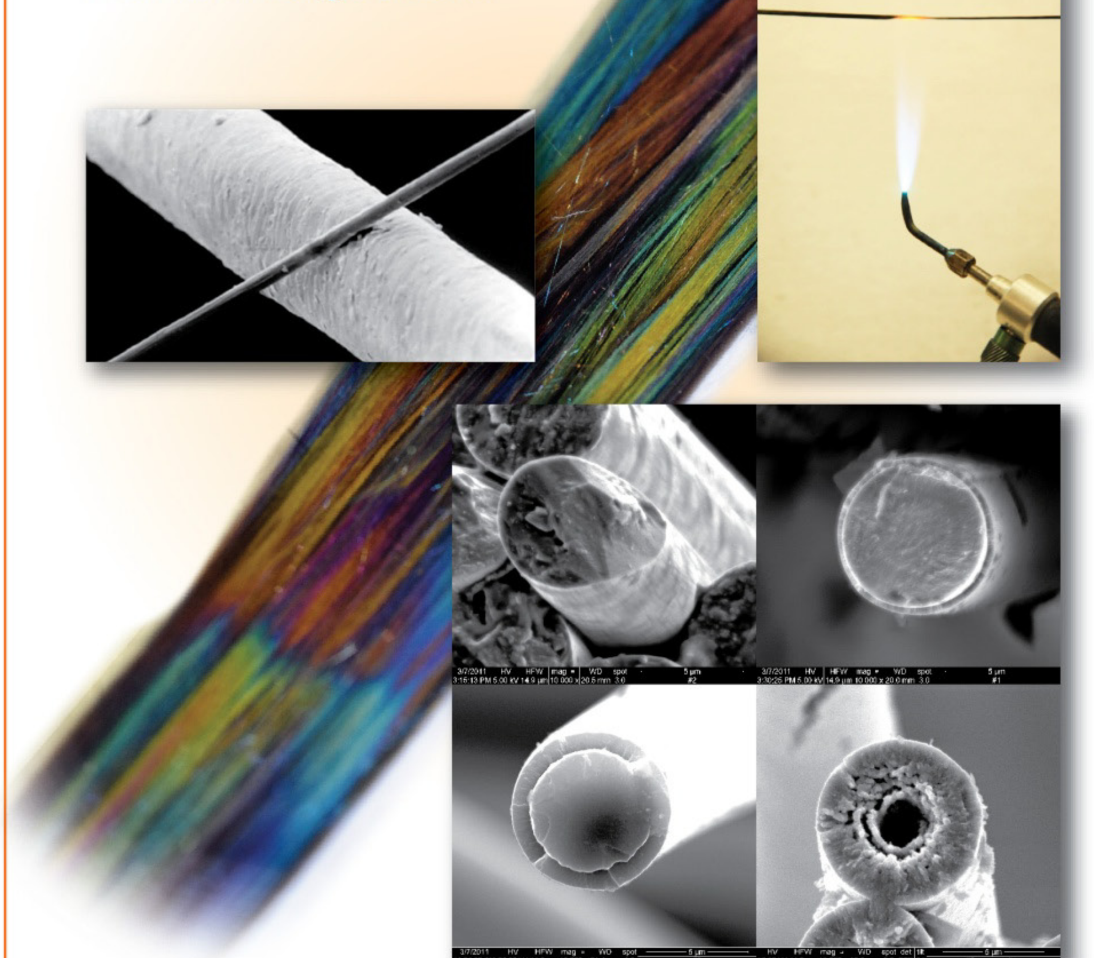
Alpha Silicon Carbide Direct

Producing A New Ceramic Fiber Reinforcing Material



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2012 R&D 100 AWARDS ENTRY FORM

1. SUBMITTER INFORMATION

A. Primary Submitting Organization

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B. Joint Submitter(s)

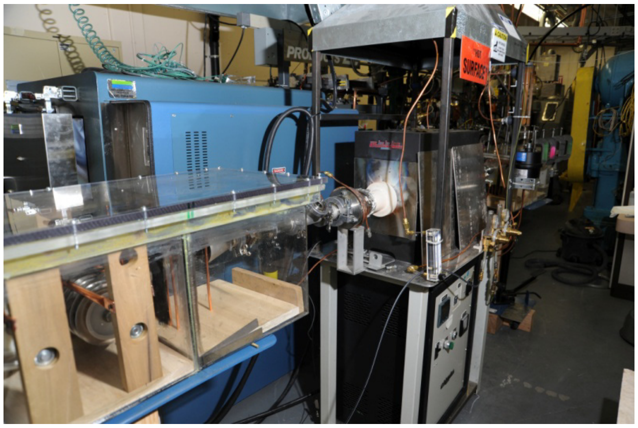
None

2. PRODUCT INFORMATION

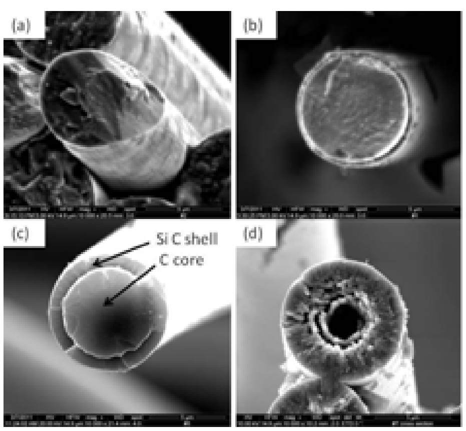
A. Product Name

SiC Direct (full name: Alpha Silicon Carbide Direct Continuous Fiber Process). Click the following link to view video: www.inl.gov/sicdirect

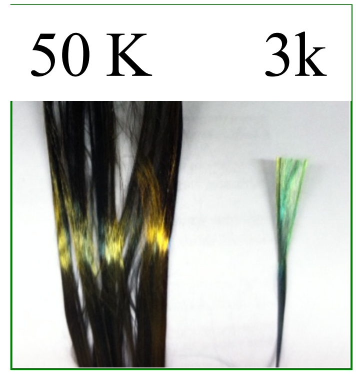
B. Product Photo



*INL researchers Dr. John Garnier and Dr. George Griffith used this small 18-inch furnace with a 6-inch hot zone operating at 1700°C to successfully test and prove **SiC Direct.** The process has proven to be scalable by converting multiple carbon fiber tows containing 3,000 filaments to 50,000 filaments each and commercially viable as low cost raw materials are used in producing uniform, high quality alpha silicon carbide (α-SiC) fibers.*

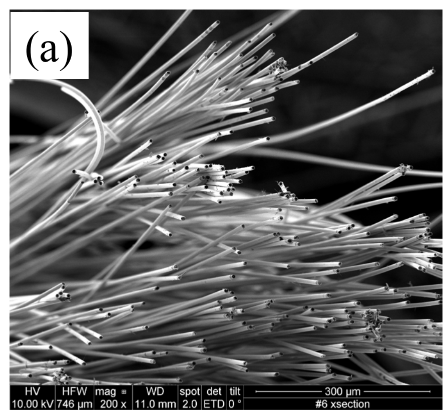


Scanning Electron Microscope (SEM) photomicrographs of cross sections of C fibers during conversion to α-SiC.
(a) As-received (unconverted) solid PAN-based carbon fibers.
(b) Partially converted carbon fiber with ~200 nm thick SiC shell.
(c) Partially converted C fiber with 1 μm (1,000 nm) thick SiC shell.
(d) Fully converted hollow SiC tube.



alpha SiC tows

The **SiC Direct** process has been demonstrated to produce alpha silicon carbide fiber in varying tow sizes. A tow contains smaller filaments, about 6-7 micron in diameter. This image shows results of simultaneous processing of two different alpha SiC tows, one containing 50,000 filaments (left) and the other 3,000 filaments,



This is an Electron Microscope photomicrograph of α-SiC fibers produced in a 3,000 filament (tow) at the Idaho National Laboratory's research laboratory using the **SiC Direct** continuous conversion process

3. PRODUCT DESCRIPTION

SiC Direct is a manufacturing technology materials and process *breakthrough* allowing continuous or pre-formed production of high-strength-to-weight, thermal resistant, affordable alpha (α) phase silicon carbide fibers from low-cost commercial carbon fibers.

4. PRODUCT FIRST MARKETED OR AVAILABLE FOR ORDER

SiC Direct Federal Business Opportunity (FedBizOps) announcement, Dec. 21, 2011:

> Battelle Energy Alliance (BEA), the managing and operating contractor for the Department of Energy (DOE) at the Idaho National Laboratory (INL) is seeking expressions of interest from potential licensees wishing to discuss a potential license agreement for the purpose of commercializing the technology described below. This is not an opportunity to provide goods or services to BEA or DOE. This solicitation will close to response sixty days after publication. Parties interested in obtaining additional information should send their request to: Gary W. Smith, INL Senior Commercialization Manager

Invention Summary (FedBizOps): BEA has developed a process for making continuous micron diameter alpha silicon carbide (SiC) material directly in a fiber form starting from a carbon fiber substrate. Using controlled process conditions of temperature, gaseous atmosphere and form of the carbon fiber substrate, BEA can produce an alpha SiC fiber. Continuous carbon fibers can be drawn through a furnace containing a controlled heating, reaction and atmosphere zone. Fully converted SiC filaments have a narrow hollow core which can increase overall the fiber mechanical strength and partially converted fibers will have a SiC outer layer over a carbon core fiber. The fiber will retain strength during the conversion, thus making the process suited to continuous processing and low fabrication cost. This novel process for making a unique form of alpha SiC fiber is not available commercially today.

On Oct. 6, 2011, a presentation was given at the ASME Nuclear symposium in a public forum that included U.S. manufacturers (***Synthesis and Analysis of α-SiC Components for Encapsulation of Fuel Rods and Pellets).*** Provided in Support Documentation.

INL inventors also presented at the USACA 36th Annual Conference on Composites, Materials, and Structures *(U.S. Citizens Only / ITAR Restricted Sessions)* Jan 24, 2012 • Session on Structural Ceramics **(*Synthesis and Analysis of Alpha Silicon Carbide Fiber for Use in MMC and CMC Composites).*** Provided in Support Documentation.

5. PREVIOUS R&D 100 NOMINATION FOR THIS PRODUCT

No.

6. PRINCIPAL INVENTOR TEAM LEAD

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7. PRODUCT PRICE

Currently, commercial alpha silicon carbide (α-SiC) fibers are not produced or available for sale. No known commercially viable process to produce α-SiC fibers has been invented and made available in the commercial market, until now. So, no direct price comparison for **SiC Direct** can be made to a competitor.

The only commercially-available material for a price comparison is beta silicon carbide (β-SiC) fiber, which requires an expensive pyrolysis production process using silicon-containing polymers, such as polysilazane and polycarbosilane ($4,000 per pound). For example, high quality Hi-Nicalon type S (β-SiC) fibers imported from Japan sell for $6,135 per pound and require a 3-6 month lead time. This situation forces careful decision making in purchasing β-SiC fibers for highly selective use, plus the user must get in line for material delivery.

In comparison, the cost for producing higher quality α-SiC fibers using **SiC Direct,** INL's direct conversion process of silicon fibers, could be as low as $100 per pound at a production level of 100,000 pounds per year.

This conservative cost estimate is based on investments for a modest manufacture scale-up effort of producing 200,000 pounds per year and includes energy savings as a baseline against the current energy intensive β-SiC fibers.

This projected production cost is more than 60 times less costly than the current selling price of the β-SiC fibers, which can be more than $6,000 per pound.

Price Affordability. Much lower production costs, a significant increase in availability, high strength-to-weight ratio and excellent performance in high temperature environments are expected to create significant demand for α-SiC fibers, as well as a rapid expansion in both the application and use of silicon carbide fibers. As a result, **SiC Direct** could cause both a very large number of existing products to be redesigned for manufacture and a barrage of new products to appear, leveraging the advantages of α-SiC fibers.

Process and Product Price Estimates

This cost estimate calculation is the result of a detailed planning exercise. Final costs for initial production efforts by a licensee are expected to be somewhat higher, hence the estimated price of as low as $100 per pound for producing 100,000 pounds per year. Many other conditions or decisions could affect the price per pound, but this provides insight into the economy of INL's **SiC Direct** innovative process.

EQUIPMENT	BASE	CAPITAL MULTIPLER			
	ESTIMATED COST ($K)	1,000 lb/ YEAR	10,000 lb/ YEAR	100,000 lb / YEAR	500,000 lb / YEAR
Bobbin machines (additive by lb/yr)	$20	1	1	5	5
High Temperature Furnaces	$400	1	2	3	5
Post fiber sizing equipment	$50	1	2	4	5
QC equipment	$350	1	1	3	3
Factory Improvements	$350	1	1	3	10
Total Capital Costs ($K)	$1,170	**$0**	**$1,620**	**$3,600**	**$6,900**
Factory Space(sq ft)	10000	0.5	0.5	1	1.5
Estimate Space Cost (lease $k = @ $20/sq ft/yr)	**$200**	**$200**	**$100**	**$200**	**$300**
Electrical (0.25/kwhr)	24,000	24,000	48,000	72,000	120,000
Electical cost ($K)	**$6**	**$6**	**$12**	**$18**	**$30**
Process Gases (future re-cycle)		**$23**	**$114**	**$227**	**$758**
Raw materials (RG SiO2) majority %		**$8**	**$80**	**$800**	**$4,000**
Raw materials (RG Si) minor %		**$9**	**$88**	**$875**	**$4,375**
Raw materials (PAN C) 100%		**$13**	**$127**	**$1,273**	**$6,364**
Other Mfg Cost ($K)					
Support Cost (base labor # = 4, 5, 6, 8 full time)	160	160	200	240	320
Sales and Marketing (labor = 1, 1, 2, 4 full time)		$265	$265	$630	$760
G&A (estimate @ 50% labor)	$80	$213	$233	$435	$540
Estimated Mfg Cost ($K) (sum all 1st year capital and materials and operating.	$1,616	$896	$2,838	$8,298	$24,346
Manufacturing Cost per pound		**$896**	**$284**	**$83**	**$49**
1st year Base Fiber Sales Value (Use lower quality Standard SiC Nicalon @ $1200/kg = $545 lb as baseline. Break even between year 1 and 2 at nominal 6,000 lb/year production total. Note that Hi Nicalon is $13,500/kg)		$400	$4,000	$40,000	$200,000
Facility size		**Lab Test facility**	**6K SF facility**		**10K SF facility**

8. PATENTS OR PATENTS PENDING ON THIS PRODUCT –

Note: This is also available in Appendix E.

United States Patent Application Attorney Docket 2939-10059US (BA-479; Oct. 8, 2011

METHODS OF PRODUCING SILICON CARBIDE FIBERS, SILICON CARBIDE FIBERS, AND ARTICLES INCLUDING SAME, Inventors: John E. Garnier and George W. Griffith

(Patent abstract is provided here and in Appendix E. The applications for this pending patent have not been published by the patent office and have not been made available for public information at this time. For this reason, the complete text of the filed patent is not included.)

ABSTRACT OF THE DISCLOSURE

BA-479 (U.S. Patent Application No. 12/901,309 filed October 8, 2010): Methods of producing silicon carbide fibers. The method comprises reacting a continuous carbon fiber material and a silicon-containing gas in a reaction chamber at a temperature ranging from approximately 1500°C to approximately 2000°C. A partial pressure of oxygen in the reaction chamber is maintained at less than approximately $1.01 \times 10_2$ Pascal to produce continuous alpha silicon carbide fibers. Continuous alpha silicon carbide fibers and articles formed from the continuous alpha silicon carbide fibers are also disclosed.

