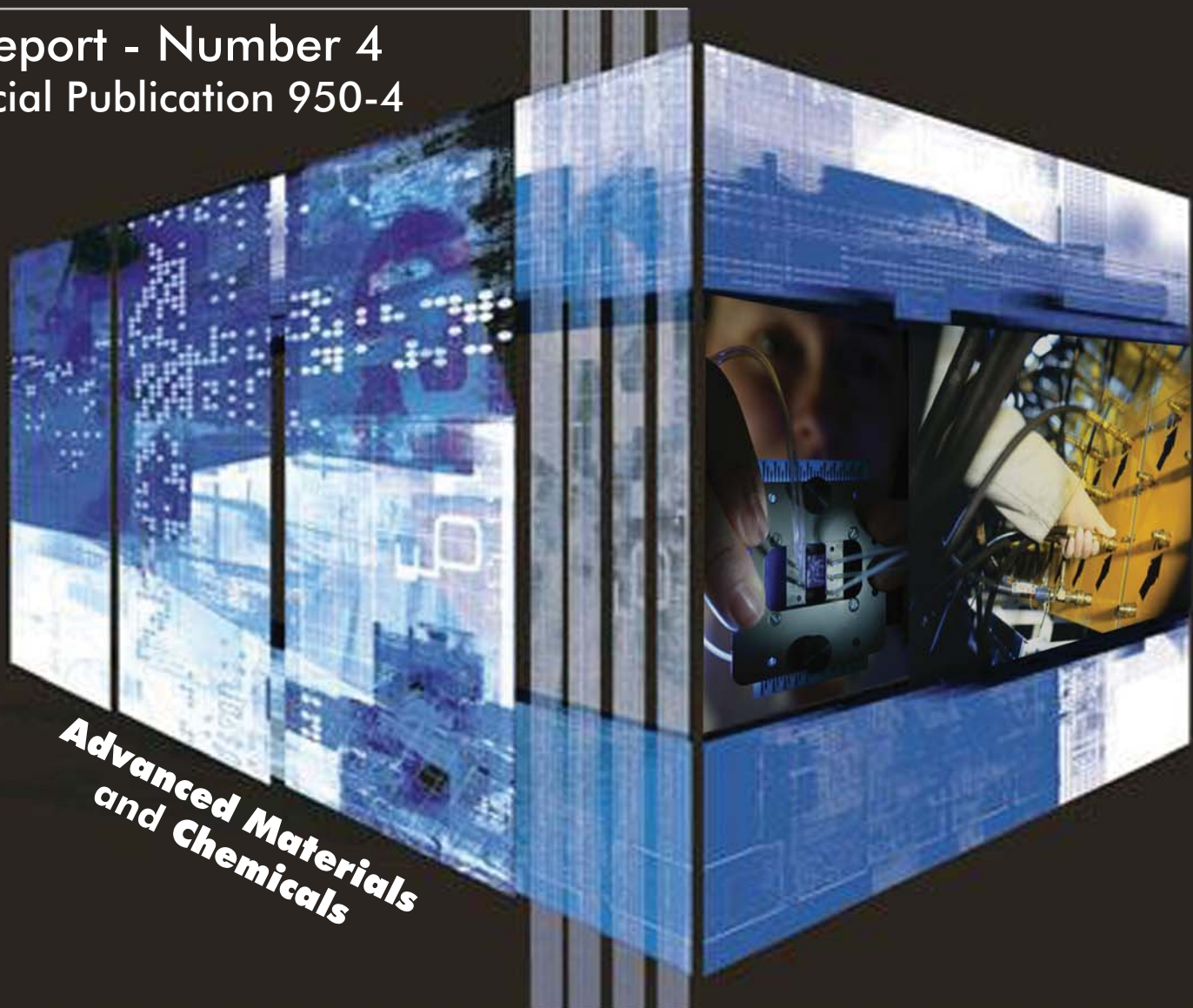


Performance of the Third 50 Completed ATP Projects

Status Report - Number 4
NIST Special Publication 950-4



**Advanced Materials
and Chemicals**

September 2006



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ACKNOWLEDGEMENTS

We are pleased to announce the completion of the next chapter in ATP's portfolio of status reports for completed projects. This compilation consists of the third batch of 50 "mini case studies" written to investigate the results and impacts of ATP's investment in innovative technologies. The goal of each status report is to provide the reader with a basic understanding of the technology, while also identifying any economic benefits that may have resulted from the ATP-funded project.

This process is a daunting one, requiring the efforts of many both inside and outside of ATP. The majority of the project was made possible by the former and current members of the status report team: Tony Colandrea, Stefanie Cox, Nashira Nicholson, Rick Rodman, Susan Stimpfle, and Virginia Wheaton. We would especially like to thank ATP Division Directors Michael Schen (Information Technology and Electronics Office) and Linda Beth Schilling (Chemistry and Life Sciences Office), and ATP Deputy Director Lorel Wisniewski, for their contributions to this project. Much appreciation also goes to the hard work of the others involved in preparing the status reports: project managers, reviewers, copy editors, and company representatives.

But these efforts aren't without rewards. As the portfolio of ATP status reports grows, we gain insight as to the role ATP plays in bridging the funding gap. We are confident this showcase of the third batch of 50 completed projects will help build on our understanding of ATP-funded innovations across many technology areas. We hope that you learn as much about the process of early-stage technology development, commercialization, and outcomes for the economy as we have in preparing these status reports.

Sincerely,

The image shows two handwritten signatures in black ink. The first signature is "Lee F. Bowes" and the second is "Stephanie Shipp".

Lee Bowes, Economist
Stephanie Shipp, Director,
Economic Assessment Office
Advanced Technology Program

INTRODUCTION

Industry has proposed 6,924 projects to the ATP since 1990, of which 768, or 11 percent, have been selected by the ATP for funding. The number of participants for these funded projects totaled 1,511, with approximately an equal number of subcontractors. This study focuses on the third group of 50 projects that were completed and provides combined statistics for all 150 completed projects studied to date.

ATP: A Partnership with Industry

The ATP attracts challenging, visionary projects with the potential to develop the technological foundations of new and improved products, processes, and even industries. The ATP partners with industry on this research, fostering collaborative efforts and sharing costs to bring down high technical risks and accelerate technology development and application. These are projects that industry in many cases will not undertake without ATP support, or will not develop in a timely manner when timing is critical in the highly competitive global market. The program funds only research, not product development. The ATP is managed by the National Institute of Standards and Technology, an agency of the Commerce Department's Technology Administration.

ATP awards are made on the basis of a rigorous competitive review, which considers the scientific and technical merit of each proposal and its potential benefits to the U.S. economy. The ATP issues a proposal preparation kit that presents and explains the selection criteria to prospective applicants and provides guidance on preparing proposals.¹ U.S. businesses conceive, plan, propose, and lead the projects. Government scientists and engineers who are expert in the relevant technology fields review all proposals for their technical merit. Business, industry, and economic experts review the proposals to judge their potential to deliver broadly based economic benefits to the nation—including large benefits extending beyond the innovator (the award recipient).

The ATP delivers benefits to the nation along two pathways: 1) a direct path by which the U.S. award recipient or innovator directly pursues commercialization of the newly developed technologies; and 2) an indirect path which relies on knowledge transfer from the innovator to others who in turn may use the knowledge for economic benefit. Either path may yield spillover benefits. The ATP looks to the direct path as a way to accelerate application of the technology by U.S. businesses. It looks to the indirect path as a means of achieving additional benefits, or benefits even if the award recipient fails to continue. The ATP's two-path approach to realizing national benefits offers advantages: one path may provide an avenue for benefits when the other does not, and both paths together may yield larger, accelerated benefits as compared to having a single route to impact.

Project Evaluation

The ATP, like other federal programs, is required by law to report on its performance.² The ATP established its evaluation program soon after it began, even before evaluation was widely required by Congress. The Economic Assessment Office (EAO) of ATP plans and coordinates the evaluation of funded projects. It is assisted in this effort by leading university and consulting economists and others experienced in evaluation.

Performance is measured against the program's legislated mission. Emphasis is placed on attempting to measure benefits that accrue not only to the direct award recipients, but also to a broader population, i.e., spillover benefits. This emphasis reflects the fact the public funding covers part of the costs of these projects, and, therefore, a relevant question is how the broader public benefits from the expenditure.

This report constitutes one element of the EAO's multi-faceted evaluation plan: status reports. The purpose of status reports is to provide an interim assessment of the status of ATP-funded projects several years after they are completed. Although the ultimate success of the ATP depends on the long-run impacts of the entire portfolio of ATP projects, the performance-to-date of this partial portfolio provides some initial answers. This study contains an evaluation of 150 completed projects: the results of the 100 projects from the Status Report – Numbers 2 & 3, and the results and status reports of a third batch of 50 projects. These reports address the questions of what has the public investment of \$321 million in the 150 projects produced several years after completion of the research, and what the outlook is for continued progress?

Study Approach

From the moment that ATP funded its first group of 11 projects in the 1990 competition, program administrators, the administration, Congress, technology policymakers, industry, and others in this country and abroad were keenly interested in the outcome. But technology development and commercialization are lengthy processes, and it takes time to produce results.

As more ATP-funded projects are completed and move into the post-project period, sufficient time has elapsed for knowledge to be disseminated and progress to be made towards commercial goals. Thus, it is now possible to compile more complete aggregate portfolio statistics, and analyze these statistics with regard to implications for overall program success.

At the core of this study are 50 mini-case studies covering each of the completed projects. Each of these briefly tells the project story, recounting its goals and challenges, describing the innovators and their respective roles, and assessing progress to date and the future outlook. Photographs illustrate many of the projects.

Although the particulars vary for each project, certain types of data are systematically collected for all of them. Consistent with ATP's mission, the evaluation focuses on collecting data related to the following dimensions of performance:

- **Knowledge creation and dissemination**, which is assessed using the following criteria: recognition by other organizations of a project's technical accomplishments; numbers of patents filed and granted; citations of patents by others; publications and presentations; collaborative relationships; and knowledge embodied in and disseminated through new products and processes.
- **Commercialization progress**, which is gauged in terms of the attraction of additional capital for continued pursuit of project goals, including resources provided by collaborative partners; entry into the market with products and services; employment changes at the small companies leading projects and other indicators of their growth; awards bestowed by other organizations for business accomplishments of project leaders; and the analyst's assessment of future outlook for the technology based on all the other information.

The approach is to provide, in an overview chapter, the aggregate statistics of interest across a set of 150 projects, such as the total number of patents and the percentage of projects whose technologies have been commercialized. In addition, the aggregate statistics are combined to produce composite project metrics for overall performance. The composite performance scores allow one to see at a glance the robustness of a project's progress towards its goals. Underlying the simple scores is a wealth of data.

Sources of Information

Data for the projects were collected from many sources: ATP project records; telephone interviews with company representatives; interviews with ATP project managers; company websites; the U.S. Patent and Trademark Office; in-depth project studies conducted by other analysts; academic, trade and business literature; news reports; filings at the Securities and Exchange Commission; and business research services, such as Dun and Bradstreet, Hoover's Online, Industry Network, and CorpTech. Each one of the individual project write-ups was reviewed for accuracy by the project's lead company and ATP staff.

Study Limitations and Future Directions

Since developments continue to unfold for most of these projects, the output measures for the cases may have changed significantly since the data were collected. The cases provide a snapshot of progress several years after the completion of the ATP-funded projects.

Although undertaken at different calendar dates, the reports are written within about the same interval of time after ATP funding ended. Yet, different points in each technology's life cycle may be captured, depending on the technology area. Information technology projects, for example, may be expected to be further along than advanced materials and chemical projects. Examined at a later time, there may be less (or more) difference in the accomplishments among projects in different technology areas.

This study tracks outputs leading to knowledge dissemination but it does not assess the actual commercialization efforts by others who acquire the knowledge. The tracking of commercialization efforts is limited to the direct path of impact (i.e., commercialization by the award recipients or innovators).

“Completed” and “Terminated” Projects Defined

Projects do not necessarily finish in the order funded. For one thing, they have different lengths, ranging from approximately two years to no more than five years. For another, they are required to file a final report with the ATP and have financial and other paperwork completed before project closeout. The financial closeout is done through the National Institute for Standards and Technology (NIST) Grants Office, which notifies the ATP that it considers the project completed. This study assesses the first 150 projects the Grants Office declared “completed.”

Not all ATP projects reach completion; some are stopped short and are classified as “terminated.” Some of these were announced as award winners but never officially started. Other projects got off the ground but were closed for various reasons with a substantial amount of the technical work still unfinished. These terminated projects are assessed according to the principal reasons they stopped before completion. They are treated in Appendix B. While the terminated projects are generally regarded as unsuccessful, some produced potentially useful outputs.

Report Organization

The report has been divided into separate technology area “editions” in order to provide a smaller, more targeted compilation. However, the overview still provides a summary overview of the performance of the 150 completed projects as a group. It identifies some major outputs that appear useful as indicators of the degree of project success, and it uses these outputs in a prototype project performance rating system. A preview also notes some of the broad-based benefits that this portfolio of projects is producing and likely to produce. For additional background, the make-up of the portfolio of projects in terms of technologies, organizational structure, company size, and other features is provided.

The individual project reports, within the particular technology area, follow the overview. The reports highlight major accomplishments and the outlook for continued progress. A detailed account of the project under review is given, with attention to technical and commercial goals and achievements, information about technology diffusion, and views about the role played by ATP funding. A performance rating is assigned to each project based on a four-star scoring system. The rating depends on the accomplishments of the project in creating and disseminating new scientific and technical knowledge and in making progress toward generating commercial benefits, as well as the outlook for continued progress.

Three appendices provide supporting information. Appendix A provides a listing of technical and commercial achievements of each completed project. Appendix B provides a discussion of the terminated projects throughout ATP's existence. Appendix C provides a list of the first 150 completed projects and the respective composite performance ratings. The listed is sorted in descending order of performance rating, then by company name.

1. The current edition of the kit and other program materials may be obtained on ATP's website (www.atp.nist.gov), by e-mail (atp@nist.gov), by phone (1-800-ATP-Fund or 1-800-287-3863), or by mail (ATP, NIST, 100 Bureau Drive, Stop 4701, Gaithersburg, MD 20899-4701).
2. The Government Performance and Results Act (GPRA) is a legislative framework for requiring federal agencies to set strategic goals, measure performance, and report on the degree to which goals are met. An overview of the GPRA is provided in Appendix 1 of the General Accounting Office Executive Guide, Effectively Implementing the Government Performance and Results Act, GAO, Washington, D.C., GGD-96-118, 1996

Overview of Completed Projects

PART 1

Project Characteristics

This report provides an overview of the first 150 ATP-funded projects to reach completion. These projects reflect an investment of more than \$621 million that was shared about equally by ATP and industry.

Of the initial 150 projects, 75 were led by small businesses that submitted single-company-applicant proposals to ATP. Eighty-seven percent involved collaborative relationships with other firms, universities, or both. Sixty-seven percent were funded in ATP's General Competitions.

In terms of classification by type, 25 percent of the projects were "Electronics, Computer Hardware, or Communications", while "Advanced Materials and Chemicals" accounted for 23 percent. "Manufacturing", "Information Technology", and "Biotechnology" each constituted about 17 percent of the remaining projects.

(The 150 completed status reports discussed in this chapter can be found online at <http://www.atp.nist.gov/> under funded projects.)

Single Applicants and Joint Ventures

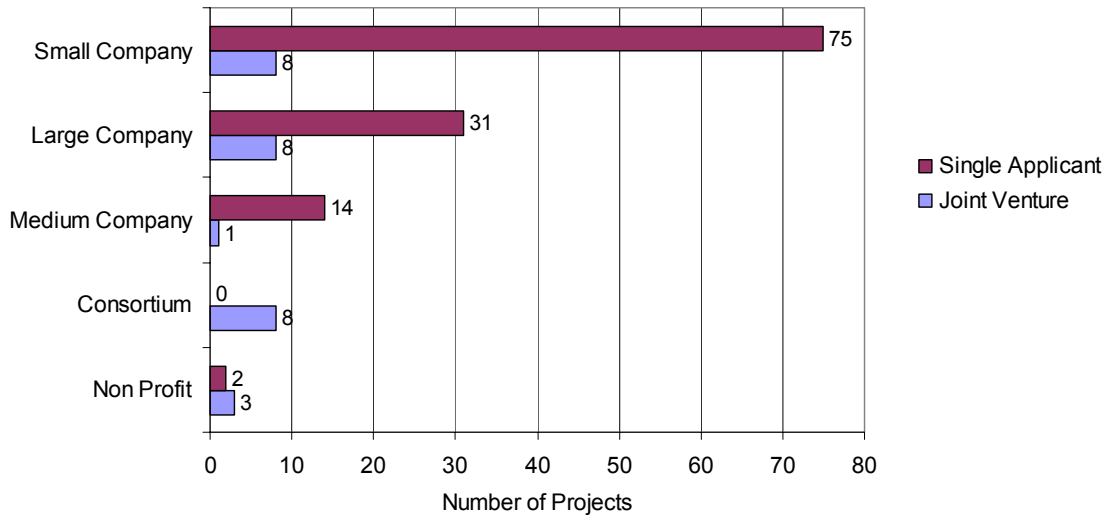
"Single-applicant projects," make up 81 percent of the first 150 ATP-funded projects; these projects were subject to an upper limit on ATP funding of \$2 million and a time limit of 3 years. Nineteen percent of the 150 projects were joint ventures. Each of these projects had a minimum of two for-profit companies sharing research and costs for up to 5 years. Typically, the joint-venture membership included other for-profit companies, universities, and nonprofit laboratories. These projects, free of the funding constraint, tended to take on larger problems for longer periods of time.

Project Leaders

Figure 1-1 illustrates how project leadership of single-applicant and joint-venture projects was distributed among the various types of organizations. Small companies led most of the projects—75 of the 122 single-applicant projects and 8 of the 28 joint-venture projects. "Small" follows the Small Business Administration's definition and includes companies with fewer than 500 employees. Large companies—defined as Fortune 500 or

equivalent firms—led 31 of the single-applicant projects, or 25 percent, and eight of the joint ventures, or 29 percent. Medium-sized companies led only 14 single-applicant projects and one joint venture. Consortia led eight of the joint venture projects. Nonprofit institutions led two of the single-applicant projects¹, and three joint ventures.

Figure 1-1
Number of Single-Applicant and Joint-Venture Projects by
Type of Leadership



Source: Advanced Technology Program First 150 Status Reports

A Variety of Technologies

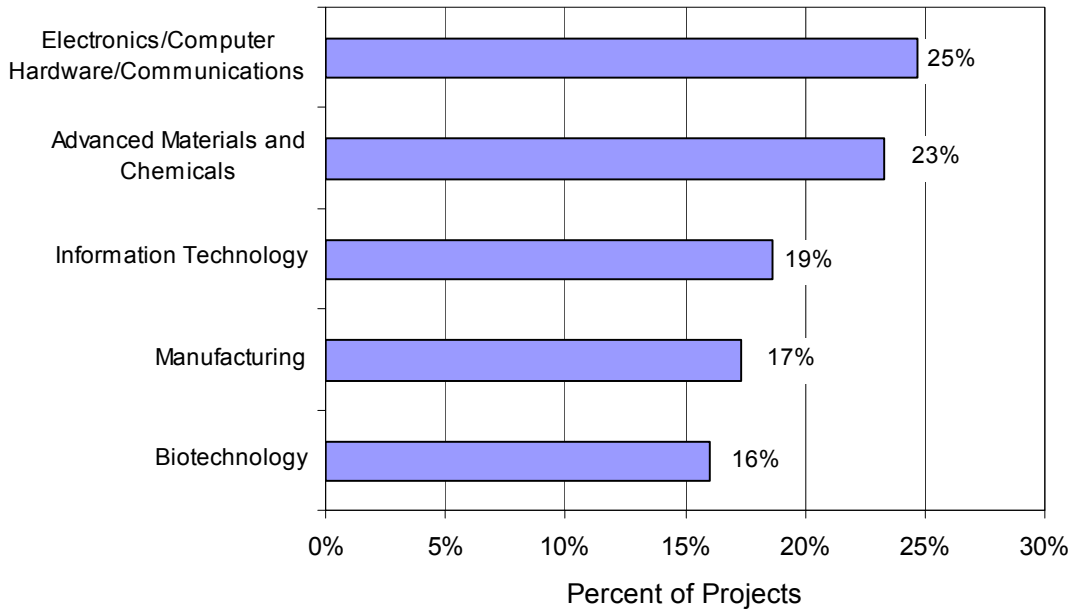
The 150 completed projects fall into the five technology areas used by ATP for classification purposes. Figure 1-2 shows the percentages of completed projects by technology area. The highest concentration, accounting for 25 percent of the total, is in “Electronics, Computer Hardware, or Communications.” This category includes microelectromechanical technology, microelectronic fabrication technology, optics and photonics, and other electronics projects.

“Advanced Materials and Chemicals” account for 23 percent of the projects. “Information Technology,” “Manufacturing,” and “Biotechnology” account for, 19, 17 and 16 percent respectively of the 150 projects. The Manufacturing category includes areas such as energy conversion and energy generation and distribution, in addition to machine tools, materials handling, intelligent control, and other discrete manufacturing. The Advanced Materials and Chemicals category includes the subcategories of energy resources/petroleum, energy storage/fuel cell, battery, environmental technologies, separation technology, catalysis/biocatalysis, and other continuous manufacturing technologies, as well as metals and alloys, polymers, building/construction materials, and

¹ From the 1991 competition, when nonprofits were eligible to lead ATP projects.

other materials. The category of Biotechnology includes areas such as bioinformatics, diagnostic and therapeutic, and animal and plant biotechnology.

Figure 1-2
Distribution of Projects by Technology Area



Source: Advanced Technology Program First 150 Status Reports

The technology make-up of these 150 projects differs from that of the larger ATP portfolio of projects in part because the composition of ATP applicants and awardees over time changes. Of the first 150 completed projects, 67 percent come from ATP's General Competitions that were open to all technologies, while 33 percent come from ATP's focused program competitions, which were held from 1994 through 1998. These competitions funded technologies in selected areas of focus, such as in Motor Vehicle Manufacturing Technology and Digital Video in Information Networks.

It should be noted that while the five major technology areas are used to classify the projects, most of them are not easy to classify. Most ATP projects involve a mix of technologies and interdisciplinary know-how.

Collaborative Activity

Although only 19 percent of the 150 projects were joint ventures, 87 percent of all projects had collaborative arrangements. As shown in Table 1-1, 49 percent of the projects involved close research and development (R&D) ties with universities. Sixty-one percent reported collaborating on R&D with companies or other nonuniversity organizations. Slightly less than half the projects formed collaborative relationships with other organizations for commercial pursuit of their ATP-funded technologies. Thirty-five

percent of projects had collaborative relationships with both universities and nonuniversities for either R&D or commercial purposes.

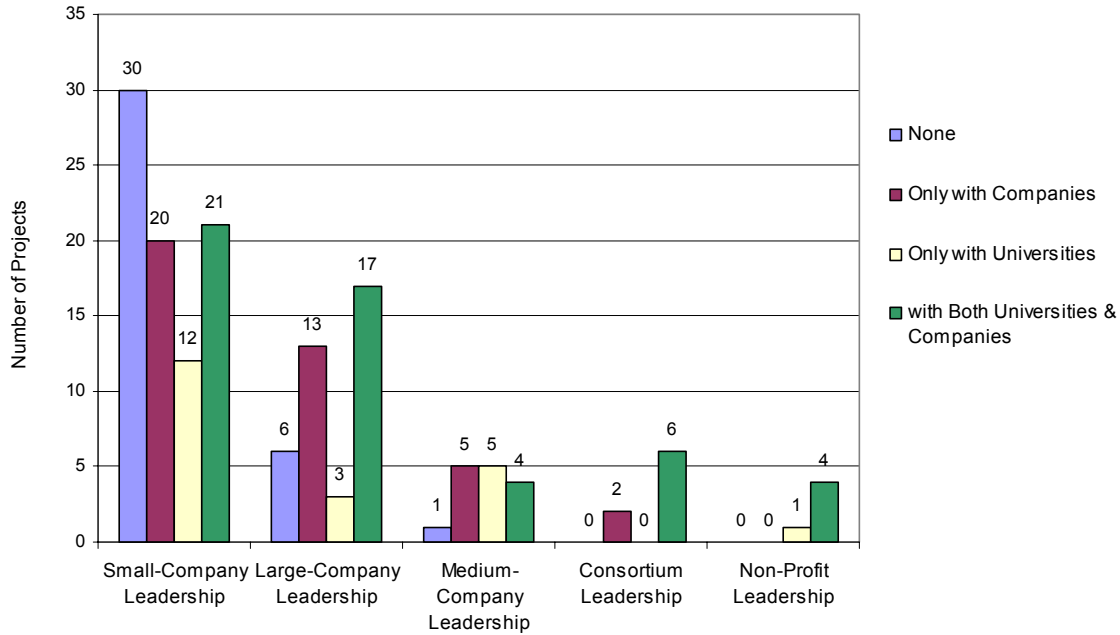
**Table 1-1
Collaborative Activity**

Type of Collaboration		Percentage
A)	Collaborating on R&D with other companies or nonuniversity organizations	61%
B)	Close R&D ties with universities	49%
	Collaborating on R&D with other companies or nonuniversity organizations OR close R&D ties with universities (A or B)	75%
	Collaborating with both universities and non-university organizations (A and B)	35%
C)	Collaborating on commercialization with other organizations	46%
	Collaborating in one or more of the above ways	87%
Note: This assessment of collaborative relationships likely understates the numbers because it focused on the project's lead organization and probably missed some of the informal collaborative relationships of other participants.		

Source: Advanced Technology Program First 150 Status Reports

For more detail, Figure 1-3 illustrates the types of collaboration undertaken by projects with different forms of project leadership. It highlights the fact that under all forms of project leadership, projects were highly likely to involve collaboration with other companies. About 43 percent of the projects led by small and large companies involved university collaboration, while the share rose to 60 percent for projects led by medium-sized companies, and 75 percent for consortium-led projects.

Figure 1-3
Number of Projects with R&D Collaborations by Type of Collaboration
and Type of Project Leadership



Source: Advanced Technology Program First 150 Status Reports

Costs of the Projects

As shown in Table 1-2, ATP and industry together invested in excess of \$621 million on the 150 projects. They shared almost equally in project costs, with ATP providing a slightly larger share. ATP spent an average of \$1.72 million per single-applicant project and an average of \$3.97 million per joint-venture project. Across the 150 projects, the average total cost (ATP plus industry) per project was \$4.14 million. Estimated benefits attributed to ATP from just a few of the 150 projects for which quantitative economic benefits have been provided exceed ATP’s funding for all of the 150 projects. In addition, there is considerable evidence of large project benefits that have not yet been quantified.

Approximately 45 percent of single-applicant projects had total research costs under \$3 million. These projects had an ATP share that ranged from a little more than \$.5 million to \$2 million. Slightly less than 50 percent had total research costs greater than \$5 million, and one project had total research costs greater than \$30 million. ATP’s share of these costs were \$2 million or more for 50 percent of the projects and were \$5 million or higher for 36 percent. For one of the projects, ATP’s share exceeded \$10 million. Joint ventures, which made up only 19 percent of the total number of projects, accounted for 35 percent of total ATP funding.

Table 1-2
ATP Funding, Industry Cost Share, and Total Costs of 150 Completed Projects

	Single Applicant Projects	Joint Venture Projects	Total Projects
ATP Funding (\$ Millions)	210.1	111.1	321.2
Industry Cost Share (\$ Millions)	184.6	115.2	299.8
Total Project Costs (\$ Millions)	394.7	226.3	621.0
ATP Share of Costs	53%	49%	52%
Industry Share of Costs	47%	51%	48%
Average Project Funding Provided by ATP (\$ Millions)	1.72	3.97	2.14
Average Project Cost-Share Provided by Industry (\$ Millions)	1.51	4.11	2.00
Average Project Funding Provided by Overall (\$ Millions)	3.24	8.08	4.14

Source: Advanced Technology Program First 150 Status Reports

PART 2

Gains in Technical Knowledge

One of ATP's major goals is to build the nation's scientific and technical knowledge base. Each of the 150 completed ATP projects targeted a number of specific technical goals designed to achieve a new or better way of doing things. The knowledge created by each project is the source of its future economic benefit, both for the innovator and for others who acquire the knowledge. It is a good starting place for assessing completed projects.

(The 150 completed status reports discussed in this chapter can be found online at <http://www.atp.nist.gov/> under funded projects.)

New Technologies and Knowledge Gains

Knowledge gains by the projects are diverse and encompass the five major technology areas. The technologies developed in the 150 projects are listed in column C in Tables A-1–A-5 in Appendix A. The set of tables provides the reader with a convenient, quick reference to the entire range of technologies. The entries are arranged alphabetically, by project lead company using the five technology areas. As was mentioned earlier, most of these projects are interdisciplinary, involving a mixture of technologies and generating knowledge in multiple fields.

Even those projects that were not fully successful in achieving all of their research goals, or those that have not been followed by strong progress in commercialization, have achieved knowledge gains. Moreover, some of the projects carried out by companies that have since ceased operations or stopped work in the technology area yielded knowledge, as indicated primarily by the presence of publications and patents. In these cases the direct market routes of diffusion of knowledge gains through commercialization by the innovators are likely lost. However, the indirect routes—whereby others acquire and use the knowledge—remain.

Of What Significance Are the Technical Advances?

Measuring the significance of technical advances is challenging. One factor that challenges measurement is the length of elapsed time that typically separates an R&D investment and its resulting long-term outcomes. In the interim period, various short-run metrics may serve as indicators that project results appear to be on track toward achieving long-term goals. One metric that has been used to signal the significance of a project's technical achievements is formal recognition in the form of an award from a third-party organization.

Thirty awards for technical accomplishments were made to participants for achievements related to ATP-funded projects. Participants in 19 of the 150 projects received awards for their technical achievements. Participants in seven of the projects received multiple technical awards. Table 2-1 lists the awards made to these projects by third-party organizations in recognition of their technical accomplishments.

**Table 2-1
Outside Recognition of Technical Achievements of the First 150 Completed Projects**

Project Awardee	Year	Awarding Organization	Award
American Superconductor	1996	<i>Industry Week Magazine</i>	Technology of the Year award
American Superconductor	1996	<i>R&D Magazine</i>	One of the 100 most important innovations of the year
Automotive Composites Consortium (a Partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)	1999	<i>Popular Science Magazine</i>	Best of What's New for the Chevrolet Silverado composite truck box, "a breakthrough in the use of structural composites"
Cincinnati Lamb, UNOVA (Lamb Technicon)	1999	<i>Industry Week Magazine</i>	Top 25 Technology and Innovation Award
Communication Intelligence #1	1997	Arthritis Foundation	"Ease-of-Use Seal of Commendation" for the development of natural handwriting technology, for use by disabled people who have trouble with keyboard entry
DuPont	1993	<i>Microwave & Rf Magazine</i>	One of the Top Products of 1993, for high-temperature superconductivity component technology
Ebert Composites	1999	Civil Engineering Research Foundation	Charles Pankow Award for Innovation in Civil Engineering
Engineering Animation	1994	<i>Computerworld Magazine</i>	Smithsonian Award, for the use of information technology in the field of medicine
Engineering Animation	1995	Association of Medical Illustrators	Association of Medical Illustrators Award of Excellence in Animation
Engineering Animation	1995	International ANNIE Awards	Finalist, received together with Walt Disney, for best animations in the film industry
Engineering Animation	1996	<i>Industry Week Magazine</i>	One of the 25 Technologies of the Year, for interactive 3D visualization and dynamics software used for product development
GM Thermoplastic Engineering Design (Engineering Design with Thermoplastics)	2001	Internal GM R&D Award	Campbell Award for "Process Modeling and Performance Predictions of Injection-Molded Polymers"
GM Thermoplastic Engineering Design (Engineering Design with Thermoplastics)	2001	Society of Plastics Engineers	Best Paper Award from the Product Design and Development Division

Project Awardee	Year	Awarding Organization	Award
HelpMate Robotics	1996	<i>Discover Magazine</i>	One of 36 finalists for Technology of the Year, for the HelpMate robot used in hospitals
HelpMate Robotics	1997	Science Technology Foundation of Japan	Japan Prize, to CEO Joseph Engelberger, for "systems engineering for an artifactual environment"
Illinois Superconductor	1996	<i>Microwave & Rf Magazine</i>	One of the Top Products of 1996, for cellular phone site filters and superconducting ceramics
Illinois Superconductor	1997	American Ceramic Society	Corporate Technical Achievement Award
Integra Life Sciences**	1999	New Jersey Research and Development Council	Thomas Alvin Edison Award
Kopin Corporation	1998	<i>Electronic Products Magazine</i>	"Product of the Year" Award for expanding functionality of portable devices including PDAs, cell phones, and pagers
Kopin Corporation	1998	<i>IndustryWeek Magazine</i>	"25 Technologies of the Year" Award
Kopin Corporation	1999	<i>Photonics Spectra Magazine</i>	"25 Most Technically Innovative Products" Award for the CyberDisplay 320C
Kopin Corporation	2003	Consumer Electronics Show 2003	"Best Innovation" Award for the 44-inch LCoS HDTV
Molecular Simulations	1996	Computerworld Magazine	Finalist for Smithsonian Award, the 1996 Innovator Medal
NCMS	1994	Institute for Interconnecting & Packaging Electronics Circuits	Best Paper of Conference Awards
Perceptron (formerly Autospect, Inc.)	1998	International Body Engineering Conference	Best Paper Award
Strongwell Corporation	1998	Composite Fabricators Association Conference	Best of Show Award
The Dow Chemical Company	2004	Department of Commerce, NIST/Brookhaven	Gold Medal for Scientific/Engineering Achievement for Dr. Daniel Fischer's work on "a unique national measurement facility for soft X-ray absorption spectroscopy enabling breakthrough materials advances"
Xerox Palo Alto Research Center	2003	<i>JavaWorld</i>	Editors' Choice Award for the Most Innovative Java Product or Technology
X-Ray Optical Systems (XOS)	1995	<i>R&D Magazine</i>	R&D top 100
X-Ray Optical Systems (XOS)	1996	<i>Photonics Spectra Magazine</i>	Photonics Circle of Excellence Award

Source: *Advanced Technology Program First 150 Status Reports*

**The award went to Dr. Kohn of Rutgers University for his collaborative work with Integra on the project.

Examples of Projects with Knowledge Gains

Xerox Palo Alto Research Center: Xerox Palo Alto Research Center (PARC) expanded its research on modularity with a cost-shared award for \$1.7 million from ATP's Component-Based Software Focus Program. The project began in 1995, and the researchers developed two prototype applications that extracted system-wide concerns into separate modules with their own code. They called this approach aspect-oriented programming (AOP).

As ATP funding ended, PARC began working with the Defense Advanced Research Projects Agency to create a general-purpose language and tool, which PARC patented and called AspectJ. This product:

- Is freely available through IBM's eclipse.org web site
- Has six trade books devoted to it
- Won the JavaWorld Editors' Choice Award for the Most Innovative Product or Technology in 2003
- Is used aggressively by IBM in developing new software products

AOP is well recognized in the computer industry and has eight patents associated with it. More than a dozen universities in North America and in the United Kingdom include it in their curricula. Although the average computer user does not know or care about aspects, programmers' use of AOP in designing web sites will bring speed, reliability, greater customization, and savings. End users receive better services, delivered more quickly, at a lower cost.

Orchid BioSciences (formerly Molecular Tool, Inc. Alpha Center): A small company, Molecular Tool, applied for and was awarded \$1.9 million under the ATP Tools for DNA Diagnostics focused program in 1995, in order to compress most of the functions of SNP analysis that were being done in the 20-foot by 15-foot biotechnology laboratory onto a 1-square-inch glass chip..

Molecular Tool successfully developed a patented prototype SNP analysis tool in 1998 and gained the attention of the biotechnology industry. Orchid BioComputer (later renamed Orchid BioSciences) purchased Molecular Tool in 1998 to acquire the ATP-funded equipment and the company's project-related knowledge.

In 2000, Orchid BioSciences was performing DNA analyses using a single nucleotide polymorphism (SNP) analysis tool, which performed more than 800,000 DNA analyses per day. Orchid's SNP scoring tool, called SNPstream, analyzes up to 100,000 data points for increased accuracy. Furthermore, a typical result showed one in several billions statistical probability, increased from one in a million. SNP technology has had high-profile applications:

- Used to attempt to identify the remains of some New York City World Trade Center victims of 2001, which could not be identified by conventional DNA analysis due to sample degradation.
- Used in assisting major metropolitan police departments in forensics, including Los Angeles, Houston, and England's Scotland Yard. Also developed advanced forensic applications to identify individuals from unsolved crimes using degraded DNA samples for the Federal Bureau of Investigation. Orchid's express DNA service provides forensic DNA analyses in five business days compared with the standard four to five weeks.
- Used for the United Kingdom's scrapie genotyping program to help sheep farmers use selective breeding to eliminate the disease scrapie from their flocks. The company has genotyped over 1 million sheep to date.

The societal benefits of SNP analyses are growing. Typical DNA analysis cost has been reduced by approximately 70 percent, and the time it takes to perform DNA analysis has been reduced by approximately 75 percent, such that DNA analysis can now provide results in about a week (reduced from 4 weeks). Police departments are able to solve cold cases, because SNPstream is able to analyze DNA from degraded samples. It is hoped that pharmacogenetic applications (studying genetic variations related to the onset of disease, and pharmaceuticals) will improve medical treatment.

SciComp: ATP provided in cost-shared funds to \$1.9 million to SciComp to develop a software synthesis technology that would simplify the process of mathematical modeling.

SciComp, Inc successfully incorporated simplified mathematical modeling (representing a mathematical device or process) into software for the derivative securities industry. Called SciFinance, this solution includes tools that can automate the pricing of complex derivative securities, organize libraries of pricing codes, and provide risk-management analysis.

As of 2004, SciFinance includes six financial products, four of which incorporate the ATP-funded synthesis technology and two that enhance the other products.

SciComp's software synthesis technology improved the productivity of mathematical modelers by tenfold. SciComp has been awarded two patents based on ATP-funded technology development, and the company has shared knowledge through nine published papers and made several presentations at conferences.

As of 2004, the volume of derivative securities trading has continued to grow, resulting in increased demand for software tools to assist in the pricing of complex derivative structures. SciComp is one of only a few companies that provide these tools.

PART 3

Dissemination of Knowledge

If knowledge from the projects is disseminated—either through products and processes commercialized by the innovators or through publications, patents, and other modes of knowledge transfer—it may benefit other producers in the economy and, subsequently, consumers. The resulting national benefits may go far beyond the returns to the innovating firms and the benefits to their customers.

(The 150 completed status reports discussed in this chapter can be found online at <http://www.atp.nist.gov/> under funded projects.)

Multiple Ways of Disseminating Knowledge

New knowledge developed in a project can be diffused in a variety of ways. This section discusses two principal means: through patents filed and granted by the U.S. Patent and Trademark Office (USPTO) and cited by others, and through preparation of technical papers that are published or are presented at conferences. Collaborative activity among research and commercial partners, treated in Part 1, is another way by which knowledge is disseminated. Another way is through the observation and reverse engineering of the new goods or services produced directly by the innovators and their partners, discussed in Part 4. Among the other important ways—not explicitly covered here—in which knowledge developed in a project can be diffused are informal interactions among researchers, suppliers, customers, and others; movement of project staff to other organizations; distribution of nonproprietary project descriptions by government funding agencies; and project-related workshops and meetings.

Pathways of knowledge dissemination allow others to obtain the benefits of R&D without having to pay its full cost. When the technology is particularly enabling—in the sense of providing radically new ways of doing things, improving the technical bases for entire industry sectors, or being useful in many diverse areas of application—the spillover benefits to others are likely to be particularly large. The generation of spillover benefits, or positive externalities, from technological advancement is an important argument for public support of enabling technologies.

Balancing Intellectual Property Protection and Knowledge Dissemination

ATP encourages broad dissemination of knowledge produced in ATP-funded projects because it increases the number of potential users of the knowledge and, therefore, may increase national benefits. At the same time, ATP does not force innovating companies to compromise their ability and willingness to pursue early commercial applications of the technology by giving away all of their intellectual property. After all, these companies,

which contribute a substantial share of the costs, have agreed to tackle difficult research barriers and to take the technology to the marketplace as rapidly as possible.

Thus, it is not surprising that the amount of knowledge dissemination varies among the projects. Most of the projects pursue some forms of deliberate knowledge dissemination, such as publishing scientific papers, giving presentations, and forming collaborative relationships. Most projects also engage in considerable unintended knowledge dissemination; for example, as a company's scientists move and work among other companies and universities; as myriad formal and informal discussions occur; as others reverse-engineer their products; and through mergers and acquisitions of the innovating companies.

Public Disclosure of Patent Filing Information

When applying for a patent to protect intellectual property, an inventor must explicitly describe the invention. Because patent law requires that the invention is both novel and useful, the inventor must demonstrate that the invention is essentially different from any other invention and must describe how it can be used. When the USPTO grants a patent, the full application text describing how the invention may be used and how it is related to other technologies is put into the public record and becomes a medium through which knowledge is transferred to others. Hence, patents serve to disseminate knowledge.

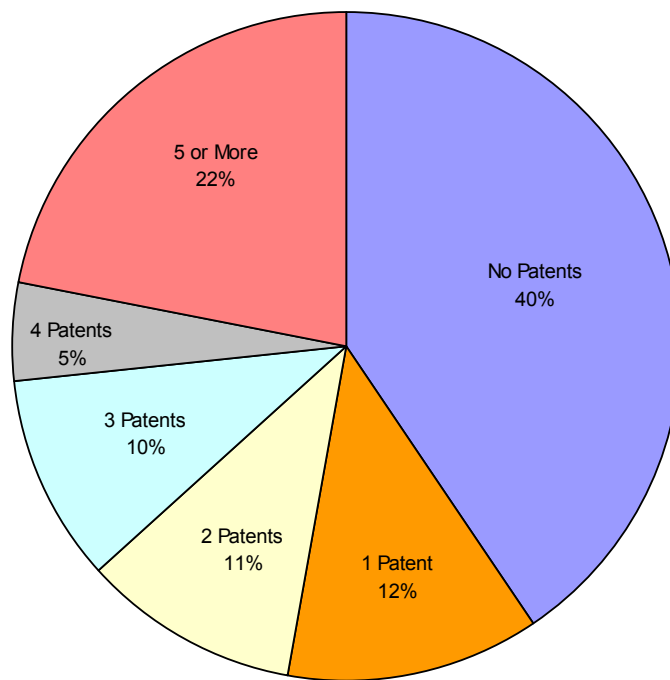
At the same time, patent data are not perfect signals of knowledge creation and dissemination. The decision to seek patent protection for intellectual property is influenced by many factors, including the ease with which others can copy the property's intellectual content and the difficulty of defending the patent position from infringement. Some companies may decide that patent protection is not worth its expense or that a strategy of trade secrets and speed-to-market is more effective. Conversely, patents may be filed as the basic ideas are forming, and trade secrets used in later stages. Furthermore, the importance of patents as a strategy varies among technology areas; for example, patents figure more strongly in electronics and manufacturing than in computer software. The absence of a patent does not mean that intellectual property was not created. But the presence of a patent is a signal that it was created. Despite the limitations, patent statistics serve as useful indicators of knowledge creation and dissemination, and they are widely used by researchers.

Of the 150 completed projects, 89 had filed 500 patents at the time the study data were collected.² Eighty-one of the projects had among them a total of 347 patents granted, or 70 percent of the total filed. Thirty-two of the projects had filed a total of 153 patents for which a final decision on granting was still pending.

² Patents filed and not yet granted are included here, in addition to those filed and granted, despite the fact that there is no public disclosure until patents are actually granted. The reason for including patents filed and not yet granted is to help offset the problem that there are substantial differences across industries in the lag time between patent filing and granting.

Figure 3-1 displays the distribution of the 150 projects by the number of patents filed, whether granted or not yet granted. More than half the projects have filed one or more patents. Participants in 12 percent of projects had filed a single patent, 26 percent had filed 2 to 4 patents each, and 22 percent had filed 5 or more patents. Forty percent of the projects had not filed a patent.

Figure 3-1
Distribution of Projects by Number of Patents Filed



Source: *Advanced Technology Program First 150 Status Reports*

Knowledge Disseminated by Patents as Revealed by Patent Trees

Each published patent contains a list of previous patents and scholarly papers that establish the prior art as it relates to the invention. The citations provide a way to track the spread of technical knowledge through patents granted to ATP-funded projects. By following the trail of the patent referenced, it is possible to construct what looks much like a horizontal genealogy tree.

Once the pool of ATP-related patents was identified, computerized tools made available by the USPTO were used to track subsequent patents that refer to each of the ATP-related patents as prior art and the links recorded.³ The process is then repeated in turn for each of these patents, until the chain of references is complete. Next, the information is

³ The references to prior patents contained in a published patent are based on information supplied by the applicant and on research by USPTO researchers. There is no way to distinguish between the two sources and no indication that one tends to dominate the other. (USPTO telephone interview with ATP staff, February 11, 2000.)

converted into a graphic format that illustrates the diffusion of knowledge along the path from ATP project patents in the tree.

With the passage of additional time, new branches may emerge as outgrowths of earlier patents. To the extent that later patents are dependent on the earlier ones, the patents in the tree represent developments in knowledge that would not have occurred, or at least not in the same timeframe, had ATP not stimulated the creation and dissemination of that platform knowledge.

Patent Tree Illustrating Knowledge Dissemination

The patent tree in Figure 3-2 shows citations of a patent that came out of an ATP-funded project led by **Texas Instruments, Inc.** during which the company developed a special insulating material, known as aerogel, to overcome problems with interconnect delays as a result of the continuing trend toward miniaturization. The company overcame impediments to aerogel processing early in the project, but in 1997, an industry competitor announced that it would begin using copper interconnect wiring in future integrated circuit designs. After the ATP-funded project Texas Instruments shifted focus away from aerogels for aluminum and began to develop copper interconnects.

The patent tree illustrates how an ATP-funded project whose direct path appears to have slowed or has come to a standstill nevertheless has the potential to remain influential along an indirect path of knowledge utilized and cited in subsequent patents. As the patent tree illustrates, a number of other companies are referencing the Texas Instrument patent, and the potential for beneficial impact from the research continues.

