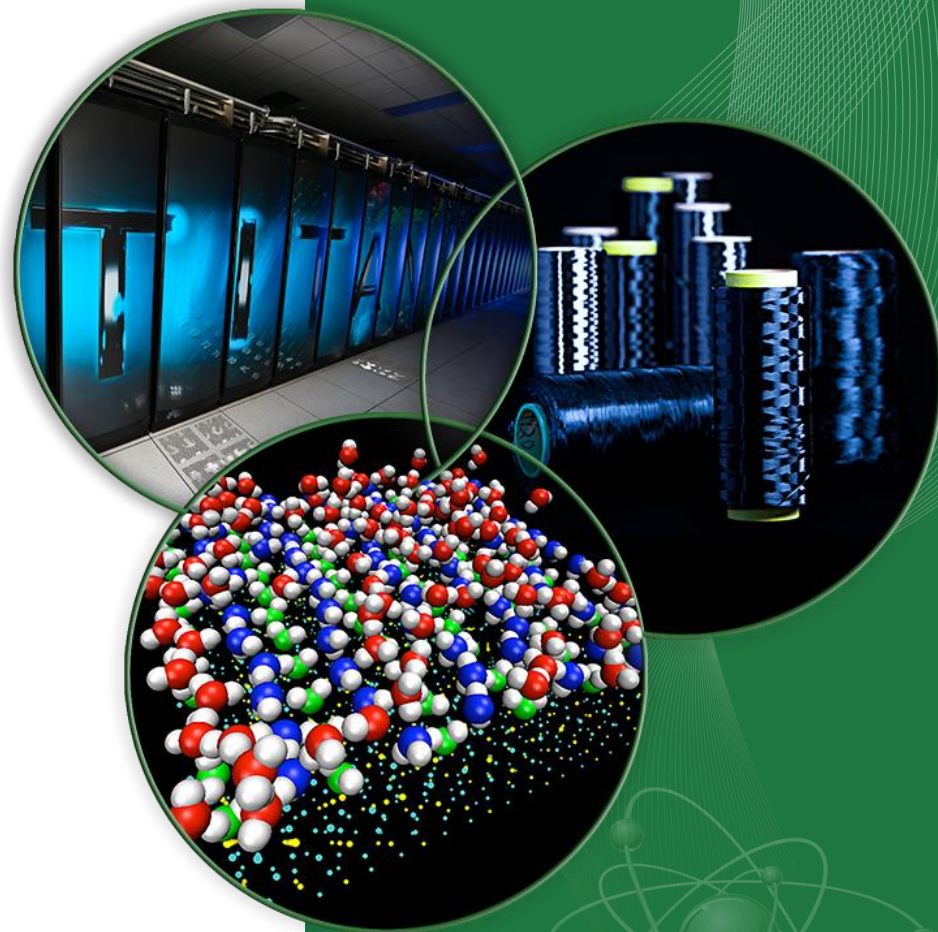


# Improving Carbon Fiber Production: High-Quality Carbon Fiber at a Significantly Reduced Price

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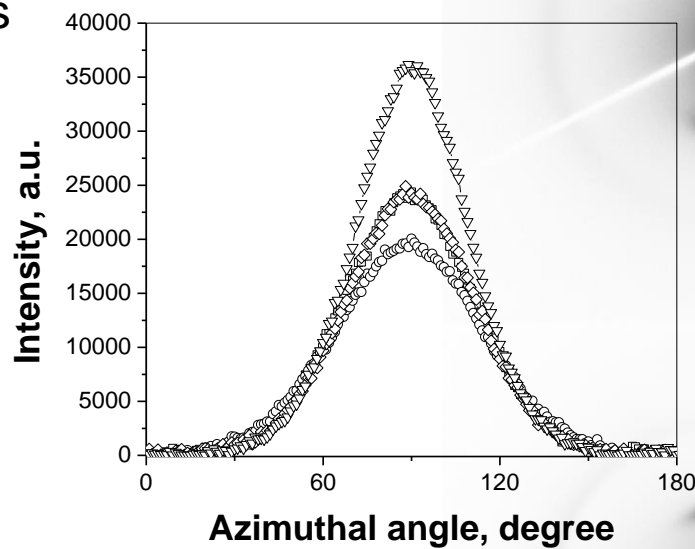
October 3, 2017



# Opportunities and remaining challenges with textile-PAN as carbon fiber precursors

ORNL and its commercializing partners manufacture carbon fibers from textile precursors.