United States Patent Application Attorney Docket 2939-10288US (BA-530; March 1, 2010; Via Electronic Filing)

METHODS OF PRODUCING CONTINUOUS BORON CARBIDE FIBERS, CONTINUOUS BORON CARBIDE FIBERS, CONTINUOUS FIBERS COMPRISING BORON CARBIDE, AND ARTICLES INCLUDING FIBERS COMPRISING AT LEAST A BORON CARBIDE COATING; Inventors: John E. Garnier, George W. Griffith

(Patent abstract is provided here and in Appendix E. The applications for this pending patent have not been published by the patent office and have not been made available for public information at this time. For this reason, the complete text of the filed patent is not included.)

ABSTRACT OF THE DISCLOSURE

BA-530 (U.S. Patent Application No. 13/215,967 filed August 23, 2011): Methods of producing continuous boron carbide fibers. The method comprises reacting a continuous carbon fiber material and a boron oxide gas within a temperature range of from approximately 1400°C to approximately 2200°C. Continuous boron carbide fibers, continuous fibers comprising boron carbide, and articles including at least a boron carbide coating are also disclosed.

9. SiC DIRECT – PRIMARY FUNCTION

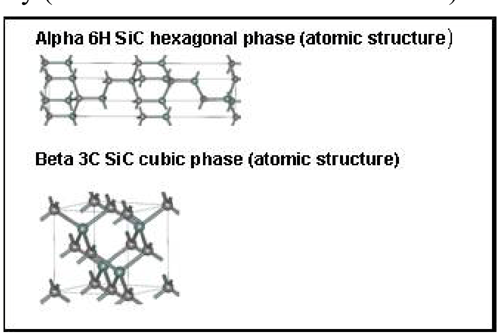
Continuous Production of Alpha Silicon Carbide (α-SiC*) Fibers*

Human history has been characterized by the discovery of new materials and the processes for producing and using them. That is how history bestowed titles on the Stone, Bronze and Steel Ages. In the 20th century, technological advances enabled the Space Age to pursue exploration of the stars using newly developed materials, including use of thermal insulative tiles that permitted the Space Shuttle to safely reenter the Earth's atmosphere.

Ceramics, glasses, polymers, composites, metal alloys and aggressive investigation of material microstructures brought new capabilities to space exploration, medicine, military and manufacturing. All have required diligent research to deliver these significant advances, which have had major international economic impact and created successful commercial competitors with technological advantages.

Advanced manufacturing processes and materials are the cutting edge in maintaining America's global competitive advantage that leads to greater productivity, new products and businesses, plus career opportunities with new skills.

Silicon Carbide – Tomorrow's Age? Today, the chemical, mechanical, and thermal properties of silicon carbide (SiC) fiber make it an attractive degradation-resistant ceramic for a wide variety of aerospace, defense, industrial and nuclear applications. SiC fibers are possible in alpha (α-SiC, hexagonal phase) and beta (β-SiC, cubic phase) crystal structures for commercial use in high temperature semiconductors, abrasives and wear components, heating elements, reinforcement in composites, armor, turbine engines, rockets, and jewelry (moissanite as a diamond simulant).



SiC fibers are super-materials and have an amazing diversity of qualities depending on the crystalline form. They are:

- exceptionally hard, durable and strong (strength-to-weight ratio – 1/3 the weight of steel and 3-6 times stronger),
- chemically inert and light weight,
- thermal shock resistant,
- high temperature resilient (α-SiC fiber's up to 2730°C sublimation point),
- excellent electrical conductors or insulators (depending on their crystal formation during manufacturing and final configuration),
- wear resistant and/or abrasive ,
- mechanically and thermally stable in radiation environments for long-term use,
- composed of and produced from relatively inexpensive materials,
- corrosion resistant,
- capable of high strain (α-SiC stretches 2 percent as individual fiber or 10 percent in larger fiber groups; β-SiC 1 percent),
- variable in compositional forms and application (e.g. 250 molecular polytypes, delivers many attractive qualities inherent in the various crystalline forms to produce a wide variety of products).

Although use of these fibers as reinforcement in metals yields significant strength-to-weight advantages over monolithic metals, the application of SiC fibers in metal and ceramic matrix composites is currently limited due to the high cost of producing commercial SiC fiber, e.g. as much as $6,000 per pound for β-SiC fibers. In fact, SiC fibers currently are only available in β-SiC form as imports from Japan where they are produced through expensive pyrolysis of silicon-containing polymers.

SiC Direct. Now, Idaho National Labs (INL) researchers have developed an ***"amazing breakthrough"*** patent-pending process technology, called **SiC Direct**, which fabricates α-SiC fiber super-materials. It is a simple and affordable direct conversion process that uses inexpensive raw materials (carbon fibers, silicon granules and silicon dioxide sand).

INL researchers have invented a continuous process that can be used to manufacture α-SiC fibers in lengthy coiled fiber (up to 1,800 meters or longer) and woven into desired patterns for even greater durability and more varied use. This patent-pending process also can be employed in a batch processing to produce α-SiC products in various forms, called pre-forms, (e.g. sheets, large three-dimensional forms such as support posts, and many other shapes).

In **SiC Direct** a mixture of silicon dioxide (SiO_2) with silicon (Si) is heated in an alumina crucible using a high temperature tube furnace to generate an oxidative silicon oxide (SiO) vapor.

Carbon Fibers - As spun polyacrylo-nitrile (PAN) filaments are heated to very high temperature (2000°C to 3000°C) the carbon begins to graphitize forming narrow hexagonal graphene sheets which eventually merge to form a single, columnar carbon filament as illustrated in Figure 1, below. In forming a carbon filament, the graphene sheets are folded and shaped into a fiber form, followed by heating to high temperature.

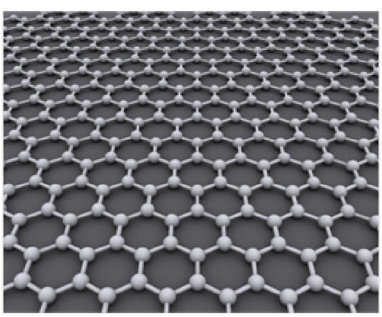


Figure 1: Graphitized carbon forming graphene hexagonal sheets.

Commercially available, the PAN-based carbon fibers contain 3,000 individual carbon (C) filaments, each about 6-7 microns (µm, 6-7,000 nanometers) in diameter. These provide the source of carbon fiber.

Why We Get α-SiC? In the **SiC Direct** process, these carbon fibers are exposed to SiO vapor in an argon carrier gas at elevated temperatures (1600°C) for a short time, which converts it to α–SiC. The fiber draw rate can be adjusted to vary local fiber reaction time with SiO.

In the **SiC Direct** continuous process the reaction between SiO gas and C molecules initiates at the carbon filament surface. The thermodynamic response is from the presence of hexagonal graphene carbon, which results in the formation of hexagonal α–SiC as the dominant phase. However, this does not rule out the formation of some low beta cubic phase of SiC.

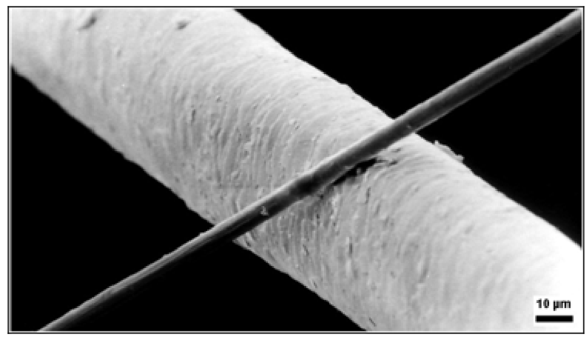


Figure 2: A single 6-micron diameter carbon pan fiber filament is positioned on top of a human hair for comparison. The SiC Direct process has demonstrated simultaneous conversion of groups of both 50,000 and 3,000 filaments.

So, as the carbon (graphene-based) filaments are exposed to SiO vapor in an argon carrier gas at elevated temperature for a short time, conversion to the hexagonal crystalline form, α–SiC, occurs.

In the alpha **SiC Direct** process the fiber draw rate can be adjusted to vary local fiber reaction time with SiO.

This direct process continuously converts C filaments into SiC fiber, beginning at the exposed surface of the filaments to form a shell and then progressing through the entire fiber. A shell measuring ~200 nanometers (nm, 0.02 microns) takes about 4 minutes of exposure in the high temperature furnace.

After about 12 minutes of exposure, a sample with a 1 µm (micron or 1,000 nm) thick SiC shell is made. As dwell time at 1600°C is increased, the fiber is completely converted to SiC. The fiber adopts a tubular geometry as the atomic diffusion of silicon through the forming SiC layer is less than the rate of atomic carbon diffusion outward. Thus, this process is suitable for making both SiC as well as silicon carbide-carbon (SiC/C) composite fibers at high production rate by the direct conversion of the fibers (Figure 3).

Thousands of meters of silicon carbide single filament fibers and multiple fiber groups already have been produced in demonstrated production testing. More tests and demonstrations are underway using continuous **SiC Direct** to experiment further in fabricating fibers at relatively low process temperatures and using low cost gas-phase reactants.

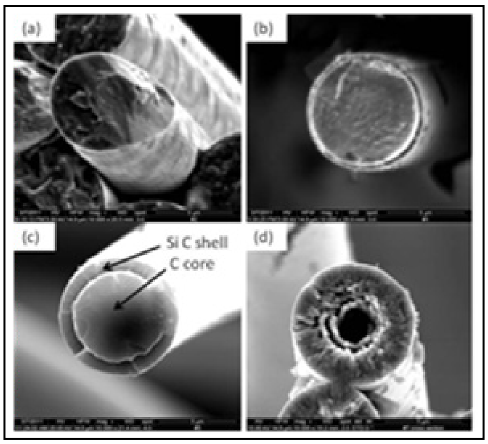
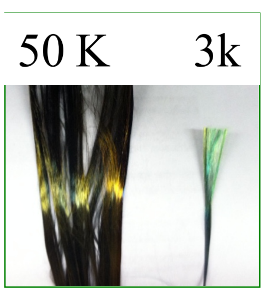


Figure 3: Scanning Electron Microscope (SEM) photomicrographs of cross sections of C fibers during the SiC Direct conversion process to SiC.
(a) As-received (unconverted) solid PAN-based carbon fibers. (b) Partially converted carbon fiber with ~200 nm thick SiC shell. (c) Partially converted C fiber with 1 μm thick SiC shell. (d) Fully converted hollow SiC tube.



alpha SiC tows

Figure 4: The SiC Direct process has been demonstrated to produce alpha silicon carbide fiber in varying tow sizes. A tow contains smaller filaments, each about 6-7 microns in diameter. This image shows results of simultaneous processing of two different alpha SiC tows, one containing 50,000 filaments (left) and the other 3,000 filaments (right).

An affordable method of producing silicon carbide fibers could significantly expand their use in products that already include abrasives, cutting tools, structural material, auto parts, electric systems, electronic circuit elements, thin filament pyrometry, heating elements, jewelry, steel production, catalysts, as well as nuclear fuel particles and cladding.

INL's testing and manufacturing plan calculates production costs for the higher quality α-SiC that could deliver fibers costing as low as $100 per pound. This compares to the selling price of $500-$6,000 per pound from overseas suppliers, depending on the material used for production.

INL's 'breakthrough' process to produce α-SiC uses about 60 percent less energy than current β-SiC production methods by eliminating costly polymer precursors and multiple high temperature processing steps. This advance comes at a significant time in that Department of Energy experts estimate an increased need for an economical SiC fiber to be as high as 120,000 pounds per year for nuclear applications alone.

Initially, the primary function of this innovative technology was to produce affordable α-SiC fibers and then incorporate these more readily available fibers into aluminum by using low cost spray forming methods (another INL patented invention). The purpose was to form continuous SiC fiber reinforced metal matrix composites to determine suitability for manufacturing and additional processes.

The success of **SiC Direct** in producing α-SiC fibers opens an expanding array of uses, ranging from heavy manufacturing to the potential for providing low cost, high area substrates for catalytic surfaces. Identification of applications has just begun and may introduce an entirely new age of materials and material production processes.

SiC Direct was tested and introduced as a market opportunity in 2011 through paper presentations at ASME and then an announcement in the Federal Business Opportunity publication (Dec. 21, 2011). Interest is growing rapidly, as is the application, for this amazing process and its expanding products.

Why now and not before? INL scientist, engineer and entrepreneur, Dr. John Garnier has worked his entire career in leading edge material research and had significant leadership roles in three major corporations (Standard Oil, E.I. DuPont de Nemours and Honeywell).

He and nuclear energy researcher George Griffith sought to make affordable α-SiC materials which could enhance INL nuclear reactor design efforts, fuel designs and reactor operations. Seeking a simple production process that was not capital intensive and could produce affordable α-SiC fibers, the duo began to eliminate approaches.

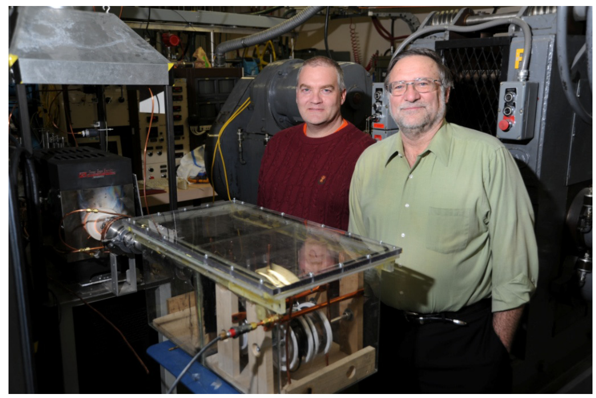
They quickly settled on trying a direct conversion approach, leveraging a small research furnace in one of INL's lab spaces. Their idea proved successful.

Commenting on discovering this elegant and simple silicon carbide fiber continuous production process, Dr. Garnier said, "I've spent my long and interesting career working in specialized materials. At INL it all came together for me to try this production method, because I had the resources of a national laboratory right there. It is amazing how well it works and we are calling the process **SiC Direct**."

Garnier and Griffith made extensive tests on the proprietary process and soon learned that **SiC Direct** exhibited robust process control, while at the same time produced a high quality uniform fiber product. This enhanced its potential for successful scalability, which was proven by the production of coils of SiC fiber as long as a mile (1,800 meters). Then, they turned their attention to expanding it to the batch processing of a wide variety of shapes, which also was successful

INL presented a paper on the base technology titled: "Synthesis and Analysis of Alpha Silicon Carbide Components for Encapsulation of Fuel Rods and Pellets", K. McHugh. J. Garnier, G. Griffith (INL); presented at the Proceedings of the ASME Small Modular Reactor Symposium, SMR2011, Sept. 2011 (see Support Materials).

On Jan. 24, 2012, Garnier presented another paper on SiC Direct to the 36th International Conference and Exposition on Advanced Ceramics and Composites in Daytona Beach, FL (Jan. 22-27, 2012). Both presentations created exceptional interest in the new process, as has INL's announcement in the Dec. 21, 2011 publication of an opportunity in the FedBizOps.



***SiC Direct** research team (left to right): George Griffith and John Garnier.*

The potential for economical manufacturing of current products and creation of new products incorporating this vastly improved material is exceptional and may well introduce a very exciting future in many industries.

10. SiC DIRECT – OPERATIONS, THEORY, AND CHEMICAL REACTIONS

Silicon carbide (SiC) is a refractory ceramic with a vast array of commercial applications because of its exceptional diversity of qualities. The super-material exhibits tremendously high-strength-to-weight ratio (1/3 density of steel and 3-6 times stronger), hardness, chemical inertness, and attractive thermal properties with high conductivity, low expansion, and thermal shock resistance. These and other qualities make it useful for an impressive variety of applications, including abrasives and wear components, high-temperature semiconductors, armor, jewelry (as a diamond simulant), and reinforcement in composites.

