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Low Cost Carbon Fiber Research in the LM Materials Program Overview

20 May 2009

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2009 Projects

Full Scale Development of Textile Based Precursors - PAN-VA

Lignin-Based Low-Cost Carbon Fiber Precursors

Advanced Stabilization of Carbon Fiber Precursors

Advanced Oxidation of Carbon Fiber Precursors

Precursor and Fiber Evaluation

LCCF – Commercialization

Stimulus Package, Critical Path & Next Projects

A 10% mass reduction translates to a 6-7% increase in fuel economy or may be used to offset the increased weight and cost per unit of power of alternative powertrains

Increasing Severity of Challenge ←

Increasing Impact on Mission ↑

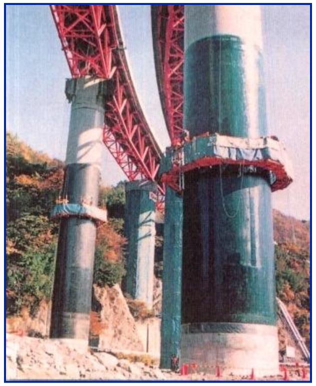
Material Options	Critical Challenges				
Carbon-fiber Composites	Low-cost fibers	High-volume Mfg.	Recycling	Joining	Predictive Modeling
Aluminum	Feedstock Cost	Manufac-ing	Improved Alloys	Recycling	
Magnesium	Feedstock Cost	Improved Alloys	Corrosion Protection	Manufac-ing	Recycling
Advanced High-strength Steels	Manufacturability	Wt. Red. Concepts	Alloy Development		
Titanium	Low-cost Extraction	Low-cost Production	Forming & Machining	Low-cost PM	Alloy Development
Metal-matrix Composites	Feedstock Cost	Compositing Methods	Powder Handling	Compaction	Machining & Forming
Glazings	Low-cost Lightweight Matls.	Noise, T° struc. models simulations	Noise reduction techniques	UV and IR blockers	
Emerging Materials and Manufacturing	Material Cost	Mfg-ability	Design Concepts	Performance Models	

Chart is provided courtesy of Robert McCune - Ford Motor Company

U.S. Department of Energy
Energy Efficiency and Renewable Energy
Bringing you a prosperous future where energy is clean, abundant, reliable, and affordable

Common Issues and Needs

Civil Infrastructure
Rapid Repair and Installation, Time and Cost Savings



Bio-Mass Materials
Alternative Revenue
Waste Minimization

FUELING OUR FUTURE BIOMASS 2008

Fiber Cost
Fiber Availability
Design Methods
Manufacturing Methods
Product Forms

Hydrogen Storage
Only Material With Sufficient Strength/Weight

Power Transmission
Less Bulky Structures
Zero CLTE

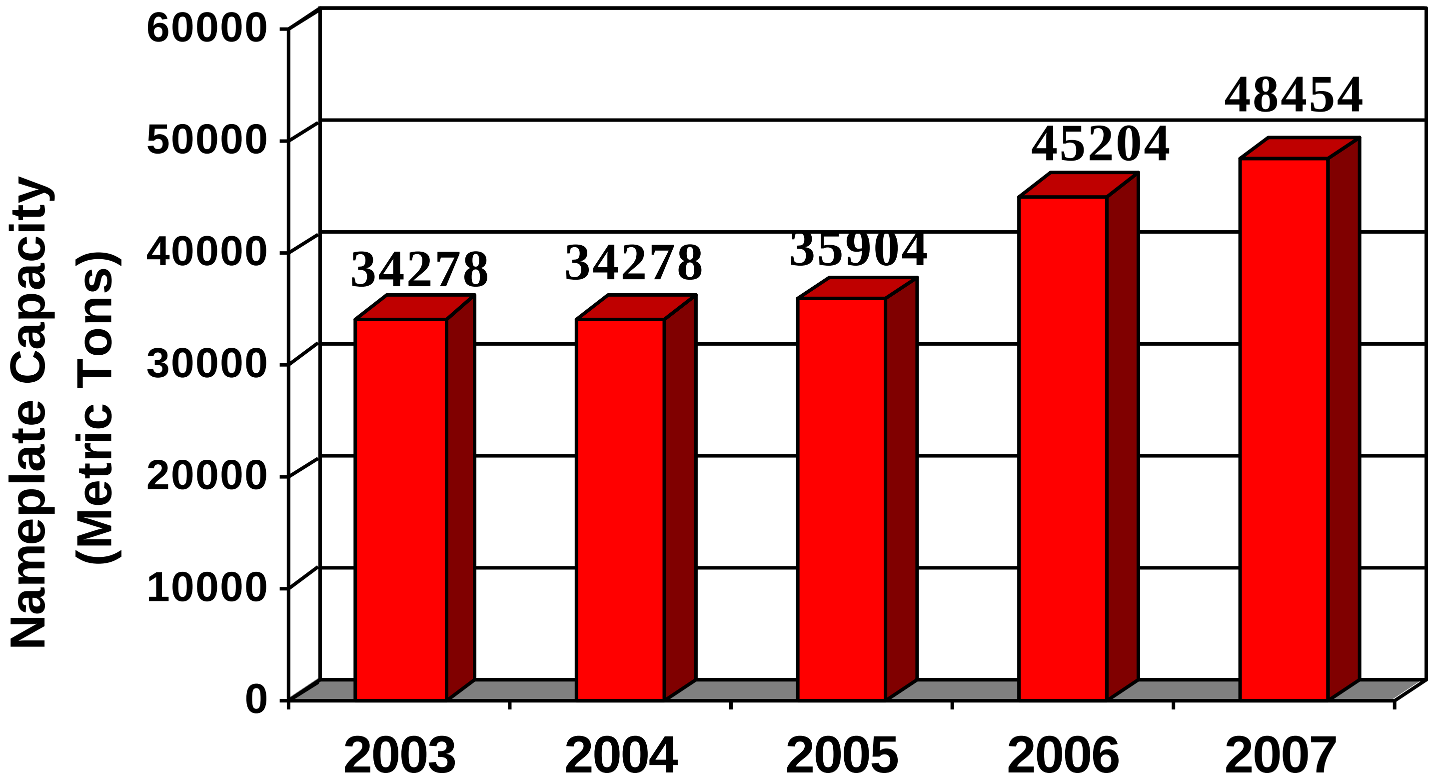
Oil and Gas
Offshore Structual Components

Wind Energy
Needed for Longer Blade Designs

Vehicle Technologies
Necessary for 50+% Mass Reduction

Carbon Fiber Capacity

10 lbs of CF on Each North American Vehicle would consume world supply.



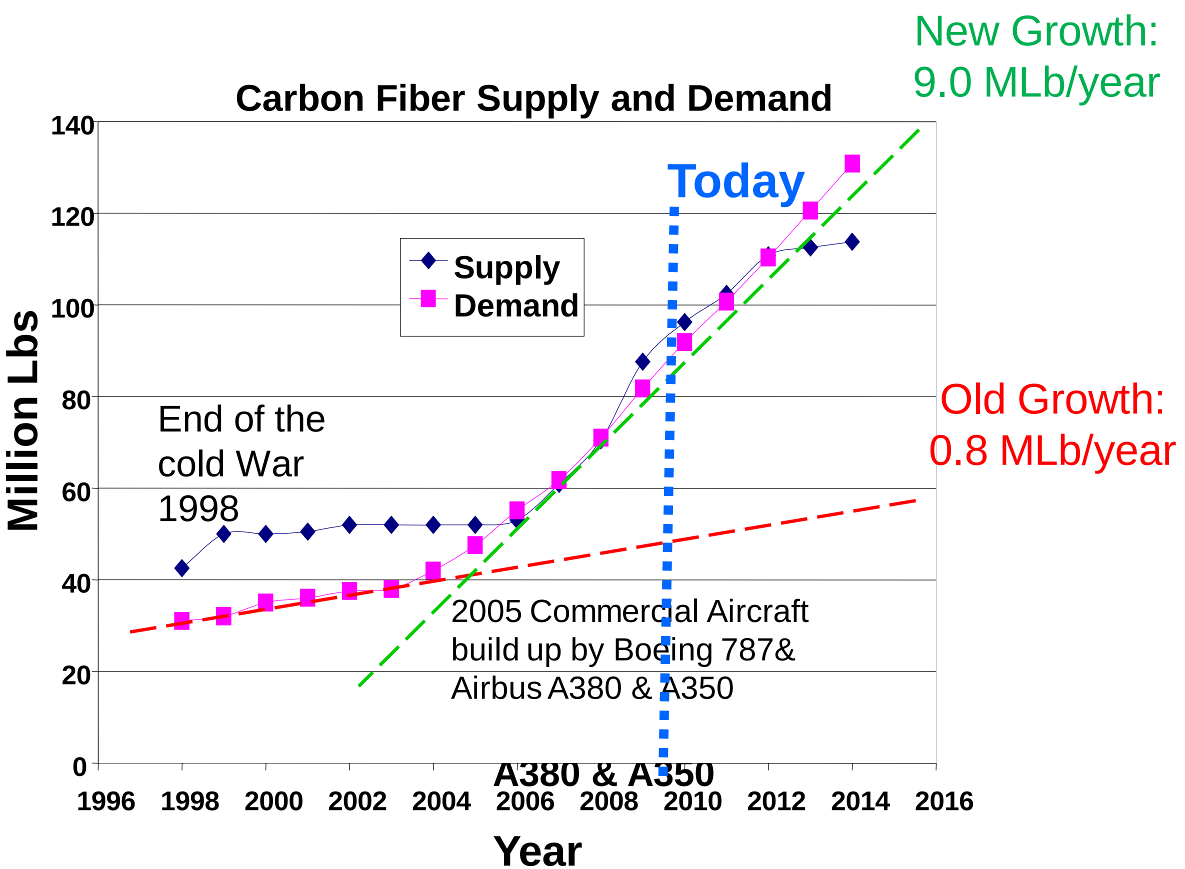
Source: U.S. Department of Defense, "Polyacrylonitrile (PAN) Carbon Fibers Industrial Capability Assessment", OUSD(AT&L) Industrial Policy, October 2005.

