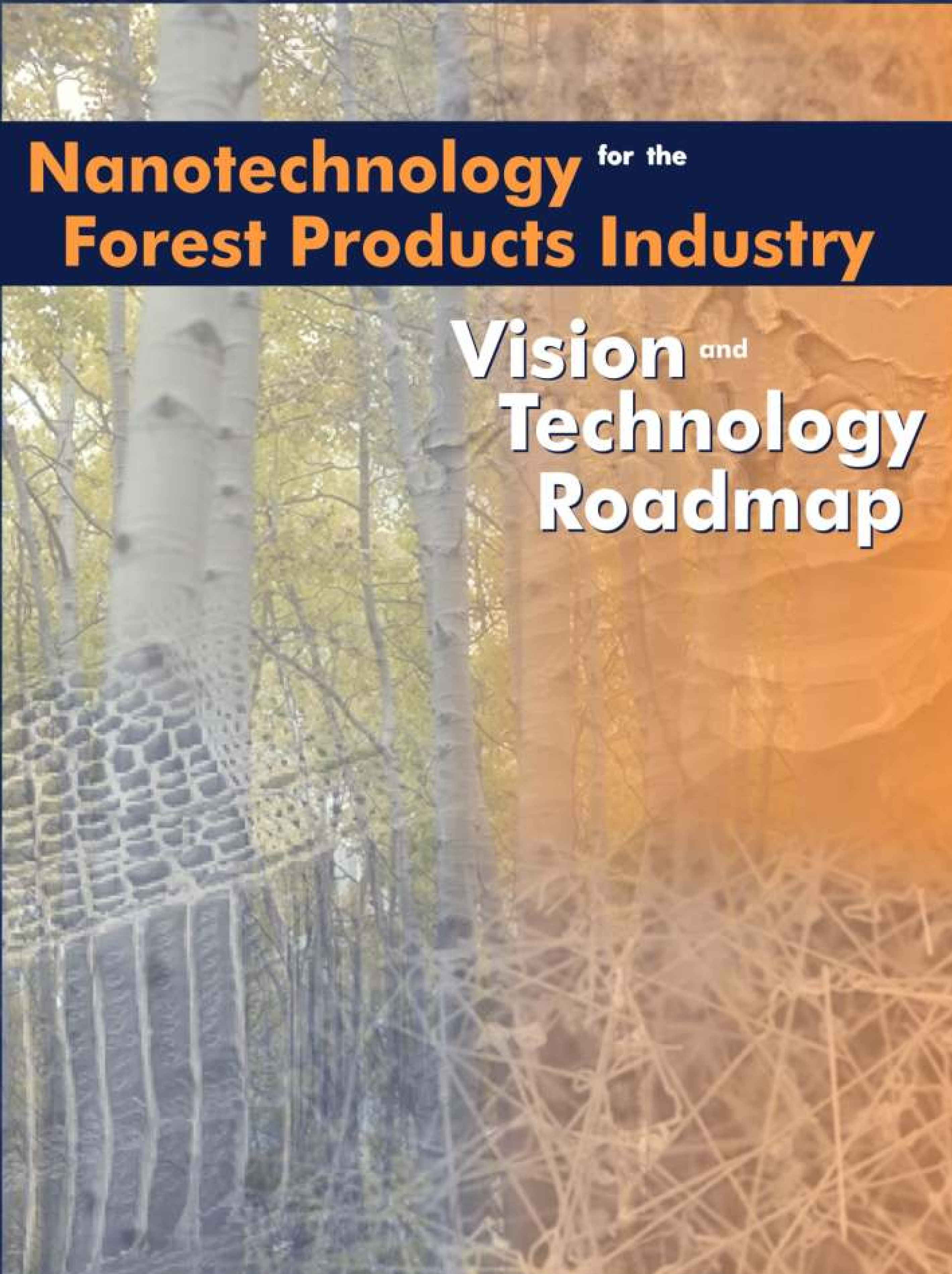
Nanotechnology for the Forest Products Industry

Vision and Technology Roadmap



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Executive Summary

Introduction

Nanotechnology is defined as the manipulation of materials measuring 100 nanometers or less in at least one dimension. Nanotechnology is expected to be a critical driver of global economic growth and development in this century. Already, this broad multi-disciplinary field is providing glimpses of exciting new capabilities, enabling materials, devices, and systems that can be examined, engineered, and fabricated at the nanoscale. Using nanotechnology to controllably produce nanomaterials with unique properties is expected to revolutionize technology and industry.

The forest products industry relies on a vast renewable resource base to manufacture a wide array of products that are indispensable to our modern society. American paper and wood products companies produce over 225 million tons of products each year that touch every aspect of our lives, contribute over $240 billion per year to the gross domestic product, and employ over 1.1 million Americans. Emerging nanotechnologies offer the potential to develop entirely new approaches for producing engineered wood- and fiber-based materials. They can also enable the development of a wide range of new or enhanced wood-based materials and products that offer cost-effective substitutes for non-renewable materials used in the manufacture of metallic, plastic, or ceramic products. Nanotechnology could transform the forest products industry in virtually all aspects—ranging from production of raw materials, to new applications for composite and paper products, to new generations of functional nanoscale lignocellulosics. Research and development (R&D) in nanotechnology is critically important to the economical and sustainable production of these new generations of forest-based materials—materials that will meet societal needs while improving forest health and contributing to the further expansion of the biomass-based economy.

A nanometer is a billionth of a meter, or 80,000 times thinner than a human hair.

Nanotechnology can be used to tap the enormous undeveloped potential that trees possess—as photochemical "factories" that produce rich sources of renewable raw materials using sunlight, water, and carbon dioxide. The consumption of carbon dioxide in the production of these raw materials provides a carbon sink for this important greenhouse gas. By harnessing this potential, nanotechnology can provide benefits that extend well beyond fiber production and new materials development and into the areas of sustainable energy production, storage, and utilization. For example, nanotechnology may provide new approaches for obtaining and utilizing energy from sunlight—based on the operation of the plant cell. Novel new ways to produce energy, chemicals, and other innovative products and processes from this renewable, domestic resource base will help address major issues facing our nation, including national energy security, global

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climate change, air and water quality, and global industrial competitiveness.

Potential Uses for Nanotechnology in Forest Products

Potential uses for nanotechnology include developing intelligent wood- and paper-based products with an array of nanosensors built in to measure forces, loads, moisture levels, temperature, pressure, chemical emissions, attack by wood decaying fungi, et cetera. Building functionality onto lignocellulosic surfaces at the nanoscale could open new opportunities for such things as pharmaceutical products, self-sterilizing surfaces, and electronic lignocellulosic devices. Use of nanodimensional building blocks will enable the assembly of functional materials and substrates with substantially higher strength properties, which will allow the production of lighter-weight products from less material and with less energy requirements. Significant improvements in surface properties and functionality will be possible, making existing products much more effective and enabling the development of many more new products. Nanotechnology can be used to improve processing of wood-based materials into a myriad of paper and wood products by improving water removal and eliminating rewetting; reducing energy usage in drying; and tagging fibers, flakes, and particles to allow customized property enhancement in processing.

Potential Uses

- Intelligent products with nanosensors for measuring forces, loads, moisture levels, temperature, et cetera.
- As building blocks of products with substantially enhanced properties.
- As coatings for improving surface qualities to make existing products more effective.
- As basis for making lighter-weight products from less material and with fewer energy requirements.

Many challenges stand in the way of exploiting the potential benefits of nanotechnology in the forest products industry and much research will be needed to move forward in this arena. Researchers will need to address technical challenges such as the lack of fundamental understanding of lignocellulosic material formation at the nanoscale and the absence of adequate technology for measuring and characterizing these materials at the nanoscale. Participants in this effort will need to come from not only academia but from industry and government as well; they will need to come together to form an infrastructure and move forward as a cohesive unit working simultaneously towards a single goal—the advancement of nanotechnology into the forest products industry.

Advancing the nanotechnology research agenda efficiently and effectively will require gaining consensus on research needs and priorities among the forest products industry, universities with forest products research and education departments and programs, technology developers and suppliers, research institutes and laboratories serving the forest products industry, and mission-oriented federal agencies with supportive goals, such as the National Science Foundation, the U.S. Department of Agriculture (USDA), and the U.S. Department of Energy (DOE). In building consensus, the forest products sector can capitalize on the good working relationships that the forest products industry has with its university research community, and with federal agencies such as the USDA Forest Service; the USDA Cooperative State Research, Education, and Extension Service (CSREES); the DOE Industries of the Future Program; and the DOE Biomass Program. In addition, the forest products sector can take advantage of the linkages it has with research communities across the globe. As the industry's operation and markets become more and more global in nature, international cooperation and collaboration is imperative.

Increased cooperation must also occur between the forest products and nanotechnology research communities, the federal departments and agencies with ongoing programs in nanotechnology R&D, and the National Nanotechnology Initiative (NNI). Linkages between the forest products sector research communities and the NNI umbrella centers and user facilities (such as those sponsored by the National Science Foundation, DOE, and National Institutes of Health) are critical to capturing synergies, enhancing accomplishments, and avoiding needless duplication of facilities and other resources.

Workshop

In a first step towards reaching the goals of applying nanotechnology in the forest products industry, a workshop to explore opportunities and research needs was convened on October 17-19, 2004, at the National Conference Center in Lansdowne, Virginia. Over 110 leading researchers with diverse expertise from industry, government laboratories, and academic institutions from North America and Europe were in attendance. Workshop objectives were as follows:

- Develop a vision for nanotechnology in the forest products industry
- Develop a roadmap for nanotechnology in the forest products industry (identify potential applications and uses, identify knowledge gaps and the research needed)
- Interest federal funding entities in nanotechnology as applied to forest products industry manufacturing processes and lignocellulosic materials
- Foster cooperation and collaboration among industry, academia, and government to fill knowledge gaps

This document represents a report of the workshop, and the first roadmap of technological needs and research priorities for nanotechnology applied to forest products manufacturing processes and forest-based lignocellulosic materials. Workshop participants identified the fundamental research challenges in nanoscale lignocellulosic biopolymer structures, novel surface phenomena, biosynthesis, systems integration, education, and introduction of nanomaterials into the marketplace. An overview of the Forest Products Nanotechnology Roadmap is shown in Figure 1.

Workshop participants also identified some of the unique properties and characteristics of wood lignocellulosic biopolymers that make them an exciting avenue for research, including:

1) Lignocellulosic biopolymers are some of the most abundant biological raw materials, have a nanofibrillar structure, have the potential to be made multifunctional, and can be controlled in self-assembly.

2) Lignocelluloses as nanomaterials and their interaction with other nanomaterials are largely unexplored.

3) New analytical techniques adapted to biomaterials are allowing us to see new possibilities.

It is hoped that this vision and roadmap will inspire researchers to pursue these opportunities and encourage the formation of collaborative research programs.

Vision Statement

To sustainably meet the needs of present and future generations for wood-based materials and products by applying nanotechnology science and engineering to efficiently and effectively capture the entire range of values that wood-based lignocellulosic materials are capable of providing.

Figure 1. Overview of the Forest Products Nanotechnology Roadmap

Vision for Nanotechnology in the Forest Products Industry

To sustainably meet the needs of present and future generations for wood-based materials and products by applying nanotechnology science and engineering to efficiently and effectively capture the entire range of values that wood-based lignocellulosic materials are capable of providing.

Vision Elements

- Novel lignocellulosic nanomaterials, produced from our renewable forest resource base, are routinely used by a variety of manufacturers as economically viable alternatives to non-renewable, energy-intensive materials such as metals, plastics and ceramics
- Nanoscale architecture and properties of lignocellulosics are utilized to improve the manufacturing process efficiency and product functionality of a wide array of commercial products needed by human kind
- Nanoscale materials and nanotechnology are used to achieve the most efficient and effective use of forest-based materials and manufacture of all forest products
- Nanotechnology has enabled the U.S. forest products industry to revolutionize manufacturing processes and product offerings, making it globally competitive and a key contributor to sustainable, environmentally-safe economic growth in the U.S. and around the world

Strategy

Adapt and deploy existing nanotechnologies

- Reduces costs by leveraging existing capital investment
- Shortest time for deployment
- Exploits existing nanotechnology knowledge base
- Adds value and functionality to existing products

Create and deploy novel new nanotechnologies

- Exploits the unique nanoscale components and properties of wood
- Enables new generations of cost-effective products and materials
- Exploits the full potential of wood as a material
- Achieves maximum efficiency of material use

Priority Activities

- Develop consensus on nanotechnology R&D priorities for the forest products industry with respect to:
  - Liberating nanodimensional cellulose fibrils
  - Using nanomaterials, nanosensors, and other applications of nanotechnology to improve the raw material and energy efficiency of conversion processes
  - Achieving directed design of biopolymer nanocomposites
  - Developing biomimetic processes for synthesizing lignocellulosic-based nanomaterials
  - Utilizing self-assembly of nanodimensional building blocks for functional structures and coatings
  - Exploiting the nanoscale architecture of lignocellulosics
  - Biofarming lignocellulosic materials with unique multifunctional properties
- Develop a portfolio of short-, mid-, and long-term R&D that is focused on high-impact, high priority activities most critical to commercial producers of nanomaterials and nanoproducts
- Link the forest products sector R&D community with the broader community of nanotechnology R&D
- Establish a $40 - $60 million per year nanotechnology R&D program oriented towards forest-based materials and manufacturing processes

1—Overview of the Forest Products Industry[1]

The U.S. forest products sector is often described as a mature industry, with moderate profit opportunities and stable revenues. Research and investment have the potential to reinvigorate this key American industry, which is largely based on renewable, carbon-neutral raw materials, and expand its global opportunities in the decades ahead. These research and development (R&D) efforts must focus on the most exciting new industrial technology to come along in years—the use of nanomanufacturing techniques, which are expected to revolutionize traditional industrial processes over the next decades.

Industry Overview

- A mature industry that plays a vital role in U.S. economy.
- Energy use remains disproportionately high—a very energy-intensive sector.
- Pressures from global competitiveness demand advances in process technologies.
- Nanotechnology could revitalize the industry.

Strategic Drivers for Nanotechnology in the Forest Products Industry

Strengthening U.S. Industrial Competitiveness and Sustainability

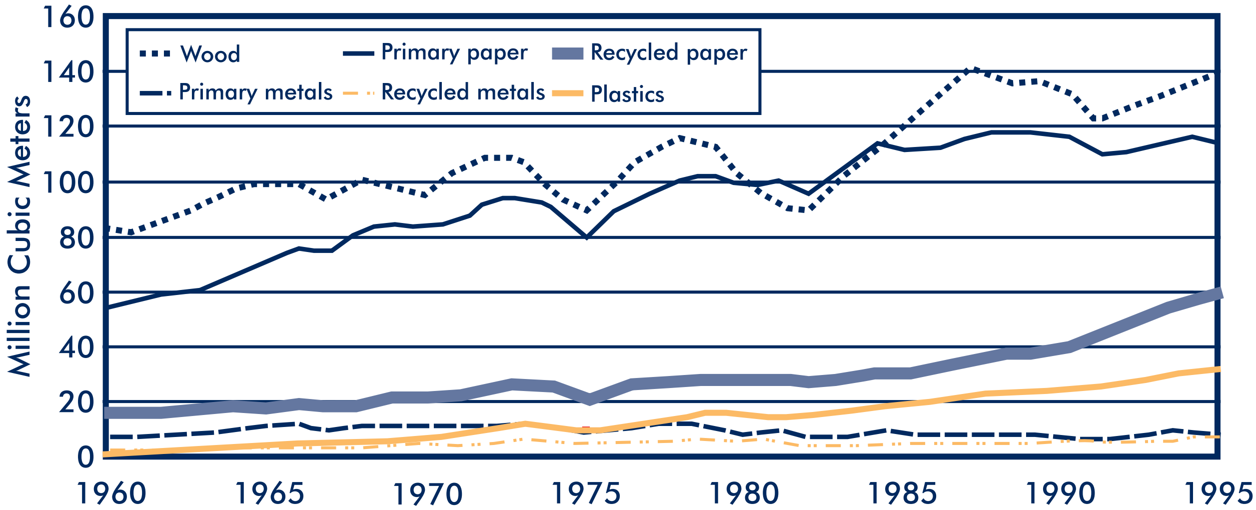
Nanotechnology represents a major opportunity to generate new products and industries in the coming decades. The ability to see materials down to atomic dimensions and determine and alter how materials are constructed at nano- and atomic scales is providing the opportunity to develop new materials and products in unprecedented ways. In the past, materials scientists concentrated efforts on simple, single-crystals and homogeneous materials that were easier to understand and could be analyzed by the techniques of the time. We now have much improved tools to investigate and understand how wood, a composite cellular material, is synthesized in a tree; how the molecular and nanoscale components are assembled; and how this architecture and assembly controls material properties.

The many thousands of products derived from our forests are ubiquitous and are taken for granted in our everyday world—the hallmark of a great product and great material. Nanotechnology now offers the opportunity to re-invent how we utilize wood and wood-based materials and the industry that converts it to the myriad of products in use today. It can enable the development of a wide range of new or enhanced wood-based materials and products that offer cost-effective substitutes for non-renewable materials used in the manufacture of metallic, plastic, or ceramic products.

By employing nanotechnology to revitalize the forest products industry, we can strengthen one of America's core manufacturing competencies (Figure 2). The U.S. has a massive infrastructure in place for growing, harvesting and processing wood products, which provides a key employment base in almost every state. This infrastructure provides a fundamental strategic advantage to build on for preserving the global economic competitiveness of this industry.

[1] Matos and Wagner 1998; Wagner 2002; United Nations 2005; United Nations 1997; U.S. Department of Energy 2004; Paperloop 2004; McNutt and Cenatempo 2003; Ince et al. In press.

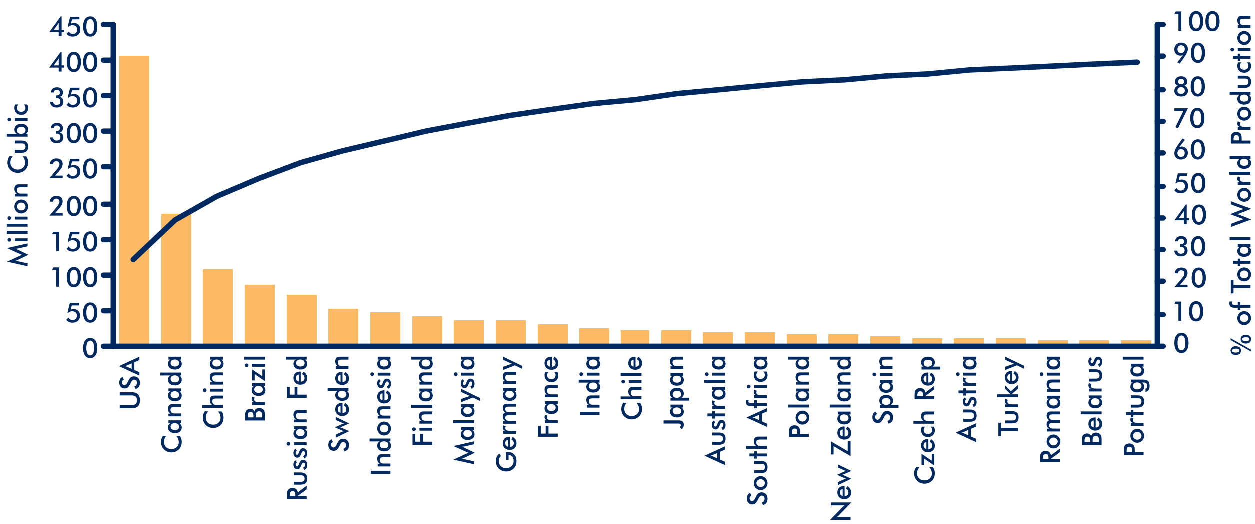
Figure 2. Consumption of Materials in the U.S., 1960-1995



Source: Matos and Wagner 1998

Large forest resources combined with prudent forest management and a good system of roadways, canals, and railways has allowed the U.S. to develop and maintain the world's largest forest products industry. As shown in Figure 3, even Canada—our largest competitor—produces only a fraction of the U.S. industrial roundwood harvest. Other major competitors, such as Brazil, Indonesia, Finland and Sweden, produce even smaller fractions. At the current rate of timber production (400 million cubic meters (m^3) per year), the U.S. is still far short of depleting the almost 31 billion m^3 of standing timber in America's forests, which is being added to at the rate of over 850 million m^3 per year. Not only could more wood be obtained from U.S. forestlands without detriment to the environment, but there would also be a variety of positive environmental benefits. More intense forestry practices such as the use of fast growing

Figure 3. Industrial Roundwood Production, 1997



Source: United Nations 1997

tree species and plantations would be expected to further increase forest productivity and increase our ability to sustainably produce timber.