Unlike most ceramic materials, it also performs well in a nuclear reactor environment as SiC maintains excellent stability during extended exposure in radiation environments, is composed of low activation elements, and retains its strength and shape under high-radiation dose conditions.

SiC also exhibits polymorphism and, in fact, more than 250 molecular polytypes have been identified, a major asset making its suitable for such diverse uses. Alpha silicon carbide (α-SiC) is the most common polymorph and is formed at temperatures greater than 1700°C. It has a hexagonal crystal structure. Beta silicon carbide (β-SiC) is formed at temperatures below 1700°C and results in the cubic 3C-SiC (β) structure.

> **Silicon carbide has two crystalline phases.**
>
> **Alpha silicon carbide (α-SiC) has a hexagonal crystalline structure known to be phase stable to its sublimation (phase transition from solid to gas) temperature at about 2730°C.**
>
> **Beta silicon carbide (β-SiC) has a cubic crystalline structure that reaches its sublimation temperature about 1700°C and experiences phase transition to alpha at 2200°C.**

SiC formed in hexagonal stacking arrangements (4H, 6H, etc.) are grouped together as α-SiC with the 6H polytype being the most prevalent. Many fabrication routes exist for bulk SiC, including carbon reduction of silica (Aecheson Process), chemical vapor deposition, thermal evaporation/deposition (Lely Process), and pyrolysis of polymers. In general, β-SiC is formed at lower temperatures (<1600°C) and α-SiC polytypes at higher temperatures (2200–2500°C).

The properties of SiC depend on its crystal structure, density, processing method and purity. It also can vary widely depending on the crystal structure (α-SiC or β-SiC; see below).

SiC fibers are very strong (~3 GPa tensile strength) and stiff (~420 GPa tensile modulus), but are quite expensive. Unfortunately, until now, α-SiC fibers are not available at all. The only commercial method for making β-SiC fibers is the pyrolysis of expensive silicon-containing polymers, such as polysilazane and polycarbosilane ($4,000 per pound). As noted earlier, Hi-Nicalon type S fibers, which are β-SiC, are imported from Japan cost $500 to $6,000 per pound and require a 3-6 month lead time.

Currently, all available commercial fibers are β-SiC. The **SiC Direct** process offers a commercially viable method to form α-SiC that greatly simplifies processing and uses inexpensive raw materials (carbon fibers, silicon granules, and silicon dioxide sand). Conversion costs are estimated to be about $60-70 per pound for processing and calculations for 100,000 pound production per year effort could deliver product for a cost as low as $100 per pound (see paragraph 7).

Also, energy savings using **SiC Direct** is exceptional and calculated at requiring 60 percent less than current manufacturing of β-SiC fibers. The current β-SiC fiber manufacturing process uses high cost, energy-intensive polymer raw materials that convert through multiple high temperature furnace passes and post annealing.

Beyond the energy savings in producing α-SiC fibers, **SiC Direct** offers significant savings in manufacturing of materials. Its application in the U.S. manufacturing of flat plate aluminum helps understand the potential impact of domestically producing and using α-SiC fibers.

Significant energy savings could be made by moving from the conventional ingot processing of flat rolled plate aluminum (sheet/plate/foil).

*Properties of SiC Polytypes**

Polytype	3C (β)	4H	6H
Crystal structure	Zinc blende (cubic)	Hexagonal	Hexagonal
Space group	T^2_d-F43m	C^4_{6v}-$P6_3mc$	C^4_{6v}-$P6_3mc$
Pearson symbol	cF8	hP8	hP12
Lattice constants (Å)	4.3596	3.0730; 10.053	3.0730; 15.11
Density (g/cm³)	3.21	3.21	3.21
Bandgap (eV)	2.36	3.23	3.05
Bulk modulus (GPa)	250	220	220
Thermal conductivity (W/(m·K))	360	370	490

* "Properties of Silicon Carbide (SiC)", Ioffe Institute, 06/06/2009. http://www.ioffe.ru/sva/nsm/semicond/sic/.
Park, Yoon-Soo *et al.* (1998). *SiC materials and devices*. Academic Press. pp. 20–60. ISBN 0127521607.

For example, if α-SiC fibers were used for a structural reinforcement in aluminum alloys (e.g. continuous fiber reinforced aluminum metal matrix composite - CFRAMMC) in producing 50 percent of the 5.5 million metric tons of this flat aluminum manufactured in 2003, the following impact could be realized:

- Saving 52.5 trillion British Thermal Units (BTUs) a year, which represents a 30 per cent annual energy savings or equivalent to 8.5 million barrels of oil,
- Increasing strength-to-weight ratio by about 30 percent, plus
- Reducing carbon emissions by 7.6 million metric tons per year.

(Figures based on U.S. Energy requirements for Aluminum Production: Historical Perspective, Theoretical Limits and Current Practices, prepared for the Industrial Technologies Program, U.S. Department of Energy, Energy Efficiency and Renewable Energy by BCS, February, 2007).

Operations and Chemical Reactions.

As outlined earlier in paragraph 9, **SiC Direct** is adaptable for use in a continuous operation or batch operation, depending on the form of product desired.

A schematic of the two approaches (continuous and batch) for making α-SiC are shown in Figure 5. A mixture of silicon dioxide ($SiO2$) with silicon (Si) is heated in an alumina crucible in a high temperature tube furnace to generate an oxidative SiO vapor, which produces the following reaction:

$Si (l) + SiO_2 (s) \rightarrow 2SiO (g)$

Commercial polyacrylonitrile (PAN)-based carbon fibers containing 3,000 individual carbon (C) filaments, each about 7 μm (microns) diameter (Figure 3a) are exposed to SiO vapor in an argon carrier gas at elevated temperature for a short time and converted to α –SiC, producing the following reaction:

$SiO (g) + 2C (s) \rightarrow \alpha\ SiC (s) + CO (g)$

For background history on development of (alpha) SiC, in in the mid 1980s a small business joint venture company Keramont attempted to produce a silicon fiber by heating of short sections of carbon in a closed batch fired furnace. However, the joint venture was terminated after majority acquisition by a major foreign chemical company and the company closed. Given no commercial market for SiC fibers at this time further process research in the process was terminated Dr. Garnier had observed this testing regimen as the second attempts to make a SiC fiber whose structure was not determined. The process apparently failed because of the extended time the carbon fibers spent in the batch fired furnace. Later around 1994, a company called MER revisited the batch furnacing process making several short length (inches) of SiC fibers for a government program involving neutron radiation testing of various fibers including SiC. In this project the structure of the SiC fibers (beta or alpha) were not reported, only that after neutron radiation exposure the MER fibers were intact. Prior to development efforts by Keramont and MER, the first attempt to make an alpha SiC fiber (high L/D ratio) was by Standard Oil Engineered Materials that successfully produced alpha silicon carbide rod (1/16 inch dia x 10 inch) in the mid-1980s using a pressureless sintering approach similar to how Hexaloy-SA is made today. Later as it was determined that the pressureless sintering process could not be scaled to smaller fibers or longer lengths and research was terminated.

SiC Direct uses a very different method to produce α-SiC, as described above and shown in Figure 5.

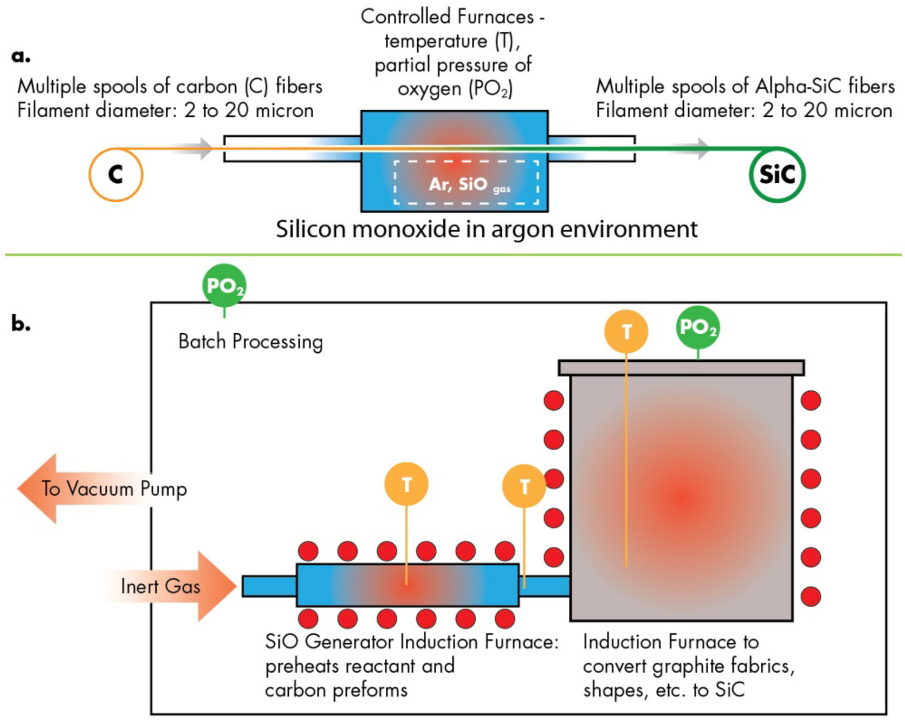


Figure 5. Approaches used to convert carbon fibers to SiC. (a) Continuous direct conversion to SiC fibers. (b) Continuous batch conversion of fibers, fabrics, and other shapes.

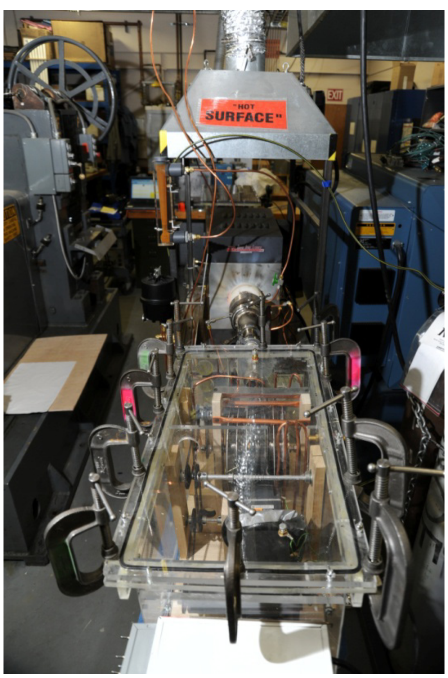


Figure 6: SiC Direct was successfully tested using this small 18-inch furnace with a 6-inch hot zone operating at 1700°C.

The fiber draw, or production rate, is adjusted to vary the resident time and temperature in the furnace hot zone, dependent on the quantity of SiO available (Figure 6). This variance results in continuous conversion of C to SiC, beginning at the exposed surface of the filaments to form a shell and progressing through the entire fiber. A shell measuring ~200 nm, is illustrated in Figure 7b after about a 4-minute exposure.

As reaction progressed (about 12-minute exposure), a sample with a 1 µm thick SiC shell was made (Figure 7c.) As dwell time at 1600°C was further increased, the fiber was completely converted to SiC and adopted a tubular geometry as shown in Figure 7d. Thus, the process is suitable for making both SiC as well as SiC/C. The shell thickness develops uniformly from the individual filaments making up a fiber bundle, which indicates the reactant vapor has penetrated the inter-filament gaps.

Using Electron Dispersive Spectroscopy (EDS), an analysis of the fibers indicated that the converted material was characterized by 1:1 stoichiometric (balanced chemical reaction)

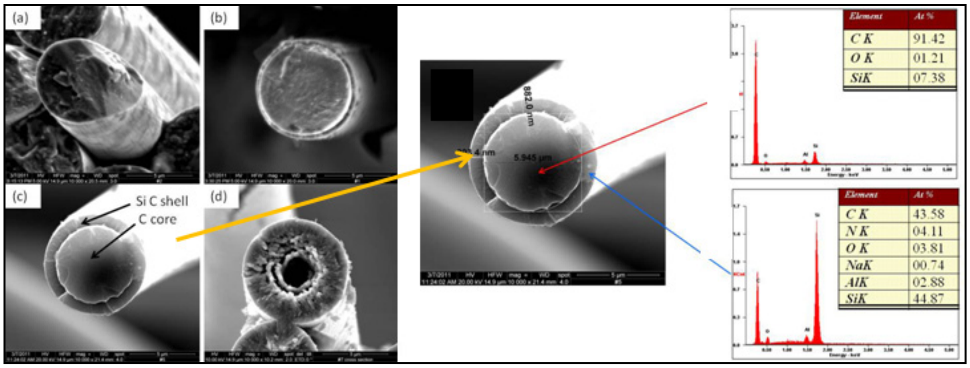


Figure 7 Scanning Electron Microscope (SEM) photomicrographs of cross sections of C fibers during conversion to SiC. (a) As-received (unconverted) solid PAN-based carbon fibers. (b) Partially converted carbon fiber with ~200 nm thick SiC shell. (c) Partially converted C fiber with 1 µm thick SiC shell. (d) Fully converted hollow SiC tube. SEM/EDS analysis of a partially converted filament (right) confirms stoichiometric conversion of C to SiC.

amounts of Si and C with residual Na, Al, N and O materials retained from the coating on the starting C fibers. These products may be useful and/or desired by specific customers; however, pure SiC products can be produced by using different carbon fiber sources.

Analysis indicated the predominant phase was α-SiC with 6H polytypes dominating.

Experimental Evidence of α-SiC. The researchers conducted many tests to confirm production of α-SiC. INL's X-ray Diffraction (XRD) analysis confirms the presence of SiC as "moissanite," a hexagonal form that simulates diamond qualities and has many potential industrial and jewelry uses. Moissanite has visible surface colors from irradiant green to purple. The highest quality appears as a colorless material. Figure 8 displays INL laboratory results from process run number two.

SiC Direct permits customization of products

SiC Direct is robust permitting production adjustments by varying the rate of drawing the carbon fiber source through the furnace and temperature manipulation. In addition, INL has developed two α-SiC fiber direct processes, continuous and batch operational forms, by which researchers have demonstrated that a variety of forms and shapes can be processed.

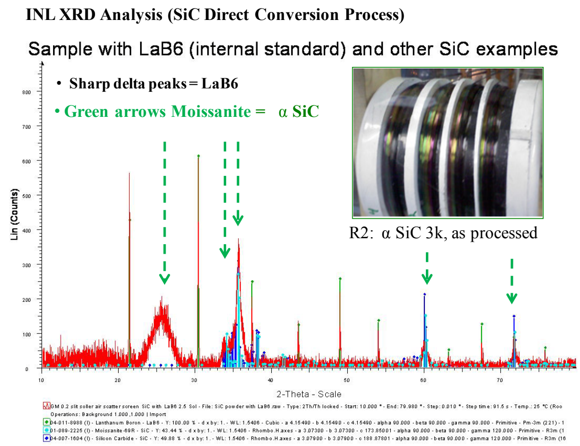


Figure 8: Multi-fiber (tow production) per run with confirming X-ray diffraction (XRD) analysis showing presence of alpha SiC fibers created in hexogaonal form.

Manufacture of α-SiC fiber in quantity enables its adaptation to many different forms. It can be used uni-axially, woven or braided into many different shapes and forms, such as tubes, plates and tapes.

In fact, the **SiC Direct** process may prove to be capable of also producing quality β-SiC materials at lower temperatures for use in appropriate products that do not require the material characteristics at the α-SiC fiber level. Production of β-SiC was not the goal of INL's initial research and its production has yet to be tested and evaluated.