Carbon Fiber Price Goal

Vehicle Materials Priority

$5 - $7 Per Pound

Strength: ≥ 250 Ksi
Modulus: ≥ 25 Msi
Strain: ≥ 1%



Source: High Performance Composites

CARBON FIBER – Program Planning

In Early FY2007 the ACC funded a cost model with Kline & Company.

Cost model was used to construct a "Critical Path" for Lower Cost carbon fiber program implementation.

Results of That Cost Model:

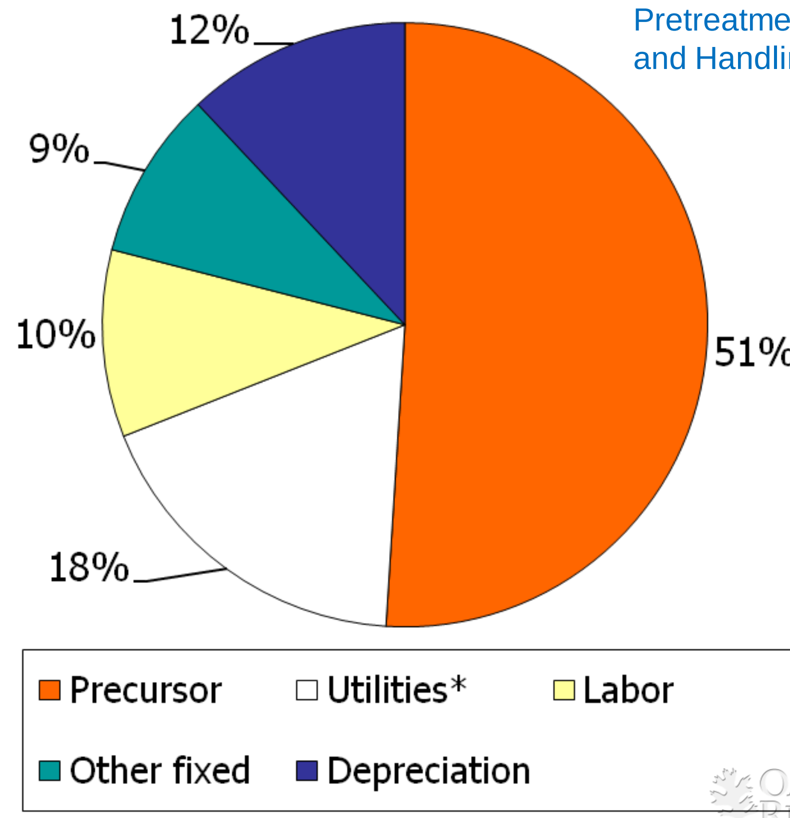
Carbon Fiber Costs (Baseline)

Diagram from Harper International

Spooling & Packaging	Surface Treatment	Carbonization/ Graphitization	Stabilization & Oxidation	Precursors
$0.61 (6%)	$0.37 (4%)	$2.32 (23%)	$1.54 (16%)	$5.04 (51%) Includes Pretreatment and Handling

Baseline - $9.88

- With conventional processing using a carbon fiber-grade (CF) PAN, precursor is over **50%** of the carbon fiber cost



4 Elements of Cost Reduction

1. Scale of Operations
2. Precursors
3. Conversion
4. Manufacturing of Composite

* Data From Kline & Company

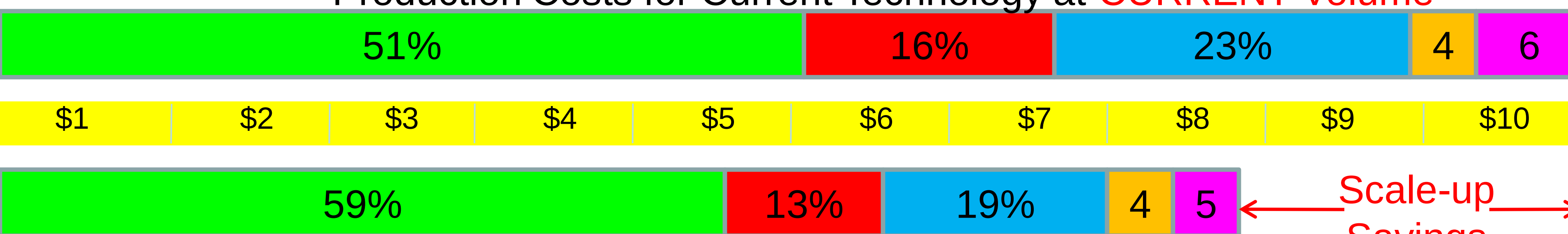
Carbon Fiber Costs (Scale of Operation)

Diagram from Harper International

* Data From Kline & Compar

Precursors	Stabilization & Oxidation	Carbonization/ Graphitization	Surface Treatment	Spooling & Packaging
$5.04 Baseline Today - $9.88	$1.54	$2.32	$0.37	$0.61
$4.64 High Volume - $7.85	$0.99	$1.48	$0.33	$0.41

Production Costs for Current Technology at CURRENT Volume

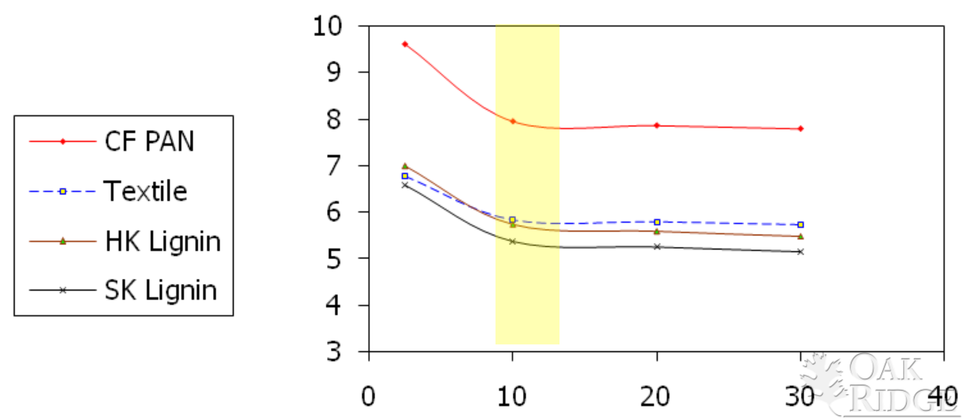


Production Costs for Current Technology in HIGH Volume

Significant Cost Reduction can be achieved by increased Scale-up of Plant and Line Size

But

<u>Not All the Needed Cost Reduction</u>



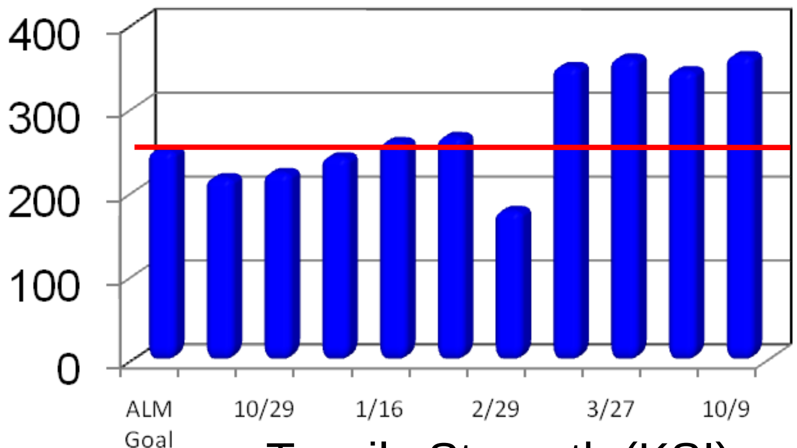
Current Research (Precursors)

More Affordable Precursors are Needed

<u>3 Current Precursor Options</u>

1. Textile Grade PAN (MA or VA formulations)
2. Lignin Based Precursor (Hardwood or Softwood)
3. Polyolefins (not shown on chart)