Improving Industrial Environmental Performance

Trees utilize carbon dioxide (CO_2) from the atmosphere, water and nutrients from the soil, and the energy of the sun (via photosynthesis) to produce the nano-dimensional arrays that comprise the three-dimensional cellular composite materials known as wood. Increasing our use and dependence on wood and wood-based materials for new generations of nanomaterials and products can provide a number of environmental benefits. For example, trees provide an efficient way to sequester CO_2 and lock it up in wood. Forests also provide an efficient and effective means of controlling water run-off from rainfall, which helps to recharge aquifers, maintain flows of surface waters, and prevent erosion of valuable topsoil. Other environmental benefits of properly managed commercial forestlands include forest fire hazard mitigation, improving forest health and condition, and slowing conversion of privately-held forest land to non-forest uses by providing increased economic returns for forestlands. In addition to the benefits provided by forests, new or enhanced wood-based materials offer a renewable alternative in a world that will see exponential growth in demand for consumer goods and products in developing countries. Nanotechnology can also be used to make manufacturing processes more efficient and effective, enabling products to be made with substantially less raw material and energy inputs, and increasing the ability of these products to be recovered and recycled.

The Industry Today

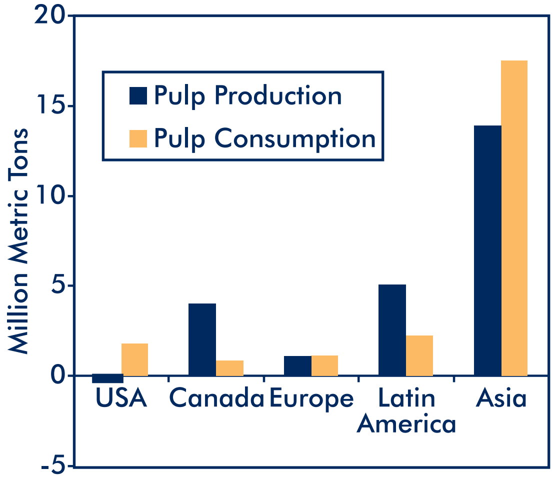
The U.S. forest products industry produces thousands of products that are essential for everyday needs in communication, education, packaging, construction, shelter, sanitation, and protection. The United States is the world's leading producer of lumber and wood products used in residential construction and in commercial wood products such as furniture and containers. The United States is also the leader in the pulp and paper business, producing approximately 28 percent of the world's pulp and 25 percent of the world's paper and paperboard. Total U.S. shipments are valued at $243 billion annually, of which nearly two-thirds, or $156 billion, come from the pulp and paper sector.

Forest products manufacturing continues to play a vital role in the U.S. economy. As a major national employer, the industry operates thousands of manufacturing facilities throughout the country, ranging from large, state-of-the-art paper and board mills to small, family-owned saw mill operations. In this sector more than 1.1 million workers are employed in good-paying jobs. Pulp, paper, composite, and saw mills are particularly vital to rural areas, where they are often a region's leading employer. In all, forest products account for 1.2 percent of the total U.S. Gross Domestic Product (GDP).

Yet it has become ever more difficult for the industry to generate the capital it needs to stay competitive in an increasingly global marketplace. Along with increased global competition, the cost of energy, raw materials, and labor has escalated in the United States, placing severe competitive pressures on U.S. producers. As a U.S. Department of Energy (DOE) report notes, "Energy-intensive industries face enormous competitive pressures that make it difficult to make the necessary R&D investments in technology to ensure future efficiency gains."

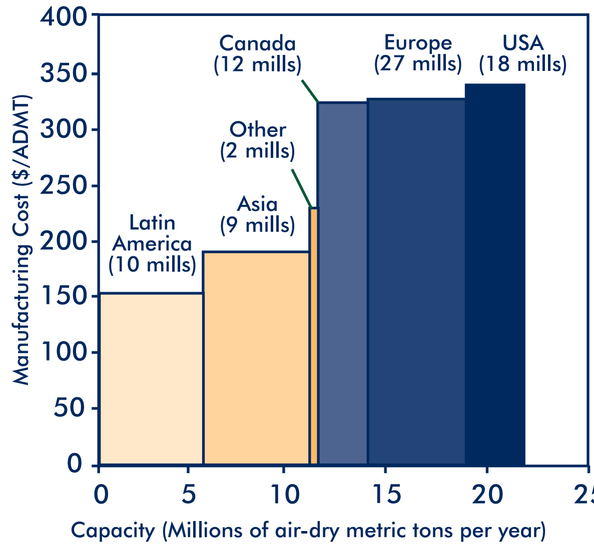
Additionally, while the United States still maintains a relatively low-cost position in the cost of wood, relative to many of its competitors, equatorial nations such as Brazil and Indonesia have developed fast-growing wood species such as eucalyptus

Figure 4. Changes in Pulp Production 1990-2000



Source: Paperloop 2004

Figure 5. Bleached Hardwood Kraft Market Pulp Manufacturing Cost Curve



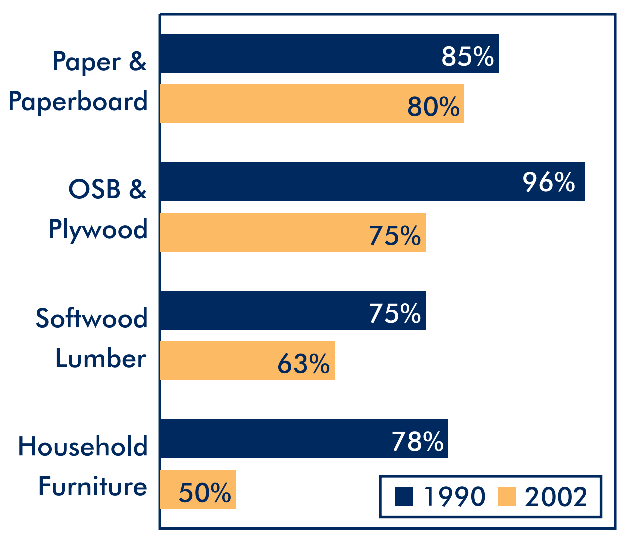
Source: Ince, P. et al. In press

and acacia that have put U.S. wood producers at a competitive disadvantage. Moreover, the United States has one of the highest labor costs in the world. Without significant gains in productivity, U.S. industry will continue to lose out to offshore production of traditional pulp and paper products. Figures 4 and 5 illustrate global shifts in pulp production from 1990 to 2000 and the 2003 global cost-capacity curve for hardwood market pulp producers.

Forest products have traditionally been a strong export market for U.S. manufacturers, but global competition from lower-cost producers is increasing. By 2001, the U.S. paper industry exported $18 billion; however, imports totaled $33 billion. For wood products, exports were valued at $3 billion and imports at $10.6 billion. As shown in Figure 6, U.S. wood and paper products producers have lost a significant percentage of the U. S. market to imports. Clearly, R&D to create newer, higher-value products for global markets is imperative for U.S. manufacturers.

Recent trends on Wall Street have offered other challenges to reinvestment and growth. The industry's high capital intensity and the short-term focus on quarterly results tend to limit the industry's ability to take risks and to invest in new technology research and development. The industry's response has been to pursue pre-competitive, collaborative R&D in partnership with government agencies such as the DOE, and with public and private research universities. The results have been extremely fruitful.

Figure 6. Loss of U.S. Markets to Imports



Domestically produced shares of U.S. consumption in four leading forest product sectors from 1990 to 2002

Source: Ince, P. et al. In press

Energy Efficiency Pays Off

The significance of energy use to the forest products industry, and to the pulp and paper sector in particular, can hardly be overstated. Fully a third of all energy used in the United States is consumed in industrial processes, and forest products ranks among the top eight energy-intensive industries, along with chemical, mining, steel, and petroleum refining. At least 18 percent of U.S. industrial energy use can be attributed to forest products manufacturing—and of the 3.2 quads used by the industry in 1998, 2.7 quads were consumed in pulp and paper processes.

New technologies that reduce energy consumption will improve the industry's economic competitiveness and also reduce the nation's overall energy consumption. Already, the industry has made great strides, decreasing primary energy intensity by 27 percent since 1972 while at the same time dramatically expanding output. Process efficiency advances such as cogeneration (the industry generates more than half of its energy and two-thirds the electricity it uses on-site through steam and cogeneration systems) and the replacement of fossil fuel (down 17 percent) with biomass (up 19 percent) have aided this effort. Contributing to this effort are the partnerships with the DOE and leading academic research institutions, as well as the ever-growing portfolio of energy-saving and cost-cutting techniques and the more than 100 collaborative research projects that have been funded since 1994 as part of the Forest Products Industry of the Future Program. This successful partnership can provide both a model and a springboard for a new area of emphasis on nanotechnology.

2—Vision for Nanotechnology in the Forest Products Industry

Vision Statement

To sustainably meet the needs of present and future generations for wood-based materials and products by applying nanotechnology science and engineering to efficiently and effectively capture the entire range of values that wood-based lignocellulosic materials are capable of providing.

Nature has utilized nanostructures since the earth began to cool 4.5 billion years ago and has blessed us with a rich legacy of examples to stimulate our imaginations. These range from the microstructures of minerals to the intricate molecular mechanisms of life. While it is now possible for us to manufacture structures that do not occur in nature, we are strongly guided by the immense variety of those that do. Some of the most important applications of biotechnology are likely to be the tuning up of useful cellular machinery that nature has not yet had time to evolve to its most efficient form. We have been doing something similar for a century and a half with organic molecules – dyes, for example, or synthetic fibers – and Japanese metallurgists were inventing new microstructures much earlier than that to create edged tools and weapons of legendary quality. They were not aware of the nanoscale origins of their products, but they were producing them just the same.

Today

Some of the most important developments in nanotechnology are occurring at the interface between biological and inorganic systems. The current emphasis of the new branch of chemistry know as “nanotechnology” is the development of macro-scale materials with nano-scale structures. The distinctive feature of nanoscience is the increased understanding and technical control of nanoscale structure and functionality. This is not about new materials but about new processes, new forms, and new functionalities for old materials.

Modern analytical and microscopy tools are allowing us to see, in more detail, the nature of wood fibers down to nano- and atomic scales. We can now appreciate the fact that they are made of nanodimensional components that produce the unique properties of wood. Indeed, paper, paperboard, and other wood-based materials are typically made from a range of components that inevitably have some degree of nanodimensional scale, put together empirically to make valuable performing substrates.

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Nanotechnology In Forest Products

- Traditional manufacturing works from the top down—nanotechnology works from the bottom up, manuipulating molecules to achieve precise and novel effects.
- Nanotechnology is the most promising breakthrough towards production growth since the Internet—some say a **second industrial revolution**.
- Last year alone, 7,000 research papers were published on nanotechnology.
- To reach its goals, the forest products industry must align with the greater nanotechnology research community.

In just a few years, nanotechnology has moved from science fiction into the forefront of research and new product applications. Already, it is considered the most promising breakthrough toward productivity growth since the Internet became part of the workplace. Last year alone, more than 7,000 research papers devoted to nanotechnology were published, and the pace of development is quickening as well.

While predictions vary, it is clear that applications for nanotechnology are closer to reality than the public realizes, and may even qualify as a second industrial revolution. Because self assembled nano-structures require researchers to work at the atomic or molecular scale, it changes the very definitions of raw materials and manufacturing processes. Manufacturing traditionally builds things from the top down, hewing lumber from trees, extracting stone from quarries, assembling computer chips from silicon. Nanotechnology works from the bottom up, manipulating molecules and atoms to achieve precise and novel effects, improving and altering existing materials.

Already, nanotechnology is being incorporated into a variety of products. Computer and cell phone chips have nanoscale circuits; cotton khakis contain nanosized particles that repel stains and water; automobile manufacturers have employed a nano-finish so its cars never need waxing. Nano particles or fibrils are found in stronger automobile bumpers, more effective sunscreens, bouncier tennis balls, and more powerful golf clubs. The National Science Foundation (NSF) predicts that within a decade nanotechnology will provide a $1 trillion market, and provide two million new jobs. Federal research funding will average $1 billion a year over the next four years—one of the largest infusions for industrial R&D since the early days of the space program.

Tomorrow

The vision for the forest products industries is to better utilize all the components that are available in wood and wood-based materials. New methods for liberating these materials, including nanodimensional cellulose fibrils, macromolecules, and nanominerals, will be needed in order to use the techniques developed for other nanomaterials as platforms for creating new wood-based materials and products. Nanotechnology holds the promise of changing virtually all of the processes by which wood and paper products are now made, transforming the sector from a resource-based to a knowledge-based industry with much greater prospects for long-term stability.

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Research

Research is already showing the way to increase performance and add value in a host of traditional forest products sector products. Initiatives to provide greater strength, water resistance, fire-retardancy, and new forms of packaging are showing great promise, as described in the subset of emerging opportunities presented below.

- New methods to produce biodegradable polymers and perform surface/interface modification of wood and pulp fibers could lead to biomaterials with attractive structural and functional properties (e.g., clay nanocomposites).
- New types of adhesives and surface coatings could provide enhanced durability, resistance to moisture and decay, and fire retardancy. Indeed, nanocomposite fire retardant treatments are already available and may be adaptable to wood products.
- Nanosized particles could replace current chemical treatments for preserving wood products with direct impregnation of titanium dioxide, zinc oxide, and other particles shown to improve wood longevity. This will be especially useful given that many countries are already banning current forms of preservative-treated wood.
- Nanocellulose fibrils are the principal structural elements of wood. Understanding how they are organized with other cell wall materials to provide the bulk properties of wood will allow reconstructing traditional wood and wood-based materials into new shapes and applications.
- For paper, paperboard, and composites new polymerization techniques can allow for the synthesis of reinforced fibers compatible with either water or organic liquids. Incorporating biochemical and biomimetic techniques can make these products more recyclable while also improving performance.
- Among the many possibilities suggested to date for new woodfiber-based products incorporating nanomaterials are moisture-resistant cell-phone components, advanced membranes and filters, improved loudspeaker cones, and additives for paints, coatings, and adhesives.

It should be noted that many of these new techniques may draw substantially on the wealth of the advanced chemistry already in use in the industry. As one report concludes, “It should be remembered that substantial parts of the cell wall structure engineered during traditional pulping, bleaching and fiber processing are in the nanometer range.”

Partnerships

Moving ahead in the area of nanotechnology, the forest products industry must seize the opportunity to link with larger nanotechnology research and industrial communities such as the ongoing efforts of the National Nanotechnology Initiative (NNI). The NNI is a visionary R&D program that coordinates the activities of 22 federal agencies and a host of collaborators from academia, industry, and other organizations. The total federal funding investment for the NNI is $988 million in fiscal year 2005 and a request of $1,052 million in fiscal year 2006. The goals of the NNI include:

- Maintain a world class research and development program aimed at realizing the full potential of nanotechnology
- Facilitate transfer of new technologies into products for economic growth, jobs, and other public benefit

Moving ahead in the area of nanotechnology, the forest products industry must seize the opportunity to link with the larger nanotechnology research and industrial communities such as the National Nanotechnology Initiative (NNI).

- Develop educational resources, a skilled workforce, and the supporting infrastructure and tools to advance nanotechnology
- Support responsible development of nanotechnology[2]

By linking with communities such as the NNI, the forest products industry will be able to expand its knowledge of nanotechnology, pool its resources with those of others pursuing common R&D goals, and advance its own agenda towards its short-, mid-, and long-term goals for nanotechnology in the industry.

[2] National Science and Technology Council, Committee on Technology, Subcommittee on Nanoscale Science, Engineering and Technology, *National Nanotechnology Initiative Strategic Plan*, December 2004.

3—R&D Strategy

Research in nanotechnology for the forest products industry will be focused on the fundamental composite material in wood: lignocellulose. Lignocellulose is an abundant material that is nanodimensional at the basic level—these dimensions hold the keys to the ability to develop materials from the bottom up. In addition, many existing products, such as paper and paperboard, have empirically evolved from the use of micro-scale materials, such as microfibers and clay fillers that have turned out to incorporate nanodimensional fibrils and particles. Substantial improvements in performance and economy can be achieved by successfully using these nano-dimensional components more intelligently.

Two Approaches

The R&D strategy for the forest products industry encompasses two approaches:

1) Nanotechnologies developed in the broad NNI effort will be adopted and deployed into materials, processes and products used in or produced by the current forest products industry. The expected gains of this research direction will be in improving processing efficiencies, improving end-use performance of existing products, and some degree of new product development using much of the existing capital infrastructure—with some minor-to-moderate modifications and additions.

2) Develop completely new materials or product platforms using the improved knowledge of nanoscale structures and properties of the materials used in the forest products industry and other industries. This direction potentially will lead to radically different products, processing techniques, and material applications.

R&D Strategy

- NEEDED: Fundamental and cross-cutting research for basic understanding of material properties at the nanoscale.
- NEEDED: New concepts and design methodologies for nanoscale tools and devices.
- Nanotechnology by Design will yield new tools for precisely building material function around end-use application.
- Networking with other interested parties is vital.

TWO APPROACHES—

(1) Incorporate knowledge and technologies developed through the NNI effort into forest products industry materials and processes.

(2) Focus on completely new platforms for radically different products, processing techniques, and applications.

Key Research Challenges

Major scientific and engineering breakthroughs will be required to take advantage of the opportunities that nanoscience offers the forest products industry. The Nanotechnology for the Forest Products Industry Workshop participants identified fundamental research challenges in nanoscale lignocellulosic biopolymer structures, novel surface phenomena, biosynthesis, systems integration, education, and introduction of nanomaterials into the marketplace. The following discussion provides a synthesis and summary of the research challenges and opportunities that were identified at the workshop in the various concurrent breakout sessions.

The research challenges span a range of scientific focus areas including:

- Fundamental Understanding and Analytical Tools
- Nanomaterials by Design
- New Nanoscale Building Tools
- Nanotechnology for Manufacturing

A summary of the major research opportunities identified by workshop participants is shown in the box below.

Summary of Major Research Opportunities

- ☑ Directed design of biopolymer nanocomposites (e.g., combining lignocellulosic materials with other nanomaterials).
- ☑ Use of self-assembly of nanodimensional building blocks to produce functional structures and coatings (can also take advantage of installed industry infrastructure for rapid technology transfer and adoption).
- ☑ Nanoscale architecture from renewable resource biopolymers (e.g., creating novel biopolymers; active functional surfaces; and synergistic coupling of biopolymers with inorganic nanomaterials).
- ☑ Biofarming lignocellulosic nanomaterials with unique multifunctional properties by understanding and exploiting the architecture and ultrastructure of plant cell walls.
- ☑ Liberating nanodimensional cellulose fibrils with a view to exploring the anticipated beneficial properties, for example, cellulose nanofibrils appear to offer very high strength, up to one-quarter the strength of carbon nanotubes.
- ☑ Develop biomimetic processes for synthesizing an array of nanodimensional lignocellulosic materials.
- ☑ Using nanomaterials, nanosensors, and other applications of nanotechnology science and engineering to dramatically improve the efficiency of forest products raw material conversion processes by reducing energy consumption in processing by 50 percent, using up to 60 percent less raw materials per unit of product output, and reducing product degrade/off-specification.
- ☑ Developing, enhancing, and adapting physical, chemical, optical, and electrical property instrumentation and analytical methodologies used in nanotechnology and nanoscience to lignocellulosic biopolymers' unique nanofibrillar and cellular morphology.

Fundamental Understanding and Analytical Tools

Cross-cutting fundamental research that combines biology, physics, chemistry, materials science, computer science, and engineering will be vital.

As an R&D effort organized around the unique properties of lignocellulose and its processing into consumer products, research is needed to develop fundamental understanding of nano-biostructures and processes, nanobiotechnology, and techniques for a broad range of applications in biomaterials, biosystem-based electronics, agriculture, energy, and health. Lignocellulosics are challenging materials insofar as they have an architecture comprised of mixtures crystalline polymeric materials, oriented molecules, and randomly designed components. Analytical techniques being developed in the studies of "soft matter" and nanotechnology will be of great value. But it must be recognized that new approaches will be needed to help elucidate the fundamental structures involved. In this respect it will be necessary to reach out to disciplines previously not strongly linked with forest products. Increasingly, biologists, physicists, chemists, materials scientists, and engineers will need to work together to provide the range of techniques and skills needed. A major resource to be included will be national laboratories, where a number of x-ray, neutron, and advanced-light sources will enable a more detailed analysis of lignocellulosics. Some fundamental areas of research will include:

- Progress in the study of biological and biologically inspired systems in which nanostructures play an important role. This includes developing an understanding of the relationships among chemical composition, single molecule behavior, and physical shape at the nanoscale and in terms of biological function and material properties.
- The study of cell biology and nanostructured tissues, as well as synthesis of nanoscale materials based on the principles of biologically guided self-assembly. Biosynthesis and bioprocessing offer fundamentally new ways to manufacture nanostructured products, including novel biomaterials, improved delivery of bioactive molecules, nanoscale sensory systems, biochips, and the modification of existing biomolecular machines for new functions.
- Genomic modifications of trees or other lignocellulosic feedstock that modify the characteristics of the components in wood to better suit the requirements for processing or end-use products.
- Development of more efficient ways to liberate and stabilize nanodimensional cellulose fibrils so that they can be used most effectively.

