Defense Systems
Information Analysis Center

# DSIAC TECHNICAL INQUIRY (TI) RESPONSE REPORT

## Cellulosic Precursor Carbon Fibers

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# ABOUT DSIAC

The Defense Systems Information Analysis Center (DSIAC) is a U.S. Department of Defense Information Analysis Center sponsored by the Defense Technical Information Center. DSIAC is operated by SURVICE Engineering Company under contract FA8075-14-D-0001.

DSIAC serves as the national clearinghouse for worldwide scientific and technical information for weapon systems; survivability and vulnerability; reliability, maintainability, quality, supportability, and interoperability; advanced materials; military sensing; autonomous systems; energetics; directed energy; and non-lethal weapons. We collect, analyze, synthesize, and disseminate related technical information and data for each of these focus areas.

A chief service of DSIAC is free technical inquiry (TI) research, limited to 4 research hours per inquiry. This TI response report summarizes the research findings of one such inquiry. For more information about DSIAC and our TI service, please visit www.DSIAC.org.

# ABSTRACT

The Defense Systems Information Analysis Center (DSIAC) received a technical inquiry requesting information on the need for cellulosic precursor carbon fiber and where this need may be. The inquirer indicated that his/her company had a process to domestically produce fibers like viscose rayon fibers used historically in military missiles and National Aeronautics and Space Administration solid rocket motors, though viable need was required before production. DSIAC contacted subject matter experts (SMEs) from Texas Research Institute, Inc. Austin; Johns Hopkins University Applied Physics Laboratory Energetics Research Group; National Renewable Energy Laboratory; Oak Ridge National Laboratory; and various academic institutions. DSIAC also searched for information on cellulosic fibers using open sources and the Defense Technical Information Center Research and Engineering Gateway. The search results and SME responses were then compiled in a response report that was delivered to the inquirer.

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# 1.0 TI Request

## 1.1 INQUIRY

Is there still a strong need for cellulosic precursor carbon fibers (CFs) for use in rocket nozzles?

## 1.2 DESCRIPTION

The inquirer requested information on possible needs for cellulosic precursor CFs that are typically produced for rocket nozzles. These fibers were once domestically produced from historic viscose rayon, also known as the North American Rayon Corporation (NARC) aerospace-grade rayon, using a toxic process. The fibers were used in Army and Air Force missiles and National Aeronautics and Space Administration (NASA) solid rocket motors (SRMs) [1].

# 2.0 TI Response

Defense Systems Information Analysis Center (DSIAC) staff completed literature searches using the Defense Technical Information Center (DTIC) Research and Engineering (R&E) Gateway and open sources to find articles relevant to the inquiry topic using the following search terms: “cellulosic,” “precursor,” “carbon fiber,” and “rocket nozzle.” Closed technical inquiries and other DSIAC website sources (e.g., journals, web articles, and state-of-the-art reports [SOARs]) were also examined for relevant information or subject matter expert (SME) contacts.

## 2.1 BACKGROUND

### 2.1.1 Introduction to Cellulosic Precursor CFs

Cellulosic fibers were first introduced to the market in the 1960s and were produced from rayon. Rayon is a natural, cellulose-based material (e.g., wood pulp, cotton linters, leaves, inner pith of bamboo plants, etc.) that is chemically processed to produce semisynthetic fibers. Even though the raw materials cost is lower for rayon-based fibers, a lower yield pushes production costs above polyacrylonitrile (PAN)-based CFs. This fact is one of the reasons that rayon-based CFs have limited commercial production (1−2% of all CF production). Due to lower tensile strengths than PAN-based CFs, rayon-based CFs are not used in structural applications. Instead, rayon fibers are used in the rocket and missile industries for ablative applications (e.g., heat shields and rocket nozzles) since the fibers can withstand high temperatures and the erosive gases of SRMs. There are two required stages and one optional stage of production. Stabilization of the precursor (i.e., low temperature oxidation) and carbonization (i.e., longitudinal orientation and development of the crystalline ordering) are required; graphitization is an optional stress that will increase the modulus of elasticity [2].

### 2.1.2 Potential Markets for Cellulosic Precursor CFs

Cellulosic CFs were classically used to form ablative rocket nozzles and heat shields. However, NARC ceased production of the fiber after it was unable to financially comply with Environmental Protection Agency (EPA) regulations. According to a *CompositesWorld* article, NASA’s stockpile of NARC fiber has been dwindling since production the fiber stopped in the 1990s. Therefore, new processes to create cellulosic CF could be of interest and used for SRMs in the next-generation Space Launch System [3].