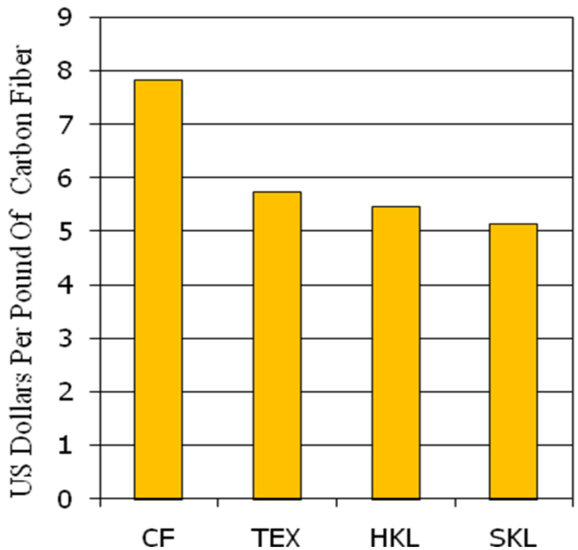
Carbonized Textile Precursor



Tensile Strength (KSI)

Current Carbonized
<u>Textile Properties:</u>
Strength: 370+ KSI
Modulus: 32.0+ MSI

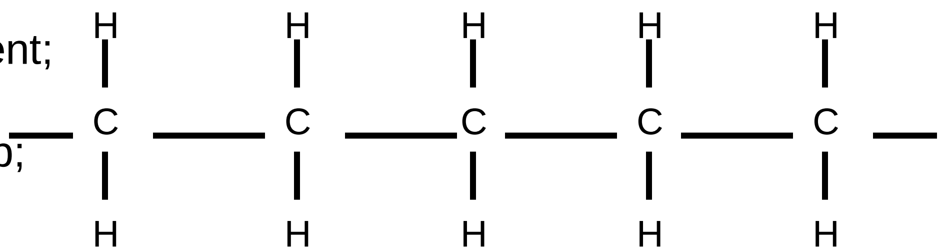
Alternative Precursors and Conventional Processing



Processed Precursor Fibers from a Hardwood/Softwood Lignin Blend.

PE:

86% C Content;
65-75% Yield
$0.50-$0.75/lb;
Melt Spun

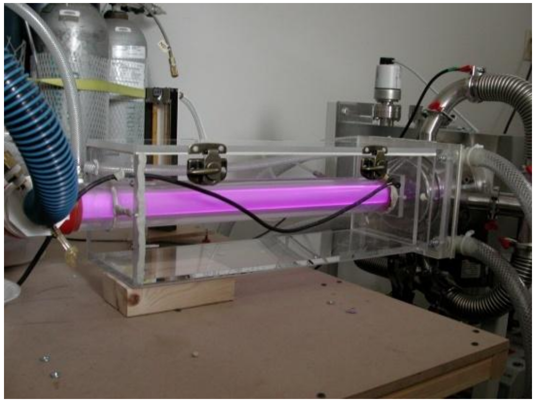


Current Research (Conversion)

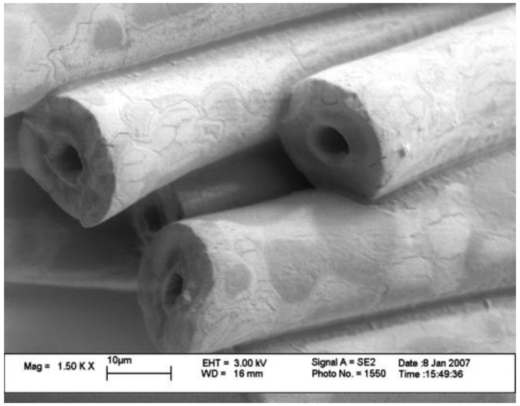
Alternative Processing Methods Under Development

4 Processing Options

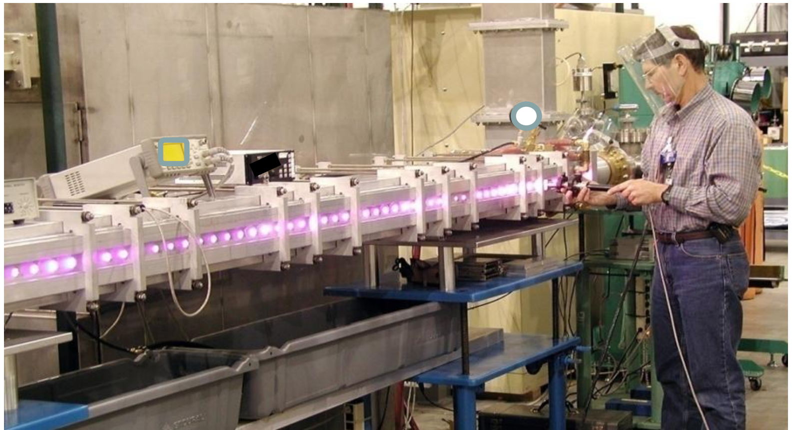
1. Advanced Stabilization
2. Plasma Oxidation
3. MAP Carbonization
4. Surface Treatment (Not on graph)



Early Generation Oxidation Module

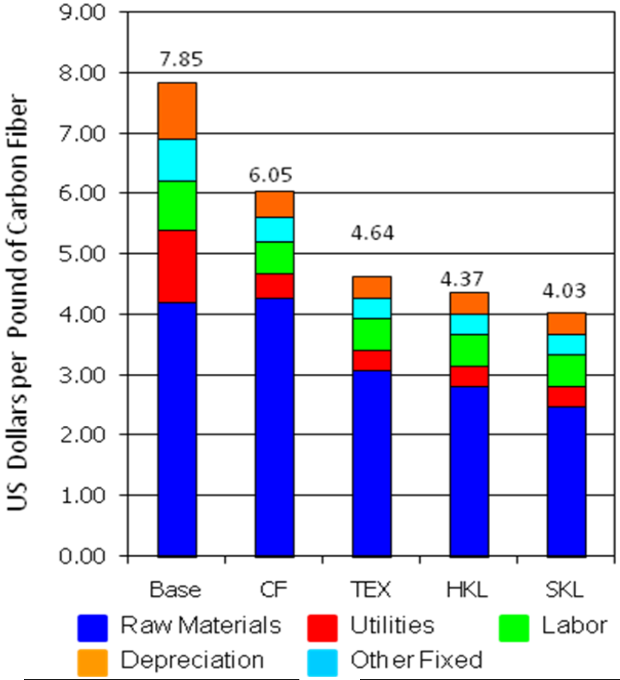


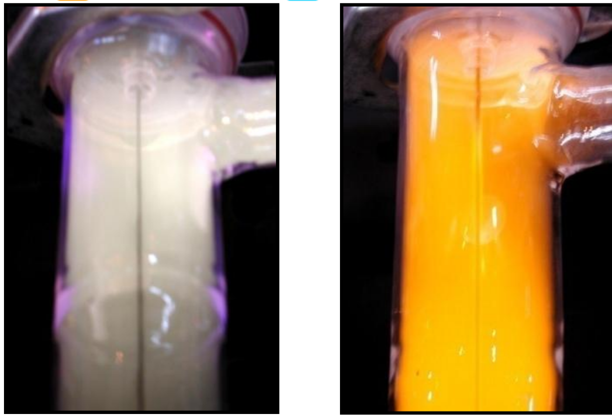
Advanced Stabilization



MAP Carbonization/ Graphitization Unit

Alternative Processing





Advanced Surface Treatment

Current Research (Recent Invention Disclosures)

1920	Naskar, Paulauskas, Ludtka, Ludtka, & Eberle	Electromagnetic processing of carbon precursors for controlled properties of carbon fibers	LM & H2
1924	Naskar, Paulauskas, Janke, & Eberle	Polyolefin based precursors and the processing of the materials for production of carbon fibers	LM & H2
1926	Naskar, Paulauskas, & Eberle	Nano-reinforced high-strength carbon fibers	LM & H2
1973	Naskar, Paulauskas, Janke, & Eberle	Novel compositions for PAN based carbon fiber precursors	LM & H2
2051	Baker, Menchhofer & Baker	In-situ Incorporation of Carbon Nanotubes into Lignin Fibers	LM
2060	Menchhofer, Baker, & Montgomery	Carbon Nanotubes Grown on Various Fibers	LM
2069	Paulauskas & Naskar	Rapid stabilization/oxidation of lignin based precursors for carbon fibers	LM
2212	Several	Carbon Fiber Composites with Enhanced Compression Strength	LM
2239	Several	Polyolefin-based Flame Retardant Material	LM & H2
2241	Naskar & Paulauskas	Extremely Flame Retartant Material from PAN Fibers via Advanced Oxidation	LM

Carbon Fiber Budgets Completed Projects

Project	Thru FY07	FY 08	Total
Melt Spinnable Precursors	1809	0	1809
Coal Based Precursors	950	0	950
LCCF from Textile Precursors	3630	0	3630
Lignin Purification	540	0	540
LCCF from PAN-MA Precursors	0	50	50
Tow Splitting	120	0	120
LCCF for Hydrogen Storage	600	0	600
LCCF Integration / Users Facility	1500		1500
Other Spin-off Proprietary Projects	600	200	800
TOTALS	8549	1050	9599