Nanomaterials by Design

"Nanomaterials by Design" is a uniquely solutions-based research goal. As described in the nanomaterials roadmap developed by the chemicals industry, "*nanomaterials by design* refers to the ability to employ scientific principles in deliberately creating structures (e.g., size, architecture,) that deliver unique functionality and utility for target applications."[3] This research area will focus on the assembly of building blocks to produce nanomaterials in technically useful forms, such as bulk nanostructured materials, dispersions, composites, and spatially resolved, ordered nanostructures. It will yield a new set of tools that can provide nearly limitless flexibility for precisely building

Nanomaterials by Design

Creating "Nanomaterials by Design" is a uniquely solutions-based goal. It will yield a new set of tools that can provide nearly limitless flexibility for precisely building material function around an end use application.

[3] U. S. Department of Energy and Chemical Industry Vision2020 Technology Partnership, Chemical Industry R&D Roadmap for Nanomaterials by Design: From Fundamentals to Function, December 2003.

material function around an end-use application. Such a powerful, function-based design capability holds the potential to solve critical, unmet needs throughout society. Techniques being developed in the areas of self-assembly and directed self-assembly will allow us to use the building blocks available in the forest products industry to manufacture materials with radically different performance properties.

New Nanoscale Building Tools

Novel concepts and design methodologies are needed to create new nanoscale devices and integrate them into architectures for various operational environments. These require a profound understanding of the physical, chemical, and biological interactions among nanoscale components. Research in this area includes development of new tools for sensing, assembling, processing, manipulating, and manufacturing. It will also require integration along scales, controlling and testing of nanostructures and devices, design and architecture of concepts, software specialized for nanosystems, and design automation tools for assembling systems containing large numbers of heterogeneous nanocomponents.

Novel concepts and design methodologies are needed to create new nanoscale devices.

Manufacturing at the Nanoscale

Research in this area will focus on creating nanostructures and assembling them into nanosystems and then into larger-scale structures. This research should address understanding nanoscale processes, developing novel tools for measurement and manufacturing at the nanoscale, developing novel concepts for high-rate liberation and stabilization of nanoscale building blocks, and understanding the processing of nanostructures and nanosystems, as well as the scale up of nanoscale processing methods.

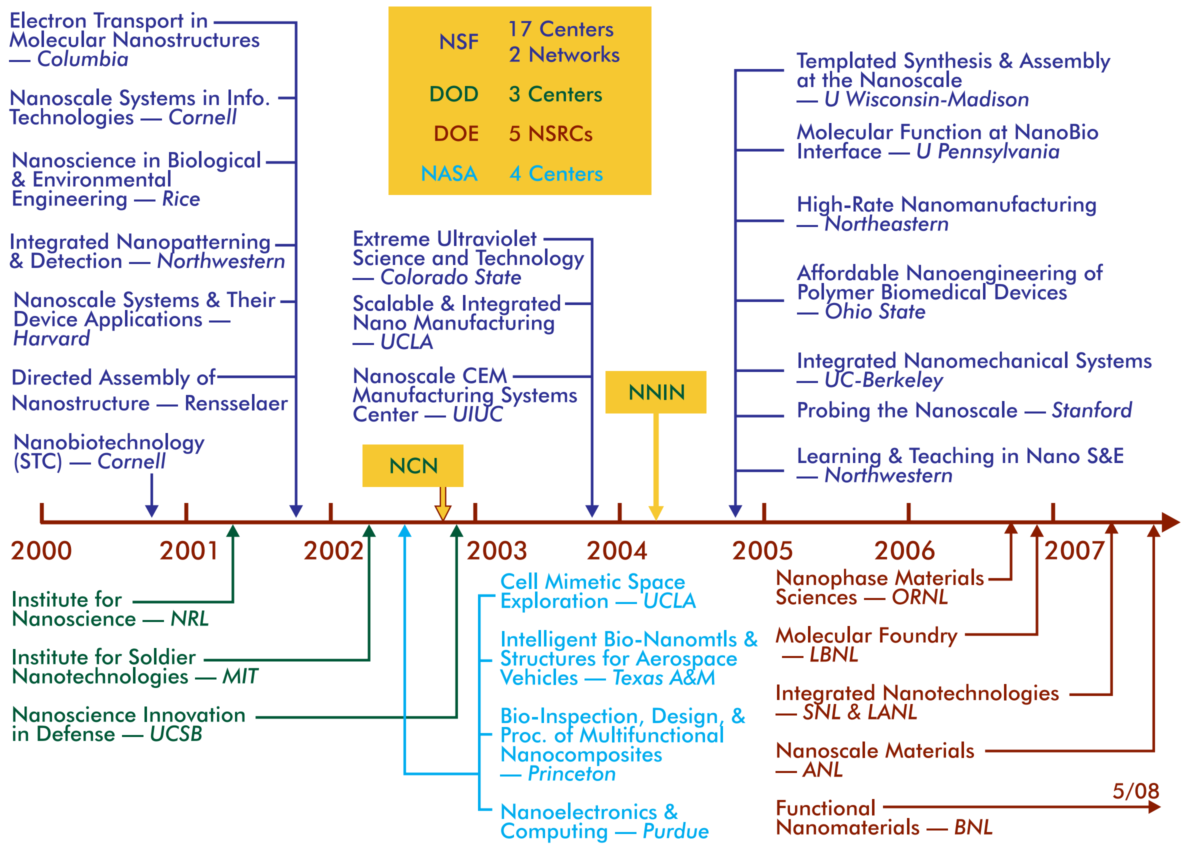
Fostering Networks and Collaboration

To make the vision a reality, it will be necessary to facilitate intensive coordination and integration among interdependent and multidisciplinary research areas. A Steering Group could be used to foster this coordination and guide research along the lines identified by this and future roadmaps. More importantly, it will be vital to network with centers already funded as part of the NNI (Figure 7) so that full advantage can be taken of the investments in facilities and programs already under way. Examples include the nanofabrication facility at Pennsylvania State University, where researchers are investigating the production of cellulose nanofibrils, the self-assembly group at Harvard, where basic principles are being developed, and the nanocomposites groups at Rensselaer Polytechnic Institute and Wright Patterson Air Force Base. In each case, there are existing bodies of knowledge that can be leveraged by the forest products industry. Networks will include the R&D User Centers established by the U. S. Department of Energy, the National Science Foundation (NSF) and the National Institute of Standards and Technology. These facilities, such as those provided by the NSF's National Nanofabrication Infrastructure Network (NNIN) (Figure 8), make staff, facilities, and equipment necessary for nanoscale research accessible to researchers at businesses and academic institutions around the country.

Networking with existing NNI centers is vital.

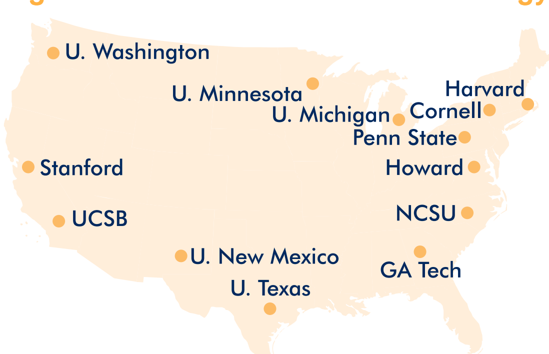
Figure 7. NNI Centers and User Facilities

The NNI has developed a substantial network of research centers, user facilities, and related infrastructure throughout the United States. Since the inception of the NNI in 2001, NSF, DOD, and NASA have established 24 research centers. Five DOE user facilities are under construction, with interim operational capability online already at some of these facilities. NSF has established a 13-university network of user facilities, the National Nanotechnology Infrastructure Network (NNIN), as well as the 7-university Nanotechnology Computational Network (NCN). In 2004, the total number of users of both the NCN and NNIN facilities and expertise exceeded 6300.



Source: National Science and Technology Council 2004

Figure 8. National Nanotechnology Infrastructure Network



The National Nanotechnology Infrastructure Network (NNIN) provides provides open on-site and remote access to teaching tools and instrumentation as well as capabilities for fabrication, synthesis, charaterization, design, simulation, and integration to users in academia, small and large industry, and government. The NNIN also has extensive education, training, and outreach capabilities.

R&D Focus Areas

The Steering Committee for the *Nanotechnology for the Forest Products Industry Workshop* considered a number of different options for organizing the technical focus areas for the breakout discussion sessions. The following five R&D focus areas were selected on the basis that they 1) provide the best path forward for a nanotechnology roadmap by helping to identify the underlying science and technology needed, and 2) foster essential interactions among visionary, interdisciplinary research and technology leaders from industry, academia, research institutions, and government.

1) **Polymer Composites and Nano-reinforced Materials**—Combining wood-based materials with nanoscale materials to develop new or improved composite materials with unique multifunctional properties.

2) **Self-Assembly and Biomimetics**—Using the natural systems of woody plants as either the source of inspiration or template for developing or manipulating unique nano-, micro-, and macro-scale polymer composites via biomimicry and/or direct assembly of molecules.

3) **Cell Wall Nanostructure**—Manipulating the cell wall nanostructure of woody plants in order to modify or enhance their physical properties and create wood and wood fibers with superior manufacturability or end-use performance.

4) **Nanotechnology in Sensors, Processing, and Process Control**—Using non-obtrusive, nanoscale sensors for monitoring and control during wood and wood-based materials processing, to provide data on product performance and environmental conditions during end use service, and to impart multifunctional capabilities to products.

5) **Analytical Methods for Nanostructure Characterization**—Adapting existing analytical tools or creating new tools (chemical, mechanical, electrical, optical, magnetic) that accurately and reproducibly measure and characterize the complex nanoscale architecture and composition of wood and wood-based lignocellulosic materials.

A sixth focus area is also included here as a part of the R&D Strategy: **Collaboration in Advancing Programs and Conducting Research.** This section emphasizes the importance of collaboration and cooperation among researchers from various disciplines and organizations, including universities, research institutes, National Laboratories, and government agencies and departments.

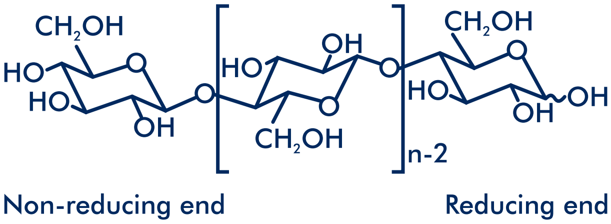
The following sections describe the R&D focus areas, including the key technical challenges and research priorities.

R&D Focus Area 1
Polymer Composites and Nano-Reinforced Materials

Description

Wood can be viewed as a polymeric composite of cellulose, hemicelluloses, protein, and lignin, as well as a composite of nanofibrils, microfibers, tracheids, vessel elements, and parenchyma cells (See Figures 8-11). In addition, wood and wood-based materials, such as glued laminated beams, oriented strandboard, plywood, medium density fiberboard, hardboard, paper, and paperboard, are increasingly being manufactured as composites of non-wood and wood-based materials. Non-wood materials, used as films, fillers, and matrices, include a wide array of materials such as clays, calcium carbonate, waxes, high-density polyethylene, titanium dioxide, adhesives, resins, Portland cement, polypropylene, polyethylene terephtalate, et cetera.

Figure 8. The Chemical Structure of Cellulose



Source: Fibersource (www.fibersource.com/f-tutor/cellulose.htm)

Figure 9. Cellulose Nanofibrils Within a Plant Cell Wall

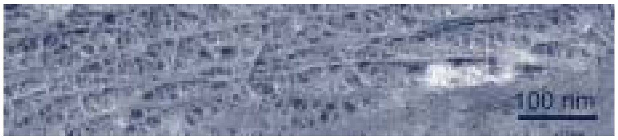
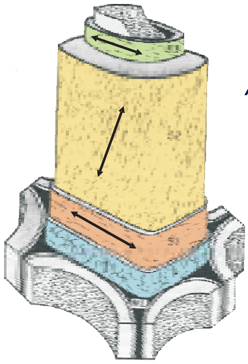


Image courtesy of Candace Haigler and Mark Grimson, North Carolina State University, Raleigh, NC

Figure 10. Diagram of a Wood Fiber



Arrows indicate the orientation of cellulose microfibrils in the different layers of the secondary wall.

Adapted from Smook 1992

Figure 11. Assemblies of Tracheids, Ray Cells, and Parenchyma Cells in Wood

Images courtesy of W.C. Brow Center, State University of New York

The availability of new nanomaterials offers the forest products industry the opportunity to improve its existing composite products and create new high-value and high-performance composite products. High-performance, nano-based chemistry and additives for coatings, for example, are producing surface enhancements and providing more flexible and effective use of valuable raw materials. The development and use of nanomaterials and nanotechnology offers forest products producers opportunities for reduced material and energy inputs; added functionality to wood, wood-based composites, and pulp, paper, and paperboard products; and improved process efficiency—all of which will help secure the sustainability and viability of the forest products industry.

R&D Focus Area 1 - continued
Polymer Composites and Nano-Reinforced Materials

Research Goal and Objectives

The overall goal of this focus area is to utilize—via adapting, developing, measuring, and implementing—a wide array of nanomaterials and nanotechnologies that will 1) improve the end-use performance of current wood, wood-based composites, pulp, paper, and paperboard products; 2) allow development of new generations of high-value, high-performance products from forest-based materials; and 3) reduce the overall costs to manufacture wood, wood-based composites, pulp, paper, and paperboard products.

The goal is to develop the capability to use a wide array of nanomaterials that improve end-use performance of current wood-based products, allow development of new generations of high-performance products, and reduce manufacturing costs.

Individual objectives include:

- Utilize, develop, and investigate novel wood-based and non-wood-based nanoscale materials with enhanced properties (e.g. films, coatings, fillers, matrices, pigments, additives, and fibers)
- Determine the physical, chemical, mechanical, optical, magnetic, and electronic properties of nanoscale lignocellulosic structures and lignocellulosic nanofibrils in wood
- Liberate nanodimensional cellulose fibrils from the lignocellulosic matrix existing in wood
- Investigate the ability of wood nanofibrils to be converted into carbon nanotubes, nanotubules, and nanowires
- Understand the inter-relationships between lignocellulosic nanoscale material characteristics and resulting product end-use property improvement
- Utilize, develop, and investigate use of nanoscale materials and nanotechnology to improve conversion efficiencies of wood and wood-based materials to final products such as by reducing raw material needs and process energy consumption

Outcomes & Impacts

The following examples, while by no means all-inclusive, illustrate some of the potential applications of successful R&D in this area.

1) Value-added, durable products and products with improved properties that would benefit the consumer (for example, the addition of nanoscale fillers to polymeric materials yields dramatic strength increases even at low addition levels [<5 percent]. If strength properties of wood, wood composites, paper, and paperboard could be dramatically improved, the material content of products could be decreased by up to 60 percent without losing end-use performance).

2) Improved water removal processes and other processing efficiencies in paper products nanotechnology that would lower energy costs by 50% (and, thus enhance environmental sustainability). Water removal (typically accomplished via thermal drying process) usually constitutes the biggest portion of the energy costs required to produce wood, wood-based composites, pulp, paper, and paperboard products. Modification of water absorption leading to faster draining fibers and 100 percent solid coatings would minimize the energy

R&D Focus Area 1 - continued
Polymer Composites and Nano-Reinforced Materials

required and maximize the productivity of paper and wood products converting and finishing processes.

Key Research Challenges

The primary barriers to achieving the goals of the Polymer Composites and Nano-Reinforced Materials focus area can be grouped into three categories: technical, organizational, and behavioral.

1) *Technical*—The number of nanoscale materials currently available in the marketplace capable of providing the desired properties or benefits to the forest products industry is limited. Also, there is generally a lack of experience within the forest products industry with the methodologies needed to characterize and develop nanomaterials with beneficial properties.

2) *Organizational*—Cross-disciplinary teams of scientists and technologists are needed, along with research funding and facilities, to accomplish the goals.

3) *Behavioral*—Many forest products companies are unwilling to participate in anything but pre-competitive research directives; therefore, research projects must be selected carefully. Also, because of the nature of the industry (high-volume production, low-profit-per-ton, and capital-intensive, large-scale equipment and facilities), sufficient economic and technical feasibility studies will have to be performed before any new technologies are implemented.

Cross-disciplinary teams of researchers are required to carry out needed research most efficiently and effectively.

R&D Priorities

Novel Materials

Develop and investigate novel materials with enhanced properties (e.g., films, coatings, fillers, matrices, pigments, additives, and fibers—especially lignocellulosic nanofibrils).

This research would be both fundamental and developmental in nature. Cross-disciplinary teams of material scientists, biological scientists, polymer chemists, paper scientists, forest products technologists, and chemical and mechanical engineers would need to share their expertise and expand the knowledge base of the development group. A range of advanced tools including particle characterization, electrokinetic properties, high-power microscopes, surface spectroscopy, pressure reactors, small-angle X-ray scattering, and high-consistency mixers would be needed to perform this research. It is estimated that it would take 3 to 10 years to develop and characterize beneficial materials. The work would best be accomplished at or among institutions capable of forming cross-disciplinary teams and in possession of the majority of the needed equipment. Cross-disciplinary teams would need to work with industrial partners and a variety of national research laboratories to further expand their capabilities.

R&D Focus Area 1 - continued
Polymer Composites and Nano-Reinforced Materials

Novel Materials for Processing Equipment

Develop and investigate novel materials for processing equipment.

This research would be of both a fundamental and developmental nature. It would require materials scientists, polymer chemists, paper scientists, forest products technologists, and chemical and mechanical engineers to form cross-disciplinary teams that would work with equipment and other suppliers on novel materials for processing equipment. For example, pilot-paper machine and coating equipment could be adapted to implement the new processes equipment that would be needed for performing the studies. This would be shorter-term research focused on studying the operational and economic benefits of advanced nanomaterials on wood processing, panel products composites manufacturing, and paper-making and coating equipment.

Inter-Relationships Between Nanoscale Material Characteristics and End-Product Properties

Develop and understand the inter-relationships between nanoscale material characteristics and the resulting product end-use property improvements.

This research focuses on determining the inter-relationships between nanoscale material characteristics and resulting product end-use property improvements. As nanoscale materials become increasingly used in forest products to attain new and improved end use performance, inclusion of these materials needs to move away from iterative trial and error product development approaches where such nanoscale materials are included in test sample products followed by assessing resulting product properties. If one can create a database of nanoscale material properties and elucidate the relationships between constituent nanoscale material properties and product end-use properties, one can employ scientific principles and computational modeling in deliberately creating new and improved products that deliver unique functionality and utility for targeted end-use applications without the need to go through iterative trial and error product development experimentation.

This nanomaterials database, constituent material interaction, and end-product performance modeling research would be carried out by researchers with backgrounds and skills in forest products technology, pulping and papermaking science and engineering, chemical and mechanical engineering, materials science, mathematical modeling, computational modeling, and metrology. The work would require the use of a laboratory equipped with up-to-date physical-property, surface-testing, and other metrology equipment. Small-scale pilot-plant and wet laboratories would be needed to create composite end products for testing and evaluation. For example, coating labs and printing facilities would be needed to generate samples for testing coating and performing paper-print testing and analysis.

Implementing New Materials

Determine the best way to implement new materials.

This research would be more developmental and applied in nature. It would require the formation of cross-disciplinary teams of material scientists, forest products technologists, paper scientists, and chemical and mechanical engineers. The research teams could work with cooperators at universities, federal laboratories, or commercial pilot-plant facilities equipped

R&D Focus Area 1 - continued
Polymer Composites and Nano-Reinforced Materials

with appropriate pilot equipment (e.g., paper machines, coaters, formers, presses, and auxiliary equipment) to determine the best way to implement the nanomaterials being developed by other cross-disciplinary teams. If equipment modifications are not needed, the research could be accomplished in a shorter period of time. However, if significant modifications to existing equipment need to be made, projects may extend into a longer lifetime.

Economic and Life-Cycle Models

Develop economic and life-cycle models for forest-based nanoscale materials and products.

