Precursor and Fiber Evaluation

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ORNL

March 20, 2009

Project ID lm_05_warren

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Oak Ridge National Laboratory

Overview

Timeline

- Start- 2008
- End- 2015
- Percent complete – ~ 15%

Barriers

- Barriers addressed
  A. High cost of carbon fiber
  B. High volume manufacturing of carbon fiber

Budget

- $200k/yr

Partners

- ORNL
- Some equipment mfrs have provided complimentary or discounted equipment

Objectives/Milestones

- Develop, maintain, and operate a full suite of hardware for the development and evaluation of carbon fiber conversion protocols of a range of precursors

Date	Milestone or Go/No-Go Decision
Sept-08	Precursor evaluation system in routine use evaluate conversion protocols
Mar-09	2500ºC furnace installed
Sept-09	Fibers delivered to ACC upon request

Oak Ridge National Laboratory

Technical Approach

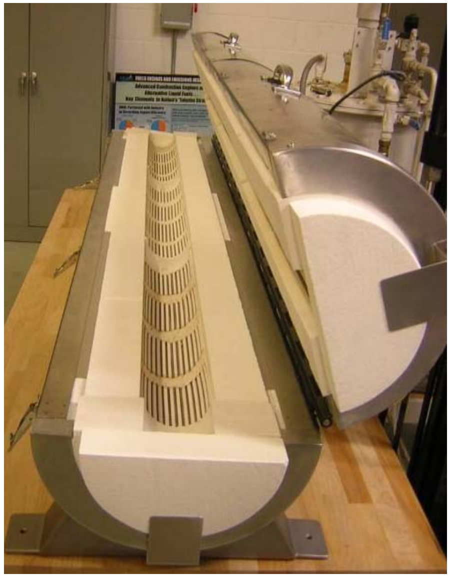
- Maintain and operate conventional pilot line, advanced carbonization unit, and precursor evaluation system
- Evaluate new precursors and develop the initial conversion protocols using small quantities of precursor
  - Precursor evaluation system enables conversion trials on single-filament or few-filament precursor samples, in single-shift operation

Conventional Pilot Line

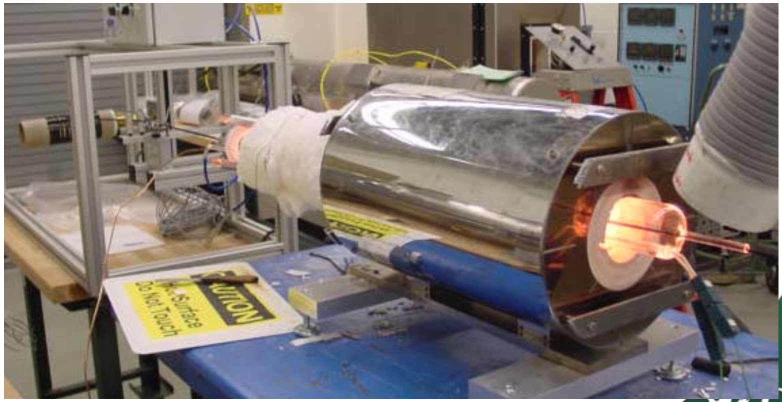
- 1:20 scale of a commercial grade production line
- Capacity for 8 tows
- Preferred tow size ≥ 3k

Precursor Evaluation System

- Designed for development of conventional processing recipes with limited quantities of precursor
  - Residence time, temperature, atmospheric composition, and tension are independently controlled in each furnace
  - Can process single filament up to thousands of filaments
  - Precise tension control and stretching capability allows stretched/tensioned processing of ~20-filament tows
  - Temperature capability from room temperature to 1,700ºC; 2,500ºC furnace received March 2009
  - Single-shift operation



This has become our “workhorse” equipment system



MAP Carbonization System

MAP carbonization hardware continues to be maintained and operated for demonstration purposes