Not ALM Funding – Not figured in Totals

Carbon Fiber Budgets – Current & Planned

Project	Thru FY07	FY 08	FY 09	FY 10	FY 11	Total
Lignin Purification	540	0	0	400	0	940
Lignin-Based LCCF Precursors ✓	5648	525	900	975	900	8948
Full Scale Development of PAN-VA Precursors ✓	200	500	500	0	0	1200
Full Scale Development of PAN-MA Precursors	0			200	500	700
Commercialization of Precursors ✓		450	100	500	TBD	1050+
Microwave Assisted Plasma (MAP)	4400	0	0	0	1250	5650
Carbon Fiber Surface Tailoring	0	0	0	200	200	400
Advanced Oxidation of CF Precursors ✓	1900	975	1415	1150	1900	7340
Advanced Stabilization of CF Precursors ✓	1005	290				1295
Other Spin-off Proprietary Projects ✓	800	N/A	N/A	N/A	N/A	N/A
Precursor & Fiber Evaluation ✓	0	200	200	200	200	800
Polyolefin Precursors	0	0	200	400	600	1200
TOTALS	13693	2940	3315	4025	5550+	29523+

Not ALM Funding – Not figured in Totals

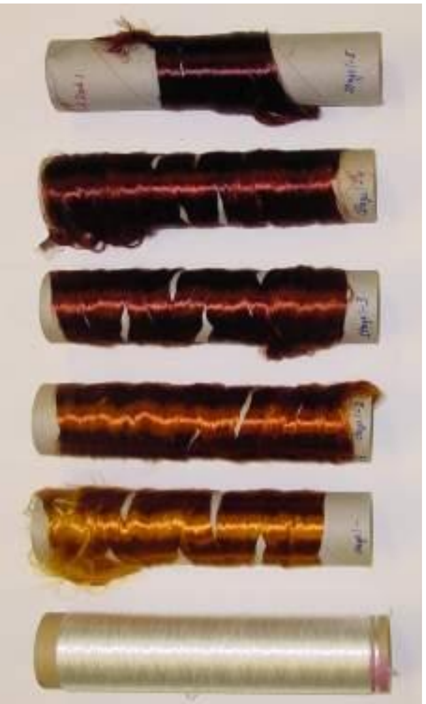
✓ Today's Presentations

Presentations

Time	Agreement Number	**Technical Presentation**	**Briefing**	**Presenter(s)**	**Funding ($) FY 09**
1:45 – 2:00		Overview of Low-Cost Carbon Fiber (LCCF)	LM - 02	Dave Warren	
2:00 – 2:15	16622	LCCF – FISIPE PAN VA Precursors		Dave Warren	500,000
2:15 – 2:45	8987	LCCF from Renewable Resources	LM – 03	Fred Baker	900,000
2:45 – 3:15	13323	Advanced Stabilization of Carbon-Fiber Precursors	LM – 04	Felix Paulauskas	1,415,000
	9442	Advanced Oxidation of Carbon-Fiber Precursors		Felix Paulauskas	
3:15 – 3:25	17859	LCCF – Precursor & Fiber Evaluation	LM – 05	Cliff Eberle	200,000
3:25 – 3:35	16623	LCCF – Commercialization		Cliff Eberle	150,000
3:35 – 3:45		LCCF – DOE Planning & Critical Path Forward		Dave Warren	

Project 16622

Low Cost Carbon Fiber
Textile Based Precursors PAN-VA



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Textile Precursors – Purpose, Barrier, Approach

Project 16622

Purpose: Scale-up chemical modification of textile precursors, in conjunction with an industrial partner. Identify and develop textile based precursor. Develop optimal processing parameters. Incorporate both in commercial production facilities at industrial scale.

Barriers: New precursors are needed for carbon fiber manufacturing cost reduction. They must be scaled for industry production and conversion parameters must be optimized.

Approach:

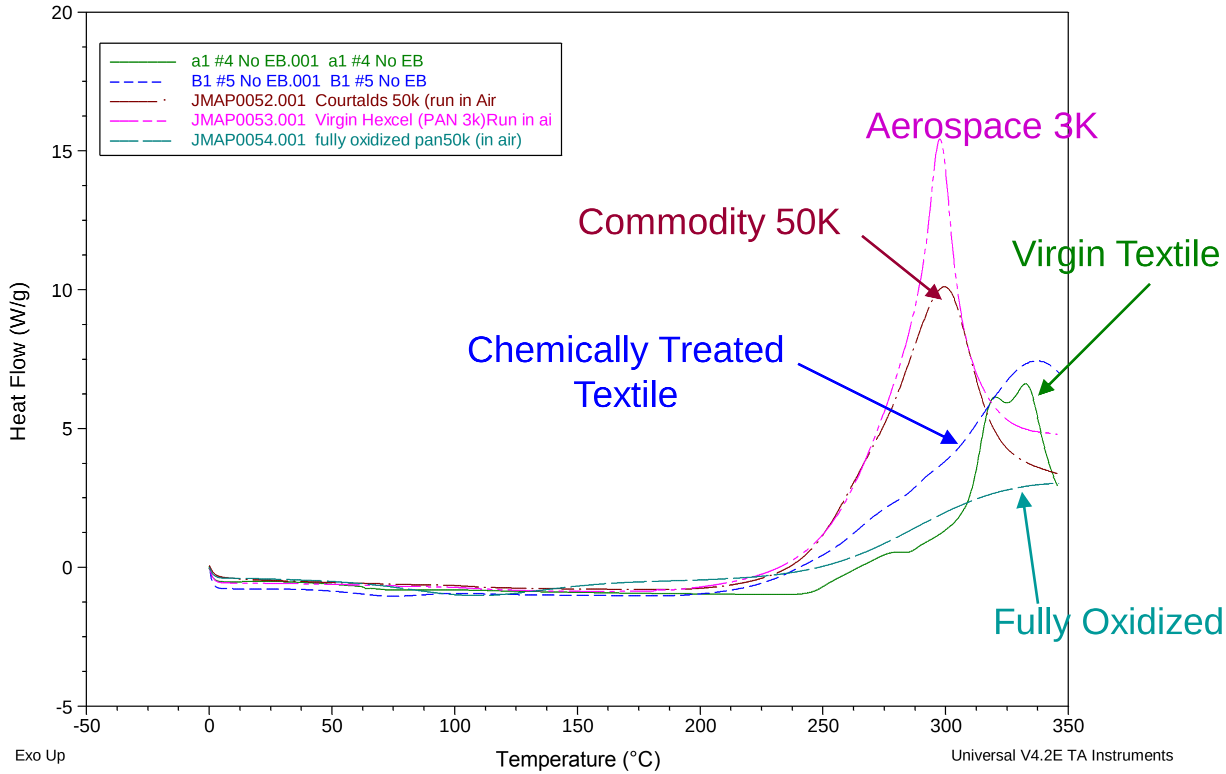
1. Identify appropriate textile PAN formulation working with a textile producer.
2. Develop chemical modification protocol.
3. Develop conversion protocol (time-temperature-tension, 3T profile)
4. Incorporate modification in “precursor” plant.

Budget: $ 500K **Project Dates:** Jan 2007 - October 2009

Textile Precursors

Project 16622

Early Work Done by Hexcel 2001-2004 under LM Subcontract



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

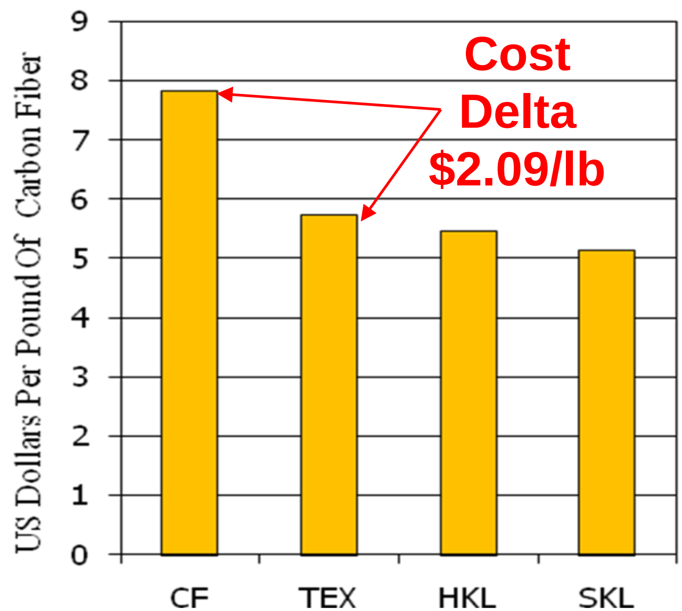
Chemically treated textile could be undergo oxidative stabilization in less time but a slightly higher temperature.