To be successfully used in consumer products and end uses, wood-based products and materials, including nanoscale materials, must be technically and economically viable as well as socially and environmentally acceptable. The vast majority of work identified in this forest products industry roadmap is justifiably focused on technical issues and overcoming technical barriers. However, research to assess and model impacts and the economic viability of using nanoscale materials in forest products is also needed. Such research will help identify the most critical areas in which to focus research to reduce costs, attain needed end-use performance characteristics, and overcome any negative environmental impacts in production and use. Life-cycle assessments are increasingly being used to assess and quantify the environmental impacts of materials and products and, hence, contribute to achieving social acceptability. Because success is expected with respect to use of nanotechnology and nanoscale materials within the forest products industry, it is necessary that research on life-cycle assessment and determining and overcoming any unacceptable environmental impacts needs to be carried out. Interdisciplinary teams comprised of forest products technologists, pulp and paper scientists and engineers, business majors (including Masters of Business Administration), economists, chemical engineers, mechanical engineers, and environmental engineers would best perform this research. The interdisciplinary teams should work with such groups as the National Council for Air and Stream Improvement (NCASI) and The American Forest and Paper Association (AF&PA) to develop economic and life-cycle models for nanomaterial-containing products.

R&D Focus Area 2
Self-Assembly and Biomimetics

Description

Much can be learned by studying the naturally occurring nanostructures found in forest biomass. Learning how they self-assemble and developing methods that use this self-assembly will be critical to manufacturing new products from this renewable resource. Through biomimicry, we will also be able to take advantage of the efficiencies of these natural structures.

Learning how woody plants self-assemble and developing methods that use self-assembly is critical to developing novel nanoscale lignocellulosic-based biomaterials.

The major constituents of woody materials are cellulose, lignin, and the hemicelluloses. Of these, cellulose is by far the most predominant and is, in fact, the world's most abundant polymer. Cellulose is a renewable resource, and has many unique properties that result from its organization at a supermolecular level. Under the guidance of the cell, the cellulose chains self-assemble into partly crystalline nanofibrils within the plant cell wall. Deviations from perfect crystallinity exist and, rather than being truly amorphous, probably reflect the complex nanoscale structure of the biopolymer that is, as yet, not completely understood. These nanofibrils impart specific properties to the composite structure based on the way they are formed and are oriented within the fiber cell wall. By understanding and influencing how these structures form, and by developing practical manufacturing processes to manipulate their formation, it may be possible to develop entirely new products that take advantage of the material characteristics manifested at this scale. These material characteristics include unique optical, strength, electrical, and sorptive properties.

Research Goal and Objectives

The overall goal of this focus area is to develop a technical platform that enables self-assembly of lignocellulosic materials either singly or in combination with other materials at a nanoscale. Individual objectives will include the following:

- Create novel, functional, self-assembling surfaces on existing lignocellulosic substrates
- Develop a fundamental understanding of molecular recognition in plant growth and cell-wall self-assembly in forest products processes to create new or to enhance existing products
- Characterize self-assembled natural and synthetic materials
- Integrate micro- and nanoscale organization in new products and processes

Outcomes and Impacts

The following examples, while not all-inclusive, illustrate some of the potential applications of successful R&D in this area.

1) Barrier coatings and films/laminates for use in packaging to protect components, indicate the condition of the contents, or provide a security function.

2) Extremely light-weight, paper-like structures that not only significantly reduce the weight of existing paper products, but enable entirely new uses for fibrous webs (e.g., matrices for other polymers or ceramic materials).

R&D Focus Area 2 - continued
Self-Assembly and Biomimetics

3) An understanding of how molecules in woody biomass self-assemble leading to the use of the constituents as a chemical feedstock (as an alternative to non-renewable chemical raw materials such as petroleum).

4) By modeling the "nanofactories" found in the leaves and other living parts of the woody plant, we may be able to mimic processes such as photosynthesis and transpiration. This may enable development of more efficient methods for manufacturing foodstuffs and fuels from forest resources, as well as the production of simple and/or composite structures for the controlled passage and separation of various materials.

Key Research Challenges

The primary barriers to achieving the goals of the Self-Assembly and Biomimetics focus area can be grouped into four categories—manufacturing, materials, analysis, and nanoscience.

1) *Manufacturing*—Manufacturing in the forest products industry is characterized by large, high-throughput processes that are very capital-intensive. A major barrier is the difficulty of large-scale and high-speed production of nanomaterials.

2) *Materials*—Biomass as a starting material tends to be very non-homogeneous, which may prove to be a significant barrier to complete resolution followed by self-assembly of specific components of this resource.

3) *Analysis*—The forest products industry's ability to predict bulk material properties from single and assemblies of nanostructures is limited by the analytical methods currently available. Some may exist in other industries but are not used in forest products.

4) *Nanoscience*—Little knowledge exists regarding the self-assembly and incorporation of useful molecules into nanomaterials derived from the forest resource base.

R&D Priorities

A fundamental approach will accelerate the development of forest-based nanomaterials and new products incorporating nanomaterials. To do this, an in-depth understanding of the principles of self-assembly in these materials must be developed. To develop a mechanistic understanding of self-assembly, extensive knowledge bases in many scientific disciplines, new measuring techniques, modeling and the correlation of nano, micro-, and macro-scale properties must be developed. Key R&D priorities as described below.

Manufacturing

Optimize development and product life cycles in manufacturing.

This will require methods that make maximum use of current manufacturing systems and meet the production-rate requirements of the industries. To do this, the thermodynamics and kinetics of self-assembly must be quantified and modified and the macro- and nanoscales during processing will need to be reconciled.

Materials

Develop methods to neutralize the impact of the heterogeneous nature of the starting material.

R&D Focus Area 2 - continued
Self-Assembly and Biomimetics

This may include methods that are insensitive to non-homogeneity or that incorporate homogenization of the starting material as a first step.

Analysis

Develop data bases sufficiently large to permit modeling.

To do this structure/property relationships will have to be developed for lignocellulosic materials. High throughput analytical methods will also have to be developed. This will require multidisciplinary solutions. In addition, we will need to correlate what happens at the micro- and nanoscales to the macro scale.

New measurement techniques, modeling, and the ability to correlate nano- and micro-scales are needed.

Nanoscience

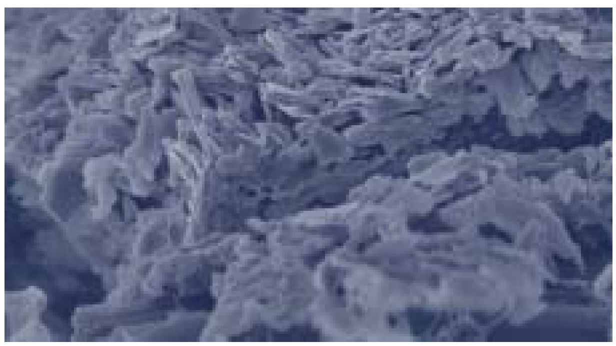
Establish/expand the discipline of nanoscience as it pertains to lignocellulosic materials, which today is virtually non-existent.

Developing this area will require multi-disciplinary partnerships. Examples include areas such as new measurement techniques (e.g., for particle/particle interactions), modeling capabilities (quantify the effects of surface chemistry and shape factors), and multi-scale self-assembly.

Linkages and Implementation

New measurement techniques, modeling, and the ability to correlate nano- and macro-scales are needed.

To gain a mechanistic understanding of self-assembly and biomimicry, fundamental knowledge will have to be developed in basic disciplines such as biology, biochemistry, biophysics, polymer science, surface and colloid science, thermodynamics, kinetics, and organic materials science. This will require the virtual creation of a new multidisciplinary field as it relates to the forest resource. Specialized equipment and analytical capabilities currently used in other fields and even the development of new techniques and instrumentation specific to this area will be needed. Long-term cooperative efforts between academia, research institutions, and the network of nanotechnology laboratories (currently under development) will also be needed.



An electron microscope cross section image of a paper coating

Image courtesy of IMERYS

R&D Focus Area 3
Cell Wall Nanotechnology

Description

Research activities in the Cell Wall Nanotechnology focus area seek to understand and exploit the architecture and processes of consolidation of wood cell walls, which are the primary determinants of the material properties of wood and wood fibers. Wood cell walls are nanocomposites of cellulose, hemicelluloses, protein, and lignin. They form the basis of the forest products industry and its renewable resources. Wood cells contain unique nanomanufacturing protein complexes that use activated glucose to assemble cellulose nanofibrils, the most abundant renewable material resource on earth. These remarkable cellulose nanofibrils exhibit a modulus roughly one quarter to one fifth of that of a carbon nanotube, yet they are produced naturally without the need for energy-consuming, high-temperature processing.

Manipulating the architecture and processes of consolidation of wood cell walls is key to achieving superior physical properties at nano-, micro-, and macro-scales.

Recent studies of interactions between the major cell wall constituents, together with evidence that the assembly of the cell wall is genetically encoded, suggest that the formation of cell walls involves highly orchestrated nanoprocesses. Research illustrates that the molecular and nanoarchitectural details of these cell walls differ between cell types and tissues and species. At the present time, our understanding of the native structures, their molecular diversity, and the mechanisms of biogenesis and hierarchical assembly in woody plant cell walls is very rudimentary.

Improved understanding of the essential nature of the plant cell wall and the nanoprocesses involved in its formation converges with emerging nanoscience, bringing about new challenges and opportunities. New information and insight concerning nanoscale order and assembly of the lignocellulosic cell walls is needed. This knowledge will: (a) advance opportunities to control structure within the genetically engineered plants; (b) suggest new uses of wood and its constituents individually; and (c) provide system prototypes for biomimetic nanoscale engineering processes to produce materials in entirely new ways. It is possible to imagine material production processes that use proteins directly, or the as-yet-unknown protein operation mechanisms, allowing fibrils and composite materials to be manufactured and engineered at the nanoscale outside the cell. Such approaches may dramatically change the manufacturing of wood products by reducing energy consumption, eliminating the delignification process, and conferring the ability to industrially engineer material properties on the nanoscale. Ultimately, research into these areas will benefit the public through value-added traditional products, novel manufacturing processes, and innovative products, as well as optimized plantation forests with minimized impact on natural ecosystems.

The goal is to develop the capacity to manipulate the nanoarchitecture of secondary walls of woody plants.

Research Goal and Objectives

The overall goal of this focus area is to develop the ability to modify the nanoarchitecture of the secondary walls of woody plants to achieve significant improvements in properties, and to adapt these properties to different applications.

R&D Focus Area 3 - continued
Cell Wall Nanotechnology

Achieving this goal depends on controlling the processes of cell wall assembly through genetic and environmental manipulation. To accomplish this it will be necessary to achieve a deep and integrated understanding of the complex and highly coupled processes responsible for consolidation of the cell walls and how these processes vary in different species and tissues and under different environmental growth conditions. New methods for investigating the constituents and separating them surgically at the nanoscale need to be developed. Finally, it will be necessary to take advantage of all the new instrumental and technical methods available for investigating nanostructures as they occur in their native conditions without disruption. Ultimately, genetics, cell biology, biochemistry, biophysics, and materials engineering, must be linked in order to establish causal relationships between molecular phenomena and the diverse structures and mechanical properties of plant cell walls at the nanoscale.

Specific objectives will include but are not limited to:

- Characterize the consolidated cell wall structure without resorting to reductionist methods that result in the loss of information regarding the nature of individual constituents or the coupling of the different constituents
- Establish the relationship between genetic information and its phenotypic expression at the nanoscale level with regard to mechanisms of biosynthesis and the structure and organization of the constituents of the cell wall
- Identify the influence of environmental conditions on native cell wall properties and stabilizing properties under variable environmental conditions
- Establish a more solid foundation for relating the genomic information from *Arabidopsis thaliana* to corresponding genomic information for commercially important species of wood
- Develop new methods of investigating cell wall structure by non-invasive microscopic and spectroscopic measurements that do not require isolation of the individual components in order to acquire information concerning structure and mechanical properties
- Develop new techniques for separating the cell wall constituents without altering the native structures
- Develop methods to economically extract lignin without substantially altering its structure

Outcomes and Impacts

The following examples, while not all-inclusive, illustrate some of the potential applications of successful R&D in this area.

1) Improved product diversity and properties through engineering of wood feed stock to meet product-specific requirements (e.g., generation of thinner walls in southern pine, changed ratio of wall components, more flexible fiber, more or less absorptive fiber).

2) Enhanced use of non-merchantable timber, wood processing residues, and other agricultural residues as sources of biomass to compensate for declining fossil fuels.

3) Improved ecological sustainability through reduced energy costs to extract lignin (currently accounts for one half of paper manufacturing costs), minimization

R&D Focus Area 3 - continued
Cell Wall Nanotechnology

of undesirable chemical byproducts in wood processing, and faster growing trees that retain industrially useful properties to maximize productivity of plantation forest land.

4) New methods for extracting and isolating the lignin and hemicelluloses in a form that is marketable (may have unique properties for applications in the industrial markets for phenolic-based polymers and polysaccharides).

5) Improved economic vitality of local industry and job creation through innovative, high-value processing and fabrication of wood and wood-based products.

Key Research Challenges

The most challenging part of what lies ahead is to establish new paradigms to enable examination of wood cell wall structures at the nanoscale that will be meaningful to the wood science community. The wood science community has historically organized its foundational knowledge at the microscale and the molecular scale, with only speculative models to bridge the gap. With the development of vastly more powerful instruments for examining structure at the nanoscale level, it is now necessary to discard the speculative models and establish new paradigms that are validated by experimental observations. Ultimately, we must obtain a comprehensive view of the chemical, mechanical, microscopic, and crystallographic nature of cell wall structure in the native state and during processing.

Some of the specific challenges to be addressed by research in the Cell Wall Nanotechnology focus area are described below.

1) The speculative models that have been in frequent use over recent decades will not prove useful—they are based on adaptation of many of the paradigms developed in studies of synthetic polymers.

2) Many phenomena that can occur at the nanoscale level do not lend themselves to analysis in terms of the concepts of the classical thermodynamics of macroscopic systems (e.g., the aggregation of cellulose in the native state).

3) Traditional computer modeling of the atomic scale does not readily deal with molecular and fibrillar interactions at the nanoscale. Novel nanoscale modeling methods for fibrous composites need to be developed as a means of understanding native cell wall structure, alterations during processing, and conceptualization of value-added materials.

4) Because cellulose has a predominant importance in the forest products industry and is the skeleton around which other cell wall constituents are organized, it becomes important to understand the processes by which the cellulose is formed.

5) It is equally important to understand how the processes of formation and assembly of the other cell wall constituents are coupled with the deposition of the cellulose to form the hierarchically more complex sub-layers of the cell wall.

6) Knowledge of changes in composition and structure will not necessarily lead to new value added products unless it is known how these changes alter the physical and mechanical properties of cell walls. This relationship of chemical and

R&D Focus Area 3 - continued
Cell Wall Nanotechnology

structural changes to mechanical properties can lead to development of improved wood and fiber products.

R&D Priorities

Regulating Cellulose Nanofibril Formation

Investigate the process of formation of cellulose nanofibrils, including genetic, biochemical, cellular, and biophysical regulation.

Here we include both the primary synthesis of the molecules and the process that brings about their nucleation into a unique, unusually stable nanofibrillar form that has rarely been duplicated in a non-biotic environment. Cellulose biogenesis is effectively a nanomanufacturing process accomplished by a multi-protein complex that is highly integrated into the cell structure. We need to identify all the essential proteins and, their mechanistic interaction, the regulatory mechanisms for the protein complex, and how it is influenced by environmental factors. We need to accurately characterize the diverse nanoscale structures of native cellulose and determine how variability is controlled. A number of well-characterized model systems exist that can continue to be the subjects of biological research in this area as well as in understanding the biophysics of fibril formation. Ongoing research to synthesize cellulose *in vitro* from isolated cellular constituents should be extended to characterization of all the constituents of the active complex and reconstituting it outside the cell solely from identified components. In addition, efforts have begun toward generating dynamic computer models of an active cellulose synthesizing complex. Such efforts will be aided by development of methods for theoretical protein modeling at the nanoscale that do not depend on solved crystallographic structure and higher resolution imaging of the native cellulose synthesizing complex.

Regulating the Synthesis of Other Cell Wall Constituents

Characterize the processes that regulate the formation of the other constituents of the cell wall and the manner in which they are coupled with the deposition of cellulose.

It is important to identify and understand the enzymes and cellular processes that control the synthesis and interaction of hemicelluloses and lignin. To aid this research, systems need to be developed allowing heterologous expression or reconstitution of polymer-synthesizing systems so that functions of altered genes can be rapidly tested. Important questions include identification of where in the cytoplasm, plasma membrane, or exoplasmic/cell wall space is each enzyme system found and determination of the shape, surface area, degree of aggregation and/or crystallinity for all the polymeric components in addition to understanding their interaction. Potential practical benefits of modifying existing constituents or of adding nanomaterials into the cell wall to generate novel and useful composite properties should also be explored.

R&D Focus Area 3 - continued
Cell Wall Nanotechnology

Assembling and Consolidating the Woody Cell Wall

Determine the manner in which the processes of assembly and consolidation are guided by the expression of genomic information, the biophysical interactions of the synthesized molecules, and the emerging mechanical properties.

The impact the composite structure of the cell wall and its components have on the mechanical properties of the plant cell and fiber material obtained from woody plants is important for many applications. Fundamental questions include how the additional polymeric constituents influence the aggregation of cellulose and the formation of the plant cell wall. It is clear that cell wall consolidation involves the transformation of components that are first deposited in a highly hydrated environment into a very tightly aggregated environment within which the degree of hydration is controlled by the characteristics of the consolidated structure. It is important to determine how this process is regulated allowing it to be highly predictable within particular cell types and species yet also subject to environmental regulation. Other questions include how the native properties of the cell wall are changed during industrial processing, and if this can be more advantageously controlled. Moreover, an understanding of the interaction of additives such as adhesives used in wood products with the cell wall structure at the nanoscale could allow the properties of wood products to be improved.

For understanding the consolidated structure of cell walls in the native state or in mutated form, new tools are needed. Some of these are already under development (e.g., recombinant fungal hydrolases against diverse plant cell wall polysaccharides are being characterized, and monoclonal antibodies are being generated that recognize a greater diversity of carbohydrate epitopes). Development of computer models for nanoscale interactions of fibrous components and for nanoscale mechanical properties will also help to advance this research area.

Developing Efficient Experimental Systems

Exploit appropriate model systems to rapidly accumulate fundamental knowledge.

For genomic studies, the model plant *Arabidopsis thaliana*, which has a close evolutionary relationship to Poplar, is a valuable tool because it has secondary cell walls with nanoscale structure that parallels commercially important woody species. Research has already identified induced mutations in Arabidopsis that cause alterations in secondary walls, particularly the cellulose and lignin components. Equally important is the genomic characterization of model tree species and the development of widely accessible methods for rapid testing of gene function directly in transgenic trees. Genomic analysis must ultimately be resolved to cellular understanding of the roles of the encoded proteins, and here more unusual model systems like the cellulose-synthesizing moss *Physcomitella patens* may have particular value. Other model systems involving bacteria may also be useful for rapid testing and provide additional insight into cellulose synthesis. For example, the bacteria *Acetobacter xylinum* produces large quantities of cellulose and has been used extensively to study cellulose biosynthesis.

Applying Novel Instrumental Methods

Apply new instrumental methods to study the cell wall native state without significantly altering its structures.

R&D Focus Area 3 - continued
Cell Wall Nanotechnology

Much remains to be accomplished in this area. Many of the instrumental methods developed to date have been for investigations of inanimate structures and inorganic systems. Methodologies that are more sophisticated than those currently used in the wood science community will be needed to isolate the different constituents in as close to their native state as possible and for examine the nanoscale structure of non-disrupted cell walls. In some cases, this may require the development of new instruments or measurement techniques that avoid the reductionist approaches used in the well-established methods of wood chemistry. Even more far-reaching possibilities include nanodevices fabricated to search for and report interactions between molecules in normal and altered cell walls. Such devices could possibly be engineered to allow high throughput analysis of cell wall structure and properties, including intact plants. These capabilities would significantly improve our understanding of cellulose fibril synthesis and the formation of the plant cell wall. Understandings of cell wall mechanical properties can be investigated via use of nanoindentation.

Developing New Nanocomposites

Develop cell walls as models and materials for nanoscale assembly of new composites.