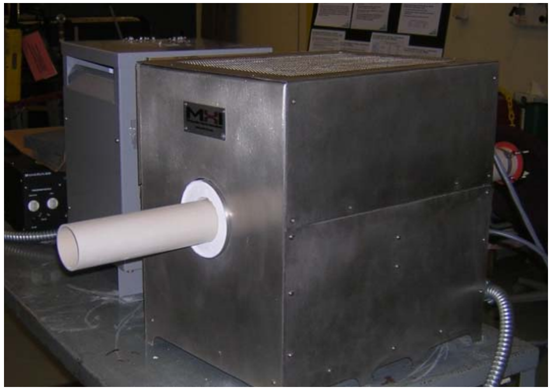
Special Capabilities

Laboratory Box Oven
for Tow Oxidation

Low-Force Tension
Controller

Dancing Tension
Controller

~ 2,500 ºC Furnace
(Similar Model)



New 1,750 ºC Furnace

Primary Accomplishments

- Pilot line
  - Added remote monitoring capability
  - Delivery system for multiple spools of precursor fibers
  - Independent tensioning of multiple large tows
- Precursor evaluation system
  - Textile PAN, proprietary lignin, and polyolefin precursor trials conducted
  - 1,750ºC replacement furnace received
  - 2,500ºC tube furnace received
- MAP carbonization system
  - Frequent demo operations

Future Work

- **Rest of FY09**
  - Install and commission 1,750 ºC and 2,500 ºC furnaces in new precursor evaluation lab space
  - Tune pilot line operation with multiple tows
- **FY10**
  - Relocate existing precursor evaluation equipment to new lab space
  - Add differential stretching capability to conventional pilot line

Date	Milestone
Mar-09	2,500 ºC furnace installed
Sept-09	Fibers delivered to ACC upon request

Date	Milestone
Mar-10	Complete relocation of existing precursor evaluation equipment
Sept-10	Differential stretching equipment ordered

Summary

- Major accomplishments
  - Trialed textile PAN, proprietary lignin, and polyolefin precursors in precursor evaluation line
  - Upgraded pilot line for improved multiple-tow operation
  - Ordered and received 1,750 ºC and 2,500 ºC furnaces for precursor evaluation system
- Continuation plans
  - Continue maintaining, operating, and upgrading these systems as appropriate to meet the needs of ongoing projects and future programmatic needs

Low Cost Carbon Fiber Commercialization

Dave Warren and Cliff Eberle

ORNL

March 20, 2009

Project ID # 16623

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Overview

Timeline

- Start- 2008
- End- 2016
- Percent complete – ~ 10%

Barriers

- Barriers addressed
  A. Deployment of low cost carbon fiber

Budget

- FY08 $450k
- FY09 $100k

Partners

- ORNL
- Numerous prospective partners

Objectives

- **Develop and execute partnerships and strategies leading to rapid LCCF commercialization and deployment**

Date	Milestone or Go/No-Go Decision
Dec 2011	First commercial sales of textile PAN precursor fibers
Dec 2014	Groundbreaking on high-volume plant for conventional manufacturing of lignin-based carbon fiber
Dec 2016	First commercial orders for advanced conversion equipment

Technical Approach

- Partner with industrial end users to ensure that we are delivering products that meet their needs
- Develop an IP portfolio and partnership/licensing strategy that delivers compelling value to potential partners and fulfills our sponsor's mission
- Publish the results of our work in journals, at conferences, workshops, symposia, etc.
- Work with industrial partners in CRADAs and other partnership mechanisms to accelerate and complete development and "technology handoff" to manufacturers
- Partner with government and industry to develop a national demonstration facility that will deliver sufficient quantities of low cost carbon fiber to prove that it can be used effectively in downstream manufacturing processes to deliver affordable, high quality products

Commercialization Status

- ORNL has an ongoing partnership with FISIPE to introduce textile PAN precursor fibers – commercialization imminent
- ORNL is currently negotiating a CRADA with a partner, with the goal to significantly accelerate the development and commercialization of lignin-based precursor
- ORNL is in negotiation with a prospective new entrant into carbon fiber manufacturing, that plans to begin with textile PAN fibers and later adopt other technologies as they mature
- ORNL is engaged in serious "due diligence" discussions, that cannot be accurately characterized as negotiations, with several companies
  - Conversion equipment manufacturers
  - Prospective precursor manufacturers
  - Prospective carbon fiber manufacturers
- ORNL routinely fields calls (weekly to monthly) from companies considering the use of LCCF technology; in many cases these are prospective new entrants into the carbon fiber industry

