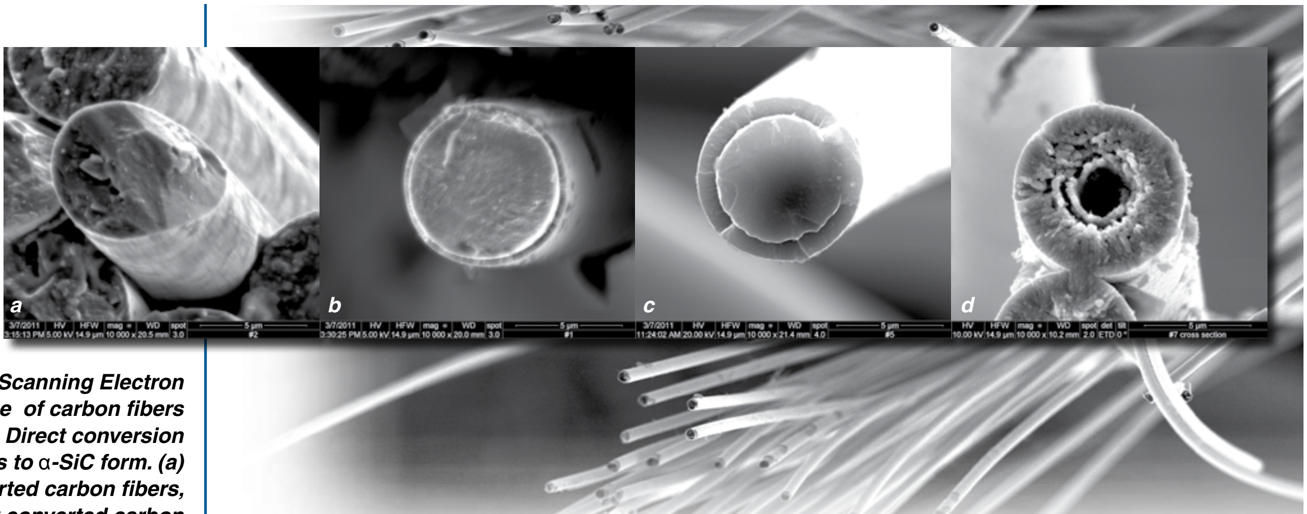

Figure 1: Scanning Electron Microscope of carbon fibers during SiC Direct conversion process to α-SiC form. (a) Unconverted carbon fibers, (b) Partially converted carbon fiber (α-SiC/C) with ~200 nm thick SiC shell, (c) Partially converted C fiber with 1 micron thick SiC shell. (d) Fully converted SiC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

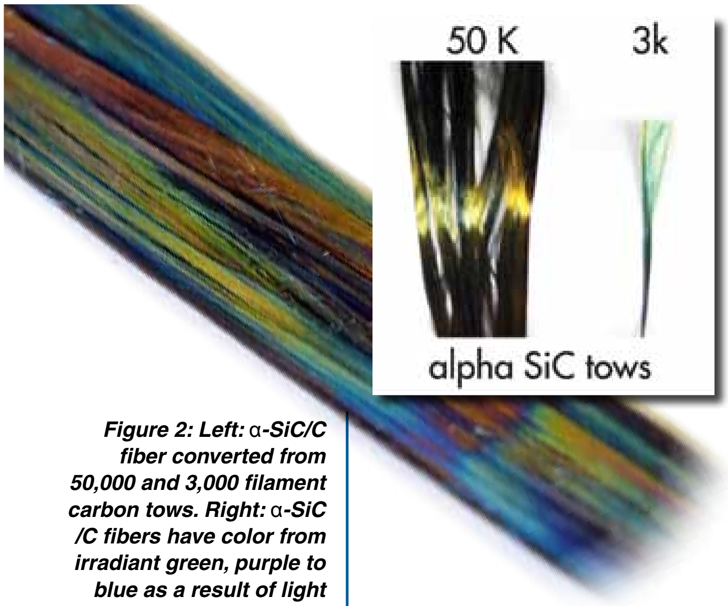

Figure 2: Left: α-SiC/C fiber converted from 50,000 and 3,000 filament carbon tows. Right: α-SiC /C fibers have color from irradiant green, purple to blue as a result of light scattering from sub-micron SiC grains and residual elements, such as N2 (green) .

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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 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

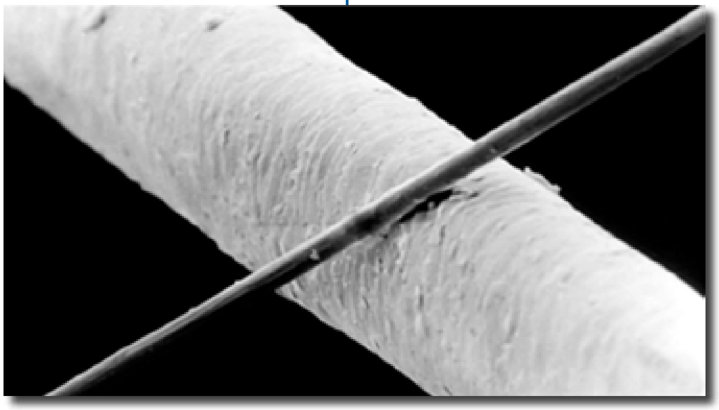

Figure 3: A single 6-micron diameter carbon pan fiber filament is positioned on top of a human hair for comparison. INL's α-SiC Direct process has demonstrated conversion with tows containing 3,000-50,000 filaments.