Figure 3-2
Patent Tree for Texas Instruments, Inc. - Patent 5,894,173
Project Impact After Innovator Reduced Activity

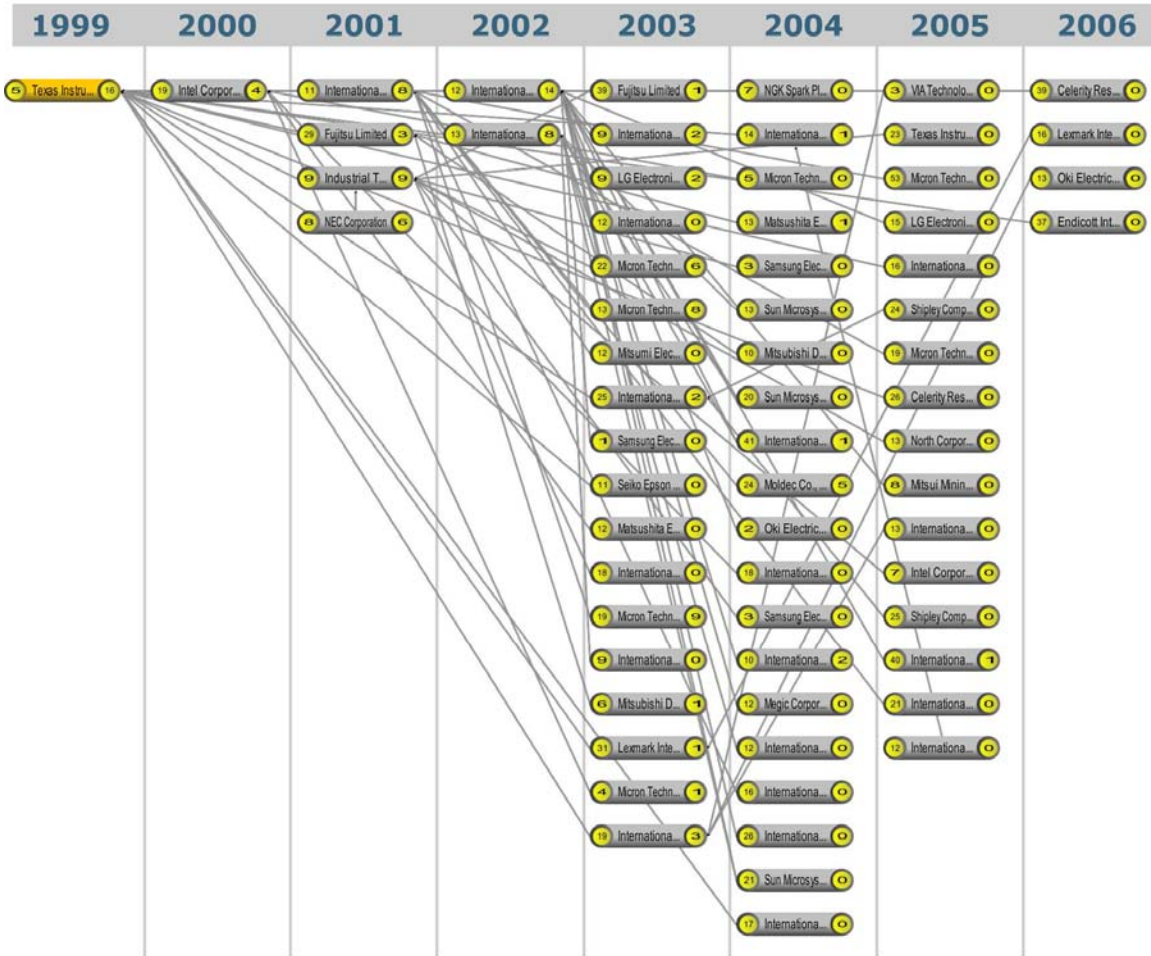


Figure 3-3
Patent Tree for Large Scale Biology Corporation - Patent 5,993,627
Project Impact Where Innovator Went Bankrupt

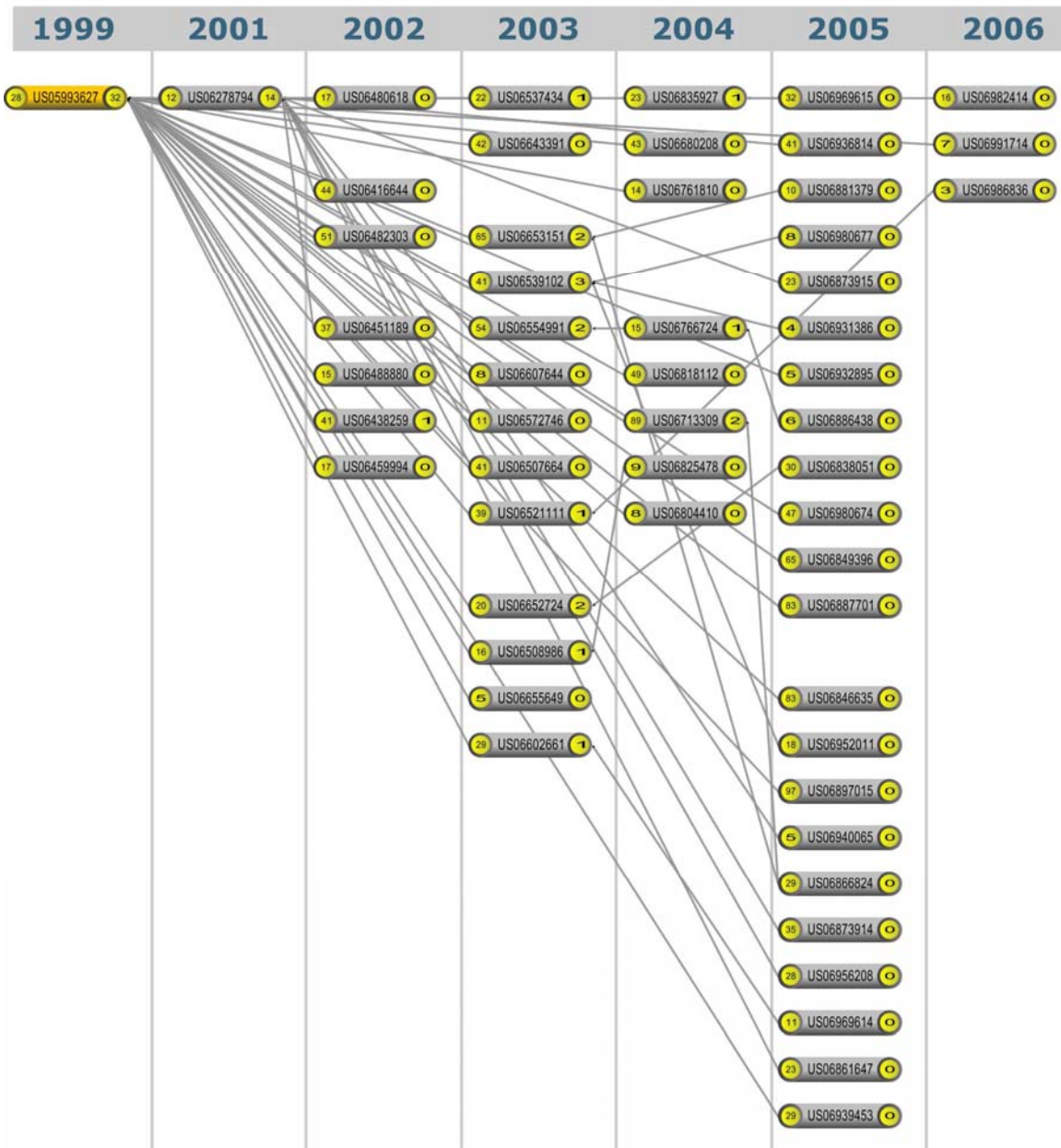


Figure 3-3 shows citations of a patent resulting from a project led by **Large Scale Biology Corporation**. Though the company went bankrupt, the patent tree illustrates how knowledge can outlive its creator and continue to be disseminated. An observer who equates business success of the innovator, one-to-one, with ATP project success may be mistaken, because the indirect path may nevertheless produce important benefits.

Patent Tree Illustrating Extensive Knowledge Flows

Figure 3-4 illustrates just how complex knowledge dissemination through patent citations can become. The path shown is for a patent resulting from an ATP-funded project led by **JDS Uniphase (formerly SDL, Inc.)** and **Xerox Corporation**. With the ATP award, the research team successfully developed high-performance, multibeam red laser diodes; two alternative methods for monolithic integrations of red, infrared, and blue emitters; and several valuable intermediary technologies. From these successes, the ATP-funded project built a strong U.S. technology base for multiple laser applications. Eighty-four inventions from this project have been commercialized into numerous products. This single Xerox patent resulted in approximately 110 citations.

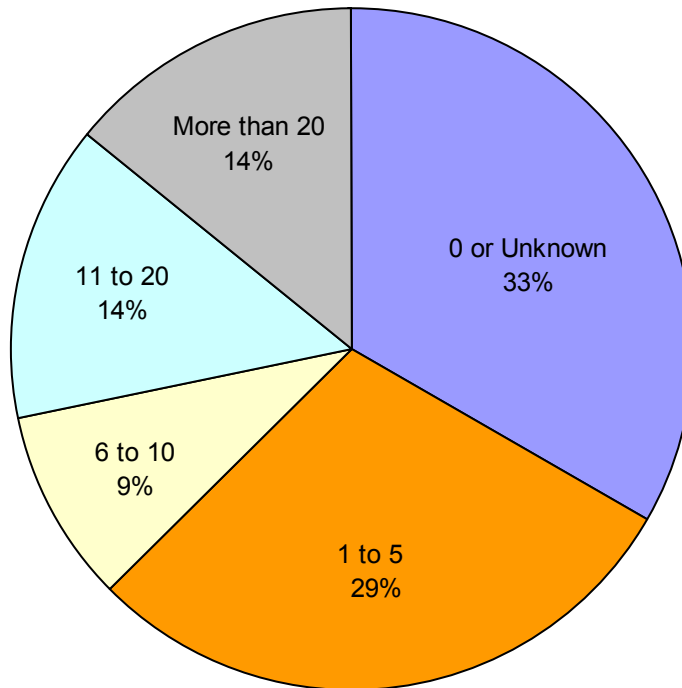
For projects that have received a patent or patents, access to patent trees is available through the individual status reports on the NIST ATP website (http://statusreports-atp.nist.gov/basic_form.asp). Although representing only one aspect of knowledge dissemination, the patent trees extend awareness of the influence of the new knowledge.

Knowledge Dissemination through Publications and Presentations

Participants in almost 66 percent of the 150 projects had published or had presented papers in technical and professional journals or in public forums. Participants in more than half of all projects had published, and the number of publications totaled at least 831 papers. Participants in nearly 47 percent of the projects had given project-related presentations, and the number of presentations totaled at least 739. Overall, publications and presentations for these 150 projects equaled or exceeded 1570.

Figure 3-5 gives the distribution of projects by their numbers of publications and presentations. Twenty-nine percent of the projects each had between one and five papers published or presented. Nine percent had between 6 and 10 papers published or presented, and another 14 percent had between 11 and 20. At the high end, 14 percent of projects each had more than 20 papers published or presented. Thirty-three percent had no known presentations or publications.

Figure 3-5
Distribution of Projects by Number of Publications and Presentations



Source: Advanced Technology Program First 150 Status Reports

Knowledge Dissemination through Other Means

Aside from publishing, presenting, and patenting, ATP-funded projects have a high rate of collaborative activities. Eighty-seven percent of the projects showed some type of collaboration (see Table 1-1). With so many partners, collaborators, and subcontractors involved, it would be difficult to secure the information. The involvement of so many participants in the projects provides rich avenues of further interaction, and those interactions in turn may increase knowledge flows through personal and professional contacts.

When the government enters into an agreement with an organization, certain information about the agreement is generally made public. Such is the case with ATP and company cost-sharing partnerships. Nonproprietary information has been disclosed to the public for each of the 768 projects funded by ATP in 44 competitions held from 1990 through September 2004 (project information is available on the ATP website⁴). Further, new nonproprietary project descriptions are added to the site as new awards are made. Evaluation reports, such as this one, are also available at ATP's website and provide information to the public.

⁴ <http://jazz.nist.gov/atpcf/prjbriefs/listmaker.cfm> or <http://atp.nist.gov> (go to Funded Projects Database).

PART 4

Commercialization of the New Technology

New technical knowledge must be used if economic benefits are going to accrue to the nation. This generally means that a new product or process is introduced into the market by the innovating firm, its collaborators, or other companies that acquire the knowledge. In competitive markets, the producer is typically unable to capture all the benefits of a new product or process, and the consumer reaps part of the benefits. The higher up the supply chain the innovation occurs, the more value-added steps there are before final consumption, and the more intermediate firms in the supply chain may benefit, in addition to the final consumer.⁵

(The 150 completed status reports discussed in this chapter can be found online at <http://www.atp.nist.gov/> under funded projects.)

Commercialization of Products and Processes—A Critical Step Toward National Benefits

When a product or service incorporating new technology reaches the marketplace, a buyer can learn a great deal about the technology. The mere functioning of a new product reveals some information. Intentional investigation, including reverse engineering, reveals even more. More than 60 percent of the 150 projects reviewed for this study had some commercial products or processes based on ATP-funded technology already on the market. Therefore, product use and examination are providing others with information about the new technologies.

Ninety-one of the projects had already spawned or expected to bring to market 222 new products or processes when the data for this report were collected. Companies in 18 additional projects expected to achieve their first commercialized results shortly⁶, and

⁵ For a detailed treatment of the relationship between spillover benefits (knowledge, market, and network spillovers) and commercialization, see Adam B. Jaffe, *Economic Analyses of Research Spillovers: Implications for the Advanced Technology Program*, GCR 96-708, (Gaithersburg, MD: National Institute of Standards and Technology, December 1996). He notes: “Market spillovers will not be realized unless the innovation is commercialized successfully. Market spillovers accrue to the customers that use the innovative product; they will not come to pass if a technically successful effort does not lead to successful commercialization” (p. 12). In commenting on spillovers that occur because new knowledge is disseminated to others outside the inventing firm, he observes: “Note that even in the case of knowledge spillovers, the social return is created by the commercial use of a new process or product, and the profits and consumer benefits thereby created” (p. 15).

⁶ “Shortly” refers to the time when the question is asked. Since Status Reports are written about 5 years after ATP funding ends, the perspective is the same for all status reports. So, when a company answers that

companies in 17 projects that had already commercialized their technology expected to add new products and processes soon. Thus, 73 percent of the projects had spawned one or more products or processes in the market or were expected to do so shortly, for a total of 245 products or processes either on the market or expected shortly after the time the data were collected. Table 4-1 summarizes the commercialization results.

**Table 4-1
Progress of Participating Companies in Commercializing the
New Technologies**

Degree of Progress	Number of Projects	Number of Products/Processes
Project has resulted in at least one Product/Process on the market AND additional Products/Processes are expected soon	17	63
Project has resulted in at least one Product/Process on the market, but no additional Products/Processes are expected soon	74	159
Project is expected to result in a Product/Process on the market soon, but no Product/Process is currently on the market.	18	23
Total Projects that have resulted in Products/Processes on the market OR are expecting to have Products/Processes on the Market soon.	109	245

Source: Advanced Technology Program First 150 Status Reports

A number of additional years have passed since the data for the first 150 projects were collected. Since that time, further developments have doubtless occurred with these projects, which have changed their commercialization results. This overview reports commercial progress of the first 150 projects, all at approximately comparable times following their completion.

A Quick Glance at the New Products

A variety of new products and processes resulted from the projects. For a convenient, quick reference, brief descriptions of the new products or processes for each project are listed in column D in Tables A-1–A-5 in Appendix A. For each new product or process, the new technology on which it is based is also listed in the tables, in column C.

they expect a product or process on the market soon or shortly, they are referring to new product commercialization in the next 3 to 12 months.

Commercialization: A Critical Step, but Not the Final Word

Commercializing a technology is necessary to achieve economic benefit, but it does not ensure that the project is a full success from the perspective of either the company or ATP. Widespread diffusion of the technology may or may not ultimately follow the initial commercialization. Nevertheless, it is significant that these products and processes are actually on the market.

Rapidly Growing Companies

Rapid growth often signals that a small innovating company is on the path to taking its technology into the market, and one dimension of company growth typically is its employment gains.⁷

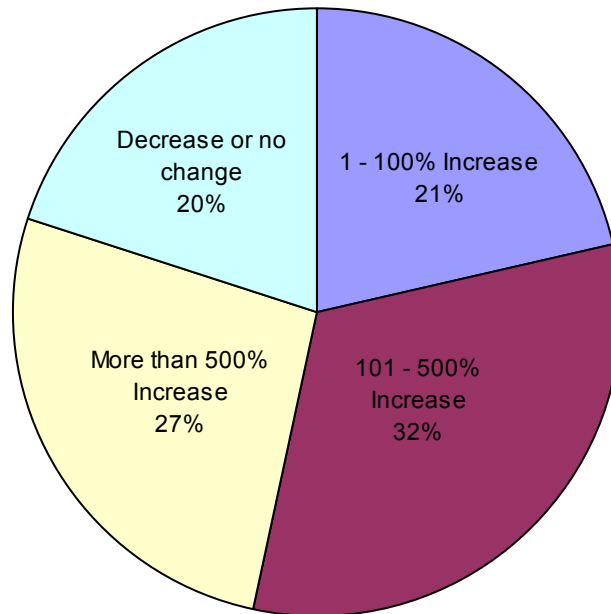
Figure 4-1 shows employment changes for the 75 small-company, single-applicant ATP award recipients.⁸ Twenty-seven percent of these companies experienced job growth in excess of 500 percent from the beginning of the project until several years after the project had completed. Thirty-two percent—the largest share—experienced job growth in excess of 100 percent, ranging up to 500 percent. Mergers and acquisitions accounted for 20 percent, or nine of the 45 projects that experienced substantial job growth (substantial job growth being in excess of 100 percent).

Not all the small companies grew. A little more than one-quarter of them experienced no change or a decrease in staff. Several of the companies that were small when they applied to ATP grew so rapidly they moved out of the small-size category. As a group, of the 75 small single-applicant companies, 45 companies at least doubled in size; 14 of them grew more than 1,000 percent. ATP helped these companies develop advanced capabilities, which they subsequently leveraged into major business endeavors.

⁷ Employment within the small companies is considered as an indicator of commercial progress. Assessing macroeconomic employment gains from the technological progress stimulated by the 150 projects is beyond the scope of this report.

⁸ Employment changes in joint ventures, larger companies, and nonprofit organizations are less closely tied to the success of individual research projects, and, therefore, are not included in the employment data in Figure 4-1.

Figure 4-1
Employment Change at Small Companies that Received a
Single-Applicant Award



Source: Advanced Technology Program First 150 Status Reports

The following examples illustrate the potential impact of ATP funding on the employment growth of funded companies.

Incyte Corporation grew from 4 to 215 employees due to the development of flexible techniques for manufacturing chem-jet-based microarrays. The technique synthesizes large arrays of specific DNA fragments suitable for medical diagnosis, microbial detection and DNA sequencing, and for creating supplies of detachable oligonucleotides for subsequent use. (Project number 94-05-0019)

Nanophase Technology increased employment from 2 employees at the start of the ATP project to 61 employees at the time the status report was written. The employment is a result of Nanophase's development of a technology that enabled a 25,000-fold increase in the development of nanoscale materials and a 20,000-fold reduction in cost. (Project number 91-01-0041)

Capital Attraction

Attraction of additional capital is another signal that a company is positioned to make further progress. Of the 150 projects, 104 had attracted additional capital to further pursue development of their technologies. Additional funding came variously from collaborative partners, venture capitalists, public offerings of stock, other governmental departments including state government programs, and other sources.

Members of the **Genosensor Consortium** attracted additional internal funding after successfully developing a technology for automated DNA sequence analysis during the ATP-funded project. (Project number 92-01-0044)

eMagin Corporation received a \$3 million grant from the U.S. Air Force after successfully developing microdisplays that have been integrated into hundreds of medical, commercial, and military applications. (Project number 93-01-0154)

ABB Lummus attracted additional internal capital after the ATP project as a result of the company's successful development of a new, environmentally superior process to manufacture alkylate using solid-acid catalysts. (Project number 95-05-0034)

The Dow Chemical Company also attracted additional capital due to the methodologies developed during the ATP project to create a direct, economical, single-product oxidation process incorporating a silver-based catalyst for conversion of propylene to propylene oxide. (Project number 95-05-0002)

PART 5

Overall Project Performance

The individual performance of the 150 completed projects has varied, as, measured by the creation and dissemination of knowledge and the accelerated use of that knowledge for commercial purposes. Some of the award-recipient companies grew by leaps and bounds as they translated their knowledge gains from ATP-funded research into profitable and beneficial products, services, and production processes. Some continued to strive toward hard-to-achieve goals, while others showed little outward signs of further progress. A few that achieved impressive research accomplishments later failed in the commercialization phase. However, the achievements of the more successful projects, with their impressive new performance capabilities resulting in lower costs and higher quality products and processes, appear to have much more than compensated for the less successful projects. There is considerable evidence that the benefits attributable to ATP from the 150 completed projects substantially exceed their costs.

(The 150 completed status reports discussed in this chapter can be found online at <http://www.atp.nist.gov/> under funded projects.)

Composite Performance Scores

During the intermediate period covered by this analysis—after project completion but before long-term benefits have had time to be realized—ATP uses a Composite Performance Rating System (CPRS) to help gain a sense of how projects in the portfolio have performed overall thus far against ATP’s mission-driven multiple goals.⁹ In this intermediate period of project life cycles, the focus is on progress toward the goals of 1) knowledge creation, 2) knowledge dissemination, and 3) commercialization. The CPRS uses a weighted composite of output data systematically collected for each of the 150 projects—some of which have been presented in aggregate form in the preceding sections of this overview—to assess overall performance of the portfolio of completed projects in this intermediate period.

The output data serve as indicator metrics of progress toward achieving goals. Examples of available indicator metrics signaling progress toward the creation and dissemination of knowledge are a) awards for technical excellence bestowed by third-party organizations,

⁹ For an in-depth treatment of the CPRS, which was developed in prototype for ATP’s use, see Rosalie Ruegg, *Bridging from Project Case Study to Portfolio Analysis in a Public R&D Program*, NIST GCR 03-851 (Gaithersburg, MD: National Institute of Standards and Technology, 2003).

b) patent filings, c) publications and presentations, d) knowledge dissemination from potential reverse engineering of new and improved products/processes on the market or expected soon, and e) collaborative activity. Available indicator metrics signaling progress toward commercialization of the new technology include a) attraction of additional capital, b) employment gains, c) project-related company awards for business success, d) moving products and processes into the market, and e) analysts' outlooks for future progress by the award-recipient companies.

Weights are assigned to the indicator data, which are combined to produce a composite numerical score that is then converted to a zero- to four-star rating for each project. A score of one star or less signals poor overall performance; two stars, moderate performance; three stars, strong performance; and four stars, outstanding performance. The distribution of CPRS scores computed for each project in a portfolio of projects is then examined, and the results taken as indicative of overall portfolio performance.

The resulting CPRS ratings provide an easy-to-grasp highlighting of portfolio performance in the intermediate period. They call out those projects that have exhibited outstanding or strong outward signs of progress towards long-run program goals during the years covered and those that have exhibited moderate or few signs of progress. However, the ratings are imperfect and should be viewed as only roughly indicative of overall performance.

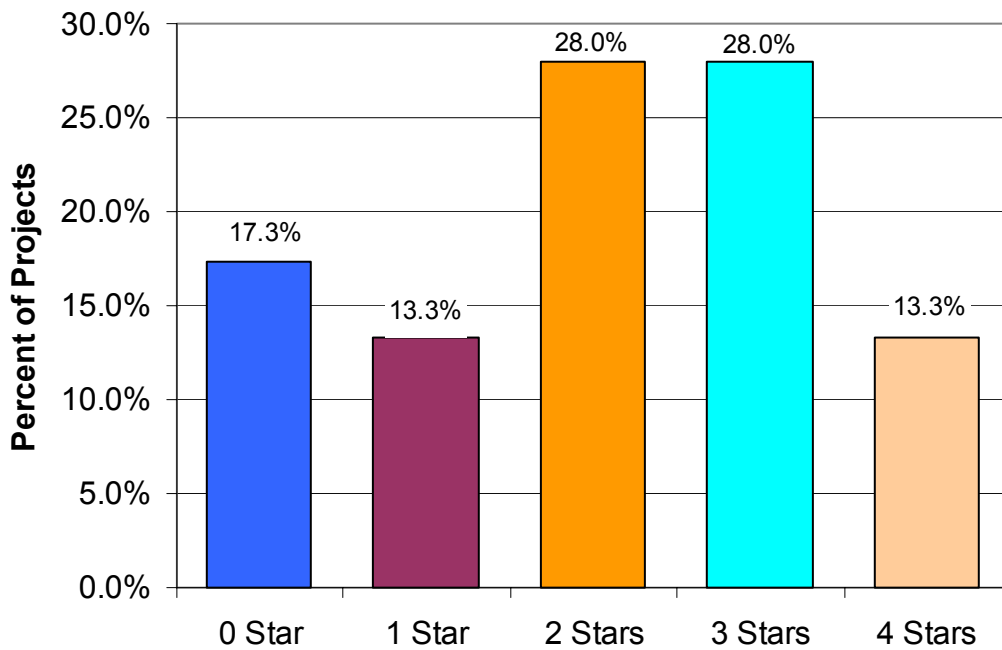
The performance metrics are consistent with the view of varying degrees of success—with knowledge creation and dissemination constituting partial success, and a continuation into commercialization constituting a fuller degree of success in terms of project progress. Some companies carried out their proposed research with a degree of success during the time of ATP funding, but then did not continue pursuit of their project's larger goals after ATP funding ended. At this stage of evaluation, ATP considers such projects only partial successes, because the direct path for achieving project goals is truncated. Such projects are not among the higher scorers in this report. It is possible, however, that developments along the indirect path (diffusion of knowledge from the project through publications, presentations, patents, and licensing) may nevertheless occur—particularly if a project produced effective knowledge transmitters, such as patents and publications. It is also possible that a company may work in secrecy for a long period of time with no visible outputs and then suddenly explode on the scene with a single output that will yield large societal benefits.

Limiting factors include the extent to which not all relevant effects are captured; moreover, the use of indicator metrics is constrained by data availability, the development of the weighting system is empirically driven rather than theoretically based, and the ratings do not directly measure national benefits. The degree of correlation between a project's performance score and its long-run societal benefits is impossible to know at this time. Projects with the same scores are not necessarily equal in their potential benefits. They are, however, somewhat comparable in terms of the robustness of their progress to date.

Scoring the First 150 Completed Projects

The distribution of CPRS scores for ATP's first 150 completed projects is shown in Figure 5-1. Combining the two and three-star categories shows 56 percent of projects performed at a moderate level. Thirteen percent of the projects performed at a high (four-star) level and approximately 30 percent of the projects scored one star or less, perhaps not surprising for companies taking on difficult goals.

Figure 5-1
Distribution of Projects by Star Rating



Source: Advanced Technology Program First 150 Status Reports

The 20 four-star projects overall include 16 single-applicant projects led by small companies and four joint ventures, two led by a consortium and two led by small companies. Leaders of these top-scoring projects are listed in Table 5-1.

**Table 5-1
List of Four-star Projects**

Aastrom Biosciences, Inc.	Nanophase Technologies Corporation
American Superconductor Corp.	National Center for Manufacturing Sciences (NCMS)
Automotive Composites Consortium (a Partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)	Orchid BioSciences (formerly Molecular Tool, Inc. Alpha Center)
Cerner Corporation (formerly DataMedic - Clinical Information Advantages, Inc.)	SciComp, Inc.
ColorLink, Inc.	SDL, Inc. and Xerox Corporation
Cree Research, Inc.	Third Wave Technologies, Inc.
Engineering Animation, Inc.	Tissue Engineering, Inc.
Integra LifeSciences	Torrent Systems, Inc. (formerly Applied Parallel Technologies, Inc.)
Kopin Corporation	Xerox Palo Alto Research Center
Large Scale Biology Corporation (formerly Large Scale Proteomics Corporation)	X-Ray Optical Systems (XOS), Inc.

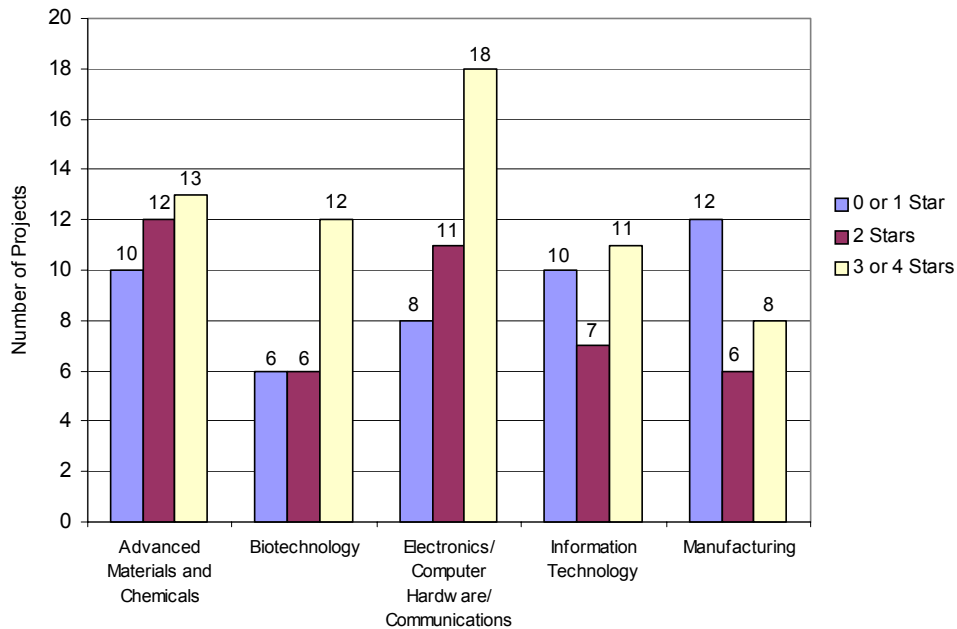
The three-star projects included 35 single-applicant projects and 7 joint-venture projects. Of the single-applicant projects, 25 were led by small companies, two by medium companies, and eight by large companies. Of the joint ventures, two were led by small companies, two by an industry consortium, two by a large company, and one by a nonprofit organization.

A few projects with low CPRS ratings had impressive technical achievements as indicated by the receipt of a third-party technical award, though most of the technical awards went to those with the highest overall ratings. In contrast, all of the awards for business acumen went to the projects with CPRS ratings of three or four stars

Performance by Technology Areas

Overall project performance in the intermediate period covered by the study varied by technology area, as illustrated in Figure 5-2. Of the 24 Biotechnology projects, 12 were three- or four-star projects. Of the 37 Electronics projects, half scored high. Of the 26 Manufacturing projects, close to third scored high, but 46 percent scored low. The 35 projects in the Advanced Materials and Chemical group were more evenly divided into high, low, and moderate scorers. The 28 Information Technology projects had 11 projects that were high-scoring projects, 7 moderate-scoring, and 10 low-scoring projects. Differences in life cycles among the technology areas may account for part of the performance differences, but the relatively small number of projects in each category does not support the drawing of robust conclusions about how projects in the different technology areas will perform.

Figure 5-2
Number of Composite Scores by Technology Area



Source: Advanced Technology Program First 150 Status Reports

Project Performance Translated into Economic and National Security Benefits

Photonics

ATP has provided cost-sharing funding to more than 120 photonics projects since 1991¹⁰. To access the economic benefits from a portion of these projects, the author adopted a cluster study approach to combine the methodological advantages of detailed case studies and of higher level overview studies. The following five projects were selected for analysis: Capillary Optics for X-Ray focusing and Collimating; MEMS-Based Infrared Micro-Sensor for Gas Detection; Infrared Cavity Ring-Down Spectroscopy; Optical Maximum Entropy Verification; and Integrated Micro-Optical Systems.

Findings from the study indicate that U.S. industry and consumers, and the nation, will enjoy at least \$33 of benefits for every dollar of ATP's \$7.47 million investment in the cluster of five projects. ATP technology translates into \$1.90 already realized benefits generated for every dollar of ATP's investment in the five projects.

Component-Based Software (CBS)

Developing the capacity to build large software systems from assemblies of smaller, reusable, independent components is an important strategy to reduce software system

¹⁰ Pelsoci, Thomas, M., *Photonics Technologies: Applications in Petroleum Refining, Building Controls, Emergency Medicine, and Industrial Materials Analysis*. NIST GCR 05-879 (Gaithersburg, MD: National Institute of Standards and Technology, September 2005).

costs, increase system reliability, and enable lower cost upgrades. Three projects included among the first 150 Status Reports were part of a portfolio of 24 projects that was included in an in-depth economic case study conducted by RTI.¹¹ These projects were led by **Reasoning Inc.**, **TopicalNet, Inc. (formerly Continuum Software)**, and **HyBrithms (formerly Hynomics Corp.)**.

Across the entire CBS portfolio, RTI's economic study estimated \$840 million in net-present-value benefits and a benefit-to-cost ratio at 10.5, suggesting that the investment in the portfolio of projects as a whole was worthwhile. The net-benefits estimate is based on the cost of all 24 projects, but the benefits of only 8 were the subject of the detailed case study. In addition, the study found other benefits that were presented qualitatively, namely, enhancing the credibility of the mostly small software firms that were funded and assisting firms in strengthening their planning and management functions.

Reasoning Inc., TopicalNet Inc. (formerly Continuum Software), and Hynomics Corp. (formerly HyBrithms) had commercialization activities underway when RTI conducted its study. Their costs, but not their benefits, were included in RTI's aggregate portfolio net-benefit measure, because they were not among the eight projects selected by RTI for the portfolio benefits assessment. Thus, the RTI study results, at best, suggest that the three projects are part of a portfolio of projects found to be valuable. Of the three projects, two are rated as three-star performers, and one is a two-star performer.

It is also informative to look at how some of the other projects that were rated as top performers have progressed since the original data were compiled and the CPRS ratings calculated. Additional projects are profiled below.

Scalable Parallel Programming

One of the top-performing projects among the first 50 completed projects, originally profiled in Volume 1, was a project led by **Torrent Systems, Inc.** Although Torrent had fewer knowledge-dissemination outputs than the other top-performing projects, its exceptional commercialization efforts boosted it into the four-star group. The project developed a component software system that insulates programmers from the complexities of parallel programming while allowing them to use it productively in scalable applications. Torrent delivered this new capability in its software product, *Orchestrator*TM. An early user of the new software, United Airlines, was able to increase its revenue by \$100 million per year as a direct result of using *Orchestrator*TM.¹²

When revisited in Status Reports, Volume 2, Torrent's technology was reported to be enabling e-businesses and other companies to process and analyze unlimited volumes of data. Torrent was listed in *Computerworld's* "100 Hot Emerging Companies" in 1998 and received a number of other awards recognizing both its software technology and business acumen.

¹¹ White and Gallaher, November 2002.

¹² Information from Hoover's Online company search and Torrent's website, current August 31, 2000.

Since that time, Torrent, which had only two employees when it received its ATP award, has been acquired for a purchase price of \$46 million by Ascential Software Corp., a global company with a market capitalization of \$1.1 billion, headquartered in Westboro, Massachusetts.¹³ According to Ascential's Chairman and CEO, Peter Gyenes, "Torrent's patented and proven parallel processing technology is a perfect complement to the rich feature set within our data integration solution, DataStage."¹⁴ According to additional public statements by the company, Ascential has integrated Orchestrate™ into its DataStage XE product family, with the result that customers will be able to integrate data of virtually any volume and complexity, with infinite scalability, and turn growing amounts of data into valuable information assets.

United Airlines, first a Torrent customer and then an Ascential customer, is using Orchestrate™ and an IBM parallel-processing computer to design a system for managing airplane seat assignments. A statement by Bob Bongirno, managing director of applications development for United Airlines, which is posted at the Ascential Software Corp. website provides a user's perspective of the importance of the product:

"At United, we analyze 'astronomical' amounts of data every day through our Orion system to determine the optimum seat availability and price across tens of millions of passenger itineraries," he said. "For Orion and our other data-intensive applications, we demand a parallel processing technology that is robust and reliable enough to process massive data volumes on very large systems and will provide a state-of-the-art data integration foundation that helps us manage all our disparate data sources and accelerates the development of new applications. The combination of technologies from Torrent and Ascential holds great promise for meeting the data processing needs of customer-centric organizations like United."

Thus the commercialization path has grown more complex for this ATP-funded technology as the technology has been combined with other software elements. At the same time, the impact potential of the technology appears strong. According to Doug Laney, META Group Vice President, the worldwide market for data integration was projected to grow from \$900 million in 2001 to \$1.3 billion in 2004,¹⁵ and the technology platform funded in part by ATP appears well positioned to play a role in serving this growing market. Those projections were well-founded. Ascential grew rapidly in 2004, with a 46 percent increase in total revenue. In March 2005, Ascential agreed to be acquired by IBM for approximately \$1.1 billion, strengthening IBM's fast-growing information integration business.¹⁶ (Project number 94-06-0024)

¹³ Standard and Poor's stock report on Ascential Software Corp.

¹⁴ Press Release, November 28, 2001, available on-line at www.ascentialsoftware.com, Press Center.

¹⁵ Ibid.

¹⁶ Company press release, "IBM to Acquire Ascential Software." March 14, 2005. (<http://ibm.ascential.com/news/pr.html/view/1107>)

High-Temperature Superconducting (HTS) Wire

The project led by **American Superconductor Corporation (AMSC)** is another of the top-rated 100 completed projects profiled originally in Status Report Volume 1. At the time Volume 1 was being written, the company was beginning to launch its commercialization effort. Since then, the company has reportedly continued making impressive advances, building the world's first high-volume HTS wire manufacturing plant with a capacity to manufacture 20,000 kilometers of wire per year when it is fully equipped. This new manufacturing capacity is said to give potential customers the ability to accelerate their schedules for launching commercial products incorporating HTS wire by making the product available to them in commercial quantities, at commercial prices.¹⁷ AMSC's products and services listing now shows a vertically integrated portfolio that includes HTS wire, motors, generators, synchronous condensers, industrial power quality solutions, power conversion, and transmission grid solutions.

A press release issued October 1, 2003, announced that AMSC had received additional funding from the Department of Defense (DOD) and Department of Energy (DOE) to support further manufacturing scale-up for second-generation HTS wire. According to Dr. Paul Barnes, U.S. Air Force Superconductivity Team Leader, ensuring that the United States will have a reliable supply of the second-generation HTS wire is expected to be central to the development of many future military systems, including lightweight high-power generators and advanced weapon systems. According to James Daley, manager of the Superconductivity program at DOE, the technology is also expected to play an important future role in upgrading the nation's power grid.¹⁸ (Project number 91-01-0146)

Visualization Software

As in the preceding examples, **Engineering Animation, Inc. (EAI)**, leader of another of the top-performing projects and originally profiled in Status Report Volume 1, continued to aggressively and successfully pursue applications of its award-winning imaging software capabilities developed in the ATP-funded project. Founded by two professors and two graduate students in 1990, EAI had 20 employees at the time ATP made the award. According to company officials, the ATP award allowed it to significantly extend its capabilities in computer visualization and computations dynamics and to form important collaborative relationships that enabled it to leverage the technology in many different directions. The company used its ATP-funded technology to improve the training of doctors as well as to guide medical procedures. Furthermore, patients reportedly had better outcomes when the visualization software was used during their surgical procedures.

In 1999, the company employed approximately 1,000 staff members and had sales of \$71 million. At that time, EAI had extended and deployed its award-winning visualization

¹⁷ Information provided by the company at its website, www.amsuper.com.

¹⁸ Company press release, October 1, 2003.

capabilities to develop a virtual factory technology implemented at Ford Motor Company. This application of the software enabled faster design and analysis of factory models.

On October 23, 2000, EAI was acquired by Unigraphics Solutions Inc. for \$178 million. Subsequently, through acquisition and merger, Unigraphics and another software services company, SDRC, became a combined subsidiary of Electronic Data Systems Corporation (EDS), the world's largest information technology outsourcing services company, which has a worldwide infrastructure and 138,000 employees.¹⁹ Unigraphics and SDRC were combined to form EDS's fifth line of business, Product Lifecycle Management (PLM) Solutions. This union provided, through Unigraphics NX software, a unified approach to extended enterprise collaborations enabling the modeling and validation of products and their production processes digitally from initial concept to finished parts. Thus, EAI followed the business model for growth of merging with a much larger company.²⁰ An online search revealed that previously developed EAI products and books remain on the market. (Project number 91-01-0184)

Examples of strong projects from among the three and four -star group are described below. These, too, appear to be delivering important economic benefits.

Improving Software Efficiency through Reusable Components

An example is a four-star project led by **Xerox Parc** which is credited with developing aspect-oriented programming (AOP) and later developed products that incorporated its principles. After the ATP funded project ended Xerox developed AspectJ, an open-source language based on AOP. Aspect J extends Java; and is being further developed and used in IBM's software applications and by many others. Eight patents emerged from this ATP-funded project and more than 3,250 articles or books have been written about AOP. In June 2003, AspectJ won the JavaWorld Editors' Choice Award for the Most Innovative Product or Technology Using Java. (Project number 94-06-0036)

Miniature LCSs Enhance High-Definition Displays

Another four-star project with continued strong commercialization was led by **Kopin Corporation**. Kopin formed a joint venture with Philips, and together with their subcontractor, Massachusetts Institute of Technology facilitated a paradigm shift in high-definition display technology. During the ATP funded project, Kopin and Philips combined existing monochrome liquid crystal displays (LCDs), with color, signal processing, and high-definition technology. Independently, Philips successfully commercialized high-resolution projection HDTVs using the ATP-funded technology. Kopin also successfully applied the ATP-funded enabling technology in numerous applications including miniaturized display applications for use in viewfinders for camcorders and digital cameras, wearable computers, virtual reality games, and military

¹⁹ Prior to the acquisition of Unigraphics, EDS was the major company stockholder. Information found at www.eds.com.

²⁰ Ibid.

applications. LCD projection display technology is a key product differentiator in U.S. electronics manufacturing. (Project number 94-01-0304)

Structural Composites for Large Automotive Parts

As a result of the ATP funded project the **Automotive Composites Consortium-ACC**, (A partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors) successfully produced a prototype box for a pickup truck that is stronger and more durable than steel, does not rust, is visually attractive, requires no bed liner, and improves fuel efficiency through its light weight (36 pounds, or 33 percent, lighter than steel). This pickup truck box gave the ACC member companies (General Motors [GM], Ford, and Chrysler, which later became DaimlerChrysler) the knowledge and tools to develop commercial products and to continue innovative research, based on this initial success. Applications of this successful ATP-funded technology include strong, lightweight components for aircraft, firefighter helmets, and marine motor covers. Project researchers shared their developments through one granted patent and several articles and presentations. As public acceptance of tough, durable composites increases, applications are expected to broaden. (Project number 92-01-0040)

To these examples, other promising technologies may be added—technologies that improve productivity, facilitate better weather forecasts, improve communications, enable new drug discovery, reduce energy costs, and improve health and safety.

What Difference Did ATP Make?

ATP aims to improve the international competitiveness of U.S. firms by funding projects that would not take place in the same timeframe, on the same scale, or with the same goals without ATP's support. A project may be successful in terms of achieving its goals, but if the same accomplishments would have occurred in the same timeframe without ATP, then the program has not had the intended effect. For this reason, evaluation studies of ATP—as well as other government programs—should apply the principle of “additionality” to correctly distinguish between benefits that would likely have occurred anyway and those benefits that are reasonably attributable to ATP.

In preparing the 150 individual mini-case studies, analysts asked project leaders about the role ATP funding played in their projects. Throughout the project selection process, beginning with the application, ATP presses the questions of why the project requires ATP funding, why funding is appropriate, what will happen if ATP funding is not provided, and how the expected outcome will differ with and without ATP involvement. During the evaluation process, these questions are again pursued retrospectively, i.e., what happened that was different as a result of ATP? Applied prospectively, the results are hypothetical. In evaluation studies, the results may be based on counterfactual survey and interview questions, such as those posed in the status report case studies. Evaluation studies have also used control group techniques, which provide more reliable evidence of the additional impacts of ATP.²¹

²¹ See *Survey of Applicants 2002*, NIST GCR 05-876, (Gaithersburg, MD: National Institute of Standards and Technology, June 2005).

Forty-six percent of the respondents indicated their projects would not have happened at all without ATP funding. Indeed, some participants said their companies would have gone out of business had the ATP award not been made.

Thirty-eight percent of the respondents said they would have attempted the project at some later date or at a slower pace and that ATP funding enabled them to accelerate the technology. Table 5-1 shows the project time savings attributed to ATP for those projects that reported they would have proceeded without ATP funding. With ATP, the projects avoided delays ranging from six months to five years and more. The acceleration of some of the projects may seem short; however, the value of even a small acceleration can be substantial. Speed in developing and commercializing a technology can also mean increased global market share for U.S. producers.

Table 5-2
Effect of ATP Funding on Expected Timing of Research

Effect on Project		Number of Projects
Would not have conducted Project without ATP funding		69
Would have proceeded without ATP funding, but with a delay*:		57
	Length of Delay	
	6 months	1
	12 months	3
	18 months	7
	21 months	3
	24 months or more	10
	More than 5 years	11
	Delay, but time unspecified	22
No Response		24
Total		150

Source: Advanced Technology Program First 150 Status Reports

*Another factor potentially influenced by ATP funding (the scope and scale of the project) was not explicitly covered.

**The Printed Wiring Board Joint Venture project had a split response: half the tasks would not have been done at all and half would have been delayed by at least a year. This result is recorded conservatively in Table 5-1 as a two-year delay.

A number of companies also reported other effects of their ATP awards. Some reported that receiving their award enhanced their ability to raise additional capital. Some reported that their award helped them form collaborative relationships for research and commercial activities. Others reported that receipt of their ATP award had enabled them to gain in international competitiveness.

What Constitutes Success and Failure for ATP?

Because individual project failure must be allowed and tolerated in a program that focuses on overcoming challenging technical barriers to innovation, it is essential to take a portfolio approach to assessing ATP. Moreover, success should be assessed against the legislated mission of the program.

Four general tests, and several additional specific tests—all derived from ATP’s mission—if applied after sufficient passage of time, should reveal the extent to which ATP has successfully met its mission, as described below.

Test 1: Has the portfolio of ATP-funded projects overall produced large net social benefits for the nation?

Test 2: Has a substantial share of net social benefits accrued to citizens and organizations beyond ATP direct award recipients?

Test 3: Did ATP make a substantial positive difference in the size and timing of the benefits?

Test 4: Has the portfolio of ATP-funded projects enhanced United States’ economic and technological competitiveness?

Additional specific tests of success include the following: Did the projects produce new scientific and technical knowledge? Did ATP increase collaboration? Were small businesses able to participate? Were manufacturing capabilities improved?

While the ultimate answers to these success “test questions” depend on the long-run impacts of the entire portfolio of ATP projects, the performance-to-date of the sub-portfolio of 150 projects provides emerging answers.

There is mounting evidence that the tests for program success are being met. First, there is strong evidence that social benefits of the portfolio are large and exceed program costs. Second, there are benefits extending well beyond those captured by the direct award recipients: there is substantial evidence of knowledge and market spillovers as others cite the project patents and use the products. Third, there is evidence that ATP has made a significant difference in the amount and timing of benefits, as well as having other beneficial impacts on the companies. Fourth, there is some evidence of improvements in the competitiveness of U.S. companies.

The performance ratings show that the majority of the projects continued to make progress in the several years after ATP funding ended. Moreover, the portfolio has been shown to contain a core group of highly active and productive projects that are successfully accomplishing their high-risk project goals.

ATP awarded a total of \$621 million to the 150 completed projects. Questions of keen interest are what is the public investment producing in the way of benefits, and are the tests for program success being met? Estimated benefits attributed to ATP from just a few of the 150 projects for which quantitative economic benefits have been provided exceed ATP's funding for all of the 150 projects. In addition, there is considerable evidence of large project benefits that have not yet been quantified.

This completes the portfolio view of ATP. Appendix A that follows provides an overview of the 150 individual projects that make up the portfolio. Appendix B describes reasons that some ATP-funded projects did not proceed to completion. Appendix C lists the first 150 completed projects along with their CPRS star ratings.

ABB Lummus Global, Inc. (formerly ABB Lummus Crest)

A Safe, Environmentally Friendly Method to Produce Alkylate

High-octane alkylate is used as a gasoline blend stock to raise octane and reduce automobile emissions. In 1995, refiners typically used liquid acid catalysts (hydrofluoric acid or sulfuric acid) to promote and enhance the speed of the chemical reactions to produce alkylate. However, liquid acids pose serious safety and handling problems, including potential life-threatening accidents. ABB Lummus Crest (the name was changed to ABB Lummus Global Inc. in 1995) submitted a proposal to the Advanced Technology Program (ATP) to develop a solid acid catalyst, which would be a safer, more economical substitute catalyst for the alkylate process. ABB would develop a thin-film catalyst layer to be deposited on a small pellet-like carrier. Previous attempts to develop solid acid catalysts had failed due to pore blockage and short catalyst life. Numerous technical risks included identifying an acceptable inert support structure for the thin-film catalyst, activating the catalyst, and developing a means to regenerate the catalyst at the refiner's site. If successful, ABB believed the solid acid catalyst process could retrofit existing U.S. alkylate plants. If retrofitted in all 112 domestic plants, the process could save approximately \$580 million in processing costs per year.

ATP awarded cost-shared funding for a three-year project as part of a focused program, "Catalysis and Biocatalysis Technologies," in 1995. By the end of the project, ABB was able to deposit a thin layer of active catalyst on a small, inert, pellet-shaped support and regenerate the catalyst quickly. They were granted a patent covering the method of preparation for this catalyst. Furthermore, the company developed a reactor design to perform the process efficiently for which they applied for patent coverage. Based on their achievements, ABB continued development and formed a joint venture in 2001 with Akzo Nobel (a catalyst manufacturer whose name was changed in 2004 to Albemarle Corp. through an acquisition) and Fortum Oil and Gas, an energy company (whose name was changed to Neste Oil Corporation in 2005, as a result of a restructuring). The joint venture partners developed and optimized a solid acid alkylation demonstration plant that produced 10 barrels per day from 2002 to 2004. As of 2005, the joint venture was in negotiations with several large energy companies to establish the first commercial-scale plant to produce upwards of 10,000 barrels per day. Analysts predict that growing global demand for alkylate will require 40 to 50 new plants by 2015.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* * *

Research and data for Status Report 95-05-0034 were collected during December 2004 – February 2005.

Liquid Acid Catalysts Pose Significant Risks

Gasoline is manufactured by combining several petroleum-based components to meet octane, oxygenate, and environmental standards. Alkylate is an ideal component, because it makes fuels more environmentally sound and is very high in octane

(higher than 90). Demand for fuel containing increasing amounts of alkylate has grown as the number of fuel-environmental standards has increased.

Developed during World War II to produce high-octane gasoline for aircraft, conventional methods to process alkylate rely on liquid acid catalysts, either hydrofluoric

or sulfuric acids. (Catalysts increase the speed and efficiency of the chemical reactions.) Later, alkylate's main application evolved as a component in unleaded and premium grades of automobile gasoline. However, both hydrofluoric acid and sulfuric acid are very corrosive and can cause serious injury through skin contact or inhalation. A few well-publicized accidents in the United States involving hydrofluoric acid in the late 1980s resulted in significant leakage, exposure, and injury. Thus, production methods that use liquid acid pose a potential threat to plant workers and to the surrounding communities. Extensive safety precautions and complex permit processes can make liquid acid methods expensive. In addition, liquid acid processing results in an unwanted heavy hydrocarbon (oil) byproduct called acid-soluble oil, which can be difficult to recover and dispose.

Two types of solid catalysts had already been studied unsuccessfully. One type consisted of active materials adsorbed on inert supports of alumina or silica. One difficulty with this type of solid catalyst was the high cost of the active components. Furthermore, the catalyst reacted slowly with the support in the presence of moisture and then lost activity. Disposing of the spent catalyst posed environmental problems. The second type of solid catalyst contained acidic (heteropoly acids) or "superacid" supports. The active catalyst constituents did not leach out, and they could be regenerated conveniently. The main problem with these solid catalysts, however, was that their initial high activity decreased rapidly, because heavy alkylation byproducts blocked the pores of the catalyst support.

