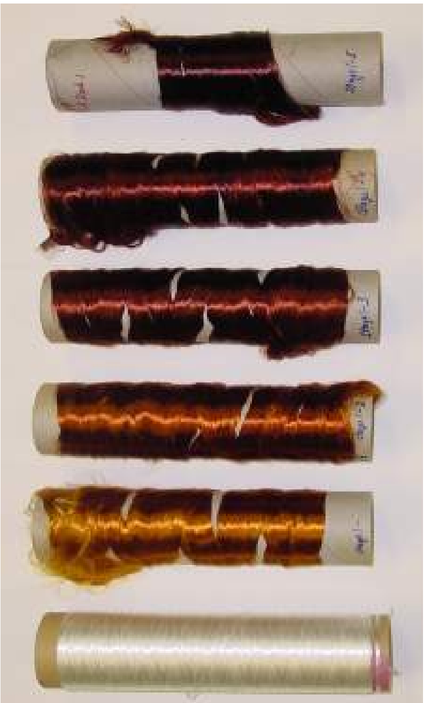
Low-Cost Carbon-Fiber Integration / Users Facility And Commercialization of Textile Precursors



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Integration – Purpose, Barrier, Approach

Purpose:

Aid Commercialization of all developed LCCF related projects by integrating each processing development or new precursor into the existing system for manufacturing carbon fiber. Be able to incorporate an alternative processing step or material into the way it is currently done in industry. Be able to do this for large tow or evaluation of small samples.

Barriers:

New processing techniques will not be adopted by industry until they are proven, by demonstration, to be robust, reliable and able to produce the desired product. No capability to process small, new precursor samples.

Approach:

1. Establish conventional processing capabilities.
2. Design lab space to allow for substitution of processing techniques and materials.
3. Upgrade equipment to support project needs.
4. Establish capability to evaluate small quantities of potential new precursors.

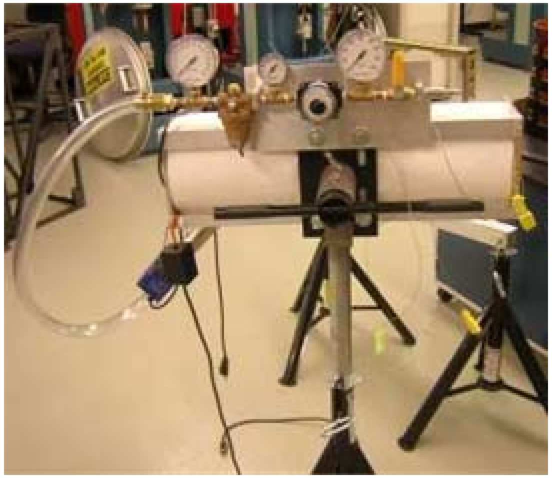
Budget: $ 200K

Integration – Accomplishments

FreedomCAR Materials

Established Conventional Processing Line

1. Purchased 1/20th scale line from Toho.
2. Installed at ORNL.
3. Carbon-proofed facility.
4. Upgrade Line. Higher speed winders, exhaust system upgrade, continuous gas supply, added surface treatment, automated controls.
5. Processed conventional and textile grade precursor.