As we understand the subtleties of assembly of cell walls at the nanoscale level, we need to be alert to the possibilities that some of these processes may lend themselves to scaling to a higher level, as well as to the possibilities that they may provide models for nanoscale assembly of other materials, whether synthetic or natural. That is, they may provide excellent models for the development of new nanocomposites with unusual properties. The understanding of the physical and chemical properties of the cell wall constituents may also provide a path for engineering materials with new bulk or surface properties needed for emerging applications. Examples include sensors, packaging materials, biocompatible or anti-microbial materials, or substrates whose surface properties have been tailored for compatibility with other electronic or optoelectronic materials or devices. It is known that cellulose fibrils exhibit piezoelectric properties. Piezoelectric materials are widely used for physical, chemical, and biological sensing devices. More fundamentally, cell wall molecules could possibly be used as nanobioelectronic devices. An understanding of the properties and assembly of cell wall molecules may enable the realization of these functionalities.

R&D Focus Area 4
Nanotechnology in Sensors, Processing, and Process Control

Description

The ability to monitor the environment and conditions occurring during the manufacturing and use of wood-based products will help lower production cost and add greatly to the functional value of goods and services. Access to information could be greatly expanded through the availability of non-obtrusive sensors that are small and affordable. It should be possible to take advantage of what is being developed in other areas for use in the forest products arena. Additionally, we can anticipate that the deeper understanding of the properties of lignocellulosic materials at the nanoscale will reveal properties or materials that can be incorporated into sensors or used as part of a network communicating local conditions.

The ability to efficiently and effectively use nanoscale sensors to monitor processing and end-use conditions will reap enormous benefits and cost reductions.

Nanostructured catalysts could be used in processing to efficiently and effectively disassemble wood into its various components for optimal downstream processing. For example nanostructured catalysts could be used to selectively remove lignin; separate lignocellulose into its constituents (cellulose, hemicelluloses, and lignin); or possibly even liberate cellulose nanofibrils. Examples of the benefits achieved from such nano-controlled disassembly include enhanced material properties (e.g. lignin and hemicelluloses could be liberated at near native state) and reduced environmental impacts. For example, energy costs would be reduced in pulping if such nanostructured catalyst disassembly avoided the high temperature processing (>100^{o}C) employed with currently used delignification technologies. The need for pulp bleaching might also be minimized or avoided.

Manufacturing costs could also be reduced by employing nanotechnology to prevent cellulose and hemicellulose degradation and yield loss due to over-processing. For example, it is possible that cellulose or other-types of nanofibrils/nanomaterials could be grafted onto wood fiber surfaces. These grafted nanofibrils/nanomaterials could reduce or eliminate the need to do additional mechanical fiber refining via improving fiber-fiber network bonding or allowing a reduction in the amount of fiber needed to produce products. Water removal (drying) via use of thermal (steam) energy is a big cost in the production of most forest products. If nanomaterials could be used to eliminate all rewetting in the press nip (such as in wet press felts), significant manufacturing energy savings could be achieved. For example, self-assembling temperature-sensitive nano-polymers could be attached to fiber surfaces in order to convert hydrophilic fiber surfaces to hydrophobic surfaces at temperatures above a selected low critical solution temperature (LCST) so that water can be more easily squeezed out of fibers network. When temperature returns to that below the LCST, the nano-polymer changes to hydrophilic and serves to improve fiber-fiber network bonding.

Research Goal and Objectives

The goal of this focus area is to develop the capacity to adapt and use nanoscale sensors and nanomaterials to reduce manufacturing costs and improve quality and performance while imparting multifunctionality to forest products.

R&D Focus Area 4 - continued
Nanotechnology in Sensors, Processing, and Process Control

Some specific objectives include the following:

- Develop feedstocks and wood-based products that have sensing capability at the nanoscale—this research would be focused on raw material manipulation or modification genetically so that the building block of feedstock has sensing capabilities to provide moisture control, et cetera
- Develop lignocellulosic-based sensors. This research is focused on fundamental understanding of lignocellulosic materials so that it can be modified chemically to produce sensing-specific capability, such as temperature, or self-healing
- Develop the capability to effectively and efficiently incorporate a variety of functional nano materials with wood-based materials and paper at the macro, micro, and nanoscale to make high-performance, high-value products
- Develop or deploy durable, rugged, and low-energy or passive nanosensors that can be incorporated into wood and paper-like products to add value. Research will include monitor or control of temperature, pressure, volatile organic compounds (VOCs), moisture content, mold, and insect attacks. Research will also include fiber tagging to increase fiber recyclability/trackability and fiber surface characteristics for identification
- Develop rugged, robust sensors to monitor processes at the nanoscale and sampling procedures for their effectiveness. This goal is related to general sensor development for monitoring nanoscale behavior or process in wood products manufacturing. The research is more or less related to Analytical Methods for Nanostructure Characterization (R&D Focus Area 5)
- Develop and employ highly selective nanostructured catalysts to efficiently and effectively delignify or separate wood into its constitutive components in environmentally preferable ways (e.g. no environmentally troublesome byproducts are produced; high temperature processing is not needed; product yields are high; and energy consumption is greatly reduced compared to current processes)
- Develop nanotechnologies that can modify fiber surface nanostructure and fiber-fiber network bonding ability so that wood fiber mechanical refining energy and fiber useage can be significantly reduced
- Explore the use of nanofibrils/ nanomaterials in forest products unit operations such as water removal as a means to significantly reduce energy consumption

Progress in nanoscale research involving lignocellulosic materials will be significantly enabled by metrology that allows observation and monitoring of nanoscale features and properties.

Outcomes and Impacts

Wood and lignocellulose-based materials can be used as a substrate or as the sensor itself. The manufacturing costs for producing forest products will be greatly reduced through increasing final product yields and reduced energy consumption. The following examples illustrate some of the potential applications:

R&D Focus Area 4 - continued
Nanotechnology in Sensors, Processing, and Process Control

1) Nanosensors incorporated into wood-based materials that can provide very early warning of either mold or termites.

2) Nanoscale processes that can provide self-healing once the structure is being attacked by mold or termites.

3) Nanosensors on packaging materials that can detect conditions such as food spoilage or medical package tampering or exposure to unsafe conditions.

4) Intelligent papers that have memory capabilities or are responsive to radio or electronic signals.

5) Nanosensors that can provide fiber tagging for recycling, forensic, or counterfeiting applications.

6) Nanostructured catalysts that can liberate cellulose nanofibrils from wood as well as selectively remove lignin and/or hemicelluloses in environmentally preferable ways.

7) Nanofibrils/nanomaterials that can be used in forest products processing to significantly reduce manufacturing energy and materials consumption.

Key Research Challenges

The primary barriers to achieving the goals of Nanotechnology in Sensors, Processing, and Process Control can be generally categorized in three areas:

1) *Technical*—A lack of basic knowledge of the nano-scale architecture and formation processes of wood and wood components exists, such as a fundamental understanding of cell wall structure and self-assembly. There is also a limited number of scientists and technologists with expertise to conduct and apply the needed research on nanosensors in the forest products industry.

2) *Cultural*—The forest products industry is conservative and risk-averse

3) *Social*—Regulatory issues and the perception of the wood, pulp, and paper industry as low-technology limits driving forces for change.

R&D Priorities

Basic Research in Food and Medical Product Contamination

Identify microbial species or chemical/optical/physical agents that are unique fingerprints or signatures of food spoilage, medical contamination, or product degradation, and develop methodologies for incorporating these agents into non-obtrusive, low-cost, robust nanosensors for food and medical packaging materials.

Much research in this area may already be available (e.g., assessments of classes of chemical signatures, an indicator that will identify the chemical signature, diagnostic information for health care practitioners, feedback for remote health care, contamination by pathogens, sterility indicator). However, more may be required, especially in integrating the knowledge to wood-fiber science or paper-coating chemistry and biosensor research to develop paper-deployable molecular-level sensing capabilities of the identified species or agent related to spoilage or contamination.

Basic Research in Wood-Fiber Science

Investigate genetic and chemical modifications of wood lignocellulose materials to enable basic sensing capabilities and self regulation (e.g., for moisture, temperature, VOCs).

R&D Focus Area 4 - continued
Nanotechnology in Sensors, Processing, and Process Control

Research is needed on modifying fiber surfaces so that they can easily interact with other species to build nanosensors on the surface or nanosensors that are responsive to radio and electronic signals. Knowledge developed in fiber cell wall structure and self-assembly research should be integrated into this effort.

Research in Coating Technology and Materials

Investigate and develop paper and wood product coating technology and coating materials that can deploy nanosensors to these products through mechanical or chemical means.

Research is needed in developing new coating material and novel coating deployment techniques (such as inkjet printing of conductive materials on paper), as well as paper-surface modification to be receptive of new coating or printing materials and techniques. Basic research in new "ink" or coating material interaction with fibers is also needed.

Research in Fiber Tagging

Investigate and develop fiber tagging techniques (e.g., through coating or fiber modification) to enable fiber separation and identification for recycling, counterfeiting, or forensic applications.

Examples include "nanotechnology watermarks" that code paper or wood products for recycling or that identify or certify "chain of custody."

Basic Research in Data Synthesis

Study and develop methods to synthesize data from multimillions of nanosensors in order to generate useful information for action or process control.

This work will require research in mathematics and computer science related to signal processing and data synthesis.

Research in Nanostructured Catalysis

Develop cost effective, efficient, environmentally-preferable and highly-selective nanostructured catalysts for disassembling wood and lignocellulose.

Nanostructured catalysts are needed that are able to liberate cellulose nanofibrils from wood, remove lignin, and have the ability to separate lignocellulose into its constituent components at high yield and in near native-state. Understanding the principles that control nanocatalysis is key to developing more effective catalysts. In this way rates of reaction can be greatly increased as well as increasing selectivity.

Research in Forest Products Processing to Achieve Manufacturing Cost Savings

Carryout research on the use of nanomaterials in conjunction with unit operations in the processing of wood and wood-based materials.

Areas of particular importance include preventing degradation and yield loss, improving water removal, decreasing fiber refining, and fiber modification to achieve significant energy and materials savings in the manufacture of wood fiber-based forest products.

R&D Focus Area 5
Analytical Methods for Nanostructure Characterization

Description

Progress in nanoscience and nanotechnology is significantly enabled by tools that allow visual observation and manipulation of the nanosized features and measurement of properties at the nanoscale. Each of the preceding four topics Polymer Composites and Nano-Reinforced Materials; Self-assembly and Biomimetics; Cell Wall Nanotechnology; and Nanotechnology in Sensors, Processing, and Process Control—has needs for tools which are able to describe the size and shape (morphology) and composition and chemistry at the nanometer scale of lignocellulosic materials, as well as take measurements of mechanical, electrical, and electronic properties.

Although general statements about nano-scale analysis tools already available can be made, it is important to recognize that it is often necessary to develop or adapt tools (and specimen preparation) to unique scientific or analysis questions. While the scientist or engineer always wants more information than is available at any specific time, it is often productive to identify how the answers to specific well-focused questions can be addressed. Thus, this focus area centers on general issues and identifying analytical challenges.

Two related but fundamentally different types of nanoscale analysis questions exist. Frequently, questions are asked about the structure, chemistry, or properties of a specific nanosized object. Either that specific object is of interest or it is assumed to be an example of other similar objects. However, for industrial applications, it is equally important and possibly more important to be able to determine representative properties and distributions of properties that occur in a collection of nanoscale objects. When these nanosized objects are incorporated into larger functional systems, it is a particular challenge to understand the nature of these objects in the overall systems and how changes at the nanoscale alter overall system properties.

Lignocellulosic materials are complex structures and, as natural materials, can vary significantly in properties within and between species. Many of the techniques being developed in the areas of soft-matter physics and nanotechnology need to be adapted and developed to meet the challenges presented by lignocellulosic materials.

Research Goal and Objectives

The overall goal of the analytical methods focus area is to develop the nanoscale characterization methods and physical (mechanical, electrical, magnetic, optical) and chemical property measurements and techniques necessary to adequately characterize complex wood and wood-based lignocellulosic materials, alone or used in conjunction with other organic or inorganic materials, at the nanoscale in three dimensions over relevant time and length scales. Specific objectives include:

1) Adapt currently available physical and chemical property instrumentation used in nanotechnology and nanoscience to lignocellulosic nanofibrillar and cellular morphology.

2) Utilize the intense light sources and neutron scattering tools being developed at national laboratories to gain deeper understanding of the nature of lignocellulosic materials.

3) Adopt and adapting the techniques used in the area of soft matter physics, such as light scattering and rheology measurements, to quantify the structures encountered in lignocellulosic materials.

R&D Focus Area 5 - continued
Analytical Methods for Nanostructure Characterization

4) Adopt techniques from biological analysis, such as phage display, to characterize lignocellulosic surfaces in terms of bonding sites.

5) Achieve three-dimensional imaging capabilities for lignocellulosics at nanoscale.

6) Achieve nanoscale lignocellulosic measurement methods that are scientifically sound and artifact-free.

The goal is to develop characterization methods, measurements, and techniques for the complex nanoscale architecture and composition of wood and wood-based materials.

The understanding and use of the unique properties of cellulose, hemicellulose, lignin, and wood extractives in more advanced applications requires adequate characterization and control of these properties at the nanoscale. Analysis and characterization tools have proven to be the essential and sometimes limiting capability that facilitates the development of nanoscience and nanotechnology in specific areas. It is essential that the range of tools currently applied to more conventional materials be made accessible to those working with forest products. This can be facilitated by gathering the information available on tools and centers where these tools are available. In addition, development and training efforts will enable a new generation of tools to be developed or applied to lignocellulosic materials.

Research cooperation and collaboration and information sharing is essential in making rapid progress.

Key Research Challenges

The following include some of the more prominant challenges in this focus area:

1) Characterization of self-assembled (bottom up) nano-, micro-, and macro-systems such as lignocellulosics is inherently more complex than engineered (top down) materials and, therefore, analysis issues and structure property relations are more challenging.

2) The need to determine nanoscale morphology suggests use of microscopy or scattering techniques. The desire for chemical information suggests use of various forms of spectroscopy. Unfortunately, the organic/polymeric nature of lignocellulosic material places special constraints and demands on the tools that can be used and the manner in which they can be used. One of these constraints is that lignocellulosic materials (like other organic/polymeric materials) are subject to damage by electrons, ions, and photons. As a result, exposure must be limited and/or evidence of damage and rate of damage must be determined.

3) The type of information needed determines the applicable tools. A chemical analysis that reports simply the presence of carbon and oxygen is of very little value. Specific functional analysis is required: aromatic carbon, carbonyl groups, carboxylic acids, hydroxyl groups, et cetera. Fortunately, many well-established tools for characterizing materials on a nanometer scale exist because the needs of the semiconductor

R&D Focus Area 5 - continued
Analytical Methods for Nanostructure Characterization

industry have promoted their development and utilization. However, they must still be adapted to organic polymeric materials.

R&D Priorities

Compendium of Available Tools

Create and maintain a compendium of available analysis tools.

It is recognized that many types of analysis methods exist that can be used to characterize materials at the nanoscale. Many of these methods are not commonly available to workers in the forest products industry. Because it is important to researchers and engineers to understand what is available and how different techniques might be used, a frequently mentioned need is a compendium of characterization tool descriptions that includes their outputs, limitations, and specimen requirements. A comprehensive list may be difficult to produce and maintain, however, because new tools are constantly being developed, and existing tools and methodologies are always being improved. Nonetheless, the beginning of such a compendium is provided in Appendix F. Also included in the appendix is a listing of user facilities where some special instruments are available for research use.

Improved Measurement of Hemicellulose

Develop techniques and tools to measure hemicellulose polymer structure and properties at the nanoscale.

Hemicelluloses are inherently difficult to describe for several reasons. Although glucose is the principal monomer, other sugars are also incorporated. Much of the polymer is linear; however, branches exist, and the repeat structure is irregular. Furthermore, some side chains are acetylated. Hemicelluloses are also much lower in molecular weight than cellulose, and they are somewhat water-soluble and subject to modification by hydrolysis.

The morphology of woody plant cell walls is complex.

Measurement of Lignin

Develop techniques and tools to measure lignin structure and properties at the nanoscale.

Lignin is the most poorly defined plant cell wall component. It appears to be a highly cross-linked polymer composed of phenylpropene monomers (*p*-coumaryl, coniferyl, and sinapyl alcohols) produced late in the growth of plant cells and embedded in a complex cell wall structure. It could be described as the product of free radical polymerization; however, no consensus exists on the details of the process or molecular structure. Part of the reason is that lignin is such an intractable material. It is not soluble and must be isolated from cellulose and hemicellulose by aggressive destruction of those components. It is subsequently broken into small fragments that can be characterized. During this process, there is always some concern that alteration of the original polymer has occurred.

R&D Focus Area 5 - continued
Analytical Methods for Nanostructure Characterization

Methodologies and Instrumentation

Develop methodologies and instrumentation to determine cell wall morphology and measure properties at the nanoscale.

The morphology of plant cell walls even at the micrometer scale is complex. There are many different types of cells that perform different functions and change function as the plant matures. In a very crude approximation, a plant stem is composed of tubular cells fused together by lignin-rich layers (middle lamella). The walls of the tubes consist of a few distinct layers composed of varying amounts of hemicellulose and lignin reinforced by cellulose nanofibrils.

The morphological information obtained at the various lengths of scale must be integrated and self-consistent.

There are several types of technical challenges for the description of cell wall structure, which are illustrative of the difficulties involved in working with any wood nanomaterial. One would like to describe each distinct wall layer, but we do not know how to isolate the layers without damage. Even to prepare an ultrathin cross section (*ca.* 100nm) of a cell for study by transmission electron microscopy or x-ray microscopy is a difficult task. Alternative methods for preparing cross sections must be developed.

The size and shape of plant cell walls offer a challenge for scanning probe microscopy. The vertical range of most scanning probe microscopes is on the order of 5 µm. This is smaller than the diameter of most cell lumens, which range from about 10 to 30 µm. This limits the portions of a specimen that are approachable by the scanning tip. The irregularity of cell structure also presents problems for scanning probe microscopy. These irregularities often result in contact with the side of the scanning tip rather than the point, which results in images that are very difficult to comprehend.

Achieving a realistic representation is a common problem in microscopy. There is widely held consensus that morphological information from different length (size) scales must be integrated. This implies using different microscopical techniques and working from low resolution toward high resolution to be certain that the fields studied in high resolution are not unique.

Compositional information is needed in addition to the description of size, shape, and structure. Perhaps the most confounding problem is that cellulose and hemicellulose are chemically similar (both polymers of simple sugar). If one is concerned with the composition of nanomaterials, then one is most likely to be employing spectroscopical methods; solution chemical analysis does not seem applicable. The differences in vibrational and electronic spectra are very small; mass spectra may exhibit better selectivity. Perhaps some means of selectively labeling one material may improve discrimination. Antibodies tagged with gold have been employed in some electron microscopy studies.

Secondary ion mass spectrometry (SIMS) is another means of obtaining chemical information with high resolution imaging. It is very surface-sensitive (1-10 nm) and has fair lateral resolution (50-100 nm) and very good chemical specificity. Sputtering with small ions (e.g., helium, argon, cesium) creates

No single spectroscopic technique will be sufficient to characterize woody plant materials at the nanoscale.

R&D Focus Area 5 - continued
Analytical Methods for Nanostructure Characterization

damage deep beneath the surface which has frustrated the use of SIMS for composition-depth profiling or organic/polymeric materials. Recently, progress has been made in using 'cluster ions' like SF_5, Au_n, and buckyballs as sputtering ions. These cluster ions are very efficient at sputtering and reduce damage to a very shallow layer. SIMS can be very useful for obtaining three-dimensional imagewise chemical information in organic/polymeric materials.

Strategies that Employ Multiple Techniques

Develop and deploy new collaborative strategies for analysis involving multiple techniques.

The need to combine spectroscopy and microscopy and/or apply multiple techniques and disciplines is a recurring theme in the discussion of nano-scale analysis tools. To date, no single spectroscopical technique has been demonstrated to be self-sufficient. The application of more than one instrument provides good validation of the accuracy of results. For example, SIMS provides good chemical specificity, but the detection limits for different species varies widely. X-ray photoelectron spectroscopy (XPS, ESCA) provides more uniform sensitivity but has poor chemical specificity. The combination of both techniques provides robust chemical information. Oftentimes, the most useful nanoscale studies combine information on morphology and chemistry. Computer modeling of experimental results can also be extremely helpful.

Many of the tools we need are not commonly available and have been developed to support the semiconductor industry. This suggests that progress will require collaboration between scientists who may have to struggle to find a common language.