Textile Precursor – Purpose

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

Textile Precursors could be readily brought to market because:

- Textile mills already in existence making material in 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.



Project 16622

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

Textile Precursor – Accomplishments (FISIPE)

Began: January 2007 Partner: FISIPE (PT)

FISIPE

Guided FISIPE in the chemical pretreatment.

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

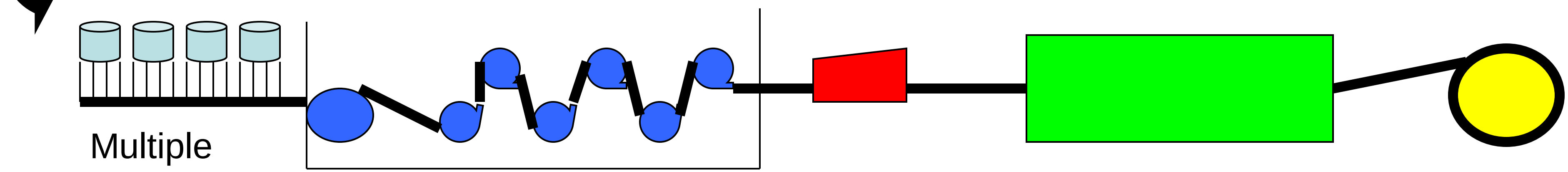
Due to export control restrictions, FISIPE does not know the conversion conditions. We will work directly with the converter to transfer that.

Project 16622

Textile Precursors

Project 16622

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



Multiple Spinnerettes

Solvent Extraction, Washing and Tensioning

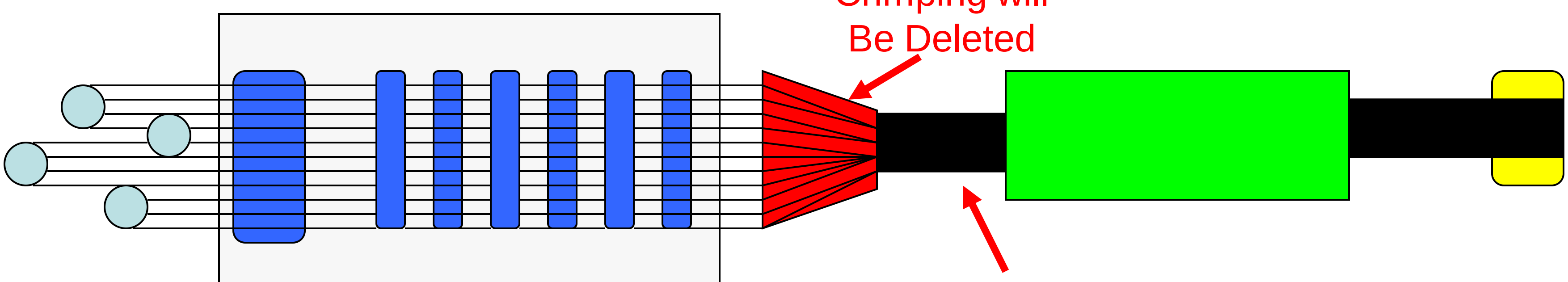
Crimping

Drying with Tension

Spooling or Bailing in Bulk

Slightly Modified versions of polymer selected

Gathering during Crimping will Be Deleted

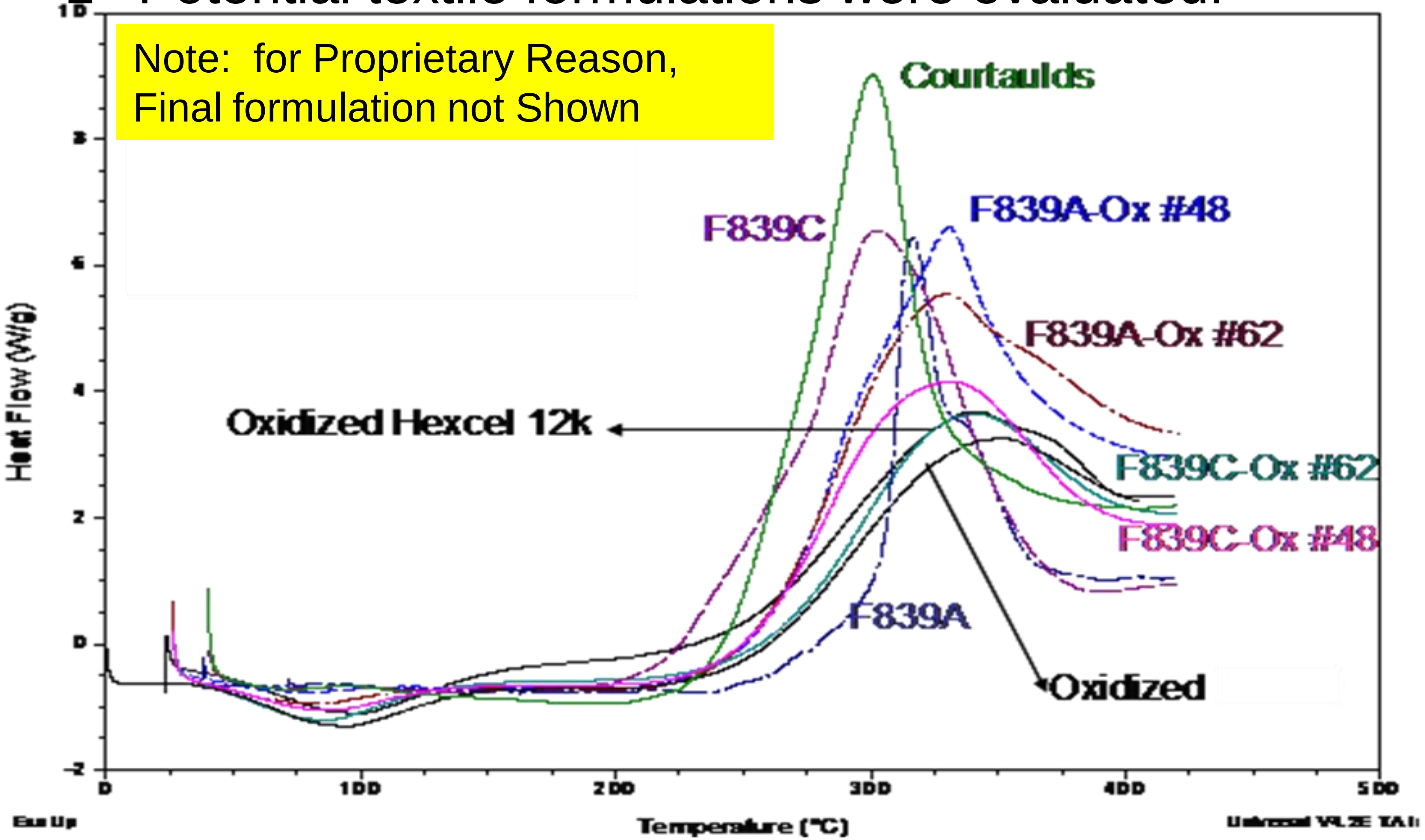


Chemical Pretreatment

Project 16622

1st Potential textile formulations were evaluated:

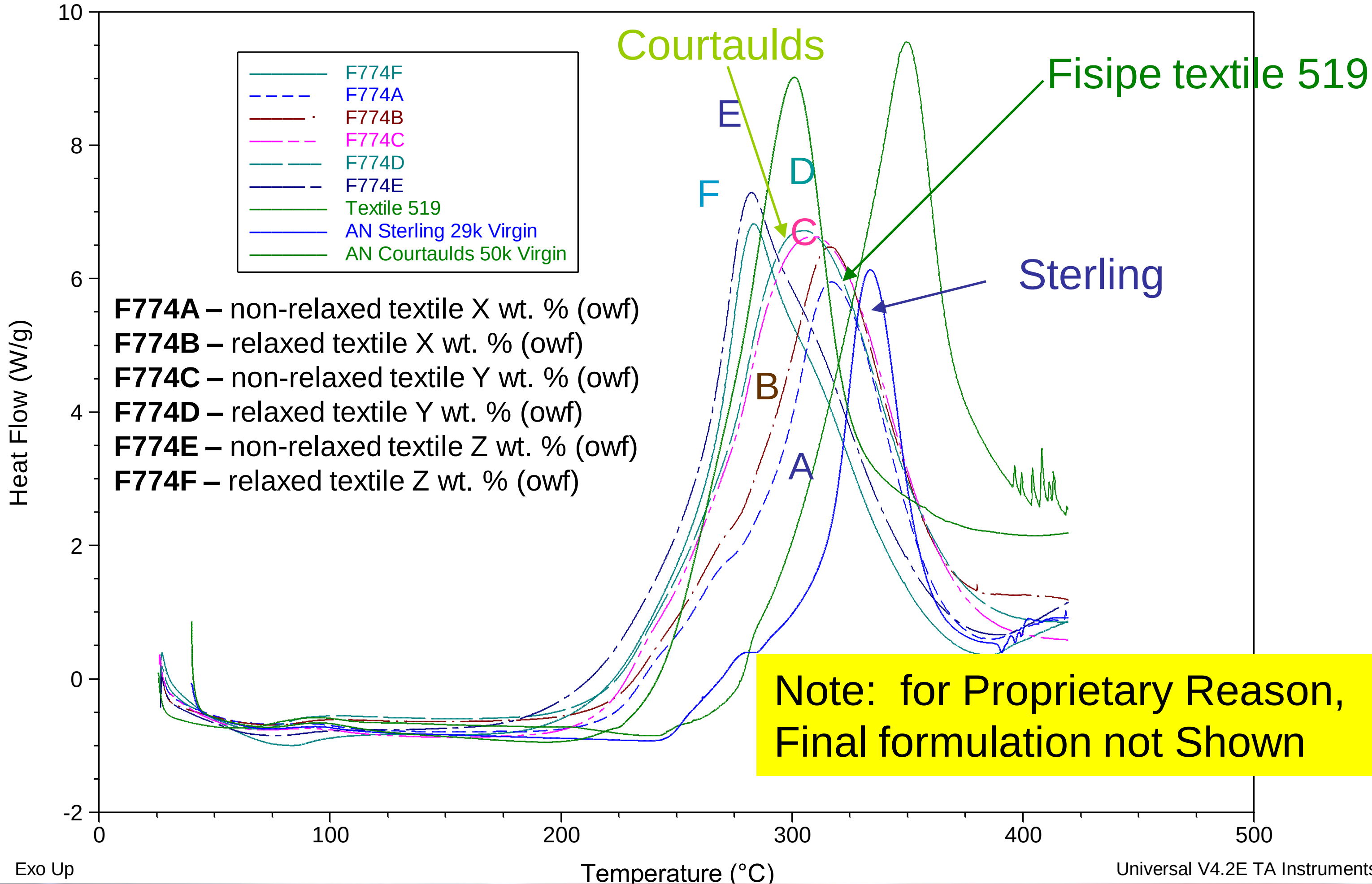
Note: for Proprietary Reason, Final formulation not Shown



Early Pretreatment Trials

Project 16622

Next Chemical Pretreatment Trials were Conducted:



Note: for Proprietary Reason, Final formulation not Shown

Textile Precursors

Project 16622

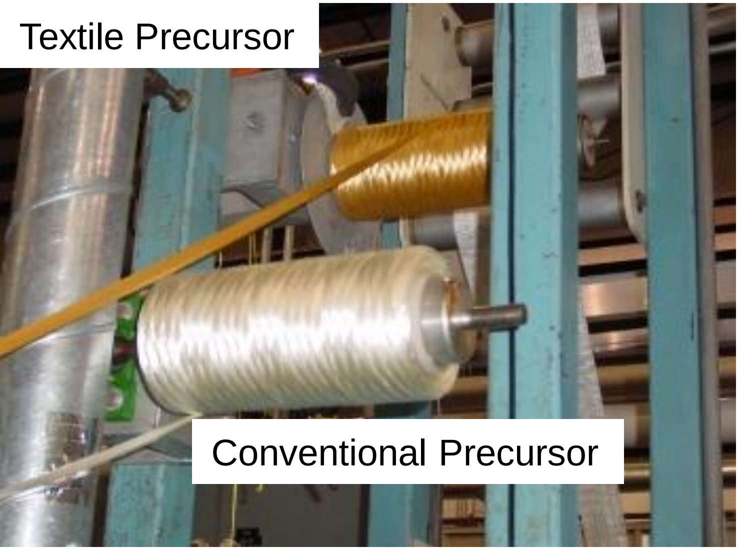
In January 2007, we began some initial work with FISIPE once it became apparent that commercialization of textile precursor is the quickest route to significant cost reduction.

Chemical Modification has now been scaled from the pilot line below to one of their full scale lines in their production facilities.

Textile Pilot Plant

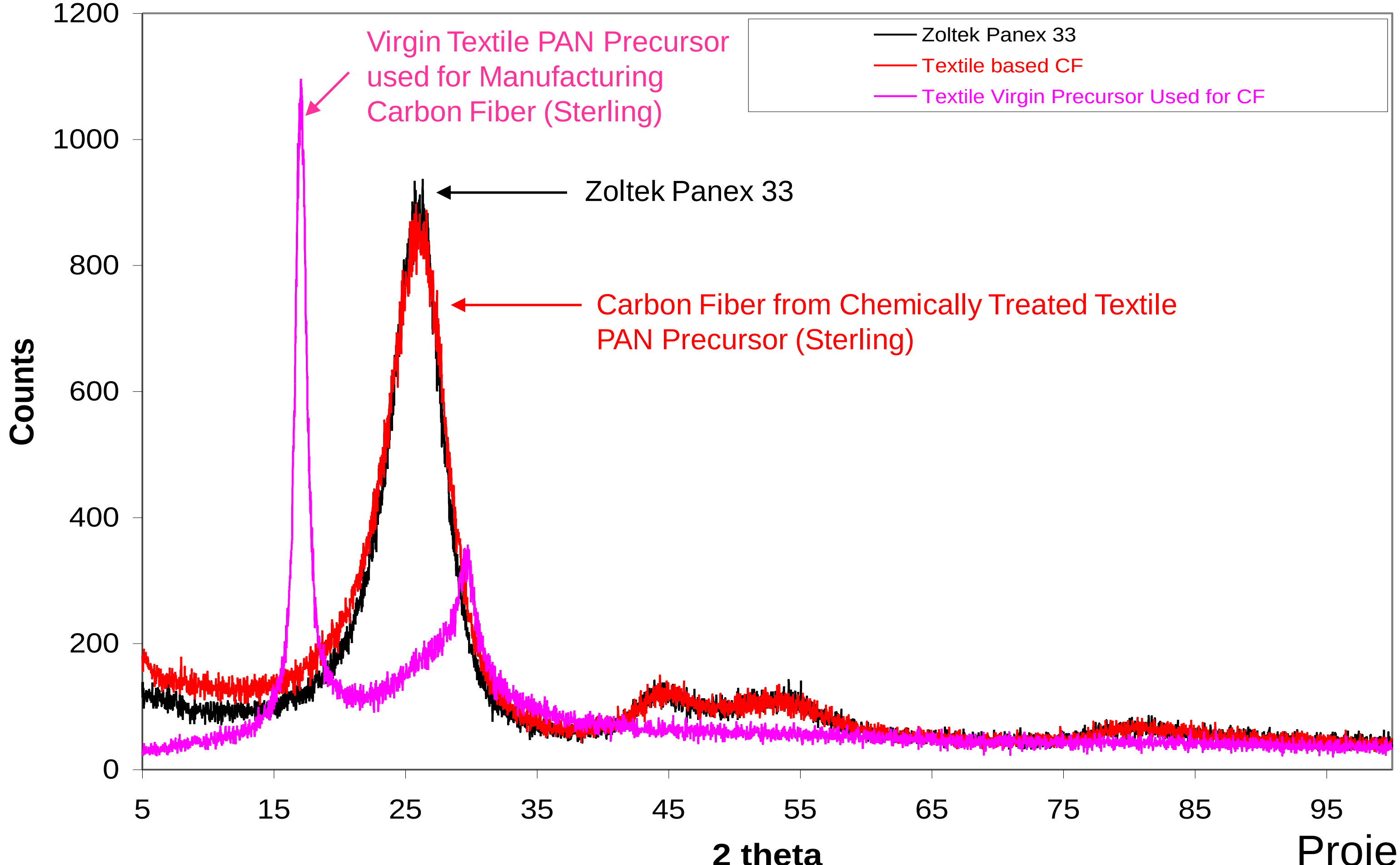
Textile Precursors

Project 16622



- Textile PAN_VA Co-monomer
- Formulation proprietary to FISIPE
- 26,600 filament tow size
- Chemically treated with a proprietary solution treatment at “normal” processing temps
- Oxidized and Carbonized using both the Precursor Evaluation Line and the Pilot Line
- Test data from both tow tests and single filament tests

Wide Angle XRD Profiles: Comparison Between Commercial Panex 33 and Textile Grade Carbon Fiber