Sizing Dryer

Carbon Proofing

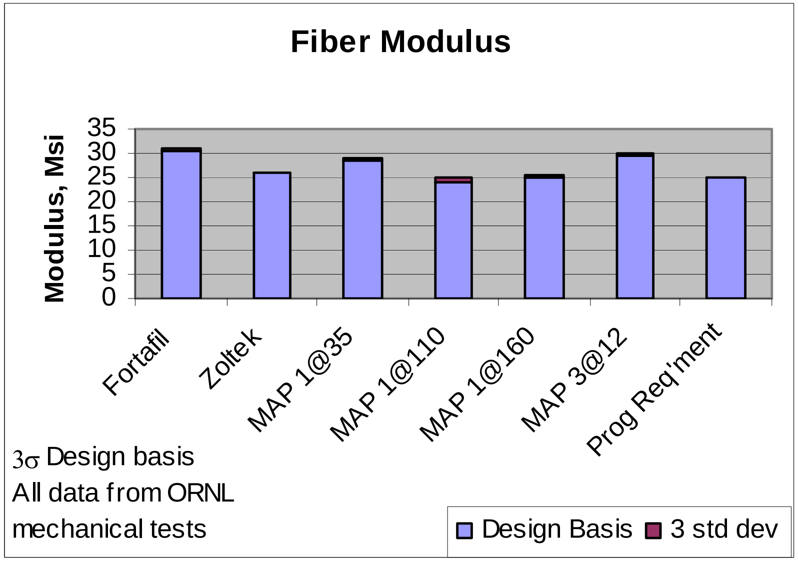
Installed Conventional Line

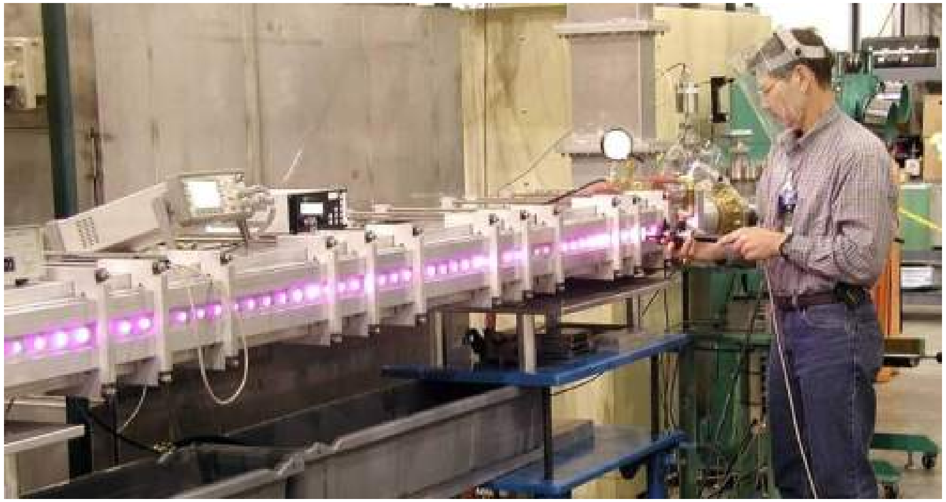
Integration – Accomplishments

FreedomCAR Materials

Advanced Technology Pilot Line

1. Installed the first subsystem to the advanced technology line.
2. Scaled up unit for the Microwave Assisted Plasma (MAP) technology which is used for carbonization.
3. Located beside the conventional pilot line.
4. Installed higher speed winders.
5. Processed material using MAP for carbonization and conventional processing for the remainder of the manufacturing.
6. Manufactured and delivered plaques for testing to the ACC.





Current MAP Reactor

Integration – Accomplishments

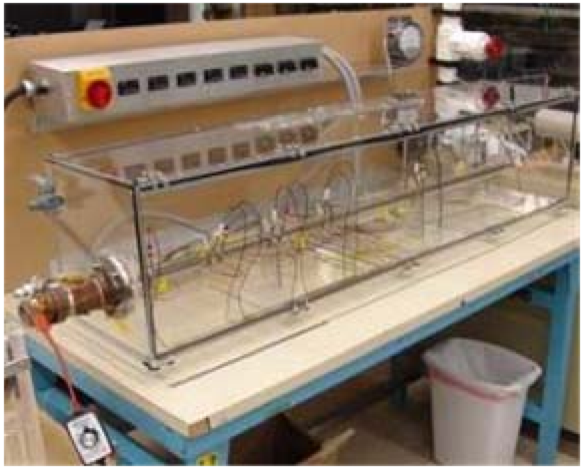
Large Line requires > 1K tows with a 20 hour start-up time. Tow breakage requires complete shutdown and restart.