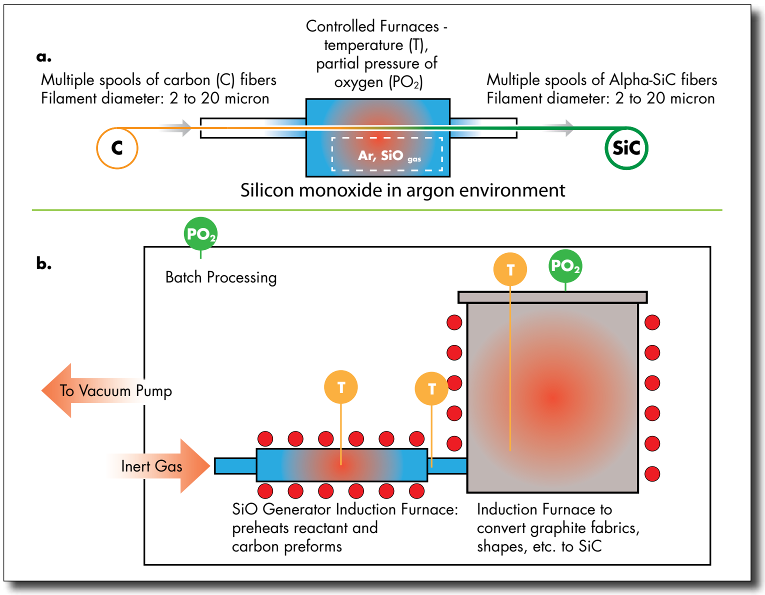

Figure 4: SiC Direct converts carbon fibers to α-SiC:(a) Continuous direct conversion to α-SiC fibers, (b) Continuous batch conversion of fibers, fabrics, and other pre-form shapes.

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 (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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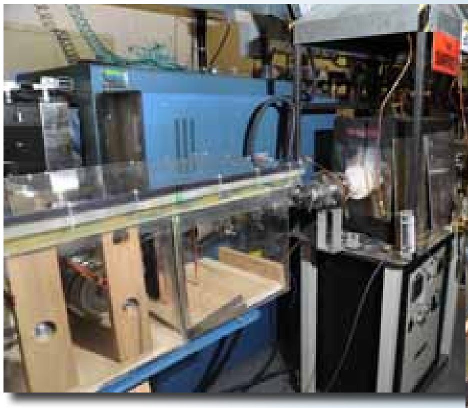

Figure 5: SiC Direct uses a high temperature furnace, right, to continuously convert carbon filaments into alpha silicon carbide fibers, which are coiled on spools, left and below.

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.

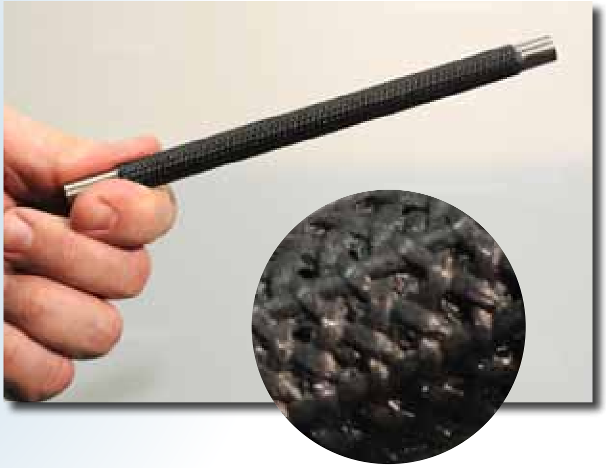

Figure 7: SiC Direct produces low cost α-SiC that permits customization of the fibers. This SiC overbraid on a metal tube demonstrates an advanced application for nuclear fuel-sized rods (patent pending). It is known that SiC will not degrade in radiation environments and has high temperature resilience.

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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.

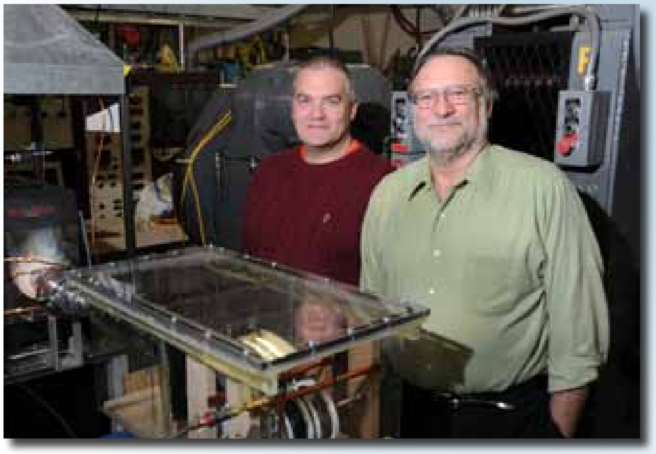

Figure 9: Inventors, left to right, George Griffith and John Garnier display process furnace, left, and α-SiC fibers on spools, right.

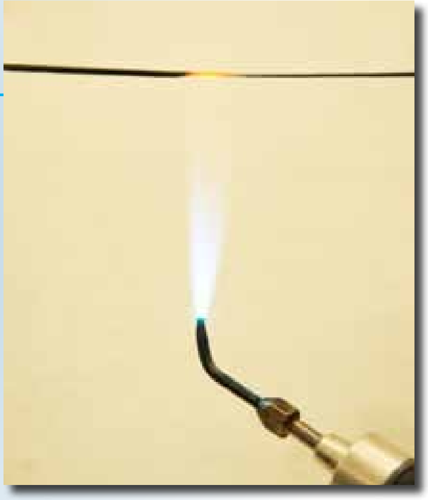

Figure 8: α–SiC fibers have high temperature resilience and do not experience phase change up to 2730°C, making the materials attractive for use in radiant heaters, boilers, pumps, and other high temperature conditions.