R&D Focus Area 6
Collaboration in Advancing Programs and Conducting Research

In conducting the research identified in the five preceding R&D focus areas—polymer composites and nano-reinforced materials; self-assembly and biomimetics; cell wall nanotechnology; nanotechnology in sensors, processing, and process control; and analytical methods for nanostructure characterization—it is essential that research collaborations and sharing of science and technology knowledge occurs. Collaboration and cooperation needs to occur among:

- Individual researchers
- Researchers with differing disciplines
- Basic and applied researchers and research teams
- Research institutions including universities, research institutes, and national laboratories
- Industry, universities, research institutions, and federal agencies and departments
- All of these groups from countries around the world

Table 1 shows a partial listing of research organizations and their possible roles in advancing the development and application of nanotechnology in the forest products industry sector.

Table 1. Roles and Responsibilities of Potential Research Partners

National Science Foundation	• Develop programs around basic research needs • Focus on pre-competitive research • Provide funding for university basic research and education • Work with/coordinate with other federal agencies, industry, and research institutes/laboratories • Disseminate science and technology information
Department of Energy (DOE) and DOE National Laboratories	• Participate in multidisciplinary and multi-institutional R&D programs • Conduct basic and applied research • Provide access to user facilities by other researchers • Provide funding and other resources for basic and applied research • Collaborate with/coordinate with other federal agencies, universities, research institutes/laboratories, and industry • Disseminate science and technology information
USDA Forest Service	• Participate in multidisciplinary and multi-institutional R&D programs • Conduct basic and applied research • Provide funding and other resources for basic and applied R&D • Provide access to facilities by other researchers • Collaborate with/coordinate with other federal agencies, universities, research institutes/laboratories, and industry • Disseminate science and technology information
USDA Cooperative State Research Education and Extension Service (CSREES)	• Develop programs to meet basic and applied research needs • Provide funding for basic and applied R&D • Collaborate with/coordinate with other federal agencies, universities, research institutes/laboratories, and industry • Disseminate science and technology information
Industry (Forest products companies and their suppliers)	• Participate in multidisciplinary and multi-institutional R&D programs • Collaborate with/coordinate with federal agencies, research institutes/laboratories, and universities • Provide partial funding for R&D • Help guide R&D into fruitful commercial product areas • Implement nanoscale science and technology as appropriate • Participate in multidisciplinary and multi-institutional R&D programs
Universities with materials nanotechnology programs	• Conduct basic and applied research • Provide access to facilities by other researchers • Train students in nanotechnology • Collaborate with/coordinate with other universities, federal agencies, research institutes/laboratories, and industry • Link with universities with forest products and pulp/papermaking programs • Disseminate science and technology information
Universities with forest products and pulp/papermaking programs	• Participate in multidisciplinary and multi-institutional R&D programs • Link with basic science nanotechnology researchers at other universities, research institutes/laboratories, and national laboratories • Conduct basic and applied R&D • Provide access to facilities by other researchers • Leverage resources as appropriate • Engage graduate and undergraduate students in the research • Train students in nanotechnology • Disseminate science and technology information
Research Institutes and Research Laboratories	• Participate in multidisciplinary and multi-institutional R&D programs • Conduct basic and applied research • Provide funding and other resources for basic and applied R&D • Provide access to facilities by other researchers • Collaborate with/coordinate with other federal agencies, universities, other research institutes/laboratories, and industry • Disseminate science and technology information
International Research Organizations	• Participate in multidisciplinary and multi-institutional R&D programs • Conduct basic and applied research • Provide funding and other resources for basic and applied R&D • Provide access to facilities by other researchers • Collaborate with/coordinate with other researchers, universities, research institutes/laboratories, and industry • Disseminate science and technology information

4—Implementation Plan: Next Steps and Recommendations

Next Steps

To efficiently and effectively advance the nanotechnology research agenda for the forest products industry, this roadmap should be used as a starting point for further engaging key stakeholders and stakeholder groups in dialogue, consensus building, and partnership building. The following are some of the key stakeholder groups:

- Forest products industry—primary producers, converters, suppliers, and collective industry groups such as the American Forest and Paper Association (AF&PA), the Southern Forest Products Association, the APA (Engineered Wood Association), the Composite Panel Association, the China Clay Producers Association, and the Georgia Mining Association
- Federal Departments and Agencies—the USDA Forest Service, USDA Cooperative State Research, Education and Extension Service, Department of Energy (DOE) and its national laboratories, National Science Foundation (NSF), and National Institute of Science and Technology (NIST)
- University and Research Institute/ Laboratory Communities—1) universities with forest products and pulp and paper departments and programs, umbrella university groups such as the Pulp and Paper Education and Research Alliance and the Society of Wood Science and Technology, and research institutes and laboratories focused on the forest products industry; 2) the established research communities already involved in nanotechnology research, development, demonstration, and deployment, such as the various Nanotechnology Research Centers located at universities and Federal national laboratories
- International research communities involved in nanotechnology research and/ or focused on the forest products industry

In addition, technical societies can help provide important opportunities for interactions, dialogue, and technical information exchange through conferences, workshops, technical courses, and symposia. Examples of technical societies include the Technical Association of the Pulp and Paper Industry and the Forest Products Society. These two groups regularly schedule conferences, workshops, and continuing education courses to serve the needs of

Next Steps and Recommendations

- Consensus on needs, priorities, and an agenda among all key stakeholders will ensure proper organization, allocation, and deployment of resources.
- To advance the nanotechnology research agenda, partnerships should be established among industry, government, and academia.
- This roadmap is a living document that should be reexamined by convening experts every 3 to 5 years.
- Strong, focused leadership in the form of a Steering Committee is essential to achieving the goals outlined in this roadmap.

primary pulp, paper and wood products producers, suppliers, and converters as well as the basic and applied research communities serving the forest products sector. Technical societies serving the nanotechnology communities include such organizations as the Materials Research Society, the American Chemical Society, the American Society of Mechanical Engineers, and the American Institute of Chemical Engineers.

In building consensus on nanotechnology opportunities and R&D priorities within the forest products industry itself, the industry can capitalize on already established working relationships between forest products companies and universities and federal agencies with active forest products research programs. The industry has established a set of technological themes in its Agenda 2020 Initiative overseen by the AF&PA. Nanotechnology is part of this research agenda. While not all forest products companies currently take part in the Agenda 2020 initiative, it does provide forums for the broader industry to participate. Agenda 2020 fosters collaborative, cost-shared research on pre-competitive priorities of the forest products industry.

Increased linkages need to be made between research communities of the forest products sector and the broader community of nanotechnology researchers in order to capture synergies, enhance accomplishments, and avoid needless duplication of facilities and efforts. This broader community of nanotechnology researchers includes established university nanotechnology research centers; federal departments, agencies, and laboratories having ongoing programs in nanotechnology R&D; federal laboratories with nanotechnology user facilities; and the National Nanotechnology Initiative (NNI).

From these linkages and interactions, consensus on the technological roadmap needs to be achieved among all the key stakeholders, or little will be accomplished other than uncoordinated piecemeal activities. Finding, allocating, organizing, and deploying resources is much easier when a broad consensus exists among the key stakeholders for critical technological needs, priorities are set for exploiting marketplace opportunities, and a visionary, forward-thinking agenda is developed.

Consensus on research direction will dictate proper research, development, and deployment.

Recommendations

This technology roadmap provides a starting point for systematically focusing the many potential and diverse efforts in nanotechnology for the forest products industry. It identifies priority needs and research directions for the next five, ten, fifteen years. It should be viewed as being a dynamic, living document and should be reexamined by convening experts every three to five years—experts who will review the industry's progress, redefine goals, and assess accomplishments versus resources available and resources expended.

A portfolio of research projects commensurate with the size and impact of the forest products industry is needed.

A critical first step in moving nanotechnology for the forest products sector forward is to gain consensus on what the specific focus should be for the short term, mid term, and long term. It is important that efforts be focused on high-impact, high-priority activities that will be the most critical to commercial producers of nanomaterials and nanoproducts. Achieving consensus on critical activities should be accomplished by engaging all the key stakeholders in assessing market potentials, determining technological barriers and the feasibility of overcoming them, identifying assets and resources available, identifying champions for the various program activities, defining funding needs, and identifying risk factors.

Specific next steps include:

1) Identifying and prioritizing specific avenues of promising research

2) Initiating a portfolio of R&D projects that is commensurate with the size and importance of the forest products industry

3) Developing effective funding strategies to support collaborative multi-disciplinary research activities, demonstration and validation of technology, and education of a workforce skilled in developing and applying nanotechnology for the forest products industry

4) Identifying precompetitive technological needs to help set and focus research targets

Realizing the potential of the emerging nanotechnology industry will require a solid, supporting foundation in instrumentation and measurement methodologies. Significant challenges must be addressed to apply nanoscale instrumentation to wood and lignocellulose.

Strong, focused leadership will be required to make implementation of this roadmap a reality. Steps should be taken to establish a steering group that includes key stakeholder groups and key funding groups. The steering group would serve as a focal point and champion for the overall national roadmap and aid in accelerating nanotechnology in the forest products industry. The steering group should include representatives from individual forest products companies, the AF&PA, Pulp and Paper Education and Research Alliance, Society of Wood Science and Technology, USDA Forest Service, USDA CREES, DOE national laboratories, NSF, NIST, and appropriate technical societies.

Strong, focused leadership will be required to make implementation of this roadmap a reality.

The forest products industry should seek to become part of the NNI and participate in its activities. The industry should have as its goal to establish a $40 to $60 million-per-year nanotechnology R&D program under the NNI by 2008.

The forest products industry should have a nanotechnology R&D program of $40 to $60 million per year.

This roadmap represents the first step in communicating the needs and opportunities associated with applying nanotechnology in the forest products industry. Appropriate representatives from the forest products sector (including members of industry, universities, and federal agencies) should begin to interact and increase contacts with the existing nanotechnology research community. In this regard, the USDA Forest Service will seek to participate on the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee of the National Science and Technology Committee.

Appendices

Appendix A: Workshop Agenda

American Forest & Paper Association

TAPPI people resources solutions

Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

Forest Products Laboratory

Nanotechnology Workshop for the Forest Products Industry

The National Conference Center
18980 Upper Belmont Place
Lansdowne, VA 20176

October 17-19, 2004

Sunday, October 17

5:30 pm Dinner in Guest Dining

OPENING SESSION AND INTRODUCTORY SPEAKERS – *General Session Room N3-365*

7:00 pm Welcome – **Phil Jones**, *IMERYS,* and **Ted Wegner**, *USDA Forest Service, Forest Products Laboratory;* Workshop Co-Chairs

7:10 pm USDA Nanotechnology Roadmap – **Hongda Chen**, *National Program* Leader, Bioprocessing Engineering, USDA-CSREES

7:40 pm Overview of Roadmapping Process – **Shawna McQueen**, *Energetics*

8:10 pm Workshop Deliverables – **Phil Jones** and **Ted Wegner**

8:30 pm Adjourn for Evening

Monday, October 18

6:30-8:00 am Breakfast in Guest Dining

PLENARY LECTURES – *General Session Room N3-365*

8:00 National Nanotechnology Initiative: Overview and Planning for the Future – **Mihail Roco**, *Chair, National Science & Technology Council's Subcommittee on Nanoscale Science, Engineering & Technology*

8:45 Department of Energy Nanotechnology Programs – **Paul Burrows**, *Pacific Northwest National Laboratory*

9:30 Nanobiomaterials – **Art Ragauskas**, *Georgia Institute of Technology*

10:15 Discussion

10:30 Break

10:45 BREAKOUT SESSIONS MEET

- Polymer Composites and Nano-reinforced Materials – **Art Ragauskas** and **Margaret Joyce**, Session Chairs – *Room N3-365*
- Self-Assembly and Biomimetics – **Wolfgang Glasser**, **Derek Gray** and **Pete Lancaster**, Session Chairs – *Room N3-247*
- Cell Wall Nanotechnology – **Rajai Atalla** and **Candace Haigler**, Session Chairs – *Room N3-148*
- Nanotechnology in Sensors, Processing and Process Control – **Yulin Deng**, **Steve Kelley**, and **JY Zhu**, Session Chairs – *Room N3-555*
- Analytical Methods for Nanostructure Characterization – **Jim Beecher** and **Tim Rials**, Session Chairs – *Room N3-249*

12:30 Lunch – Guest Dining

1:30 CONTINUE BREAKOUT SESSIONS

3:30 Break

3:45 Progress Reports from the Breakout Sessions – Session Chairs/ Representatives – *General Session Room N3-365*

5:30 Announcements – **Phil Jones**, **Ted Wegner**, and **Jane Kohlman**

Monday, October 18—Continued

5:30 – Dinner – Guest Dining

Evening Speakers – *General Session Room N3-365*

7:00 Nanotechnology – The European Forest Products Perspective – **Tom Lindstrom**, *STFI*

7:30 Forest Products Industry Perspectives for Nanotechnology – **Del Raymond**, *Weyerhaeuser*

Tuesday, October 19

6:30-8:00 am Breakfast in Guest Dining

8:00 CONTINUE BREAKOUT SESSIONS

10:30 Break

Morning Speaker – *General Session Room N3-365*

10:30 Nanotechnology – The National Nanotechnology Initiative – **Sharon Hays**, *Deputy Associate Director, Technology Division of the Office of Science & Technology*

11:00 Roadmapping Report-outs from the Breakout Sessions – Session Chairs/Representatives – *General Session Room N3-365*

12:30 Lunch – Guest Dining

1:30 Depart

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Nanotechnology in Sensors, Processing and Process Control
Chairs: Yulin Deng and JY Zhu

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Appendix D: Selected Workshop Presentation Summaries

The following are summaries of selected presentations from the Workshop. The complete presentations can be found at www.nanotechforest.org.

USDA Nanotechnology Roadmap – Hongda Chen, National Program Leader, Bioprocessing Engineering, USDA-CSREES

Nanotechnology, as a new enabling technology, has the potential to revolutionize agriculture and food systems in the United States. Agricultural and food systems security, disease treatment delivery systems, new tools for molecular and cellular biology, new materials for pathogen detection and protection of the environment are examples of the important links of nanotechnology to the science and engineering of agriculture and food systems. Some overarching examples of nanotechnology as an enabling technology are:

- Production, processing, and shipment of food products can be made more secure through the development and implementation of nanosensors for pathogen and contaminant detection.
- The development of nanodevices can enable the keeping of historical environmental records and location tracking of individual shipments.
- Systems that provide the integration of "Smart Systems" sensing, localization, reporting, and remote control can increase efficiency and security.

The USDA is a partner agency of the National Nanotechnology Initiative (NNI). The Cooperative State Research, Education and Extension Service (CSREES) has identified specific priority research areas in agriculture and food systems, several of which can directly benefit from research in nanotechnology. Research areas, which are highlighted in the USDA Nanotechnology Roadmap, are complementary to and supportive of the goals and missions of CSREES and the Experiment Station Committee on Organization and Policy (ESCOP). Research areas include: pathogen and contaminant detection, identity preservation and tracking, smart treatment delivery systems, smart systems integration for agriculture and food processing, nanodevices for molecular and cellular biology, nanoscale materials science and engineering, environmental issues and agricultural waste, and education of the public and future workforce.

National Nanotechnology Initiative: Overview and Planning for the Future – Mihail Roco, Chair, National Science & Technology Council's Subcommittee on Nanoscale Science, Engineering & Technology

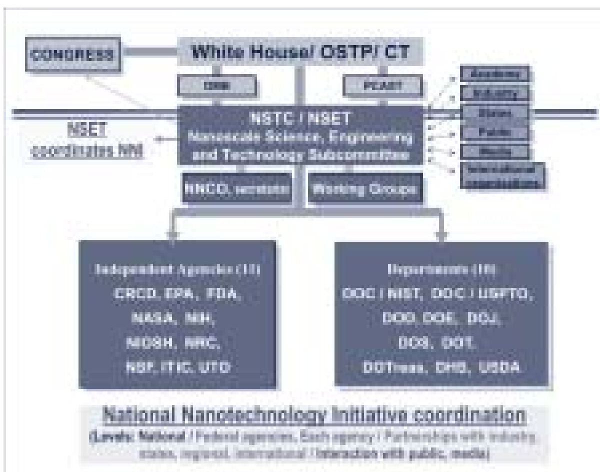
The vision of the NNI is a future in which the ability to understand and control matter on the nanoscale leads to a revolution in technology and industry. Toward this vision, the NNI will expedite the discovery, development, and deployment of nanotechnology in order to achieve responsible and sustainable economic benefits, to enhance quality of life, and to promote national security. The initiative is a multi-agency, multidisciplinary program that supports research and development (R&D), develops infrastructure, and promotes education, knowledge diffusion, and commercialization in nanotechnology.

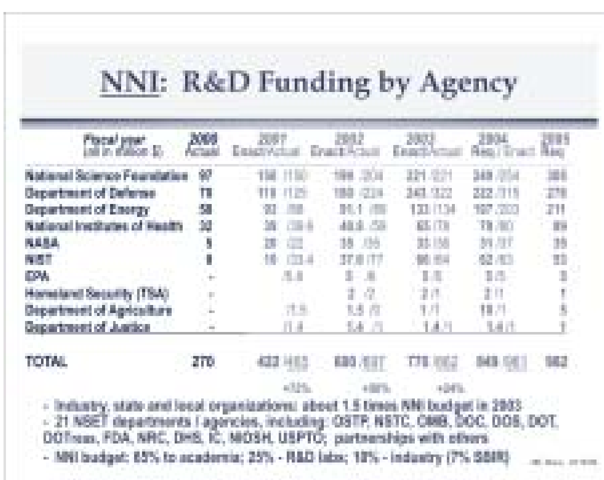
Concomitant with development of new technology options, the NNI is addressing nanotechnology's various societal dimensions. Interagency coordination is managed through the Nanoscale Science, Engineering, and Technology (NSET) Subcommittee of the National Science and Technology Council (NSTC) Committee on Technology.

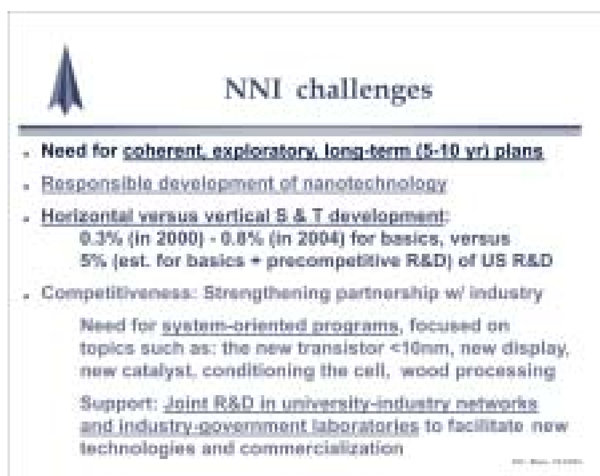
The goals of the NNI are as follows:

- Maintain a world-class R&D program aimed at realizing the full potential of nanotechnology.
- Facilitate transfer of the new technologies into products for economic and public benefit.
- Develop educational resources, a skilled workforce, and the supporting infrastructure and tools to advance nanotechnology.
- Support responsible development of nanotechnology.

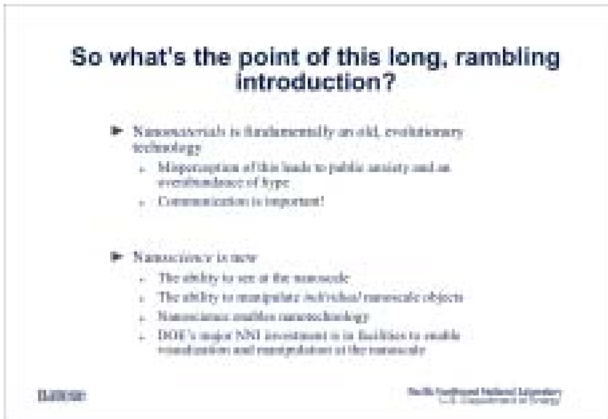
The NNI will provide a balanced and coordinated investment in the program component areas and in a broad spectrum of applications. This will ensure that the United States remains a global leader in the responsible development of nanotechnology and secures the resulting benefits to the economy, to national security, and to the quality of life of all citizens.