Precursor Evaluation Line

1. Procured and Installed equipment for processing small scale precursor samples.
2. Added tensioning and restraining capabilities.
3. Processed conventional PAN, textile PAN and proprietary precursor samples.

Graphitization Furnace

Clamshell Furnaces



Transparent Furnaces

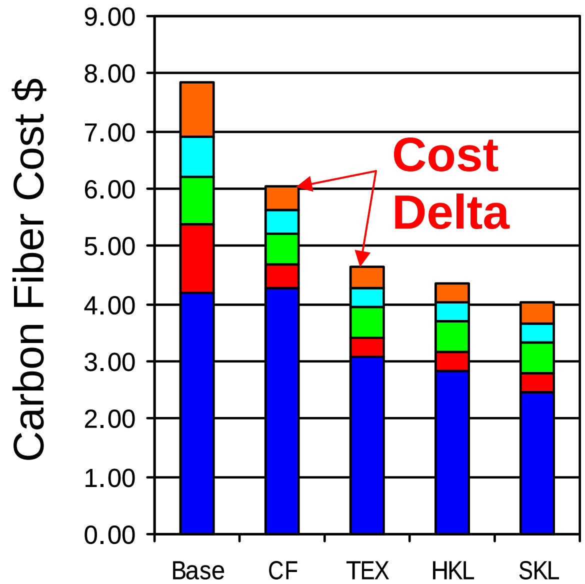
Due to quick start & stop the Precursor Evaluation Line is the "Workhorse" for precursor development

Textile Precursor – Purpose

Early 2007 the Kline Cost model revealed that significant cost savings could be yielded by using a textile based precursor.

Textile Precursors could be readily brought to market because:

- The textile mills are already in existence and make material in the required volumes.
- Textile precursor requires a single step pretreatment during manufacture to render it carbonizable.
- There is a large demand for lower modulus fiber in a number of industries making commercialization likely.
- The precursor could be converted to carbon fiber using conventional processing equipment.



The Program made a Strategic Decision to delay further integration activities and focus on implementing textile based precursor development and commercialization.