ABB Lummus Proposes to Develop Solid Acid Catalysts

ABB Lummus Crest (ABB) was an engineering and construction company with customers in the oil and gas refining industry. ABB proposed to develop a new, environmentally superior process to make alkylate. Company researchers had to address the following technical risks:

- Identify acceptable inert support structure for the thin layer of alumina catalyst
- Identify an appropriate activation procedure to form the active catalyst

- Produce a catalyst with sufficient concentration of active sites per unit of reactor volume
- Maintain catalyst activity for a target 1,000 kg alkylate per 1 kg of catalyst
- Regenerate the catalyst on site at the refinery
- Improve the catalyst's safety and environmental characteristics (compared with hydrofluoric and sulfuric acid)
- Integrate the catalyst with the refineries' reactors, in order to retrofit existing plants that currently rely on hydrofluoric and sulfuric acid

Because of these risks, ABB was unable to fund the research internally. ATP awarded cost-shared funding for a three-year project beginning in 1995 as part of a focused program, "Catalysis and Biocatalysis Technologies." If successful, ABB believed solid acid alkylation could save U.S. refiners an estimated \$580 million in processing costs per year based on retrofitting 112 domestic alkylation facilities. Another potential benefit of solid acid catalysis would be reducing or eliminating the unwanted heavy oils that result from liquid acid processes. The primary benefit would be increased safety for communities and plant workers, because solid acid processing does not have the health and safety risks that are associated with transporting and utilizing liquid acids.

ABB Completes Laboratory-Scale Reactor

In 1995, soon after the start of the project, ABB Lummus Crest merged with ABB Global Engineering to form ABB Lummus Global Inc. During the first year of the project, ABB intended to develop the catalyst supports, the methods of forming the thin layer of oxide film on the supports, and the procedure for activating the catalyst. During the second and third years, ABB would develop the proposed solid acid catalyst and would focus on laboratory catalyst testing, regeneration studies, reactor engineering studies, catalyst manufacturing, process engineering, and process economics to guide research. Contact with potential clients would provide feedback to make adjustments to the process during development. ABB began to explore solid acid catalysis with the following partners: Glatt Air

Techniques, Inc., the Department of Ceramics Engineering at Rutgers University, Niro Inc., Norton Chemical Process Products Corp. (later renamed Saint-Gobain NorPro Corp.), and Vector Corp. ABB also relied on consultants at Applied Research and Technology and formed a key cooperative relationship with Akzo Nobel, a catalyst manufacturer, in 1996.

ABB's research covered five primary areas:

- **Thin-film formation and catalyst activation.** ABB developed the methodology to form thin-film oxide or other catalyst precursors on a suitable support. Vector Corp. provided thin-film coating processes. Niro, Inc. conducted alternative film formation tests, but these did not produce satisfactory thin-film coatings. Glatt Air Techniques, Inc. conducted more successful film formation tests in a Wurster Fluid Bed Coater. Norton Chemical Process Products Corp. supplied the inert alpha alumina pellets onto which the coatings are applied. ABB prepared the active chlorinated alumina thin-film catalyst that had the desired acidity and stability and applied it to the support.
- **Surface features' effects on catalyst performance.** ABB measured the relationship between surface features of thin-film catalysts in order to improve their performance for alkylation. A good catalyst has high activity and selectivity. Activity refers to the capacity of a chemical to take part in a chemical reaction; selectivity refers to the catalyst's ability to promote a desired reaction rather than undesired reactions that reduce the yield of the desired product. ABB was able to demonstrate that solid acid alkylation resulted in higher selectivity than sulfuric acid alkylation. Activity remained stable.
- **Solid acid catalyst testing.** ABB tested five catalysts: beta zeolite, paraffin isomerization type catalyst, a proprietary catalyst from Hydrocarbon Technologies Inc., another alumina catalyst, and a proprietary catalyst developed by Akzo Nobel. Because the Akzo Nobel catalyst showed the most promise, ABB prepared and tested a thin film adaptation of it.
- **Catalyst regeneration.** A significant problem for solid acid alkylation was that these catalysts fouled

quickly, lost activity, and required frequent regeneration. ABB needed to find a way to clean the catalyst and bring it back to fresh activity. The company demonstrated low-temperature regeneration with hydrogen, performing five cycles of alkylation and regeneration. Results during the project indicated good recovery of catalyst activity after each regeneration, with good stability.

- **Reactor engineering.** ABB developed a kinetic model and investigated several commercial reactor concepts. They built and tested a series of reactors with varied flows of reaction products and filed a patent application for their optimized reactor system. They designed and built a laboratory-scale reactor to simulate one stage of a commercial reactor.

In 1997, ABB and Akzo Nobel initiated a cooperative program to evaluate the alumina thin-film catalyst and an alternative, proprietary Akzo-developed catalyst. Their intent was to move forward jointly in developing, piloting, and commercializing the final preferred alkylation process.

Catalyst production methods that use liquid acid pose a potential threat to plant workers and to the surrounding communities

For the remainder of the ATP-funded project, ABB and Akzo conducted further testing of three catalysts: chlorinated thin-film alumina catalyst (the main focus of this project), a proprietary catalyst developed by Akzo Nobel, and a thin-film adaptation of the Akzo Nobel catalyst. They developed a reactor concept for a commercial solid acid alkylation plant for each of the three alternatives. The companies optimized feed materials and process parameters for alkylation. They developed and demonstrated regeneration cycles on a laboratory scale and finally selected the catalyst that had the best regeneration characteristics. ABB received a patent for their technology advances and published their results in academic journals. Key accomplishments from the ATP-funded research included the production of efficient, solid acid catalysts on inert substrates; demonstration of the catalyst regeneration process; and the reactor design concepts. These advances could not have been accomplished without ATP support.

Researchers Seek a Third Joint Venture Partner

After ATP funding ended in 1998, ABB and Akzo continued developing their solid acid alkylation process. Prior to commercialization, ABB and Akzo needed to resolve size and capacity scale-up issues and develop business plans for manufacturing with solid acids. Their goal was to achieve at least economic parity with hydrofluoric and sulfuric acid process methods. They developed a concept for a demonstration plant, which included technical designs, capacity goals, and general layout. The goal was to determine whether large-plant manufacturing was feasible after achieving success in the demonstration plant and determining the likelihood of technical and economic success in a commercial plant. At this point, another partner was needed, so that ABB and Akzo could develop a prototype plant located at or near an existing refiner.

ABB believed solid acid alkylation could save U.S. refiners an estimated \$580 million in processing costs per year.

Fortum Oil and Gas joined the joint venture in 2001. Fortum was an energy company with an ongoing interest in developing solid acid catalysts. The company had participated in similar prototype plant development efforts about 10 years earlier. Together, the three companies developed a prototype plant located in Porvoo, Finland (see illustration) that began operation in 2002. The plant used the reactor design concepts and catalyst regeneration methods developed during the ATP-funded research. The prototype plant provided the complete cycle of alkylation, including catalyst regeneration, at a fully scalable capacity. If successful, the concepts would be scaled up to a commercial-scale process, which produces upwards of 10,000 barrels of alkylate per day (one barrel equals 42 U.S. gallons). Furthermore, handling and safety concerns were dramatically reduced at the prototype plant, as the safe-to-handle solid acid catalyst pellets were utilized in place of the conventional toxic and corrosive liquid acids. The pellets could be delivered in plastic bags or metal drums.



The photo on the left shows an outside view of the solid acid alkylation pilot plant built and operated by the joint venture partners from 2002 to 2004 in Porvoo, Finland. The plant was used to optimize and demonstrate the process. An internal view of the reactors is shown on the right.

The demonstration plant proved that the solid acid catalyst yields high-quality alkylate, with maximized activity and stability. The process was robust, with low sensitivity towards feedstock variation and common impurities. One key to the process success is cyclic reactor operation; that is, alternating periods of alkylation and mild catalyst regeneration, which allows the plant to operate continuously and maintain product quality.

Joint Venture Set to Commercialize “AlkyClean”

In October 2004, ABB, Akzo Nobel, and Fortum Oil and Gas completed their demonstration project. (The Akzo Nobel refinery catalyst business was purchased by a U.S. firm, Albemarle Corp., in 2004. Fortum was renamed Neste Oil Corporation in 2005.) The joint venture partners announced publicly that they had developed a new, environmentally superior process to make alkylate, which achieved a similar alkylate product at a cost that was comparable to the liquid acid catalyst process. Moreover, the process used is safer, because the solid acid catalyst pellets for this “green technology” are neither toxic nor corrosive, unlike liquid acids, which must be transported to the refinery and utilized under strict safety precautions. Furthermore, the solid acid process “...generates gasoline of the highest quality. Moreover, this robust process is reliable, no unnecessary byproducts are produced, and plant investment is considerably lower than with the old technology,” said Philip Angevine, manager of the Ultra-Clean Fuels program at ABB. ABB, Albemarle, and Neste expect that their solid acid alkylation process, called “AlkyClean,” will fill a crucial refining niche.

Because AlkyClean processing is economically competitive with liquid acids, without the environmental safety concerns, ABB Lummus and its joint venture partners anticipate that oil refinery firms will choose their technology for new alkylation plants. ABB will license the new technology for both new plants and retrofitting existing plants. The technology will be typically transferred via an engineering design package, with drawings and all the specifications and instructions to build and operate the plant. ABB representatives will support the plant as technical consultants throughout the life cycle of the unit.

Alkylation Demand Is Expected to Rise

As of 2004, the U.S. catalyst market was growing at 4.5 percent per year, boosted by demand for catalysts that help to reduce pollution. The increase in global demand for alkylate by 2015 will reach an estimated 400,000 to 500,000 barrels per day (or the equivalent of 40 to 50 new alkylation plants). The AlkyClean process is economically competitive with existing hydrofluoric and sulfuric acid processes. Analysts predict that alkylate will gain market share as refineries update their processes to meet more stringent environmental regulations for fuels anticipated by 2010 to 2015. Furthermore, its commercial potential will grow as existing plants age. For example, energy companies are likely to find it more difficult to renew permits at existing hydrofluoric acid and sulfuric acid alkylation plants because of environmental, safety and homeland security concerns. These concerns could necessitate new plant construction or retrofitting where applicable.

Conclusion

ABB Lummus Crest (renamed ABB Lummus Global in 1995) successfully developed solid acid alkylation catalyst technology as an alternative to existing liquid acid processes that rely on corrosive and toxic hydrofluoric and sulfuric acids. ABB received one patent for this technology and published its findings in academic journals. ABB formed a joint venture with Akzo Nobel (a catalyst manufacturer, later acquired by Albemarle Corp.) and Fortum Oil and Gas (an energy company, renamed Neste Oil under a restructuring in 2005) to operate a successful demonstration plant from 2002 to 2004. At the prototype plant, they produced

alkylate at a reliably scalable fraction of a commercial plant that generally produces about 10,000 barrels per day. As of 2005, the joint venture was seeking agreements to establish the first commercial plants. Analysts predict that the alkylate market demand will grow by the equivalent of 40 to 50 new plants by 2015, due to rising environmental standards and increasing demand for fuel. Many of the existing 170 plants worldwide (most are in the United States) will need to be replaced or retrofitted within that time, as well, so the commercial potential for this technology remains strong.

PROJECT HIGHLIGHTS

ABB Lummus Global, Inc. (formerly ABB Lummus Crest)

Project Title: A Safe, Environmentally Friendly Method to Produce Alkylate (Thin-Film Solid Acid Catalyst for Refinery Alkylation)

Project: To develop a long-life, solid acid catalyst for use in the economical, environmentally friendly production of high-octane refinery alkylate.

Duration: 9/1/1995–8/31/1998

ATP Number: 95-05-0034

Funding(in thousands):**

ATP Final Cost	\$1,504	46%
Participant Final Cost	<u>1,800</u>	54%
Total	\$3,304	

Accomplishments: ABB Lummus Global, Inc. (formerly ABB Lummus Crest) achieved 100 percent of its technical goals:

- **Thin-film formation and catalyst activation.** ABB formed thin-film oxide or other catalyst precursors on inert support pellets and achieved desired acidity and stability.
- **Catalyst performance.** ABB demonstrated that solid acid alkylation resulted in higher selectivity than sulfuric acid alkylation. Selectivity means that the catalyst promotes desired reactions rather than undesired reactions, so that the highest purity product is achieved.
- **Testing solid acid catalysts.** ABB tested five catalysts and selected a proprietary catalyst developed by Akzo Nobel, which was the most efficient.
- **Catalyst regeneration.** Solid acid catalysts fouled quickly and were deactivated. A key milestone for this project was developing a means to regenerate catalysts (cleaning them and bringing them back to activity). ABB demonstrated good recovery of catalyst activity after each regeneration, with good stability.

- **Reactor engineering.** ABB developed models and commercial reactor concepts for its various catalyst samples. After selecting the best catalyst, they optimized the reactor system. They built a laboratory-scale reactor to simulate a commercial reactor.

After ATP funding ended, ABB continued its collaboration with Akzo Nobel, a catalyst manufacturer. Together, they formed a joint venture with Fortum Oil and Gas, an energy company, in 2001 to demonstrate and optimize the solid acid catalyst based alkylation process technology at a demonstration-scale plant located near an existing refinery. The demonstration plant operated from 2002 to 2004 (Fortum was renamed Neste Oil Corp. in 2005) with the following results:

- The joint venture developed "AlkyClean catalyst," which are solid acid catalyst pellets that are delivered to the refinery in plastic bags or metal drums. Use of the pellets eliminates the transporting, processing and handling risks associated with toxic and corrosive liquid acids.
- The solid-acid-catalyst-based AlkyClean process yielded high-quality alkylate, at a cost that is economically comparable to liquid acid processes.
- The solid acid catalyst process showed low sensitivity towards feedstock variation and impurities.
- Alternating cycles of alkylation and catalyst regeneration allowed the plant to operate continuously and maintain product quality.

ABB filed three patent applications from this ATP-funded technology, with the following one patent awarded:

- "Catalyst and method of preparation" (No. 5,935,889: filed October 4, 1996; granted August 10, 1999)

Commercialization Status: ABB has now proven and optimized solid acid alkylation at its demonstration-scale plant. Together with joint venture partners Albemarle Corp. and Neste Oil Corp., ABB is negotiating with energy companies to establish the first commercial plants, which will produce upwards of 10,000 barrels per day.

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

PROJECT HIGHLIGHTS

ABB Lummus Global, Inc. (formerly ABB Lummus Crest)

Outlook: The outlook for solid acid catalyst alkylation is strong. Alkylate can meet the rising demand for high-octane and low-emission gasoline. Solid acid alkylation is a safer, cost-effective alternative to existing hydrofluoric acid and sulfuric acid processes. Construction of 40 to 50 new alkylation plants is anticipated by 2015. In addition, as the existing 112 U.S. alkylation plants age, environmental safety and homeland security concerns will increase the likelihood of refiners choosing ABB's AlkyClean technology.

Composite Performance Score: * * *

Focused Program: Catalysis and Biocatalysis Technologies, 1995

Company:

ABB Lummus Global, Inc.
1515 Broad St.
Bloomfield, NJ 07003

Contact: Vincent J. D'Amico

Phone: (973) 893-2702

Subcontractors:

- Glatt Air Techniques, Inc.
Ramsey, NJ
- Department of Ceramics Engineering
Rutgers University
Piscataway, NJ
- Niro, Inc.
Columbia, MD
- Saint-Gobain NorPro Corp. (formerly Norton Chemical Process Products Corp.)
Akron, OH
- Vector Corp.
Cranbury, NJ

Publications: ABB researchers disseminated their findings through the publications listed below.

- Ercan, C., F. M. Dautzenberg, C. Y. Yeh, and H. E. Barner. "Mass-Transfer Effects in Liquid-Phase Alkylation of Benzene with Zeolite Catalysts," *Industrial and Engineering Chemistry Research*, v. 37, no. 5, 1724-1728, May 1998.

- D'Amico, V. J. "New Solid Acid Alkylation Process for Motor Gasoline Production," *ABB Review*, 71-76, February 2000.
- Raseev, S. D., D. G. Suci, and S. Raseev. *Thermal and Catalytic Processes in Petroleum Refining*. New York: Marcel Dekker, 2003.
- Dautzenberg, F. M. and P. J. Angevine. "Encouraging Innovation in Catalysis," *Catalysis Today*, August 2004.

Presentations: ABB researchers also shared their findings through the presentations listed below.

- Dautzenberg, F. M., H. E. Barner, and Maraschino, M. "Integration of Catalysis and Reaction Engineering for New Refinery Technology," CATCON'98, Houston, TX, June 1998.
- Barner, H. E., C. Ercan, A. Khonsari, J. T. Kwon, L. L. Murrell, A. Westner, and F. M. Dautzenberg. "Thin Film Solid Acid Catalyst for Refinery Alkylation," ATP Fall Meeting, Atlanta, GA, November 1998.
- Broekhoven, E. I., V. J. D'Amico, P. J. Nat, H. Nousiainen, and J. Jakkula. "The AlkyClean Process: A New Solid Acid Catalyst Gasoline Alkylation Technology," National Petrochemical and Refiners Association Annual Meeting, San Antonio, TX, March 2002.
- Dautzenberg, F. M. "Hydro- and Dehydrogenation of Large Volume Petrochemicals," Roermond Conference on Catalysis, Rolduc-Kerkrade, The Netherlands, June-July 2002.
- Dautzenberg, F. M. "Intra-reactor Process intensification," Symposium for Emerging Technologies, Antwerp, Belgium, May 2003.
- D'Amico, V. J., E. H. Broekhoven, and H. U. Nousiainen. "AlkyClean Solid Acid Alkylation: Will It Finally Become a Reality?" Akzo Nobel Catalysts SCOPE Symposium, Florence, Italy, June 2004.

Air Products and Chemicals, Inc.

Cheaper, More Efficient Oxygen Through Novel Production Method

The ability to separate oxygen from air has proven invaluable to many industries, because using pure oxygen in high-temperature furnaces improves their efficiency and reduces emissions. Although the benefits of using oxygen are substantial, its widespread use has been hindered by the high cost of oxygen-separation technologies. In 1992, Air Products and Chemicals, Inc. (APCI), a leading supplier of industrial gases, demonstrated a new approach to oxygen separation in the laboratory. APCI's approach was based on ion-transport membranes (ITMs) that efficiently produce high-purity (greater than 95 percent) oxygen at a high temperature. The company's new approach was limited to basic laboratory trials and had yet to be incorporated into a functional prototype. Therefore, in order to advance the research into this promising technology, APCI submitted a proposal to the Advanced Technology Program (ATP). In their proposal, they highlighted the potentially significant, industry-wide impacts of their proposed technology, as well as the technical risks.

In 1993, ATP awarded APCI cost-shared funding for a three-year project to advance the development of the ITM technology. Although APCI was not able to develop a functional ITM prototype by project end, the ATP-funded project did result in two achievements: the development of ceramic-steel seals and the development of designs and parameters to determine the most efficient method to remove contaminants from oxygen. These accomplishments helped to further APCI's research into oxygen-separation techniques by several years and encouraged the company's management and other agencies to support additional research and development for the ITM technology. The Department of Energy has supported the effort with a funding commitment for three separate three-year research phases between 1998 and 2007 to investigate new oxygen-separation methods.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

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Research and data for Status Report 93-01-0041 were collected during October - December 2001.

Significant Market Exists for Lower Cost, High-Purity Oxygen

In 1991, the daily production of gaseous and liquid oxygen in the United States was nearly 450 billion cubic feet, or about 51,500 tons. Costly high-purity (greater than 95 percent) oxygen was used in many industries such as steel, glass, and aluminum production and municipal waste incineration. These industries relied on oxygen for its ability to produce high temperatures essential to the efficient operation of furnaces and incinerators. The electronics industry used ultra-high-purity oxygen to produce a thin oxide layer on the

surface of silicon wafers during the fabrication of complementary metal-oxide semiconductors and other circuits. Power-generation and oxygen-enriched combustion applications used pure oxygen in the integrated gasification combined cycle. It was predicted that the total market would grow significantly if lower cost high-purity oxygen could be produced.

Other industries that would benefit from less costly pure oxygen included waste water treatment, pulp and paper manufacturing, lake and river revitalization, and hazardous waste incineration. While these sectors' use of oxygen was minor compared to the steel industry,

they represented potential high-yield growth in an area that would positively impact the environment. However, these new industries would only benefit from the use of oxygen if new, low-cost oxygen-separation processes could be developed.

Existing Oxygen-Separation Processes Hinder Widespread Adoption

The technology used for the commercial production of oxygen from air varied. For example, cryogenic distillation was the process used in large-tonnage production (100 to 2,000 tons per day) of high-purity oxygen. For small-tonnage production (less than 100 tons per day), the pressure swing adsorption (PSA) or vacuum swing adsorption (VSA) methods were used. Processes to produce less than one ton per day were also available. However, all of these methods had drawbacks that prevented cost-effective high-purity oxygen production, and they were prohibitively expensive for many potential applications.

Industries would only benefit from the use of oxygen if new, low-cost oxygen-separation processes could be developed.

Since 1989, Air Products and Chemicals, Inc. (APCI), a leading supplier of industrial gases, had been pursuing unique ways to separate oxygen from air. They believed that a low-cost oxygen-separation process for scale-up purposes could be developed. APCI's proposed approach was to incorporate an ion-transport membrane (ITM) into a prototype oxygen-separation unit. They believed that only the ITM technology could achieve totally selective separation and thus a pure oxygen product. Their proposed separation process involved compressing the feed air, pulling a vacuum on the product oxygen, or a combination of the two. While this process was similar to the other separation processes used at the time, APCI calculated that the use of mixed oxide ITM could result in a cost savings of at least 30 percent over PSA, VSA, or cryogenic distillation. That significant price reduction would allow numerous additional applications for pure oxygen, and the oxygen-production industry would be revolutionized.

Funding Support Needed for High-Risk Oxygen Separation

In 1992, APCI realized that if their research into ITM technology were to continue at a sustainable pace, they would need additional capital. Although APCI's management believed in the technology, they could not justify risking such a high percentage of their research and development (R&D) budget on a technology that was still 10 years from commercialization. In addition, the technical risks of this project were very high. Each step of the project would require the development and incorporation of many novel subcomponents into a finished product, each carrying its own obstacles and complications. Therefore, the company submitted a proposal to ATP in 1993 and was awarded a three-year project with approximately \$2 million in funding assistance.

APCI Identifies a Four-Phase Development Program

In 1989, when APCI began its R&D into the ITM technology, they envisioned a four-phase development program that would include the following:

Phase I: Fundamental Membrane/Process Development-internal funds

Phase II: Laboratory Prototype Development Unit (PDU)-with ATP funding help

Phase III: Technology Demonstration Plant-with Department of Energy (DOE) funding

Phase IV: Commercialization-internal funds

Phase I was solely funded by APCI and concluded in 1993. During that four-year phase, the company made significant technology advances in powder preparation of membrane compositions, dense-membrane fabrication, and thin-film-membrane deposition. Furthermore, APCI gained an increased understanding of the transport mechanisms of oxygen through the membrane. This, in turn, led to the development of proprietary membrane structures and ITM module designs.

APCI viewed phase II as a crucial step in the further development of the ITM technology. This stage included the design, construction, and testing of a laboratory

PDU. To help achieve its goal for this phase, APCI contracted with Ceramatec, Inc. to aid in the development of the ITM module, which was the key component of the proposed prototype. Ceramatec, a private company based in Salt Lake City, Utah, brought extensive ceramic expertise to the project.

APCI believed that only the ITM technology could achieve totally selective separation and thus a pure oxygen product.

As the ATP project progressed during phase II, APCI and Ceramatec collectively worked on the components that would be used in the PDU. Both companies faced problems in developing several components that were integral to the success of the project. Although APCI was not able to develop a functional PDU based on the ITM technology during the ATP project, the company did develop ceramic-steel seals for the PDU and the designs and parameters to determine the most efficient method to remove contaminants from oxygen.

Moreover, additional benefits from this ATP project included confirmation of the concept of a thin-cell membrane's approach to the small-scale development of proprietary technology (which is still being pursued) and an increase in the body of knowledge and experience in the technology, which is considered invaluable by APCI. The completion of phase II in 1997 marked the end of the ATP project.

Phase III was to have begun after the ATP project was completed and was intended to benchmark the work accomplished during phase II. The construction and operation of a commercial-scale membrane unit in the range of 1 to 10 tons of oxygen per day was to have been demonstrated.

Phase IV would have launched the full commercialization of the technology. Although APCI did not develop a functional PDU, their accomplishments during this project encouraged additional R&D support that continues today. APCI continues to strive for a 30-percent cost reduction for oxygen separation. Their current strategy is to begin commercialization initiatives after this reduction in cost is achieved.

Given APCI's progress during phases I and II, the company adjusted its original timeline. Rather than move forward with a technology demonstration plant in phase III and commercialization in phase IV, the company attracted DOE funding for additional research into creating an oxygen-separation system that could achieve a 30-percent cost reduction.

Additional Post-Project Support Advances ITM Technology

The collaboration between APCI and Ceramatec on the ITM technology continued after the ATP project, with APCI employees working on-site at Ceramatec. Furthermore, many additional participants are engaged with APCI to advance the technology and the knowledge gained during the ATP project, including Texaco, McDermott Technology, Eltron Research, Concept RERC, University of Pennsylvania, and Pennsylvania State University. Additional governmental agencies are also supporting further development of this technology.

APCI continues to leverage its ATP-funded work in its current project with DOE. The DOE project, which was awarded in 1998, is divided into three phases, each lasting three years. The first phase focused on selecting the final materials for the scale-up of a full-size ITM membrane. For phase I, the total investment by both APCI and DOE was \$25 million, with each contributing approximately \$12.5 million. The second phase of the project entails scaling up ITM modules, which comprise many small ITM membranes; demonstrating that the new modules can enable a 30-percent cost savings over conventional air-separation technologies; and illustrating that the production of five tons of high-purity oxygen a day is possible. The third phase will demonstrate that 25 tons of oxygen can be processed per day and that the new process can be used with gas turbines, an idea initially conceived during the ATP project.

Conclusion

Air Products and Chemicals, Inc. (APCI) began its research and development (R&D) of the ion-transport membranes (ITM) technology in 1989. By 1993, the company had invested approximately \$8.3 million in the development of the technology. The development of a

functional laboratory prototype development unit that used the ITM technology, which was the ultimate objective of the ATP-funded project, was not successful. However, the company did achieve two milestones during the project: the development of ceramic-steel seals and the development of designs and parameters to determine the most efficient method to remove contaminants from oxygen. These accomplishments have been instrumental in continuing the R&D for ITM technology. These seals and design and parameter methods, as well as the additional knowledge gained during the project, have enabled further R&D by APCI and others. To help disseminate this knowledge, the company published several research disclosures in two professional journals in March 1995.

The company attracted DOE funding for additional research into creating an oxygen-separation system that could achieve a 30-percent cost reduction.

In 1998, APCI entered into a contract with the Department of Energy to continue its R&D of this technology, and the company has partnered with several businesses to further its efforts. APCI is still committed to its original objective to develop an oxygen-separation technology that will generate high-purity (greater than 95 percent) oxygen with a 30-percent cost savings over traditional processes such as pressure swing adsorption, vacuum swing adsorption, and cryogenic distillation.

PROJECT HIGHLIGHTS

Air Products and Chemicals, Inc.

Project Title: Cheaper, More Efficient Oxygen Through Novel Production Method (Energy-Efficient Oxygen Production Using Novel Ion-Transport Membranes)

Project: To design, build, and test a laboratory prototype air-separation unit for producing high-purity oxygen using high-temperature ion-transport membranes (ITMs) that results in a 30-percent savings in cost over traditional methods.

Duration: 03/01/1994-02/27/1997

ATP Number: 93-01-0041

Funding** (in thousands):

ATP Final Cost	\$1,998	42%
Participant Final Cost	<u>2,721</u>	58%
Total	\$4,719	

Accomplishments: Air Products and Chemicals, Inc. (APCI) was not able to develop a prototype development unit (PDU) based on the ITM technology. However, the company developed ceramic-steel seals that will be incorporated into the PDU and developed processes to remove contaminants from oxygen. Moreover, APCI published research disclosures in March 1995 in two articles.

Commercialization Status: APCI is continuing its research and development (R&D) so that future commercialization is possible. However, the company does not intend to pursue commercialization initiatives until a 30-percent decrease in production cost is achieved.

Outlook: APCI, its subcontractor Ceramatec, its many additional business partners, and the Department of Energy (DOE) are continuing R&D of this technology. APCI has concluded the first phase of a three-phase project with DOE that began in 1998 and is expected to end in 2007.

Composite Performance Score: *

Company:

Air Products and Chemicals, Inc.
7201 Hamilton Boulevard
Allentown, PA 18195

Contact: Brian O'Neil

Phone: (610) 481-5683

Subcontractor:

Ceramatec
West Salt Lake City, Utah

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

Automotive Composites Consortium (a Partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)

Developing Structural Composites for Large Automotive Parts

In 1994, U.S. auto manufacturers believed that structural composites were the key to the future production of lightweight, fuel-efficient vehicles. Structural composites, which are blends of a polymer with glass fibers added for strength and stiffness, would result in a vehicle that weighed less and was more corrosion-resistant to one made with steel parts. In addition, one complex composite part could replace many steel subcomponent parts, thereby reducing assembly cost. U.S. manufacturers Chrysler, General Motors (GM), and Ford were collaborating as the Automotive Composites Consortium (ACC) to develop structural (load-bearing) polymer composite technology through high-risk, cooperative, pre-competitive research programs. Existing composite parts were limited in both size and load-bearing capacity. These parts could only be produced in low volumes due to high scrap rates and long production cycle times. The ACC researchers proposed to develop a prototype pickup truck box as an example of a large, strong, and durable structural component. Their research and development would require collaboration across disciplines and massive equipment. The ACC applied to the Advanced Technology Program (ATP) for a two-year project as part of the 1994 focused program, "Manufacturing Composite Structures." ATP awarded the funding in 1994 and the project began in 1995.

During this successful project, the ACC, along with suppliers, developed the processes, tools, and data necessary to produce innovative structural composite materials. The project to develop the truck box led to the establishment of the National Composite Center (NCC) in 1996, which was funded in part by the State of Ohio. After ATP funding ended in early 1997, the ACC continued developing the prototype truck box for three more years with support from the Department of Energy, NCC, and the three auto manufacturers. Extensive testing proved the pickup truck box's ruggedness and durability. Since 2001, GM and DaimlerChrysler (Chrysler merged with Daimler-Benz to become DaimlerChrysler in 1998) have commercialized several components using this technology, with more expected in the future. Ford has continued development, but has not yet commercialized products in the U.S. market. Benefits of this technology have extended beyond the auto industry. For example, the rugged composite material is being used for airplane parts, marine parts, and firefighter helmets.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

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Research and data for Status Report 94-02-0027 were collected during May – June 2004.

Composite Component Production Was Evolving

Composites are created by combining two or more materials to produce a new material that has improved properties. For example, the combination of a polymeric resin and glass fibers results in a material with

increased stiffness and strength. A wide range of automotive parts, such as soft seating materials and hard structural components, are made from polyurethane composites. Polyurethanes are formed when isocyanates (highly reactive derivatives of organic acids) are combined with polyols (alcohol-based

chemicals). One process used to manufacture parts made of polyurethanes is called reaction injection molding (RIM). During the RIM process, the combined material is quickly injected into a complex-shaped mold under heat and pressure. Varying the speed, pressure, and temperature produces components with different mechanical properties.

As early as the 1970s, the automotive industry used RIM to produce dashboards and body panel components that had a pleasing appearance and good paint-adhesion properties. By the 1980s, manufacturers were looking for ways to gain fuel efficiency by reducing vehicle weight while maintaining structural integrity. Typically, a 10-percent weight reduction results in an improvement in fuel economy of about three to seven percent. In order to produce load-bearing components, the next step was to incorporate RIM technology with a glass fiber mat or “preform” to produce a strong polyurethane-glass fiber reinforced composite. This process was called structural reaction injection molding (SRIM). In the 1980s and early 1990s, the polyurethane reaction was too fast to mold large parts. Manufacturers had produced some medium-sized simple parts (for example, a bumper beam support, floor pans, and seat backs), but these parts were not attractive, so they had to be used in applications that were not directly visible to the consumer.

By 1994, manufacturers wanted to make larger, attractive, and strong SRIM polyurethane composite components. However, major advances in molding technology and in glass fiber preform technology would be required in order to make parts that were durable enough to replace steel. The new SRIM parts would need to include glass fibers as reinforcement (the primary load carriers) and a matrix of the reacted polyurethane. The polyurethane surface would be visible to the consumer and needed a finished look.

Auto Manufacturers Propose a Prototype Pickup Truck Box

Chrysler, Ford, and General Motors (GM) were collaborating as the Automotive Composites Consortium (ACC) to develop structural polymer composite technology for automotive applications through high-risk, cooperative, precompetitive research programs. The consortium approached ATP in 1994 for funding under a focused program, “Manufacturing

Composite Structures.” They proposed a project to develop a manufacturing process that would include analyzing, designing, testing, and demonstrating high-volume, low-cost methods to manufacture a composite pickup truck box and tailgate assembly. This strong, durable, lightweight truck box would be the first of potentially many structural automotive body components. Composites resist dents, scratches, corrosion, and rust, which are typical problems in steel vehicles. Moreover, composite structures weigh less.

However, this precompetitive technology was unproven and entailed significant risk, because of the many design aspects that had to work together, such as fiber placement, preform molding, RIM processes, and the chemistries of polyurethane combinations. Scrap rates of 30 percent and long production mold cycle times of 20 to 40 minutes of typical liquid molding processes had to be reduced. If successful, this project would, for the first time, facilitate high-volume production of large, complex composite parts.

ATP approved the proposal and development started in 1995. The project relied on more than 20 subcontractors to provide materials, equipment, and testing. If successful, this newly established supplier base and infrastructure would ensure the rapid commercialization of many strong, durable, lightweight components after the end of the project. The new technology would help manufacturers and suppliers produce lightweight, fuel-efficient, cost-competitive automotive products, which would help to advance the U.S. auto industry’s global competitiveness. Manufacturing techniques developed during this project could also be applied to a broad range of additional products for aerospace, furniture, medical, and recreational applications.

Researchers Develop Glass Fiber and Polyurethane Processes

Consortium researchers began with computer-aided design (CAD) techniques and finite element analysis (FEA) to develop a virtual prototype truck box (see Figure 1). Software parameters needed to account for detailed part geometry, the properties of a glass fiber framework for the pickup truck box, and a polyurethane matrix to hold the fibers together and to make an attractive surface.



Figure 1. Computer-aided design of the truck box and tailgate. Ribs add strength and stiffness.

The ACC researchers had four primary objectives:

- Use glass-fiber preforming technology developed in a parallel ACC project. The researchers would combine the glass fibers and a binder (glue) on a screen to make the preform, which they would heat to 300°F to 400°F. They would move the preform to the mold where pressure formed it to its final shape.
- Develop processing technology to inject the polyurethane into the mold containing the preform and to adjust cure times, speeds, pressure, glass fiber contents, and temperatures.
- Develop joining technology using structural adhesives to integrate the composite part with the metal body of the truck.
- Develop technology for high-volume, low-cost production, which required experimenting with polyurethane chemical combinations and proportions.

Major advances in molding technology and in glass fiber preform technology would be required in order to make parts that were durable enough to replace steel.

Researchers required specific attributes of preforms:

- Conformability. The pickup truck box mold had a complex shape, and the glass fiber perform had to properly fit this shape. For example, the bottom surface was corrugated to add stiffness and strength.

- Loft (thickness). Researchers needed the preform to be compact, but to fully fill the space between the mold surfaces.
- Uniformity. Researchers needed the fibers to be oriented in many directions to achieve uniform strength and stiffness to meet performance requirements and to have the same fiber content throughout the part for good resin flow.
- Net Size. Researchers needed a preform that fit against the edge of the mold to avoid the need for in-mold or post-mold trimming, and to prevent resin from “racing” along the edge of the mold.
- Cycle time. Researchers required an adequate supply of preforms to match the proposed part production rate of 15 parts per hour.

Researchers Develop SRIM Technology

The ACC preforming operation provided high-quality, net-shape glass fiber preforms that needed no preparatory work prior to molding (see Figure 2 below). The use of high quality preforms is important to achieve a rapid SRIM molding process with a low scrap rate.



Figure 2. Loading a glass fiber preform onto the molding tool in preparation for polyurethane injection. For high-volume production the loading could be automated.

Researchers achieved the target four-minute cycle time to produce the pickup truck box and tailgate inner shell. They balanced polyurethane reaction characteristics (such as temperatures, gel time, and cure time), process conditions (including injection rates, number and location of injection ports, and press tonnage), and preform characteristics (such as fiber volume percentage and consistency).

The tailgate preform consisted of two glass-fiber preform shells wrapped around a foam core. The low-density inner foam core provided structural integrity to the molded part. Researchers were able to shape, trim, and assemble the tailgate shells in under the four-minute cycle time required. They optimized material properties to achieve weight and cost savings. They performed a study with the University of Detroit's Mercy Polymer Institute to reduce cycle times for the core, based on properties of polyurethane foam. Ultimately, they produced 50 dimensionally accurate foam cores. Future work would focus on chemical adjustments to the foam to reduce cycle time.

Three key parameters were identified for mold-filling characteristics: resin viscosity, preform permeability (allowing liquid polyurethane to pass through), and clamping force (pressure). During their work on varying pressure, the researchers discovered that too little pressure resulted in incompletely filled molds (see Figure 3 below). They proposed to use a 500-ton SRIM press, but a computer model showed it would provide too little pressure. They needed additional funding if they were to purchase a larger SRIM press. Fortunately, the State of Ohio provided significant funding to create a composite manufacturing demonstration facility in 1996 (during the ATP-funded project), called the National Center for Composite Systems Technology (later renamed the National Composite Center [NCC]). The ACC pickup truck box was the first project carried out at the NCC demonstration facility. In 1997, the NCC acquired a 1,000-ton SRIM press to use on the ACC project. However, additional development was still needed to complete the prototype pickup box. After the conclusion of the ATP-funded project in 1997, the ACC continued to work at the NCC. Researchers would eventually estimate that they needed a 2,000-ton press to consistently force the resin down the vertical walls.



Figure 3. Example of an incompletely filled box molding, which was a result of too little pressure.

Development Advances Prototype Pickup Box

Although the prototype pickup truck box was not yet completed when the ATP-funded project ended in 1997, the ACC and its suppliers had made significant progress and anticipated eventual success. They had accomplished the following: developed a unique design for an all-composite box assembly, populated a database of ACC-measured materials' properties; identified material, design, and processing parameters; fabricated a development plaque mold and production quality molds for the box and tailgate; identified an appropriate structural adhesive; and strengthened their supplier base capability. They received one patent for ATP-funded developments and shared knowledge with the industry through publications and presentations. They had equipment in place at the NCC and had identified the next steps to validate the performance of the prototype. For example, they knew that they needed a heavier press and that they would need to develop an "injection-compression" process in which polyurethane would be injected into the mold before closing the mold completely. No models existed to simulate the injection-compression process in detail. Researchers at NIST were attempting to create and verify a software tool that could do this. Subsequent to the ATP program the ACC received \$3 million in additional funds from the Department of Energy, as well as ongoing internal funding from each of the three auto manufacturers to continue the development. Moreover, they continued chopped-fiber preforming and SRIM molding development work at the NCC.

SRIM Composites Compare with Steel

SRIM composites have both advantages and disadvantages when compared to steel. Automating the SRIM processes decreased the initial cost of developing new complex-shaped, consolidated structural composite parts. Composites resist dents, scratches, corrosion, and rust, problems that have traditionally plagued pickup truck owners. Comparable steel parts would have to be assembled from multiple subcomponents that are welded together (and steel subcomponents require expensive stamping dies). Moreover, automated SRIM processes have a cost advantage for small production runs of composite parts for specialized vehicles. This is beneficial when auto manufacturers need to produce more variety and

specialization in models in order to meet consumer demand. Composites also weigh less. However, the lower cost of steel still provides an advantage in large production volumes. Furthermore, consumers have traditionally believed that steel is strong, while plastic is “cheap.” Manufacturers needed to market the benefits of lightweight, tough, durable, long-lasting composites to the public in order to further its acceptance.

Manufacturers Pursue Development, Testing, and Commercialization

In 1997, the ACC, supplier companies and NCC, continued developing the SRIM technology and met the goals of the program. The ACC prototype SRIM composite box weighed less than a corresponding structure in steel (a 36-pound savings on the pickup truck box, or 33 percent), met the performance requirements of a truck box, and had inherently better durability characteristics than steel. The ACC demonstrated the feasibility of its productivity goals and a cost model estimated comparable cost of a unique steel box produced at the target volume of 50,000 units per year.

In a proprietary program GM conducted pickup truck box testing and development in 1998 and 1999, investing more than \$60 million. Engineers tested 48 pickups in some of the worst operating environments in North America: phosphate and sulfur mines, chemical environments, and tar sand fields. They drove the trucks for two years and 1.2 million miles in temperatures that ranged from -40°F to 170°F. All the boxes sustained minimal damage, which validated the composite’s performance (“Composites Build a Tougher Truck,” *Composites Technology*, April 2002, pp. 32-36). GM’s SRIM composite pickup truck box won an award from *Popular Science* magazine in 1999 for the “Best of What’s New.” The magazine called it “a breakthrough in the use of structural composites.” GM released the truck box to the public as an \$850 option on the 2001 Chevrolet Silverado (see Figure 4). At the same time, GM released the SRIM composite midgate (a door that folds down to expand cargo room) on the Chevrolet Avalanche (standard on all models, 53,000 units were sold in 2001; 90,000 in 2002; and 93,000 in 2003). GM also released inner tailgate sections on the Cadillac Escalade EXT (also standard, 546 units were sold

in 2001, 13,000 in 2002, and 11,000 in 2003). The Silverado pickup truck box option was discontinued in 2003.



Figure 4. The Chevrolet Silverado pickup truck box and tailgate, manufactured from SRIM composite, were released to the public as an option in 2001.

Ford has also focused on the preform technology. The company has developed marketable products, but has not yet commercialized any products in the U.S. market.

DaimlerChrysler has commercialized floor covers for its 2005 minivans that are manufactured with SRIM technology. Its “Stow ‘n Go” system allows consumers to fold down the second- and third-row seats into the floor in the Chrysler Town & Country LX and the Dodge Grand Caravan SXT. DaimlerChrysler expects to sell more than 250,000 “Stow ‘n Go” minivans per year, which would be the highest volume application of this technology to date.

Although manufacturers have not calculated direct fuel savings from these components, as more composite components are implemented over time, the weight savings (15 to 33 percent) may result in noticeable fuel economy increases in the range of 4 to 20 percent.

Industries Discover New Applications for SRIM

In addition to automotive applications, benefits of the ATP-funded SRIM technology have also extended to other industries. Boeing is using SRIM composites to manufacture parts for the Air Force C-17 cargo plane. Compared to previous composite parts, the SRIM tail cone cost 80 percent less and a SRIM access door cost 46 percent less and was 9 percent lighter. It is anticipated that additional SRIM aircraft parts will be manufactured in the future. Boeing is expanding SRIM

composite use in its new 787 “Dreamliner” series passenger planes currently under development (see Figure 5 below). This is the first airliner with the majority of its large assemblies made of composite materials, including its 22-foot-wide fuselage and its super-efficient wings. Using SRIM composite parts results in a 3-percent improvement in fuel efficiency and an overall 20-percent fuel savings compared with the 747 model. The Dreamliner can carry up to 289 passengers, and its first commercial flight is anticipated in 2008.



Figure 5. Boeing’s 787 “Dreamliner” uses SRIM composites for structural parts, increasing fuel efficiency by 3 percent. Overall fuel savings is 20 percent compared with the 747. Its first commercial flight is anticipated in 2008.

SRIM composites have also been used in firefighter helmets manufactured by Lion Apparel. In addition to eliminating two steps in the manufacturing process, production efficiency was increased by 35 percent, the raw material inventory reduced, and labor costs reduced. The scrap rate was reduced from 20 percent to less than 3 percent. The resulting helmets were 15-percent lighter than previous composite helmets and had 15-percent greater impact resistance.

The prototype composite box weighed less than a corresponding structure in steel, met the performance requirements of a truck box, and had inherently better durability characteristics than steel.

SRIM composites have also contributed to benefits in the marine industry. SeaRay, a division of Brunswick Corp., uses SRIM technology to produce boat motor covers in response to upcoming U.S. Environmental Protection Agency emissions standards for the manufacturing process. By 2007, manufacturers must use closed molding processes to minimize volatile organic compound emissions. This technology replaces existing spraying in an open atmosphere.

The ACC and NCC continue to enhance SRIM technology by building on the ATP-funded developments. As of 2004, for example, they are examining the use of carbon fiber in place of glass fiber for a heavily stressed support piece used between the front and rear doors on a four-door sedan. Carbon fiber is stiffer and stronger than fiberglass and provides a weight savings of 50 to 60 percent over steel.

Conclusion

Chrysler (now DaimlerChrysler), Ford, and General Motors (GM) jointly formed the Automotive Composites Consortium (ACC) in 1988 to develop innovative polymer composite technology. They wanted to use this technology to reduce the weight and corrosion in automobile and truck parts compared with existing steel structural parts, because reduced weight would improve fuel economy. The ACC aimed to make composites more affordable. The group applied to ATP under a focused program, “Manufacturing Composite Structures” in 1994 for a two-year project to develop a prototype pickup truck box using a process called structural reaction injection molding (SRIM) as an example of a lightweight and tough structural component. This project began in 1995 and helped to establish the National Composite Center (NCC) in 1996 in Ohio, which focuses on developing cost-competitive composite materials and processes. Researchers produced net-shape glass fiber preforms; produced finished sample plaques; and met requirements for shape, thickness, strength, stiffness, and scrap. Following the conclusion of the ATP-funded project, the ACC and NCC acquired the needed equipment and successfully produced prototype pickup truck boxes in 1999. GM released the SRIM pickup truck box to the public as an option on the Chevrolet Silverado pickup truck in 2001, achieving a 33-percent weight savings over steel (however, they discontinued the option in 2003). The company simultaneously released standard parts for the Chevrolet Avalanche and Cadillac Escalade. DaimlerChrysler has also released commercial SRIM components for its 2005 minivans. Additional applications of this successful ATP-funded technology include strong, lightweight components for aircraft, firefighter helmets, and marine motor covers. Project researchers shared their developments through one granted patent and several articles and presentations. As public acceptance of tough, durable composites increases, applications are expected to broaden.

PROJECT HIGHLIGHTS

Automotive Composites Consortium (A partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)

Project Title: Developing Structural Composites for Large Automotive Parts (Automotive Composite Structures: Development of High-Volume Manufacturing Technology)

Project: To develop a composites manufacturing process called structural reaction injection molding (SRIM) into a cost-effective means to produce large automotive structural parts, such as the box of a pickup truck.

Duration: 3/1/1995 - 2/28/1997

ATP Number: 94-02-0027

Funding** (in thousands):

ATP Final Cost	\$2,575	47%
Participant Final Cost	<u>\$2,855</u>	53%
Total	\$5,430	

Accomplishments: With ATP funding, the Automotive Composites Consortium (ACC) successfully produced a prototype box for a pickup truck that is stronger and more durable than steel, does not rust, is visually attractive, requires no bed liner, and improves fuel efficiency through its light weight (36 pounds, or 33 percent, lighter than steel). This pickup truck box gave the ACC member companies (General Motors [GM], Ford, and Chrysler, which later became DaimlerChrysler) the knowledge and tools to develop commercial products and to continue innovative research, based on this initial success. In addition, the SRIM project contributed the following:

- ATP support of the project helped to establish the National Composite Center (NCC) in Ohio in 1996, which promotes, develops, and applies advanced composite technology to aerospace, defense, automotive, and commercial markets. NCC also received funding from the State of Ohio.
- ACC received \$3 million follow-on research funding from the Department of Energy from 1997 to 2000 to complete the program.
- NCC conducted related research with the Air Force Research Laboratory at Wright-Patterson Air Force Base for aerospace composite preform parts, beginning in 1997. They demonstrated significant cost reductions in 1999 for the Air Force C-17 cargo aircraft. For example, a tail cone cost 80 percent less, and a fighter access door cost 46

percent less and was 9 percent lighter than previous composite parts.

- GM won a "Best of What's New" award from Popular Science magazine for the Chevrolet Silverado composite pickup truck box in 1999; the magazine lauded it as "a breakthrough in the use of structural composites."
- ACC began work in 2001 at NCC on using carbon fiber in the B-pillar (the support piece between the front and rear doors) on a four-door sedan. Carbon-fiber-based SRIM is the next generation of lightweight, strong, and durable structural composite parts; they are stiffer and stronger than steel, with a 50- to 60-percent weight savings. A 10-percent overall weight savings is expected to increase fuel efficiency by 3 to 7 percent.

The ACC received one patent for SRIM technology from this ATP-funded project:

- "Self-contained constant stress/constant strain test fixture" (No. 5,798,463: filed February 12, 1997; granted August 25, 1998)

Commercialization Status: The original technology has been evolving through additional research and new applications. Some examples of commercial products that rely on the lightweight, high-strength, corrosion-resistant, and flexible qualities of this SRIM technology include the following:

- Pickup truck box and tailgate assembly for the Chevrolet Silverado went into production as an option in the 2001 model year, discontinued in 2003. The commercial box weighed 50 pounds less than the conventional welded steel box (33-percent savings). The tailgate is 15 pounds lighter than steel and has a 1,000-pound load-carrying capacity (compared to 600 pounds for steel).
- Midgate (a door that folds down to extend cargo space) for the Chevrolet Avalanche, beginning in the 2001 model year. Sold 53,000 units in 2001, 90,000 in 2002, and 93,000 in 2003.
- Inner tailgate sections for the Cadillac Escalade EXT hybrid sport utility vehicle, beginning in the 2001 model year. Sold 546 units in 2001, 13,000 in 2002, and 11,000 in 2003.