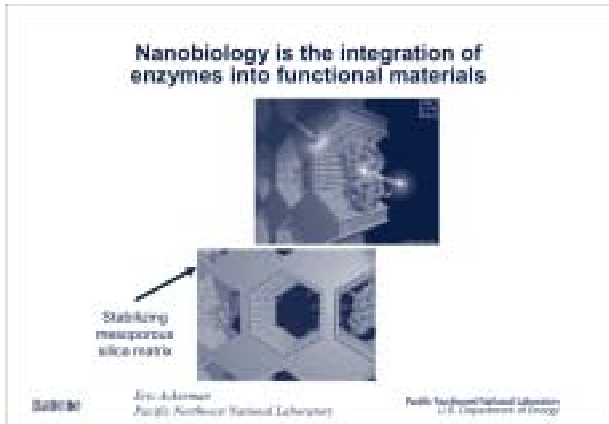


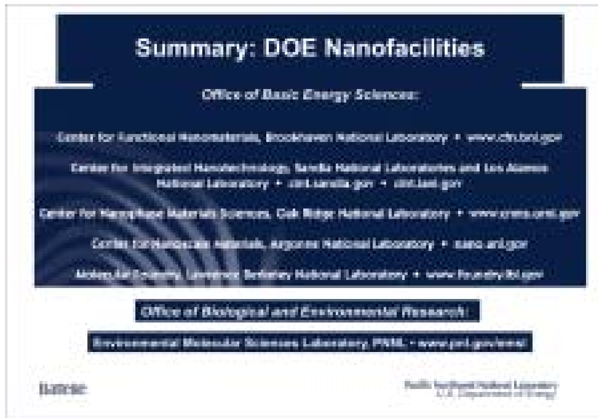




Department of Energy Nanotechnology Programs – Paul Burrows, Pacific Northwest National Laboratory







The National Nanotechnology Initiative – Sharon Hays, Deputy Associate Director, Technology Division of the Office of Science & Technology

The federal government's investments in science and technology have been guided by several fundamental principles. These principles include the following: a) sustain and nurture America's world-leading science and technology enterprise through pursuit of specific agency missions and through stewardship of critical research fields and scientific facilities; b) strengthen and expand access to high-quality science, mathematics, and engineering education, and contribute to preparing the next generation of scientists and engineers; c) focus on activities that require a federal presence to attain national goals, including national security, environmental quality, economic growth and prosperity, and human health and well being; and d) promote international cooperation in science and technology that will strengthen the advance of science and achievement of national priorities.

Nanotechnology will likely have a broad and fundamental impact on many sectors of the economy. The NNI incorporates long-term research leading to new fundamental understanding and discoveries of phenomena, processes, and tools for nanotechnology, and applies them towards grand challenges that support agency missions. The NNI creates centers and networks of excellence to encourage research networking and shared academic users' facilities, develop enabling infrastructures to accelerate commercialization, and prepare a new generation of skilled workers with the multidisciplinary perspectives necessary for rapid progress in nanotechnology.

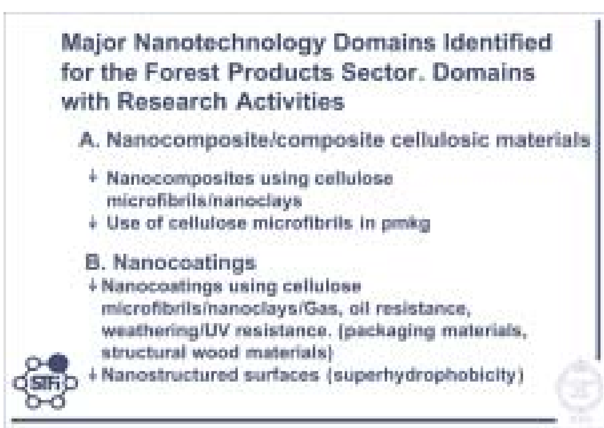
The President signed the 21st Century Nanotechnology Research and Development Act, which put into law programs and activities supported by the NNI. Consistent with this legislation, in 2005 the Initiative will continue to focus on fundamental and applied research through investigator-led activities, multidisciplinary centers of excellence, and infrastructure development, and will continue to support activities aimed at assessing the societal implications of nanotechnology, including ethical, legal, public and environmental health, and workforce-related issues. The President's Council of Advisors on Science and Technology (PCAST) reviews the multi-agency nanotechnology R&D programs and articulates a strategic plan for the program, defining specific grand challenges to guide the program and identifying metrics for measuring progress toward those grand challenges.

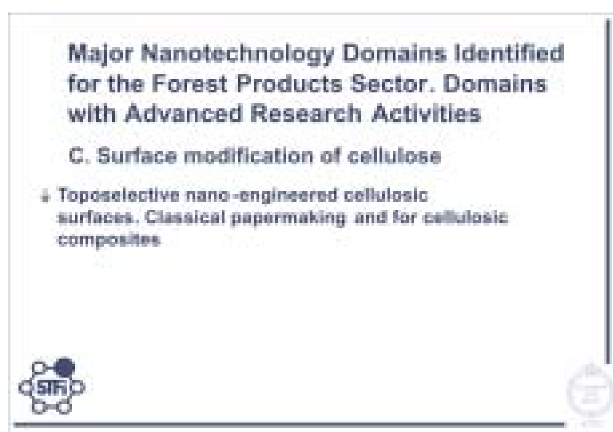
The President's 2005 Budget provides $1 billion for the multi-agency NNI, a doubling over levels in 2001, the first year of the Initiative. This investment advances our understanding of nanoscale phenomena—the unique properties of matter that occur at the level of clusters of atoms and molecules – and enable the use of this knowledge to bring about improvements in medicine, manufacturing, high-performance materials, information technology, and energy and environmental technologies. Agency investments must be consistent with interagency planning documents such as the NNI implementation plan.

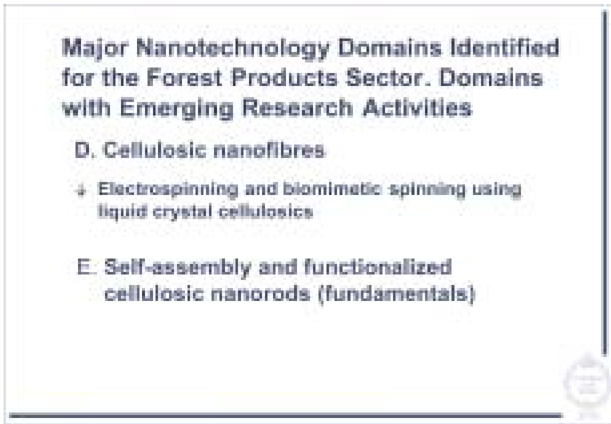
Forest Products Industry Perspectives for Nanotechnology – Del Raymond, Weyerhaeuser

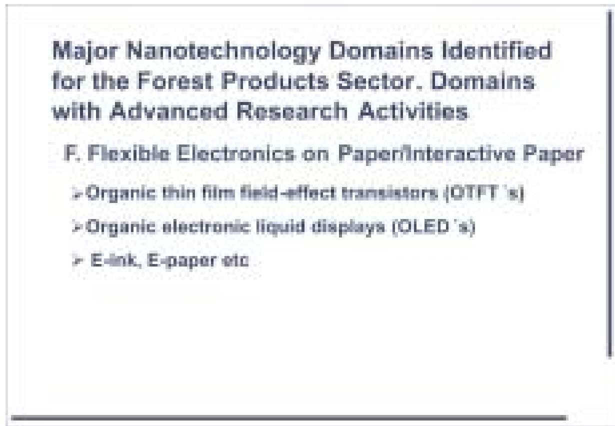
Nanotechnology – The European Forest Products Perspective – Tom Lindstrom, STFI

Nanotechnologies and nanosciences represent a new multi-disciplinary and integrative approach to materials science and engineering, as well as designing new systems and processes by exploiting effects at the nanoscale and controlling the structure and self-assembly of materials. Europe enjoys a strong position in the nanosciences that needs to be translated into a competitive advantage for European industry. The objective is twofold: to promote the creation of a European nanotechnology-enabled industry, and to promote the uptake of nanotechnologies into existing industrial sectors. Research may be long-term and high-risk but will be oriented towards industrial application and co-ordination of efforts at the European Union (EU) level. Encouraging industrial companies, including start-ups, will be pursued through the promotion of strong industry/research interactions in consortia undertaking projects with substantial critical mass. Research and development activities should promote development of new professional skills. For an effective development, European universities may have to adapt with respect to education and training in nanosciences and nanotechnologies. Whenever appropriate, ethical, societal, communication, health, environmental and regulatory issues, in particular metrology and measurement traceability aspects, should be addressed.

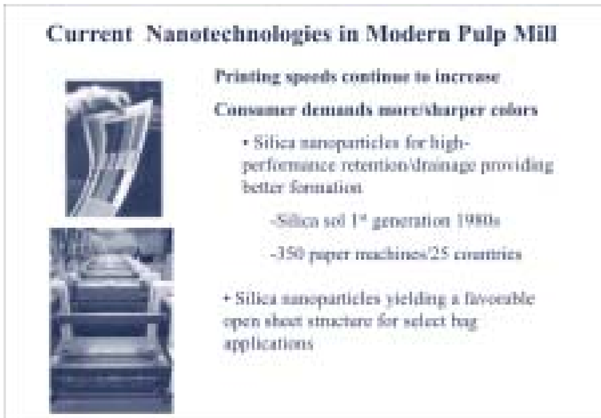


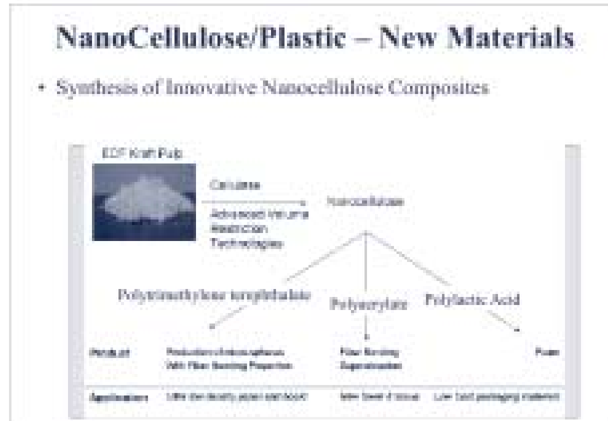


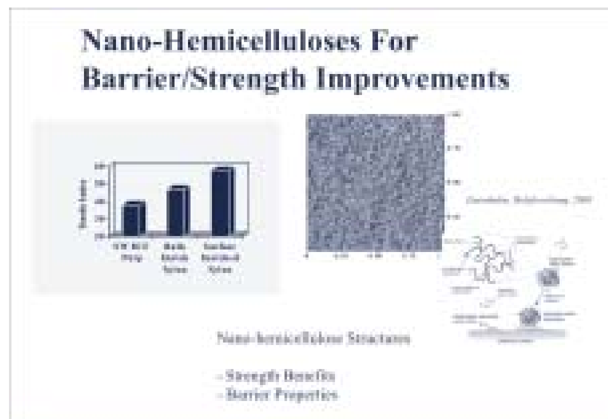

Major Nanotechnology Domains Identified for the Forest Products Sector. Domains with Emerging Research Activities
D. Cellulosic nanofibres
Electrospinning and biomimetic spinning using liquid crystal cellulosics
E. Self-assembly and functionalized cellulosic nanorods (fundamentals)

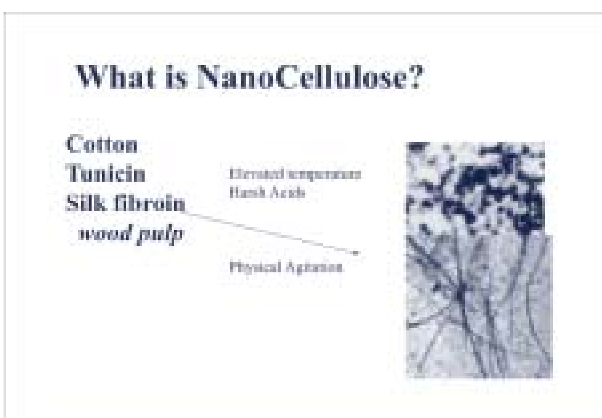

Major Nanotechnology Domains Identified for the Forest Products Sector. Domains with Advanced Research Activities
F. Flexible Electronics on Paper/Interactive Paper
Organic thin film field-effect transistors (OTFT´s)
Organic electronic liquid displays (OLED´s)
E-ink, E-paper etc

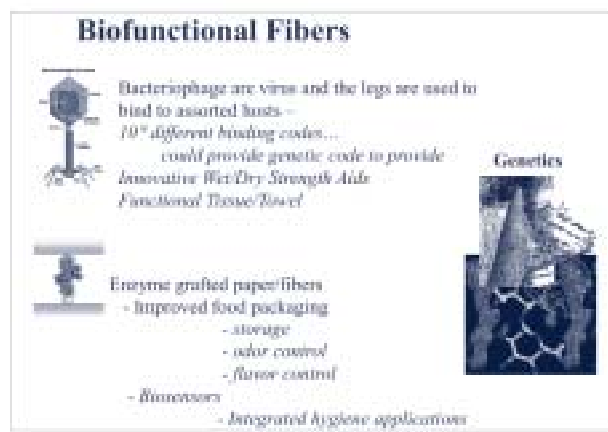
Nanobioterials—-Arthur J. Ragauskas, Institute of Paper Science and Technology — Georgia Institute of Technology, Atlanta, GA











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Appendix F: Tools for the Characterization of Nanometer-Scale Materials

Before discussing some of the contemporary tools which are available and might advance our understanding of cell wall structure, lets review some limitations. When employing a technique in which photon or particle beams are used to form images by transmission, the specimen must be thin.

Another challenge is discriminating different materials. Lignin and holocellulose are distinguishable because lignin has mostly aromatic carbon structures, and celluloses are mostly aliphatic carbon structures. There are few differences between cellulose and hemicelluloses: molecular weight, branching, some side groups, non-glucose sugar monomers.

Another general shortcoming is the interpretation of microscopy/spectroscopy without an understanding of the probe/specimen interaction. This was mentioned many times during the Workshop discussions. It was suggested that computational models should be employed for interpretation. This appears to be particularly problematic in microscopy because images tend to be interpreted as a scene before our eyes lit by the sun.

Duchesne and Daniels recently reviewed wood cell wall structure, the techniques used to learn that structure and some of the limitations (Duchesne 1999). Some tools which may be useful in advancing our knowledge are reviewed below.

Specimen Preparation

Drying

Drying methods for wood and artifacts created by critical-point drying, freeze drying, and air drying are reviewed by Duchesne and Daniel (Duchesne 1999).

Microtomy

It is a serious challenge to prepare 20- to 30-nm cross sections of homogeneous materials by ultramicrotomy. The challenge is greatly increased with heterogeneous and fibrous materials. The microtome knife is really initiating a crack which will grow along the path of least resistance (Jesior 1986). The presence of fibrous materials such as cellulose fibrils will redirect the progress of the crack.

For thin cross sections, which are appropriate for visible light microscopy, the common practice is to ‘soften’ wood by swelling in water or other liquid mixtures. This practice may not always be appropriate. An alternative means of ‘softening’ wood could be to heat the specimen. This is equivalent to the widely employed practice in synthetic polymer microtomy–cryoultramicrotomy–of lowering the specimen temperature to achieve the desired hardness.

Distortions introduced by microtomy have been systematically examined (Jesior 1986), and means to reduce distortion with diamond and glass knives have been reported (Matzelle 2000; Matzelle 2003). H. Sitte, who has extensively studied microtomy and directed the design of commercial instruments, has thoroughly reviewed microtomy practice (Sitte 1996).

Polishing

Grinding and polishing are frequently used to prepare flat specimens for analysis (ASTM 1960). Specimens of metals and alloys for SEM/EDX or optical microscopy are routinely prepared by cutting cross sections and polishing the surface with abrasives of increasing fineness (**Metallographic sections**). There is no special difficulty if all phases of the specimen have similar hardness.

These techniques have been adapted for preparing large cross sections of paper by embedding the paper in epoxy resin (Williams 2000; Rothbard 2003).

Focused Ion Beam Cutting

Ions with kilovolt energy interact with solid specimens in a number of ways. The primary ions implant within the specimen and displace specimen atoms in the process. Along this interaction path, secondary ions and neutrals are sputtered from the specimen along with electrons and photons. Ultimately, some of the energy raises the specimen temperature. These interactions can have useful effects and some create damage (Meingaills 1987).

Focused ion beams (**FIB**) are often used in the semiconductor electronics industry for manufacturing and specimen preparation. Typically, 30-keV gallium ions focused to about 100 nm with beam currents of nanoamperes (nA) are used to cut specimens. In the usual process, stair-stepped depressions are carved into the specimen with a rastered ion beam on either side of a thin (~ 100 nm) section to be studied. Finally, the edges of the thin section are cut free from the specimen, which is recovered and mounted on a transmission electron microscopy grid (Giannuzzi 1999; Overwijk 1993).

Most of the published examples involve inorganic semiconductor materials (Veirman 1999; Phaneuf 1999) or inorganic composites (Kim 2000). There are reports of biological specimen cross sections–human hair and housefly eye (Ishitani 1995). An FIB was used to prepare a cross section of color photographic film which was subsequently imaged by the ion beam (Phaneuf 1999). John Henry Scott showed a wood cross section which was prepared by FIB in his presentation at the Workshop.

Estimates of the damage caused by FIB can be made by Monte Carlo simulations (Kim 2000) or by determining the depth to which gallium ions are implanted. These have been reported to agree well (Giannuzzi 1999). Specific results depend upon material composition and ion energy, but the ion implantation depth is usually about 20 nm, and atom displacements are confined to about 30 nm for gallium ions in low atomic number materials. The depth of the damage layer is strongly dependent upon ion accelerating voltage and the incident ion milling angle (McCaffrey 2001). Single crystal silicon layers cut by FIB still show evidence of crystallinity where the ion damaged layers would be expected to be amorphous (Giannuzzi 1999).

Another consideration for biological materials is that the specimens will necessarily be exposed to vacuum during preparation. However, this may not be a concern because the preparations are intended for TEM, STXM, or EXAFS examination where further vacuum exposure will occur.

The focused ion beams are often used to decompose gases such as $W(CO)_6$ at the specimen surface to deposit metal decoration on the specimen for protection or to reduce charge accumulation.

Microscopy and Spectroscopy

Scanning Probe Microscopies

Scanning probe microscopy techniques, especially tapping mode atomic force microscopy, are the most frequently mentioned techniques for characterizing nanometer-scale structures. These techniques are reported in well over 10,000 research papers each year. Their potential use is evident, and the potential for abuse is much more subtle. Like all microscopy techniques, SPM can be self-satisfying—an image will be obtained (often just what you were looking for); the instruments are readily available and convenient to operate.

The early use of AFM by Hanley and Gray *et. al.* to describe wood cell structure illustrates some of the specimen preparation problems and limitations (Hanley 1992; Hanley 1994). Cell structures were subjected to physical and chemical treatment in the preparation process. Although such methods are commonly used, it leaves concern that the resulting specimen is an accurate representation of native plant morphology. The AFM images illustrate the problem of probe tip shape convolution with specimen morphology.

Interpretation of the images requires a thorough understanding of the probe/specimen interactions. Quantitative modeling of the contrast mechanism is encouraged. The use of other microscopy techniques along with SPM is beneficial in two ways: 1) examination at larger scale will establish a context for high-resolution studies and help to select representative fields, 2) other imaging methods will reinforce the SPM findings because scanning probes frequently create artifacts.

This is a case where collaboration with an experienced SPM microscopist may be the best path to understanding.

Fortunately, there have been many reviews of this field recently (Poggi 2004; Meyer 2004). One excellent and comprehensive review by an SPM-pioneer offers a good place to begin learning of the promise and pitfalls of SPM (Colton 2004). This review begins with scanning tunneling microscopy (**STM**) and follows its evolution and concludes with the measurement of mechanical properties utilizing nanoindentation methods.

Atomic Force Microscopy

Atomic force microscopy (**AFM**) is the most often applied SPM method for describing molecular solids or biological specimens. In its first mode, contact AFM, the stylus tip was maintained in contact (or very near contact) with the specimen as in a miniature profilometer. Currently, it is most often used in a tapping mode in which the stylus and supporting cantilever are set into vibration near their resonant bending frequency (nominally ~ 100 kHz).

The tip makes only intermittent contact with the specimen surface. The tip/specimen interactions alter the amplitude, resonance frequency, and phase angle of the oscillating cantilever. The relative vertical position of the tip is moved to maintain constant amplitude of oscillation; this is often described as amplitude modulation (**AM-AFM**). The reduced surface forces result in less specimen damage. The change in vibration phase (i.e., the delay between the cyclic motive force applied to the cantilever and the resultant movement of the cantilever) often is used to discriminate between areas with different composition on the specimen surface. The phase image thus contains chemically sensitive information. However, the interpretation of this information is not always clear (Colton 2004; Raghavan 2000).

Frequency modulation (**FM-AFM**) is another dynamic mode of operation in which the cantilever amplitude is maintained constant while the stylus/specimen interaction alters the frequency. FM-AFM is used with the stylus near the specimen surface but not in contact. An exhaustive review of dynamic AFM, including theory and operation, is available (Garcia & Perez 2002).