Funds Shifted Accordingly

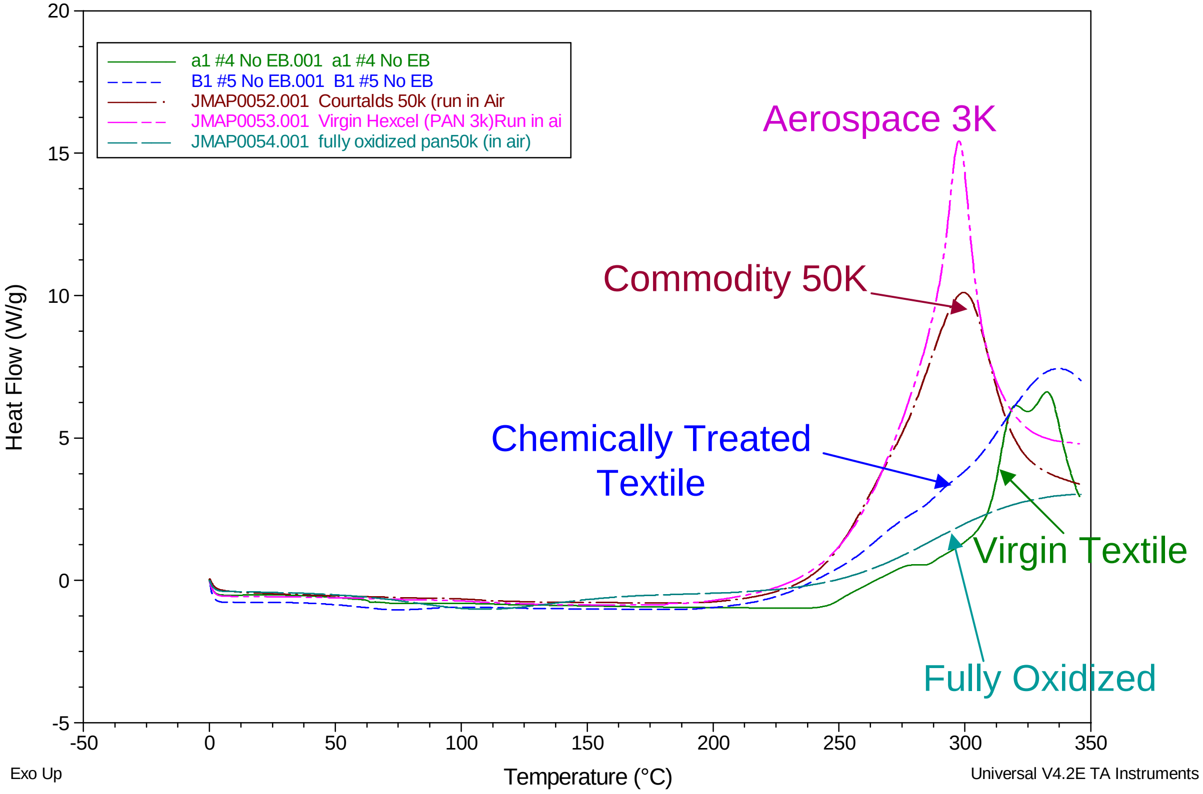
Textile Precursor – Barrier

FreedomCAR Materials

Hexcel did earlier work for ALM on developing a Chemical pretreatment that renders textile PAN carbonizable.

Steepest part of slope determines speed of stabilization. Location of ramp up start & peak determine oxidative stabilization temp range.

Chemically treated textile could undergo oxidative stabilization in <u>less time</u> but at a slightly higher temperature.



Precursor from the Sterling Textile Plant in Pensacola, Florida.

Textile Precursor – Barriers

- Process has been demonstrated using textile PAN
  - 28K samples used successfully were collected wet at Sterling
  - They were chemically treated in a lab at Hexcel, then dried
  - They were then processed in an experimental line

Oxidation residence time was reduced from 85 minutes to 50 minutes.

		TEXTILE		CONVENTIONAL		Program Goals
		Spool 1	Spool 2	Zoltek Panex 33	Fortafil F3(C)	
Modulus	**($x\ 10^6$ lb/in^2)**	**30.8**	**30.2**	**26.1**	**31.1**	25
Ultimate Strength	**($x\ 10^3$ lb/in^2)**	**398.6**	**393.4**	**407.8**	**485.3**	250
Elongation at Break	**(%)**	**1.25**	**1.27**	**1.5**	**1.5**	1.0

Barriers: Developing in-line chemical modification; Adjusting modification to particular PAN formulations; Splitting Large tows to manageable size; Time-temperature-tension profiles for conversion to carbon fiber, Scale-up.

Textile Precursor – Tech Transfer

There is significant demand for a lower cost carbon fiber for a variety of industries.

Adaptation will take time since processing methods and specific applications can only be developed after there is fiber at certain price points.

Automotive

Wind Energy

Power

Oil & Gas

Consumer and Sporting Goods

Infrastructure

Discussions with Potential Partners at Different Levels:

<u>Textile Manufacturers:</u>
FISIPE, S.A. (Portugal)
Bluestar Textiles (UK)
Sterling (US – Closed)

<u>Carbon Fiber Manufacturers</u>
SGL Fibers (UK)
Owens Corning (Belgium & US)
Great Lakes Carbon Fiber (US)
Samsung (S. Korea)

Textile Precursor – Accomplishments (FISIPE)

Once it became apparent that commercialization of textile precursor is the quickest route to significant cost reduction, we began some initial work with FISIPE January 2007.

FISIPE uses a VA co-monomer

Guided FISIPE in installing the chemical bath which is now in their pilot line facility and in optimizing the chemical pretreatment.