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

PROJECT HIGHLIGHTS

Automotive Composites Consortium (A partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)

- Floor sections for the "Stow 'n Go" system to fold down second- and third-row seats in the Chrysler Town & Country LX and the Dodge Grand Caravan SXT, beginning in the 2005 model year. Sales in excess of 250,000 units are anticipated.
- High-performance aircraft components by Boeing that include a C-17 airplane tail cone and an access door.
- Structural components on Boeing's 787 "Dreamliner" series passenger planes under development. Composites save 3 percent in fuel efficiency and contribute to an overall 20-percent fuel savings. Commercial flight is anticipated in 2008.
- Fire helmet shells manufactured by Lion Apparel (15-percent lighter than previous composite helmets with 15-percent greater impact resistance).
- Boat motor covers manufactured by the SeaRay Marine Division of Brunswick Corp. The SRIM process reduces volatile organic compound emissions compared with existing processes.

Outlook: The outlook for continuing the commercial development of the SRIM technology is good. As structural composite parts prove their strength and durability and as consumer demands for fuel efficiency increase over time, analysts predict that consumers will be more accepting of composite structural parts.

Composite Performance Score: * * * *

Focused Program: Manufacturing Composite Structures, 1994

Company:

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Company:

General Motors Research and Development
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Company:

Ford Motor Company
Research and Advanced Engineering
Manufacturing and Processing Department
2101 Village Road, MD3135
Dearborn, MI 48121-2053

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Subcontractor:

- Altair Engineering
Troy, MI
- American GFM
Chesapeake, VA
- American Sunroof Corporation
Southgate, MI
- ATI Systems, Inc.
Madison Heights, MI
- Bayer Chemical
Pittsburgh, PA
- Bucciero & Associates, PC
Troy, MI
- Chemstress Consultants, Inc.
Akron, OH
- Collins & Aikman (formerly Textron Automotive)
Americus, GA
- Delsen Testing Laboratories, Inc.
Glendale, CA
- Excel Pattern Works, Inc.
Dearborn, MI
- GenCorp Automotive Research
Akron, OH
- Gilchrist Metal Fabricating
Hudson, NH
- Hi-Tech Mold & Engineering, Inc.
Grand Rapids, MI
- H.S. Die and Engineering
Grand Rapids, MI
- Intelligent Structures, Inc.
Plymouth, MI
- Magna International
Southfield, MI

PROJECT HIGHLIGHTS

Automotive Composites Consortium (A partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)

- MSX International (formerly MascoTech)
Auburn Hills, MI
- National Composite Center
Kettering, OH
- RP/C Alliance Corp.
Southfield, MI
- SIA (Sovereign Specialty Chemicals, now part of Henkel)
Chicago, IL
- Structured Solutions
Troy, MI
- Tooling Technology Center
Windsor, Ontario, Canada
- Troy Tooling, Inc.
Rochester Hills, MI
- Mercy Polymer Institute
University of Detroit
Detroit, MI
- Weber Tools & Mold
Midland, Ontario, Canada
- SAMPE (publication and conference), Midwest Conference, Dearborn, MI, p. 342, September 12-14, 2000.
- Iobst, S. A., C. H. Mao, and D. L. Denton. "Use of Real Time Data Acquisition to Optimize the SRIM Process for a Pickup Truck Box." *Proceedings of SAMPE* (publication and conference), Midwest Advanced Materials and Processing Conference, Dearborn, MI, p. 355, September 12-14, 2000.
- "ACC Successfully Demonstrates SRIM Technology: ACC Development Leads to OEM Application F3P Fiber Preforming Used for the Aston Martin Vanquish." *USCAR Newsletter*, Spring 2001.
- "ACC Successfully Demonstrates SRIM Technology: Process Is Cost-Effective and Efficient; Pickup Box 25 Percent Lighter than Steel." *USCAR Newsletter*, Spring 2001.
- Brosius, D. "Composites Build a Tougher Truck." *Composites Technology*. p. 32-36, April 2002.

Publications: Researchers shared their knowledge about SRIM through the following publications:

- Lee, D. "National Composite Center Produces Pickup Truck Bodies." *Society for the Advancement of Material and Process Engineering (SAMPE) Journal*, March/April 1999.
- Romanchik, D. "Tests Characterize Composite Materials and Bonds." *Automotive Test Report*, May 1999.
- "USCAR Composite Technology Commercialized by GM." Press release from U.S. Council for Automotive Research (USCAR), February 2, 2000.
- "Truck Makers Choose Different Routes for Composite Cargo Boxes." *Composites Technology*, p. 19, March-April 2000.
- Denton, D. L., C. H. Mao, D. E. Willertz, N. G. Chavka, J. S. Dahl, E. D. Kleven, T. J. Dearlove, C. A. Di Natale, E. M. Hagerman, S. A. Iobst, J. A. Schroeder, and R. A. Bergen. "Development of a Cost-Effective SRIM Manufacturing Process for a Composite Pickup Truck Box." *Proceedings of*

Presentations: Researchers also shared knowledge through the following presentations:

- Denton, D. L., C. H. Mao, D. E. Willertz, N. G. Chavka, J. S. Dahl, E. D. Kleven, T. J. Dearlove, C. A. Di Natale, E. M. Hagerman, S. A. Iobst, J. A. Schroeder, and R. A. Bergen. "Development of a Cost-Effective SRIM Manufacturing Process for a Composite Pickup Truck Box." SAMPE Conference, Midwest Conference, Dearborn, MI, September 12-14, 2000.
- Iobst, S. A., C. H. Mao, and D. L. Denton. "Use of Real Time Data Acquisition to Optimize the SRIM Process for a Pickup Truck Box." SAMPE Conference, Midwest Advanced Materials and Processing Conference, Dearborn, MI, September 12-14, 2000.
- Iobst, S. A., C. H. Mao, and D. L. Denton. "Optimizing the SRIM Process for the ACC Composite Pickup Truck Box Molding," presented at the Lightweight Materials in Automotive Applications Workshop, Windsor, ON, June 4, 2001.

Bekaert Advanced Coating Technologies
(formerly Advanced Refractory Technologies, Inc., Advanced Coating Division)

Advanced DLN Films for a Wide Range of Industrial Needs

In the early 1990s, diamond-like nanocomposite (DLN) films, a new group of thin-film coating materials with unique and versatile properties, were developed by Russian scientists in collaboration with Advanced Refractory Technologies, Inc. (ART), a supplier of advanced materials and technologies, and universities and laboratories in the United States. In 1992, ART acquired exclusive rights to the DLN films and began to aggressively pursue their technical and commercial development.

By 1995, many potential applications for DLN films had been identified, and ART sought financial assistance to further explore these possibilities. After obtaining limited funding through private investors, ART applied to the Advanced Technology Program (ATP) and was awarded cost-shared funding in 1995. This project was a high-risk endeavor because the entire field of diamond-like film coatings was new at the time, and DLN technology was in an early stage of development. However, the domestic market potential for DLN coatings was estimated to exceed \$100 million annually. By the end of the ATP-funded project in 1997, ART had developed DLN technology and then successfully marketed it in 2000. That same year, the division of the company that produced diamond-like coatings was purchased by Bekaert Corporation (which later became Bekaert Advanced Coating Technologies). As of 2003, the company sells a number of products that utilize DLN technology.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* * *

Research and data for Status Report 95-01-0131 were collected during January - March 2003.

DLN Films Have Superior Characteristics

In the late 1980s, a new group of thin-film coating materials was discovered in Russia. By the early 1990s, these diamond-like nanocomposite (DLN) films were being developed by Russian scientists in collaboration with Advanced Refractory Technologies, Inc. (ART), a supplier of advanced materials and technologies, and universities and laboratories in the United States. DLN films are the most adaptable of thin films. They can be combined with a wide range of metals and ceramics. They also adhere to many substrates, are very hard, resist corrosion, remain stable at high temperatures, and can be produced with a broad range of electrical conductivities.

At the time, there were many thin films that were similar to DLN, including diamond and diamond-like carbon

(DLC). Although these competitors had many useful characteristics, they also had drawbacks. Diamond films were crystalline and very hard, and they conducted heat well. However, they did not adhere to many substrates, they did not easily accept other elements into the film coating, and they were expensive. DLC had characteristics ranging from amorphous (formless) to highly crystalline. It was used in applications such as Ray-Ban sunglasses. However, these films were also inflexible and exhibited relatively poor adhesive and electro-optical properties.

DLN Films Suitable for a Wide Range of Applications

Because DLN films had many superior characteristics, ART wanted to expand the industry's understanding of them and wanted to establish improved manufacturing

techniques for the films. Moreover, the company sought to develop targeted applications in four specific performance areas:

- Reduced friction in moving parts, such as bearings and seals
- Increased corrosion and environmental protection of photovoltaic solar panels and sensors
- Improved stability and performance of field-emission devices for flat panel displays
- Improved dielectric materials for X-ray lithography

The company's overall goal was to establish DLN technology as an economically viable process that could yield coatings with a high benefit-to-cost ratio in a wide range of applications. ART needed to meet this goal within a reasonable period of time to effectively compete with overseas manufacturers. The company's ability to minimize the number of iterations required for specific property combinations in DLN films and to demonstrate the manufacturability of new formulations would enable it to reduce the length of time to commercialize the technology.

DLN films were being developed by Russian scientists in collaboration with Advanced Refractory Technologies, Inc.

ART realized that the development of DLN technology was a high-risk endeavor. Diamond-like film coatings were new, and DLN technology was in an early stage of development. By 1995, the company had been able to obtain only limited funding for the development effort through private investors. Additional funding would enable ART to accelerate the technology development cycle, shorten the product optimization cycle, and increase the manufacturability and affordability of DLN film coatings. ART turned to government contract opportunities, but these programs were unable to provide the amount of funding the company needed. Therefore, in 1995, ART applied to ATP and was awarded \$2 million in cost-shared funding.

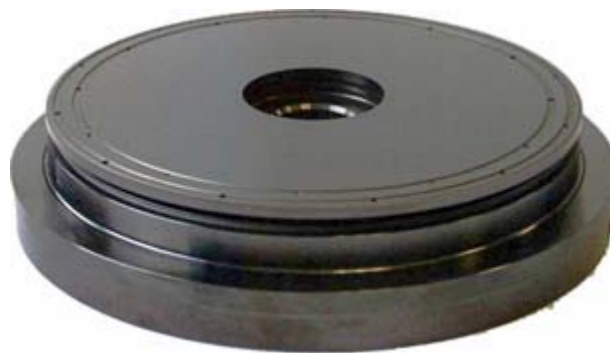
DLN Technology Promises Broad-Based Benefits

ART believed that once DLN technology was developed, its market value would exceed \$100 million

annually. The use of DLN to improve the tribological (i.e., reduced friction and wear) performance of materials was also expected to have significant market and economic implications.

ART Develops New DLN Technology

ART sought to develop, within a reasonable amount of time, an economically viable technology to produce coatings that would provide significant advantages in many applications. The company planned to accomplish this by gaining a better understanding of DLN films and their relationship to manufacturing processes and product properties, developing theoretical and empirical models, and improving the deposition process.



An example of a DLN coating on a mold for CDs.

By the end of the ATP-funded project, ART had successfully developed the DLN technology. The company had established specifications and film compositions for a variety of applications. It had also extensively characterized the film properties and performance in a large database, and it then used the data to devise optimal DLN coatings for specific applications.

ART also attempted to demonstrate that it could manufacture DLN coatings by achieving targeted deposition rates, yields, and utilization. The company succeeded in demonstrating specific deposition rates of DLN on silicon.

Post-Project Commercialization Is Significant Success

At the end of the ATP-funded project in 1997, ART attempted to commercialize the DLN technology through two applications: blades used in electrosurgery

(i.e., surgery conducted with electricity) and flat panel displays. For electrosurgical blades, the DLN coating was expected to provide several advantages over the two types of blades currently in use: blades that had no coating, which had a limited life, and blades coated with Teflon, which had a tendency to delaminate and leave residue after surgery. Conversely, DLN-coated blades provide a smooth cut, are reusable, have a long life, and require much less power for cutting. ART anticipated that the use of DLN-coated blades would be widespread because, at the time, more than 80 percent of invasive surgeries in the United States used electrosurgery. ART also believed that DLN-coated blades would help to improve the standard of care available in the healthcare industry.

In January 1998, after the conclusion of the ATP-funded project, ART established a business venture for electrosurgical blades and aggressively pursued manufacturing and scale-up plans. However, in 1999, after experiencing coating adhesion problems, the company decided against pursuing this venture.

ART attempted to commercialize the DLN technology through two applications.

For the flat panel displays application, DLN coatings can be used on spacers in field emission devices to improve picture clarity and appearance. ART aggressively pursued product development and hoped to design and construct a prototype that a U.S. customer could use to compete with overseas manufacturers in this multibillion dollar industry. However, the customer, which had been developing its own technology, ultimately decided not to pursue the DLN technology.

ART began to successfully commercialize DLN technology in 2000 by promoting its use in semiconductor applications. That same year, the division of the company that produced diamond-like coatings was purchased by Bekaert Corporation, a global group headquartered in Belgium, that had experience using DLN technology. In 1996, Bekaert had licensed the technology from ART for plastic molding applications in Europe, where the technology had been

used for several years. The new division became Bekaert Advanced Coating Technologies division.

From 2000 to 2002, the new division's revenue grew from approximately \$60,000 to well over \$1 million. To accommodate the increased demand for DLN-coated products, Bekaert began constructing a coating facility in Amherst, NY, that was four times the size of the facility used by ART. Bekaert also hired production personnel to meet the increased demand.

DLN technology could yield a high benefit-to-cost ratio in a wide range of applications.

In 2003, the division is selling a number of products with DLN coatings:

- Molds for CDs and DVDs in which the coating reduces abrasion and wear in the transfer of data, decreasing the need for machine maintenance
- Coatings on blow-molding components used in manufacturing plastic juice bottles where the coating is used inside the mold to reduce sticking and to retard plastic residue buildup, resulting in increased productivity and reduced maintenance
- Coatings for components used in semiconductor cluster tools

The division is also using DLN technology to coat tools used in metal-forming applications, such as soda and beverage cans. The DLN coating extends the life of the tool, resulting in increased productivity. According to Bekaert management, "The division has had an extraordinary growth rate. From 2001 to 2002, it was 120%. This year [2003], it has been slightly lower, but better than 50% growth is expected."

Since the ATP-funded project began in 1995, ART has filed for and been granted four patents for the DLN technology. The company has also made presentations and has published a paper about DLN's tribological, electrical, thermal-stability, and corrosion-resistance properties.

Conclusion

By the end of the ATP-funded project, Advanced Refractory Technologies, Inc. (ART) had successfully reached its goal of developing diamond-like nanocomposite (DLN) technology. ART had established specifications and film compositions for a variety of DLN applications. In 2000, ART sold the division that produces diamond-like coatings to Bekaert Corporation. In that same year, the new division of Bekaert (Bekaert Advanced Coating Technologies) began to successfully market DLN coatings. Today, in 2003, the division is selling a number of products, including coatings on molds for CDs and DVDs, coatings on blow-mold components used in manufacturing juice bottles, coatings on semiconductor process components, and coatings on metal-forming tools.

PROJECT HIGHLIGHTS

Bekaert Advanced Coating Technologies (formerly Advanced Refractory Technologies, Inc., Advanced Coating Division)

Project Title: Advanced DLN Films for a Wide Range of Industrial Needs (Diamond-Like Nanocomposite Technology)

Project: To develop scientific understanding and basic process-control information to enable the use of diamond-like nanocomposite (DLN) films, a new family of thin-film materials, in a wide range of industrial applications.

Duration: 8/1/1995-12/31/1997

ATP Number: 95-01-0131

Funding (in thousands):

ATP Final Cost	\$1,996	54%
Participant Final Cost	<u>1,677</u>	46%
Total	\$3,673	

Accomplishments: During this project, Advanced Refractory Technologies, Inc. (ART) successfully developed DLN coating technology. The company established improved manufacturing techniques for DLN films and developed several applications, such as electro-surgical blades and flat panel displays.

Since 1995, ART has been granted the following patents:

- "Capacitive thin films using diamond-like nanocomposite materials"
(No. 5,638,251: filed October 3, 1995, granted June 10, 1997)
- "Electrically tunable low secondary electron emission diamond-like coatings and process for depositing coatings"
(No. 6,013,980: filed May 9, 1997, granted January 11, 2000)
- "Fluorine-doped diamond-like coatings"
(No. 6,468,642: filed December 2, 1998, granted October 22, 2002)
- "Electrically tunable low secondary electron emission diamond-like coatings and process for depositing coatings"
(No. 6,486,597: filed October 21, 1999, granted November 26, 2002)

In addition, the company has published and presented five papers that describe the results of its work.

Commercialization Status: Bekaert Advanced Coating Technologies is currently selling a number of products with DLN coatings. These include components that are used in manufacturing CDs, DVDs, polyethylene terephthalate juice bottles, and metal cans and components used in semiconductor cluster tools.

Outlook: Since 2000, when Bekaert Advanced Coating Technologies began to successfully market the DLN coatings, the division has grown steadily, with commercial revenues exceeding \$1 million in 2002. The revenue growth, which has resulted in additional jobs at Bekaert, is expected to continue at a steady pace in 2003. According to Bekaert management, the coatings are "just now finding a market segment." The division is realizing the benefits of the research and development conducted during the ATP-funded project, and management "is confident that the technology will only mature and there will be more and more products."

In the future, Bekaert is considering other uses of the technology. For example, applications include using it on tools that produce pharmaceutical tablets to eliminate abrasion from the medicinal powder; for automobile engine components to decrease friction and increase the life of the parts; and for biomedical applications, such as surgical tools, to increase the efficiency and life of the tool.

Composite Performance Score: * * *

Number of Employees: 15 employees at project start in the division (ART), 18 at Bekaert as of January 2003.

Company:

Bekaert Advanced Coating Technologies
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Amherst, NY 14226

Contact: Chandra Venkatraman

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Bosch Corporation (formerly AlliedSignal Braking Division)

Casting Process Innovations to Lower Cost of Aluminum Automotive Parts

In the early to mid-1990s, the U.S. Government set a far-reaching goal for fuel efficiency at 80 miles per gallon by 2010. At that time, however, most automobiles achieved about 20 miles per gallon. In order to close the fuel-efficiency gap and also to meet the growing demand for reductions in emissions, automobile parts manufacturers began to examine using aluminum more extensively in the manufacturing process. Individual parts producers were examining methods to rid aluminum of porosity and oxide-related defects, but there was no concerted effort across the industry to fix the problem quickly.

To address this problem, the Advanced Technology Program (ATP) created a focused program, "Motor Vehicle Manufacturing Technology." The program would foster innovation in manufacturing processes that could slash time-to-market with major shifts in manufacturing techniques, such as the move to lightweight components for body and power train parts. AlliedSignal Braking Division (now Bosch Corporation) joined with two suppliers, Stahl Specialty Company (now ThyssenKrupp Stahl Company) and The Top Die Casting Company, to form a joint venture to reduce the cost of aluminum automotive parts and to speed their use for braking systems on automobiles and trucks.

The joint venture received an ATP award in 1995 to develop more efficient techniques to produce defect-free aluminum automotive parts. Unfortunately, the technical challenges were too numerous and too difficult to overcome, and the outlook for the technology is uncertain. However, the ATP-funded project eliminated the porosity problems, decreased the overall number of defects in aluminum, and enabled research into aluminum casting to advance two years ahead of where it would have been without ATP support.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

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Research and data for Status Report 95-07-0020 were completed during October - December 2001.

Inefficient Casting Techniques Increase Costs for Aluminum Parts

In the early to mid-1990s, the preferred methods of fabricating aluminum framework components for automobiles and trucks were die casting and permanent-mold casting. While these processes were expected to grow in importance as the demand for lighter vehicles increased, both processes imposed excess costs on the use of aluminum because they created significant amounts of pores and defects in the castings. Castings with exposed surface pores and defects are typically detected at the foundry and discarded before they are used. However, those castings with defects under the surface can only be

detected after machining and, sometimes, after assembly. The cost of discarding a machined casting includes the total cost of both the aluminum casting and the prior investment in machining. Moreover, the cost of discarding a fully assembled component is far higher. These costs could be dramatically decreased if the amount of pores and defects produced by the original casting operations could be reduced.

Increased Use of Aluminum Would Improve Vehicle Fuel Efficiency

By the mid-1990s, the national objectives of increased fuel efficiency and reduced vehicle emissions were forcing manufacturers to adopt lighter weight materials

in the fabrication of vehicle components and structures. Components such as wheels and brakes were specific targets for weight reduction, because a reduction in the mass of these components has a multiplicative effect in improving the vehicle's noise, vibration, and harshness performance, while reducing its fuel consumption. If the persistent, costly problems of impure aluminum could be solved, the metal, with its low density and high mechanical integrity, was expected to displace cast iron in the fabrication of numerous mechanical, hydraulic, and pneumatic components of passenger cars, light trucks, and heavy trucks. If the industry succeeded in creating a method for manufacturing aluminum that reduced the imperfections to just one small defect per part, the cost of using aluminum would decline dramatically, increasing its use in automobile and truck parts. The overall result would be lighter, more fuel-efficient vehicles.

AlliedSignal Partnered to Reduce Aluminum Defects

In 1995, AlliedSignal, a producer of parts for all types of automotive transportation, decided that parts manufacturers could reduce the cost of aluminum significantly faster through a concerted team effort. Therefore, AlliedSignal recruited two companies to form a joint venture project to correct the flaws in the aluminum-casting process. The first company, Stahl Specialty Company (now ThyssenKrupp Stahl Company), is a large firm that uses permanent-mold casting for aluminum. The second company, The Top Die Casting Company, is a small, privately held firm that uses die casting for aluminum. Both of these participants manufacture a variety of near net-shape aluminum castings for the U.S. car and truck industries. AlliedSignal used many of these parts to manufacture truck brakes, turbochargers, and automotive braking systems.

Die casting and permanent-mold casting imposed excess costs on the use of aluminum.

The impetus behind this joint venture was the ATP's focused program, "Motor Vehicle Manufacturing Technology," which targeted high-risk research projects to improve materials, assembly processes, and systems integration to reduce time-to-market for new vehicles.

The focused program called for reduced waste and accelerated cost reductions that would be achieved 200 percent faster than the current market rate.

The joint venture's proposed development project identified two tasks that were designed to reduce the concentration of pores and defects per part from an average of one large and three small defects to one small defect. AlliedSignal estimated that achieving this lower defect level would reduce the cost of aluminum manufacturing by 10 percent. The joint venture also proposed to create computational models of the die and permanent-mold casting processes to establish the "intelligent process" conditions and die designs that would minimize the porosity and defect concentration in a wide range of cast component shapes. These models would also generate improvements in the melting, holding, and casting practices in order to prevent defects from forming during the cooling process.

ATP Support Necessary for Joint Venture to Proceed

Without support from ATP, the three companies would not have been able to pool their resources and work together as a team. Internal business processes and priorities would have prevented a focused effort of parallel, complementary research that was designed to double the existing rate for achieving weight reductions in automobiles and trucks. The proposed joint venture met the criteria of the ATP focused program and addressed an extremely important challenge facing the suppliers of the U.S. automotive industry: to reduce the cost of cast aluminum components. The joint venture's proposal claimed that maintaining this cost improvement with the introduction of shorter production runs and new component designs would require developing and maintaining a synergy between the design and casting processes. In 1995, ATP awarded the joint venture \$0.9 million in cost-shared funds to establish this materials processing synergy and to work to reduce the overall cost of aluminum for automobile and truck parts.

Some Technical Risks Are Overcome, but Aluminum Not Yet Pure Enough

The technical challenges in reducing waste in die casting and permanent-mold casting are similar.

Establishing intelligent processes for casting, as well as devising improved techniques to prevent defects during cooling, had been unsuccessfully attempted at individual foundries. However, this joint venture hoped to succeed where others had failed by leveraging each entity's specific knowledge. Together, the joint venture participants generated robust, finite element models of each casting process in order to develop new die designs. These models related the controllable casting parameters to the microstructure, expected oxide inclusions, and residual stresses of cast aluminum to attempt to reduce pores that formed during the post-casting cooling process. As a result of these process improvements, oxide particulates and absorbed gasses were reduced significantly in the melting process (zero rejects for porosity).

Achieving this lower defect level would reduce the cost of aluminum manufacturing by 10 percent.

At the end of the ATP-funded project in 1997, the new processes had reduced the defects in cast aluminum from one large and three small defects per part to two small defects per part. Though this was a significant improvement in purity, the aluminum defects were still too prevalent to generate the cost savings needed to use aluminum in AlliedSignal's braking systems. The company was able to eliminate up to 85 percent of the defects from valve body designs using the new processes. While that reduction in defects and scrap was promising, other structures could not be produced as cost efficiently. Therefore, all products still required continuous testing and screening, and the new process did not result in cost reductions.

While the AlliedSignal joint venture was unable to produce multiple-part braking system products, there were some individual successes. The Top Die Casting Company reported that they used the new casting process to manufacture air brake valves, brackets, and other component parts, resulting in reduced scrap and reduced cost to the customer. They reported being one to two years ahead of where they would otherwise have been in aluminum-casting techniques. The company committed several hundred thousand dollars to research this process between 1997 and 2001, when they hoped to learn to cast aluminum in their cold

chamber machines. The Top Die Casting Company would not have undertaken this research without the base of knowledge acquired during the ATP-funded research. Additionally, Stahl Specialty Company reported using the ATP-funded tooling design on a separate product line that it began selling in late 1997.

Participants Shared Their Project Knowledge

Throughout the duration of the ATP-funded project, the participants shared their experiences and knowledge with others in the industry. The Top Die Casting Company published an article in *Modern Metals* magazine and attended several annual National Institute of Standards and Technology-sponsored meetings for the industry, where the company's representatives spoke with attendees about its findings. Stahl Specialty Company shared its knowledge through consulting and touring arrangements with other foundries, as well as through presentations at three conferences, including the American Foundry Society and the Aluminum Casting Research Laboratory.

All products still required continuous testing and screening, and the new process did not result in cost reductions.

In 1996, the AlliedSignal Braking Division was sold to a German company, Bosch Corporation, and in 1997 it became a subcontractor rather than a joint venture partner. Although the company participated fully as a subcontractor and performed to expectations, it was not in a position to share its findings.

Conclusion

Although the AlliedSignal joint venture sought to create improved braking systems for automobiles and light trucks by decreasing the total cost of aluminum production, the project did not reach its overall goals. Aluminum production costs were not reduced by 10 percent, because the new process could not achieve the target reduction in defects that would eliminate the costly inspection and rejection processes that follow machining. Research into processes to reduce aluminum defects did continue after the close of the ATP-funded project, although no new product lines resulted.

PROJECT HIGHLIGHTS

Bosch Corporation (formerly AlliedSignal Braking Division)

Project Title: Casting Process Innovations to Lower Cost of Aluminum Automotive Parts (Low-Cost, Near Net-Shape Aluminum Casting Processes for Automotive and Truck Components)

Project: To develop design and process innovations in die casting and permanent-mold casting to minimize microstructural defects, thereby improving the quality and lowering the cost of cast aluminum components for cars and trucks.

Duration: 9/30/95-12/30/97

ATP Number: 95-07-0020

Funding (in thousands):

ATP Final Cost	\$ 898	36%
Participant Final Cost	<u>1,589</u>	64%
Total	\$ 2,487	

Accomplishments: Participants in this joint venture were unable to reach their goal of a 10-percent reduction in the cost of aluminum manufacturing as a step toward increasing overall fuel efficiency in automobiles and trucks. However, they were able to create a synergy between design and casting processes that resulted in the following accomplishments:

- Elimination of porosity problem (zero rejects for porosity)
- Reduction from one large and three small defects per part to two small defects per part
- Acceleration of research by two years ahead of where it otherwise would have been through parallel research efforts
- Reduction of defects in a specific type of valve body design by up to 85 percent
- Commitment of hundreds of thousands of research dollars by The Top Die Casting Company to continue the ATP-funded research between 1997 and 2001; they reported being one to two years ahead of where they would otherwise have been in aluminum-casting techniques

- Manufacture of air brake valves, brackets, and other component parts by The Top Die Casting Company, resulting in reduced scrap and reduced cost to the customer
- Small process improvement at Stahl Specialty Company

In addition, the Top Die Casting Company published an article in *Modern Metals* magazine and attended several annual National Institute of Standards and Technology-sponsored meetings. The Stahl Specialty Company made presentations at three conferences, including the American Foundry Society and the Aluminum Casting Research Laboratory.

Commercialization Status: The technical challenges of this project were too numerous and difficult to overcome. As a result, AlliedSignal created no new products for brakes using the technology developed under the ATP-funded project. The Top Die Casting Company produced some components using the new processes, such as air brake valves and brackets. Stahl Specialty Company used one step of the aluminum manufacturing process to assist in aluminum filtration. That process had a small impact on several of the company's product lines.

Individual joint venture participants used the technology and process improvements developed through this ATP-funded project to create new castings with a reduced number of defects. However, the number of defects was not low enough for commercial acceptance. After additional research and development, the individual participants hope to be able to commercialize aluminum automobile parts that meet the 10-percent cost-reduction goal. As of December 2001, however, major research initiatives had ended in favor of other industry priorities, though improved aluminum purity remains a priority for the future of the industry.

Outlook: Due to continuing technical challenges in producing purer aluminum through the casting process, the outlook for this technology is uncertain. Improvements in purity did occur, and Stahl Specialty Company's manufacturing process did improve somewhat. However, it is unclear whether the industry will continue to build upon these innovations.

PROJECT HIGHLIGHTS
Bosch Corporation (formerly AlliedSignal Braking Division)

Composite Performance Score: * *

Focused Program: Motor Vehicle Manufacturing
Technology, 1995

Companies:

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Stahl Specialty Company
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Contact: Rich Andriano
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The Top Die Casting Company
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Contact: Joe Pieve
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BP Amoco (formerly Amoco Corporation, Resource Center)

Process to Lower the Cost of Olefin-Paraffin Separations

Light olefins, which are unsaturated, chemically active hydrocarbons, are the principal building blocks for the petrochemical industry. They are produced in greater quantities than any other organic chemicals and form the basis for many chemical products such as polyesters, surfactants, and antifreeze. Before they can be used in a product, light olefins such as ethylene and propylene must be purified by separating them from paraffins (a residue obtained from certain petroleum crudes). Traditionally, the U.S. olefins industry has used distillation processes to separate olefins from paraffins, but these processes are difficult and costly due to the extreme temperature and pressure conditions that are usually required.

In the 1970s, Amoco Corporation became interested in the facilitated transport process as an alternative to distillation processes. After conducting research, the company was able to demonstrate the technical feasibility of this process; however, it also experienced problems with its use. The complexing agent, silver nitrate, was costly, and cellulose acetate hollow-fiber bundles used for the contactor (the vessel used to bring the substances into contact) were not strong enough. Amoco suspended its research efforts, but by 1993, the price of silver had decreased and hollow-fiber technology had improved, making this an opportune time for the company to continue its research of the facilitated transport process. The project, which would also involve the testing of new chemical agents, was too risky for private investors, but the company believed it would be a good fit for the Advanced Technology Program (ATP). That year, Amoco applied for and was awarded cost-shared funding from ATP to develop a low-cost process for separating light olefins from the corresponding paraffins using facilitated transport and microporous polypropylene hollow-fiber contactors. Amoco believed this effort could result in increased earnings of more than \$60 million a year for the company from additional olefin recovery. Net benefits to U.S. chemical process industries were expected to approach approximately \$1 billion a year.

By the project's end in 1997, Amoco had successfully developed a high-efficiency contactor that could be used with silver nitrate. The company had also, with its subcontractor SRI International, developed two new complexing agents that were cost competitive with silver nitrate when used for facilitated transport. In spite of these successes, in 1998 Amoco discontinued its research into the new technology. The company had experienced costly operating problems with the new process and was unable to demonstrate its economic feasibility.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* *

Research and data for Status Report 93-01-0234 were collected during February 2003.

Early Tests of Facilitated Transport Identify Problems

Much of the cost of producing light olefins is associated with purifying the olefins by separating them from

paraffins (the residue from certain petroleum crudes). This step has traditionally been performed by distillation, which is usually achieved by refrigerating the olefins and paraffins at very low temperatures or by using trays to separate olefins from paraffins of the same carbon

number. Both methods are costly. Cryogenic refrigeration is capital and energy intensive, and the refrigeration compressors are expensive to maintain. Distillation with trays is capital intensive because it requires the use of many trays.

In the late 1970s, Amoco Corporation began to examine ways to improve its olefin-paraffin separation process in order to reduce costs. The company became interested in facilitated transport, a less costly alternative to distillation. With facilitated transport, a chemical agent, such as silver ion, is used to form a chemical association with electrons in the olefins. This association, formed inside a contactor, results in a water-soluble complex of olefins, which is transferred from the contactor to a second vessel where it is separated by lowering the pressure inside the vessel and/or by adding heat. Neither step requires an extreme of pressure or temperature.

To test this new technology, Amoco set up a facilitated transport pilot plant for olefin-paraffin separations. At the pilot plant, the company demonstrated the technical feasibility of the process; however, using silver nitrate as the complexing agent at high temperatures was costly. Moreover, cellulose acetate hollow-fiber bundles, which were used as the gas-liquid contactor in the process, weakened and burst with prolonged contact with the aqueous silver nitrate complexing agent. Because of these problems, Amoco suspended its development of the technology.

Market and Technological Changes Encourage Renewed Research

By 1994, the price of silver had decreased and advances had been made in hollow-fiber technology. Polypropylene fibers were available and contactors made from these fibers were rugged and could withstand high differential pressures, thereby improving the efficiency of olefin-paraffin separation. Based on these changes, Amoco believed it should resume its research and development effort to achieve a breakthrough in olefin-paraffin separation costs.

Amoco's goal was to develop a commercially viable process for separating and purifying light olefins, especially ethylene and propylene, using facilitated transport and microporous polypropylene hollow-fiber

contactors. The company also wanted to develop new water-soluble complexing agents.

Amoco recognized that developing this process would involve a high level of risk because the technology was new and would rely on the use of as-yet undeveloped chemical complexing agents. However, if successful, the company anticipated it would earn more than \$60 million per year from additional olefin recovery. Net benefits to the U.S. chemical process industries overall were expected to eventually approach \$1 billion per year. In addition, the cost savings resulting from this new technology would enable the U.S. olefins industry to gain a sustainable competitive advantage in the global marketplace.

Due to the risk involved in the project, as well as its potential for significant broad-based benefits, Amoco applied to ATP in 1993 for funding assistance and was awarded \$1.25 million for a three-year project.

New Separation Process Promises Broad-Based Benefits

Amoco believed that developing a facilitated transport process for olefin-paraffin separations would affect a wide range of technologies in the United States. The U.S. industries that would directly benefit from decreasing olefin costs included the polymer and petrochemical industries, as well as the packaging film, wire and cable coatings, toys, waste bags, chewing gum, squeeze bottles, electrical insulation, piping, blow-molded products, and injected-molded products industries.

Amoco's goal was to develop a commercially viable process for separating and purifying light olefins using facilitated transport

The lower cost and increased efficiency of olefin-paraffin separations would also increase the global competitiveness of the U.S. olefins industry. In 1994, the United States was the world's largest producer of ethylene, with an annual capacity of approximately 35 billion pounds. The global demand for this chemical was nearly 140 billion pounds per year and was predicted to grow by 4.4 percent through 1995. However, other areas of the world, including East Asia and the Middle

East, were steadily increasing their capacity to produce ethylene; this was expected to have a significant effect on the U.S. ethylene industry.

Emerging processes such as natural gas utilization, which was unprofitable at the time, would realize significant benefits from facilitated transport. Several of the technologies used to convert natural gas into liquid transportation fuel were capital intensive and yielded low rates of return. A facilitated transport process could greatly enhance the economics of these technologies to enable the production of olefins from domestic natural gas instead of imported oil. Thus, the success of Amoco's proposed research would not only help maintain the competitive position of U.S. industries, but could also lower U.S. dependence on imported oil.

Scientific and Technical Issues Are Identified

In order to make facilitated transport separations commercially viable, Amoco needed to address several critical scientific and technical issues. First, the complexing agent had to be stable, had to demonstrate chemical integrity, and could not degrade when heated. The complexing agent also had to be immune to poisoning by acetylenes and sulfur compounds, because process streams in the petrochemical industry were always contaminated with such compounds. Subcontractor SRI International had identified transition metal compounds that were thermally and chemically stable. The derivatives of these compounds could yield improved complexing agents, but developing an economical, large-scale synthesis of such compounds would be a technical challenge.

Cost savings resulting from this new technology would enable the U.S. olefins industry to gain a sustainable competitive advantage in the global marketplace.

Second, large, rugged contactor units containing porous polymer hollow fibers, which demonstrated high, sustainable olefin fluxes when processing impure olefin feedstreams, had to be developed. A porous polypropylene contactor manufactured by the Celgard Division of Hoechst Celanese was a promising candidate, but development work was needed to

demonstrate that it could be used in an industrial process stream for an extended period of time. One of Amoco's key concerns was the swelling of the fibers by aromatic hydrocarbons that were likely to be present in industrial process streams.

Third, integration of the facilitated transport process into existing olefins plants had to be tested. Successful pilot integration with an industrial facility was necessary for Amoco to realize the potential cost savings.

Improved Complexing Agents Tested at Bench-Scale Facility

To accomplish its goal, Amoco built an experimental bench-scale unit to test the separation of olefins from paraffins in various Amoco process streams using the facilitated transport process. At the same time, SRI conducted research to develop improved complexing agents. SRI was to synthesize and screen up to 10 new complexing agents and to deliver enough of the most promising complex for testing in Amoco's bench-scale unit.

In Amoco's experimental unit, two fiber bundles were used in an absorber/desorber arrangement. The operating temperature of each bundle was controlled through recirculating baths. A vacuum was used in the desorber to allow olefins to escape from the complexing agents while liquid pumps circulated the aqueous complexing agent solution.

In preliminary experiments, silver nitrate was used as a complexing agent because even though it was expensive, Amoco knew it would work. As other complexing agents became available through research at SRI, they were tested in the experimental unit. Initially, Amoco used synthetic mixtures of hydrocarbons to simulate the various process streams used in olefins plants and oil refineries. Poisoning studies were conducted by spiking the feeds with anticipated poisons (e.g., sulfides, acetylenes, or amines). These poisoning studies were considered essential in establishing the structural integrity of the complexing agents under the demanding conditions of an industrial environment. As progress was made, the company obtained actual feed samples from the plants and tested them in the experimental facility.

Facilitated Transport Requires Further Research

By the end of the project, Amoco had achieved two important objectives. First, silver nitrate could be used as a facilitating agent in the high-efficiency contactors manufactured by the Celgard Division of Hoechst Celanese. These contactors exhibited economic olefin fluxes and purity when processing impure olefin feedstreams in both bench-scale tests and initial pilot plant tests at an industrial site. Second, SRI had successfully developed two new complexing agents that were water soluble, were able to bind olefins under realistic operating conditions, and were cost competitive with silver nitrate when used for facilitated transport. Amoco tested each complex and deemed one candidate, molybdenum sulfide dimer, the best choice for further work because it had superior solubility, chemical stability, and shelf life.

Amoco was unable to demonstrate the economic feasibility of using this new technology.

However, work still remained on integrating the facilitated transport process into existing olefins plants. A pilot plant demonstration in a commercial olefins plant had to be completed; contactor and facilitating agent lifetimes had to be determined to accurately assess the economic viability of the technology; and a detailed productivity response to key process variables, such as temperature, pressures, transmembrane pressure, and flow rates, had to be precisely measured to determine optimal operating conditions.

Also, Amoco needed to demonstrate that the alternative complexing agent candidate was effective on both the smaller bench scale and the pilot plant scale. Finally, additional research on poison resistance and more effective complexing agents was needed. This area demonstrated great promise, but the technical risks were still quite high.

Amoco Discontinues Separation Process Development

For a year after the ATP funding ended, Amoco continued its research of facilitated transport for olefin-paraffin separations using both the silver nitrate complexing agent and molybdenum sulfide dimer. In

work on the technology. Although the process was technically sound, the company was experiencing costly operating problems. The hollow-fiber contactors were not robust enough, and the candidate complexing agent was expensive. Furthermore, the hollow fibers and limited thermal stability failed to alleviate the problems with silver nitrate that the company had experienced prior to the project, such as susceptibility to sulfide and acetylene poisoning. Thus, Amoco was unable to demonstrate the economic feasibility of using this new technology for olefin-paraffin separations.

Knowledge Disseminated Through Presentation and Patents

Based on its work on the ATP-funded project, Amoco made a presentation entitled "Facilitated Transport Process for Low-Cost Olefin-Paraffin Separations" at the ATP Catalysis, Biocatalysis, & Separations Technology Review in October 1996. Amoco has also been granted three patents as a result of its work on the project.

Conclusion

By the end of the ATP-funded project, Amoco had achieved several of its objectives. It was able to use silver nitrate as a facilitating agent in high-efficiency contactors and had developed a promising new complexing agent that would potentially cost less than silver nitrate when used for facilitated transport. However, the company was unable to demonstrate the commercial viability of separating olefins from paraffins using facilitated transport. Thus, it did not commercialize the technology. Nevertheless, Amoco believes that the knowledge it gained from the ATP-funded project, especially about chemical agents, may be useful in future studies on separation.

PROJECT HIGHLIGHTS

BP Amoco (formerly Amoco Corporation, Resource Center)

Project Title: Process to Lower the Cost of Olefin-Paraffin Separations (Facilitated Transport Process for Low-Cost Olefin-Paraffin Separations)

Project: To capitalize on recent advances in chemistry and hollow-fiber manufacture to develop a new, low-cost process for separating light olefins from the corresponding paraffins.

Duration: 4/15/1994-4/14/1997

ATP Number: 93-01-0234

Funding (in thousands):**

ATP Final Cost	\$1,250	50%
Participant Final Cost	<u>1,250</u>	50%
Total	\$2,500	

Accomplishments: By the end of the project, Amoco had achieved the following objectives:

- Developed a high-efficiency contactor that could be used with silver nitrate and established olefin flux targets under realistic operating conditions. The target flux, recovery, and separation factors were achieved and surpassed at the laboratory bench scale.
- Developed two new promising complexing agents through a subcontract with SRI International. After testing each complexing agent, Amoco decided to continue development of one of them, molybdenum sulfide dimer, because of its superior solubility, chemical stability, and shelf life.

In addition, Amoco made a presentation entitled "Facilitated Transport Process for Low-Cost Olefin-Paraffin Separations" at the ATP Catalysis, Biocatalysis, & Separations Technology Review in Boulder, Colorado in October 1996.

Since 1998, Amoco has been granted the following patents:

- "Unsaturated hydrocarbon separation and recovery process"
(No. 5,744,685: filed October 15, 1996, granted April 28, 1998)

- "Unsaturated hydrocarbon separation and recovery process"
(No. 5,863,420: filed October 15, 1996, granted January 26, 1999)
- "Process for synthesis of molybdenum sulfide dimers"
(No. 5,962,364: filed July 30, 1997, granted October 5, 1999)

Commercialization Status: The chemical agents that were developed during this ATP-funded project have not been commercialized.

Outlook: At this time, it is unclear whether the chemical agents developed during this ATP-funded project can be economically applied to olefin-paraffin separations with facilitated transport. However, Amoco believes that the knowledge gained about the chemical agents may be useful in future studies on separation.

Composite Performance Score: * *

Company:

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Contact: Vince Kwasniewski

Phone: (630) 961-7403

Subcontractor:

SRI International
Menlo Park, CA

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

Catalytica Energy Systems (formerly Catalytica, Inc.)

Development of Improved Catalysts Using Nanometer-Scale Technology

In 1994, eight percent of the U.S. gross domestic product was attributed to the chemical and petroleum-refining industries, which produce gasoline, lubricants, diesel fuel, polymers, plastics, and chemical intermediates and products. Of particular concern in these industries was the efficiency of catalysts in a manufacturing process. Catalysts control and enhance the speed of chemical reactions. Improvements in catalyst efficiency could increase product yields, decrease waste byproducts, and potentially lower energy consumption associated with a given process. Catalytica, Inc. and Microfluidics International Corporation formed a joint venture to address this issue. Potential spillover applications of the improved catalysts could benefit the biotechnology, pharmaceuticals, cosmetics, chemicals, coatings, electronics, and food industries. As small companies, Catalytica and Microfluidics alone could analyze only a few catalyst applications by measuring their outcomes. Understanding how a new manufacturing approach could improve catalysts required comprehensively studying the reaction parameters, such as solution concentrations, solution feed rates, and level of acidity or alkalinity. This could only be done with outside funding.

Catalytica and Microfluidics applied to the Advanced Technology Program (ATP) in 1994 and were awarded cost-shared funds in order to complete a three-year study of a new process concept to improve catalyst manufacturing. Beginning in 1995, the two companies worked with collaborators and subcontractors, which included several major chemical and petroleum manufacturers, to test the concepts. The companies developed and patented a new catalyst production reactor, called a Multiple Stream Mixer/Reactor (MMR), which produced solid catalyst materials that were more efficient and shared knowledge through publications and presentations. However, the technology was sometimes incompatible with customers' existing manufacturing methods, and Catalytica (later renamed Catalytica Energy Systems) abandoned the technology in 1999 due to lack of funding for continued development. Microfluidics continued developing the MMR and commercialized it in 2004 for laboratory-scale use. Future commercial potential will focus on manufacturing phase-pure, uniform nanomaterials in diverse applications, such as pharmaceuticals, superconductors, pigments, and ceramics. The company anticipates it will have an industrial production model ready for the market by 2005 or 2006.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* *

Research and data for Status Report 94-01-0190 were collected during October-November 2004.

Catalyst Inefficiencies Result in Waste

The chemical and petroleum-refining industries produce gasoline, lubricants, diesel fuel, plastics, and chemical intermediates and related products. The fuels produced are essential to heat our buildings and to operate the nation's cars, trucks, buses, aircraft, trains, and boats.

Other petroleum-derived products are used in antifreeze, tires, cleaning products, cosmetics, polymers, and plastics. In 1993, these industries generated global annual sales of more than \$400 billion, with one-third from U.S. manufacturing and sales. This accounted for approximately eight percent of the U.S. gross domestic product.

Catalysts are a key element in the processes used to manufacture many chemical and petroleum products. They control and enhance the speed of chemical reactions by using small amounts of a stable substance, such as metal oxide, which remains unchanged at the end of the reaction. Catalysts enhance the speed of the chemical reaction to transform one chemical into a useful new chemical, a process called conversion. The use of commercial catalysts in production began in 1936, and since then, the industry has adopted many processes, using hundreds of catalysts to increase efficiency in producing fuels and chemicals (John S. Magee and Geoffrey E. Dolbear, *Petroleum Catalysis in Nontechnical Language*, 1998). In most conventional chemical reactors, inadequate mixing and conversion rates can limit the value and performance of a chemical reaction. As a result, product yields are sub-optimal, and unwanted by-products are produced, resulting in higher costs. Improving catalysts is often a key to more efficient chemical reactions.

Catalytica Proposes to Manufacture More Active Catalysts

Catalytica, Inc. and Microfluidics International Corporation formed a joint venture in 1994 and submitted a proposal to ATP, seeking cost-shared funding to pursue improved catalyst manufacturing. The Catalytica team proposed to collaborate with a subcontractor, Worcester Polytechnic Institute, to develop more active catalysts, which would increase the product-manufacturing rate and/or achieve higher product quality. A more selective catalyst would produce fewer by-products as well as improve raw material utilization, optimize energy efficiency, and reduce purification and disposal costs.

One approach to improving the efficiency was to reduce the catalyst's particle size. Reducing particle size provides increased surface area, which speeds the chemical reaction, because more sites on the particle become available to promote the desired chemical reaction. For example, consider a cube of sugar that is approximately 1.6 cm on each side (or about 2.5 cm² surface area on each of the six faces of the cube).

The total surface area of the powdered sugar cubes is increased to about 2,500 cm², larger than a standard newspaper page. Similarly, greater catalyst surface area increases the potential reaction rate.

Team Proposes to Use Microfluidizer

One form of catalyst manufacturing involves a catalyst emulsion (with the consistency of whipping cream) that is pumped through a nozzle and sprayed into a large tank swept with hot air. The water in the emulsion evaporates quickly and solid catalyst particles fall to the bottom of the tank. The technical details of forming these precipitating solids are complex; they include managing solution feed concentrations, solution feed rates, temperature, and solution pH range (degree of alkalinity or acidity). Successful catalysts are generally made from mixtures of elements, usually a metal, metal oxide, or metal sulfide that catalyzes a desired reaction. Small amounts of other elements are added to further improve desired reaction rates. These additives are called promoters (to speed up desired reactions) or poisons (to slow down undesired reactions that produce waste).

Catalytica and Microfluidics proposed to use an existing instrument called a Microfluidizer to produce catalyst precursors in a crystallized form. These precursors could be produced in sizes ranging from 5 to 50 nanometers (a nanometer is 1/1000 of a micrometer). The two companies believed that using the Microfluidizer would result in catalysts that have greater chemical purity and structural uniformity. The Microfluidizer had already been used to emulsify liquids, disperse solids, de-agglomerate particles (split them apart), and rupture biological cell membranes. At the time, Catalytica and Microfluidics researchers believed that similar results could be obtained to improve manufacturing of catalysts for oil and chemical refining.