Workshop on Low Cost Carbon Fiber Composites for Energy Applications

- Held in Oak Ridge 3 – 4 March 2009
- ~ 80 attendees from a wide range of industry and government
- Short plenary session followed by four ~ day-long breakout sessions for facilitated brainstorming
- Agenda, presentations, and breakout reports posted to the web at http://events.energetics.com/carbon_fiber09/
- Final report has been posted or soon will be posted
- Generated a high degree of apparent enthusiasm and momentum for accelerated commercialization of low cost carbon fiber composites technology
- Follow-up planning and actions underway

Workshop on Low Cost Carbon Fiber Composites for Energy Applications (2)

Automotive talk – opening plenary

“This is REALLY HARD stuff…”

“It’s been a long day!”

“Are we ready to vote?”

“I think we can agree on that”

“Can we go home yet?”

Infrastructure Need

- ORNL and partners have the facilities necessary to develop low cost carbon fiber composites technology
- A facility is needed to provide sufficient quantities of low cost carbon fiber to end users and/or their suppliers that they can prove it will work in their manufacturing processes and deliver affordable, high quality composite structures
  - Conventional conversion line to make fibers from alternative precursors, esp. lignin, polyolefins, and unconventional PAN
  - Melt spinning line to provide adequate quantities of melt spun, low cost alternative precursors
  - Space for the future addition of advanced conversion process modules

Future Work

- Continue development and execution of partnerships to rapidly commercialize low cost carbon fiber composites
- Develop and manage IP portfolio to deliver maximum value to partners and sponsor
- Continue publication of our work and its results in appropriate journals and venues
- Complete follow-up of Workshop on Low Cost Carbon Fiber Composites for Energy Applications
- Partner with industry as appropriate on the development of a National Demonstration Facility for low cost carbon fiber composites technology
- Work with industry partners to effectively and rapidly deploy the technology at full scale

Summary

- We have a unique opportunity to significantly accelerate the commercialization and deployment of low cost carbon fiber composites technology
- ORNL is actively developing partnerships to achieve rapid commercialization
  - Several end use industries
  - Throughout the value chain
- The recent Workshop on Low Cost Carbon Fiber Composites for Energy Applications generated momentum that we are working to maintain and exploit