Outside of ablative and thermal resistance applications, other countries (e.g., Japan, China, Australia, Finland, etc.) have explored other uses for cellulosic precursor CFs that may not require the high strengths of other CFs. Cellulose-based CFs for carbon-carbon composites (CCCs) could potentially be used in hypersonic reentry vehicles, rocket propulsion, and aircraft brakes [4]. Widespread automotive and wind energy applications that do not require the >3 gigapascals (GPa) of ultimate tensile strength are offered by PAN-based CFs [5].

In Japan, researchers have been developing products with cellulose nanofibers such as disposable diapers and ballpoint pens, and cellulose nanofibers could potentially be used in food, cosmetics, and flexible displays. cellulose nanofibers has also been used as a strengthening resin in rubbers and plastics [6]. Furukawa Electric intends to use cellulose-based CFs for automotive interiors, electronics components, and body exterior panels, as part of the $3.5 billion market of replacing fiberglass composites [7]. Chemviron Carbon, located in the United Kingdom, has used viscose rayon fibers for knitted or woven activated carbon fabrics (ACFs) as an adsorbent material in chemical protective clothing. Applications for ACFs include the following [8]:

- Combat suits;
- First responder suits;
- Escape masks;
- Undergarments;
- Gloves;
- Overgarments;
- Chemical, biological, radiological, and nuclear (CBRN) filtration media;
- CBRN decontamination wipes;
- Low weight reduced respirator canisters;
- Antimicrobial wound dressings;
- Missile decoy media.

Similarly, CF/cellulose composite papers were produced using papermaking techniques. Hot fibers were used to modify the CF/cellulose composite paper's mechanical properties [9].

## 2.2 SMEs

### 2.2.1 National Laboratories and Government Agencies

#### Oak Ridge National Laboratory (ORNL)

Two technology centers at ORNL have research focuses on CF development and advancement in partnership with the U.S. Department of Energy (DoE). The Carbon Fiber Technology Facility, housed at ORNL, allowed the Manufacturing Demonstration Facility and the National Transportation Research Center to focus on alternative precursors, advanced and energy-efficient conversion processes, and scaling. The thermal conversion line provided a baseline for standard modulus PAN, but was designed with the flexibility to accommodate lignin, polyolefin, and pitch precursors. It can also be readily upgraded to convert rayon and high-modulus PAN precursors [10, 11]. See Table 1 for more information on ORNL's Manufacturing Demonstration Facility, and Table 2 for more information on ORNL's National Transportation Research Center.

**Table 1: ORNL Manufacturing Demonstration Facility Contact Information [12]**

| | |
|---|---|
| **Organization** | Manufacturing Demonstration Facility |
| **Address** | 2370 Cherahala Blvd<br>Knoxville, TN 37932 |
| **Email** | manufacturing@ornl.gov |

**Table 2: ORNL National Transportation Research Center Contact Information [13]**

| | |
|---|---|
| **Organization** | National Transportation Research Center |
| **Address** | 2360 Cherahala Blvd.<br>Knoxville, TN 37932 |
| **Phone** | (865) 946-1500 |
| **Email** | transportation@ornl.gov |

### National Renewable Energy Laboratory (NREL)

After finding multiple papers published on CF manufacturing by scientists at the National Renewable Energy Laboratory (NREL), DSIAC contacted a principal scientist at the National Bioenergy Center of NREL. However, the NREL research is focused on bioderived acrylonitrile (ACN), another precursor for CF, though NREL mentioned that Southern Research was exploring a cellulosic route to produce CF as a rayon replacement [14].

## 2.2.2 Industry and Private Organizations

### Institute for Advanced Composites Manufacturing Innovation (IACMI)

With the assistance of ORNL's Sustainable Transportation Program, DSIAC contacted the Director of the Materials and Process Technology Area at ORNL, who is located at IACMI.

The Director of the Materials and Process Technology Area stated that IACMI doesn't know of a current potential market for CFs made from cellulosic precursors, though several research institutions are attempting to produce cellulose-based CFs that would satisfy a market need (i.e., the balance of cost and performance) and lead to future market development. See Table 3 for IACMI contact information.

A few other institutions conducting relevant research include the University of Tennessee-Knoxville, North Carolina State University, Iowa State University, Washington State University, and the University of British Columbia. Innventia in Sweden is also notable for its relevant research, and there is also interest in Scandinavia and Asia for CF manufacturing [15].