Project Success Could Lead to Increased Energy Efficiency and Cost Savings

If successful, the technical advances from this project would also change the processing infrastructure from

the traditional large mixing tanks to smaller units with lower capital costs, resulting in reduced catalyst manufacturing costs. Catalysts with greater purity and uniformity would have higher selectivity and conversion rates when used in chemical reactions. If catalyst selectivity (the ability to promote the desired reaction) could be enhanced, then purification, waste, and disposal costs could be reduced, and energy efficiency and resource use could be optimized. For example, in a typical commodity chemical plant that produces 200 million pounds of product per year, a five percent improvement in selectivity would reduce waste emissions by 10 million pounds per year and save several million dollars annually in raw materials and fuel costs.

Catalytica and Microfluidics faced significant risks. On their own they would be limited to addressing a few catalyst opportunities by focusing on proprietary problems defined by a limited set of clients, with fewer chances of a more enabling technical success. ATP support would allow a comprehensive process analysis. Learning to understand and manipulate the processes would be essential to extending and optimizing the technology for catalyst precursor preparation and understanding the impacts of catalyst structure on catalyst performance. ATP awarded the joint venture partners cost-shared funding as part of a general competition in 1994, and the project began in 1995.

Project Team Develops a New Reactor

The Microfluidizer, a mechanical device initially developed by Arthur D. Little, Inc. worked by combining two chemical solutions, forming two streams of the premixed solutions, and forcing the streams to collide and mix under high speed, pressure, and shear (particles in the solutions hitting each other repeatedly at different angles with each collision). The result was uniform nanometer-scale precipitated solid particles. However, after Catalytica and Microfluidics started testing, they discovered that some of the synthesis methods used by catalyst manufacturers proved to be incompatible with the Microfluidizer.

Furthermore, as researchers pursued this technology, they found an unexpected problem. The Microfluidizer allowed the solutions to make contact before entering the reaction chamber, which caused the reaction to begin too soon. The Microfluidizer was able to produce good texture, distribution, and uniformity, but it was not efficient at controlling the chemistry. Solutions began precipitating before entering the chamber, and some of the desired high-speed, high-shear impact was lost.

Catalytica and Microfluidics proposed to pursue development of a new mixer/reactor that would keep the solution streams separate until they entered the reaction chamber. The new reactor could apply intense mixing to the precipitate-forming reaction itself, not before, thereby permitting fast, controlled, continuous reaction chemistries. The reactor would allow the development and manufacture of nanomaterials. The two solutions would enter the inlet, pass through high-shear microchannels under high pressure, and collide in an intense energy field. Company researchers predicted that resulting materials with nanometer-scale dimensions would have smaller crystallite size (affording more surface area), improved homogeneity, and greater chemical purity. Developing the mixer/reactor became an important focus of the project.

Researchers Target Key Industrial Catalysts

During the ATP-funded project, Catalytica and Microfluidics analyzed the catalysts used in the chemical and oil-refining markets. They made business/research contacts with 10 major chemical and catalyst manufacturers who agreed to perform much of the testing at their facilities, which would reduce internal staff and equipment costs for the Catalytica team. These potential customers were, at the same time, informal research collaborators. If successful, these customers would purchase the technology from Catalytica and Microfluidics. Together, they studied reactions to produce the following materials:

- Mixed metal oxide catalysts used for synthesizing chemicals, with a goal of five-percent yield improvement.
- Nanocrystalline zeolites used for catalytic cracking of petroleum to make gasoline components, with a goal of 25-percent productivity improvement.
- Colloidal-size catalysts used for residuum processing (residuum are the oil products that remain after petroleum has been distilled), with a goal of 10 cents/barrel cost reduction.
- Catalysts with controlled phase purity and reduced crystallite size for syngas manufacture. Syngas (or synthesis gas) is a mixture of carbon monoxide and hydrogen that is generated in coal gasification and some types of waste-to-energy facilities. Syngas is an intermediate step of natural gas to manufacture liquid fuels and other chemical products. Hydrogen is also produced from natural gas as an intermediate step in manufacturing basic chemicals.
- Catalyst support materials used to improve surface area and porosity (these included gamma alumina, diaspore, and a low thermal expansion ceramic precursor).

A five-percent improvement in selectivity would reduce waste emissions by 10 million pounds per year and save several million dollars annually in raw materials and fuel costs.

Initial Results Are Limited

While Catalytica and Microfluidics developed the new reactor, they also continued working with each of the customer/research collaborators. Progress was difficult because of their customers' shifting priorities and deadlines. Although some progress was made, customers decided to either pursue the work internally or abandon the technology. The results of their research to produce catalytic materials are described here:

- **Mixed metal oxide catalysts for synthesizing chemicals.** Catalytica and Microfluidics prepared formulations of copper-zinc-aluminum mixed oxides. They were able to improve the mixing, and the resulting crystallite sizes were significantly smaller, with improved phase purity. This was one of the major developments of the program, which provided incentive to pursue other catalyst systems. The team made two efforts with mixed metal oxides:
 - Together with one chemical company collaborator, they worked to improve control of precipitation. The new mixer/reactor provided no catalytic benefit. The company chose to pursue internal projects instead.
 - Together with a second chemical company, they worked to increase reaction rates through the use of fine-grained particles and further reduced the size with the new mixer/reactor. Preliminary results showed some difference in catalytic performance, but not dramatic improvements. The technology was incompatible with the collaborator's in-place synthesis methods, so the project was abandoned.
- **Nanocrystalline zeolites.** Hydrocracking is used to produce superior-quality, stable lubricants. The process works by reacting a petroleum feedstock with hydrogen, in the presence of a catalyst, at high temperatures (400-425°C) and pressures (3000+ psi [pounds per square inch]). The process eliminates impurities. The team made two efforts to perform hydrocracking with zeolites:
 - Together with one oil company, they worked to synthesize zeolite Y, which had a large potential market. Processing with the Microfluidizer increased the rate of high-quality, zeolite synthesis, but only marginally decreased crystallite size.

The researchers achieved some enhancements in catalyst yield, but test product samples had limited reproducibility.

- Together with a catalyst manufacturer and a chemical company, their initial results showed significant stability advantages, although follow-up testing was less favorable. The chemical company discontinued the project.
- **Colloidal catalysts.** Catalytica and Microfluidics worked with another oil company to develop improvements in catalysts used in residuum processing. Researchers sought to provide “drop-in” catalyst replacements, but this effort was abandoned because margins for improvement were too small to be cost effective.
- **Syngas catalysts.** In principle, the smaller the crystallite size, the higher the activity of the catalyst. However, very small copper crystallites are unstable in syngas reactions. The team had to balance initial activity and long-term stability. They made two efforts in the area of syngas catalysts:
 - Together with one catalyst manufacturer, they created promising test materials with high catalyst stability.
 - Together with a chemical company, they evaluated the synthesis of copper-zinc-aluminum mixed oxide catalysts. They prepared a series of catalyst samples, and resulting materials showed superior activity maintenance. However, the chemical company ultimately decided to proceed with internal developments instead of pursuing the ATP-funded technology.
- **Catalyst supports**(diaspore and low thermal expansion ceramic precursors). These are fine white powdery substances with high fusing temperatures used in ceramic coatings.

- The team reduced diaspore synthesis times from days to a few hours. Catalytica proposed to construct a continuous pilot-scale unit for a catalyst support manufacturer, but the manufacturer declined.
- A small company wanted to develop specialty low-thermal-expansion ceramics as exhaust port liners for diesel engines. They needed a low-cost route to produce the material in 100-kilo quantities. Researchers investigated two compositions: one based on barium-zirconium-silicon phosphate and another based on calcium-strontium-zirconium phosphate. Ultimately, the effort was discontinued due to the extensive washing steps needed to purify the material.

By 1998, at the end of the ATP project, Catalytica and Microfluidics had developed a prototype mixer reactor, called the Multiple Stream Mixer/Reactor (MMR), and used it with their customers/research partners. They received three patents for this technology and disseminated knowledge in several academic journals and presentations. The project continued for another year with approximately \$350,000 of internal joint venture funding. However, without the financial backing of a large, full-time research team, they were unable to make the strides necessary to validate the technology. The researchers still believed that the basic premise of the project was valid: ultraturbulent mixing of two or more solutions would result in precipitation and increased homogeneous nucleation (the initial stage of precipitate formation) with a larger number of small crystallites of precipitate. However, they were unable to prove this during the project’s limited timeframe. Customers who manufactured catalysts were hesitant to outsource their research and development, and some of their existing synthesis methods proved to be incompatible with both the Microfluidizer and the new MMR. Catalytica (later renamed Catalytica Energy Systems) abandoned the technology in 1999, but Microfluidics continued.

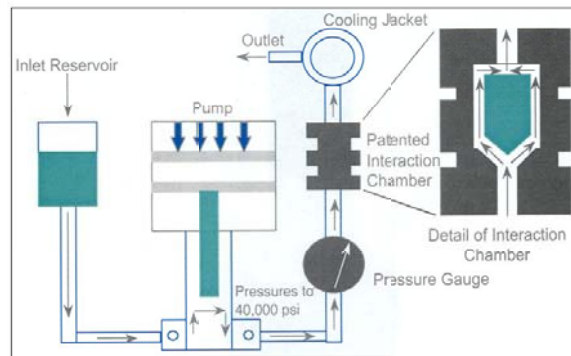
MMR on the Brink of Success

Commercialization was difficult in a competitive, commodity environment, but Microfluidics (later a division of MFIC Corporation) continued to develop the MMR. After some delay due to internal business changes, the market began to express interest in their reactor. In 2003, the company performed testing for a biotechnology firm using the ATP-funded technology.

Catalytica and Microfluidics had developed a prototype mixed reactor, called the Multiple Stream Mixer/Reactor (MMR), and used it with their customers/research partners.

MFIC introduced its patented MMR to the market in 2004 as a continuous chemical reactor (see illustrations below). Each solution feed stream reaches velocities of 80 to 300 meters per second. The two streams collide and combine efficiently in a patented reaction chamber. Increasing the level of supersaturation (higher than normal solute concentration dissolved in a solvent) through high-intensity mixing can result in an increased nucleation rate and a decrease in crystallite size of the precipitated product. In addition, intense mixing also increases homogeneity in pH and/or concentration. As a result, the properties of the product, in terms of phase purity, uniformity of composition, and uniformity of crystal size, are also improved. The MMR may become a standard device for conducting chemical reactions, especially to produce nanoparticles. This degree of reaction chemistry can reduce waste and lead to efficient manufacturing of new nano-structured materials in scalable quantities.

Irwin J. Gruverman, Chairman and CEO of MFIC, said, “[The MMR] permits creation of incredibly small structures that allow reactions to occur very rapidly at the nanometer scale, and that produce the smallest, most uniform and purest reaction products... [W]e can produce these nanomaterials quickly, phase-pure, and very uniform... We will enable new products, improve old products, and... revolutionize the way people do reaction chemistry.... The [MMR] advantages include much lower capital cost, because it is a continuous process unlike the batch processes normally used today to perform reaction chemistry.



Photograph and schematic of MFIC's patented MMR System, which permits fast, controlled, continuous reaction chemistries. The MMR aids the development and manufacture of nanomaterials. Two streams of liquid enter the inlet, pass through high-shear microchannels (up to 40,000 psi), and collide in an intense energy field (see schematic). Resulting materials have uniquely small crystallite size, improved nano-scale homogeneity, and improved chemical purity

Our technology is more energy-efficient, is easier to control and facilitates making pure material with minimal contamination.... The higher operating pressure allows us to put much more energy into the stream and get more uniform, stable microemulsions and nanosuspensions.”

In addition to catalysts, potential applications for materials produced in the MMR include the following:

- **Pharmaceuticals.** Solids can be recrystallized, purified, and then dispersed as nano-suspensions in an injectable medium. Nebulized (liquid converted to a fine spray) inhalable drugs can be prepared with nanoparticles.
- **Superconductors.** Particle uniformity at the nanometer scale improves performance.
- **Photographic media.** Resolution and performance depend on crystalline component size and uniformity.

- **Planarization abrasives.** In semiconductor manufacturing, it is critical that deposition layers be free of defects. Abrasive crystals are used to process the underlying surfaces of semiconductor layers.
- **Pigment synthesis.** Paint quality (such as appearance and wear) depends on uniform particle pigment properties.
- **Ceramics.** Nanoparticles minimize thermal expansion (increase in size due to an increase in temperature) and maximize isotropic strength (uniform strength in all directions).
- **Ultrapure chemicals.** Chemicals are recrystallized more completely by ultraturbulent processing.

MFIC commercialized its first laboratory MMR system in 2004, and its first major production MMR system is scheduled for commercialization in 2005 or 2006. The MMR can be custom-built to meet research and production needs. The laboratory system has a capacity of 10 gallons per hour, and MFIC projects that production systems will have throughputs of 200 gallons per hour and higher. The company demonstrated the MMR at the Massachusetts Institute of Technology's Emerging Technology Conference in September 2004 as a technology "poised to make a dramatic impact on the world."

MFIC introduced its patented Multiple Stream Mixer/Reactor to the market in 2004 as a continuous reactor.

Conclusion

Catalytica, Inc. (now Catalytica Energy Systems) and Microfluidics (now MFIC Corporation) formed a joint venture in 1994 to develop a comprehensive methodology to improve catalyst manufacturing. Catalysts are used to speed chemical reactions. More active catalysts could be used in the oil refinery and chemical manufacturing industries to increase energy efficiency and reduce waste in the manufacturing process.

The companies applied to ATP for cost-shared funding in 1994, and ATP awarded funding for a three-year project, beginning in 1995. The Catalytica team analyzed reactions for five types of catalysts, with some positive results. They used their knowledge to develop a new mixer/reactor, called a Multiple Stream Mixer/Reactor (MMR), which enabled fast, controlled, continuous reaction chemistries for smaller and purer product particles (nanometer scale, down to billionths of a meter). They received three patents for these advances and shared their results in academic journals and presentations. MFIC commercialized the MMR in 2004 for laboratory use and anticipates offering a manufacturing-scale model in 2005 or 2006. The resulting nanoparticles may be useful for many industries such as biotechnology, pharmaceuticals, cosmetics, chemicals, coatings, and electronics.

PROJECT HIGHLIGHTS

Catalytica Energy Systems (formerly Catalytica, Inc.)

Project Title: Development of Improved Catalysts Using Nanometer-Scale Technology

Project: To develop and demonstrate the manufacturability of catalysts with enhanced activity and selectivity for use in the chemical and petroleum-refining industries.

Duration: 1/16/1995–3/31/1998

ATP Number: 94-01-0190

Funding (in thousands):

ATP Final Cost	\$1,994	47%
Participant Final Cost	<u>2,262</u>	53%
Total	\$4,256	

Accomplishments: While the ATP-funded project failed to meet its original goal to increase the productivity of catalysts in the chemical and petroleum-refining manufacturing processes, Catalytica and Microfluidics did make the following technical advances in enhancing catalyst production:

- Using the existing Microfluidizer equipment, improved the mixing, homogeneity, and phase purity; resultant catalyst crystallite sizes were significantly smaller.
- Developed a prototype mixer/reactor (Multiple Stream Mixer/Reactor [MMR]) by 1998 and began testing with customer/research partners. MMR allowed fast, controlled, continuous reaction chemistries. Resulting catalysts had uniquely small crystallite size, improved nano-scale homogeneity, and improved chemical purity.
- Produced an improved MMR, in which two solution streams enter, pass through high-shear microchannels under high pressure (up to 40,000 pounds per square inch), and collide in a unique reaction chamber.

Microfluidics was awarded three patents for this ATP-funded technology:

- "Use of multiple stream high pressure mixer/reactor" (No. 6,159,442: filed August 5, 1998; granted December 12, 2000)

- "Multiple stream high pressure mixer/reactor" (No. 6,221,332: filed August 5, 1998; granted April 24, 2001)

Commercialization Status: Microfluidics' MMR may prove to be a valuable tool for the emerging nanotechnology sector, producing nanoparticles for many industries. The company sold its first laboratory MMR system in 2004 and expects the first major production MMR system to become available in 2005 or 2006.

Outlook: The outlook for MMR technology is good but clouded. Although Catalytica and Microfluidics found it difficult to enter the petroleum and chemical-refining markets, their research may benefit other industries. For example, the ultra-fine particles resulting from the MMR are potentially useful in cosmetics, pharmaceuticals, paints, and coatings.

Composite Performance Score: **

Numbers of Employees: 120 employees at project start, 85 as of December 2000 (Catalytica); 40 employees at project start, 42 as of March 1998, and 47 as of December 2004 (MFIC Corporation).

Company:

Catalytica Energy Systems (formerly Catalytica, Inc.)
430 Ferguson Drive, Bldg. 3
Mountain View, CA 94043

Contact: David Ginter
Phone: (650) 960-3000

Company:

MFIC Corporation (formerly Microfluidics International Corp.)
30 Ossipee Road
Newton, MA 02464

Contact: Irwin J. Gruiverman
Phone: (617) 969-5452

Subcontractor:

Worcester Polytechnic Institute
Worcester, MA

PROJECT HIGHLIGHTS

Catalytica Energy Systems (formerly Catalytica, Inc.)

Publications:

Catalytica and MFIC researchers disseminated their findings through the following publications:

- Moser, W. R., B.J. Marshik, J. Kingsley, M. Lemberger, R. Willette, A. Chan, J.E. Sunstrom IV, and A. Boyce. "The Synthesis and Characterization of Solid States Materials Produced by High Shear Hydrodynamics Cavitation." *Journal of Materials Research*, Vol. 10: 2322-2335, 1995.
- Gruverman, I. J. "Production of Nanostructures under Ultraturbulent Collision Reaction Conditions – Applications to Catalysts, Superconductors, CMP Abrasives, Ceramics, and other Nanoparticles." *Mat. Res. Soc. Symposium Proceedings*, Vol. 775, 2003.
- Gruverman, I. J. "Ultraturbulent Reaction Technology." *Drug Delivery Technology*, Vol. 3, No. 1, January 2003.
- Microfluidics demonstrated the MMR at the juried Massachusetts Institute of Technology's Emerging Technology Conference as a technology "poised to make a dramatic impact on the world." September 29-30, 2004.

Presentations:

They also shared knowledge through the following presentations:

- Kilner, P. H. "Liquid Acid Replacement." Solid Acid/Base Conference, Houston, TX, February 26-28, 1995.
- Moser, W. R., J. E. Sunstrom, and B. J. Marshik-Geurts. American Chemical Society, Anaheim, CA, April 4, 1995.
- Ginter, D. M., D. L. King, and P. H. Kilner. "Use of a High Intensity Multistream Mixer for the Precipitation of Catalyst Precursors Having Reduced Crystallite Size and Improved Phase Purity." American Institute of Chemical Engineers (AIChE), Miami Beach, FL, November 1998.
- Ginter, D. M., D. L. King, and P. H. Kilner. "Effect of a Novel Multistream Mixer/Reactor in Reducing Mass transfer Limitations in a Model Fast Chemical Reactions." (AIChE, Miami Beach, FL, November 1998.

Crucible Materials Corporation

Corrosion-Resistant Steel Through Powder Metallurgy Process

In the 1990s, intergranular stress corrosion cracking in alloys was a growing problem in the United States. In this type of corrosion, cracking occurs along the grain boundaries (anomalies, or uneven portions) of an alloy when it is both under stress and in a corrosive environment, such as salt water. While the need to utilize the earth's oceans for fuel, minerals, energy, and food was increasing, the ability to effectively use this natural resource would require materials that were more resistant to salt water corrosion and less expensive than those currently available. The only alloys that demonstrated resistance to corrosion in seawater were expensive and often difficult to weld or fabricate.

Crucible Compaction Metals, a powder metallurgy production facility and division of Crucible Materials Corporation, estimated that the annual economic loss from corrosion was approximately \$80 million. This included preventative actions, replacement and loss of products, and safety and product liability issues. To limit these huge economic losses, the company proposed to use a new design methodology to manufacture high-strength, high-nitrogen stainless steel that had superior intergranular stress corrosion cracking resistance in seawater and other high-chloride environments. In 1995, the company was awarded cost-shared funding from the Advanced Technology Program (ATP) to develop new alloys for stainless steel using the company's proposed process.

By the end of the three-year ATP-funded project in 1998, Crucible had developed high-nitrogen alloys that could improve the performance of stainless steel and had identified potential commercial applications for one stainless steel, SS100. Due to the cost of developing and marketing SS100, the company decided not to commercialize it at that time. Crucible is interested in developing and commercializing SS100 in the future and has continued to consider different markets for it, including the boat shaft and medical implant markets.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* *

Research and data for Status Report 94-01-0287 were collected during February-March 2003.

Crucible Proposes Improved Powder Metallurgy Process

In the 1970s, steel manufacturers were adding up to 0.12 percent nitrogen to improve the strength and corrosion resistance of stainless steel. Since then, alloys with nitrogen contents as high as 0.5 percent have been produced, further increasing corrosion resistance and strength. However, as increasing amounts of nitrogen are added to an alloy, the molten components cool and segregate into different phases (crystal structures), making it difficult to shape the alloy.

In the mid-1990s, Crucible Materials Corporation created a mathematical model to predict the corrosion resistance of steel alloys. This model predicted that increasing the nitrogen content should lead to superior corrosion resistance. Crucible proposed to develop a powder metallurgy process, using inert gas atomization to produce powder particles, followed by rapid solidification to achieve the desired homogeneous chemistry. In this process, powder is created by impinging high-pressure gas onto a molten metal stream to disintegrate the metal into drops that rapidly

form solid spheres upon cooling. Crucible believed that the powder metallurgy process, which had already been successfully used to produce segregation-free high-alloy tool steel with no workability problems, could also be applied to the development of an alloy for high-strength, high-nitrogen stainless steels.

Mathematical Model Used to Predict Experimental Alloys

Crucible's goal was to use both its mathematical model, based on thermodynamic data from recent literature, and rapid solidification technology to manufacture high-strength, high-nitrogen stainless steel that is resistant to intergranular stress corrosion cracking in seawater and high-chloride environments. To accomplish this, the company planned to use its mathematical model to create a matrix of high-nitrogen, iron-based alloys that were predicted to have high corrosion resistance. Crucible would melt and atomize 50 pounds each of 10 different experimental alloys to produce powder and use heat and high pressure to isostatically press the powders into test blocks. Each test block would be chemically analyzed, thermally processed, and tested for strength and corrosion resistance.

The results of this experiment would check the predictions of the model, and the alloy with the most promise would be atomized in an 800-pound unit to produce powder that would be extensively tested to verify the laboratory results taken from the 50-pound sample. A finished product, a near-net-shape part, would then be manufactured and sent to an end user for a field application and trial.

Crucible believed that, if successful, this development would reduce the United States' corrosion-related losses by \$80 million each year. However, there was a significant technical risk involved in undertaking the project. At the time, powder metallurgy was not performed according to a scientific model. In addition, Crucible anticipated difficulty in creating a component with enough solubility to absorb and retain sufficient nitrogen. Furthermore, the development effort would be costly and would require an extended length of time to complete. Because of the technical risk and cost factors, the company was unable to proceed without outside funding. Therefore, in 1994, Crucible applied for cost-

shared funding from ATP and was awarded \$908,000 for a three-year project that began in March 1995.

Reduced Corrosion Losses Could Lead to Broad-Based Benefits

The new high-strength, high-nitrogen stainless steel could potentially benefit a broad section of American industry by limiting the huge economic losses caused by corrosion. If successful, this development project would also reduce the need for expensive alloys; result in new methods and processes that could be applied to existing technologies; create new markets for high-strength, corrosion-resistant products; and enhance U.S. competition in overseas markets.

In 1994, the total annual consumption of stainless and heat-resistant steels in the United States was approximately 1.5 million tons. One significant production cost factor was the need to use expensive metals such as chromium, nickel, and molybdenum. Crucible anticipated that if 10 percent of the chromium and nickel in these alloys was replaced with low-cost nitrogen, a yearly cost savings of nearly \$150 million in strategic metals could be realized. Strategic metals, such as chromium and nickel, are expensive, are imported, and have limited availability. If 10 percent of the molybdenum could be replaced with nitrogen, another \$30 million could be saved. Existing highly alloyed nickel-based materials were resistant to corrosion, but had only moderate strength and were very expensive. They were used only for essential, strategic components.

Crucible believed that, if successful, this development would reduce the United States' corrosion-related losses by \$80 million each year.

Methods developed during the ATP-funded project to maximize the nitrogen content in stainless steels would represent a significant advance in metals processing. In addition, the technology could be applicable to other processes for improving corrosion resistance, such as surface treatment of stainless steels. The technology developed during this project could also be applied to increase nitrogen levels in other important alloys such as nickel-based superalloys.

Manufacturers of valves and other products, in which stress corrosion is an ongoing problem, would significantly benefit from the more corrosion-resistant and stronger stainless steel. In addition, the ability to produce both advanced alloys and near-net shapes via powder metallurgy would help domestic producers compete overseas.

Project's Technical Objectives Are Established

Crucible's goal was to develop and commercialize a more corrosion-resistant stainless steel. To meet this goal, the company established the following technical objectives:

- Apply existing thermodynamic models of melt composition and nitrogen pressure for alloys in order to design corrosion-resistant, high-strength, high-nitrogen stainless steel
- Develop the commercially viable powder metallurgy process to obtain unusually high nitrogen compositions
- Develop commercial-scale processing methods to minimize nitrogen loss during subsequent thermomechanical processing of powdered metals
- Study the microstructure of the powder, formed parts, and specimens exposed to high temperatures through the use of optical microscopy, x-ray diffraction, scanning electron microscopy, and transmission electron microscopy
- Evaluate the mechanical properties, corrosion resistance, weldability, and machinability of the formed parts

High-Nitrogen Alloys Are Developed but Not Commercialized

During the ATP-funded project, Crucible developed several alloy compositions that could potentially be used to improve the performance of stainless steel. The company also identified a number of potential commercial opportunities for one stainless steel, SS100. This included applications for Norfolk Southern Corporation, which had tested the material as a

candidate for railroad applications, and for Cameron Oil Tool, Dupont, and Westinghouse, to which Crucible had sent material for testing.

The company determined, however, that it was unable to assume the cost of developing and marketing SS100 at that time. Although Crucible was prepared to manufacture the material by using its powder metallurgy process, the company lacked the equipment needed to shape the material into the intended applications. This meant that Crucible would have to complete the process by relying on services outside the company. Crucible had also experienced some difficulties in developing SS100 (as well as the other alloys), such as maintaining a high level of nitrogen while melting and atomizing the composition, which would have added to the production cost.

During the ATP-funded project, Crucible developed several alloy compositions that could potentially be used to improve the performance of stainless steel.

In spite of the difficulties, though, Crucible believed that the work it performed in the ATP-funded project was important, and the company continued to fund the development of high-nitrogen alloys after the project ended. Crucible developed one patent as a result of its research and published several articles to explain the research to other engineers.

Conclusion

With ATP's assistance, Crucible developed several high-nitrogen alloys that may be used to produce high-strength, corrosion-resistant steel, and the company identified several potential commercial opportunities for one stainless steel, SS100. Due to the high cost of developing and marketing SS100, the company decided against commercializing it when the ATP-funded project ended. Crucible is interested in commercializing SS100 in the future and has continued to consider potential markets for it, including the boat shaft and medical implant (for example, hip or knee) markets. During the project, Crucible received one patent and published several articles describing its advances in developing the high-nitrogen alloys.

PROJECT HIGHLIGHTS

Crucible Materials Corporation

Project Title: Corrosion-Resistant Steel Through Powder Metallurgy Process (Rapid Solidification Powder Metallurgy for High-Nitrogen Stainless Steels)

Project: To develop new corrosion-resistant steels for marine and other corrosive environments using rapid solidification powder metallurgy for high-nitrogen stainless steels.

Duration: 3/15/1995-3/14/1998

ATP Number: 94-01-0287

Funding (in thousands):

ATP Final Cost	\$ 869	38%
Participant Final Cost	<u>1,430</u>	62%
Total	\$2,299	

Accomplishments:

 Crucible Materials

Corporation developed several alloys with high levels of nitrogen that demonstrated the potential to produce high-strength, corrosion-resistant stainless steel. These high-nitrogen alloys may be used in stainless-steel products and marketed in the future.

Crucible was granted the following patent:

- "High strength, corrosion resistant austenitic stainless steel and consolidated article" (No. 5,841,046: filed May 30, 1996, granted November 24, 1998)

Commercialization Status: Crucible has not yet commercialized stainless-steel products made from the high-nitrogen alloys developed during this ATP-funded project.

Outlook: At this time, Crucible plans to develop and market one stainless steel, SS100, in the future. It is considering various markets for SS100, including the boat shaft and medical implant markets.

Composite Performance Score: * *

Company:

Crucible Materials Corporation
Crucible Compaction Metals Division
1001 Robb Hill Road
Oakdale, PA 15071-3200

Contact: Dr. Frank J. Rizzo

Phone: (412) 923-2670, Ext. 116

Publications and Presentations:

Since 1995, Crucible has published or presented the following papers:

- Rhodes, Geoffrey O. and John J. Conway. "High-Nitrogen Austenitic Stainless Steels with High Strength and Corrosion Resistance." JOM. Vol. 48, No. 4, pp. 28-31. April 1996.
- Rhodes, Geoffrey O., John J. Eckenrod, and Frank J. Rizzo. "High Nitrogen Corrosion Resistant Austenitic Stainless Steels Made by Hot Isostatic Compaction of Gas Atomized Powder." Corrosion 96, The NACE International Annual Conference and Exposition. Paper No. 416. 1996.
- Rhodes, Geoffrey O., Ulrike Habel, John J. Eckenrod, and John J. Conway. "Development, Properties and Applications of High Strength Corrosion Resistant High-Nitrogen Austenitic Stainless Steels Produced by HIP P/M." Advanced Particulate Materials & Processes 1997. Proceedings of the 5th International Conference on Advanced Particulate Materials & Processes. 1997.
- Rhodes, G.O. and W.B. Eisen. "High Nitrogen Corrosion Resistant Austenitic Stainless Steels Produced by HIP P/M Processing." Materials Science Forum. Vols. 318-320, pp. 635-648. 1999.

General Motors

Thermoplastic Engineering Design

In the early 1990s, designing and molding a new production-quality thermoplastic automobile part cost hundreds of thousands of dollars and often required 5 to 10 trial-and-error cycles before a part with the correct dimensions, shape, stiffness, and strength could be manufactured. Engineers at General Motors (GM) and General Electric (GE) Plastics believed that they could apply virtual design methods to improve the thermoplastic parts development process. To transform the trial-and-error development process into a design process that utilized computer simulations, the two companies formed a joint venture. Because existing internal, piecemeal funding was inadequate to stimulate the collaborative, pre-competitive research needed to integrate all the development aspects, GM and GE applied for and were awarded Advanced Technology Program (ATP) cost-shared funding in 1992 for a five-year project called Thermoplastic Engineering Design (TED). The TED project team comprised six subcontractors, including software developers AC Technology (now Moldflow Corporation) and Hibbit, Karlsson & Sorenson, Inc. and several universities.

After the end of the ATP-funded project in 1997, the TED program successfully streamlined the development of new thermoplastic parts and shortened time to market for new parts by reducing the number of development and testing cycles, the number of test molds produced, and the number of rejected prototype parts. The TED process decreased internal testing time and improved reliability, and researchers incorporated their data into commercially available thermoplastics design software. In 1999, GM spun off Delphi Corporation, an auto parts manufacturer that currently uses the TED process to produce thermoplastic parts for many original equipment manufacturers and after-market suppliers. Global thermoplastics industry sales are strong and are expected to continue to grow at six percent annually through 2006. This provides continued opportunities to implement the TED technology. However, GM and GE Plastics have reduced their U.S. thermoplastics research and production due to strong foreign competition.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

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Research and data for Status Report 92-01-0040 were collected during July - September 2003.

Existing Parts Development Process Was Slow and Costly

Injection molding of thermoplastics involves converting plastics into products by injecting molten plastics, mixed with glass fibers for strength and stiffness, into mold cavities of complex shapes under heat and high pressure. In 1992, the automotive industry relied heavily on injection-molded thermoplastics; each vehicle contained several hundred injection-molded parts. The existing iterative process to develop a new part was time-consuming and expensive. Engineers first developed specifications, then created prototype molds,

determined the required processing conditions, tested the resulting parts, and modified the molds. They often needed to repeat this cycle 5 to 10 times before they were able to produce a satisfactory part with acceptable processing conditions. Because many thermoplastic parts change with each new model year, engineers must continually develop and test new mold designs to make the updated parts.

The development of new injection-molded components relied on the integration of two inter-related processes: flow analysis (how the part is manufactured) and structural analysis (how the part performs). Flow

analysis includes considering temperature, the flow of the hot liquid plastic in the mold, and the shrinkage and warpage that occurs in the cooling process. Engineers had to determine where to put the gates (or injection ports) in the mold design. Placement of the gates is important, because the fibers align with the flow direction, which affects the strength and stiffness of the resulting part. The thermoplastic is stronger when the fibers are aligned in the same direction. The part's strength is greatest in the direction of the fibers; in other directions, it is weaker. This directional difference in properties is called anisotropy, which was an important area of focus in the ATP-funded project. Ideally, the fibers must align in the direction that strength and stiffness are needed. Structural analysis requires testing strength in performance conditions. The initial mold design must take into account these performance strength requirements.

After a prototype part was produced, the researchers tested its performance to see how much stress and heat the part could tolerate under normal operating conditions or in an accident. If a given part failed to meet performance requirements, engineers had to redesign the mold.

In 1992, the initial development cost of a single auto body panel was as high as \$500,000, with 5 to 10 molding trials required to produce an acceptable part. If General Motors (GM) and General Electric (GE) could eliminate one trial cycle, manufacturers would save \$40,000 and six weeks of development time.

GM and GE Proposed to Integrate Thermoplastic Molding and Performance Design

GM and GE Plastics proposed to link the mold design and performance design processes through virtual simulations in order to streamline thermoplastic parts development. GM and GE had been independently conducting limited research in virtual design, but were unable to make strides due to limited funding. GE had focused on raw materials, while GM had focused on end products. The companies formed a joint venture and sought ATP support to foster collaboration on end-to-end design. In 1992, they were awarded \$5.78 million in cost-shared funding for a five-year project. The joint venture proposed to develop comprehensive virtual design processes for thermoplastics that would enable them to create the most cost-effective design

with the best processes and materials. Their proposed research involved using modeling software and compiling data on materials, molding, and the performance of the resulting parts. The research team called the process "Thermoplastic Engineering Design (TED)." They desired a mechanics-based engineering design approach similar to what the metals industry had developed over many years. However, using plastics with additives, such as fibers and fillers, increased the design complexity. Also, plastics fail differently than metals and are more sensitive to time and temperature. Design and testing required an interdisciplinary understanding of microstructure, material properties, design methods, manufacturing processing, assembly, parts performance, and recycling considerations in order to predict outcomes and reduce the trial-and-error cycle.

The development of new injection-molded components relied on the integration of flow analysis and structural analysis.

The joint venture enlisted the support of six subcontractors: AC Technology (maker of C-MOLD mold-development software [now Moldflow Corporation]); Hibbit, Karlsson & Sorenson, Inc. (maker of ABAQUS performance-testing software); Michigan State University; University of Massachusetts-Lowell; and Stanford University. ATP support facilitated collaboration between the project members and allowed research efforts to increase fourfold, to about 50 cross-disciplinary researchers and engineers. The TED project stimulated synergy between university research, materials production, mold design, and parts performance.

Joint Venture Identifies Primary Technical Goals

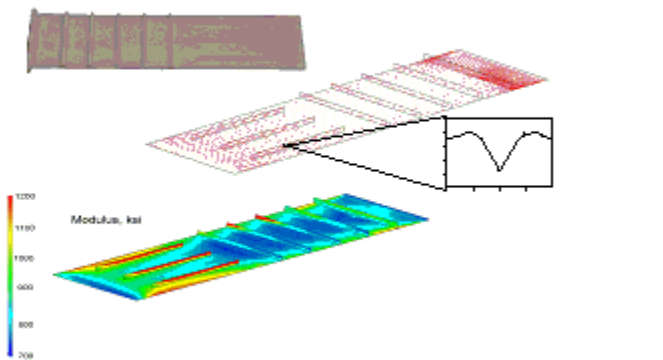
The GM and GE engineers and researchers intended to reduce the expense and development cycle time for thousands of new thermoplastic parts. ATP support allowed the TED project engineers to cooperate on three primary goals:

1. Develop comprehensive mechanics-based design models (e.g., microstructure, materials properties, fiber alignment and related strength and stiffness, parts geometry, and ultimate parts performance)

2. Assess past performance and incorporate the design knowledge into engineering tools (e.g., software simulations, design manuals, and engineering databases)

3. Test the technology in GM and GE manufacturing applications and transfer this technology to a broad manufacturer/supplier base (e.g., Does the part deform and/or break under impact? How does it perform over time? How much temperature change can it tolerate?)

To understand the relationship between processing conditions and materials characteristics, the researchers began by molding plaques (rectangular pieces of plastic) under very controlled processing conditions. The team entered the processing conditions into the C-MOLD and ABAQUS software programs and monitored microstructure, temperature, cooling time, pressure, and other variables. They developed a “TED Calculator” that compared the outcome with the predictions, making refinements as needed. The researchers then studied the results, looking at dimensions and warping, fiber length and orientation/alignment, stiffness and strength, impact resistance, and other parts characteristics. The TED process allowed them to predict these properties, so they could reduce the number of trial-and-error cycles necessary to develop new parts. After they performed a number of trials using different models, their predicted values agreed with the experimental results.



Top: Researchers used representative plaques to test thermoplastic materials for a range of properties. These experimental data are compared to computer predictions and provide the basis for improving predictive models. Middle: Process simulation predictions of fiber orientation can be represented visually. Bottom: Predicted fiber orientation, combined with material data, can be used to predict the modulus (stiffness variation) of the test plaque.

Finally, researchers tried to predict failure (e.g., breaks or cracks) based on fiber orientation and other experimental data. Software tools and new ATP developed failure theories helped to predict the variation in strength of a molded component and the in strength of a molded component and the crack initiation locations. Complex-shaped components performed differently than the experimental plaques. The researchers were able to refine the models developed in order to improve the accuracy of their predictions for any shapes.

TED Project Meets Technical Goals

The TED process made significant strides in improving the ability to use computer software to design and predict the dimensional and mechanical performance of injection-molded prototype parts. The project team accomplished its three primary technical goals, and the resulting process reduced the trial-and-error cycles for new parts development. One important outcome was the linking of the two commercial virtual prototype design software programs, C-MOLD (now Moldflow) and ABAQUS, to combine mold design and parts performance. Moldflow and ABAQUS still use the techniques developed in TED; in fact, Hibbit, Karlsson & Sorenson currently markets this Moldflow-ABAQUS interface.

The TED project stimulated synergy between university research, materials production, mold design, and parts performance.

The team compiled data from trials, and they incorporated the data into internal design manuals and databases for prototype development. Engineers used the results from tests made on numerous existing components (e.g., intake manifolds, radiator tanks, transmission covers, wiper arms, fan blades, door components, and body side moldings) to predict the performance of new parts.

TED Knowledge Is Disseminated Widely

The TED project team shared its knowledge extensively through the following:

- Provided training workshops for GM and GE Plastics engineers on how to incorporate TED strategies and tools
- Published highlighted accomplishments and results of the TED project in numerous academic publications
- Founded the National Thermoplastics Engineering Design Association, a direct output of the ATP-funded project, which has approximately 1,500 members from 300 companies and institutions, where members can learn about the TED modeling tools
- Gave public presentations at workshops and conferences and contributed to the development of a graduate-level course offered at Michigan State University

Economic Changes Stimulate Corporate Shift

In the late 1990s, U.S. manufacturers were increasingly outsourcing parts production in order to increase efficiency and reduce costs. Two years after the ATP-funded project had ended, GM spun off Delphi Corporation in 1999, an operation that included the majority of GM's thermoplastics production. Delphi now uses the TED process to produce thermoplastic parts for multiple original equipment manufacturers and after-market suppliers. GM has continued limited thermoplastics research.

Improved Thermoplastic Design Provides Economic Benefits

Reducing development cycle time and cost was the primary benefit of the TED process, but it is difficult to measure cost reduction down to the level of specific parts. However, GM, GE, and Delphi have demonstrated several specific benefits that resulted from their project research:

- Delphi now designs thermoplastic parts that have predictable fatigue life (length of time a part can sustain repeated loading), impact strength, and, in some cases, shrinkage/warpage properties.

- The TED program led to the development of a new International Standards Organization (ISO) Standard, 294-5, to generate the anisotropic mechanical data needed for thermoplastics part design.
- A Moldflow and ABAQUS interface, which was designed based on TED principles, provides engineers commercially available software to integrate virtual mold design with parts performance.
- The project research resulted in one U.S. patent and two project awards.
- Delphi continues to refine TED design principles and applies them to many re-engineered and new parts. For example, engineers were dealing with a thermoplastic radiator tank, which fits on both sides of a metal radiator. The tanks must pass a long term fatigue test. Using the TED process, the fatigue lifetime was accurately predicted by taking into account the temperature, glass fiber orientation, ABAQUS stress analysis, and an ATP developed fatigue cracking theory. In some cases tanks were also cracking during assembly to the metal radiator. Again, using the TED process, the assembly equipment was redesigned to eliminate the cracking. These ATP developed virtual part design methodologies remain in place at Delphi for all new plastic radiator tanks.
- GM is continuing limited TED research internally. At GM's Powertrain division, engineers now have a virtual part design capability to improve stiffness and strength for their injection-molded engine components. One of the GM test cases was a plastic intake manifold. Injection-molded, glass-fiber-filled plastic intake manifolds have to withstand an overpressurization test, where they are pressurized to simulate a backfire inside the manifold. GM used C-MOLD to determine the principal material directions and ABAQUS to predict the burst pressure of these parts. Engineers used TED methods to modify the design of the product, significantly raising the burst pressure. This modified design was generated in one iteration, rather than the 4-5 that might have been required without the TED modeling.

- GE Plastics provides improved raw materials and technical support to its other thermoplastics customers for use in molding processes for business equipment (e.g., monitors and printer housings), telecommunications (e.g., cellular telephones), and optical media (e.g., CDs and DVDs).

TED Development Continues

As of 2003, GE Plastics, GM, and Delphi researchers were continuing to perform research on the predictive models for virtual design. Based on the TED process, GM and GE Plastics are developing new high-performance thermoplastic parts, using new materials in order to produce parts that support heavier loads. Delphi, as a subcontractor to GM and other manufacturers, produces many thermoplastic parts using the TED process. Even after the ATP-funded project concluded, the company continues internal research and still maintains a joint development agreement with Moldflow software, which originated with the TED project. Moldflow sells this modeling software commercially.

Demand for Thermoplastics Rises as Prices Drop

The overall use of thermoplastics in automobiles has increased dramatically since the TED project began, increasing competition and reducing prices. By 1997, North American automakers were using 56 percent more fiberglass than in 1990 (352 million pounds, compared with 226 million pounds). The use of nylons in engine components in U.S.-built cars grew 24 percent from 1990 to 2002 (97 million pounds in 1990, 100 million pounds in 1995, and 120 million pounds in 2002, mostly glass- or mineral-reinforced). While the entire U.S. market for thermoplastics is projected to grow 6.4 percent annually from 2003 through 2006, the most rapid growth will be in automotive, medical, and industrial applications. The thermoplastics industry is strong and competitive, and TED strategies and tools allow manufacturers to produce these thermoplastic components more efficiently.

Conclusion

The use of thermoplastics in the automotive industry has grown steadily at approximately six percent per year since the early 1990s. Virtual thermoplastics

design had the potential to reduce high development and manufacturing costs in automobiles. In 1992, General Motors (GM) and General Electric (GE) Plastics began a collaborative project, with ATP funding support, to explore this design innovation, called Thermoplastic Engineering Design (TED). TED researchers proposed to develop new virtual design methods for thermoplastics parts development. They sought to optimize their understanding of the complex interactions between plastic materials the manufacturing process, and the part geometry (engineering application) in order to reduce time to market and improve automotive parts' performance. The joint venture successfully developed models to virtually design and manufacture prototype thermoplastic parts. These models account for temperature, material content, warping, and structural integrity during performance.

The TED researchers shared their results extensively. They incorporated their data into commercially available software, Moldflow (formerly C-MOLD) and ABAQUS, for virtual mold and performance design; this led to an interface between ABAQUS and Moldflow software, which benefits many thermoplastics designers. Furthermore, TED researchers developed a new international engineering standard, received one patent, published their results widely within the thermoplastics engineering community, formed the National Thermoplastic Engineering Design organization with approximately 1,500 members, and held many workshops.

As a result of increasing foreign competition and a need to remain competitive, in 1999 GM spun off Delphi Corporation, which produces thermoplastic components for GM and other manufacturers. While GM and GE Plastics have reduced their U.S. thermoplastics research and production, the industry continues to grow and remain competitive. Delphi currently uses the TED process to produce thermoplastic parts for many original equipment manufacturers and after-market suppliers.

PROJECT HIGHLIGHTS

General Motors

Project Title: Thermoplastic Engineering Design
(Engineering Design with Injection-Molded Plastics)

Project: To develop a scientific understanding of the relationship between processing, part geometry, microstructure, and part performance for fiber-reinforced molded thermoplastic parts and embody this knowledge in an integrated thermoplastic engineering design methodology (virtual design).

Duration: 10/1/1992-9/30/1997

ATP Number: 92-01-0040

Funding (in thousands):

ATP Final Cost	\$5,784	48.4%
Participant Final Cost	<u>6,166</u>	51.6%
Total	\$11,950	

Accomplishments: With ATP funding, General Motors (GM), Delphi Corporation (formerly part of GM), and General Electric (GE) Plastics have developed models and generated data for "virtual design" in order to improve the design and development of thermoplastic automotive parts. The project team linked two commercial software tools, Moldflow (formerly C-MOLD) and ABAQUS, with new failure theories for plastics in order to integrate mold design with parts performance. The companies continue to enhance these software tools, and failure models which benefits thermoplastics engineers in general.

Researchers implemented the Thermoplastic Engineering Design (TED) process within GM and GE Plastics both to rework existing parts and also to develop new parts. They formed the National Thermoplastics Engineering Design Association (NTEDA) to transfer the TED methods to design engineers at interested U.S. manufacturers. They won the following awards for their achievements:

- Best Paper Award from the Society of Plastics Engineers Product Design and Development Division, May 2001. J. F. O'Gara, P. H. Foss, and J. P. Harris for "Reinforced Plastic Design: Microstructure and Stiffness of Tensile Bars," Annual Technical Conference (ANTEC) '01 Preprints, Society of Plastics Engineers.
- Campbell Award, 2001. P. H. Foss and C. C. Mentzer for "Process Modeling and Performance Predictions of Injection Molded Polymers." This internal GM research and development award recognizes researchers for outstanding contributions to pure or applied science.

GE received the following patent for technology related to the ATP-funded project:

- "Devices and methods for measurements of barrier properties of coating arrays"
(No. 6,383,815: filed April 4, 2001; granted May 7, 2002)

The project also resulted in the development of a new International Standards Organization (ISO) Standard:

- "Injection molding of test specimens of thermoplastic materials-Part 5: Preparation of standard specimens for investigating anisotropy" (ISO 294-5, out of technical committee 61; Stage date 12/13/2001)

This project led to many publications and workshops, which are listed at the end of this report.

Commercialization Status: The results of the TED project significantly affected commercial software for design simulations, linking Moldflow, a molding-process software, with ABAQUS, a performance-testing software. Hibbit, Karlsson & Sorenson sells the interface, called "ABAQUS Interface for Moldflow." These virtual design tools have shortened development time and have improved the performance of thermoplastic parts, which has benefited many manufacturers. The project's focus was on improving the development process, so the project has impacted many parts (e.g., Delphi's thermoplastic radiator tank and many other parts; GM's injection-molded plastic intake manifold and other engine components; GE Plastics' improved raw material, which is used in business equipment, optical media, and telecommunications devices).

Outlook: The outlook for TED technology is good. The technology is available to plastics engineers through the commercial off-the-shelf software, Moldflow and ABAQUS. GM, GE Plastics, and Delphi Corporation continue to develop and use the technology internally. Benefits of the ATP-funded technology continue to accrue for consumers: sales of thermoplastics are high, prices are low, and parts last longer. Thermoplastics sales are expected to grow at 6.4 percent per year through 2006, with strong foreign competition.