Low Cost Carbon Fiber
Research in the LM Materials Program
Critical Path

20 May 2009

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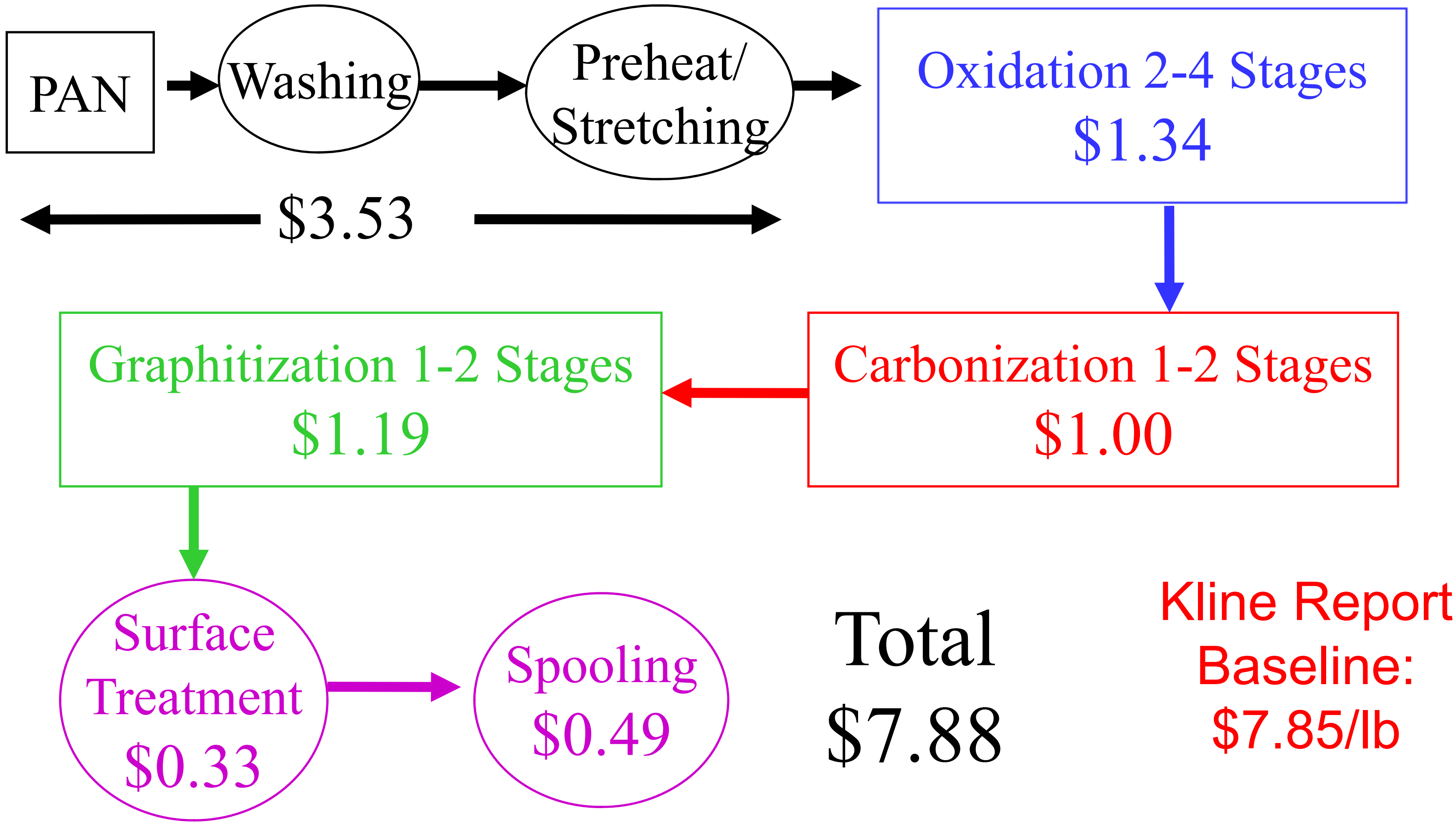
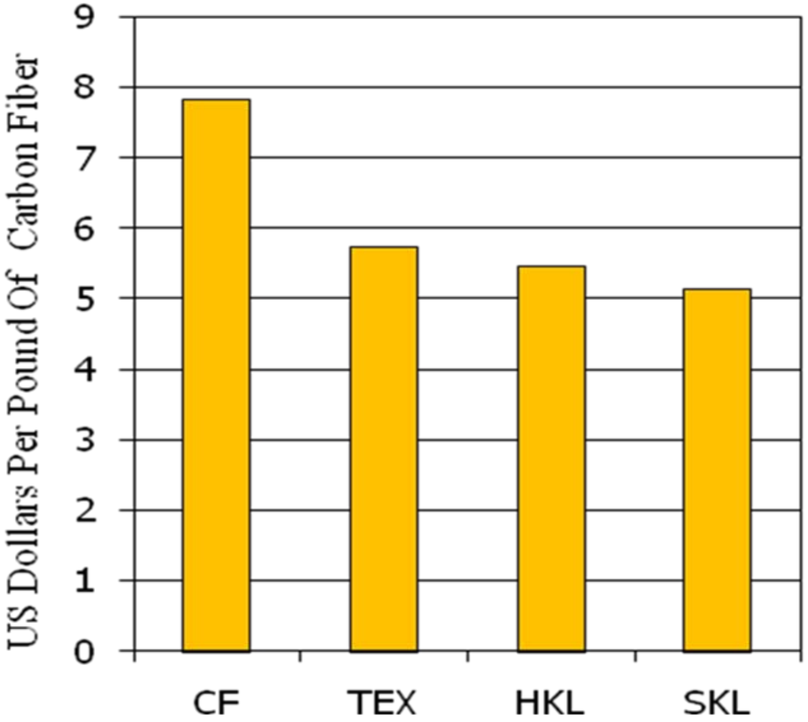
Carbon Fiber - Production Costs