Figure 9. Examples of fiber pre-forms that SiC Direct is capable of producing are shown above demonstrating simple fiber strands (1-inch diameter braid, left) and a woven form (8-inch pre-form, right).

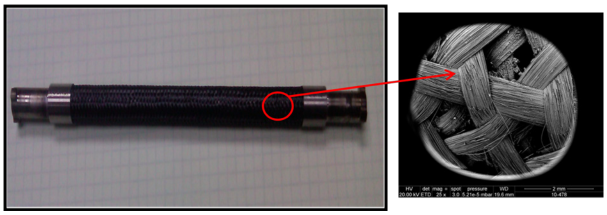


Figure 10. SiC Direct produces α-SiC that permits customization of the fibers. This tri-axial α-SiC overbraid on a metal tube demonstrates that SiC Direct fiber can be manufactured and formed in shapes to surround nuclear fuel-sized rods. There is exceptional potential for its use in nuclear reactors to protect fuel in reactor operating conditions, enhancing safe operations and significantly reducing the potential for fuel meltdowns (α-SiC does not degrade in radiation environments and has high temperature resilience up to 2700°C).

SiC Direct delivers the capability of producing α-SiC fibers, but is exceptionally enhanced by using INL's patented process, spray forming, in combination with conventional hot pressing or hot rolling. For example, **SiC Direct** permits the use of SiC and SiC/C fibers to be incorporated into aluminum matrices to form continuous fiber-reinforced aluminum matrix composites (CFRAMCs). This suggests that **SiC Direct** may be adaptable to many other structural uses. This is discussed more in Product Use, paragraph 12.

SiC Direct has been demonstrated, tested, and analyzed to prove that α-SiC can be economically produced in an environmentally friendly way. As important, INL researchers have advanced this "breakthrough" beyond discovery and initial application, proving scalability and commercial viability (already having produced α-SiC fibers in thousands of meters of length in a relatively small furnace). Their analysis reveals high quality production, low cost for affordability and availability, and exceptional insight to applicability (discussed in Product Use, paragraph 12).

11. SiC DIRECT – BUILDING BLOCKS

SiC Direct began as a conversation between two INL researchers, a materials scientist and a nuclear engineer. They were seeking solutions to a national laboratory challenge – selecting and/or creating new materials that solve a long list of requirements to improve operations, safety and manufacturing for nuclear reactors.

This super-material needed to be:

- high temperature resilient (above 2000°C) and effectively insulative,
- mechanically and thermally stable in radiation environments for long-term use,
- affordable (e.g. composed of and produced from relatively inexpensive materials),
- chemically inert and light weight,
- exceptionally strong and durable, and
- very hard and wear resistant.

So, the first building block in producing **SiC Direct** was the extensive career experience of INL scientist, engineer and entrepreneur Dr. John Garnier, who has worked his entire career in leading edge material research. He has had significant leadership roles in three major corporations (Standard Oil, E.I. DuPont de Nemours and Honeywell).

Partnering with him was a key, insightful and driven nuclear energy researcher, Dr. George Griffith, an experienced commercial nuclear reactor operator. Griffith was determined to find solutions to the international challenges revolving around the nuclear reactor material needs listed above.

These two proffered a series of choices in approaching the selection and/or creation of new materials. They sought to identify affordable materials which could enhance INL nuclear reactor design efforts, fuel designs and reactor operations. Starting by inventorying known materials and their qualities, they cataloged and reviewed manufacturing processes for all candidates.

Historically, the search for new materials or refinement of processes has produced exceptional materials. For example, aluminum was considered an exotic material when it was first produced in the early 1800's and was very expensive until refined manufacturing processes made it affordable. Today, aluminum daily commodity prices hover around one dollar per pound.

After their comprehensive review, the duo focused on alpha silicon carbide (α-SiC), which offered significantly variable material forms and qualities (e.g. 250 molecular polytypes are available permitting exploitation of the many attractive qualities inherent in the various crystalline forms to produce a wide variety of products). This was a challenge in itself because while α-SiC was known to exist; none has been produced commercially and is not available. Garnier had ideas about possible production methodologies based on his experience with silicon carbide in the 1980's and 1990's.

Seeking a simple process that was not capital intensive and produced affordable α-SiC fibers, the two researchers began to eliminate approaches and focused on plausible production methods. They quickly settled on trying a direct conversion approach, leveraging a small research furnace in one of INL's lab spaces. Garnier and Griffith went to Kevin McHugh in INL's Energy Efficiency and Industrial Technology department who had a 10 KW furnace with a six-inch chamber to try what became their patent-pending **SiC Direct** process. Their idea proved successful in directly converting carbon fiber sources into high quality, uniform α-SiC.

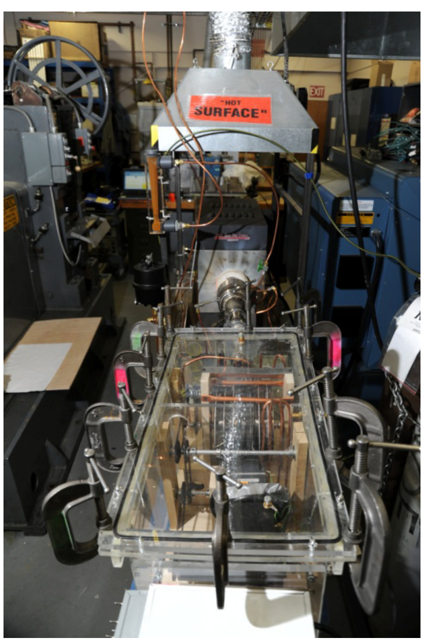


Figure 11. INL's 10-KW furnace was used to test the patent-pending SiC Direct process that directly converts carbon fiber materials into α-SiC fiber. Moving from an initial one-inch long fiber length, INL now produces α-SiC fiber thousands of meters in length. INL has produced continuous α-SiC fibers as long as a mile using the SiC Direct process, Figure 12, right.

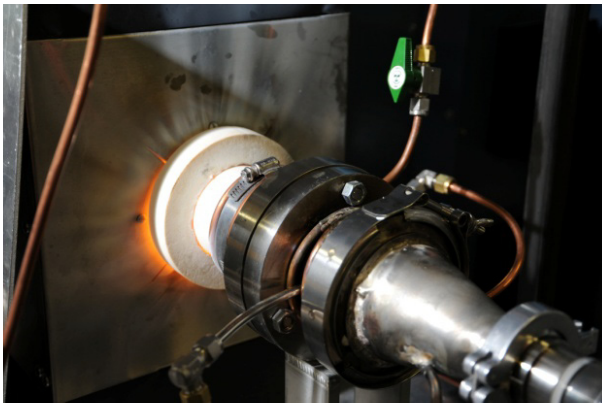
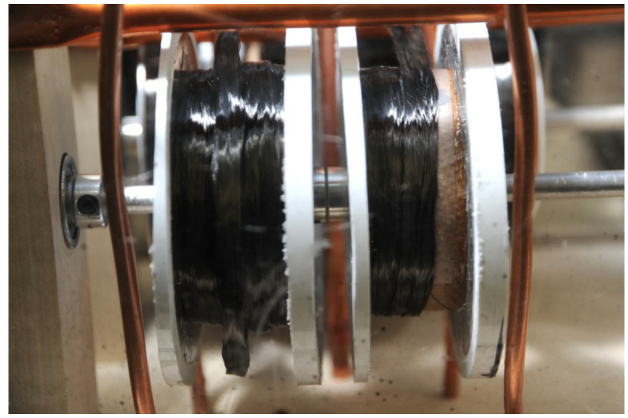
 

Figure 13. The carbon fiber is drawn through a furnace with silicon oxide vapor at about 1600°C and then coiled, Figure 14, right. After testing various temperature and draw rates, researchers learned that adjustments for varying quality of alpha silicon carbide fiber can be produced, depending on requirements.

Their first successful test produced a one-inch length of α-SiC fiber. The next attempts were calibrated and soon they produced a ten-foot length, which quickly gave way to production rates of five inches per minute that delivered α-SiC fiber at 3,600 inches (300 feet). Their goal was to produce a continuous, mile-long (e.g. 1,800 meters in length) converted α-SiC fiber. By Jan 2012, they have produced thousands of meters of α-SiC fiber.

Commenting on discovering this elegant and simple silicon carbide fiber production process, John Garnier said, "At INL it all came together, allowing us to develop the **SiC Direct** production method, because we had the resources of a national laboratory right there. It is amazing how well this method works in producing this super material that has so much potential."

Garnier and Griffith made extensive tests on the proprietary process and soon learned that **SiC Direct** production method exhibited robust control, while at the same time produced a uniform product of the highest quality. This enhanced its potential for successful scalability, which was proven by the production of much longer coils of SiC fiber (1,800 meters) and then expanding it to the batch processing of a wide variety of shapes. Large complex shapes and sizes are limited to the furnace size thus allowing this production method to be adapted to a wide range of commercial production needs.

Each researcher had specific areas of focus – Garnier research of new materials to advance manufacturing and product availability, Griffith materials that could make operating nuclear reactors safer, more efficient, easier to build and longer lasting. Their research continues to refine production of α-SiC fibers and finding applications that could make life better for people.

In the process they created new intellectual, patent-pending property for Idaho National Laboratory to make available for commercialization.

12. PRODUCT COMPARISON: COMPETITORS, MATRIX, IMPROVEMENTS, LIMITATIONS

A. Competitors

There is no known commercial manufacturing process to produce alpha silicon carbide (α-SiC) fibers, hence no commercial α-SiC fibers are available for sale anywhere.

Instead, the only commercial material available for comparison in price is beta silicon carbide (β-SiC). While these are high quality fibers in comparison to other materials available today, they are more limited than α-SiC fibers. In addition, β-SiC fibers require an expensive pyrolysis production process using silicon-containing polymers, such as polysilazane and polycarbosilane ($4,000/lb). For example, high quality Hi-Nicalon type S fibers imported from Japan cost $500 to $6,135 per pound and require a 3 to 6 month lead time. This high cost limits the use and application in purchasing β-SiC fibers, plus the purchaser must get in line for material delivery.

The β-SiC fibers materials are synthesized in an energy-intensive pyrolysis process using very costly polymers containing silicon. They are commercially available, including Sylramic fibers from COI industries in San Diego, CA and Hi-Nicalon and Hi-Nicalon S fibers from Nippon Carbon in Tokyo, Japan. These are distributed through COI Ceramics, Inc. (San Diego, CA) and Tyranno Fiber from Ube Industries, Ltd. (Tokyo, Japan), respectively.

INL's **SiC Direct** process compares very favorably to imported commercial β-SiC fibers with estimated production costs yielding SiC fibers for as low as $100 per pound (see paragraph 7). The demand for an economical SiC fiber has been estimated by DOE-Nuclear Energy to be as high as 120,000 lbs per year for nuclear applications alone.

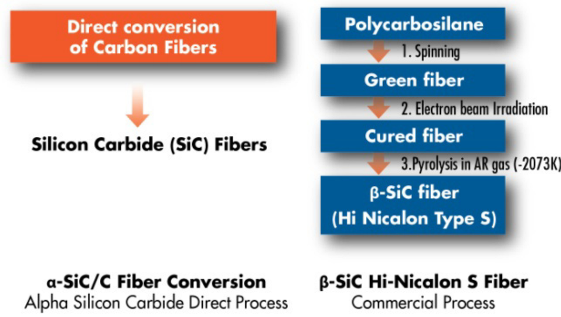


Figure 15. This block diagram illustrate the process simplification and comparison of current and conventional β-SiC fibers processing, highlighting the elimination of unit operations using SiC Direct.

Incorporation of α-SiC fibers into ceramic matrix composite and metal matrix composites could also substantially increase demand for products, if they can save weight, increase durability and strengthen materials useful in transportation and construction. It is estimated that metal matrix composites utilizing α- SiC may reduce vehicle chassis weight by as much as 20-30 percent.

During 2012, INL plans more development through additional α- SiC demonstrations producing thousands more meters of SiC fibers, fabricated at relatively low process temperatures by direct conversion of carbon fibers using low-cost reactant materials. Experimentation is planned to consider materials that can be used in many other products.

B. Competitive Comparison:

Carbon and Silicon Carbide Fiber Properties

Property	Units	Carbon	Carbon	Alpha SiC/C (from SiC Direct	Alpha SiC (from SiC Direct)	Beta SiC						
		PAN ASC4	PAN IM10	SiC + C (ASC4)	(SiC fully converted ASC4)	Nicalon	Hi-Nicalon Type S	Tyranno ZMI	Tyranno SA (SiC + Al)	Sylramic (SiC + TiB2+ B4C)	SCS-6 SiC/C	SCS-Ultra
Cross-Section		Round	Round	Round	Round + Hollow core	Round	Round	Oval	Oval	Round	Round	Round
Aspect Ratio		Continuous	Continuous	Continuous	Continuous	Continuous	Continuous	Continuous	Continuous	Continuous	Continuous	Continuous
Weave or Braid		Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	No
Weave or braid then convert		NA	NA	Yes	Yes	No	No	No	No	No	No	No
Diameter	um	6.9	7	4.4-7.1	Derived from C fiber 5-7.8	14	14	11	7.5 - 10	10	150	142
Density	g/cc	1.79	1.79	2.2	2.94	2.55	2.74	2.48	3.1	2.95	4.1	3.08
Maximum Service Tempera-ture	°C	2000+	2000+	Estimate 2000+	Estimate 2000+	1200	1800	1400	1400	1400	1400	1400
Thermal Conductiv-ity	W/m-K	6.83	6.14	TBD	TBD	7.77	7.77	2.52	64.6	45	not listed	not lsited
Elongation at Break	%	1.8	2.1	1.9	0.9	not listed	not listed	1.7	0.7	not listed	0.9	0.9
Thermal Expansion	PPM/°C	-0.63	-0.7	TBD May be similar to ASC4	TBD	3.9	3.9	4.0	4.5	not listed	4.1	4.1
Tensile Strength	MPa	4460	6964	3345	TBD	3000	2600	3400	2800		3,900	5,900
	ksi	647	1010	485	TBD	435	377	493	406	400	560	850
Tensile Modulus	GPa	221	303	166	TBD	210	420	200	380		380	415
	msi	32	44	24	TBD	30	61	29	55.1	45	56	60
Fiber Cost Estimate. Nicalon/ Tyranno $ based on Yen/$ ratio	$/lb	< $50	<$500	< $ 90	< $ 90	$ 500	<$6,315	< $1,000	<$4,000	< $1,000	< $2,000	< $2,500

C. Alpha Silicon Carbide Improvements

First and foremost, **SiC Direct's** continuous production process offers improvement in both the availability and cost of α-SiC fibers. As emphasized earlier, **SiC Direct** has demonstrated an innovative capability of producing these high quality fibers available and in significant quantities. Add to that an affordable price (e.g. as low as $100 per pound) and manufacturers could begin making new and better products for use by consumers, governments and international organizations.

In addition to availability and cost, one of the most important advancements is the variability of product that can be produced using this revolutionary process. Manufacturers could license the process or order specific products from component suppliers that may significantly enhance their competitiveness, manufactured products and energy savings.

For example, automakers may be able to reduce the weight of their vehicles (all types – diesel, gas and electric), increasing gas mileage. A 20 – 30 percent weight reduction in diesel or gas powered vehicles could translate into as much as 25 percent increase in fuel economy (e.g. 32 MPG becomes 40 MPG). Electric vehicles may well be able to go beyond the 25 percent increase in distances with each charge.

SiC Direct also permits further development of the alpha SiC fiber by expanding the use of carbon PAN fiber precursors, such as the Hexcel IM 9 and IM 10 carbon fiber. These types of fibers have significantly higher mechanical strength (1010 ksi) as compared to other carbon PAN fibers at 200 to 500 ksi).