Composite Performance Score: * * *

PROJECT HIGHLIGHTS

General Motors

Company:

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Composites Processing Group
Materials and Processes Lab
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Contact: Howard W. Cox

Phone: (586) 986-1203

Subcontractors:

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1080 Main Street
Pawtucket, RI 02860

Michigan State University
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East Lansing, MI 48824-1226

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Mechanical Engineering Department
Terman Engineering Center 509
Stanford, CA 94305-4021

University of Massachusetts-Lowell
Plastics Engineering Department
One Lowell Avenue
Lowell, MA 01854

Publications and Presentations:

The joint venture members held numerous workshops to share project knowledge with design engineers at GM, GE, and engineering firms, including the following:

- Trantina, G. G., and M. G. Wyzgoski. "Engineering Design with Injection Molded Thermoplastics," Society of Plastics Engineers (SPE) ANTEC '94 Preprints, May 1994.
- Foss, P. H., H. H., Chiang, L.P. Inzinna, C. L. Tucker, and K. F. Heitzmann. "Experimental Verification of C-MOLD Fiber Orientation Predictions," presentation at the C-MOLD Users' Group meeting, Dearborn, MI, September 1994.
- Foss, P. H., R. M. Shay, and C. M. Mentzer. "Experimental Verification of C-MOLD Shrinkage Predictions," presentation at the C-MOLD Users' Group meeting, Dearborn, MI, September 1994.
- Trantina, G. G., and M. G. Wyzgoski. "Engineering Design with Injection Molded Thermoplastics for Automotive Application," 1994 International Body Engineering Conference, Detroit, MI, September 1994.
- Held the first TED workshop on "Mechanical and Dimensional Performance of Injection-Molded Thermoplastic Parts" on February 23, 1995 in Troy, MI with more than 70 GM engineers. Held two additional GM workshops for Delphi in Rochester, NY and Anderson, IN.
- Held first NTEDA workshop on March 7, 1995 for 50 engineers representing a variety of plastics industries. Held 12 workshops in 1995 that addressed approximately 550 engineers and designers.
- Foss, P. H., J. P. Harris, J. F. O'Gara, L. P. Inzinna, E. W. Liang, C. M. Dunbar, C. L. Tucker III, and K. F. Heitzmann. "Experimental Verification of C-MOLD Modulus and Fiber Orientation Predictions," presentation at the C-MOLD Users' Group meeting, Dearborn, MI, September 1995.
- Shay, Jr., R. M., P. H. Foss, and C. C. Mentzer. "Comparison of C-MOLD Predictions and Experimental Shrinkages," presentation at the C-MOLD Users' Group meeting, Dearborn, MI, September 1995.
- Liang, E. W., V. K. Stokes, L. P. Inzinna, J. F. O'Gara, and J. P. Harris. "Tensile Modulus of Injection-Molded Short-Fiber Reinforced Thermoplastics-Experiment and Prediction," 1995 RETEC, Atlanta, GA, September 13, 1995.
- Held GM workshops on October 31, 1995 and November 2, 1995.
- Hasan, O. A. "The Mechanical Behavior of Rubber-Modified Thermoplastics," invited lecturer at Drexel University, November 1995 and ASME Winter Meeting, November 1995.

PROJECT HIGHLIGHTS

General Motors

- Submitted research on fiber length and orientation measurements to the Measurement Science Conference in Anaheim, CA, January 1996.
- Held NTEDA workshop at the Design Engineering Show in Chicago, IL on March 20, 1996. By that time, NTEDA had 1,500 members from 300 different companies and institutions.
- Michigan State University developed a graduate-level course, "The Design for Manufacture of Injection-Molded Parts," based on the TED project, 1996.
- Held technical sessions, "Fatigue of Glass Fiber Reinforced Injection Molded Plastics I. Stress-Lifetime Data" and "II. Tensile Versus Flexural Loading," SPE ANTEC Meeting, May 2001.
- Held a technical session for product design and development, "Product Design and Development Methods to Optimize Plastics Part Designs: Development of an ISO Standard for Determining Anisotropic Properties of Glass-Filled Thermoplastics," May 5, 2003 conference for ANTEC.
- Bushko, W. C., and V. K. Stokes. "The Effects of Differential Mold Temperatures on the Warpage of Injection-Molded Parts," presented at the ASME International Congress and Exposition, Chicago, IL, November 6-11, 1994. Paper included in *Advances in Computer-Aided Engineering (CAE) of Polymer Processing*, edited by K. Himasekhar, V. Prasad, T. A. Osswald, and G. Batch, MD-Vol. 49/HTD-Vol. 283, pp. 137-161, American Society of Mechanical Engineers, NY, 1994.
- Bushko, W. C., and V. K. Stokes. "Solidification of Thermoviscoelastic Melts. Part I: "Formulation of Model Problem," *Polymer Engineering and Science*, Vol. 35, No. 4, pp. 351-364, February 1995. Part II: "Effects of Processing Conditions on Shrinkage and Residual Stresses," pp. 365-383.
- Foss, P. H., H. H. Chiang, L. P. Inzinna, C. L. Tucker III, and K. F. Heitzmann. "Experimental Verification of C-MOLD Modulus and Fiber Orientation Predictions," *Society of Plastics Engineers (SPE) ANTEC '95*, Preprints, May 1995.
- Woods, J. T., R. P. Nimmer, and K. F. Ryan. "The Development and Validation of Rate Dependent Brittle Failure Criterion for Polycarbonate and Polyetherimide," ANTEC 1995, Boston, MA.

The team made the technology widely available through many publications:

- Bushko, W. C., and V. K. Stokes. "The Effects of Differential Mold-Surface Temperatures on the Warpage of Packed Injection-Molded Parts," *Proceedings of the Society of Plastics Engineers 52nd Annual Technical Conference*, San Francisco, CA, May 2-6, 1994, pp. 506-512.
- Bushko, W. C., and V. K. Stokes. "Shrinkage of Packed Injection-Molded Parts: Simulation of Gate Freeze-Off Effects," *Proceedings of the Society of Plastics Engineers 52nd Annual Technical Conference*, San Francisco, CA, May 2-6, 1994, pp. 559-564.
- Stokes, V. K. "The Hot-Tool Welding of Acrylonitrile-Butadiene-Styrene," *Proceedings of the Society of Plastics Engineers 52nd Annual Technical Conference*, San Francisco, CA, May 2-6, 1994, pp. 1327-1332.
- Bushko, W. C., and V. K. Stokes. "The Effects of Boundary Conditions on the Dimensional Changes and Residual Stresses in Injection-Molded Polycarbonate Parts," *Proceedings of the Society of Plastics Engineers 53rd Annual Technical Conference*, Boston, MA, May 7-11, 1995, pp. 484-490.
- Stokes, V. K. "Experiments on the Hot-Tool Welding of Polycarbonate," *Proceedings of the Society of Plastics Engineers 53rd Annual Technical Conference*, Boston, MA, May 7-11, 1995, pp. 1229-1233.
- Stokes, V. K. "Toward a Weld-Strength Data Base for Vibration Welding of Thermoplastics," *Proceedings of the Society of Plastics Engineers 53rd Annual Technical Conference*, Boston, MA, May 7-11, 1995, pp. 1280-1284.

PROJECT HIGHLIGHTS

General Motors

- Bushko, W. C., and V. K. Stokes. "The Effects of Boundary Conditions on the Shrinkage and Residual Stresses in Injection-Molded Parts," presented at the ASME Joint Applied Mechanics and Materials Summer Meeting, Los Angeles, CA, June 28-30, 1995. Paper included in Current Research in the Thermo-Mechanics of Polymers in the Rubbery-Glassy Range, edited by M. Negahban, AMD-Vol. 203, pp. 129-158, American Society of Mechanical Engineers, NY, 1995.
- Shay, Jr., R. M., P. H. Foss, and C. C. Mentzer. "Comparison of C-MOLD Predictions and Experimental Shrinkages," 1995 Automotive RETEC Preprints, SPE, November 1995.
- P. H. Foss. "Experimental Verification of Finite Element Predictions of Shrinkage, Fiber Orientation and Modulus," Autofact 1995 Preprints, SME, November 1995.
- Bushko, W. C., and V. K. Stokes. "Solidification of Thermoviscoelastic Melts. Part III: Effects of Mold Surface Temperature Differences on Warpage and Residual Stress," Polymer Engineering and Science 36 (3): 322-335, February 1996.
- Bushko, W. C., and V. K. Stokes. "Solidification of Thermoviscoelastic Melts. Part IV: Effects of Boundary Conditions on Shrinkage and Residual Stresses," Polymer Engineering and Science 36 (5): 658-675, March 1996.
- Hasan, O. A. "Use of Deformation Maps in Predicting Time-Dependent Response of Thermoplastics," ANTEC, 1996.
- Foss, P. H., J. P. Harris, J. F. O'Gara, L. P. Inzinna, E. W. Liang, C. M. Dunbar, C. L. Tucker III, and K. F. Heitzmann. "Prediction of Fiber Orientation and Mechanical Properties Using C-MOLD and ABAQUS," ANTEC '96 Preprints, SPE, May 1996.
- Shay, Jr., R. M., P. H. Foss, and C. C. Mentzer. "Comparison of C-MOLD Predictions and Experimental Shrinkages: Fiber-Filled Materials," ANTEC '96 Preprints, SPE, May 1996.
- Wyzgoski, M. G., and G. E. Novak. "Fatigue Fracture of Reinforced Plastics: Measurements and Predictions," ANTEC '96 Preprints, SPE, May 1996.
- V. K. Stokes. "Cross-Thickness Vibration Welding of Polycarbonate, Polyetherimide, Poly(Butylene Terephthalate), and Modified Polyphenylene Oxide," Polymer Engineering and Science 37 (4): 715-725, April 1997.
- VerWeyst, B. E., C. L. Tucker, and P. H. Foss. "The Optimized Quasi-Planar Approximation for Predicting Fiber Orientation in Injection-Molded Composites," International Polymer Processing, 12:238-248, 1997.
- VerWeyst, B. E. Ph.D. Dissertation, "Numerical Predictions of Flow-Induced Fiber Orientation in Three-Dimensional Geometries," University of Illinois at Urbana-Champaign, 1998.
- VerWeyst, B. E., C. L. Tucker, P. H. Foss, and J. F. O'Gara. "Fiber Orientation in 3-D injection Molded Features: Prediction and Experiment," International Polymer Processing, 14:409-420, 1999.
- VerWeyst, B. E. and C. L. Tucker, III. "Fiber Suspensions in Complex Geometries: Flow/Orientation Coupling," Can. J. Chem. Eng., 80:1093-1106, 2002.
- Wyzgoski, M. G., J. A. Krohn, and G. E. Novak. "Fatigue of Glass Fiber Reinforced Injection Molded Plastics I. Stress-Lifetime Data," Polymer Composites Journal, accepted for publication, 2004.
- Wyzgoski, M. G., J. A. Krohn, and G. E. Novak. "Fatigue of Glass Fiber Reinforced Injection Molded Plastics II. Tensile Versus Flexural Loading," Polymer Composites Journal, accepted for publication, 2004.

Research and data for Status Report 92-01-0040 were collected during July - September 2003.

Honeywell (formerly AlliedSignal, Inc.)

PIM Process Used to Manufacture Ceramic Components

Manufacturers continually seek to develop more resilient and cost-effective materials to use in their products. Ceramics have specific characteristics that make them a plausible replacement for several materials, including steel. Ceramics products are lighter, less corrosive, more resilient in high-temperature environments, and sustainable in nonlubricated environments. In 1991, plastic powder injection molding (PIM) production in the United States was a \$20 billion industry. In contrast, the use of PIM in the ceramics industry was relatively small at \$10 million annually. Honeywell (formerly AlliedSignal, Inc.), however, believed that an entire ceramics-based parts industry could be created, based on the existing PIM process for plastics, and had the potential to grow into a \$1 billion market within 10 years.

Therefore, in 1993, Honeywell proposed to the Advanced Technology Program (ATP) a three-year project to develop a cost-effective ceramic PIM process. Honeywell, which pioneered this technology, needed ATP funding because of the high technical risk of the work. Upon completion of the project in 1997, Honeywell had realized measurable results in several areas, including feedstock production and component manufacturing for a few small parts. The new process has proved most effective in minimizing machining for high-volume, labor-intensive parts.

Over time, commercialization based on this project's technical success is being realized in several sectors far beyond the original scope of this project: ceramic chinaware, spark plugs, oxygen sensors, ball bearings, manufacturing components, engine components, and bio ceramics. Honeywell also used the knowledge gained to expand its product development into metal PIM. This ATP-funded project helped to catapult the small ceramic PIM industry from approximately \$10 million annually to one that in 2002 was estimated to exceed \$160 million, with almost 10 percent annual growth.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* *

Research and data for Status Report 93-01-0104 were collected during October - December 2001 and April - May 2003.

PIM Process Could Lower Cost of Ceramics Production

Ceramics have the potential to replace materials such as steel in many applications because they are lighter, less corrosive, more heat-resistant, and sustainable in nonlubricated environments. Moreover, ceramics are less likely to be rejected by the human body than other materials. Over the last several decades, scientific improvements have led to fundamental material technology advances that have made it possible to use ceramics in a wide variety of automotive, consumer,

industrial, aerospace, military, and medical products. Although the adoption of ceramic-based products had increased by 1993, manufacturers in several industries had experienced difficulty finding a means to cost-effectively produce these components.

Honeywell (formerly AlliedSignal, Inc.) believed that by applying powder injection molding (PIM), the process used successfully by the plastics industry, to ceramics production, they could create a responsive, low-cost, high-volume manufacturing base for shaping ceramic materials into engine components and could quickly build an entire ceramics-based parts industry. They

projected that, if successful, the ceramics PIM industry could grow from its existing annual market base of \$10 million to \$1 billion in 10 years. However, due to the project's potential for failure, Honeywell was hesitant to fund the project and needed to find a way to attract outside collaborators. Therefore, Honeywell applied for funding from ATP and, in 1994, received a three-year award of approximately \$1.6 million to develop ceramic PIM technology.

Collaboration Provides Framework for Success

To attain their objective of developing a cost-effective ceramic PIM process, Honeywell and its subcontractors (Pennsylvania State University; Cincinnati Milacron; Autolite, a subsidiary of Honeywell; Golden Technologies; and Honeywell Aerospace), needed to achieve these specific milestones and results:

Milestone: Develop cost-effective ceramic feedstock formulations (alumina and silicon nitride), which possess rheological (dealing with the deformation and flow of matter) and molding characteristics similar to those of plastic feedstock.

Result: Selected and characterized raw materials and formulations.

Milestone: Develop a predictable shrinkage control process for finished products. **Result:** Identified key molding variables, conducted molding trials, and attained reproducible size and improved shrinkage tolerance to produce near-net-shape ceramic components; incorporated gel strength-enhancing additives to improve the stiffness of molded parts and developed new alloys for molding machine components to minimize rust contamination; defined the relationships between gel strength, blend formulation, time, and temperature stability.

Milestone: Develop a safe binder-removal process. **Result:** Carefully configured dies to release water and minimize blister formation.

Milestone: Achieve aging and molding characteristics for ceramic PIM that are similar to those characteristics in plastic PIM. **Result:** Injection molding gives better component density than slip casting; achieved molding machine process controls to create high-quality alumina and silicon nitride

compounds; developed best practices for designing molds using water-based ceramic PIM.

Milestone: Incorporate plastic PIM equipment into the ceramic PIM process by making only minor modifications. **Result:** With slight modifications, used plastic PIM machines to complete preliminary high-precision prototype molding of both alumina and silicon nitride ceramic compounds at Autolite, Golden Technologies, and Ceramic Components; completed preliminary prototypes for two additional customers, demonstrated the process, and determined production rates for a wide variety of specialty electrical parts.

The project team that Honeywell selected to accomplish these tasks included the following members:

- **Pennsylvania State University**, which has an established center for materials research with a technology base in rheological and tribological science (dealing with the design, friction, wear, and lubrication of interacting surfaces in relative motion, as in bearings or gears)
- **Cincinnati Milacron**, which is the largest U.S. manufacturer of injection molding equipment
- **Autolite**, which is the largest U.S. manufacturer of alumina spark plugs
- **Golden Technologies**, which is a major manufacturer of alumina ceramic products
- **Honeywell Aerospace**, which was developing a manufacturing base for advanced silicon nitride ceramics

Ceramics have the potential to replace materials such as steel in many applications.

Throughout the project, each team member played an integral role in developing feedstock and manufacturing prototype components. Several team members, including Autolite, Honeywell Aerospace, and Honeywell's in-house research team, regularly

developed various feedstock formulations. As these formulas were developed, Golden Technologies and Pennsylvania State University conducted molding trials and studies, which measured the materials' rheological characteristics, heat capacity, thermal conductivity, and other properties important for conducting mold-fill modeling. Test and trial results revealed inefficiencies that were quickly corrected, and eventually the team members obtained the desired characteristics. Reaching this milestone made PIM machine testing with alumina and silicon nitride feedstock formulations possible. With slight modifications, the plastic PIM machines were used to produce several small ceramic components.

Technical Accomplishments Encourage Feasibility of Broad-Based Production

Although the project team consistently achieved and improved upon the prototype production of small products and components, they aspired to develop methods and processes to enable the production of large components and products. In early 1997, near the project's completion, molding trials at Bemis Manufacturing Company successfully fabricated large-size ceramic components by using plastic molding machines. The trials supported the notion that plastic molding equipment could also be utilized in ceramic PIM and provided encouragement to other plastic molders to expand their product lines and benefit from the use of preformulated feedstock.

Superior Feedstock Formulations Reduce Costs

Honeywell and its team members developed several ceramic feedstock formulations for use in PIM equipment. The new feedstock formulations, composed of silicon nitride and alumina, retained superior molding, shrinkage, and pressure characteristics that have been successful in reducing development costs for several products in various industries. These include mechanical applications (e.g., seals, nozzles, spark plugs, and sensors), manufacturing applications (e.g., stamping punches and guide rollers), and even bio-ceramics (e.g., artificial bones for humans). Honeywell and its partners sold feedstock to plastic PIM manufacturers that had switched to or had incorporated ceramic PIM into their manufacturing processes.

However, the volume and profit margins were too low, and the project team ultimately discontinued this practice.

Manufacturers in several industries had experienced difficulty finding a means to cost-effectively produce these components.

Approximately 40 to 50 manufacturers have adopted these new processes. Those that are using the new preformulated feedstock formulations include Saint Gobain Advanced Ceramics (formerly Carborundum), Coors Ceramics, Norton Ceramics, Kyocera Inc., and NTK Ceramics. The superior feedstock compositions used by these manufacturers have enabled the development of various products that now provide more heat-resistant, longer lasting parts to consumers. This ATP-funded project brought credibility to the process, and several other companies have developed their own formulas. Depending on the geometry of a part, it may be produced by slipcasting, gel casting, die pressing, or PIM. The PIM process is cost effective for high-volume parts with complicated shapes.

Analyses Prove Ceramic PIM's Viability

After the Honeywell team overcame several obstacles and reached many of its milestones, they completed four marketing and business case analyses on ceramic PIM. They conducted the analyses for specific products believed to have significant industry-wide impacts and large and immediate return-on-investment. These products included oxygen sensors, 8.5-mm spark plugs, and entire spark plug facilities. The results of the analyses on the products and manufacturing facilities proved to be invaluable. The four marketing and business case analyses are summarized below:

- Analyzed a retrofit of Honeywell's Fostoria spark plug facility to accommodate the use of injection molding. Comparing conventional 8.5-mm spark plugs to PIM spark plugs revealed that conventional spark plugs cost more than those developed by the PIM processes; therefore, the plant was partially retrofitted. The plant began producing racecar spark plugs made with insulators that had been injection molded.

- Assessed developing oxygen sensor thimbles for oxygen sensors using injection-molding methods. The analysis revealed that significant cost savings could be realized if injection molding was adopted. In 1998, one million oxygen sensor thimbles were produced per year using a traditional zirconia material system. The analysis predicted that manufacturers would be able to increase production to 1.147 million thimbles per year using the injection-molding process. Furthermore, a materials savings of 33 percent and a scrap savings of 80 percent could be gained, for a 14.7-percent reduction in fixed costs.
- Evaluated insulators for three types of oxygen sensors. The analyses determined that a 30-percent savings for the three insulators would exceed \$300,000 per year.
- Predicted that the manufacturing cost of bone china dinnerware with complex shapes, such as teacups, could be reduced by 10 to 40 percent.

Ceramic PIM Finds Widespread Acceptance

By 1996, acceptance of ceramic PIM within the industry was rapidly increasing. In fact, in June 1996, beta site testing of alumina formulations at five different ceramic fabricators validated strong commercial possibilities. This bolstered the confidence of several industries, such as consumer goods and automotive, to incorporate this technology into new and existing products, such as housewares and automobile gears.

Throughout the project, chinaware makers had displayed an interest in the development of ceramic PIM. In 1996, Honeywell demonstrated the ability to produce injection-molded bone china ceramic formulations in the shape of teacups that have a one-step, integrally molded handle and foot. The company indicated that this new process would provide a 10- to 40-percent cost reduction in dinnerware products that have complex shapes. Since that time, several companies have adopted the technology, including Lenox China in the United States, Wedgwood China in England, and Villeroy & Boch in Germany.

Continuing Impact of the Molding Techniques

Since the ATP-funded project ended in 1997, the company (now as Honeywell) has continued to produce ceramic PIM automotive parts (e.g., spark plugs and oxygen sensors) and aerospace parts for gas turbine engines (e.g., nozzles, seals, and shafts). They have patented and licensed this technology to other firms, which has led to significant growth in the industry (\$160 million in 2001). This project brought credibility to the ceramic PIM process, which is now the method of choice when a company produces complex, high-volume ceramic components. Approximately 40 to 50 companies currently use the technology, and, of those, about 6 use the formula developed in this project. In addition, the ceramic PIM process is used to produce ball bearings, manufacturing components (e.g., stamping punches and guide rollers), and bio ceramics (e.g., artificial bones for human replacement surgery). Furthermore, Honeywell, in collaboration with Rutgers University, used this process to develop metal PIM, which led to the growth of a start-up company, Latitude Manufacturing Technologies.

In 1998, Honeywell produced and sold more than 800,000 oxygen sensors that contain injection-molded ceramic parts (insulators and thimbles). As predicted in the four marketing and business case analyses that Honeywell conducted, the company reduced its production costs by 30 percent, which resulted in a savings of \$300,000.

Conclusion

This pioneering ATP-funded project proved the viability of applying powder injection molding (PIM) from the plastics industry to the ceramics industry. Furthermore, the process is now used in a wide variety of practical applications in the automotive, aerospace, manufacturing, and medical industries. The spillover resulting from this technology has initiated significant growth in the ceramic PIM industry, from \$10 million in 1994 to \$160 million in 2002, and has led to the use of the process with metal. This new industry is growing at a steady pace because of the increasing benefits of reduced manufacturing costs and increased parts life available to original equipment manufacturers and consumers.

PROJECT HIGHLIGHTS

Honeywell (formerly AlliedSignal, Inc.)

Project Title: PIM Process Used to Manufacture Ceramic Components (Ceramic Technology for Broad Based Manufacturing)

Project: To develop and test a user-friendly technology base for net-shape injection molding of complex ceramic parts using water-based injection molding systems for alumina and silicon nitride.

Duration: 04/01/1994-03/31/1997

ATP Number: 93-01-0104

Funding** (in thousands):

ATP Final Cost	\$1,600	50%
Participant Final Cost	<u>1,596</u>	50%
Total	\$3,196	

Accomplishments: Honeywell (formerly AlliedSignal, Inc.) and its team members developed several feedstock formulations for use in ceramic powder injection molding (PIM) equipment. The new feedstock formulations, composed of silicon nitride and alumina, retain superior molding, shrinkage, and pressure characteristics. Consequently, the new feedstock has reduced development costs for several products in various industrial sectors, including chinaware, spark plugs, and oxygen sensor components.

One patent was approved as a result of this project, which formed the basis for additional patent applications following project completion:

- "Gel strength enhancing additives for agaroid-based injection molding compositions" (No. 5,746,957: filed February 5, 1997, granted May 5, 1998)

In addition, Honeywell has presented and published results from the ATP-funded project to increase the industry's knowledge of this technology. These presentations and publications include:

- Fanelli, A., and C. P. Ballard. "Advances in Water-based Powder Injection Molding." 1995 International Powder Injection Molding Symposium. State College, PA: July 19-21, 1995.

- Ballard, C. P. "Water-Based Ceramic Injection Molding Compounds." Society of Manufacturing Engineers. Detroit, MI: March 27-29, 1996.
- Ballard, C. P. "Ceramic Injection Molding Meets the Demand for Manufacturing Complex Shapes." Ceramic Industry. March 1997.
- Ballard, C. P. "Targeting Ceramic Technology for Business Growth." The Gorham Advanced Materials Conference on Advanced Ceramics for the New Millennium. Atlanta, GA: March 10-12, 1998.
- Ballard, C. P., and M. Zedalis. "Advances in Powder Injection Molding." ANTEC'98, Society of Plastic Engineers. Atlanta, GA: April 26-30, 1998.
- Ballard, C. P. "Advances in Ceramic Powder Injection Molding." PIM 98 Powder Injection Molding: An Advanced Manufacturing Technology. University Park, PA: April 27-29, 1998.

Commercialization Status: Since the completion of this project, ceramic PIM technology has been incorporated into a range of products that far exceeds the expectations of the project team. Applications include chinaware, spark plugs, oxygen sensors, ball bearings, manufacturing components (e.g., stamping punches and guide rollers), engine and machine components (e.g., nozzles, seals, shafts, valves, and heating units), and bio ceramics (e.g., artificial bones for human replacement surgery). Moreover, several additional companies have developed products and/or feedstock using the processes and technology resulting from this ATP-funded project. These companies include Norton Ceramics, Morgan Crucible, Vesuvius Group, A. P. Green Industries, Baker Refractories, National Refractories, and Saint Gobain (formerly Carborundum). Component and product development is increasing steadily. This project helped to catapult the small ceramic PIM industry from approximately \$10 million annually in 1994 to one that, in 2002, was estimated to exceed \$160 million. This represents an annual growth rate of almost 10 percent. In addition, the ceramic PIM process spawned a spin-off technology, manufacturing metal components using metal PIM.

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

PROJECT HIGHLIGHTS

Honeywell (formerly AlliedSignal, Inc.)

Outlook: The ceramic PIM industry is growing steadily as competition increases. The outlook for the ceramic PIM industry and for Honeywell's ceramic PIM process is excellent.

Composite Performance Score: * *

Company:

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Contact: Remco Vanweeren

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Subcontractor:

- Pennsylvania State University
- Cincinnati Milacron
- Autolite (subsidiary of Honeywell)
- Golden Technologies
- Honeywell Aerospace

Honeywell International Inc., Ceramic Components
(formerly AlliedSignal, Inc.)

Innovative Molding Technique to Produce Ceramic Turbine Components

In the mid-1990s, U.S. manufacturers in the turbine component industry wanted to develop ceramic industrial turbine components that would be lighter, longer lived, and more heat-resistant (permitting greater fuel efficiency and less pollution) than the alloy components in use at that time. Traditional molding techniques for ceramic turbine components required custom machine work and could not generate enough savings to achieve production costs equivalent to super alloy components that would enable marketplace penetration. It was estimated that foreign firms had a two- to three-year head start into researching and developing ceramic components for turbines and that they would soon produce ceramic parts that would overcome this cost barrier, consequently shrinking U.S. manufacturers' market share.

Honeywell International Inc., Ceramic Components (formerly AlliedSignal, Inc.) submitted a proposal to the Advanced Technology Program's (ATP) 1995 focused program competition for "Materials Processing for Heavy Manufacturing". The company proposed to develop and refine a ceramic molding process called "aqueous injection molding" (AIM) to achieve low-cost fabrication of silicon nitride ceramic components.

At the completion of the ATP-funded project, Honeywell had successfully implemented the AIM process improvements, which then required engine test validation funding to lead to commercial production of ceramic turbine splitter vanes. However, the process was unable to reliably mold aircraft engine turbine blades.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* *

Research and data for Status Report 95-07-0003 were collected during October - December 2001.

U.S. Manufacturers Losing Ground to Foreign Competitors

In 1995, structural ceramics were seen as relatively high-cost, high-performance materials that could provide significant improvements in efficiency and performance, as well as reduce emissions and pollution. These improvements could substantially increase the domestic and international market share of U.S. heavy equipment manufacturing firms. However, unless the cost of producing structural ceramics dropped significantly, the material would not be adopted in turbine engines, and these improvements would not be realized. Foreign manufacturers had begun research

into structural ceramics in the early 1990s and were several years ahead of U.S. manufacturers. U.S. firms feared that the cost of foreign-based manufacturing would continue to decline, which would increase the foreign-based market share in this growing industry.

New Technology Could Increase U.S. Market Share

The focus of Honeywell's ATP project was to develop an aqueous injection molding (AIM) process that could achieve a tenfold reduction in the cost of silicon nitride turbine blades for industrial turbine engines. A number of complex-shaped silicon nitride components were being fabricated at Honeywell in small volumes (under

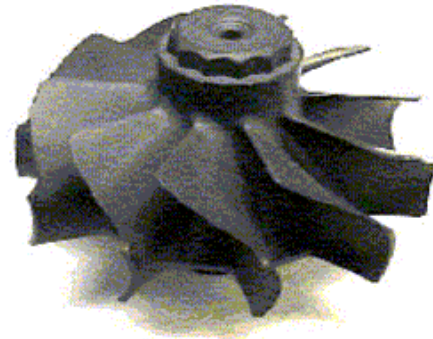
100 parts per order) before the start of the ATP-funded project. However, the existing process used to produce these parts involved slip-casting parts larger than necessary in plaster molds with extensive machining of "green" bisque stock. This resulted in costs that were 10 times higher than what the market would bear. These high manufacturing costs were due to extensive processing labor, low overall process yields, high equipment costs, and the machining of extensive amounts of waste material that could not be reused.

This process would have a significant impact on the industry's ability to manufacture high-quality, complex, ceramic components at low cost and with higher yields.

Honeywell's proposed process would form components close to the final shape of the part to minimize custom machining and wasted raw material. It was expected that the successful development of this process would have a significant impact on the industry's ability to manufacture high-quality, complex, ceramic components at low cost and with higher yields. The initial target application was to produce cost-effective silicon nitride components in industrial gas turbines and turbo-alternators. The end users in the utility power and transportation industries would be the ultimate beneficiaries, because ceramic component implementation in these applications would lower component costs, increase performance and efficiency, and decrease nitrogen oxide and carbon dioxide emissions. Moreover, Honeywell asserted that the successful development of the AIM technology would place U.S. companies in a competitive or superior position for manufacturing heavy-duty engines for vehicles, aircraft, and power plants. This improved position in the engine manufacturing market could lead to an increase in employment in the United States.

Technology Has Potential for Wide-Ranging Economic Impact

Honeywell applied to ATP for cost-shared funding under ATP's 1995 focused competition in "Materials Processing for Heavy Manufacturing". ATP funding was sought to accelerate the process improvements to AIM.



The existing process required custom machining of "green" bisque ceramics.

Before the start of the ATP-funded project, government and industrial studies had identified advanced materials, including structural ceramics, as critical technologies for maintaining U.S. economic strength in the manufacturing sector. Structural ceramic components were already in production in the United States for applications such as cutting tools, heat exchangers, filters, and heat engine components. The market was expected to grow at a rate of 10 to 15 percent annually for the next decade. However, two foreign companies were ahead of U.S. manufacturers in the research and development of the advanced materials used in turbines and turbo-alternators. Without significant improvement by U.S.-based industries, the nation's manufacturing market share in heavy equipment was expected to decline. On the other hand, if Honeywell were able to successfully develop an AIM process, U.S. manufacturers could use these components to gain a competitive edge in major manufacturing markets such as chemicals, construction, electronics, power generation, environmental controls, aircraft, recreational goods, and surface transportation.

Honeywell Identifies Technical Barriers to New Technology

Significant technical issues needed to be addressed during the ATP-funded project in order to achieve the tenfold reduction in costs for silicon nitride complex-shaped components. The five most significant barriers were:

1. Producing feedstock with consistent and stable properties
2. Consistently forming the feedstock into void-free pellets

3. Developing a mold design that would assure complete mold filling while minimizing defect formation and uniform packing to minimize distortion during sintering
4. Creating a consistent injection-molding process to achieve reproducible, defect-free molded components (e.g., no voids, no leaks, no cracking)
5. Minimizing cycle times

Solving each technical barrier would be considered incremental improvements. The challenge, however, of integrating all of the individual improvements into a process that ran without human intervention, with 80 percent fewer cycle steps and generating a tenfold cost reduction, was daunting and risky. It was precisely this challenge that Honeywell needed to meet to remain competitive and even to surpass foreign manufacturers.

Project Research Plan Specifies Three Critical Development Tasks

The Honeywell research plan defined three tasks to be completed over a 27-month period:

1. Refine the AIM technology for silicon nitride to achieve consistent high yields. This meant refining individual process steps and developing and implementing improved intelligent process controls to optimize the silicon nitride AIM fabrication process.
2. Operate the optimized AIM fabrication process for 12 months to evaluate the process's capability and to make further refinements based on process output results.
3. Evaluate a number of the components manufactured during task two; this work would be performed by Solar Turbines, a purchaser of turbine blades for industrial engine use. Solar Turbines would perform a 500-hour engine test to determine component performance, quality, and consistency.

Project Encounters Both Unexpected Barriers and Successes

Tasks one and two started as planned but later experienced some problems and caused delays that extended the period of performance for Honeywell

during the course of the ATP-funded project. The preparation process for AIM feedstock, the actual molding mixes, and the mold designs created during tasks one and two led to improvements in flow and stability, as well as reduced cracks. However, the team still found cracks in the parts, unfilled spaces, and distortion. Although they continued testing, adjusting feedstock, and upgrading a number of parts, the following three technical problems remained, which made it difficult to achieve the dimensional accuracy required:

1. Bulging part platforms and bases due to feedstock/binder problems
2. Part distortion due to poor powder packing
3. Lot variation in feedstock and molding performance

By the close of the ATP-funded project in 1998, Honeywell had achieved a 52.7-percent yield of turbine blade parts with no defects, but still had consistent problems with cracked or deformed blades. The parts' size and geometry were not suitable for the AIM process. Therefore, Honeywell experienced difficulty in achieving engine quality hardware for Solar Turbines to insert into the engine and complete the performance test.

Nevertheless, the team was able to develop the following key criteria for parts that could be made cost effectively using the AIM process:

- Parts with a maximum dense cross-section less than 0.25 inches
- Parts that can be constrained to a specific shape during densification
- Complex, small parts less than 2 inches in any dimension
- High-volume, complex-shaped components
- Parts that cannot be made in other ways

Although the team was not able to complete the project, they did predict production costs for a turbine blade, assuming the technical performance requirements could be met. Solar Turbines requested a quotation for

5,000 blades per year at a price of less than \$200 each. Based on a volume of 7,500 per year to allow for defects, the slip-casting/machining method would cost approximately \$436 each. AIM would cost \$175, a savings of 60 percent. The potential for success existed.

Honeywell had achieved a 52.7-percent yield of turbine blade parts with no defects, but still had consistent problems with cracked or deformed blades.

After the conclusion of the project, the AIM technology did allow the company to manufacture some small, complex parts, such as diffuser splitter vanes for potential use in commercial aircraft engines, which they had been unable to form prior to the ATP-funded project. The original aluminum part had an erosion problem, which would benefit from the harder, more wear-resistant silicon nitride replacement. Estimated potential annual production sales were \$140,000. Honeywell also worked with power-generation turbine companies to evaluate additional components, such as blades and nozzles. Honeywell Ceramic Component's production unit grew from 4 percent of sales in 1995 to 38 percent of sales in 1998.

Honeywell Shares Project Knowledge

During the ATP-funded project, Honeywell shared its knowledge and prototypes with Solar Turbines and published technical information about the AIM technology in the *American Ceramic Society Bulletin*, the *Journal of the American Ceramic Society*, and in the *American Society of Mechanical Engineers' Journal of Applied Mechanics*. Honeywell presented information about its technical achievements at the International Gas Turbine Conference, the American Ceramic Society Annual Meeting, and the American Society of Mechanical Engineers Turbo Expo. As a result of the ATP-funded project, Honeywell reported being two to three years ahead of where it would have been in the development and refinement of AIM without ATP support. Thus, if the project had been more successful, it could have assisted in closing the research gap with foreign competition as well as putting Honeywell and its

customers in a position to challenge foreign-based competitors and increase U.S. manufacturing market share.

Conclusion

Honeywell successfully utilized the aqueous injection molding (AIM) process to fabricate ceramic splitter vanes in 1998, after the close of the ATP-funded project, and had plans to obtain funding for engine qualification testing. They also developed plans to commercialize other small, complex high-volume ceramic turbine components, such as nozzles and blades. Consistent technical problems with the larger turbine blades, however, stalled the original project at the prototype stage due to cracking and distorting. Eventually, with continued research, Honeywell planned to use the AIM technology in making varied components like nozzles and blades, as well as unique wear parts for microturbines and stationary turbines.

PROJECT HIGHLIGHTS

Honeywell International Inc., Ceramic Components (formerly AlliedSignal, Inc.)

Project Title: Innovative Molding Technique to Produce Ceramic Turbine Components (Aqueous Injection Molding for Low Cost Fabrication of Silicon Nitride Components)

Project: To develop and refine an aqueous injection molding (AIM) process for low-cost silicon nitride components, enabling their use in high-performance turbine engines for auxiliary power units and other turbo machinery, such as stationary power-generating systems.

Duration: 9/30/1995-9/29/1998

ATP Number: 95-07-0003

Funding** (in thousands):

ATP Final Cost	\$ 738	40%
Participant Final Cost	<u>1,128</u>	60%
Total	\$1,866	

Accomplishments: This ATP-funded project assisted Honeywell, a major U.S. manufacturer of ceramic components, in furthering its ceramic processing research knowledge. This eventually closed the three-year research gap between Honeywell and its foreign-based competition. As a result of its ATP-funded research, in 1998, Honeywell successfully implemented the AIM process improvements for ceramic splitter vanes. After the close of the project in 1998, Honeywell planned to devote several million dollars over the next 5 to 10 years to overcome the remaining technical barriers to the aqueous injection molding of small ceramic turbine components and wear parts for microturbines and stationary turbines.

In order to share some of the knowledge gained during the ATP project, Honeywell published information about its technical achievements in several engineering journals and presented its findings at three conferences during the course of the research.

Commercialization Status: As a result of its research into AIM, Honeywell implemented process improvements for the fabrication of ceramic splitter vanes, a part that the company was not able to fabricate before the start of the ATP-funded project. They had plans to commercialize other small, complex, high-volume parts like blades and nozzles. The Ceramic Components Unit of Honeywell increased production from 4 percent of sales in 1995 to 38 percent in 1998.

Outlook: Through the ATP-funded project, Honeywell recovered research and development time lost to foreign-based competitors and was able to fabricate one ceramic component produced by AIM. Manufacturing additional complex parts will require more development time, and there is still some uncertainty whether AIM will result in the manufacture of a substantial number of turbine parts. Small size, complexity, and high sales volume will be the keys to cost effectiveness. The outlook for this technology is uncertain.

Composite Performance Score: * *

Focused Program: Materials Processing for Heavy Manufacturing, 1995

Company:

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** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

IBM T.J. Watson Research Center

Enhancing Polymers to Achieve Improved Conductivity

In 1993, engineers at the IBM T.J. Watson Research Center believed they were close to a breakthrough in their research into conducting polymers. Conducting polymers, a form of plastic that would carry electric current like metals, were created in the late 1970s, but could not be easily manufactured and, once made, were not stable. Early experiments with acid-doped polyaniline (PANI), one such polymer, produced positive results and indicated that PANI might be able to carry electric current.

The research engineers believed that, with the appropriate acid dopants added to PANI, they could induce a crystalline PANI structure with conductivity similar to copper wire. If the IBM T.J. Watson Research Center succeeded, they would finally bridge the gap between conducting polymer theory and practice. However, IBM's conductivity goal represented a substantial technical risk. Researchers had been trying to reach that goal for nearly 20 years without success.

Given the high risk, company executives would not commit enough funds for the extensive research required to develop PANI. Therefore, the IBM engineers joined with professors at the University of Pennsylvania and the Ohio State University to submit a proposal to the Advanced Technology Program (ATP), and, in 1993, ATP awarded funds for a three-year project.

By the end of the ATP-funded project, the researchers had met some of their goals for developing easily manufactured PANI that could conduct electric current in a fast and stable manner. Specifically, IBM researchers enhanced conductivity, solubility, and thermostability. As a result of this success, executives at the IBM T.J. Watson Research Center invested an additional \$4 million for research into PANI. In 1997, the Research Center also licensed a water-soluble version of the polymer to Monsanto, a plastics manufacturing company. Although no products resulted from this licensing effort, Monsanto did find a use for PANI as an anti-corrosion primer.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* * *

Research and data for Status Report 93-01-0149 were collected during January - February 2003.

Decades-Old Material Never Achieved Full Potential

Conducting polymers, a unique class of electronic materials that emerged in the late 1970s, promised many next-generation products. For example, conducting polymers had the potential to allow plastics to replace metals in many applications such as plastic wires and cables, rechargeable batteries, and electronic circuitry for semiconductor applications, as well as shielding against electrostatic discharge and electromagnetic interference. Conducting polymers could not be used in these applications, however,

because they were either unstable, difficult to process, or too expensive. They had never reached their full potential of bringing the advantages of plastics to the electronic applications that traditionally required metal.

PANI Promises Advantages Over Other Polymers

In 1993, IBM T.J. Watson Research Center engineers proposed a research plan to bridge the gap between metal-like conductivity and plastic-like flexibility, processability, and recycleability. Initial research on the conducting polymer polyaniline (PANI) showed that it

had significant advantages over other polymers. For example, PANI was highly soluble and easy to synthesize, and its properties could be tailored to specific applications. The challenge remained to dope PANI with different acids to dramatically increase its thermal stability and ability to conduct electricity. Much of the prior research had involved changing the overall chemical backbone of conducting polymers without regard to the molecular structure of the polymer. IBM T.J. Watson Research Center engineers believed that the polymer's structure could be the key to enhanced strength and improved characteristics. The engineers believed they could create PANI that had a highly ordered, crystalline structure with the desired properties.

Broad-Based Benefits Would Impact U.S. Economy

The research engineers believed that the benefits from a successful research project would impact the U.S. economy in three stages. During the first stage, the PANI derivative with improved thermal and environmental stability could displace metal and carbon fillers in the aerospace industry by shielding components from potentially dangerous electrostatic discharges. The second stage of economic benefits would be achieved if PANI could be created with increased conductivity. Blends of PANI with other polymers would also have wide applicability for electromagnetic interference shielding in the form of molded structural elements for a variety of products, including computer equipment, consumer electronics, and aerospace structural composites.

Conducting polymers could not be used in many applications because they were either unstable, difficult to process, or too expensive.

A third stage of benefits would occur if the conducting polymers that had enhanced thermal and environmental stability also had conductivity that was close to that of copper. If this were achieved, it would open up a broad range of applications in which PANI could displace metallic conductors or enable new technologies based on its novel properties. For example, hazardous, lead-containing solder used to attach microelectronic components to printed circuits could be replaced with a highly conductive, environmentally safe polymer. Microcircuits could use photo-imageable PANI to

replace metal wiring. This same form of PANI could also be used for the circuits that interconnect semiconductor devices.

Internal Funding Unavailable for High-Risk Research

IBM T.J. Watson Research Center engineers decided to pursue a dual-stage research process in order to achieve the proper crystalline form ideal for conductivity. The first step in the process was to design and synthesize dual-functional dopants that would increase the strength of PANI and help the polymer chains self-assemble in a crystalline fashion. The second step would involve the use of dopants that could link across crystalline structures to "lock" the crystalline chains in place for even stronger polymers. Each approach would attempt to strengthen the material and increase its stability. If both succeeded, the engineers believed stability and conductivity would then increase to commercially acceptable levels.

The engineers believed they could create PANI that had a highly ordered, crystalline structure with the desired properties.

The engineers had just begun researching PANI's properties in 1993. In order to carry out parallel research on dual-functional dopants, the Research Center would have been forced to take on risk that was too high for internal funding requirements. Scarce research resources would not be available for a parallel research process. IBM T.J. Watson Research Center engineers felt more could be accomplished with a parallel research program than they could accomplish alone. Therefore, to pursue the parallel research plan, they sought university partners and applied to ATP for support. The university partners included the University of Pennsylvania and the Ohio State University. These professors have specialized expertise in the area of conducting polymers, and, through this project, their students would gain unique industrial research skills in this field.

The goal of the proposed research program was to achieve the properties needed to commercialize conducting polymers. These properties included thermal and environmental stability of the conducting form of the polymer; processability in a variety of

solvents, as well as the ability to use several processing techniques to broaden applications; and conductivity over a wide range of conductivity applications, from electrostatic discharge to the high-risk goal of conductivity equivalent to metal. Because these goals were crucially important to industry and required outside funding to focus enough attention on the project to create new, innovative solutions, ATP awarded \$1.452 million to conduct parallel research over a three-year period.

Research Engineers Improve PANI and Achieve Project Goals

At the start of the ATP-funded program, the thermal stability of the conducting form of PANI was less than 150°C. The goal by the end of the project was to increase that to 250°C in order to extend PANI's applicability to higher temperature processing and to broaden its use in blends and extrusions such as polycarbonate materials. By using novel, thermally stable dopants, IBM T.J. Watson Research Center engineers reached thermostability in excess of 250°C.

At the project's start, the solubility of the base form of PANI in a common solvent was five percent. Engineers hoped for solubility closer to 20 percent, depending on the structure of PANI that the project ultimately settled upon. The increase in solubility would allow the favorable properties of PANI to permeate the final solvent and give PANI favorable properties. The scientists feared that solutions with greater amounts of PANI (more than 20 percent) would either gel immediately or would have a very limited shelf life, making them unusable for commercial applications. Lower solubility levels (closer to the five-percent industry standard before the ATP-funded project) limited the thickness that could be obtained in a final product, thereby limiting the potential uses for PANI-doped conducting materials. By the end of the research program, engineers had increased solubility to 15 percent, allowing new polymers, copolymers, and blends to be formed that could conduct electricity. This also allowed the development of the first electricity-conducting fibers from PANI.

PANI was unable to conduct much electric current at the start of the project. The high-risk goal of the research program was to enable these plastics to carry enough electric charge to compete with traditional

copper wiring. By the end of the project, PANI still could not compete with copper wiring, but conductivity had been increased by 2.5 times. This improvement enormously increased the useful range and applications of these materials.

IBM T.J. Watson Research Center engineers also successfully induced PANI to align into crystalline form during the ATP-funded project. The research showed promise in locking the crystalline chains in place for greater strength. This resulted in \$4 million in additional funding from IBM for continued research. The engineers, along with the university partners, continued this research after the close of the ATP-funded program, and their efforts resulted in the licensing of a water-soluble version of PANI to Monsanto Chemical Company to explore creating different insulating materials. While no insulating materials resulted, Monsanto uses PANI as an anti-corrosion primer on other products.

The project generated substantial new knowledge in the field of conducting polymers; resulted in 13 patent applications, 4 copyright applications, 23 presentations, and 31 publications; and led to improvements in conducting polymers' conductivity, solubility, thermostability, and strength.

Conclusion

Engineers at the IBM T.J. Watson Research Center, along with researchers at the University of Pennsylvania and the Ohio State University, collaborated in an ambitious research project to create new polymers that could replace copper conductors in electronic and telecommunications equipment. Though the engineers were unsuccessful in making a polymer with the electrical conductivity of copper, they did improve polyaniline significantly compared to where conducting polymers would have been without ATP support. Moreover, the engineers created a material that was licensed to a plastics manufacturing company. The company has subsequently used the material as an anti-corrosion primer on several products.

PROJECT HIGHLIGHTS

IBM T.J. Watson Research Center

Project Title: Enhancing Polymers to Achieve Improved Conductivity (Three Dimensional Engineering for Advanced Applications)

Project: To develop the basic technology and knowledge base for new conducting polymers with good thermal and environmental stability and an electrical conductivity approaching that of metallic solder.

Duration: 4/1/1994-3/31/1997

ATP Number: 93-01-0149

Funding** (in thousands):

ATP Final Cost	\$1,453	73%
Participant Final Cost	<u>539</u>	27%
Total	\$1,992	

Accomplishments: IBM T.J. Watson Research Center engineers worked with researchers at the University of Pennsylvania and the Ohio State University to enhance a conducting polymer of doped polyaniline (PANI). PANI represented a substantial improvement over what was available at the start of the ATP-funded research program. Specifically, engineers created PANI with the following improved characteristics that brought the material closer to a commercializeable form:

- Increased thermal stability from 150°C to greater than 250°C, making it possible to manufacture the particular crystalline form and to use it in commercial products
- Increased processability and solubility, but not enough for most commercial applications without additional research
- Increased conductivity by 2.5 times, which showed promise but did not keep up with changing market demand for even more conductivity

The IBM T.J. Watson Research Center also created a water-soluble version of PANI and licensed it to Monsanto Chemical Company. Knowledge gained during the ATP-funded project led to 4 copyright applications, 23 presentations, 31 publications, 13 patent applications, and 11 awarded patents:

- "Deaggregated electrically conductive polymers and precursors thereof" (No. 5,804,100: filed January 9, 1995; granted September 8, 1998)

- "Methods of fabrication of deaggregated electrically conductive polymers and precursors thereof" (No. 6,087,472: filed January 9, 1995; granted July 11, 2000)
- "Electrically conductive pressure sensitive adhesives" (No. 5,645,764: filed January 19, 1995; granted July 8, 1997)
- "Methods of fabrication of deaggregated electrically conductive polymers and precursors thereof" (No. 5,736,623: filed May 30, 1995; granted April 7, 1998)
- "Methods of fabrication of cross-linked electrically conductive polymers and precursors thereof" (No. 6,030,550: filed February 2, 1996; granted February 29, 2000)
- "Plasticized, antiplasticized and crystalline conducting polymers" (No. 5,928,566: filed March 22, 1996; granted July 27, 1999)
- "Polycrystalline conducting polymers and precursors thereof having adjustable morphology and physical properties" (No. 5,932,143: filed March 22, 1996; granted August 3, 1999)
- "Methods of fabricating plasticized, antiplasticized and crystalline conducting polymers and precursors thereof" (No. 5,969,024: filed March 22, 1996; granted October 19, 1999)
- "Charge transfer complexes between polyaniline and organic electron acceptors and method of fabrication" (No. 5,776,370: filed April 25, 1996; granted July 7, 1998)
- "Methods of fabricating branched electrically conductive polymers and precursors thereof" (No. 5,958,301: filed September 27, 1996; granted September 28, 1999)
- "Methods of fabrication of deaggregated electrically conductive polymers and precursors thereof" (No. 6,005,070: filed January 16, 1997; granted December 21, 1999)

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

PROJECT HIGHLIGHTS

IBM T.J. Watson Research Center

Commercialization Status: A water-soluble version of PANI was licensed to Monsanto Chemical Corporation in 1997, and IBM is pursuing further licensing opportunities.