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Textile Based Precursors

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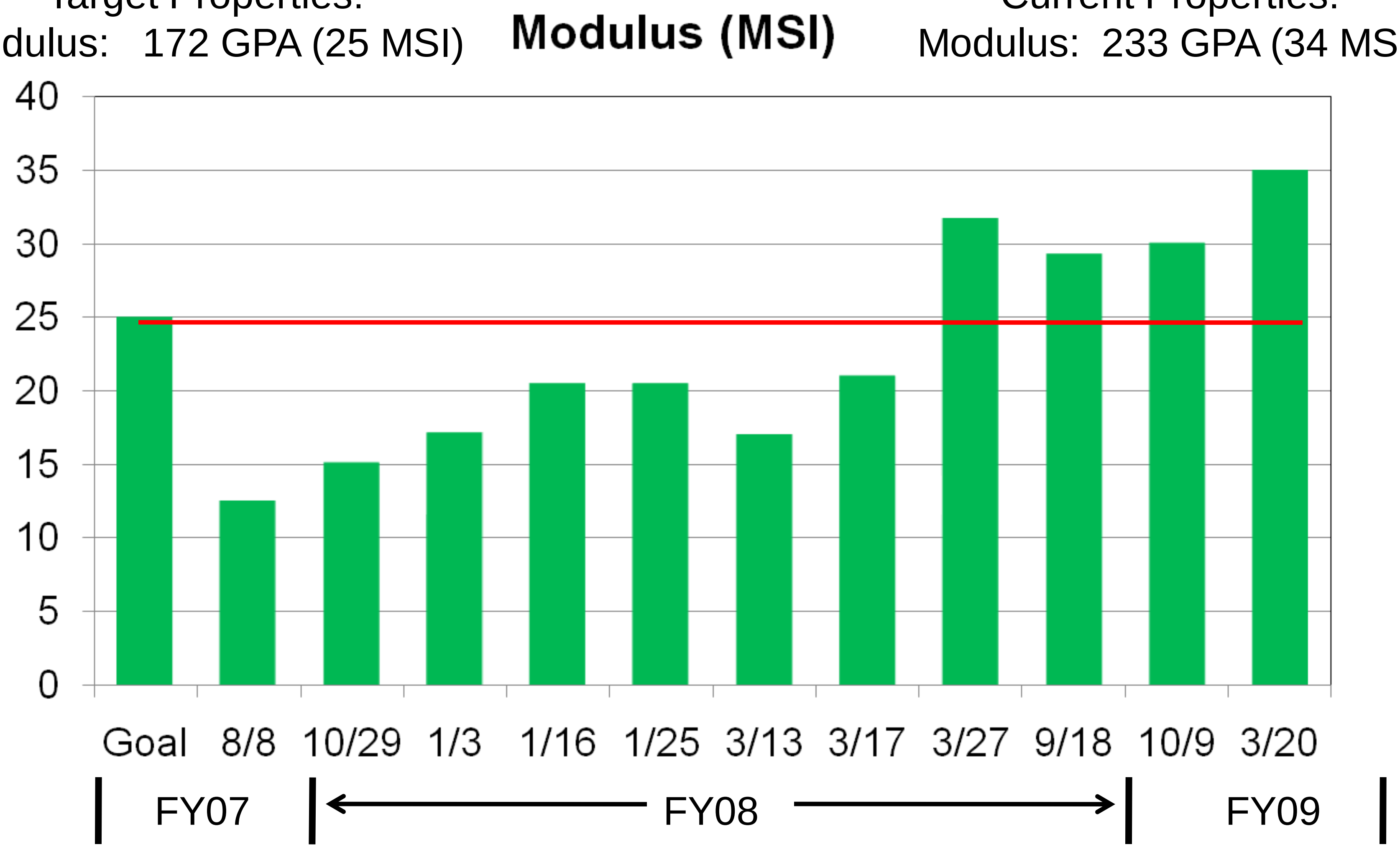
Target Properties:
Modulus: 172 GPA (25 MSI)

Current Properties:
Modulus: 233 GPA (34 MSI)

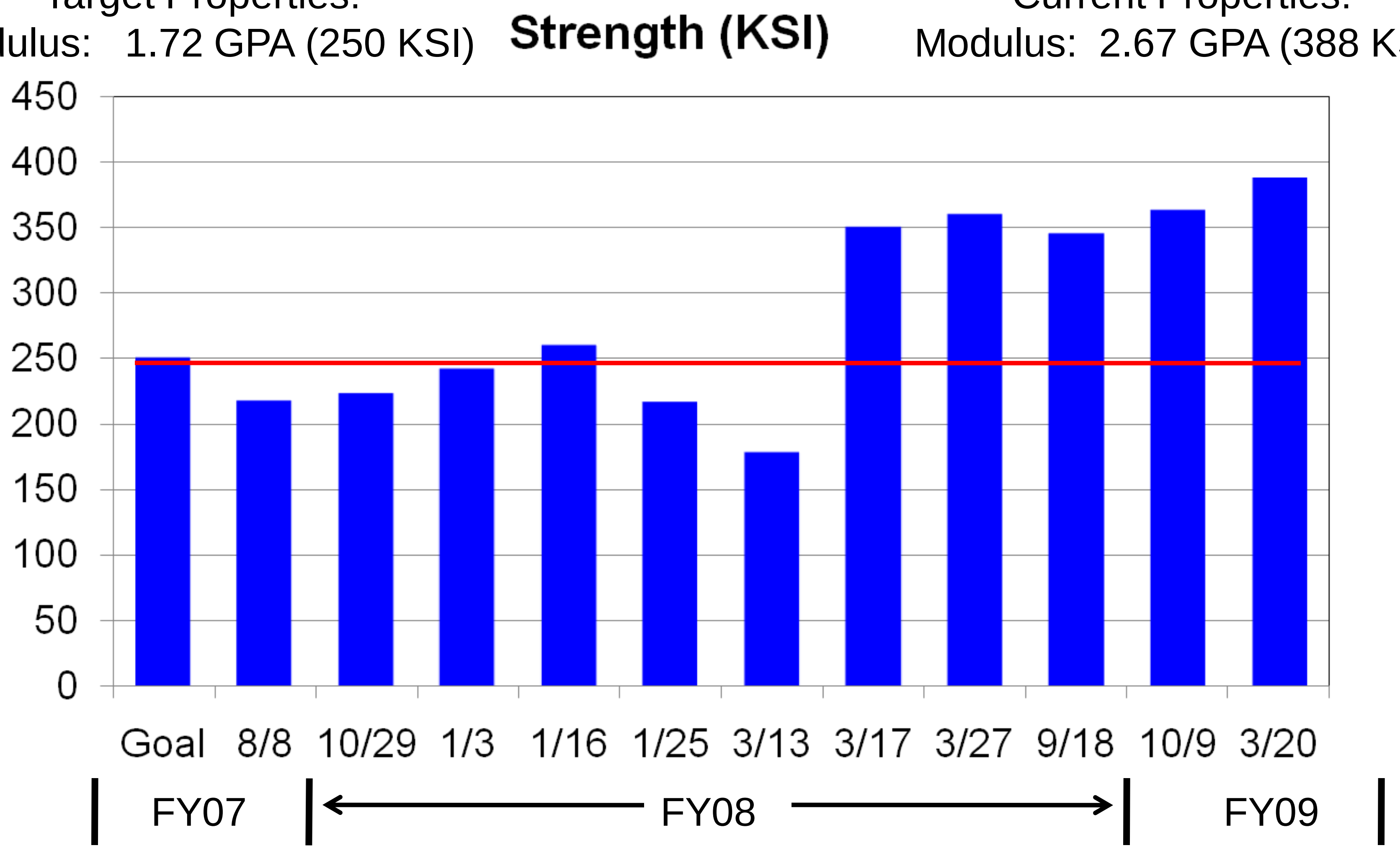


Textile Based Precursors

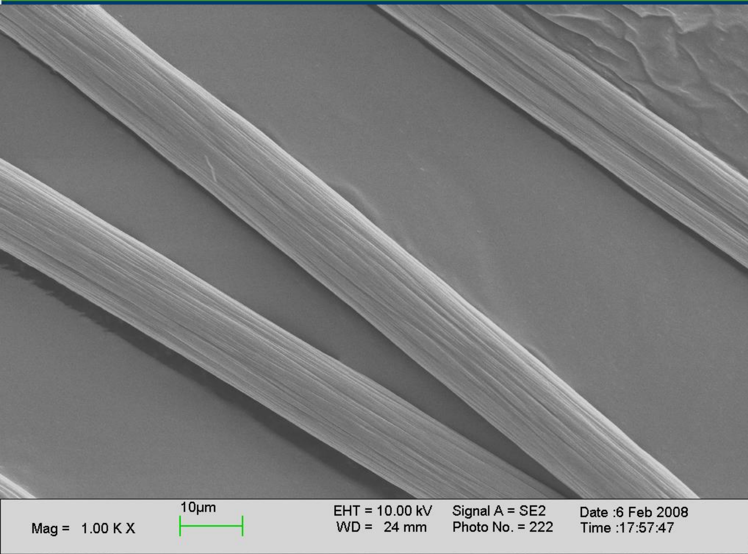
Project 16622

Target Properties:
Modulus: 1.72 GPA (250 KSI)