Having demonstrated the ability to scale-up **SiC Direct's** production process permits more improvements to be made in production schemes. Batch processing could enable a wide variety of large and small formed products (e.g. fiber for braiding in power lines, truck axels, and larger-sized products). Coiled fiber line already has been produced into the thousand of meters during a single run.

One of the most impressive examples of **SiC Direct**'s ability to affect improvements can be seen in the manufacture and use of ceramic matrix composites and metal matrix composites.

Ceramic matrix composites (CMC) overcome the technical problems associated with conventional monolithic ceramics such as alumina, silicon carbide, aluminum oxide, silicon nitride or zirconia.

Monolithic ceramics can fracture easily under mechanical or thermo-mechanical loads resulting from cracks initiated by small defects, especially in thin wall part form. Particles such as monocrystalline wiskers or platelets can be used and embedded into the matrix, however, they yield limited improvement.

With the development of carbon, aluminum oxide and beta SiC fibers, a dramatic ten-fold increase in fracture toughness and mechanical strengths have been demonstrated. These resulted in new applications as matrix forming processes such as chemical vapor infiltration and use of matrix forming polymers became available.

CMCs are used in auto parts like brake disks, mechanical slide bearings in a variety of pumps, aeronautical thrust control flaps, friction systems, components for fusion and fission nuclear reactors, and many other industrial applications.

Continuous fiber reinforced ceramic matrix composites are known as CFRCMCs. With the new alpha silicon carbide fibers CFRCMCs may be made with nearly any ceramic matrix material, such as alpha SiC, silicon nitride, boron carbide, aluminum oxide, etc. These new materials available in affordable alpha SiC fiber products may add a dynamically new and exciting addition to today's CMCs.

In addition, metal matrix composites (MMC) are composite materials having at least two constituent parts: a metal along with another metal or a different material, such as ceramic particles or fibers. MMCs have many applications including drills, tank armor, auto brakes and drive shafts, aircraft landing gears, chip modules in electronics, ceramic-to-metal seals, fuel cells and much more.

MMCs are made by dispersing a reinforcing (ceramic) material into a metal matrix. The reinforcement surface can be coated to prevent a chemical reaction with the matrix as is needed with carbon fibers. Carbon fibers are commonly used in aluminum matrix to synthesize composites with lower density and higher strength. The reinforcement in the MMC also may not always be needed to function as a mechanical strength enhancement, but also is used to change physical properties such as wear resistance friction coefficient, or thermal conductivity.

Various processes such as metal spray forming for thin sections are used in forming MMCs and other composites. Regardless of the metal forming process used for making fiber reinforced MMCs, **SiC Direct** offers a lower cost, lower density alternative to these and other coatings, especially for use in nuclear application where boron must be minimized due to boron's high neutron cross section adsorption characteristics.

More Research. Garnier and Griffith already have embarked upon research to evaluate other potential carbon sources as feedstock for alpha silicon carbon fibers and pre-forms. Their studies include looking at high quality fibers to determine whether quality improvements can be achieved and at what cost. More on this will be published in the coming months.

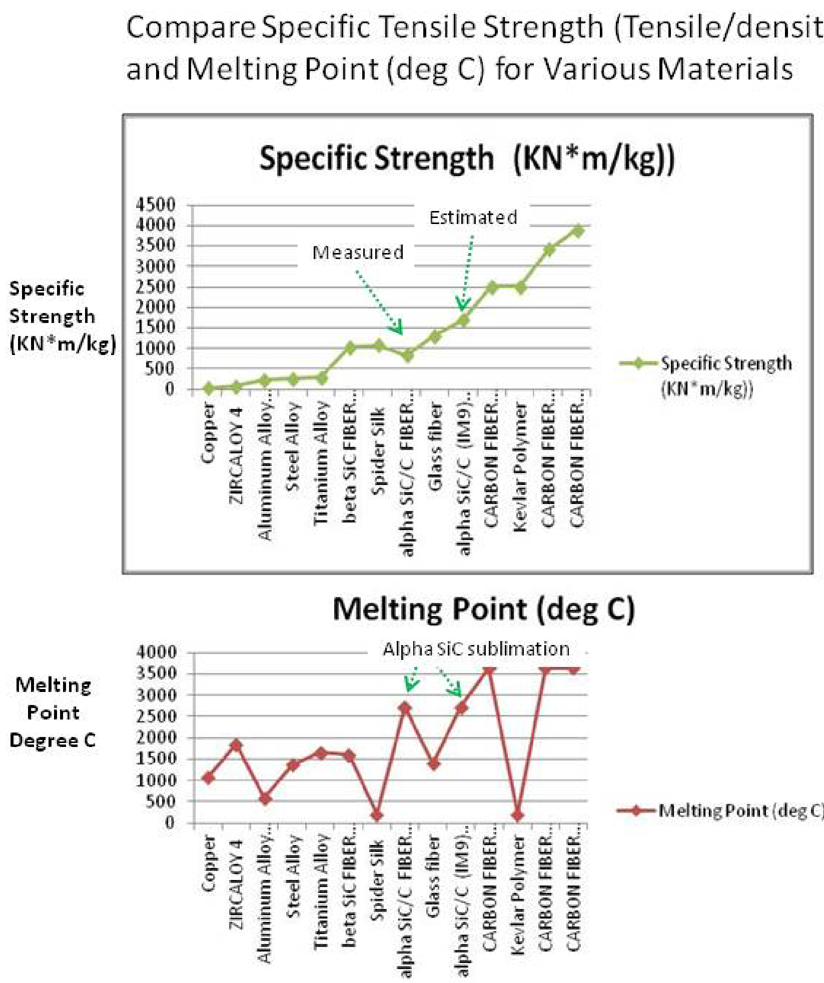


Figure 16. Comparison of the specific strength of alpha SiC with other materials

Strength of alpha SiC with other materials. The tensile strength of alpha SiC/C fiber is derived from the tensile strength of the carbon PAN fiber used. To date six different forms of PAN and pitch carbon fibers have been converted with the resulting alpha SiC/C strengths being typically 20 to 30% less than the starting carbon fibers.

Figure 16 compares the specific strength of selected materials and their respective melting points to alpha SiC and beta SiC fiber, carbon fibers and other metals. Alpha SiC/C fiber has higher specific strength by a factor of 5 to 8 than either aluminum, titanium or zirconium based alloys. Compared to carbon fiber however, alpha SiC is lower by factor of 3 to 4 as compared to the starting Hexcel AS2 and AS4 carbon fibers.

More tests are under way to evaluate the direct conversion of advanced carbon fibers, such as Hexcel IM10 fibers having specific strength of 1010 KSI. Using the trends noted on other carbon fibers converted to date, it is estimated that the resulting alpha SiC/C fiber specific strengths conservatively is expected to be 500-800 KSI. Thus, as the alpha SiC and alpha SiC/C fibers have a have a high melting point (2730^0C), this new ceramic fiber materials has the potential for use in many ultra high temperature applications above temperatures which the current family of beta SiC fibers are known to be limited to (e.g.1600^0C, see black box in figure 16).

D. Limitations

Current production using the **SiC Direct** continuous conversion has been limited by the current use of a laboratory size tube furnace (3 inch outside diameter with a 6 inch hot zone) and current stage of process development. At the moment, direct conversion rates are limited to less than 30 tows processed simultaneously reaching nominal 20 pounds per hour or 160 pounds per day using this small R&D furnace. Already, testing of more advanced methods, even within this small configuration, shows promise of producing multiple silicon carbide fiber strands, simultaneously.

However, moving to a larger configuration tube furnace or using multiple furnaces, conversion rates to hundreds of pounds per hour are feasible with minimal capital investment. A proposed manufacturing plan estimates a small 10,000 square foot facility and a two shift capability can provide 100,000 pounds of fiber at an estimated manufacturing cost as low as $100 per pound and reducing further with large production possibilities. Once licensed, more refinements in this industrial process are anticipated.

Testing for the production of other fiber materials is underway, including the development of materials such as boron carbide fiber (B4C) that has applications in the nuclear industry as a neutron adsorber or in many structural applications in aerospace due to its low density 2.46 gm/cc. These fiber materials may be able to replace or mimic critical materials that are scarce, expensive and limited in availability. If successful, this advancement could assist the nation in overcoming a major difficulty for ongoing and future manufacturing.

Currently, alpha **SiC Direct** is limited by diameter and type of commercial sources of carbon fiber presently available. Other forms of carbon fiber are being developed, including smaller and larger diameters of carbon fibers. As the starting diameter of the carbon is varied, the diameter of the completely converted alpha SiC fiber hollow fiber will change. Currently, alpha SiC fibers have hollow central diameter holes of about 1 micron when starting from the 6-7 micron carbon filament (figure 7).

Other research fibers are becoming available ranging from 3 micron to 100 micron diameters. Scaling the alpha **SiC Direct** process yields hollow cores from less than 0.5 micron to 20 micron. The hollow feature of the all alpha SiC fibers make them an ideal engineering material for hundreds of new product applications ranging from gas to bio filtering.

13. PRODUCT USE

A. Principal Application and Benefit

Silicon carbide (SiC) is a refractory ceramic with a vast array of commercial applications because of its exceptional diversity of qualities.

These super materials offer tremendous advantages in industry and product manufacturing, including:

- Very high strength to weight ratio (1/3 density of steel and 3-6 times stronger to reduce weight and increase strength),
- Thermal resistance in high temperature conditions (used in high heat manufacturing, aeronautics, gas turbines, etc),
- Configurable electrical properties from resistance to conductance for use in electronics,
- Superior resistance to nuclear gamma and neutron irradiation for use in nuclear reactors, and
- High corrosion resistance to acids such as HF and HNO3 acids.

SiC Direct, developed by Idaho National Labs (INL) researchers, offers an ***"amazing breakthrough"*** patent-pending technology that fabricates silicon carbide fiber super-materials, called alpha silicon carbide (α-SiC) fibers, in a simple and affordable direct conversion process. It uses inexpensive raw materials (carbon fibers, silicon granules and silicon dioxide sand) and relies upon standard capital equipment that is affordable and used by industrial manufacturers operating today.

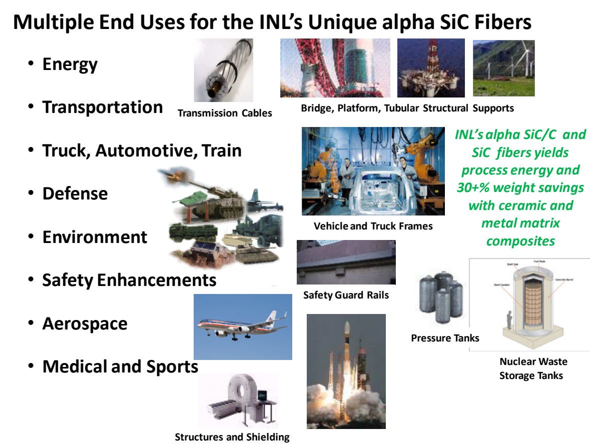
Further, **SiC Direct** produces virtually no waste requiring disposal. It produces SiO (SiC + CO) gas that leaves a trace amount of CO (carbon monoxide). The exhaust can be captured and recycled through a calcium oxide process from which calcium carbonate ($CaCO_3$) can be produced for use in other products. $CaCO_3$ is useful in neutralizing acids, adhesive and ceramic production and other industrial uses. Diverting this to other product is not prohibitive, but rather easy.

First and foremost, since α-SiC fibers are not available from any source today, **SiC Direct** makes these important new materials available.

As noted before, only β-SiC fibers have been available and those are expensive, ranging in cost from $500 to $6,000 per pound. They employ expensive and capital intensive processes to make them.

Secondly, **SiC Direct** makes α-SiC fibers affordable, which enhances their usability. The estimated cost may be as low as $100 per pound, increasing the potential for their abundance and wide use in many industries.

With the wide variety of applications, the abundant and affordable α-SiC fibers are expected to be used in many areas. The areas expected to benefit the most and the soonest from this new super material are transportation, energy, defense-aerospace, environment and industrial manufacturing. Other areas also could benefit.



Transportation. The transportation industry probably could benefit the soonest and the most, at least initially. As noted earlier, α-SiC fibers offer so many advantages to this industry including:

- As much as a 30 percent weight savings, increasing vehicle miles per gallon performance and greater distances for electrified vehicles,
- Stronger components with that weight reduction (3-6 times stronger than steel) which may increase personal safety to all drivers and passengers, and
- Increased availability of components made using critical materials such as α-SiC fibers may be of exceptional assistance in creating substitute materials.

An initial list, and certainly not comprehensive, of the types of products that could assist in transportation include:

- Vehicle frames, engine blocks, exhaust systems and safety panels,
- Lighter and stronger rail cars and semi-truck trailers,
- Energy absorbing materials for collisions,
- Specialized materials for magnet and battery production, especially catalytic surfaces, and
- Much, much more.

Energy. A second industry in line to benefit the most is energy production for some of the same reasons, but others too. In addition to the qualities listed for transportation, α-SiC fibers also may be configured to be either an insulator or conductor of electricity. They also are thermally shock resistant, resilient in high temperatures, wear resistant and have a high strain capacity. These additional qualities expand their use in the energy industry.

An initial list, and again certainly not comprehensive, of applications within the energy industry might include:

- Lighter and stronger power lines that are safer and more efficient at delivering electricity,
- Lighter and stronger power lines with a lower coefficient of thermal expansion may reduce the number of towers needed to support new power lines
- Fluid cracking catalysts in oil refining,
- Technology transitions for high-efficiency lighting,
- Components for use in currently operating nuclear reactors to extend their use,
- Permanent magnets for wind turbine generators,
- Improved composites for wind turbine blades
- Turbine engines for stationary power generation, and
- Much, much more.

Defense-Aerospace. Related in many ways, though also very different, defense-aerospace could benefit from most of the above (transportation and energy applications), as well as in other ways.

The Defense-Aerospace partial list might include:

- Blast mitigation materials for body armor, vehicle protection, and building reinforcement to increase personnel safety,
- Lighter and stronger ships, aircraft and engines for speed and efficiency,
- Weapon systems electronic components from critical materials,
- Thermal resistant materials for rockets (e.g. NASA, telecommunications satellites, etc),
- Components for shipboard nuclear reactors in submarines and aircraft carriers, and
- Much, much more.

Environment. An important complement to energy is environmental applications for α-SiC fibers. This is an area where applications are just beginning to be identified. Some applications that may suggest many others, include:

- Overall reduction of carbon emissions associated with aluminum and other manufacturing,
- Safer storage of corrosive and dangerous materials (e.g. nuclear waste, acids, etc),
- Better, more efficient, longer-lasting auto exhaust and pollution control systems,
- Potential use of captured carbon for production of α-SiC fibers, and
- Much, much more.

Industrial Applications. This list could be endless and this submission doesn't even attempt at making a lengthy list. It is clear from the work already advanced that α-SiC fibers may have a multitude of applications in industry. Just some of those might include:

- Use of smaller amounts of expensive and valuable materials like aluminum, zirconium, and titanium (e.g. replaced by α-SiC fibers saving weight and material while strengthening products),

- Aluminum based composites for stronger buildings, bridges, guard rails and other structural applications (e.g. earthquake zones),
- Industrial processing tubes and high temperature fiber membrane supports for catalysis and particulate filters,
- Pumps, heaters, boilers, and pressure vessels,
- Wear resistant materials in mining and manufacturing,
- Stronger, corrosion resistant wires and cables, and
- Much, much more.