Table 3: IACMI Contact Information [16]

| | |
|---|---|
| **Organization** | IACMI |
| **Address** | 2360 Cherahala Boulevard<br>Knoxville, TN 37932 |
| **Phone** | (865) 974-8794 |
| **Email** | info@iacmi.org |

## Texas Research Institute (TRI) Austin

TRI Austin is a DSIAC subcontractor that brings significant laboratory and materials research and development (R&D) and testing experience to DSIAC [17]. See Table 4 for the TRI Austin contact information.

Table 4: TRI Austin Contact Information [18]

| | |
|---|---|
| **Organization** | TRI Austin |
| **Address** | 9225 Bee Cave Rd.<br>Bldg B, Ste. 200<br>Austin, TX 78733 |
| **Phone** | (512) 263-3272 |
| **Email** | austininfo@tri-austin.com |

TRI Austin supplied papers of interest and contact information for other SMEs [19]. The papers of interest include the following:

The paper “NARC Rayon Replacement Program for the RSRM Nozzle, Phase IV Qualification and Implementation Status” is a review of NASA’s selection process that took place in the mid-2000s to find a replacement for the out-of-production NARC rayon CF. NASA purchased a stockpile of the fiber prior NARC discontinuing rayon fiber production to support reusable solid rocket motor (RSRM) production but wanted to have a new supplier as the stockpile dwindled. After testing 21 cellulose fiber candidates, the fiber chosen for Phase III and IV testing was Acordis Enka CCP. Concerns at the end of testing included the supplies of pulp and recent closures of other rayon plants in Europe [20].

The NREL report “Carbon Fiber from Biomass” details different CFs using biomass sources. It notes that as of 2012, the RUE-SPA-Khimvolokno in Belarus is the main rayon-based CF producer in the world, that the U.S. DoD has a vested interest in the production of these fibers, and that there is research into the production occurring at the University of Tennessee using experimental fibers from Advanced Cerametrics in Lambertville, NJ and commercial fibers from Lenzing Group. The report also explores the economics of producing fibers in the U.S., especially without importing the precursors and, instead, importing the raw materials [2].

TRI Austin mentioned that Fiber Materials Inc. produces carbon-carbon extreme materials for applications such as solid rocket motor throats, leading edges, missile nose tips, nozzle exit cones, and aircraft brakes [21]. Based on the sources provided, it is apparent that the U. S. has searched for a replacement for NARC as recently as 2005.

### Johns Hopkins University (JHU) Energetics Research Group (ERG)

DSIAC contacted SMEs at JHU ERG who provided points of contact for multiple potentially knowledgeable people at various NASA groups, as well as information related to cellulosic CFs. JHU ERG contact information is presented in Table 5. Companies that produce cellulosic CFs include the following [22]:

- Highland Industries:  Highland Industries is a U.S. company that produces woven rayon fabrics for aerospace use. It began as an exclusive relationship more than 35 years ago for woven rayon fabrics that formed the insulators for solid rocket nozzles on the Space Shuttle [23].
- Lenzig Fibers Incorporated:  According to the American Fiber Manufacturer’s Association, the only U.S. producer of Lyocell staple fibers is Lenzig Fibers Inc. [24].
- Morgan Advanced Materials & Technology:  Morgan Advanced Materials & Technology is the only company that carbonizes Lyocell fibers [25].

The steps to create cellulosic rayon fibers are fiber production, weaving, prepregging, and carbonization. The process starts with Lenzig Fibers, Inc. , then moves to Highland Industries, and then to Morgan Advanced Materials & Technology. The University of Alabama-Huntsville (UAH) would come in as an alternate to Lenzig, or to whomever they license their technology [22].

The JHU ERG representatives also provided a U.S. Army Small Business Innovative Research (SBIR) report that describes the collaboration between the University of Alabama-Huntsville and MATECH on the fiber-production process [26].

Table 5: JHU ERG Contact Information [27]

| | |
|---|---|
| **Organization** | JHU ERG |
| **Address** | 10630 Little Patuxent Parkway<br>Suite 202<br>Columbia, MD 21044-3286 |
| **Phone** | (410) 992-7300 |
| **Email** | info@erg.jhu.edu |

### Southern Research

The following as an excerpt from the article "Southern Research Project Targets Low-Cost Carbon Fibers":

> Amit Goyal, Ph.D., is leading a team of scientists at Southern Research that has developed a cheaper and cleaner process for making acrylonitrile, a precursor for carbon-fiber production.
>
> Goyal's team has devised a multi-step catalytic process that converts sugars from non-food biomass to acrylonitrile through a pathway that could be around 20 percent cheaper than the typical production method. Their process also involves a substantial reduction in greenhouse gases.
>
> The CFs produced from this raw biomass process could interest automakers, which are looking at composite materials to reduce the weight of vehicles.
>
> Goyal, manager of Southern Research's sustainable chemistry and catalysis group, Energy & Environment, North Carolina, acts as principal investigator on the $6 million Department of Energy study.