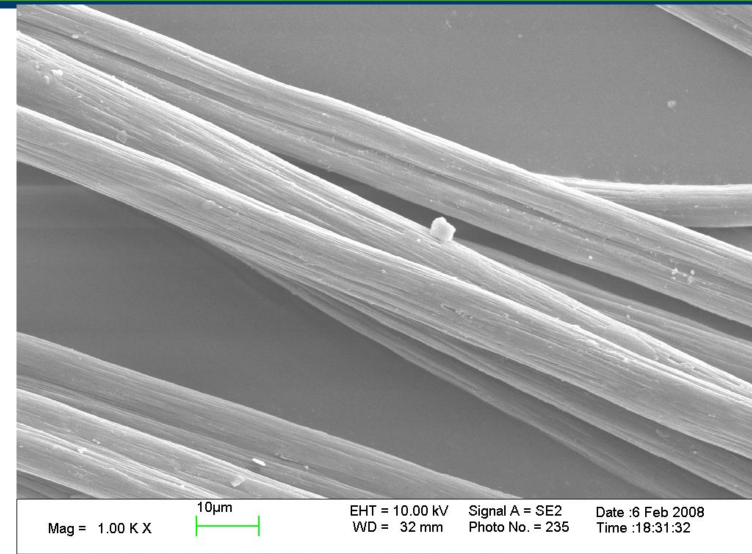
Current Properties:
Modulus: 2.67 GPA (388 KSI)



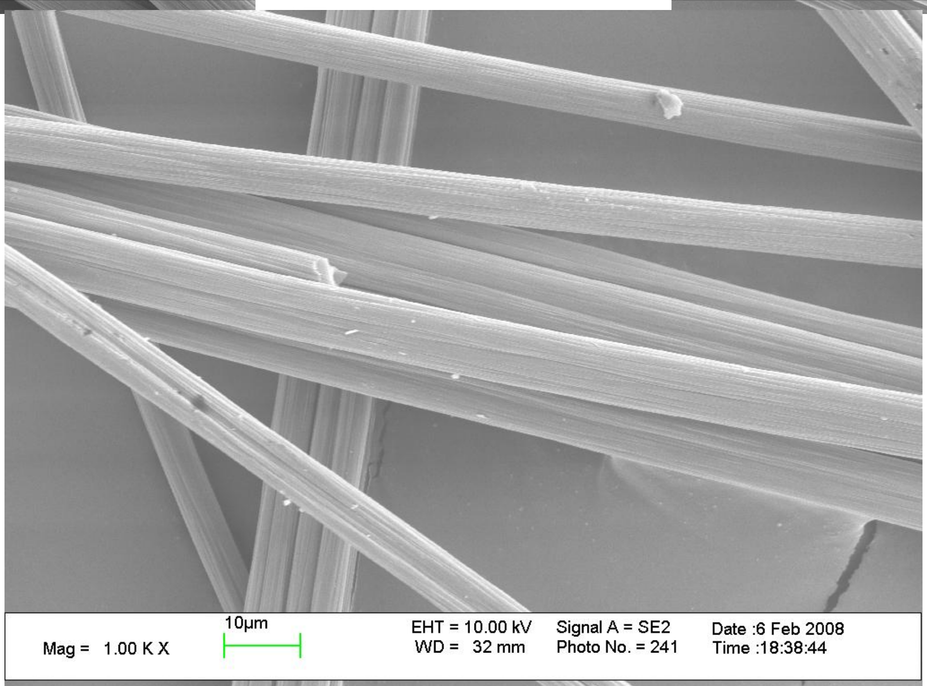
Textile Based Precursors



Oxidized textile



LT carbonized textile



HT carbonized textile

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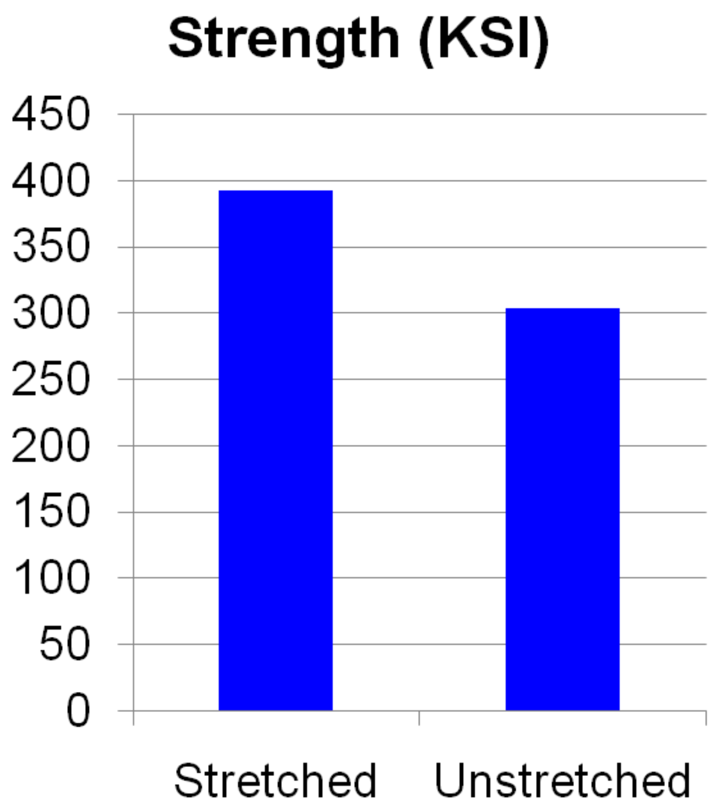
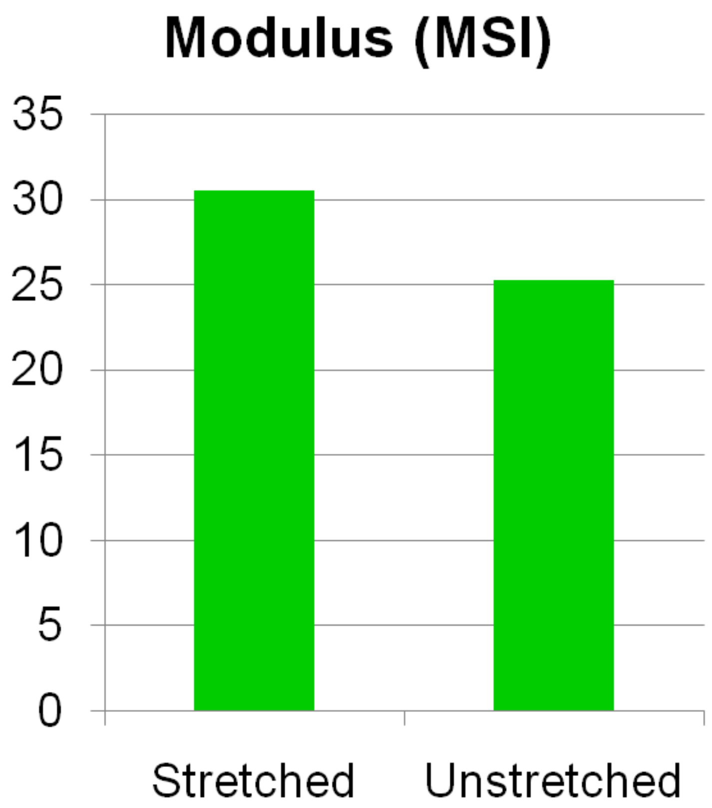
Textile Based Precursors

Project 16622

Recent work has been focused on adding Pre-Conversion Stretching.
Next is Optimizing Tensioning during Processing.





Also, determined that apparent aging effect was a non-issue.

Papers:

Project 16622

1. **"**FreedomCAR and Low Cost Carbon Fiber for Automotive Applications," Low Carbon Generation, 3M innovation Centre, London, UK , 9 February 2008.

2. "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.

3. "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.

4. "Multi-Task Research Program to Develop Commodity Grade, Lower Cost Carbon Fiber"; proceedings of the 2008 SAMPE Fall Technical Conference; September 8-11, 2008; Memphis, TN.

5. "Future Lower Cost Carbon Fiber for Autos: International Scale-up & What is Needed"; Proceedings of the 2008 SPE Composites Conference; September 16, 2008; Detroit, MI.

6. "Investment in the Carbon Fibre Business for Commercial Grade Low Cost Composites"; Proceedings of the Carbon Fibre 2008 Conference; September 30 – October 2, 2008; Hamburg, Germany.

7. "Development of Lower Cost Carbon Fiber for High Volume Applications"; Proceedings of the Composites + Polycon 2009 Conference; January 15-17, 2009; Tampa, FL.

8. "Novel Precursor Materials and Approaches for Producing Lower Cost Carbon Fiber for High Volume Industries", To be presented at and published in the proceedings of 17th International Committee on Composite Materials ICCM-17, July 27-31, 2009, Edinburgh, Scotland, UK.

Textile Precursor – Publications/Patents

Project 16622

Patents:

None

Awards:

DOE and ACC Composites Team: 2008 Outstanding Leadership Team Award for the advancement of plastics in automotive given by the Society of Plastics Engineers Automotive Division. Received November 20, 2008.

Textile Precursor – Plans and Summary

Project 16622

1. Complete development of textile VA-PAN with FISIPE.

2. Begin development of MA-PAN with at least one company.

3. Optimize current Precursor processing parameters.

4. Do preliminary work on processing of an improved precursor formulation based upon what was learned from the current work.

5. Assist carbon fiber manufacturers or potential manufacturers with the conversion protocol necessary to use textile precursors via a separate commercialization project.

6. Complete tow in-plant tow pre-splitting from 80K down to 20K or 40 K tows.