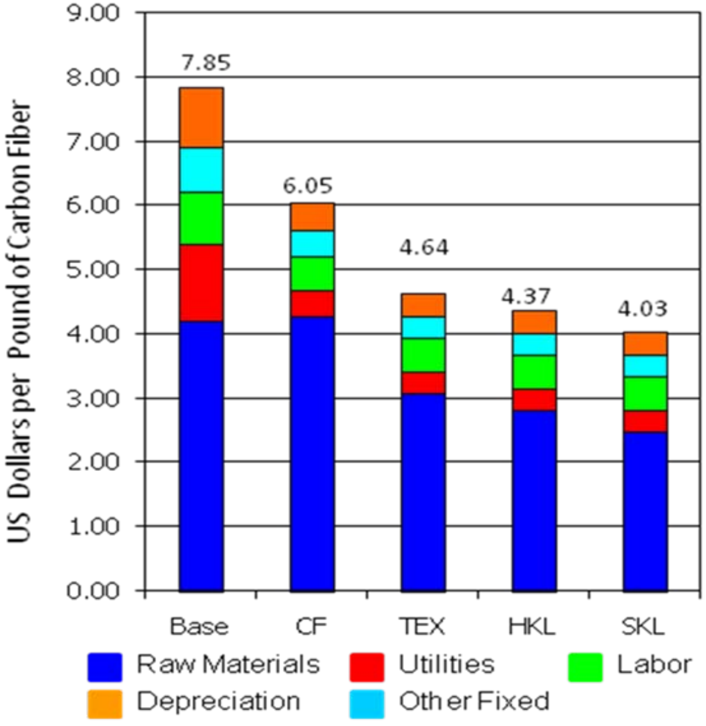


Current Research

Alternative Precursors



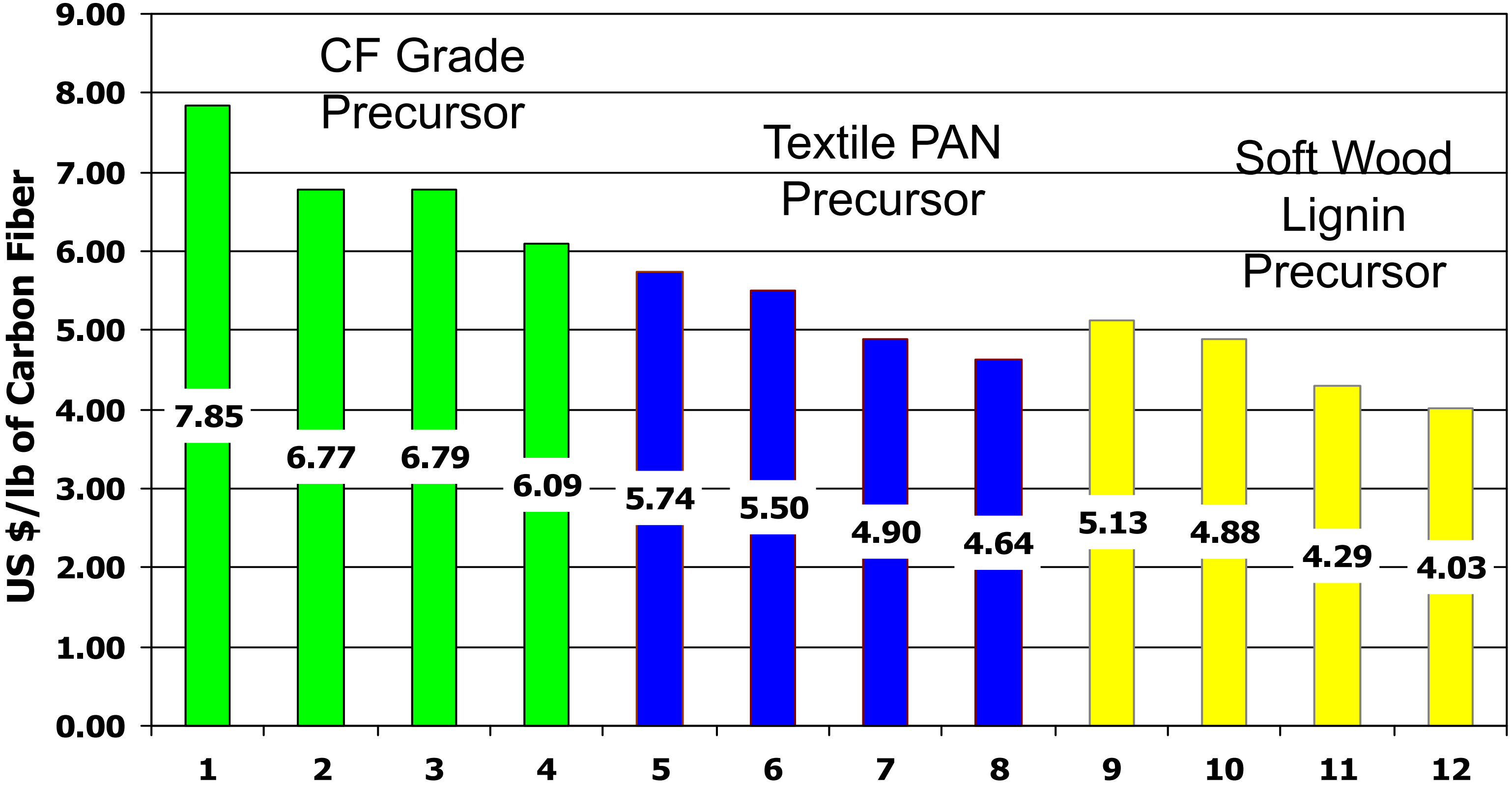
Alternative Production Methods



LOW COST CARBON FIBER MODELS
Manufacturing Costs High-Volume Case

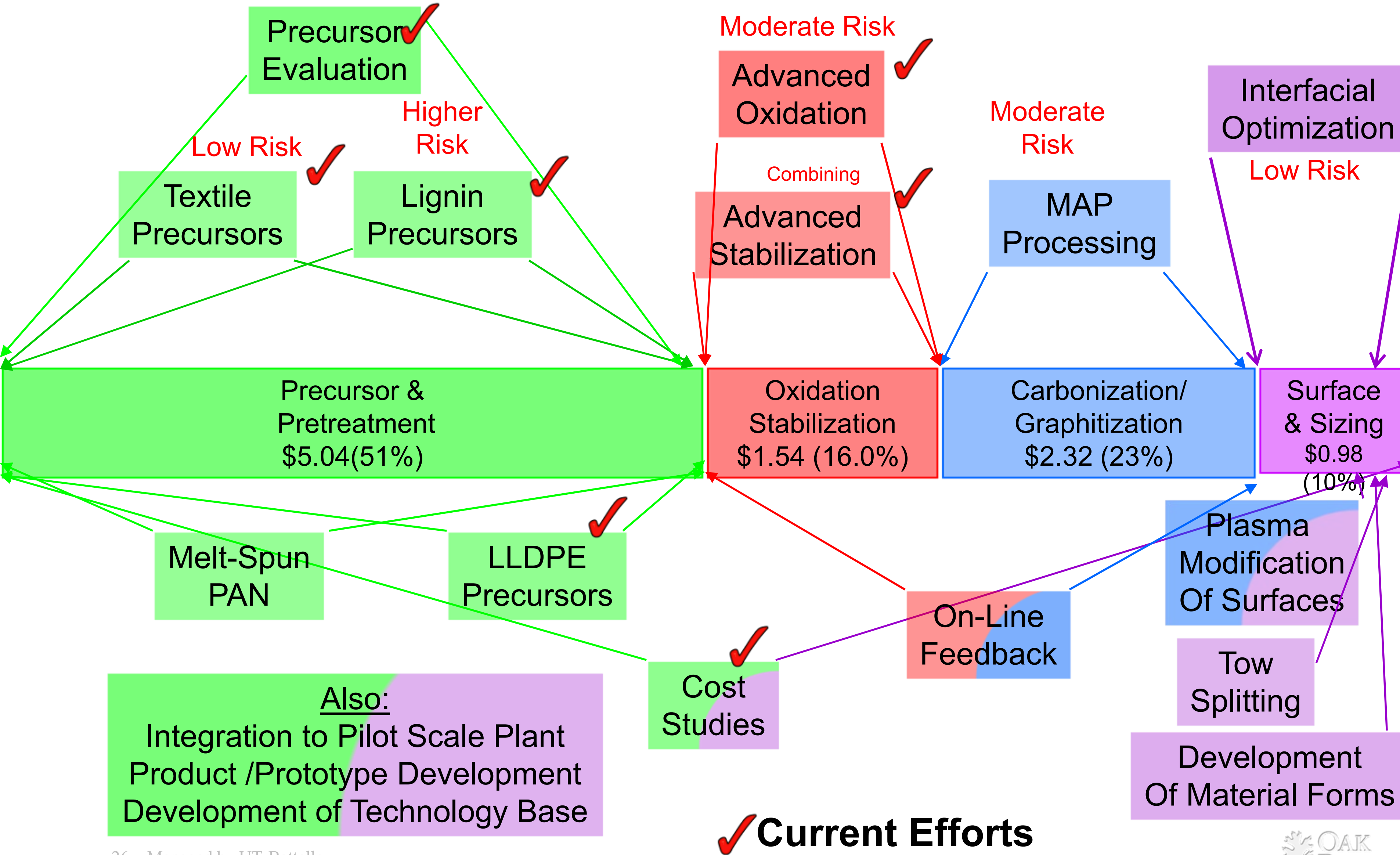
Combined Cost Projections

Key
1- Baseline
2- Plasma oxidation (PO)
3- MAP carbon-ization (MAP)
4- PO & MAP
5- Textile PAN (TP)
6- TP & PO
7- TP & MAP
8- TP, PO, & MAP
9- Softwood Kraft Lignin (SKL)
10- SKL & PO
11- SKL & MAP
12- SKL, PO, & MAP