FISIPE produces precursor which we evaluate to determine the optimum conversion conditions.

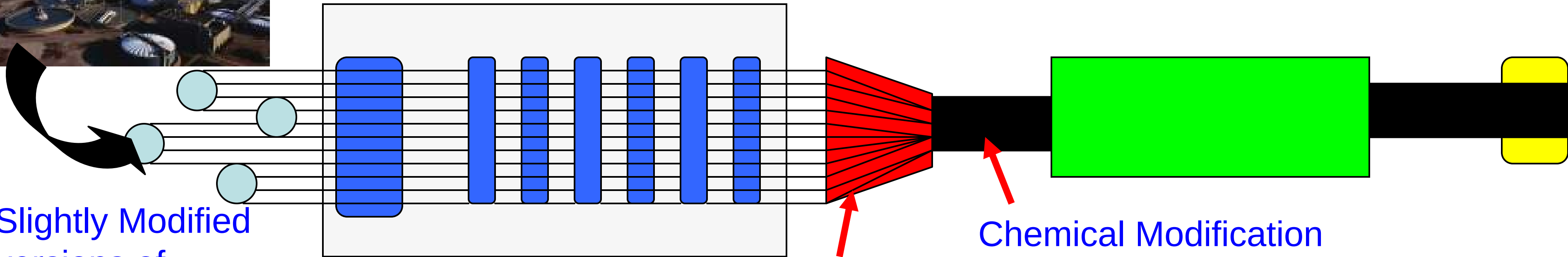
June Visit

Due to export control restrictions, FISIPE will not know the conversion conditions. We will work directly with the converter to transfer that. (Under export control license as necessary)

Budget: $500K

Textile Precursor – Accomplishments

Starts with a large “tank farm” which polymerizes PAN and other co-monomers



Slightly Modified versions of polymer selected

Gathering during Crimping will Be Deleted

Chemical Modification will occur here

Textile Pilot Plant

Currently about ½ way through development of processing time-temperature-tension profiles for fiber. Latest properties:

Strength: 249 KSI Modulus: 20 MSI (1/25 Run)

Significant proprietary interest from a number of potential converters.

Textile Precursor – Accomplishments (BlueStar)

Full Scale Development of Commercial Textile PAN-MA Precursors

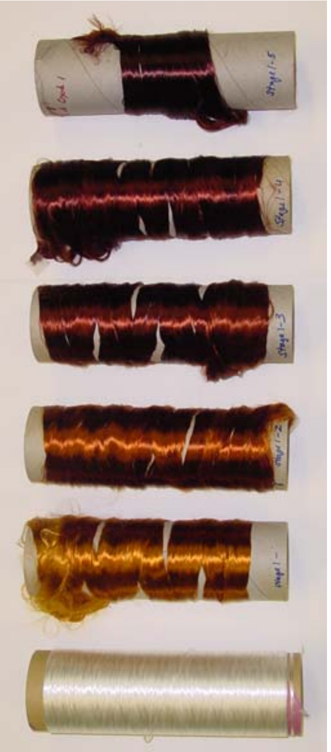
BlueStar Fibres Co. Ltd. Grimsby, UK — Neil Barker and Steve Amos
University of Sunderland, Sunderland, UK — Prof. Alan Wheatley

Bluestar uses a MA co-monomer.

Hexcel work was hand pulling a 28K tow off the line, bagging and sending to Decatur for a 1 min chemical treatment, collapsing, followed by processing.

At meeting on February 6, reached agreement to begin evaluating chemical pretreatments of Bluestar textile type material. SGL a partner in the development.

Major Concern with Chinese ownership of Bluestar.