Benefits. Also characteristic of or added to these principal applications is a lengthy list of other benefits from **SiC Direct** that could make a difference. Among these are:

- **Energy Consumption.** Fabrication of α-SiC fibers consumes 60 percent less energy to β-SiC commercial polymer processing.
- **Materials.** This process uses inexpensive materials such as silicon granules and sand with available carbon fiber materials (e.g. reduces cost considerably, as low as $100 per pound).
- **Capital Investment.** Relatively small investment is required because readily available electric or induction furnaces can be used to generate silicon oxide vapor to preheat and convert carbon fibers to SiC. Other processes for product improvements are accessible and reasonably priced.
- **Virtually No Waste.** Further, **SiC Direct** produces virtually no waste requiring disposal. The process produces SiO (SiC + CO) gas that leaves a trace amount of CO (carbon monoxide).

SiC Direct delivers a unique and new material form of alpha silicon carbide fiber, which has direct applications in all these areas and more. It provides an affordable, low-cost and better alternative to the more expensive beta SiC fibers and products in use today.

Additionally, the identification of uses for alpha SiC fibers has just begun. It enables manufacturers to reduce the amount of metals needed to produce even better products. This could apply to such materials as aluminum, zirconium, titanium, hafnium, tantalum, niobium, and, tungsten, which are used in many industrial applications today.

The key benefit is that **SiC Direct** is a robust, yet simple, manufacturing process that produces alpha SiC fibers at a very affordable cost. Reduced cost and readily available materials available is necessary for any innovative product to be rapidly adopted by industry.

B. Additional Market Applications

As described in the earlier text, it is not possible to describe all other applications as requested.

However, given the low projected capitalization required for **SiC Direct** manufacturing in the U.S., it is believed that many more applications may be discovered and employed for this new form of alpha silicon carbide fibers. Currently, expected demand is about 200,000 pounds per year or larger within five years. An increasing rate of fiber manufacturing could enable expansion to include ceramic and metal matrix composite. So, many more hardware applications for the product are expected. For example: In March 2012, GE Aviation announced a major joint venture with Nippon Carbon (Japan) to increase the supply of beta SiC fibers for use in the new GE series Lean Energy Efficient Propulsion (LEEP) turbine engines for the commercial aviation market. The beta SiC fibers enable enhanced fuel efficiency with reducing burned fuel by-product emissions.

Industrial Market. There is significant demand for ceramic and metal matrix composites today. However, these are limited by the very high cost of beta silicon carbide fibers ranging from $500 to $6,315 per pound, depending on beta SiC fiber quality.

For many applications the alpha SiC/C fibers make an ideal substitution candidate with a high strength to weight ratio greater than the all beta SiC fibers. The projected market for all SiC fibers is conservatively estimated to be more than 1,000,000 pounds of fiber per year within ten years. In fact, it is anticipated to have a special impact on green power technologies, as well as traditional power using industries.

Nuclear Energy Market. More research is planned for using alpha silicon carbide fibers and pre-forms in nuclear fuel applications, possibly fuel protection materials, reactor components and other applications for this important energy industry.

Summary. The INL scale-up manufacturing plan conservatively estimates the market for low-cost SiC fiber to grow to 200,000 pounds per year within 5-plus years as driven by industrial, energy, defense, and nuclear industry needs. The improved economics of aluminum spray forming versus conventional processing are well known and result from the very high solidification rate that eliminates segregation. The benefits of weight reduction and higher performance composite materials could accelerate market growth while significantly enhancing U.S. competitiveness and job creation.

Commercial applications of SiC fibers include stationary power turbine engines, radiant burners, diesel particle trap filters as well as its high strength to weight ratio for use in high voltage transmission cables to maximize distance between transmission towers. Today use of SiC fibers in these applications are limited by the current very high cost of beta silicon carbide fibers.

The direct conversion process for alpha SiC could be a significant step forward in developing affordable SiC fiber reinforced composites.

Military Market: The development of silicon carbide as a candidate high temperature structure fiber began in the mid 1980's through various Air Force and Navy programs for turbine engine and other applications. Some of these programs continue today. The military has demonstrated continued interest in extending high temperature structural capabilities for silicon carbide fiber as well as lowering cost. The U.S. military market application for silicon carbide fiber is large including turbines engines (> $1B) and afterburner components (> $1B). Additionally, military applications for silicon carbide fiber can extend to emissions control and armor.

The U.S. military uses of silicon carbide are numerous spanning from monolithic ceramic form for armor plate to high temperature applications of beta SiC fiber reinforced SiC ceramic composites in aerospace applications such as turbine engines and rocket nozzles.

14. SUMMARY: ALPHA SILICON CARBIDE DIRECT – IMPORTANCE, BENEFITS, FINANCIAL IMPACT

An Important Material Process

INL's **Alpha Silicon Carbide (SiC) Direct** is an important "breakthrough" that offers the potential for a new era in ceramic materials using alpha silicon carbide (α-SiC) fibers.

Previous attempts to make α-SiC fibers using various processing methods have not been successful. An elegantly simple process, **SiC Direct** continuously produces α-SiC fibers in abundance and at affordable cost.

INL's patent-pending process avoids the expense and more difficult manufacturing requirements of the far more exotic, but less capable material called beta silicon carbide (β-SiC). In fact, materials specialists could begin manufacturing α-SiC fibers almost immediately, because the process uses readily available commercial furnaces and far less expensive raw materials (widely available carbon fibers, silicon granules and silicon dioxide sand).

Researchers at INL already have produced thousands of meters of α-SiC fiber. They also have adapted it to batch process pre-woven or braided carbon forms to manufacture sheets, three dimensional forms and many other shapes. It also may be scalable for use in large manufacturing furnace operations.

Further, β-SiC materials currently are only available from overseas sources in very limited runs. Material must be ordered 3-6 months in advance and costs range from $500 to $6,000 per pound, depending on the materials required for production. For these reasons, β-SiC materials have been used very sparingly in highly specialized manufacturing efforts.

By comparison, **SiC Direct** continuously and directly converts carbon fiber into α-SiC materials at a cost that may be as low as $100 per pound, when producing 100,000 pounds or more.

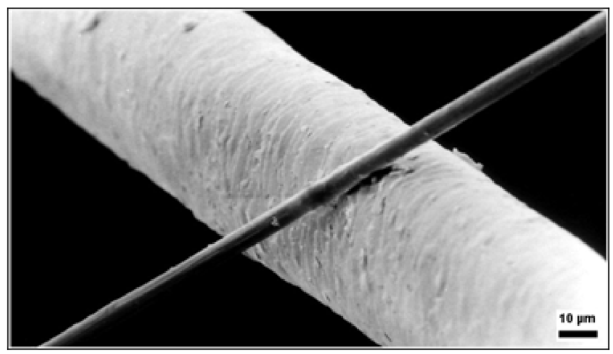
Nearly as important as INL's breakthrough production process is α-SiC's chemical, mechanical, and thermal properties, which make it an attractive degradation-resistant high temperature reinforcing ceramic for use in energy, transportation, aerospace, defense, industrial and nuclear applications (see paragraph 13).

α-SiC (and its α-SiC/C form) outperforms β-SiC with an impressive range of qualities – exceptional variance in compositional forms and application, high thermal shock resistance, high temperature resilience (sublimation temperature up to 2730°C), higher tensile strength and durability that reinforces metal matrix and ceramic matrix composites, higher thermal conductivity, chemical inertness, light weight, corrosion and wear resistance, durable abrasiveness, mechanical and thermal stability in radiation environments, and more – as the nomination text details (paragraphs 9, 10).

α-SiC/C fiber have been converted from 50,000 and 3,000 filament carbon tows. These fibers have color from irradiant green, purple to blue as a result of light scattering from sub-micron SiC grains and residual elements, such as N_2 (green).

Benefits. Affordable and available α-SiC fibers from **SiC Direct** could make a big difference. In general, four key benefits result from **SiC Direct** and use of α-SiC, including:

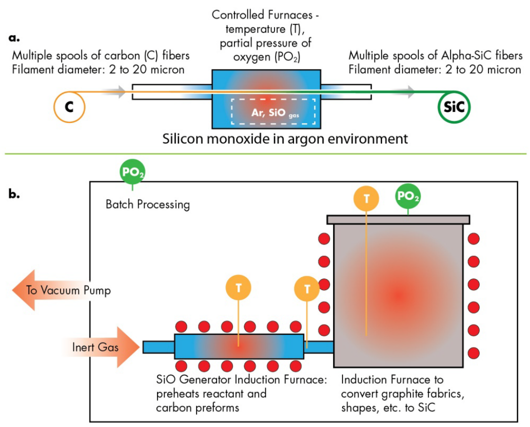
- **Energy Consumption.** Fabrication of α-SiC fibers consumes 60 percent less energy than β-SiC commercial polymer processing. Also, it saves energy in producing reinforced sheet materials and three-dimensional pre-forms.
- **Materials.** This process uses inexpensive materials such as silicon granules and sand with available carbon fiber materials.
- **Capital Investment.** No exotic materials are required for **SiC Direct.** Inexpensive commercially available electric or induction furnaces can be used to generate silicon oxide vapor to preheat and convert carbon fibers to α-SiC.
- **Virtually No Waste.** The **SiC Direct** process produces virtually no waste, but uses SiO (SiC + CO) gas that leaves a trace amount of carbon monoxide that can be recycled.
- These beneficial qualities apply to the **SiC Direct** process and the varied applications of α-SiC materials (detailed in paragraph 13) in energy, transportation, aerospace, defense, industrial, nuclear applications and other, yet to be identified, applications.



A single 6-micron diameter carbon pan fiber filament is positioned on top of a human hair for comparison. INL's α-SiC Direct process has demonstrated conversion with tows containing 3,000-50,000 filaments.

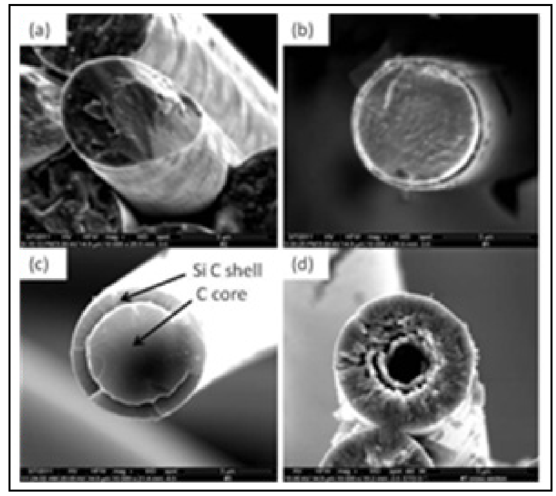
The Process - Revisited. A mixture of silicon dioxide (SiO_2) with silicon (Si) is heated in a ceramic crucible using a high temperature tube furnace to generate an oxidative SiO vapor. Commercially available polyacrylonitrile (PAN)-based carbon fiber tows are drawn through the furnace; thus, exposed to SiO vapor using an argon carrier gas at elevated temperatures (up to 1600°C). The carbon fibers convert into α–SiC, while being drawn through the furnace. The fiber draw rate (0.1 to 10 inches per minute) can be adjusted to vary local fiber reaction time with SiO to achieve a variable percentage conversion.

This direct process continuously converts carbon filaments into SiC fiber, beginning at the exposed surface of the filaments to form a shell and then progressing through the entire fiber. As dwell time is increased, the fiber is completely converted to SiC and adopts a tubular geometry.



SiC Direct converts carbon fibers to α-SiC:
(a) Continuous direct conversion to α-SiC fibers,
(b) Continuous batch conversion of fibers, fabrics, and other pre-form shapes.

This process is suitable for making both SiC and SiC/C fibers at high production rates.



Scanning Electron Microscope photomicrographs of carbon fibers during SiC Direct conversion process to α-SiC form.
(a) Unconverted carbon fibers, (b) Partially converted carbon fiber (α-SiC/C) with ~200 nm thick SiC shell, (c) Partially converted C fiber with 1 micron thick SiC shell. (d) Fully converted SiC tube.

SiC Direct saves energy, uses inexpensive materials, is environmentally friendly and requires limited capital investment. It also makes available at affordable prices a revolutionary, super fiber material so it is ready for use in redesigned and longer lasting products

This continuous direct conversion process delivers two products, fibers and three-dimensional forms. α-SiC/C fiber-based materials could propel America and the world into a new age of manufacturing and efficiency.

15. AFFIRMATION

By uploading this form to *R&D Magazine* I affirm that all information submitted as a part of, or supplemental to, this entry is a fair and accurate representation of this product.

Submitter's signature ______________________________

APPENDIXES

APPENDIX A: Submitter Information

APPENDIX B: Development Team Information

APPENDIX C: Fact Sheet

APPENDIX D: Letters of Support

APPENDIX E: Patent Abstracts

APPENDIX F: Video Script

Other supporting materials as identified – papers, presentation in a separate document.

Appendix A

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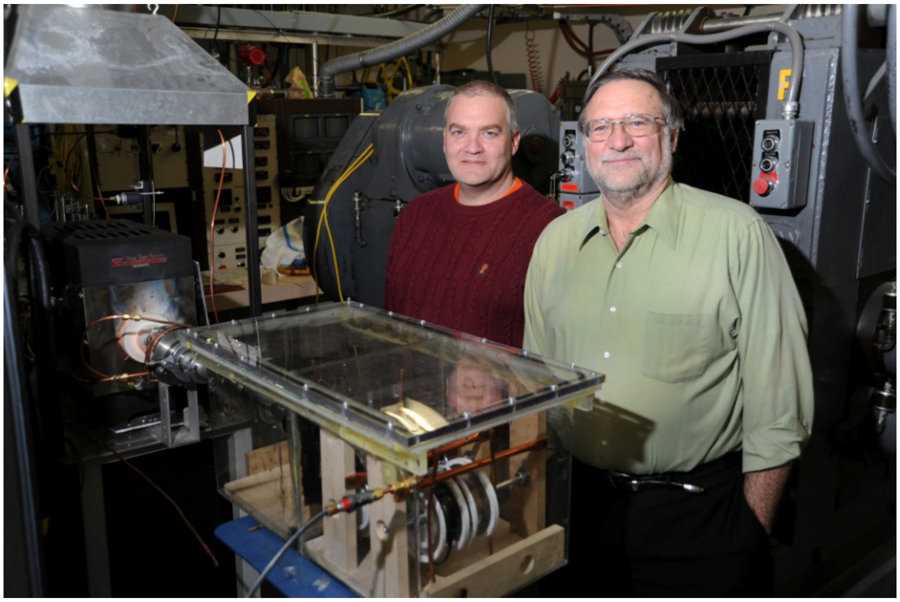
Appendix B

Development Team Information



George Griffith (left), John Garnier (right)

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Appendix C

Fact Sheet

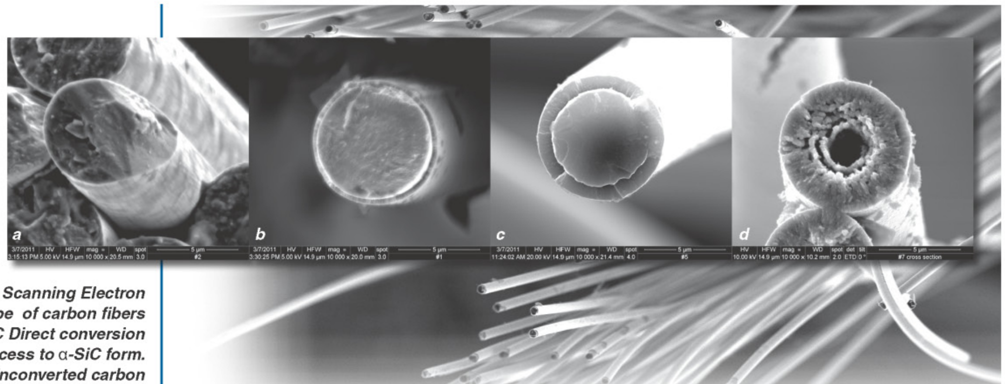

Figure 1: Scanning Electron Microscope of carbon fibers during SiC Direct conversion process to α-SiC form. (a) Unconverted carbon fibers, (b) Partially converted carbon fiber (α-SiC/C) with ~200 nm thick SiC shell, (c) Partially converted C fiber with 1 micron thick SiC shell. (d) Fully converted SiC tube.