Interactions between the stylus and specimen arise from many different mechanisms—van der Walls attraction, electrostatic, friction, viscoelastic, wetting, et cetera. It is not always evident which forces are involved. One frequent effect which is not always anticipated is the condensation of water on the specimen surface about the stylus. This results in a capillary force which is large enough to dominate the probe/specimen interaction. It is difficult to get the atmosphere dry enough to eliminate this water condensation. The most frequent practice for very high resolution studies is to work in ultrahigh vacuum (usual for atomic solids) or under fluids (water for biological materials).

AFM has been used to measure the van der Walls' force between regenerated cellulose surfaces (Notley 2004). Measurements were made in an aqueous environment with low pH and high ionic strength to suppress DLVO charge effects.

Force Spectroscopy

To learn some chemical or physical information about a specimen in addition to the topographical image, some microscopists have gained information by studying the changing forces that occur as the probe approaches and retracts from the surface. This is often called **force spectroscopy** and can be performed at selected individual locations or at each point in an entire image field (Dufrene 2002; van der Aa 2001). Stylus tips have been modified for force spectroscopy by chemical modification or by bonding cells to the surface. This has been particularly valuable in characterizing cell surfaces (Ahimou 2002; Ong 1999; Frederix 2004). Functionalized AFM tips were used to study intermolecular interactions with epoxy polymers in different liquids (Vezenov 2002).

A similar process, chemical force microscopy (**CFM**), was first described by Frisbie *et. al.* (Frisbie 1994). Chemically modified probes were used to measure the adhesive and friction forces between the probe tip and organic monolayers terminating in lithographically defined patterns of distinct functional groups.

Friction–nanotribology–is another force which has been studied using SPM (Burns 1999; Carpick 2004). An in-depth review of friction force measurement applied mostly to atomic solids was prepared by Carpick and Salmeron (Carpick 1997). The non-vertical component of stylus motion is often attributed to friction or viscosity. However, as Carpick points out: "If the sample surface is not flat, the surface normal force will have a component directed laterally and will result in contrast in the lateral force image." We can anticipate that there will not be a lot of flat surface on biological materials.

Nanoindentation

Vertical forces may be used to measure material properties of specimens such as elastic modulus or hardness. This has been done with AFM for measurement of material properties as well as identification of phases (VanLandingham 2001; Bischel 2000). The spatial resolution afforded by AFM must be tempered with inherent limitations due to the definition of stylus tip shape and the non-vertical component of force.

Recently a review of the contact mechanics relevant to SFM has been published (Unertl 1999). This emphasizes the assumptions underlying and restricting the application of most commonly used models and their implications for SFM measurements.

More rigorous and quantitative material property measurements can be obtained at a sacrifice of spatial resolution using a nanoindenter (Bhushan 1996; Colton,2004). With a nanoindenter the force on the probe and the position of the probe are measured independently. This is not the case with an AFM probe; the force on the stylus point is determined by the deflection of the cantilever which is related to the position relative to the specimen. The nanoindenter probe only moves vertically; whereas, an AFM stylus is always tilted from the vertical and often twisted by lateral force. However, nanoindenter probes are on the order of 50 nm in tip radius, which is much broader than AFM styli.

Nanoindentation has been used to quantitatively measure dynamic mechanical properties as well as static measurements on the nanometer scale (Syed Asif 2001; Syed Asif 2000; Syed Asif 1999). With the application of a small vertical oscillation to the probe, quantitative stiffness imaging of mechanical properties can be mapped at the nanoscale. This offers an excellent way to transition between the optical microscopy scale (micrometer scale) to the AFM scale (nanometer scale). These techniques are much easier to apply to flat specimens than highly textured surfaces.

Probe Shape

When characterizing non-planar specimens, the shape and size of the probe becomes important. The tip of the probe is often described as a hemisphere having a particular radius. Real probe tips are more complex and are not often characterized. However, the SPM image is a convolution of the probe tip shape and the morphology of the specimen; thus, it is important to characterize the tip (Villarrubia 1997).

Carbon nanotubes with diameters of about 1 nm have been suggested as the ultimate high-resolution probe. These probes have limitations, especially when they are not exactly perpendicular to the specimen surface. These effects have been analyzed and their use for non-contact imaging examined (Snow 2002). Isolated protein molecules on mica were imaged using carbon nanotube non-contact AFM (Bunch 2002).

Multi-walled carbon nanotubes used a probes in tapping mode AFM offer a more complex situation. The nonlinear dynamics appropriate for this interaction have been investigated experimentally and theoretically (Lee 2004).

Near-Field Scanning Optical Microscopy

The spatial resolution attainable with conventional optical techniques is limited to about half the wavelength of the light source used. For visible radiation, this results in a theoretical resolution limit of 200-300nm. Higher resolution—near-field scanning optical microscopy (**NSOM**)—can be obtained by illuminating a specimen through a very small aperture (*ca.* 50 nm) positioned very close to the specimen (about one aperture diameter away). An extensive and detailed review of NSOM is recommended (Dunn 1999).

To interpret NSOM images it is necessary to understand how these devices deliver light to subwavelength dimensions and to characterize the fields at the aperture. One of the current limitations is due to the high temperatures developed at the end of the scanning tip. Most of the radiant energy is absorbed by the conductive coating which defines the aperture; this has resulted in temperatures near the tip of nearly 500°C. NSOM tips can be used in the tapping AFM mode by synchronizing the detection with the tip vibration.

Many examples involve the illumination of specimens with NSOM and recording fluorescent emission from the specimen. Spectra from single molecules have been measured using this approach.

Scanning Electron Microscopy

Scanning electron microscopy (**SEM**) offers the possibility of nanometer resolution with little sample preparation. This is especially the case with variable pressure or environmental (**ESEM**) in which specimens can be examined in a low pressure atmosphere without any conductive metal coating. This atmosphere could be water vapor; therefore, plant or wood specimens can be examined in great detail in their native state.

The difficulty of achieving sharp images with good contrast increases with increasing atmospheric pressure. It also depends upon the experience and skill of the microscopist and the characteristics of the microscope.

There is little difference in the probability of secondary electron or backscatter electron emission for materials composed of carbon, oxygen, and nitrogen. This means that the SEM images convey little chemical information and mostly describe texture or topography.

Substantial advantage may be obtained by operating an SEM with a beam voltage in the range 0.5—5 keV. Low voltage (**LVSEM**) often affords good voltage contrast on uncoated specimens and reduces charging and damage (Goldstein 1984). The difference in secondary electron emission between polymeric materials of different composition can be optimized at low accelerating voltage (Berry 1988). The LVSEM images only interrogate a shallow surface layer because the penetration depth of these electrons is limited (Goldstein 1984).

Beam electrons stimulate the excitation of inner-shell electrons, which result in the emission of characteristic x-rays or Auger electrons. For low atomic number atoms, the probability of Auger electron emission is greater than x-ray emission. X-rays emitted by this process can be quantified imagewise by energy dispersive x-ray analysis (**EDX**). It is difficult to quantify the low energy x-rays from low atomic number elements (Goldstein 1984).

EDX analysis is somewhat complicated in ESEM because of the large number of secondary electrons generated by interactions with the gas surrounding the specimen. These secondary electrons stimulate x-ray emission from everything in the specimen chamber; this increases the amount and complexity of the background signal.

Transmission Electron Microscopy

Transmission electron microscopy (**TEM**) offers the possibility of sub-nanometer spatial resolution along with limited imagewise chemical information. The most serious limitations are that specimens must be thin enough that only single electron scattering events are likely—*ca.* 100 nm or less—and specimens must resist electron damage. Carbon replicas have been employed to avoid the problem of thick specimens (Cote 1964; Norberg 1968).

Images in TEM are created by the diffraction of electrons. The materials with which we are mostly concerned are made of carbon, oxygen, and nitrogen, which have very similar electron scattering cross sections. There is very little contrast between different components. One approach to developing image contrast is to preferentially label a component with a higher atomic number element. Lignin has been labeled using bromine, potassium permanganate, and osmium tetroxide (Duchesne 1999).

The highest resolution images of cellulose fibril morphology are a result of the examination of developing wood cells. Cells in different stages of development are rapidly frozen (so quickly that water does not form ice). The frozen specimens are cleaved, and the exposed surface is replicated with carbon and shadowed with platinum. The replica is examined by TEM (Itoh 2002)

Our current knowledge of hemicellulose disposition is from TEM of specimens which have hemicellulose labeled with gold tagged antibodies to hemicellulose (Baba 1994). The use of nanometer gold tagged antibodies is routine in biological microscopy (Baschong 1998). Software has been developed to create 3-dimensional images from a collection of rotated images containing these small gold particles (Ziese 2002).

EELS

In addition to being deflected, some beam electrons suffer energy losses due to interactions with specimens. Many beam electrons lose small amounts of energy (<50 eV) by exciting valence electrons to nearby unoccupied states. Less often, beam electrons lose energy by ionization of specimen atoms (i.e., exciting electrons from inner shell bound states). These are discrete losses and are characteristic of different elements; this gives rise to the descriptive term 'core edge.' Thus, the electron energy loss spectrum (**EELS**) for a carbohydrate would exhibit a carbon core edge near 285 eV from the primary beam energy and an oxygen core edge near 532 eV (Leapman 1992).

Different types of carbon bonding can be distinguished if the EELS spectra can be obtained with sufficient energy resolution. Thus, amorphous carbon can be distinguished from diamond or graphite, or aromatic carbon can be distinguished from aliphatic carbon. This can readily be achieved with 70 meV resolution currently available. It is best to use a field emission electron source for this purpose because they have an energy spread <.3 eV compared with the 1.0—1.5 eV energy spread (FWHM) of a thermionic electron source.

Quantitative chemically specific images may be obtained imagewise using energy filtered TEM (**EFTEM**), or a spectrum may be collected at each image pixel by scanning transmission electron microscopy (**STEM**). Rapid advancement in this instrumentation has occurred in the past 10-12 years. There is still concern about electron damage to specimens because beam currents of about 1 nA are required for acquisition times of a few minutes to obtain good signal to noise.

A new approach which is still limited by specimen thickness but promises higher resolution is **NAED**–nano-area electron diffraction. A nanometer-sized coherent electron beam was used to resolve the structure of a single double-walled carbon nanotube (Zuo 2003). The diffraction intensities were recorded and Fourier-transformed into an image. Iterative software was employed to find a unique solution to the phase problem.

X-ray Beam Probes

The intense energetic beams generated by synchrotrons can be collimated to very small dimensions (ca. 10 to 50 nm) and resolved to 0.3 electron volts (eV). Scanning transmission x-ray microscopy **STXM** has been used to describe the morphology of polymer composites (Ade 1992; Hitchcock 2002). By selecting x-radiation of different wavelengths, contrast can be developed between different materials (this is the same phenomenon as EELS, described above, except that x-rays instead of electrons are used to stimulate core electron transitions). Very thin specimens (100 – 150 nm thick) are necessary because this is a transmission probe technique. Intense high energy radiation could alter the specimens (although x-radiation is not as directly damaging as electrons).

It may be difficult to develop contrast between cellulose and hemicellulose. However, a recent study of mixtures of ethylene-butene copolymer with ethylene-octene copolymer demonstrated STXM images of separate phases of these polymers. They differ only in the length of the side chair (i.e., ethyl versus hexyl groups) (Appel 2002).

X-ray spectroscopies are often used along with STXM (Cody 1995a, 1995b). **NEXAFS** (near edge x-ray absorption) and **XANES** (x-ray absorption spectroscopy) use x-rays to excite core level electrons to unoccupied states. In near-edge spectroscopy, the fine structure at lower energy than the absorption edge is investigated with high energy resolution. This fine structure depends on many parameters such as the oxidation state, local symmetry, or ligands around the absorbing atom. The perceived difficulties are the same as those for STXM.

At energies higher than the absorption edge is a weak periodic modulation which may extend for hundreds of electron volts. This extended energy-loss fine structure (**EXELFS**) arises from a modulation in the ionization cross section caused by interference between the outgoing electromagnetic wave emitted from the ionized atom and components reflected from neighboring atoms (Leapman 1981; Joy 1986).

In x-ray photoelectron spectroscopy (**XPS**), an incoming monochromatic x-ray photon causes the removal of a core or valence electron. The escaping electron has a kinetic energy that is determined by the energy of the photon and the binding energy of the electron. XPS is an ultrahigh vacuum technique which provides information from the outermost 3- to 5-nanometer layer of the specimen. The binding energy depends upon the element and its chemical state. This is inherently a surface technique because of the low energy of the photoelectrons (Briggs 1983). It has limited chemical specificity (can distinguish lignin from cellulose but not cellulose from hemicellulose) and can be performed imagewise with resolution of about 20 nm (Fulghum 1999).

Another method for obtaining spatially resolved chemical information is **X-PEEM** (Tonner 1988). In this technique, photoelectrons emitted from a specimen are recorded as the wavelength of x-rays is changed. Synchrotron radiation is used as a source of x-rays which are dispersed with a monochrometer. This is an ultrahigh vacuum technique which requires conductive or thin (*ca.* 100 nm or less) specimens to avoid charge accumulation on the specimen (Gilbert 2000).

A good illustration of the application of X-PEEM, STXM, and AFM to the study of an immiscible blend of polymers was reported recently (Morin 2001). In addition to illustrating the value of the different techniques, it is a good example of how a more complete picture is created by combining the data from multiple techniques. Spatial resolution of 10 nm has been reported at the Wisconsin Synchrotron Radiation Center (SRC) (De Stasio 1999; Frazer 2004).

Infrared Microspectroscopy

Coupling infrared and visible light through an infrared microscope, this technique combines the chemical specificity afforded by infrared spectroscopy with the visual imaging of optical microscopy. Spatial resolution claims range from 3 to 30 micrometers (Koenig 1998). Synchrotrons may be used as a source of high-intensity infrared radiation; they provide a small spot source (*ca.* 100 mm) with between two to three orders of magnitude brightness increase over blackbody sources (Dumas 2003). In spite of the high brightness, synchrotron sources have not produced evidence of damage to biological specimens. Focal plane mercury-cadmium-telluride array detectors are routinely used to obtain spectral information imagewise.

The spatial resolution is poor compared with some other techniques, but the chemical specificity is very high. Infrared microspectroscopy is an excellent complement to other spectroscopic tools. Sample thickness greater than a few tens of micrometers can be a problem unless reflectance modes are employed.

Spectroscopic maps with 8-mm spatial resolution have been obtained using an attenuated total reflectance objective lens with a focal plane array detector (Sommer 2001). Even better spatial resolution has been attained by applying near-field optics with a modified AFM head and using synchrotron radiation (Bozec 2002).

Similar information may be obtained by Raman microspectroscopy, but often the intense laser radiation used in Raman spectroscopy creates damage in organic materials. Infrared absorbance is a single photon, direct process while the Raman process is a two-photon scattering process;

the underlying physics are different. The selection rules are different and thus the two spectroscopic methods can provide complementary information. Spatial resolution for Raman microspectroscopy can approach 1 mm. There are two common problems: 1) the excitation laser frequently causes fluorescence which masks the Raman signal and 2) the efficiency of the process is very low (about 1 photon out for 10^6 in) (Adar 2003).

Secondary Ion Mass Spectrometry

In secondary ion mass spectrometry (**SIMS**), a focused ion beam is directed to a solid surface, removing material in the form of neutral and ionized atoms and molecules (as discussed in FIB above). The secondary ions are accelerated into a mass spectrometer and separated according to their mass-to-charge ratio. The lateral resolution is between 1 micrometer and 50 nanometers in different instruments. The mass spectra provide good chemical specificity.

This is inherently a surface probe with 1- to 10-nm depth resolution. The sensitivity to different chemical species varies greatly because only ions are detected, whereas most of the products of sputtering are neutral fragments (Briggs 1992).

The use of primary beams with cluster ions (ions of high molecular weight such as Cs_n, SF_5, C_{60}, and Au_n) has improved the value of SIMS for molecular solids (Wagner 2004; Gillen 2001; Postwa 2003). Cluster ions produce greater useful signal intensity and sputter rate while limiting damage and penetration depth. Cluster ions may be used for analysis at low ion current (static SIMS) and may be used to systematically remove layers from the specimen at higher ion currents. Sputter depth profiles have been demonstrated for polymethyl methacrylate (Wagner 2004). This is a promising way of obtaining imagewise chemical data in three dimensions.

SIMS is inherently an ultrahigh vacuum technique which requires flat specimens. Some means must be employed to eliminate charge accumulation (e.g., electron flood gun or conductive screen over the specimen).

Appendix G: Nanoscience User Facilities

This is a listing of facilities where uncommon tools may be available. Rather than attempt descriptions of these laboratories, the URLs for their web sites are provided.

In addition to this list, most universities have centers where major instruments are shared among different departments. Often, non-university users can also use these facilities which usually include electron and optical microscopes, as well as surface analysis instrumentation. These centers may also provide contact with university specialists who may be potential collaborators; who are more significant help than instruments.

National Laboratories

Argonne National Laboratory
Advanced Photon Source
www.aps.anl.gov

Brookhaven National Laboratory
National Synchrotron Light Source (NSLS)
www.nsls.bnl.gov/

Lawrence-Berkley National Laboratory
Advanced Light Source
www-als.lbl.gov/als/microscopes/index.html

- visible, infrared microspectroscopy
- PEEM (Photoelectron emission spectrometer)
- imaging x-ray microscopy
- x-ray photoelectron microspectroscopy

National Institute for Standards and Technology (NIST)
SURF III synchrotron
Physics.nist.gov/MajResFac/SURF/SURF.html

High-Resolution UV and Optical Spectroscopy Facility

National Renewable Energy Laboratory (NREL)
Surface Analysis
www.nrel.gov/measurements/surface.html

Pacific Northwest National Laboratory
Environmental Molecular Sciences Laboratory
www.emsl.pnl.gov/

Universities and Research Centers

Lehigh University
www.lehigh.edu/nano/

- 13 electron microscopes
- Scienta high resolution x-ray
- photoelectron spectrometer

University of Pennsylvania
www.seas.upenn.edu/nanotechfacility/

- facilities for corporate users

University of Albany
www.albanynanotech.org/Programs/metrology/index.cfm

- six electron microscopes
- two x-ray photoelectron spectrometers
- focused ion beam
- three scanning probe microscopes
- Fourier transform infrared spectroscopy

University of Notre Dame
www.nd.edu/~ndnano/title.htm

- mostly electronics fabrication
- four electron microscopes
- atomic force microscope
- near-field scanning optical microscope
- Fourier transform infrared spectroscopy

University of Illinois
Center for Microanalysis of Materials
cmm.mrl.uiuc.edu/techniques/sims.htm

- Cameca ims 5f

University of Wisconsin
Material Science Center
www.msae.wisc.edu/mscweb/

- electron microscopes
- preparation facilities
- atomic force microscope
- x-ray photoelectron spectrometers
- focused ion beam

Synchrotron Radiation Center—Aladdin
www.src.wisc.edu

- Scienta 200 high resolution x-ray photoelectron spectrometers
- Infrared microscope
- PEEM Photoelectron emission spectrometer

Nanoscale Science and Engineering Center
www.nsec.wisc.edu

Northeastern University
Center for High-rate Nanomanufacturing
www.nano.neu.edu/

- electron microscopy
- atomic force microscopy

Pennsylvania State University
National Nanotechnology Infrastructure Network
www.nanofab.psu.edu

- electron beam, optical and probe lithography
- novel materials deposition and etching
- electron and optical microscopy
- scanning probe microscopy
- focused ion beam
- near-field scanning optical microscopy

Colorado State University
NSF Engineering Research Center for Extreme Ultraviolet Science
euverc.colostate.edu/

University of Saskatchewan
Canadian Light Source, Inc.
www.lightsource.ca/experimental/

- x-ray microscopy
- Fourier transform infrared spectroscopy

Duke University
Free Electron Laser Laboratory
www.fel.duke.edu/

- infrared FEL
- ultraviolet FEL

Louisiana State University
Center for Advanced Microstructures and Devices
www.camd.lsu.edu/

- synchrotron for x-ray microscopy and spectroscopy

Cornell University
Cornell High Energy Synchrotron Source (CHESS)
www.chess.cornell.edu/

North Carolina State University
Harold Ade research group
www.physics.ncsu.edu/stxm/stxm.html

- near edge x-ray fluorescence spectroscopy (NEXAFS)
- scanning transmission x-ray microscopy (STXM)
- photoelectron emission spectrometer (PEEM)

University of Dayton
University of Dayton Research Institute (UDRI)
www.udri.udayton.edu/

- x-ray photoelectron spectroscopy
- electron microscopy

Purdue University
Birck Nanotechnology Center
http://www.nano.purdue.edu/wps/portal/.cmd/cs/.ce/155/.s/4271/_s.155/4271

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