Budget: $150K

Project Impact

PAN	Oxidation	Carbonization	Graphitization	ST
$3.53 (44.8%)	$1.34 (17.0%)	$1.00 (12.7%)	$1.19 (15.1%)	$0.82 (10%)

Textile Precursor – Accomplishments (Companies X & Y Commericialization)

FreedomCAR Materials

Company X –

1. Approached DOE for aid in building a 40,000,000 lb/year carbon fiber plant and a 2,000,000 lb/year plant.
2. Plans were to develop 1st lines based upon textile + conventional processing.
3. Small plant to be located in US. Large plant to be located in a foreign country.
4. Main automotive customers wereEuropean automakers though several proprietary US car lines and applications were mentioned.
5. Also have a substantial customer line established in wind energy and oil and gas companies.
6. Company was Owens Corning.
7. OC decided not to proceed due to cash drain from merger with Vetrotex.

Budget: Transitioned to Company Y

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Textile Precursor – Accomplishments (Companies X & Y)

FreedomCAR Materials

Company Y –

1. A major US company.
2. Interested in building one or more large carbon fiber plants with the initial design based upon textile + conventional processing.
3. All current facilities are North American based.
4. Will use FISIPE, Bluestar or purchase a textile mill (details known but proprietary.)
5. Is assembling a team of engineers to go forward with the plant. All US citizens so export control concerns are minimized.
6. Has BOD approval for the project and major new business direction.
7. Has committed several million $'s to the project already.
8. Identity and specific plans are proprietary and are being coordinated through Vehicle Technologies management.
9. ORNL's main role is to support design teams to make sure no critical mistakes are made.

Budget: $150K

Project Impact

PAN	Oxidation	Carbonization	Graphitization	ST
$3.53 (44.8%)	$1.34 (17.0%)	$1.00 (12.7%)	$1.19 (15.1%)	$0.82 (10%)

Textile Precursor – Accomplishments (Great Lakes Carbon Fiber)

FreedomCAR Materials

Great Lakes Carbon Fiber

1. A start-up company to be located in or around Flint, Michigan..
2. Interested in building one or more carbon fiber plants with the initial design based upon textile + conventional processing.
3. Will use FISIPE, or Bluestar textile.
4. ORNL's main role is to support design teams to make sure no critical mistakes are made.
5. Only in initial planning stages.

Budget: TBD

PAN	Oxidation	Carbonization	Graphitization	ST
$3.53 (44.8%)	$1.34 (17.0%)	$1.00 (12.7%)	$1.19 (15.1%)	$0.82 (10%)

Papers:

1. "Novel Materials and Approaches for Producing Carbon Fiber", published in proceedings of 11th European Automotive Congress, Automobile for the Future, Budapest, Hungary, 30th May to 1st June 2007.
2. "FreedomCAR and Low Cost Carbon Fiber for Automotive Applications," 7th International Congress on Materials for Lean Weight Vehicles, University of Warwick, Warwick, UK , 26th to 27th September 2007
3. "The Development of Lower Cost Carbon Fiber Technologies for Automotive Applications", Proceedings of The Global Outlook for Carbon Fiber 2007, San Diego, CA, 23-25 October 2007.
4. "A Comprehensive Research Program to Develop Commodity Grade, Lower Cost Carbon Fiber", To be published in the proceeding of the ACMA Breaking New Ground: Structural Composites Applications in Defense, Infrastructure, Transportation and Corrosion-Prevention, University of Alabama-Birmingham, 4-6 March 2008.

Patents:

None

Integration/Textile Precursor – Plans and Summary

1. Complete development of textile VA-PAN with FISIPE.
2. Begin development of MA-PAN with at least one company.
3. At the conclusion of each phase produce significant amounts of material to justify to automotive companies and potential carbon fiber producers that the textile precursors are market ready.
4. Integrate carbon fiber produced from textile precursors in the ALM and OEM projects that are developing processing methods.
5. Assist carbon fiber manufacturers or potential manufacturers with the conversion protocol necessary to use textile precursors.
6. Upgrade the conventional pilot line to be able to produce larger quantities of material.