- Converted fibers exhibit broad distribution of graphite crystal orientation (misorientation angle  $\sim 42^\circ$ ) even after very high temperature conversion.
  - Depends on the draw ratio of original precursor fiber
- After such high temperature treatment, the fibers exhibit acceptable performance



Commercial CF

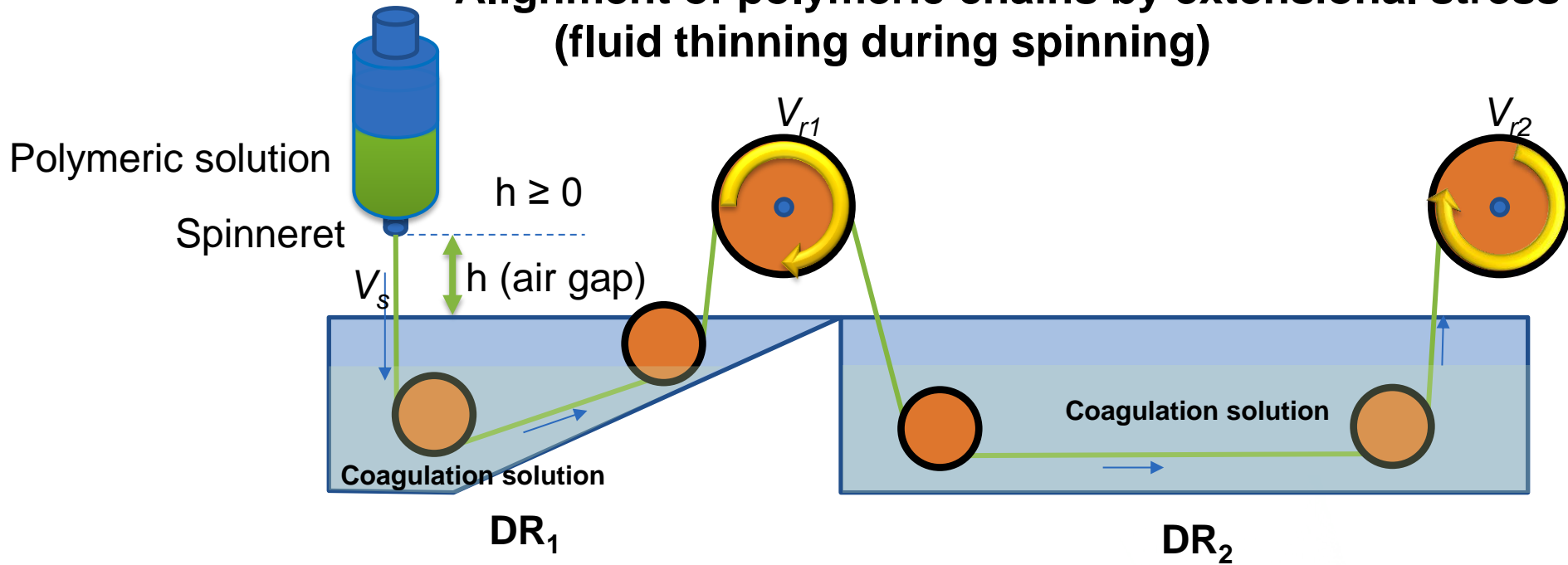
Textile-PAN-based CF

**Is there any feasible path to induce higher degree of orientation during fiber manufacturing?**

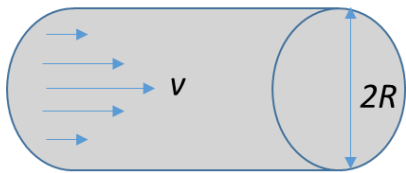
**Can we upgrade the precursor without significant capital investment?**