Alpha Silicon Carbide Direct

Producing A New Ceramic Fiber Reinforcing Material

Human history has had many identifiable ages - Stone, Bronze, Steel and Space. Soon, there may be the Alpha Silicon Carbide Fiber Age.

Silicon Carbide – Tomorrow's Age?

Today, the chemical, mechanical, and thermal properties of silicon carbide (SiC) fiber make it an attractive degradation-resistant high temperature reinforcing ceramic for use in energy, transportation, aerospace, defense, industrial and nuclear applications. SiC fibers are now possible in two forms: beta SiC (β-SiC, cubic phase) and the new alpha SiC fibers (α-SiC, hexagonal phase).

Previous attempts to make α-SiC fibers using similar processing methods have not been successful. For comparison, the beta (β-SiC) materials currently are only available from overseas sources. Production of β-SiC is very limited and expensive. Materials must be ordered 3-6 months in advance at costs up to $6,000 per pound.

β-SiC materials are used in high temperature semiconductors, abrasives and wear components, mirrors, heating elements, armor and as fiber in reinforced composites for turbine engines, rockets, and other high-end performance metal and ceramic matrix composites.

α-SiC/C, α-SiC outperforms β-SiC.

The various forms of α-SiC fiber ceramic material can outperform β-SiC fiber by:

- Varying in compositional forms and application, α-SiC has an inherently higher thermal shock resistance, including high temperature resilience with no phase change to the sublimation temperature (up to 2730°C),
- Having higher tensile strength and durability as a reinforcing fiber in metal matrix and ceramic matrix composites (true for α-SiC/C),
- Providing higher thermal conductivity,
- Having high chemical inertness and light weight (true for α-SiC/C),
- Delivering greater mechanical and thermal stability in radiation environments,
- Exhibiting extreme hardness with better fiber filament "strain-to-failure" performance (e.g. surpasses stretching of 2 percent and exceeds 10 percent in a multi-filament tow form),
- Resisting corrosion, wear and/or abrasiveness,
- Performing as an excellent electrical conductor or insulator, depending on its configuration.

SiC Direct

Now, Idaho National Laboratory (INL) researchers have developed a processing breakthrough called SiC Direct that fabricates α-SiC in continuous fiber forms as the reinforcement phase for a palette of new fiber super-materials. This simple and efficient direct conversion process

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Appendix C

Fact Sheet

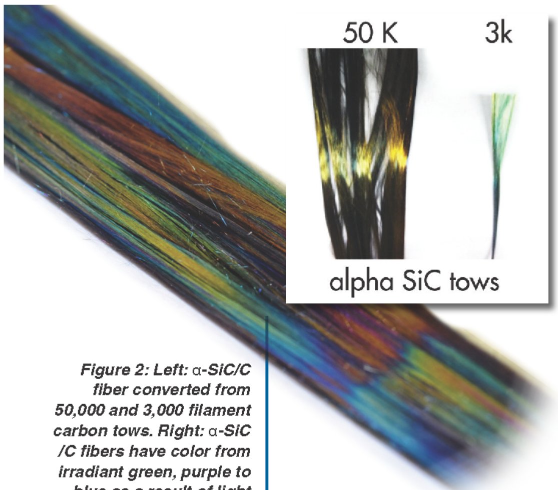

Figure 2: Left: α-SiC/C fiber converted from 50,000 and 3,000 filament carbon tows. Right: α-SiC /C fibers have color from irradiant green, purple to blue as a result of light scattering from sub-micron SiC grains and residual elements, such as N2 (green) .

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uses inexpensive raw materials (carbon fibers, silicon and silicon dioxide powder) to produce α-SiC for as little as $100 per pound.

INL's patented-pending technology is a continuous process that manufactures α-SiC fibers, coiled on spools in lengths up to a mile (e.g. 1,800 meters) or longer. Fiber can be produced using various sources of commercial small diameter (3 to 100 micron) carbon filaments in tow form (see photo), which then can be converted and braided into desired patterns.

SiC Direct also can batch process pre-woven or braided carbon forms to manufacture sheets, three dimensional forms and many other shapes. It also may be scalable for use in large manufacturing furnace operations.

Benefits

Affordable and available α-SiC fibers from SiC Direct will make a difference.

- **Energy Consumption.** Fabrication of α-SiC fibers consumes 60 percent less energy compared to β-SiC commercial polymer processing.
- **Materials.** This process uses inexpensive materials such as silicon granules and sand with available carbon fiber materials.
- **Capital Investment.** Inexpensive readily available electric or induction furnaces can be used to generate silicon oxide vapor to preheat and convert carbon fibers to SiC. Other processes for product improvements are accessible and reasonably priced.
- **Virtually No Waste.** SiC Direct produces virtually no waste. The process produces SiO (SiC + CO) gas that leaves a trace amount of carbon monoxide.

The Process

A mixture of silicon dioxide (SiO_2) with silicon (Si) is heated in a ceramic crucible using a high temperature tube furnace (Figure 4) to generate an oxidative SiO vapor. Commercially available polyacrylonitrile (PAN)-based carbon fiber tows are drawn through the furnace; thus, exposed to SiO vapor using an argon carrier gas at elevated temperatures (up to 1600°C).

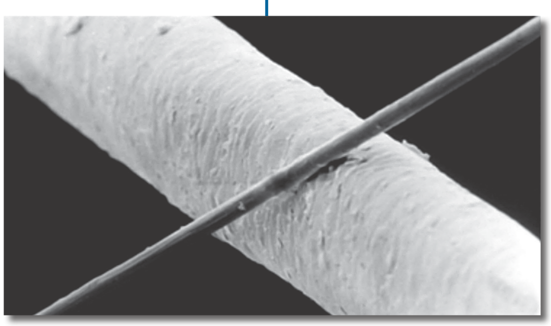

Figure 3: A single 6-micron diameter carbon pan fiber filament is positioned on top of a human hair for comparison. INL's α-SiC Direct process has demonstrated conversion with tows containing 3,000-50,000 filaments.

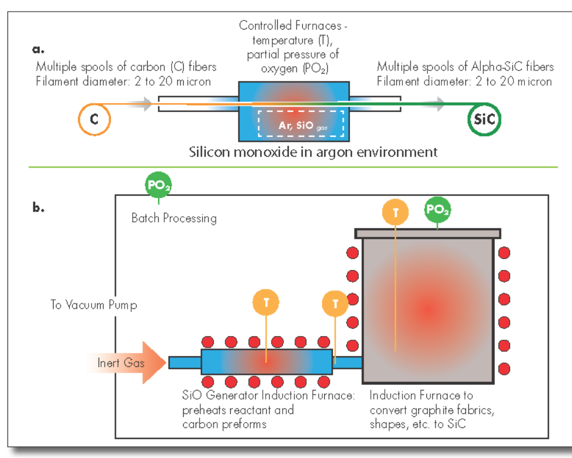

Figure 4: SiC Direct converts carbon fibers to α-SiC:(a) Continuous direct conversion to α-SiC fibers, (b) Continuous batch conversion of fibers, fabrics, and other pre-form shapes.

Appendix C

Fact Sheet

The carbon fibers convert into α–SiC, while being drawn through the furnace. The fiber draw rate (0.1 to 10 inches per minute) can be adjusted to vary local fiber reaction time with SiO to achieve a variable percentage conversion (see Figure 1).

This direct process continuously converts carbon filaments into SiC fiber, beginning at the exposed surface of the filaments to form a shell and then progressing through the entire fiber. A shell measuring ~200 nanometers (nm, 0.02 microns) takes about 4 minutes of high temperature exposure. At about 12 minutes of exposure, a sample with a 1 micron thick SiC shell is made. As dwell time is increased, the fiber is completely converted to SiC and adopts a tubular geometry (Figure 1d). This process is suitable for making both SiC and SiC/C fibers at high production rates.

Thousands of meters of silicon carbide single and multiple fibers already have been produced in demonstrated lab scale production testing.

SiC Direct Products

Many current products will be redesigned and array of new products will emerge as α-SiC material becomes available in quantity (Figure 7).

Transportation.

The transportation industry probably could benefit the soonest, including:

- Weight savings, increasing vehicle miles per gallon performance and greater distances for electrified vehicles,
- Lighter weight and stronger components (3-6 times stronger than steel) could increase personal safety to drivers and passengers, and
- Increased availability of components made using critical materials as α-SiC fibers may be useful in creating substitute materials.

An initial, and certainly not comprehensive, list of the product features desired in transportation industry includes:

- Improved vehicle frames, engine blocks, exhaust systems and safety panels,
- Lighter and stronger rail cars and semi-truck trailers,
- Energy absorbing materials for collisions,
- Specialized materials for magnet and battery production, catalytic surfaces, and more.

Energy

A second industry in line to benefit the most is energy production for some of the same reasons listed for transportation, but also α-SiC fibers can be configured to be either an insulator or conductor of electricity. They also are thermally shock resistant, resilient to high temperatures, wear resistant and have a high strain capacity. A partial product list might include:

- Lighter and stronger electrical power lines that are safer and more efficient, plus power lines with a lower coefficient of thermal expansion may reduce the need for towers supporting new power lines,
- Fluid cracking catalysts in oil refining,
- Technology for high-efficiency lighting,
- Components for use in currently operating nuclear reactors to extend their use,
- Permanent magnets for generators and improved composite blades for wind turbines,
- Turbine engines for stationary power generation, and more.

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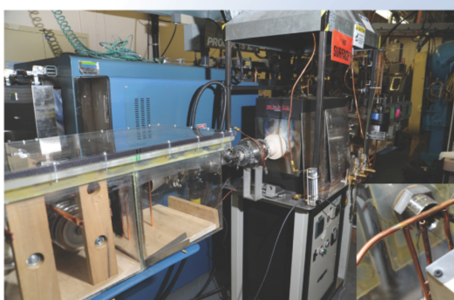

Figure 5: SiC Direct uses a high temperature furnace, above, to continuously convert carbon filaments into alpha silicon carbide fibers, which are coiled on spools, right.

Figure 6: INL's SiC Direct has produced α–SiC fibers in continuous coiled form with lengths up to a mile (e.g. 1,800 meters) or longer.

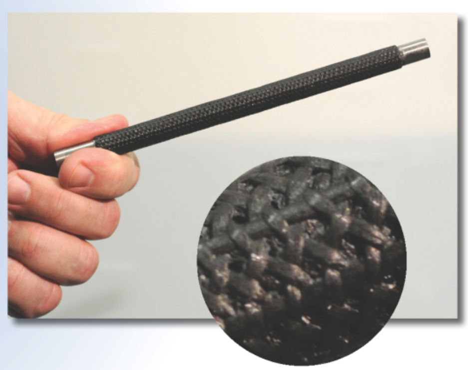

Figure 7: SiC Direct produces low cost α-SiC that permits customization of the fibers. This SiC overbraid on a metal tube demonstrates an advanced application for nuclear fuel-sized rods (patent pending). It is known that SiC will not degrade in radiation environments and has high temperature resilience.

Appendix C

Fact Sheet

NUCLEAR ENERGY

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Defense-Aerospace

Defense-aerospace could benefit from the transportation and energy applications, but also include:

- Blast mitigation materials for body armor, vehicle protection, and building reinforcement to increase personnel safety,
- Lighter and stronger ships, aircraft and engines for speed and efficiency,
- Materials for weapon systems electronics,
- Thermal resistant materials for rockets (e.g. NASA, telecommunications satellites, etc.),
- Components for shipboard nuclear reactors in submarines and aircraft carriers, and more.

Environment

An important complement to energy is environmental applications for α-SiC fibers, an area where applications are just beginning to be identified, including:

- Reduction of carbon emissions associated with aluminum and other manufacturing,
- Safer storage of corrosive and dangerous materials (e.g. nuclear waste, acids, etc),
- Better, more efficient, longer-lasting auto exhaust and pollution control systems,
- Potential use of captured carbon for production of α-SiC fibers, and more.

Industrial Applications.

This list could be extensive with multiple applications, including:

- Use of smaller amounts of expensive and valuable materials like aluminum, zirconium and titanium (e.g. use α-SiC/C fibers for high strength, while saving weight and material),
- Aluminum-based composites for stronger buildings, bridges, guard rails, other structural applications (e.g. earthquake zones),
- Industrial processing tubes and high temperature fiber membrane supports for catalysis and particulate filters,
- Pumps, heaters, boilers, and pressure vessels,
- Wear resistant materials in mining and manufacturing,
- Stronger, corrosion resistant wires and cables, and more.

SiC Direct saves energy, uses inexpensive materials, is environmentally friendly and requires limited capital investment. It also makes available at affordable prices a revolutionary, super fiber material so it is ready for use in redesigned and longer lasting products.

This continuous direct conversion process delivers two products, fibers and three-dimensional forms. α-SiC/C fiber-based materials could propel America and the world into a new age of manufacturing and efficiency.

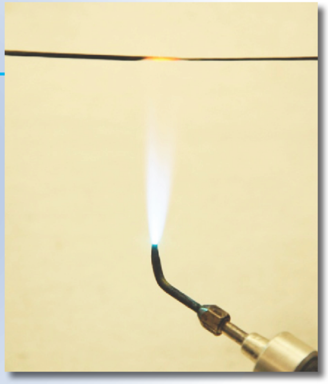

Figure 8: α–SiC fibers have high temperature resilience and do not experience phase change up to 2730°C, making the materials attractive for use in radiant heaters, boilers, pumps, and other high temperature conditions.

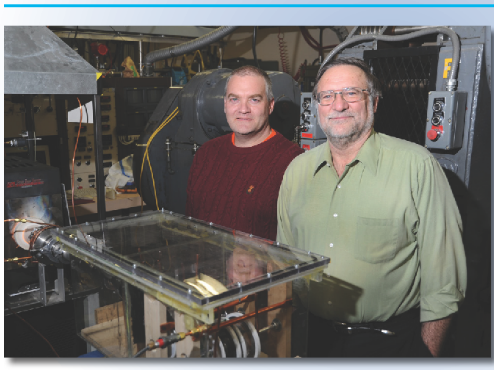

Figure 9: Inventors, left to right, George Griffith and John Garnier display process furnace, left, and α-SiC fibers on spools, right.

For more information

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John Garnier
Materials Scientist
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A U.S. Department of Energy National Laboratory

12-50164-01

Appendix D

Letters of Support

2307 OREGON STREET PO BOX 2566 OSHKOSH, WI 54903-2566
A DIVISION OF OSHKOSH CORPORATION

OSHKOSH DEFENSE

oshkoshdefense.com

February 14, 2012

Dr. John E. Garnier, Principal Investigator, Advance Nuclear Composite Hardware
National & Homeland Security Directorate
Idaho National Laboratory, P.O. Box 1625
Idaho Falls, ID 83415

Dear Dr. Garnier,

This letter is written in support of your recent advancement in the production and employment of alpha silicon carbide fibers and its nomination the Idaho National Labs for the R&D 100 Award for the reasons outlined below.