Outlook: According to the IBM T.J. Watson Research Center's final quarterly report issued before the close of the ATP-funded project, the research seemed promising. That positive outlook resulted in \$4 million in additional funding from IBM for continued research. Between 1997 and 2003, however, the research has slowed considerably, and the promise of the technology is somewhat muted by changes in market demands. While the polymer research took a large leap forward as a result of the ATP-funded research, IBM will need more licensing partners than the one-time Monsanto license in order to make a commercial impact. The outlook for this technology is uncertain.

Composite Performance Score: * * *

Company:

IBM T.J. Watson Research Center
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Phone: (914) 945-3217

Subcontractor:

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PCC Structural, Inc. (a subsidiary of Precision Castparts Corporation)

New Casting Technology to Produce Large Superalloy Components

In the mid-1990s, investment casting was used to produce superalloy components in complex shapes for aircraft and gas turbine engines. Investment casting is a process for creating near-net precision metal parts. There were, however, certain limitations to using this technology; for example, investment-cast components made with superalloys could not be scaled up for use in the largest industrial gas turbine (IGT) engines. Moreover, the technology was expensive due to the high cost of tooling. Another casting technology, sand casting, could be used to produce larger, complex industrial components at a lower cost. However, it could not be used with superalloys because of reactions between the sand mold and the traditionally reactive superalloys. In 1995, PCC Structural, Inc. (PCC), a leading producer of components made by investment casting, proposed to integrate the benefits of investment-casting technology and the economy of sand-casting technology to produce large castings for industrial equipment industries, specifically the IGT industry.

In 1995, PCC was awarded three years of cost-shared project funding from the Advanced Technology Program's (ATP) focused program competition, "Materials Processing for Heavy Manufacturing." By the end of the project, PCC had successfully demonstrated a new process and had created prototype components; however, the company did not commercialize components made with this process at that time. Deregulation of the U.S. power market in 1998 resulted in greater competition among energy producers, and manufacturers became reluctant to purchase parts made with a new technology. As of 2003, PCC is still interested in further developing and commercializing this ATP-funded technology.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

No Stars

Research and data for Status Report 95-07-0011 were collected during March - April 2003.

Larger Investment Castings Needed for Industrial Applications

Investment casting is one of the oldest metallurgical techniques. It has been used since 4000 B.C. to craft detailed objects ranging from pre-Columbian art to military parts to 20th century dental crowns and inlays. In the mid-1990s, investment casting was primarily used to produce complex parts from superalloys for aircraft and gas turbine engines. The disadvantages of this technology, however, were that the size of cast parts was limited to about two meters in diameter, and the tooling was an expensive and lengthy process.

Traditionally, large components are made with sand casting. With this technology, complex industrial components of any size can be manufactured economically. However, in the mid-1990s, superalloys could not be used because they reacted strongly with the sand mold, causing defects and requiring costly machining. Moreover, superalloys required protection from the air and had to be cast in a vacuum or inert atmosphere, while sand casting was normally performed under standard atmospheric conditions.

PCC Proposes New Casting Technology

PCC Structurals, Inc. (PCC) was a leading producer of components made by investment casting. The company wanted to combine the process advantages of investment-casting technology with the economy of sand-casting technology to cost-effectively produce large structural superalloy components for industrial equipment industries, specifically the industrial gas turbine (IGT) industry.

By 1995, land-based (non-aviation) gas turbine (LBGT) engines had achieved increased output, higher energy efficiency, and fuel flexibility through higher firing temperatures. These engines could withstand temperatures in excess of 1100°F. However, the exhaust frames for the engines were made from sand-cast stainless steel alloys. To increase the heat tolerance of the exhaust frame and to prevent oxidation, foil was added to the outside of the frame, and forced-air cooling was applied to it with a fan. However, the foil was expensive, and the cool air lowered the efficiency that the operating unit had gained with the higher temperature. In addition, the surface of the component was rough from its exposure to sand, which increased the cost of machining the component. Another problem with the sand-cast exhaust frames was the multiple joints that were attached to it. Joints that are exposed to temperature gradients are prone to developing cracks that need costly, frequent maintenance.

With PCC's proposed casting technology, an exhaust frame would be manufactured as a single casting with a high-temperature alloy, thereby eliminating the need for expensive foil and air cooling and resulting in a component with a smooth surface finish. PCC's technology would also increase the reliability of the frame and would eliminate maintenance costs for repairing the welded joints that are often made up of different metals. Furthermore, a single-cast frame would eliminate the costs of attaching, securing, and inspecting multiple parts to a frame.

The reduction in cost and maintenance of single-cast components would increase their overall value in the marketplace and make them more competitive worldwide. PCC anticipated that the annual domestic market for cast superalloy exhaust frames and other types of structural components for LBGT engines would triple in size once the new technology was developed.

Financial Assistance Needed for High-Risk Project

PCC understood that developing the new casting technology was a high-risk endeavor. Although sand casting was commonly used to make large structural components, there was a substantial technical risk associated with the sand casting of superalloys. At the time, there was little knowledge regarding either the interaction between mold materials and molten superalloys or the metal-handling requirements.

Developing a casting technology that combined the superalloy processing capabilities of investment casting with the economic advantages of sand casting would result in many benefits.

Because the project risks were more than PCC could assume at the time, the company sought financial support from ATP through the focused program, "Materials Processing for Heavy Manufacturing." This support would allow the company to develop the new casting technology 10 years sooner than if it funded the project itself.

PCC Anticipates Broad-Based Benefits

PCC believed that developing a casting technology that combined the superalloy processing capabilities of investment casting with the economic advantages of sand casting would result in many benefits. With the new casting technology, components for the largest IGT engines could be manufactured with superalloys, enabling these engines to operate at higher temperatures with greater efficiency and lower emissions. The new casting technology would also simplify the design and reduce the maintenance of components for IGTs, increasing the life cycle and lowering the cost of exhaust frames. The cost-efficient manufacturing of large superalloy castings would also decrease the overall cost of U.S. turbines, acting as an incentive to U.S. manufacturers to adopt the high-efficiency IGT technology.

Furthermore, the new technology could be applied to other large components used in combined-cycle LBGT engines, which, at the time, were manufactured with chrome steel. The technology could also be used with components in industries such as mineral processing, petrochemical, and pulp and paper.

New Casting Technology Is Developed but Not Commercialized

To meet its goal of developing a new casting technology during the ATP-funded project, PCC combined features of both investment casting and sand casting. The company made a shell for the component, which was an investment-casting technique. PCC then poured the superalloy into the shell and cast the component in an open-air environment (a sand-casting technique). Argon, an inert gas, was used as a shield to protect the superalloy component from oxidation.

The company also investigated the following:

- Use of machinable foam as an alternative to wax as a pattern; use of foam would be less expensive for large parts and would allow the shape to be modified early in the casting process.
- Appropriate sand coatings that would minimize interactions between the sand molds and the superalloys.
- Use of a furnace instead of a vacuum-shielding technique to decrease the cost of the new process.

During the ATP-funded project, PCC characterized several alloys, from which it selected one. The company also used process modeling to better understand the interrelationships between a new alloy, point geometry, and process conditions. Process modeling allowed the company to determine the most significant process parameters through modeling iterations instead of performing costly trial-and-error experiments.

By the end of the ATP-funded project, PCC had created prototypes of several different castings, with assistance from subcontractors General Electric (GE) Power Systems and Knight+Packer Inc., which provided expertise in sand-casting technology, modeling, and test evaluation. PCC had also demonstrated that one prototype would work at 1100°F.

GE Power Systems is the market leader in the IGT marketplace, commanding roughly a 50-percent share in 1998. In an effort to maintain and expand its place in the market, the company wanted to reduce the cost of the exhaust frame, one of the major components in the turbine. A strategy to reduce the cost of this component was to develop a modified investment-cast exhaust

frame that could withstand high temperatures and thus eliminate the need for a heat shield. GE had identified PCC to develop this technology with GE's assistance.

PCC believed that GE and other PCC customers such as Westinghouse would be interested in purchasing components made with the new casting technology. However, with the deregulation of the U.S. domestic power market in 1998, competition among energy producers increased and energy prices declined. Moreover, energy producers were limiting their investments in new technology. (At the time, the average cost of a new exhaust frame was \$275,000.) Thus, PCC decided not to commercialize the new casting technology in 1998. The company is, however, interested in commercializing the technology in the future, at a time when it is economically feasible to do so.

With the deregulation of the U.S. domestic power market in 1998, energy producers were limiting their investments in new technology.

Since the project ended, PCC has used the knowledge it gained to create a casting grate (a handling device that supports molds that are to be cast), based on a measurement the company developed during the ATP-funded project.

Conclusion

With ATP's assistance, PCC Structural, Inc. (PCC) created prototypes of several different castings through a new process that combined the benefits of investment-casting and sand-casting technologies. However, PCC did not commercialize components made with the new casting process. By the end of the ATP-funded project in 1998, the U.S. domestic power market had been deregulated, which resulted in greater competition among energy producers and a decline in energy prices. As energy profits became less predictable, manufacturers, such as General Electric, were less inclined to take risks and purchase components made with a new technology. As a result, orders from the industrial gas turbine industry, one of PCC's major customers, decreased. Since 1998, PCC has maintained contact with potential customers for the new components. However, at this time, there is little interest from these customers in using PCC's new casting process.

PROJECT HIGHLIGHTS

PCC Structurals, Inc. (a subsidiary of Precision Castparts Corporation)

Project Title: New Casting Technology to Produce Large Superalloy Components (Development of Casting Technology to Produce Large Superalloy Castings for Industrial Applications)

Project: To develop a casting technology that combines the superalloy processing capabilities of investment casting with the economic advantages of sand casting to achieve part sizes sufficient to produce exhaust frames for industrial gas turbine (IGT) engines.

Duration: 9/15/1995-9/14/1998

ATP Number: 95-07-0011

Funding (in thousands):

ATP Final Cost	\$1,445	68%
Participant Final Cost	<u>683</u>	32%
Total	\$2,128	

Accomplishments: PCC Structurals, Inc. (PCC) developed prototype castings using a new casting process that will allow manufacturers to produce large structural superalloy components for industrial equipment industries, such as the IGT industry.

Commercialization Status: PCC did not commercialize components made with the casting process it developed in the ATP-funded project. By 1998, when the project ended, deregulation had taken effect in the U.S. power market and profits had become less certain for energy producers. As a result, manufacturers of IGTs such as General Electric, who were expected to purchase the new components, were no longer interested because making a change in their existing equipment could affect future sales.

Outlook: In 2003, the energy market still has not recovered sufficiently to enable PCC to commercialize components made with the new casting technology. The company is, however, interested in continuing to develop the technology and to market components made with it in the future.

Composite Performance Score: No Stars

Focused Program: Materials Processing for Heavy Manufacturing, 1995

Company:

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Subcontractor:

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Knight + Packer, Inc.
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Research and data for Status Report 95-07-0011 were collected during March - April 2003.

Praxair, Inc.

O₂-Selective Materials to Lower the Cost of Oxygen

In the mid-1990s, American industries were looking for ways to save energy, cut costs, and contribute to environmental cleanliness, either through improved work processes or new technologies. For example, furnace operators in various industries were interested in converting from air to oxygen (O₂)-enriched air for their combustion processes, but were prevented from doing so by the high cost of oxygen. Oxygen or oxygen-enriched air reduces energy consumption by 25 percent to 60 percent and reduces environmental emissions. However, oxygen prices would have to be reduced by at least 25 percent to provide incentive for most furnace operators to commit the resources necessary to convert to O₂-based processes. Praxair, Inc., the second-largest supplier of industrial gases worldwide, wanted to develop highly efficient O₂-selective materials (materials used to separate oxygen from air) that could be used in sorption or membrane air separation systems. This technology could reduce the cost of oxygen by 25 percent to 50 percent, a sufficient incentive for industrial furnace operators to convert to O₂-enhanced combustion processes.

Due to financial limitations, Praxair was unable to independently fund this long-term project. In 1994, the company was awarded co-funding from the Advanced Technology Program (ATP). By the end of the three-year project, Praxair had successfully developed materials that are highly selective for oxygen. It is unclear whether this class of materials can be applied economically to air-separation systems; however, they may be useful initially in niche commercial applications. In 2003, Praxair has continued to work to bring these materials closer to commercialization through a Department of Energy-funded project in which they are attempting to separate nitrogen and oxygen from air. Although this is a slightly different application, it uses essentially the same class of materials.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

*

Research and data for Status Report 94-01-0111 were collected during October - December 2002.

Praxair Develops First-Generation Air-Separation System

In the 1950s, industrial gas companies began to recognize the potential of using noncryogenic systems for separating oxygen (O₂) from air. (Cryogenic separation is a distillation process that occurs at very cold temperatures.) By the late 1960s, Praxair, Inc., the second-largest supplier of industrial gases worldwide, had developed a pressure swing adsorption (PSA) unit for producing oxygen using molecular sieves it had invented in the 1950s. The molecular sieve materials used in current commercial PSA systems separate air by selective adsorption of nitrogen. Praxair determined that the PSA process provided a practical vehicle for using O₂-selective sorbents to generate oxygen at a reduced cost. However, there were no commercial

O₂-selective materials that met the performance requirements.

In the mid-1980s, Praxair developed the first generation of hollow-fiber membrane systems for air separation, which led to even greater interest in O₂-selective materials. Membrane systems produce nitrogen very efficiently, but the oxygen that was produced had very low purity due to limitations in the selectivity of the membrane materials.

In 1994, when Praxair applied for the ATP award, the company had already spent six years, through an in-house research program, investigating and evaluating the potential benefits of O₂-selective materials in PSA and membrane systems. This preliminary work indicated that equilibrium-based O₂-selective sorbents

containing transition element complexes (TECs) had the best chance of overcoming the high-risk technical barriers. A TEC is a combination of a transition element ion to provide an oxygen-binding site and a series of groups provided by organic components to modify the oxygen interaction characteristics.

Praxair Focuses on Advancement of O₂-Selective Materials

Praxair's goal was to develop O₂-selective materials as sorbents for PSA systems and to establish their practicality for use in membrane systems. Preliminary assessments indicated that the cost of producing oxygen could be reduced by 25 percent to 50 percent. In addition, replacing the nitrogen (N₂)-selective sorbents that were being used in PSA systems with this new material could reduce power consumption by 50 percent and production costs by more than 25 percent for oxygen, with purities in the 40 percent to 90 percent range.

Oxygen or oxygen-enriched air reduces energy consumption by 25 percent to 60 percent and reduces environmental emissions.

Although such a technological breakthrough would revolutionize the industrial gas business and the industries it supplies, the research and development (R&D) challenges would be considerable. In 1994, financial limitations had forced Praxair to channel all resources available for R&D into those activities that were most likely to succeed and that would provide the most immediate return on investment. Therefore, Praxair proposed the project to ATP for funding and received a three-year, \$1.22 million award.

Lower Oxygen Costs Would Spur Economic Growth

Praxair believed that lowering the cost of oxygen by 25 percent to 50 percent would enable many manufacturers, especially in steel-making, glass-making, and petroleum refining, to switch from air to oxygen or oxygen-enriched air for their combustion processes. This would dramatically improve their manufacturing processes, resulting in less pollution, higher productivity, and lower production costs. For example, switching to oxygen can reduce fuel

consumption by 60 percent. The total savings potential for processes that currently use oxygen and for those that could be converted to oxygen or oxygen enrichment was estimated at \$378 million annually.

In the long term, this savings could lead to economic growth, increased U.S. competitiveness, and more jobs. In the near term, as individual companies made the investments necessary to change from air to oxygen, other beneficiaries would include equipment suppliers, architecture and engineering firms, construction contractors, and material suppliers.

Material Design Guidelines Are Defined

Praxair's primary goal was to develop a new O₂-selective material that could be used for commercial air separation as a sorbent in PSA systems and that had the potential to lower the cost of oxygen by at least 25 percent. In addition, the company planned to develop high-performance membranes that could be used to separate oxygen from air and other gases via facilitated surface transport (that is, the oxygen would be transported across the membrane, assisted by O₂-selective sites embedded in the membrane). The membranes would be used if sorbent costs were too high.

Lowering the cost of oxygen would dramatically improve manufacturing processes, resulting in less pollution, higher productivity, and lower production costs.

Praxair's approach in developing both the O₂-selective sorbents and the membranes would employ TEC sites. Although there had been attempts by others to use sorbents that incorporate TEC sites, the results had not been cost effective.

Praxair defined a series of material design guidelines so that the company could assess project progress and guide materials development. A material would be considered successful if it met all of the following characteristics:

- **High (oxygen loading) under process conditions.** Oxygen loading is the equilibrium amount of oxygen sorbed by the TEC and is a function of temperature and pressure. Oxygen loading is important because it determines the amount of sorbent needed in a PSA process; thus, it affects manufacturing plant size and, therefore, the manufacturer's capital costs.
- **Fast oxygen interaction rates.** The rate at which oxygen is taken up and released affects sorbent productivity in a process cycle, power, and oxygen recovery.
- **Low cost.** The material cost should be consistent with favorable economics for commercial processes at the performance levels achieved.
- **Adequate lifetime under conditions of use.** TEC sorbent lifetime is the time between sorbent installation and replacement in a PSA system. Unless the lifetime is sufficiently long (the target was greater than 10 years), it will be a major contributor to both capital and operating costs.
- **Minimal hazards.** The sorbent should pose no threat to users or the environment under normal operation or under non-ideal conditions. Sorbent that is used up should be suitable for recycling.

At the beginning of the project, there was no single material that met all of these performance requirements. Praxair's greatest challenges were to increase the lifetime of the sorbent and decrease its cost.

Alternative Applications Considered for New O₂-Selective Materials

With the assistance of academic researchers from Clarkson University, the State University of New York, and Iowa State University, Praxair investigated a number of approaches to materials development. By the end of the project, the company had achieved some success in reaching its original goals. Praxair developed several new materials that were highly selective for oxygen. However, it was unclear at the time whether these materials could be used

economically for air separation in PSA or membrane systems. Therefore, Praxair did not pursue these goals after the ATP funding ended. However, Praxair recognized that the properties of the adsorbents produced during the ATP-funded project could potentially be used to produce high-purity nitrogen or to purify nitrogen streams that contain oxygen in a low concentration. Praxair produced approximately two kilograms of the most promising adsorbent.

The company identified a potential application in the aluminum industry that could lead to significant energy savings if the cost of high-purity nitrogen and coproduct oxygen required by the application could be reduced and the product could be commercialized. Within a year after this ATP-funded project ended, Praxair applied for and received funding from the Department of Energy to develop coproduct nitrogen and oxygen from air. Through this project, the company anticipated that it would be able to continue work on the materials developed in the ATP-funded project to bring them closer to commercialization.

Conclusion

Praxair did not reach its goals to develop a new oxygen (O₂)-selective material that could be used commercially as a sorbent for air separation in pressure swing adsorption (PSA) or membrane systems and to lower the cost of oxygen by 25 percent. However, since the ATP-funded project ended, the company has taken the properties of the adsorbents that were developed and is now using them to develop processes to produce coproduct nitrogen and oxygen from air.

Praxair developed several new materials that were highly selective for oxygen.

Since the project's conclusion in 1998, Praxair has been granted three patents for the following: a method for producing nitrogen using O₂-selective adsorbents, a separation process that uses O₂-selective sorbents, and a PSA method for producing an O₂-enriched gas.

PROJECT HIGHLIGHTS

Praxair, Inc.

Project Title: O₂-Selective Materials to Lower the Cost of Oxygen (Advanced Sorbents for Reducing the Cost of Oxygen)

Project: To synthesize, characterize, and demonstrate oxygen-selective materials for use as sorbents in air-separation systems.

Duration: 3/1/1995-2/28/1998

ATP Number: 94-01-0111

Funding** (in thousands):

ATP Final Cost	\$1,089	51%
Participant Final Cost	<u>1,060</u>	49%
Total	\$2,149	

Accomplishments: Praxair developed several new materials that are highly selective for oxygen, which have other potential applications.

Since 1997, Praxair has also been granted the following patents:

- "Method for production of nitrogen using oxygen selective adsorbents"
(No. 5,735, 938: filed January 15, 1997, granted April 7, 1998)
- "Pressure swing adsorption method for production of an oxygen-enriched gas"
(No. 6,475,265: filed October 22, 1998, granted November 5, 2002)
- "Process for separation of oxygen from an oxygen containing gas using oxygen selective sorbents"
(No. 6,183,709: filed January 4, 1999, granted February 6, 2001)

Commercialization Status: The O₂-selective materials developed during this ATP-funded project have not been commercialized. However, as of 2003, Praxair has continued work on their development through a project with the Department of Energy.

Outlook: At this time, it is unclear whether the class of materials developed under this ATP-funded project can be applied economically to the separation of air in pressure swing adsorption systems or membrane systems. According to Praxair, however, it is possible that the materials can be used commercially for niche applications. For example, the company is currently determining whether the materials could serve a role in applications such as oxygen removal from gases or in food packaging.

Composite Performance Score: *

Company:

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Subcontractors:

- Clarkson University
- State University of New York
- Iowa State University

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

Research and data for Status Report 94-01-0111 were collected during October - December 2002.

The Dow Chemical Company

Using Catalysts to Directly Oxidize Propylene to Propylene Oxide

Propylene oxide (PO), a gaseous petrochemical, is a key ingredient in polyurethane foams, coatings, sealants, and adhesives used by the housing and automotive industries. The value of products manufactured using PO derivatives was estimated at \$10 billion in 1995 (1.5 million metric tons), but existing PO production methods were slow and costly. The Dow Chemical Company (Dow) developed a plan to use silver or similar catalysts to directly oxidize propylene to propylene oxide. Catalysts accelerate the transformation of one chemical to a useful new chemical. Dow's proposed production method would be faster, cheaper, and more environmentally friendly. Because direct oxidation is hard to control, the company was unable to raise sufficient internal funds for comprehensive research and development of this high-risk process. The oxidation must produce only PO in order to succeed. Dow applied to the Advanced Technology Program (ATP) for support, and ATP awarded funding for three years as part of a focused program, "Catalysis & Biocatalysis Technologies." Researchers from the University of Michigan, Washington University, and Texas A&M University collaborated with Dow to support this project, which began in 1995.

In addition, as a member of the beamline user facility located at the National Synchrotron Light Source (NSLS) of the Brookhaven National Laboratory, Dow was able to do leading edge materials characterization as part of the ATP project to improve their understanding of how to control oxidative catalysis. The use of this facility to advance in situ catalyst characterization using soft X-ray detection technology [Near Edge X-ray Absorption Fine Structure (NEXAFS)] led to more industrial users exploring the potential of this in situ tool for catalyst characterization long after the ATP award.

Dow project researchers developed a new PO process that reacted propylene with oxygen in the presence of hydrogen. However, due to volatility in the price of hydrogen, the direct-oxidation process developed by Dow researchers is not yet cost-competitive. They did succeed in developing X-ray methods to observe chemical reaction intermediates with silver, gold, and other catalysts. They also developed a new family of oxidation catalysts. This work contributes to a better understanding of oxidative catalysis, enabling a new methodology for testing catalysts. They received six patents for their new methods and catalysts, and they disseminated their knowledge through numerous publications and presentations. Dow and other companies continued to use similar partial oxidation techniques at the NSLS facility to characterize the structure versus function relationships of new catalysts, and to more efficiently produce other industrially important chemicals for use in polymers, metal-polymer interfaces, and lubricants. As of 2004, the research on the new PO process was ongoing at Dow. The global PO market volume grew to 5.1 million metric tons in 2003 (\$238 billion) and is expected to reach 6.3 million metric tons by 2007 (\$294 billion).

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

* * *

Research and data for Status Report 95-05-0002 were collected during April – May 2004.

Propylene Oxide Is a Versatile and Practical Chemical

Propylene, a simple substance with three carbon atoms, is a flammable gas obtained by cracking petroleum molecules. One of the products formed when propylene is gently reacted with oxygen is propylene oxide (PO), a volatile, colorless gas with an ether-like odor. PO has many applications: 60 percent of the manufactured PO is used to make polyurethane foams (for upholstery), coatings, sealants, and adhesives, primarily in the automotive and housing industries; 20 percent of the PO is used to make propylene glycol, a component in fiberglass-reinforced plastics, foods, cosmetics, pharmaceuticals, airplane de-icing compounds, and hydraulic fluids; and the balance is used to produce lubricants, surfactants (detergents), and flame retardants.

Global consumption of PO had reached 1.5 million metric tons annually in the mid 1990s. Historically, consumption had been steadily growing at 6.2 percent annually, which meant that manufacturers had to increase production capacity or look for ways to produce PO more efficiently to meet demand. In a 1.5-million-metric-ton industry, a 6.2 percent annual growth rate meant increasing annual PO production by nearly 100,000 metric tons.

Current Production Is Inefficient and Energy-Intensive

In 1995, traditional processes for making PO were energy-intensive and inefficient. The processes used many pieces of equipment, involved multiple steps, and created large quantities of by-products and waste, which increased the cost of production as well as the impact on the environment. The primary production method used in the United States, called the chlorohydrin process, involved combining propylene and chlorine in a chemical reaction with water to produce an intermediate product called propylene chlorohydrin, which was then processed with caustic (sodium hydroxide) or with lime (calcium hydroxide). The output from this process includes several products: a dilute brine (sodium chloride in water) or calcium chloride, PO and propylene glycol. The resulting PO requires distillation for further purification before it can be used. Because the wastewater dilute brine produced

is about 40 times the volume of the PO, using the chlorohydrin process results in disposal problems.

The hydroperoxide process is another costly method for producing PO. This process uses ethylbenzene with oxygen to produce an organic-hydroperoxide. The hydroperoxide is used to oxidize propylene to PO, which is then purified. This process results in a large quantity of by-product, phenylmethylcarbinol, which is dehydrated to styrene, another useful chemical. Although this process has a high conversion rate (approximately 90 percent), the ratio of styrene to PO produced is approximately 2:1 (that is, 640,000 metric tons of styrene result from 285,000 metric tons of PO produced annually). Thus, the hydroperoxide manufacturing process depends on a strong market demand for both PO and styrene.

Researchers at various companies had succeeded in directly oxidizing a petrochemical similar to propylene, called ethylene. Beginning in 1937, researchers showed that ethylene could be directly oxidized to ethylene oxide (EO), a chemical similar to PO, using silver catalysts in a process that reduced raw material consumption and energy waste. Researchers had steadily improved EO production efficiency by using additives to the silver catalysts. This success stimulated the search for an analogous direct oxidation process for PO. However, because propylene is more combustible than ethylene, the catalysts used in EO production did not work for PO. Researchers had made attempts in the 1970s and 1980s, but these failed due to the lack of conversion efficiency and low selectivity to PO.

Direct Oxidation Would Cost Less and Minimize By-Products

Researchers at The Dow Chemical Company (Dow) sought methods to directly oxidize propylene to PO to minimize the numerous secondary steps and by-products of the traditional methods. Their initial research indicated that new catalyst systems that promoted silver catalysts could selectively oxidize propylene into PO. Their continued research indicated that catalysts consisting of silver and/or similar elements (for example, gold, titanium, or silica) as catalysts could use hydrogen with oxygen to oxidize propylene directly to PO. Catalysts are used to accelerate the transformation of one chemical to a

technologically useful material, for example, in transforming a gas such as ethylene into a plastic such as polyethylene. In 1995, advances in techniques for preparing and activating catalysts led Dow to request funding from ATP in order to develop these processes, which would require less energy and would result in nontoxic carbon dioxide (CO₂) and water (H₂O) as the by-products. The company was unable to raise sufficient internal funds due to the cross-disciplinary nature of the work. Outside academic specialists would assist Dow with specific project tasks. For example, Washington University researchers would conduct analytical studies of catalysts using fast reaction probing of the catalyst as well as using spectroscopy (measuring properties of molecules by probing them with different wavelengths of light). At the National Synchrotron Light Source (NSLS) user facility at Brookhaven National Laboratory in Upton, NY, Dow researchers would analyze reactions using innovative high-resolution synchrotron X-ray methods (using high-frequency electric fields and low-frequency magnetic fields). Researchers at the University of Michigan and Texas A&M University would conduct surface studies of the reactants and the catalysts.

ATP awarded funding for a three-year project, which began in 1995, as part of a focused program, "Catalysis & Biocatalysis Technologies." The ATP support allowed Dow to bring together researchers from the five organizations in a collaborative effort to study the PO reaction over various catalysts. Because direct oxidation is difficult to control, the project was considered very high-risk. Researchers needed to develop a catalyst with a long lifetime and avoid sintering the catalyst (particles sticking together forming larger particles). The oxidation must be selective, producing only PO when the propylene reacts, not other oxidation products such as propanal or acetone, and not other hydrogenation products such as propane and water. The team would study catalysts that included combinations of silver, magnesium, titanium, and gold. To compete with existing chlorohydrin-produced PO, a single-pass conversion rate of at least five percent was necessary (conversion rate is the amount of PO produced divided by the amount of propylene used). The challenge was to obtain acceptable conversion levels that produce only PO and leave the remaining propylene unreacted, so that it can be "cleanly oxidized" on the next pass.

In order to develop a one-step PO process using catalysis, Dow researchers had two primary objectives:

- Understand the ways in which oxidation catalysts work in order to gain higher production efficiency
- Synthesize the catalyst that meets performance goals

Steps to meet these objectives included the following: (1) identify and synthesize an appropriate catalyst; (2) provide a reaction environment that controls reaction heat and allows rapid diffusion of reactants; (3) achieve long catalyst lifetimes; (4) scale up catalyst production; and (5) engineer the direct oxidation process.

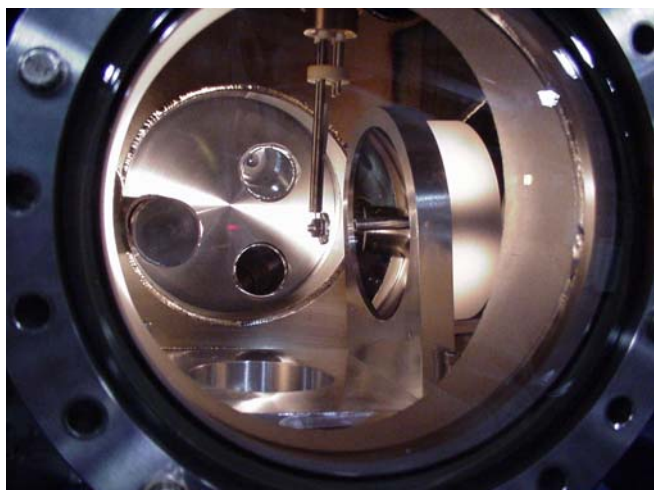
ATP Project Looks inside Chemical Reactions

Selecting the best catalysts was traditionally done by measuring the chemicals produced at the end of a trial. Adjustments and refinements to these trials are based on educated hypotheses. However, the ATP-funded researchers developed a new method to reveal the complex transformations that occur during a chemical reaction (virtually witnessing the reaction happen at various stages of the reaction). Understanding catalysis at a molecular level is vital in order to modify and develop more efficient catalysts.

The challenge was to obtain acceptable conversion levels that produce only propylene oxide and leave the remaining propylene unreacted, so that it can be "cleanly oxidized" on the next pass.

In order to observe chemical reaction intermediates, Dow researchers at the NSLS user facility placed the catalyst (silver, magnesium, gold, titanium, or a combination) in powder-like form in the vacuum-sealed chamber of a soft X-ray detection instrument (Near Edge X-ray Absorption Fine Structure [NEXAFS], see soft X-ray instrument photo). They then sealed the chamber, established a vacuum on the sample, and directed the various reactant gases (oxygen and propylene gas) into the chamber at the catalyst. The catalyst opened the double bond of the propylene molecules and allowed them to oxidize, creating PO. The soft X-ray instrument had never been used in this

way before. Intense X-rays were directed at the chamber that contained the reactants and the catalyst. By tuning the wavelength or “color” of the X-rays, the scientists could select low-energy X-rays that probed the reaction intermediates in every step of the reaction. Thus, they could “follow” the reaction’s progress, in real time, by observing the changes in the reaction intermediate’s “spectral fingerprints” on a computer screen.



The Dow/Brookhaven soft X-ray detection instrument consists of the sample stud (hanging, center, 1-cm diameter), soft X-ray detector (left, tube, 2-cm diameter), and the focusing multilayer mirror (MLM) (right, spherical mirror, 10-cm diameter). Researchers place a small amount of the powder-like catalyst on the sample stud. They close the door, establish a vacuum, and allow oxygen and propylene gas to enter the chamber. The catalyst stimulates a chemical reaction between the propylene (composed of carbon and hydrogen) and oxygen to form PO and nontoxic by-products such as H₂O and CO₂. The X-ray detector takes measurements, and the MLM screens out background spectral images.

The project scientists developed a mirror, called a normal incidence focusing multilayer mirror (MLM) to focus on particular reaction intermediates. The MLM reduced background spectral images and allowed researchers to understand how the reactants and catalysts work together under real conditions at the molecular level. Image resolution was smaller than 300 nanometers (billionths of a meter).

Dow researchers could alter PO output by making changes in temperature, proportions, and additives. They intended to use these measurement results to develop more efficient catalysts for propylene oxidation.

Catalysis Research Makes Progress

During the project, researchers discovered that adding hydrogen (H₂) as an additive with the reactant gases increased the amount of propylene that converts to PO.

Furthermore, by the end of the ATP-funded project, Dow researchers were able to control the size and dispersion of catalysts and to identify effective additives, resulting in an even higher yield of propylene to PO. The main by-products were nontoxic CO₂ and H₂O, which would eliminate the industry’s concerns about waste disposal. Researchers gained an understanding of the catalysts and developed new methods for studying the synthesis and optimization of catalysts. They worked with catalyst manufacturers to scale up production of identified catalyst compositions.

The multilayer mirror reduced background spectral images and allowed researchers to understand how the reactants and catalysts work together under real conditions at the molecular level.

Dow received six patents for their innovations, with three more patents pending. Researchers on the ATP-funded project disseminated their results widely in academic publications and through presentations. However, unexpected volatility in the price of hydrogen has been a barrier to commercialization. Some of the hydrogen additive is wasted, because instead of converting propylene to PO, it converts some propylene to propane and some of the oxygen to water. Also, researchers are still trying to develop a cost-effective catalyst with a long lifetime. Dow continues to fund this PO direct-oxidation research to achieve higher conversion rates and lifetimes. Researchers believe they may complete a process sometime between 2006 and 2014. Single-step direct oxidation is the ultimate objective of PO, so all the global competitors, such as Sumitomo, Lyondell, Degussa/Krupp Uhde, and BASF, are working to develop this technology.

Dow researchers are also using direct-oxidation techniques from this project to design new catalysts to pursue other industrially important chemicals (such as butylene oxide). They hope to develop new production methods for polymers, metal-polymer interfaces, solvents, and lubricants, as well as stronger coatings and adhesives.

Manufacturers Keep Their Production Options Open

PO production is an important business for The Dow Chemical Company, so the company invests in

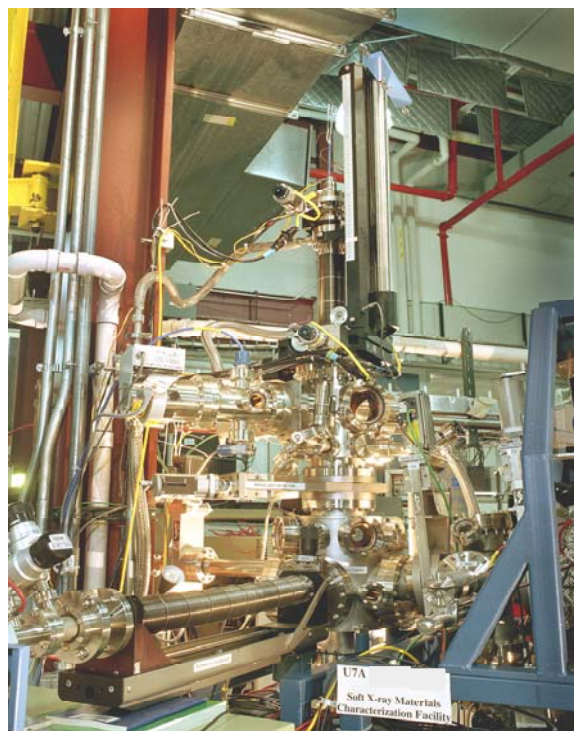
multiple manufacturing methods simultaneously. For example, the company acquired an exclusive license from a Russian firm, JSC Nizhnekamskneftekhim, to use an organic hydroperoxide-based PO manufacturing process, as reported in *Chemical Engineering*. Dow and BASF also formalized an agreement to develop and commercialize the hydrogen peroxide process for PO in 2002. The main drawback of this process is its large consumption of hydrogen peroxide, which would entail incorporating a new adjacent hydrogen peroxide plant. Dow planned to use this method in a plant on the U.S. Gulf Coast in 2004; however, this project has been delayed because Dow lowered its PO demand forecast in response to the economic downturn in North America in 2001. In commodity markets, such as PO, manufacturers compete for fractions of pennies per pound of PO. Margins are small and slight cost variations can make production methods untenable.

The scientists could “follow” the reaction’s progress, in real time, on a computer screen.

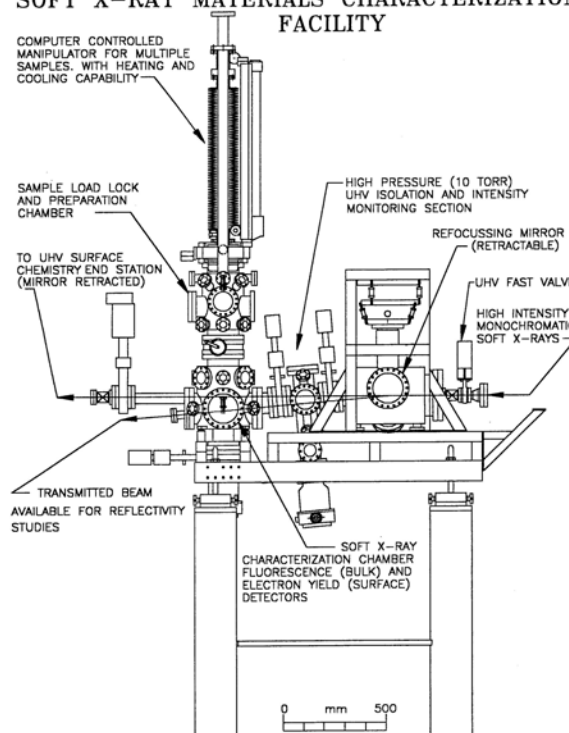
Dow and its competitors continue to pursue direct oxidation methods and alternative technologies. As of 2004, the chlorohydrin and hydroperoxide processes still dominate the global market. Direct oxidation of PO could be the easiest method, if the conversion rate can be raised high enough to achieve cost competitiveness. Dow believes that, depending on the site and availability of raw materials, any of these various technologies could become cost effective, so the company continues to explore all of the PO processes. Developing direct-oxidation PO methods remains a priority for Dow.

New In Situ Catalyst Characterization Technique Leads to Spillover Applications

Dow worked as a member of the beamline user facility located at U7A of the NSLS at the Brookhaven National Laboratory. They were able to characterize leading edge materials and improve their understanding of how to control oxidative catalysis. Collaborations at this facility advanced in situ catalyst characterization using soft X-ray detection technology (NEXAFS) and led to more industrial users exploring the potential of this in situ tool for catalyst characterization long after the ATP award.



SOFT X-RAY MATERIALS CHARACTERIZATION FACILITY



Photograph and diagram of the soft X-ray instruments. The sample is placed in a chamber at about midpoint in the tower at the center of the photograph. The soft X-ray beams enter from the right.

From 1998-2000, following the ATP project, several companies collaborated with NIST researchers to perform additional non-proprietary catalysis characterization research at the NSLS at Brookhaven National Laboratory. The collaborating companies included Texaco, Intevac, UOP, and Rohm & Haas. They continued to enhance the NEXAFS soft X-ray detection technology, automating it and making it more user friendly, and used the multilayer mirror developed in this ATP-funded project. Some examples of discoveries include the following: the ability to manipulate particles on a nanoscale (creating a patterned assembly of gold nanoparticles for specialized filters, sensors, high-efficiency solar cells, single-molecule detectors, and in the future potentially to be used for high-density information storage devices); adjust surface chemistry of biomaterials to optimize artificial polymer joints to last longer; and the electronic structure of superconductors in order to improve performance. Soft X-ray spectroscopy has also provided critical insight on performance-limiting chemistry challenges for next-generation nanometer-scale structures in integrated circuits and showed that lubricant additives were chemically interacting with the protective overcoat needed in future high-capacity computer hard disks.

The soft X-ray work broadly benefits catalysis technology by improving scientists' understanding of what makes a good catalyst under "industrial" operating conditions and has led to numerous publications. In 2004, a key NIST researcher doing research at the Brookhaven NSLS, Dr. Daniel Fischer, was awarded a Department of Commerce Gold Medal for Scientific/Engineering Achievement for his work on "a unique national measurement facility for soft X-ray absorption spectroscopy enabling breakthrough materials advances."

Conclusion

During this ATP-funded research, The Dow Chemical Company and its subcontractors made significant progress in analyzing and understanding direct-oxidation reactions to produce propylene oxide (PO), as well as developing methodologies that will affect using direct oxidation catalysis in new ways for other products. If successful, improved PO production will positively affect a huge volume and diversity of

products, such as polyurethane-foams (inside upholstery), adhesives, coatings, sealants, pharmaceuticals, cosmetics, packaging, antifreeze, soaps, solvents, and lubricants. Despite Dow's substantial additional research, the direct oxidation process for PO is not yet economically feasible due to the fluctuating cost of hydrogen. The company believes that it might successfully complete a direct-oxidation process for PO in the future (2006–2014) that will be faster and less expensive and will result in nontoxic by-products (carbon dioxide and water). Although still technically risky, if successful, direct-oxidation processes could be applied to other petrochemical gases such as butene. New conversion methods could lead to producing PO and other petrochemical derivatives with little waste and lower energy consumption.

PROJECT HIGHLIGHTS

The Dow Chemical Company

Project Title: Using Catalysts to Directly Oxidize Propylene to Propylene Oxide (Breakthrough Process for Direct Oxidation of Propylene to Propylene Oxide)

Project: To develop a direct, economical, single-step oxidation process that incorporates a silver-based catalyst to convert propylene to propylene oxide (PO).

Duration: 10/1/1995–9/30/1998

ATP Number: 95-05-0002

Funding**(in thousands):

ATP Final Cost	\$1,958	71%
Participant Final Cost	<u>802</u>	29%
Total	\$2,760	

Accomplishments: With ATP funding, The Dow Chemical Company and its subcontractors improved PO oxidation efficiency and stimulated substantial ongoing research. Researchers gained a better understanding of catalysts and made advances in their ability to observe chemical reactions in “real-time” using a soft X-ray detection instrument. Project researchers also developed a new tool that they added to the soft X-ray instrument called a focusing multilayer mirror, which filters out background spectral images.

Following the ATP-funded project, researchers from several companies and NIST continued to collaborate on non-proprietary catalyst research and development using the tools and methods of this ATP-funded project. Collaborating companies included Dow, Texaco, Intevac, UOP, and Rohm & Hass, from 1998-2000. The ongoing development resulted in numerous publications and one technical award:

- Gold Medal for Scientific/Engineering Achievement for Dr. Daniel Fischer’s work on “a unique national measurement facility for soft X-ray absorption spectroscopy enabling breakthrough materials advances.” Awarded by the Department of Commerce, NIST/Brookhaven National Laboratory, 2004.

Dow has received six patents for oxidation technologies developed during this ATP-funded project. Three additional patent applications are pending.

- “Process for the direct oxidation of olefins to olefin oxides” (No. 5,965,754: filed December 11, 1998; granted October 12, 1999)
- “Process for the direct oxidation of olefins to olefin oxides” (No. 6,646,142: filed December 7, 1999; granted November 11, 2003)
- “Process for the direct oxidation of olefins to olefin oxides” (No. 6,323,351: filed December 9, 1999; granted November 27, 2001)
- “Process for the direct oxidation of olefins to olefin oxides” (No. 6,362,349: filed October 4, 2000; granted March 26, 2002)
- “Process for the direct oxidation of olefins to olefin oxides” (No. 6,562,986: filed February 19, 2002; granted May 13, 2003)
- “Process for the direct oxidation of olefins to olefin oxides” (No. 6,670,491: filed January 14, 2003; granted December 30, 2003)

Commercialization Status: Direct oxidation of propylene to PO is still Dow’s ultimate goal. Dow researchers expect that they might complete a process sometime between 2006 and 2014. A successful process will reduce energy consumption, cost, and waste in the manufacturing of many types of plastics, lubricants, coatings, surfactants (detergents), and composite materials.

Outlook: The outlook for the direct oxidation of propylene is good, but still technically risky. If Dow can overcome the technical barriers and finalize the direct-oxidation process before its competitors are able to bring a new process to market, the company will be in a strong position to compete in the global PO market.

Composite Performance Score: * * *

Focused Program: Catalysis & Biocatalysis Technologies, 1995

** As of December 9, 1997, large single applicant firms are required to pay 60% of all ATP project costs. Prior to this date, single applicant firms, regardless of size, were required to pay indirect costs.

PROJECT HIGHLIGHTS

The Dow Chemical Company

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Subcontractors:

- Texas A&M University
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College Station, TX
- Washington University
Chemical Engineering Department
St. Louis, MO
- University of Michiga
College of Engineering
Ann Arbor, MI
- Brookhaven National Laboratory (National
Synchrotron Light Source [NSLS] user facility)
Upton, NY

Publications: Researchers disseminated their knowledge widely through 37 academic publications.

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The Dow Chemical Company

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 - "The Application of Solid State Detectors in Ultra Soft X-ray Absorption Spectroscopy." International Radiation Detectors, Torrance, CA, January 15, 1997.
 - "Soft X-Ray Surface Chemistry Characterization: Tribochemical and Thermochemical Reactions of Stearic Acid on Metal Surfaces." National Storage and Information Consortium Meeting, San Diego, CA, January 16, 1997.
 - "Chemistry and orientation of lubricants on hard disk magnetic media substrates: characterization by NEXAFS." National Storage and Information Consortium Meeting, Berkeley, CA, January 23, 1998.
 - "Dow/NIST materials characterization facility: catalysis applications." Advanced Technology Program, Catalysis and Separations Technologies Program Workshop, February 18, 1998.
 - "XANES Research at the Dow/NIST Soft X-ray Materials Characterization Facility" NSLS Users Annual Meeting, May 20, 1998.
 - "Synchrotron Based Low Z Materials Analysis." American Vacuum Society International Symposium, Baltimore, MD, November 2, 1998.
 - "Effects of Wear motion on Molecular Orientation in Ultrahigh Molecular Weight Polyethylene." NIST Orthopedic CRDA Consortium, December 16, 1998.
 - "Soft X-ray Spectroscopy Applications at Beamline U7A." The Dow Chemical Company, Midland, MI, January 12, 1999.
 - "Carbon X-ray Absorption Spectroscopy with Focusing Multilayer Mirror Detection." Osmic Inc., Troy, MI, January 13, 1999.
 - "Advanced Molecular Aspects of Polyethylene Wear: Soft X-ray Analysis." Howmedica Inc., Rutherford, NJ, April 6, 1999.
 - "Focusing Wavelength Dispersive Multilayer Detection System for Soft X-ray Absorption Spectroscopy." NSLS Users Meeting, May 24, 1999.
- Presentations:** The project also produced the following 13 presentations.
- "Soft X-ray Absorption Spectroscopy for In Situ Catalyst Characterization." Plenary Speaker, 14th International Congress on Catalysis, Baltimore, MD, June 29, 1996.
 - "Soft X-Ray Materials Characterization Facility: Catalysis Applications." Catalysis, Biocatalysis, and Separations Technology Advanced Technology Program Review, Boulder, CO, October 22, 1996.

Wyman-Gordon Company

Incremental Forging Process for Gas Turbine Applications

In the early 1990s, U.S. producers of large superalloy forgings (or disks) for the power-generation industry competed in a price-driven commodities market dominated by two foreign suppliers. At the time, these two suppliers jointly owned and used the largest commercial forging press. In order to compete with these foreign companies, Wyman-Gordon Company, one of the United States' largest metal forgers, proposed an innovative forging process and tooling design. The company sought to use its smaller press to manufacture extremely large, cost-effective, near-net-shape forgings for advanced gas turbine applications, specifically land-based gas turbines (LBGTs). They would use two nickel-based superalloys: alloy 706, which was used in current engines, and alloy 718, to be used in future engines. Building a larger forging press was not an alternative due to the exorbitant cost. Therefore, Wyman-Gordon proposed an incremental forging process that had not yet been applied to LBGTs in which only a portion of the workpiece is forged during each forging stroke.