Southern Research is working with Cytec and the New Jersey Institute of Technology on the DoE-funded study [28].

## 2.2.3 Academic Institutions

### University of Tennessee-Knoxville

The University of Tennessee-Knoxville has multiple publications related to cellulose-based CFs and has strong ties to ORNL and IACMI. The University of Tennessee-Knoxville received an EPA grant for 2004−2008 to develop cellulosic CF precursors from ionic liquid solutions. The grant resulted in three papers, one of which is included in the following list of publications of interest (third bullet) [29]:

- In the presentation “Recent Developments on Carbon Fibers from Rayon-Based Precursors” from the 2013 Carbon Fiber Workshop in Buffalo, NY, Dr. G. Bhat’s research group presented on the possibility of producing rayon fibers due to the lack of domestic manufacturers. The research explored the possibility of using experimental rayon fibers supplied by Advanced Cerametrics Inc. as a CF precursor, while also using commercial rayon fibers from Lenzing. The initial observations included a need to improve the mechanical strengths and decrease the fiber diameters [30].

- In his master’s thesis, “Pretreatment and Pyrolysis of Rayon-Based Precursor for Carbon Fibers,” K. Akato investigated two different rayon fibers as CF precursors. The objectives of the work were to investigate the stabilized stage of the produced rayon and the changes occurring during thermal decomposition, and to evaluate the effects of these changes on the properties of the carbonized fiber. The changes that take place during the conversion of the domestic fibers were compared with commercial rayon fiber [31].

- The second article in a series of three, “Rheology of 1-Butyl-3-Methylimidazolium Chloride Cellulose Solutions. II. Solution Character and Preparation,” focuses on the effects of preparation and composition on the shear rheology of cellulose in the ionic liquid 1-butyl-3-methylimidazolium chloride. The effects of three different degrees of polymerization, manual versus high shear mixing, range of cellulose concentrations, and effects of controlled amounts of lignin and hemicellulose are included in the investigation. Conclusions include the implication from the rheology that a gel phase develops at higher degrees of polymerization, higher concentration, and at lower temperatures [32].

While the University of Tennessee-Knoxville has researched efforts into cellulosic precursor CF similarly to the University of Alabama-Huntsville, there are a few other universities (i.e., Iowa State University and Washington State University) that focus on sugar or biobased lignin CF precursors for ACN or PAN.

### University of Texas

Per the suggestion of TRI Austin, DSIAC investigated Dr. Joseph Koo’s research group at the University of Texas at Austin. DSIAC determined that a plethora of Dr. Koo’s projects relate to ablation and CFs [33].

### Iowa State University

The Polymer Composites Research Group at Iowa State University is investigating the viability of using lignin-based CFs in wind turbine blades [34, 35].

### Washington State University

The Composite Materials & Engineering Center at Washington State University is researching synthesizing biobased ACN from propylene glycol [36, 37].

## 2.3 OTHER PAPERS OF POTENTIAL INTEREST

Other papers of interest in relation to the inquiry include the following:

[1] “Cellulose-Based Carbon Fibers: Increasing Tensile Strength and Carbon Yield” [38].

[2] “Cellulose-Driven Carbon Fibers With Improved Carbon Yield and Mechanical Properties.” The following is an excerpt of the abstract from the document [39]:

> The manufacture of high mechanical strength cellulose-based carbon fibers (CFs) is accomplished in a continuous process at comparably low temperatures and with high carbon yields. Applying a sulfur-based carbonization agent, i.e., ammonium tosylate (ATS), carbon yields of 37% (83% of theory), and maximum tensile strengths and Young's moduli up to 2.0 and 84 GPa are obtained already at 1400 °C. For comparison, the use of the well-known carbonization aid ammonium dihydrogenphosphate ((NH4)H2PO4), ADHP, is also investigated. Both the precursor and the CFs are characterized via elemental analysis, wide-angle X-ray scattering, Raman spectroscopy, scanning electron microscopy, and tensile testing. Thermogravimetric analysis coupled with mass spectrometry/infrared spectroscopy discloses differences in structure formation between ATS and ADHP-derived CFs during pyrolysis.

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