* From Kline & Company

Critical Path for the Automotive Program



Carbon Fiber - Research Efforts

Precursor Production

Baseline

PAN
$3.53

Options

Textile Grade PAN
$2.33

or

Hardwood Kraft Lignin
$2.07

or

Softwood Kraft Lignin
$1.73

Processing Precursor to Fiber

Conventional Thermal

Oxidation	Carbonization	Graphitization	ST
$1.34	$1.00	$1.19	$0.82

$0.60

AS PO

Advanced Stabilization
and
Plasma Oxidation

and/or

Microwave-Assisted
Plasma

MAP
$1.34

1. Polyolefin Precursors
2. Surface Activation, and Sizing

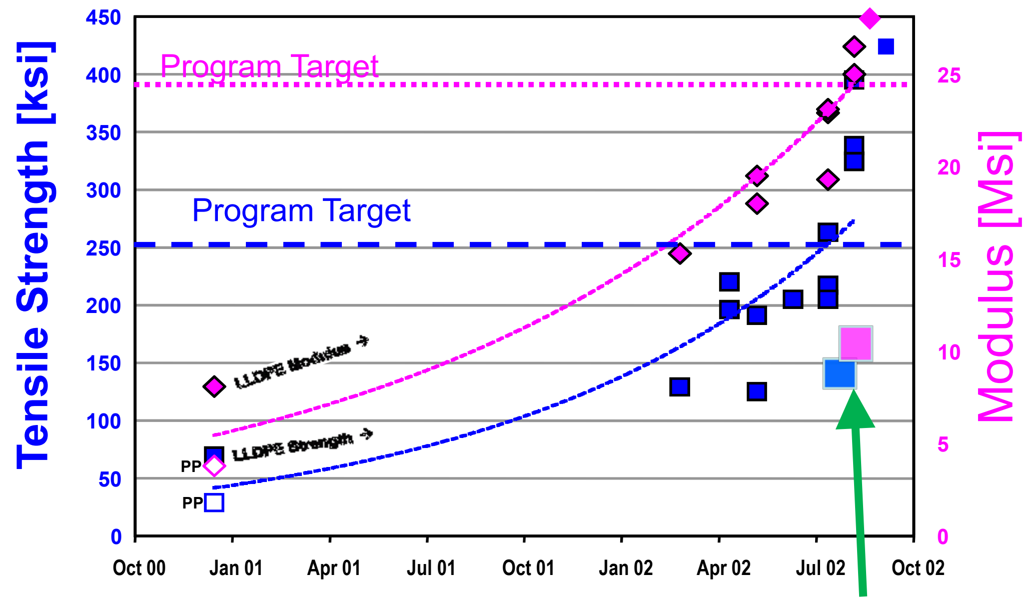
Polyolefin Precursors

Polyolefins have been investigated by others as carbon fiber precursors.
Japanese in the 70's, Hexcel in early 2000's

Obtained Properties:
30 MSI; 390 KSI

But

Required a 10 – 24 h elevated temperature sulfonation in Sulfuric Acid Making it an uneconomical process



Using 1 hour sulfonation process in proof of principal project

Dr. Naskar has developed a 1 hour sulfonation process that is:

1. not the method used in the past;
2. uses a chemical/process used in an industry that sells 1000's of tons of material for less than $0.20 a pound.
3. the process leaves the precursor ready to carbonize and by passes the entire oxidative stabilization process (80 – 120 minutes)

Polyolefin Precursors

Precursor type	Yield (%)		$/lb (as-spun)	Melt-spinnable	Best achieved properties		Problem