In 1995, the company was awarded cost-shared funding for an 18-month project from the Advanced Technology Program's (ATP) focused program competition in "Materials Processing for Heavy Manufacturing." Upon completion of the ATP project, Wyman-Gordon had successfully developed an incremental forging process using alloy 706 that achieved a 20-percent reduction in necessary materials and in overall cost. In contrast, the company was unable to commercialize LBGT forgings made with alloy 718 due to technical difficulties.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

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Research and data for Status Report 95-07-0026 were collected during fall 2001.

Foreign Competition Limits Domestic Forgers' Market Share

Forging, a process for shaping metal parts, is used to produce large quantities of identical parts, such as disks for land-based gas turbine (LBGT) engines. In 1995, U.S. producers of large superalloy forgings (disks made with alloys possessing superior mechanical properties at high temperatures) for the power-generation industry were losing market share to foreign competitors, primarily to two French companies, Aubert & Duval and Fortech. Both companies were using the same French 72,000-tonnage press. Their press had a 44-percent higher tonnage capability than the two largest presses in the United States at 50,000 tons, which were owned by Wyman-Gordon and Alcoa. The two largest presses in the world, not commercially available at the time, were in Russia; each was an 82,000-tonnage press.

France's superior forging press limited the United States' ability to compete in the global market. In fact, in 1995, annual U.S. imports of superalloy forgings totaled \$50 million. This amount, which represented a significant loss of potential sales for domestic suppliers, was expected to increase unless a dramatic change in domestic production was made.

The market for LBGTs (i.e., non-aviation gas turbines) was approximately \$75 million in 1995. Although Wyman-Gordon's annual sales in this market were \$5 million, the company believed it could develop a new technology that would increase its annual revenue stream to \$50 million within four years. This increased market share would be won at the expense of foreign suppliers of forgings. At the time, General Electric (GE) was one of the largest developers of LBGTs and was importing vast amounts of forgings from France. However, GE was seeking a domestic partner that

could provide it with forgings similar to those supplied from France. The domestic products needed to be equal to or superior in quality and be competitive in price. GE expressed interest in developing a relationship with Wyman-Gordon to supply the desired forgings. With GE's technical support, Wyman-Gordon proceeded to investigate developing a new forging technology.

Novel Incremental Forging Process Uses Smaller Press

In the early 1990s, Wyman-Gordon determined that the most cost-effective way to compete in the market was to design a process using smaller tonnage presses that could replicate the force and power of a larger tonnage press. The company planned to accomplish this by adding a rotating tooling mechanism to its existing press and by using an innovative tooling approach that would involve incremental forging, in which only a portion of the workpiece is forged during each forging stroke. This new approach would limit the amount of tonnage required to achieve a given level of deformation, enabling Wyman-Gordon's 50,000-tonnage forging press to function as a significantly larger press, comparable to the French 72,000-tonnage press. The smaller press would be able to shape forgings to a more near-net shape (i.e., close to the final shape), reducing the amount of raw material required and limiting the machining needed to achieve the geometry of a part.

U.S. producers of large superalloy forgings power-generation industry were losing market share to foreign competitors.

Wyman-Gordon had the expertise to develop such a process on its own, but its limited funds would have prolonged the development cycle. Moreover, the alternative to the new process, building a press that was larger than its competitors', was cost prohibitive. A large press was estimated to cost \$1 million per thousand tons; therefore, a 100,000-tonnage press would cost about \$100 million. Furthermore, it was crucial that Wyman-Gordon introduce its new approach within two years because international competition was threatening U.S. market share.

Wyman-Gordon Pursues External Funding Assistance

Wyman-Gordon understood that additional funding was essential to the timely development of the incremental forging process; thus, it initiated a search for funding assistance. In 1995, the company applied to ATP's focused program competition in "Materials Processing for Heavy Manufacturing." This program was set up to promote economic growth by supporting sustained, high-risk research and development to accelerate the introduction of advanced materials technologies into heavy manufacturing.

Wyman-Gordon determined that the most cost-effective way to compete in the market was to design a process using smaller tonnage presses that could replicate the force and power of a larger tonnage press.

Emphasis was placed on the introduction of both 1) advanced materials technologies into machinery and equipment products and 2) advanced machinery and equipment into the production and processing of the materials for these products. Wyman-Gordon believed its technology would meet these criteria, and the benefits resulting from this technology would impact many domestic original equipment manufacturers, as well as Wyman-Gordon. ATP awarded Wyman-Gordon \$1 million in funding assistance for an 18-month project.

Process to Reduce Raw Materials Consumption

There were three technical goals for this ATP-funded project:

- Design an incremental forging process.
- Engineer the mechanisms and tooling necessary to execute the process.
- Apply the process to the production of LBGT forgings of the nickel-based superalloy used in current engines (alloy 706), as well as the superalloy to be used in future engines (alloy 718), which would have a higher temperature capability.

Key parameters for investigation during this project included studying thermomechanical processing and near-net-shape geometry. At the time, there was limited knowledge of how incremental forging and heat treatment would affect the microstructures and mechanical properties of superalloy disks. In addition, Wyman-Gordon sought to develop a process that would maximize near-net-shape geometry. Increasing near-net-shape geometry in the forging is the most effective way to reduce the required amount of raw materials, which is the largest cost component.

Improved Computer Process Modeling Enables Development

Three-dimensional (3-D) computer process modeling of the incremental forging, which eliminated costly trial and error, was instrumental in the successful development of the forging process and of a new forging press top die (a forging tool for imparting a desired shape or form). Previous forging press designs had utilized 2-D process modeling for more than 10 years, but now, with faster and more affordable computers and software, 3-D process modeling was a valuable production tool.

Desired Results Are Achieved

Originally, Wyman-Gordon proposed and was awarded funding for an 18-month project; however, due to complications at its Houston facility and the company's ambitious expectations, the project was extended at no cost to ATP for an additional 18 months. Upon completion of the project in 1998, Wyman-Gordon had achieved its proposed goals. The company had developed and demonstrated the feasibility of a novel forging process that would enable existing presses to form near-net-shape parts. It had taken Wyman-Gordon many steps to achieve the milestones needed to achieve the desired results with this incremental forging process.

First, during the ATP-funded project, 3-D computer process modeling was used to evaluate potential designs for the incremental forging process, as well as several additional process variables. The 3-D modeling was instrumental in reducing the time and cost associated with the project. Second, microstructure and mechanical property analysis was used to examine the

effects that the incremental forging process would have on the superalloy forgings. This subscale work enhanced Wyman-Gordon's understanding of the metallurgical behavior of the two superalloys, and this knowledge is now also being used to improve forgings for other markets, such as aerospace. Third, the company designed and constructed the incremental tooling mechanism. This mechanism consistently transmits 50,000 tons of force to the forging and also allows rapid rotation and repositioning of the forging dies between each forging stroke. Fourth, Wyman-Gordon conducted a successful full-scale trial of incremental forging on stainless steel. This led to the fifth milestone of fabricating and evaluating an alloy 706 forging. The results from this forging were quite favorable. In fact, the microstructures and mechanical properties measured in the forging were equivalent to those achieved during conventional forging using much larger presses.

Wyman-Gordon understood that additional funding was essential to the timely development of the incremental forging process.

The final milestone was the fabrication of an alloy 718 forging using the incremental forging process. The company experienced some difficulty in achieving desired mechanical properties at certain temperatures for the alloy 718 forging, which was to be used in future engines operating at higher temperatures. However, when the temperature was lowered, this forging also achieved favorable results. With positive results for both alloy 706 and 718 forgings, Wyman-Gordon was poised to initiate its new method of forging for LBGT engines.

Incremental Forging Increases Wyman-Gordon's Market Share

The incremental forging process developed as a result of this project has allowed Wyman-Gordon to compete for new business. In 1995, at the start of the ATP-funded project, Wyman-Gordon held approximately 13 percent of the \$50 million market for alloy 706 forgings for LBGT applications. The reduction in input of raw materials resulting from the incremental forging process made Wyman-Gordon competitive. In addition, through the use of its new technology, Wyman-Gordon has achieved a 20-percent price reduction in LBGT forgings.

Market and Outlook for LBGT Engines Change

At the outset of this project in 1995, the market for LBGT engines was expected to grow substantially, with the majority of the growth anticipated in Asia. However, as the economy in Asia slowed, so did the demand for LBGT engines and forgings for these engines. Accordingly, Wyman-Gordon shifted its focus to domestic markets. The company expected its short-term growth in sales to result from GE's power-generation engines, which are designed to serve many markets including the aircraft industry.

Wyman-Gordon conducted a successful full-scale trial of incremental forging on stainless steel.

Wyman-Gordon is continuing to advance its successful incremental forging technology. Since the completion of the ATP-funded project, the company has improved the die materials involved in the process. Although the demand for its alloy 706 forgings has continued to increase, commercialization of alloy 718 forgings has not occurred. Wyman-Gordon encountered technical difficulties associated with alloy 718 that have prevented its commercialization.

Conclusion

In 1995, with funding assistance from ATP, Wyman-Gordon developed an incremental forging process to produce forgings for advanced gas turbine applications, specifically land-based gas turbines (LBGTs). These forgings were made with two nickel-based superalloys: alloy 706 for current engines and alloy 718 for future engines. As a result of this ATP-funded project, Wyman-Gordon can use its 50,000-tonnage press to produce large near-net-shape, nickel-based superalloy components made with alloy 706. Previously, these components could only be produced using presses with much higher tonnage capabilities, such as the 72,000-tonnage press in France.

Wyman-Gordon has increased its market share, which has enabled an increase in domestic market share across the forging value chain. In addition, Wyman-Gordon has reduced its forging costs and input materials by 20 percent. Due to current global market conditions, however, the market is flat and is expected to remain so until economic conditions improve. Despite its success with using alloy 706, the company experienced technical difficulties using alloy 718 and, thus, was unable to commercialize LBGT forgings made with this superalloy.

PROJECT HIGHLIGHTS

Wyman-Gordon Company

Project Title: Incremental Forging Process for Gas Turbine Applications (Cost-Effective Near-Net Shape Superalloy Forgings for Power-Generation Gas Turbines)

Project: To design and develop an innovative forging process and tooling technology that enables U.S. metal forgings to achieve cost-effective production of larger, near-net-shape nickel-based superalloy forgings for advanced gas turbine applications using existing presses.

Duration: 9/1/1995-5/31/1998

ATP Number: 95-07-0026

Funding (in thousands):

ATP Final Cost	\$687	65%
Participant Final Cost	<u>375</u>	35%
Total	\$1,062	

Accomplishments: Wyman-Gordon successfully developed an incremental forging process to produce near-net-shape forgings for industrial gas turbines using a lower tonnage press than was previously possible. Moreover, the company reduced input billet weight by 20 percent for alloy 706 forgings. The company also decreased forging costs by 20 percent.

Commercialization Status: Wyman-Gordon has incorporated the incremental forging process into its business operations.

Outlook: Wyman-Gordon believes that the incremental forging process is key to increasing its market share in the large industrial gas turbine forgings market. The company has not identified a new market for the incremental forging process. It believes that the process in its current state is only suitable for large industrial gas turbine applications.

Composite Performance Score: *

Focused Program: Materials Processing for Heavy Manufacturing, 1995

Company:

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Research and data for Status Report 95-07-0026 were collected during fall 2001.

APPENDIX A

Development of New Knowledge and Early Commercial Products and Processes, 3rd 50 of Status Reports

Table A-1: Advanced Materials and Chemicals; Table A-2: Biotechnology; Table A-3: Electronics, Computer Hardware, or Communications; Table A-4: Information Technology; Table A-5: Manufacturing

Table A-1. Advanced Materials and Chemicals

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
ABB Lummus Global, Inc. (formerly ABB Lummus Crest)	95-05-0034	Developed a new, environmentally superior process to manufacture alkylate, an ideal unleaded gasoline additive, using solid-acid catalysts	As of 2005, the joint venture partners were seeking commercial opportunities to build new solid-acid alkylation plants
Advanced Refractory Tech	95-01-0131	Developed a diamond-like nanocomposite (DLN) coating technology. The company established improved manufacturing techniques for DLN films and developed several applications, such as electrosurgical blades and flat panel displays	A number of products with DLN coatings are currently being sold. These include components that are used in manufacturing CDs, DVDs, polyethylene terephthalate juice bottles, and metal cans and components used in semiconductor cluster tools
Air Products and Chemicals, Inc.	93-01-0041	Developed ceramic-steel seals and processes to remove contaminants from oxygen	The company is continuing its research and development (R&D) into their prototype air-separation unit for producing high-purity oxygen so that future commercialization may be possible. However, the company does not intend to pursue commercialization initiatives until a 30-percent decrease in production cost is achieved
Automotive Composites Consortium (a Partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)	94-02-0027	Developed a composites-manufacturing process called Structural Reaction Injection Molding (SRIM) for producing large automobile structural parts, such as the box of pickup trucks	Commercialized the access door and tail cone for the Air Force C-17 cargo plane by Boeing, firefighter helmet shells by Lion Apparel, the inner tailgate sections for the GM Cadillac Escalade EXT hybrid SUV beginning in 2001, the load floor sections for the "Stow 'n Go" system to fold down second-and third-row seats in the Chrysler

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
			Town & Country LX and Dodge Grand Caravan SCT beginning in 2005, the midgate (a door that folds down to extend cargo space) for the GM Chevrolet Avalanche beginning in 2001, the motor covers for marine applications by SeaRay (Marine division of Brunswick Corp), and the pickup truck box and tailgate assembly for the 2001 to 2004 GM Chevrolet Silverado. Boeing's 787 "Dreamliner" uses SRIM composites for structural parts, increasing fuel efficiency by 3 percent. Overall fuel savings is 20 percent compared with the 747. First commercial flight is scheduled for 2008
Bosch (formerly Allied Signal)	95-07-0020	Developed a synergy between design and casting processes that resulted in the following accomplishments: elimination of porosity problem (zero rejects for porosity); reduction from one large and three small defects per part to two small defects per part; acceleration of research by two years ahead of where it otherwise would have been through parallel research efforts; and reduction of defects in a specific type of valve body design by up to 85 percent	The technical challenges of this project were too numerous and difficult to overcome. As a result, AlliedSignal created no new products for brakes using the technology developed under the ATP-funded project. The Top Die Casting Company produced some components using the new processes, such as air brake valves and brackets. Stahl Specialty Company used one step of the aluminum manufacturing process to assist in aluminum filtration. That process had a small impact on several of the company's product lines
BP Amoco	93-01-0234	Developed a process using silver nitrate as a facilitating agent in high-efficiency contactors and had developed a promising new complexing agent that would potentially cost less than silver nitrate when used for facilitated transport	Although the process was technically sound, the company was experiencing costly operating problems. Amoco was unable to demonstrate the economic feasibility of using this new technology for olefin-paraffin separations and therefore did not commercialize the technology
Catalytica Energy Systems (formerly Catalytica, Inc.)	94-01-0190	Developed catalysts with enhanced activity and selectivity for use in the chemical and petroleum-refining industries	Developed a Multiple Stream Mixer/Reactor (MMR) which may prove to be a very valuable tool for the emerging nanotechnology sector, producing nanoparticles for many industries. The company expected to sell its first major

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
			production MMR system in 2005 or 2006
Crucible Materials Corporation, Crucible Companction Metals Division	94-01-0287	Developed alloys with high levels of nitrogen that demonstrated the potential to produce high-strength, corrosion-resistant stainless steel	Commercialized high-nitrogen alloys that could improve the performance of stainless steel (SS100)
GM Thermoplastic Engineering Design (Engineering Design with Thermoplastics)	92-01-0040	Developed models and generated data for "virtual design" in order to improve the design and development of thermoplastic automotive parts. The project team linked two commercial software tools, Moldflow (formerly C-MOLD) and ABAQUS, with new failure theories for plastics in order to integrate mold design with parts performance	Commercialized virtual design tools that have shortened development time and have improved the performance of thermoplastic parts, which has benefited many manufacturers (for example, Delphi's thermoplastic radiator tank and many other parts; GM's injection-molded plastic intake manifold and other engine components; GE Plastics' improved raw material, which is used in business equipment, optical media, and telecommunications devices). The project resulted in the International Organization for Standardization (ISO) issuing a new standard (ISO 94-5)
Honeywell (formerly Allied Signal)	93-01-0104	Developed powder injection molding used in the ceramic industry for chinaware, spark plugs, oxygen sensor components, and oxygen sensor insulators	Commercialized ceramic powder injection molding technology that is being used in chinaware, spark plugs, oxygen sensors, ball bearings, manufacturing components (for example, stamping punches and guide rollers), engine and machine components (for example, nozzles, seals, shafts, valves, and heating units), and bio ceramics (for example, artificial bones for human replacement surgery)
Honeywell (formerly Allied Signal)	95-07-0003	Developed "aqueous injection molding" (AIM) process improvements for ceramic splitter vanes	Commercialized ceramic splitter vanes in 1998. They had plans to commercialize other small, complex, high-volume parts like blades and nozzles

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
IBM T.J. Watson Research Center	93-01-0149	Developed a conducting polymer of acid-doped polyaniline (PANI) with thermal stability greater than 250 degrees C from 150 degrees C, increasing processability and solubility, and increasing conductivity by 2.5 orders of magnitude	Commercialized a water-soluble version of PANI that was licensed to Monsanto Chemical Corporation in 1997, and IBM is pursuing further licensing opportunities
PCC Structural	95-07-0011	Developed a casting technology that combines the superalloy processing capabilities of investment casting with the economic advantages of sand casting and achieves part sizes sufficient to produce exhaust frames for industrial gas turbine engines	PCC did not commercialize the new casting technology. They did develop prototypes of a new casting technology that will allow manufacturers to produce large structural superalloy components for industrial equipment industries, such as the Industrial Gas Turbine industry
Praxair, Inc.	94-01-0111	Developed new materials highly selective for oxygen, including IC-2, IA-1, IA-2, and IA-3, which have the potential of meeting all characteristics of a successful material with further development	The O ₂ -selective materials developed during this ATP-funded project have not been commercialized. However, as of 2003, Praxair has continued work on their development through a project with the Department of Energy with hopes to commercialize in the future
The Dow Chemical Company	95-05-0002	Developed a direct, economical, single-product oxidation process incorporating a silver-based catalyst for conversion of propylene to propylene oxide	Dow researchers expect that they might complete a process to develop a direct oxidation propylene sometime between 2006 and 2014. A successful process will reduce energy consumption, cost, and waste in the manufacturing of many types of plastics, lubricants, coatings, surfactants (detergents), and composite materials
Wyman-Gordon	95-07-0026	Developed an incremental forging process to produce near-net shape forgings for industrial gas turbines using a lower-tonnage press than was previously possible	Wyman-Gordon has incorporated the incremental forging process into its business operations

Table A-2. Biotechnology

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
Aphios Corporation	95-01-0263	Developed a knowledge base and technology platform to tap into the pharmaceutically, industrially, and environmentally valuable chemical diversity that remains unexplored in enormous numbers of marine microorganisms	An anti-plaque solution for toothpaste or mouthwash, which is being optimized through chemistry, is the nearest product to commercialization. Novel therapeutics for multiple-disease-resistant (MDR) bacteria, influenza, HIV/AIDS, cancer, and smallpox are also undergoing trials in preclinical drug discovery and development
Cengent Therapeutics Inc. (formerly Moldyn Inc.)	94-01-0137	Developed a software that adapts a technology developed in the aerospace industry to simulations of biological molecule and drug interactions, for the purpose of qualifying drug research candidates in a more timely and efficient manner than by using trial-and-error techniques	The MD simulation software was briefly commercialized through a license to Molecular Simulations Incorporated, but failed to gain sufficient sales and was discontinued. However, Moldyn's software was incorporated with Harvard's Chemistry at Harvard Macromolecular Mechanics (CHARMM) molecular modeling tool through a licensing agreement between Moldyn and Harvard University
Dow AgroSciences LLC (formerly Mycogen Corporation)	95-01-0148	The company made strides in genetic research and demonstrated for the first time that yeast is transformable. They demonstrated that squalene could be hyper-produced in oleaginous yeast; and they gained a broader understanding of the metabolic pathways for isoprene formation in yeast	No commercialization occurred because the oleaginous yeast fermentation project was ended due to technical barriers with enzyme manipulation
DuPont Qualicon (formerly DuPont FQMS Group)	94-05-0033	Developed a functioning automated, rapid DNA diagnostic prototype system that reduced analysis time from 3 hours to 30 minutes. The system can determine the presence or absence of specific microbial contamination as a means of quality control in the food industry. However, DNA pattern results from sample testing were somewhat inconsistent and needed further development	Additional steps were required in sample preparation that negated the time saved in analysis. DuPont Qualicon ended the research into this automated system in 1998, but the company did apply some of the automation knowledge gained in this project to its ongoing alternate food-borne pathogen-testing technologies

Genosensor Consortium (c/o Houston Advanced Research Center)	92-01-0044	Developed a technology for automated DNA sequence analysis	Provided sample analysis and database services for genotyping and gene expression research to organizations such as the Schering Plough Research Institute. In 1999, consortium member Sigma Genosys began to sell Panorama Gene Arrays, which profile gene expression in human cytokines, B. subtilis, and E. coli. In 2003, Sigma Genosys sold human cancer oligoarrays. In 2003, consortium member Beckman Coulter started to commercialize arrays
Incyte Corporation (formerly Combion, Inc.)	94-05-0019	Developed a method akin to ink-jet printing for synthesizing large arrays of specific DNA fragments suitable for medical diagnosis, microbial detection and DNA sequencing, and for creating supplies of detachable oligonucleotides for subsequent use	Microarray expertise and knowledge gained in this project formed the foundation for Incyte's highly successful bioinformatics business, which operated from 1999 to 2001 (selling subscriptions to databases of DNA information). Although Incyte put the specific chem-jet microarray manufacturing techniques developed in this project on hold from approximately 1998 to 2004, the company licensed the technology to Agilent in 2001. As of 2004, Agilent was about to commercialize the ATP-funded technology in conjunction with their numerous other patented chem-jet technologies
JDS Uniphase (formerly The Uniphase Corporation)	94-05-0004	Although the attempt to develop a compact, efficient, and cheaper source of blue light for fluorescence-based diagnostic instruments and techniques for physicians and biomedical researchers was unsuccessful, the project led to the development of two unanticipated products	Commercialized the Blue Laser Module, a stripped-down, inexpensive blue laser for tabletop applications within the biotechnology industry, that reached the market in 1999 and has achieved sales as high as \$500,000 per year. They also sold the MicroBlue SLM, a specialized, low-noise blue laser for digital photo-finishing, that was first marketed in 2000 and generated \$1 million in annual sales
Large Scale Biology Corporation (formerly Large Scale Proteomics Corporation)	94-01-0284	Developed the ProGEX product line for protein identification and research. The company also completed the first version of the Human Protein Index by identifying more than 115,000 proteins from 157 medically relevant human tissues	The 2-D gel and ProGEX line of protein analysis tools has been upgraded and improved over the years. Large Scale Biology Corporation (LSBC), which acquired LSPC in 1999, still sells research products and databases created through use of technology flowing from the knowledge acquired during this ATP-funded

			project. The company performs up to one million mass spectrometry analyses of proteins per week
Medical Analysis Systems (formerly NAVIX)	95-08-0017	Developed a two-stage reaction for DNA identification and amplification. The process identifies areas of DNA that correlate with disease	Navix did not commercialize any products from its ATP-funded research. Business issues delayed research long enough for another competitor to beat Navix to the market
Monsanto (formerly Agracetus)	94-01-0074	Developed a prototype plant with elevated levels of poly-3-hydroxybuteric acid (PHB). Although the PHB concentration was not high enough for commercialization, simply raising the PHB level at all represented a technical achievement	Due to the difficulty in attaining high enough PHB levels in the cotton fibers without "crowding out" the fibers' favorable traits, no commercialization efforts resulted from this ATP-funded research.
Orchid BioSciences (formerly Molecular Tool, Inc. Alpha Center)	94-05-0034	Developed techniques for micromachining and for handling fluids on a microscopic scale to make a simple, compact DNA typing instrument	Developed the SNPstream Ultra High Through-Put (UHT), automated array-based genotyping tool. Entered the market through Orchid BioSciences in 2001. Product, intellectual property, and research and development were sold to Beckman Coulter in December 2002. As of 2004, Beckman continues to develop and enhance the system, marketing to research and clinical laboratories. Orchid BioSciences provides genetic analyses using SNPstream UHT on a fee-for-service basis (for biotech companies, pharmaceutical companies, and criminal justice agencies). Orchid's facility was providing up to 1 million SNP scores per day by the end of 2000 on a fee-for-service basis
Valentis, Inc. (formerly Progenitor, Inc.; a subsidiary of Internueron Pharmaceuticals)	94-01-0301	Developed an understanding of how the Del-1 gene regulates angiogenesis and can be used to treat ischemia	In 2003, the company completed a Phase I clinical trial and initiated a Phase II clinical trial for Del-1 angiogenesis product for the treatment of peripheral arterial disease

Table A-3. Electronics, Computer Hardware, or Communications

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
eMagin Corporation (formerly FED Corporation)	93-01-0154	Developed manufacturing techniques for large-scale, flat-panel displays based on arrays of field emitters, a sort of "flat CRT"	Commercialized two microdisplays, SVGA 3D and SVGA+ rev2. The microdisplays are integrated into hundreds of medical, commercial, and military applications. For example, firefighters see through thick smoke by looking through a thermal-imaging camera lens to find victims, even under a blanket. They can also use the lens to find the source of a fire quickly and put it out. Researchers and doctors are using the display to enhance vision for magnetic resonance imaging (MRI), endoscopic surgery, and eye surgery
INSIC (formerly NSIC) - Short Wavelength	90-01-0231	Developed optical recording standards to improve upon traditional magnetic recording	NSIC members did not commercialize optical recording devices because remaining technical obstacles would have required significant further development of the frequency-doubling technology; and by the end of the project, competition was looming from direct-lasing green and blue diode lasers
Kopin Corporation	94-01-0304	Developed liquid crystal projection display technology capable of producing high-quality, high-resolution images for high-definition TV	Commercialized the CyberDisplay 320 Monochrome, the CyberDisplay 320 Color, the CyberDisplay 640 Color, the CyberDisplay 1280 Monochrome 60" diagonal projection HDTV, the CyberDisplay 1280 Monochrome 55" diagonal projection HDTV, the CyberDisplay 1280 Monochrome 46" diagonal projection HDTV, and CyberDisplay 1280 Monochrome 43" diagonal projection HDTV
Planar Systems, Inc. (American Display Consortium)	93-01-0054	Developed a group of patterning technologies necessary to manufacture color flat-panel displays, including large-area photo exposure tools, large-area masks, wet and dry etching tools, printing tools, panel alignment methods and a final inspection tool	Subcontractor, Photonics (now Infinite Graphics, Inc. [IGI]), commercialized customized large-area photo masks for use in high-end printer circuits, calibration plates, x-ray systems, and flat-panel displays. Photonics also developed two processes: mask cleaning & laser pattern generator

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
			<p>Subcontractor, Plasma-Therm successfully commercialized dry etching processes in its Clusterlock 7000 for 6-inch wafers. The company was sold to a Swiss company, Oerlikon-Buehrle in 1999. Planar used the Plasma-Therm etcher to produce AMEL microdisplays until 2002.</p> <p>Subcontractor, YieldUp (now FSI) developed drying tools for wet-etched substrates. This is now used for flat-panel displays and primarily computer chip manufacturing. Also used in hard disk drive cleaning and photomask cleaning. Currently, the ATP-funded component is key in seven larger processing systems: ZETA Spray Cleaning System, ANTARES CX Advanced Cleaning System, EXCALIBUR Vapor HF Etching System, MERCURY Spray Cleaning System, YieldUP 4000 Immersion Etch System, YieldUP 2000 Rinse Dry Module, and YieldUP 2100 STG Rinse Dry Integration Module</p>
SDL, Inc. and Xerox Corporation	91-01-0176	<p>Demonstrated the first integration of multiple-wavelength laser diodes on a single semiconductor device. In the course of this work, the team established several intermediary technologies and accomplished important research in the field of gallium nitride (GaN)-based blue laser diodes. Demonstrated technologies include two alternative methods for monolithic integrations of red, infrared, and blue emitters; red laser diodes with powers of up to 120 mW single mode; lasers in the 700- to 755-nm range; green and blue lasers with frequency doubling; and the lasing of blue GaN diodes at room temperature</p>	<p>After the ATP-funded project, SDL commercialized several laser products that were based on technologies developed in the course of the project: a single-mode laser using facet passivation technology; a single-mode laser for PDT applications; a dual-spot single-mode laser for data storage, printing, displays, and alignment; a multi-mode laser; fiber coupled laser bars for solid state laser pumps, medical systems and displays; and a DBR laser for frequency doubling, interferometry, atomic clocks, and spectroscopy</p>
Superconductor Technologies Inc. (formerly Conductus)	91-01-0134	<p>Developed a prototype superconducting DSP switch</p>	<p>Commercialization of the technology developed and tested during this ATP-funded project was not pursued due to a lack of interest in the technology on the</p>

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
			part of the semiconductor and communications industries
Texas Instruments Inc.	94-01-0221	Developed a special insulating material, known as aerogel, to be integrated adjacent to on-chip interconnects in order to overcome problems with interconnect delay as a result of the continuing trend toward miniaturization. Texas Instruments and NanoPore developed the world's first fully automated manufacturing process to dry an aerogel quickly	The company overcame impediments to aerogel processing early in the project, but in 1997, an industry competitor announced that it would begin using copper interconnect wiring in future integrated circuit designs. Texas Instruments then shifted focus away from aerogels for aluminum and began to develop copper interconnects. Before shifting focus, however, Texas Instruments transferred its aluminum circuit aerogel knowledge to NanoPore, which later sold the rights to continue development of the product to Honeywell. Honeywell's development efforts resulted in a product that they marketed briefly in 2002 to companies for use in manufacturing semiconductors. However, Honeywell withdrew the product in 2004 after it did not fulfill its potential as a new and innovative insulator

Table A-4. Information Technology

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
Accenture (formerly Andersen Consulting Center for Strategic Research)	94-06-0012	Developed a prototype technology for reusable software components based on software architecture considerations, including formal languages to express semantics, a graphical user interface programming environment, automated techniques for assuring that the separate components are logically compatible and properly combined, and automated systems to generate executable systems	No product was commercialized as the technology focus of the industry changed shortly after the project concluded
Cerner Corporation	94-04-0008	Developed information tools to automate, validate and distribute clinical practice guidelines for mass use	Used general concepts from the ATP-funded project to execute guidelines in its Cerner Millennium product. With Cerner Millennium, clinicians are electronically alerted about potential patient safety and regulatory issues through evidence-based medical information
Cerner Corporation (formerly DataMedic - Clinical Information Advantages, Inc.)	94-04-0038	Developed a knowledge-base-driven automated coding system in the form of a software component, CHARTnote which uses MEDencode, a technology that automatically gathers, codifies, and records specific detailed information about a patient	The software is currently incorporated into and sold with approximately 7 CHARTstation products, manufactured by VitalWorks. It is also sold separately and with other products. Products include GIstation, EMstation, EYEstation, RADstation, and other areas including internal medicine and family practice, renal dialysis, and rehabilitative medicine
InStream	94-04-0018	Developed the first behavioral healthcare (BHC) Web portal for claims processing	The software product was briefly commercialized in 1998, but was quickly overtaken by competing products after a lack of funding prevented InStream from providing the necessary upgrades and market penetration to reach positive cash flow
Lucent Technologies (formerly AT&T Bell Laboratories)	94-06-0011	Developed and successfully demonstrated their software (Symphony) to develop an easy-to-use, graphics-user interface (GUI) software assembly system for nonprogrammers that handles the complexity of building reliable,	No commercialization resulted from this project because of AT&T's corporate restructuring in 1996. Lucent decided to discontinue its development of the reusable software component product

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
		custom-designed software by using libraries of reusable, software components	
SciComp, Inc.	94-06-0003	Developed a component software and a software synthesis technology for creating mathematical models in the field of scientific computing	As of 2004, SciComp offered three software tools in the SciFinance solution that incorporate the ATP-funded software synthesis technology; SciFinance also includes two additional products that enhance SciPDE and SciMC. SciComp experienced greater demand for these products as the market
Titan Systems (formerly Intermetrics)	94-04-0040	Developed a script language and a related suite of software tools to facilitate the process of developing customized home healthcare workstations for homebound or limited-mobility, chronically ill patients	A product was not commercialized. The intellectual property was acquired by HealthVision, which chose not to further develop it
Xerox Palo Alto Research Center	94-06-0036	Developed a new programming technique called aspect-oriented programming (AOP). They also developed two prototype applications of specialized computer languages	AspectJ, an open-source language that extends Java, is now used in a significant percentage of IBM's new products and is an open-source platform. PARC transferred AspectJ to the open-source eclipse.org project in December 2002

Table A-5. Manufacturing

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
Abrasive Technology Aerospace, Inc.	95-02-0053	Developed an integrated CAD/CAM approach to applying superabrasive coatings to complex surfaces of electroplated superabrasive grinding wheels	In 2000, Abrasive Technology began to market and sell electroplated superabrasive grinding wheels using the CAD/CAM technology it developed during the ATP-funded project, and still continues to do so. The company has used the new technology to produce grinding wheels for a variety of industries, including automotive and aerospace
Cincinnati Lamb, UNOVA (Lamb Technicon)	95-02-0019	Developed an experimental prototype of a flexible line boring station with intelligent tooling and controls	The BOA technology was not commercialized because auto manufacturers found less expensive machine tools to meet their specifications
General Electric Corporation R&D	95-07-0018	Developed an intelligent process for applying thermal barrier coatings to critical components in turbine engines for power plants in order to raise firing temperatures and increase fuel efficiency	GE successfully produced an improved gas turbine engine for its new H-System combined-cycle power plant, which can achieve 60-percent energy efficiency. The high-performance thermal barrier coatings developed in part using technology from this project were essential to the design of this model. GE also applied the knowledge to upgrade existing F-System plants, which achieved 56-percent efficiency. Other companies have used the process on marine aircraft and heavy diesel engines, as well as other applications
IBM Corporation	94-03-0012	Developed an automated tool kit that could be used by vendors to develop, maintain, and join interoperating families of enterprise resource planning (ERP) and manufacturing execution system (MES) applications	IBM did not commercialize its new automated tool kit. Instead, it commercialized a service based on its new Framework for Adaptive Interoperability of Manufacturing Enterprises (FAIME) technology, enterprise application integration (EAI) services
Montronix	95-02-0020	Developed a diagnostic system that can monitor the vital signs of machining operations in real time to provide a trouble-shooting aid for process engineers who are increasingly challenged to efficiently machine smaller	The developed monitoring system later evolved into a standard Montronix product line called Spectra. A key accomplishment of this project was providing free Internet-based simulated machine-tool modeling

A. Awardee Name	B. Project Number	C. Technology Developed	D. Products or Processes Commercialized or Expected to be Commercialized Soon
		volumes of a wider variety of parts	(http://mtamri.me.uiuc.edu/testbeds/testbed.intro.html). The web-based simulation is still in use by government, research and industry
United Technologies Research Center	95-06-0011	Developed a prototype handheld device to detect refrigerant leaks during manufacture of components containing refrigerant	No commercialization occurred. All three companies cited cost of development, lack of funding, competition, and uncertain market demand as contributing factors to discontinuing research into this technology. The markets for the laser emitter for the handheld unit were also limited
York International	95-06-0004	Developed a prototype heat exchanger that was 25 percent smaller and had the same heat transfer capability as the standard size. Furthermore, York developed a method and a tool that they still use in their ongoing research and development. They also demonstrated that oval-tube geometry is 10 percent more efficient for heat transfer than round tubes	Using the methods developed during this project, York developed a new commercialized plate fin, called HiQ. York uses the fin in its ECO2 rooftop heating/cooling units. Its proprietary enhancements yield approximately twice the heat transfer when compared to a standard fin. Due to the prohibitive manufacturing capital cost, York has postponed commercializing oval-tube coil technology

APPENDIX B

Reasons for Terminating ATP Projects

At the end of an ATP competition, projects are selected for award and the winners are announced. Most of these projects proceed through their multi-year research plans to completion. Some are not carried through to completion for a variety of reasons. These projects are collectively called “terminated projects.”

Between 1990 and September 2004, there were 768 ATP awards issued, of which 84²² projects ended before completion. Below is a percentage distribution by category of the reasons for termination.

Change in goals

- 54 percent ended because of changes in the strategic goals of the companies, changes in the business climate or markets, changes in company ownership, or other business-related facts.

Lack of technical progress

- 12 percent ended because of lack of technical progress, which sometimes occurs at go/no-go decision points recommended by the participant(s).

Project no longer meets ATP criteria

- 11 percent ended because changes in scope, membership, performance, or other factors meant that the project no longer met ATP’s technical and/or economic criteria.

Lack of agreement among joint venture members

- 2 percent ended because the joint venture members could not reach an agreement on some issues.

Financial distress

- 11 percent ended due to the financial distress of a key participant.

Early success

- 5 percent ended due to early success of the project!

Although projects may end early, it is not necessarily an indication of total failure. Projects that ended early produced important knowledge gains; involved integrated planning for research, development, and business activities that may have some benefit to participating companies; and entailed substantive cross-disciplinary contact among scientists and other researchers, cross-talk among technical and business staff, and negotiations among executives at different companies.

²² Included in this figure are four projects that were cancelled before the project began, comprising approximately 5 percent of the total.

These characteristics still benefit the economy by stretching the thinking and horizons of participants in the process. Companies may learn about new opportunities and apply integrated planning of research and business activities to other projects. In summary, terminated projects may have some positive impact even though they incur costs.

APPENDIX C

Composite Performance Rating System (CPRS) Star Ratings—First 150 Completed Projects

Project Number	Project Identifier (Title/Lead Organization)	Data Set	Overall Project Success
91-01-0243	Aastrom Biosciences, Inc.	1st 50	****
91-01-0146	American Superconductor Corp.	1st 50	****
94-02-0027	Automotive Composites Consortium (a Partnership of DaimlerChrysler [formerly Chrysler], Ford and General Motors)	3rd 50	****
94-04-0038	Cerner Corporation (formerly DataMedic - Clinical Information Advantages, Inc.)	3rd 50	****
96-01-0263	ColorLink, Inc.	2nd 50	****
91-01-0256	Cree Research, Inc.	1st 50	****
91-01-0184	Engineering Animation, Inc.	1st 50	****
93-01-0085	Integra LifeSciences	1st 50	****
94-01-0304	Kopin Corporation	3rd 50	****
94-01-0284	Large Scale Biology Corporation (formerly Large Scale Proteomics Corporation)	3rd 50	****
91-01-0041	Nanophase Technologies Corporation	2nd 50	****
90-01-0154	National center for Manufacturing Sciences (NCMS)	1st 50	****
94-05-0034	Orchid BioSciences (formerly Molecular Tool, Inc. Alpha Center)	3rd 50	****
94-06-0003	SciComp, Inc.	3rd 50	****
91-01-0176	SDL, Inc. and Xerox Corporation	3rd 50	****
94-05-0012	Third Wave Technologies, Inc.	2nd 50	****
92-01-0133	Tissue Engineering, Inc.	1st 50	****
94-06-0024	Torrent Systems, Inc. (formerly Applied Parallel Technologies, Inc.)	1st 50	****

Project Number	Project Identifier (Title/Lead Organization)	Data Set	Overall Project Success
94-06-0036	Xerox Palo Alto Research Center	3rd 50	****
91-01-0112	X-Ray Optical Systems (XOS), Inc.	2nd 50	****
95-05-0034	ABB Lummus Global, Inc. (formerly ABB Lummus Crest)	3rd 50	***
95-01-0131	Advanced Refractory Tech	3rd 50	***
93-01-0113	Amersham Pharmacia Biotech (formerly U.S. Biochemical Corporation)	1st 50	***
91-01-0177	Auto Body Consortium	1st 50	***
94-01-0115	Calimetrics, Inc.	1st 50	***
93-01-0055	Caterpillar Corporation	2nd 50	***
95-01-0022	Corning Tropol (formerly Tropol Corporation)	2nd 50	***
92-01-0136	Cynosure, Inc.	1st 50	***
95-02-0055	Dana Corporation	2nd 50	***
92-01-0115	Diamond Semiconductor Group, LLC	1st 50	***
98-02-0034	Digital Optics Corporation	2nd 50	***
94-01-0402	Displaytech, Inc.	2nd 50	***
90-01-0064	E.I. Du Pont de Nemours & Company	1st 50	***
94-02-0025	Ebert Composites Corporation	2nd 50	***
95-11-0012	EDO Specialty Plastics (formerly Specialty Plastics Inc.)	2nd 50	***
93-01-0154	eMagin Corporation (formerly FED Corporation)	3rd 50	***
91-01-0178	Ford Motor Company	2nd 50	***
95-07-0018	General Electric Corporation R&D	3rd 50	***
92-01-0044	Genosensor Consortium (c/o Houston Advanced Research Center)	3rd 50	***
92-01-0040	GM Thermoplastic Engineering Design (Engineering Design with Thermoplastics)	3rd 50	***

Project Number	Project Identifier (Title/Lead Organization)	Data Set	Overall Project Success
92-01-0116	Honeywell (formerly Allied Signal Inc.)	2nd 50	***
95-09-0052	Hynomics (formerly Hybrithms Corporations, formerly Sagent Corporation)	2nd 50	***
94-05-0018	Hyseq, Inc.	2nd 50	***
93-01-0149	IBM T.J. Watson Research Center	3rd 50	***
92-01-0017	Illinois Superconductor Corporation	1st 50	***
94-05-0019	Incyte Corporation (formerly Combion, Inc.)	3rd 50	***
91-01-0016	Information Storage Industry Consortium (INSIC, formerly NSIC)	2nd 50	***
91-01-0262	Kopin Corporation	1st 50	***
93-01-0101	Kurzweil Applied Intelligence, Inc	2nd 50	***
93-01-0183	MicroFab Technologies, Inc.	2nd 50	***
91-01-0224	Molecular Simulations, Inc. (formerly Biosym Technologies, Inc.)	1st 50	***
96-01-0172	Nanogen, Inc.	2nd 50	***
90-01-0166	Nonvolatile Electronics, Inc.	1st 50	***
93-01-0071	Perceptron, Inc.	1st 50	***
93-01-0205	Physical Optics Corporation (POC)	2nd 50	***
94-04-0024	PPD Informatics (formerly Belmont Research, Inc.)	2nd 50	***
92-01-0123	Sage and 3M Corporation	1st 50	***
94-02-0010	Strongwell Corporation	2nd 50	***
94-04-0046	Surgency (formerly Benchmarking Partners)	2nd 50	***
95-05-0002	The Dow Chemical Company	3rd 50	***
97-01-0087	TopicalNet (formerly Continuum Software, Inc.)	2nd 50	***
93-01-0124	Vitesse Semiconductor Corporation	1st 50	***

Project Number	Project Identifier (Title/Lead Organization)	Data Set	Overall Project Success
94-04-0027	3M Company, Health Information Systems	2nd 50	**
95-02-0053	Abrasive Technology Aerospace, Inc.	3rd 50	**
91-01-0187	AlliedSignal, Inc.	1st 50	**
90-01-0060	American Display Consortium	1st 50	**
95-01-0263	Aphios Corporation	3rd 50	**
91-01-0142	AstroPower, Inc.	1st 50	**
93-01-0250	BioTraces, Inc.	1st 50	**
95-07-0020	Bosch (formerly Allied Signal)	3rd 50	**
93-01-0234	BP Amoco	3rd 50	**
94-01-0190	Catalytica Energy Systems (formerly Catalytica, Inc.)	3rd 50	**
94-01-0137	Cengent Therapeutics Inc. (formerly Moldyn Inc.)	3rd 50	**
95-02-0019	Cincinnati Lamb, UNOVA (Lamb Technicon)	3rd 50	**
90-01-0210	Communication Intelligence Corporation	1st 50	**
93-01-0211	Communication Intelligence Corporation	1st 50	**
94-01-0287	Crucible Materials Corporation, Crucible Companction Metals Division	3rd 50	**
93-01-0091	Elsicon (formerly Alliant Techsystems, Inc.)	2nd 50	**
92-01-0022	FSI International, Inc.	1st 50	**
92-01-0074	Geltech Incorporated	1st 50	**
94-01-0147	Genzyme Corporation (formerly GelTex Pharmaceuticals, Inc.)	2nd 50	**
91-01-0034	HelpMate Robotics, Inc. (formerly Transitions Research Corporation)	1st 50	**
93-01-0104	Honeywell (formerly Allied Signal)	3rd 50	**
95-07-0003	Honeywell (formerly Allied Signal)	3rd 50	**

Project Number	Project Identifier (Title/Lead Organization)	Data Set	Overall Project Success
94-05-0004	JDS Uniphase (formerly The Uniphase Corporation)	3rd 50	**
94-01-0133	Laser Power Corporation	2nd 50	**
90-01-0212	Light Age, Inc.	1st 50	**
90-01-0121	Lucent Technologies Inc.	1st 50	**
93-01-0191	M&M Precision Systems Corporation	2nd 50	**
92-01-0053	Mathematical Technologies Inc.	1st 50	**
91-01-0088	Michigan Molecular Institute	1st 50	**
93-01-0027	Micron Optics, Inc.	2nd 50	**
95-02-0020	Montronix	3rd 50	**
95-08-0009	Nanogen, Inc.	2nd 50	**
94-01-0357	Norton Diamond Film	2nd 50	**
93-01-0054	Planar Systems, Inc. (American Display Consortium)	3rd 50	**
94-06-0026	Reasoning Systems, Inc.	2nd 50	**
90-01-0232	Saginaw Machine Systems, Inc.	1st 50	**
91-01-0263	Spire Corporation	1st 50	**
94-01-0221	Texas Instruments Inc.	3rd 50	**
94-06-0034	TopicalNet, Inc. (formerly Continuum Software, Inc.)	2nd 50	**
94-01-0063	Union Switch and Signal, Inc.	1st 50	**
94-01-0301	Valentis, Inc. (formerly Progenitor, Inc.; a subsidiary of Interneuron Pharmaceuticals)	3rd 50	**
91-01-0261	Westinghouse Plasma Corp.	1st 50	**
93-01-0041	Air Products and Chemicals, Inc.	3rd 50	*
94-04-0017	American Healthware Systems	2nd 50	*

Project Number	Project Identifier (Title/Lead Organization)	Data Set	Overall Project Success
94-02-0040	Budd Company, Design Center	2nd 50	*
92-01-0132	GE Corporate Research and Development	2nd 50	*
91-01-0069	Honeywell, Inc., Technology Center	2nd 50	*
94-03-0012	IBM Corporation	3rd 50	*
92-01-0034	Ingersoll Milling Machine Company	2nd 50	*
90-01-0231	INSIC (formerly NSIC) - Short Wavelength	3rd 50	*
94-04-0037	KOOP Foundation, Inc.	2nd 50	*
95-10-0067	KOOP Foundation, Inc.	2nd 50	*
91-01-0258	Microelectronics Center of NC	1st 50	*
95-02-0005	Perceptron (formerly Autospect, Inc.)	2nd 50	*
93-01-0045	Philips Laboratories	2nd 50	*
94-01-0111	Praxair, Inc.	3rd 50	*
91-01-0267	PreAmp Consortium	1st 50	*
91-01-0134	Superconductor Technologies Inc (formerly Conductus)	3rd 50	*
93-01-0109	Thomas Electronics, Inc.	1st 50	*
95-06-0011	United Technologies Research Center	3rd 50	*
95-07-0026	Wyman-Gordon	3rd 50	*
95-06-0004	York International	3rd 50	*
94-06-0012	Accenture (formerly Andersen Consulting Center for Strategic Research)	3rd 50	-
94-04-0025	Accenture (formerly Andersen Consulting)	2nd 50	-
92-01-0055	Accuwave Corporation	1st 50	-
91-01-0135	Aphios Corporation	1st 50	-

Project Number	Project Identifier (Title/Lead Organization)	Data Set	Overall Project Success
91-01-0025	Armstrong World Industries, Inc.	1st 50	-
92-01-0007	Calmac Manufacturing Corporation	2nd 50	-
94-04-0008	Cerner Corporation	3rd 50	-
95-01-0148	Dow AgroSciences (Mycogen Corporation)	3rd 50	-
94-05-0033	Dupont Qualicon (formerly Dupont FQMS Group)	3rd 50	-
92-01-0109	Eagle-Picher Research Laboratory	2nd 50	-
92-01-0122	ETOM Technologies, Inc. (formerly Optex Communications, Inc.)	1st 50	-
95-05-0031	General Electric Company	2nd 50	-
90-01-0126	Hampshire Instruments, Inc.	1st 50	-
91-01-0017	IBM Corporation	1st 50	-
92-01-0103	IBM Corporation	1st 50	-
94-04-0018	InStream	3rd 50	-
94-06-0011	Lucent Technologies (formerly AT&T Bell Laboratories)	3rd 50	-
91-01-0057	MediaBin (formerly Iterated Systems Incorporated)	2nd 50	-
95-08-0017	Medical Analysis Systems (formerly NAVIX)	3rd 50	-
94-01-0074	Monsanto(formerly Agrecetus)	3rd 50	-
92-01-0124	NetOptix Corporation (formerly Galileo Corporation)	1st 50	-
95-07-0011	PCC Structurals	3rd 50	-
95-07-0006	Praxair Surface Technologies, Inc.	2nd 50	-
92-01-0035	Sheffield Automation (formerly Giddings & Lewis)	2nd 50	-
91-01-0071	Thermo Trilogy Corporation	1st 50	-
94-04-0040	Titan Systems (formerly Intermetrics)	3rd 50	-