We have been following the rapid developments of this unique novel fiber, alpha Silicon Carbide as it offers a "cost effective" high temperature ceramic fiber reinforcement alternative for use in metal matrix and ceramic matrix composites as well as high temperature particulate filters and traps. We have read your technical paper presented at the ASME Symposium September 2011 on Small Modular Reactors held in Washington, DC and are reviewing your recent January 2012 presentation at the 36th Annual Conference on Composites, Materials, and Structures hosted by the United States American Ceramic Association. As we become more familiar with this new process technology to produce silicon carbide fibers by a simple direct conversion process at Idaho National Laboratory, I am encouraged by the prospects of a truly low cost ceramic reinforcement fiber for use in metal matrix composites materials. Use of this type of fiber reinforcement will read directly to higher performance, stronger and lighter weight structural materials for use in many applications, including various truck components. Our view on this new alpha SiC fiber form is that it is a breakthrough candidate for many future low cost and high performance systems.

Our company, Oshkosh is interested in your plan for expansion of the testing and evaluating this technology as it applies to our business. It appears that this technology will enable production of a new generation of alpha silicon carbide base metal matrix composite materials for use in manufacturing of advanced hardware for transportation, aerospace, chemical, defense, energy, environmental, manufacturing and medical applications.

We are confident that your alpha silicon carbide production technology will be received with enthusiasm in the highly prestigious international competition for the 2012 R&D 100 Award.

Sincerely,

David Pelko 15-FEB-2012

David Pelko, Chief Principal Engineer
Defense Engineering - Armor Technology
Oshkosh Truck Corporation
2307 Oregon Street
P.O. Box 2566
Oshkosh, WI 54903-2566

Appendix D

Letters of Support

Westinghouse

Westinghouse Electric Company
Research Technology Unit
Suite 678
600 Cranberry Woods Drive
Cranberry Township, Pennsylvania 16066
USA

Direct tel: 412-874-2887
Direct fax: 412-256-2444
e-mail: lahodaej@westinghouse.com

To:
Dr. John E. Garnier, Principal Investigator
Advance Nuclear Composite Hardware
Dr. George W. Griffith
Manager Nuclear Thermal Science and Safety Analysis
Idaho National Laboratory, P.O. Box 1625
Idaho Falls, ID 83415

Subject: Recommendation of Alpha SiC Fiber for R&D 100 Competition

February 9, 2012

Dear Dr. Garnier and Dr. Griffith,

This letter is written in support of the nomination for a R&D 100 Award for your recent advancement in the production of alpha silicon carbide fibers.

I am familiar with your new technology to produce silicon carbide fibers by a simple direct conversion process developed at the Idaho National Laboratory through the DOE Light Water Reactor Sustainability program. This alpha SiC form has an inherent single phase crystalline stability to sublimation point for alpha SiC (2,730^{0}C). This method for making alpha SiC fiber will enable cost competitive high performance engineering systems that require high temperature operation in aggressive conditions such as for nuclear, aerospace, chemical, defense, energy, environmental, and medical applications.

My company, Westinghouse Electric Company LLC, is interested in expansion of the testing and evaluation of this technology as it applies to our nuclear fuel business. This technology is a key step that will enable production of a new generation of cost competitive alpha silicon carbide base composite materials for use in advanced nuclear fuel cladding and water channels.

I am confident that your alpha silicon carbide production technology will be received with enthusiasm in the highly prestigious international competition for the 2012 R&D 100 Award.

Regards,

Edward J. Lahoda
Consulting Engineer
Research and Technology
Westinghouse Electric Company LLC

Appendix D

Letters of Support

PHYSICAL SCIENCES INC.

January 30, 2012

Dr. John E. Garnier, Principal Investigator, Advance Nuclear Composite Hardware
Idaho National Laboratory, P.O. Box 1625
Idaho Falls, ID 83415

Dear Dr. Garnier,

This letter is written in support of your recent advancement in production and employment of alpha silicon carbide fibers and its nomination for the R&D 100 Award for the reasons outlined below.

My colleague and I had attended your latest January 2012 presentation at the 36th Annual Conference on Composites, Materials, and Structures hosted by the United States American Ceramic Association (USACA-Cocoa Beach Florida) and after reading your earlier paper presented at the ASME Symposium September 2011 on Small Modular Reactors held in Washington, DC. We have become familiar with your new technology to produce silicon carbide fibers by a simple direct conversion process at Idaho National Laboratory. We also knowledge and appreciate receipt of samples of the alpha SiC/C fabric fiber to enable an early assessment by Physical Sciences Inc. on behalf to the Missile Defense Agency as a new ultra high temperature (UHT) ceramic fiber and matrix composite system. We recognize that the alpha SiC form has an inherent single phase crystalline stability to sublimation point for alpha SiC (2,730°C). Our view on this new alpha SiC fiber form is that it is a breakthrough candidate for many future low cost and high performance systems.

Our company, Physical Sciences Inc. is interested in expansion of the testing and evaluating this technology as it applies to our business. It appears that this technology will enable production of a new generation of alpha silicon carbide base composite materials for use in manufacturing of advanced hardware for aerospace, chemical, defense, energy, environmental, manufacturing and medical applications.

We are confident that your alpha silicon carbide production technology will be received with enthusiasm in the highly prestigious international competition for the 2012 R&D 100 Award.

Sincerely,

Fredrick S. Lauten

Dr. Fredrick S. Lauten
Manager and Principal Research Scientist
Physical Sciences Inc
20 New England Business Center
Andover, MA 01810

Appendix D

Letters of Support

HITCO CARBON COMPOSITES, INC

February 6, 201:

Dr. John E. Garnier, Principal Investigator,
Advance Nuclear Composite Hardware
Idaho National Laboratory, P.O. Box 1625
Idaho Falls, ID 83415

Subject: INL alpha- SiC DIRECT™ for R&D 100 award

Dear Dr. Garnier,

In support for your progress in the scale-up development and recent nomination by the Idaho National Labs of the alpha SiC DIRECT ™ process technology for the R&D 100 Award, we are pleased to provide this letter of recommendation for the reasons outlined below.

I have become familiar with your new technology to produce alpha silicon carbide fibers by a direct conversion process during my last visit to the INL. I had also attended your latest January 2012 presentation at the 36th Annual Conference on Composites, Materials, and Structures hosted by the United States American Ceramic Association (USACA-Cocoa Beach Florida). I also read your earlier paper presented at the ASME Symposium September 2011 on Small Modular Reactors held in Washington, DC. We also knowledge your recent lab level processing of HITCO 50k tow carbon samples using this direct process and look forward to evaluation of alpha SiC/C for use in both metal matrix and ceramic matrix composite systems. We recognize that in addition to carbon high temperature capabilities the 6H polytype form of alpha SiC has an inherent single phase crystalline stability to its sublimation point for alpha SiC (2,730°C). Our view on this new alpha SiC/C fiber form is a potential materials candidate for many future low cost and high performance systems.

Our company, HITCO, Inc is interested in expansion of the testing and evaluating this technology as it applies to our business. From our initial assessment this technology may enable production of a new generation of alpha silicon carbide fiber base composite materials for use advanced hardware for aerospace, chemical, defense, energy, environmental, manufacturing and medical applications.

We are confident that your alpha silicon carbide production technology will be received with enthusiasm in the highly prestigious international competition for the 2012 R&D 100 Award.

Sincerely,

Roberta Hines

Roberta Hines Business Development Manager- Advanced Ceramics
HITCO Carbon Composites / Subsidiary of SGL Carbon

1600 West 135th Street
Gardena, CA 90249-2506
USA

Phone +1 (310) 527-0700

Appendix E

Patent Pending Abstracts

United States Patent Application Attorney Docket 2939-10059US (BA-479; Oct. 8, 2011

METHODS OF PRODUCING SILICON CARBIDE FIBERS, SILICON CARBIDE FIBERS, AND ARTICLES INCLUDING SAME, Inventors: John E. Garnier and George W. Griffith

(Patent abstract is provided here. The applications for this pending patent have not been published by the patent office and have not been made available for public information at this time. For this reason, the complete text of the filed patent is not included.)

ABSTRACT OF THE DISCLOSURE

BA-479 (U.S. Patent Application No. 12/901,309 filed October 8, 2010): Methods of producing silicon carbide fibers. The method comprises reacting a continuous carbon fiber material and a silicon-containing gas in a reaction chamber at a temperature ranging from approximately 1500°C to approximately 2000°C. A partial pressure of oxygen in the reaction chamber is maintained at less than approximately $1.01 \times 10_2$ Pascal to produce continuous alpha silicon carbide fibers. Continuous alpha silicon carbide fibers and articles formed from the continuous alpha silicon carbide fibers are also disclosed.

United States Patent Application Attorney Docket 2939-10288US (BA-530; March 1, 2010; Via Electronic Filing)

METHODS OF PRODUCING CONTINUOUS BORON CARBIDE FIBERS, CONTINUOUS BORON CARBIDE FIBERS, CONTINUOUS FIBERS COMPRISING BORON CARBIDE, AND ARTICLES INCLUDING FIBERS COMPRISING AT LEAST A BORON CARBIDE COATING; Inventors: John E. Garnier, George W. Griffith

(Patent abstract is provided here. The applications for this pending patent have not been published by the patent office and have not been made available for public information at this time. For this reason, the complete text of the filed patent is not included.)

ABSTRACT OF THE DISCLOSURE

BA-530 (U.S. Patent Application No. 13/215,967 filed August 23, 2011): Methods of producing continuous boron carbide fibers. The method comprises reacting a continuous carbon fiber material and a boron oxide gas within a temperature range of from approximately 1400°C to approximately 2200°C. Continuous boron carbide fibers, continuous fibers comprising boron carbide, and articles including at least a boron carbide coating are also disclosed.

Appendix F

Video Script

1 Video Script - R&D 100 SiC Direct 031212 (4:00 minutes)

Video description	Narration Text
Narrator. Still or video of images from Stone, Bronze, Steel and Space ages. End with close up of alpha SiC.	**Human history has had many identifiable ages - Stone, Bronze, Steel and Space. Soon we may have the Alpha Silicon Carbide Fiber Age.**
Narrator or team member: Using close up of alpha SiC with text bullets: • **transportation,** • **energy,** • **manufacturing,** • **defense-aerospace, and** • **industrial operations**	**Alpha Silicon Carbide has chemical, mechanical and thermal properties that make it an attractive degradation-resistant ceramic for use in transportation, energy, manufacturing, defense-aerospace, and industrial operations.**
Hexagonal molecular drawing.	**This super-material has more than 250 molecular polytype forms that exhibit dozens of qualities and a few of those are:**
Narrator. Tensile testing video showing the meter,	• **An impressive strength-to-weight ratio – one-third the weight of steel and 3 to 6 times stronger,**
Bunsen burner thermal test of fiber	• **Resistant in high temperature conditions,**

Written/Produced by Keith Arterburn

Appendix F

Video Script

2   Video Script - R&D 100 SiC Direct 031212 (4:00 minutes)

Power lines,	• **Process configurable electrical properties from low to high conductivity,**
Nitric acid solution in bowl,	• **Highly corrosion resistant,**
Images of blue glow of nuclear reactor	• **Superior resistance to long-term radiation exposure, and more.**
Narrator: INL logo moving to video of research team in laboratory setting with names on the screen (possibly). Move to close up of alpha SiC fiber produced – vary the forms displayed from other video.	**Idaho National Laboratory researchers have invented an amazing breakthrough process to directly convert carbon fibers into alpha silicon carbide fibers – a known material that has <u>never been produced as a continuous fiber before now.</u>**
Narrator. (need suggestions for this section on video); it introduces the process which we will call Silicon Carbide Direct – not SICK Direct, even though we are using **SiC** Direct. Do not want someone lampooning this as a SICK product. Move to furnace – images of sand preparation for furnace, SiC fiber on spools.	**INL's process, called SiC Direct, uses available industrial furnaces and inexpensive domestic raw materials, like common sand, to deliver this high quality super-material for an affordable price of that could be less than $100 per pound.**

Written/Produced by Keith Arterburn

Appendix F

Video Script

3 Video Script - R&D 100 SiC Direct 031212 (4:00 minutes)

Narrator: Close up of beta SiC fibers.	**No direct competitor fiber material exists. The exotic and more expensive beta silicon fibers are limited in availability and can only be imported from overseas, some at a cost of more than $6,000 per pound.**
John Garnier on camera - recorded 021412 - AS DELIVERED.	**"SiC Direct works by taking a combination of SiO_2 sand and silicon metal heating it to a high temperature – about 1600**°C in an inert atmosphere of argon. At that point, we then draw a **commercially available carbon fiber through that atmosphere and in a few moments we get a conversion to alpha silicon carbon fiber."**
Narrator: Video of INL laboratory – lab furnace during production, John Garnier working with coiled fiber as produced.	**SiC Direct is a robust process, permitting the fiber quality to be adjusted through varying the draw rate and processing temperature.**

Written/Produced by Keith Arterburn

Appendix F

Video Script

4 Video Script - R&D 100 SiC Direct 031212 (4:00 minutes)

Narrator: video of alpha SiC coiled fiber and individuals moving handling it in the lab.	**INL researchers have produced thousands of meters of silicon carbide fiber in both single and multiple fiber groups. The alpha**
Video of team members handling woven and braided SiC samples from INL.	**silicon fibers can be woven or braided and then processed using INL's patented spray forming technique to produce metal matrix composites, tubes, plates, and various other forms with silicon carbide fiber as reinforcement material.**
George Griffith – AS DELIVERED Use nuclear fuel shaped rod or sample.	**"We have also have tested a batch processing method that permits customized shapes for many uses, including components that help make nuclear reactors safer.**
George Griffith continues – AS DELIVERED	**Alpha silicon carbide fibers and pre-forms offer the prospect of significant performance improvements and products that last longer."**

Written/Produced by Keith Arterburn

Appendix F

Video Script

5 Video Script – R&D 100 SiC Direct 031212 (4:00 minutes)

Narrator: video of autos-trains, semi-trailer trucks, gas pump,	**In transportation, autos can be made lighter and stronger significantly increasing personal safety and the efficiency of miles traveled by gas and electrified vehicles. Lighter rail cars and semi-truck trailers may reduce fuel consumption, also increasing miles per gallon.**
Narrator: Various energy video, including power lines;	**Energy systems may be improved with stronger and more conductive power lines to safely deliver electricity more efficiently.**
Video of body and vehicle armor, buildings after earthquakes. Commercial and military aircraft, ships	**Blast mitigation materials for body armor, vehicle protection and building reinforcement could be more affordable.** **Defense may be improved with lighter and stronger ships, aircraft and jets engines for speed and efficiency.**

Written/Produced by Keith Arterburn

Appendix F

Video Script

6 Video Script - R&D 100 SiC Direct 031212 (4:00 minutes)

Narrator: Industrial manufacturing – pump, heaters, heat exchangers, boilers, pressure vessels (industrial settings)	**Industrial manufacturing could be more cost efficient using less energy and much smaller amounts of valuable materials like aluminum augmented with alpha silicon fiber reinforcement. Pumps, heat exchangers, radiant heaters, boilers, pressure vessels and more may be better built and last longer.**
Narrator – Closing: Video – SiC Direct furnace and fiber coiling operations, ending with close ups of various fiber sizes and woven and braided forms.	**SiC Direct saves energy, uses inexpensive materials, and requires limited capital investment.** **SiC Direct makes available a revolutionary, super fiber material for use in redesigned and longer lasting products in two continuous direct conversion processes.**
Narrator: Alpha SiC Fiber materials on display. End with INL Logo	**Alpha silicon carbide fiber materials could propel America and the world into a new age of manufacturing and efficiency. END**

Written/Produced by Keith Arterburn