	Theoretical	Practical			Strength (KSI)	Modulus (MSI)	
Conventional PAN	68	45-50	>4	No	500-900	30-65	High cost
Textile PAN*	~ 68	45-50	1-3	No	300-400+	30	High variation in properties
Lignin*	62-67	40-50	0.40 - 0.70	Yes	160	15	Fiber handling, low strength & slow stabilization step
Polyolefin**	86	65-80	0.35 - 0.5	Yes	380	30	Slow stabilization (sulfonation) step

↑ ↑ High Yield

↑ Inexpensive

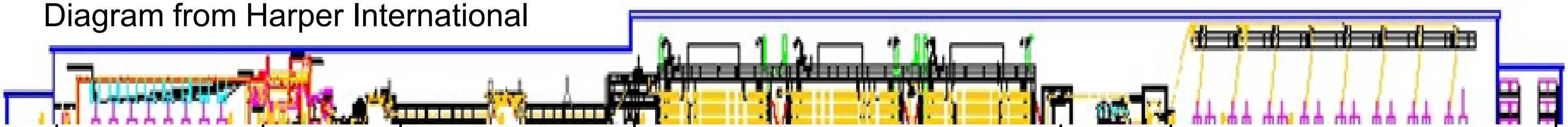
↑ ↑ Properties Proven At Small Scale

↑ Obstacle Addressed

Eliminating Oxidative Stabilization Reduced conversion time to 15 – 30 minutes

Polyolefin Precursors

Diagram from Harper International



	Spooling & Packaging	Surface Treatment	Carbonization/ Graphitization	Stabilization & Oxidation		Precursors
	$0.61	$0.37	$2.32	$1.54	Baseline Today - $9.88	$5.04
	$0.41	$0.33	$1.48	$0.99	High Volume - $7.85	$4.64
High	$0.41	$0.33	$1.48	$0.20	$3.32	$0.90
Low	$0.41	$0.33	$1.25	$0.10	$2.74	$0.65

Less Effluents
Faster throughput
Less Incineration

	Large tow CF Precursor	Small tow (<24k) CF Precursor	Textile Precursor	Polyolefin Precursor
As-Spun Fiber ($/lb)	$ 3-5	$ 4-6	$ 2-3	$ 0.50 - $ 0.60
Carbon Yield	~45%	~50%	~50%	65 - 80%
Precursor Cost ($ /lb CF)	$ 6.5-11	$ 8-12	$ 4-6	$ 0.65 - $ 0.90
Stabilization	85 - 120 min	75 -100 min	75 - 100 min	60 min **
Carbonization	Same	Same	Same	Same

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QUESTIONS?