# Precursor spinning operations

Alignment of polymeric chains by extensional stress  
(fluid thinning during spinning)



Extrusion and Capillary



$$\dot{\gamma} = \frac{4Q}{\pi R^3}$$

$$Q = v \times \pi R^2$$

$$\dot{\gamma} = \frac{4v_s}{R}$$

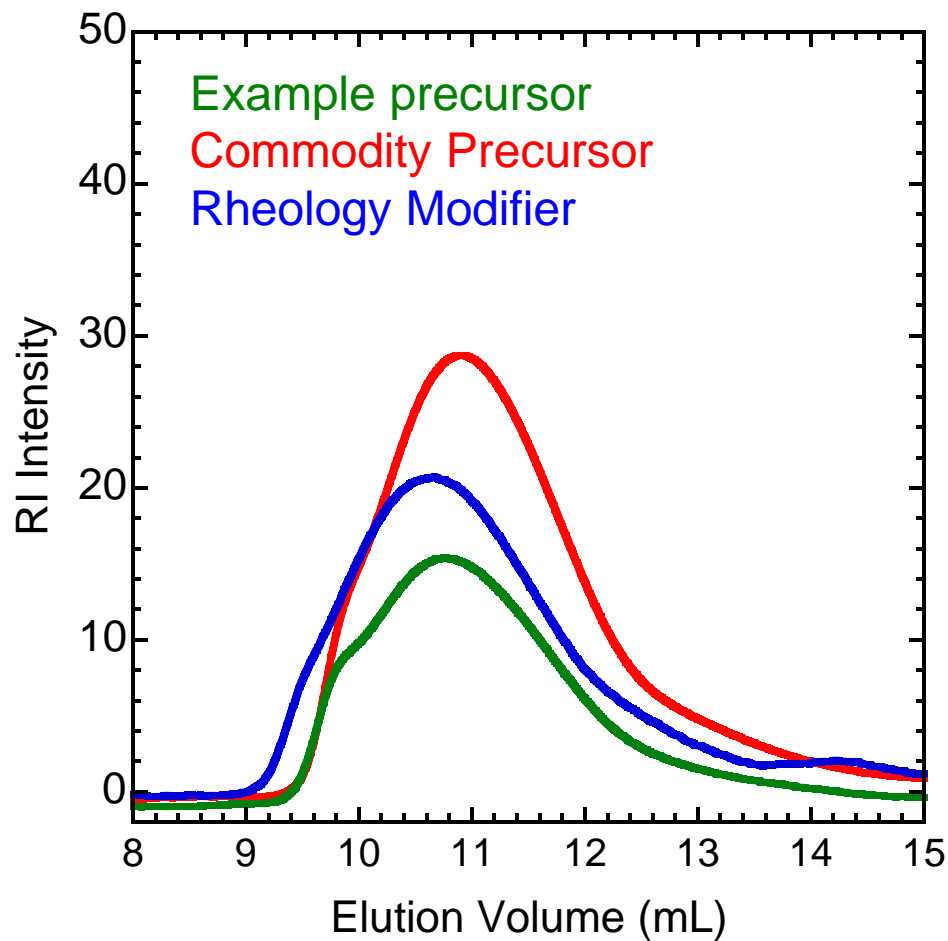
$$DR_1 \text{ (Drawing ratio)} = \frac{V_{r1}}{V_s}$$

$$DR_2 \text{ (Drawing ratio)} = \frac{V_{r2}}{V_{r1}}$$

# **We have made our first success in obtaining a composition and process that enhances PAN spinnability and extensibility**

- A PAN solution with a modifier that increases precursor extensibility.
  - Usually, processing at high shear rate while spinning and stretching the fiber causes reduction in viscosity but the stiffness in PAN jet remains poor and it limits us to accomplish significant degree of orientation (without breakage of filaments).

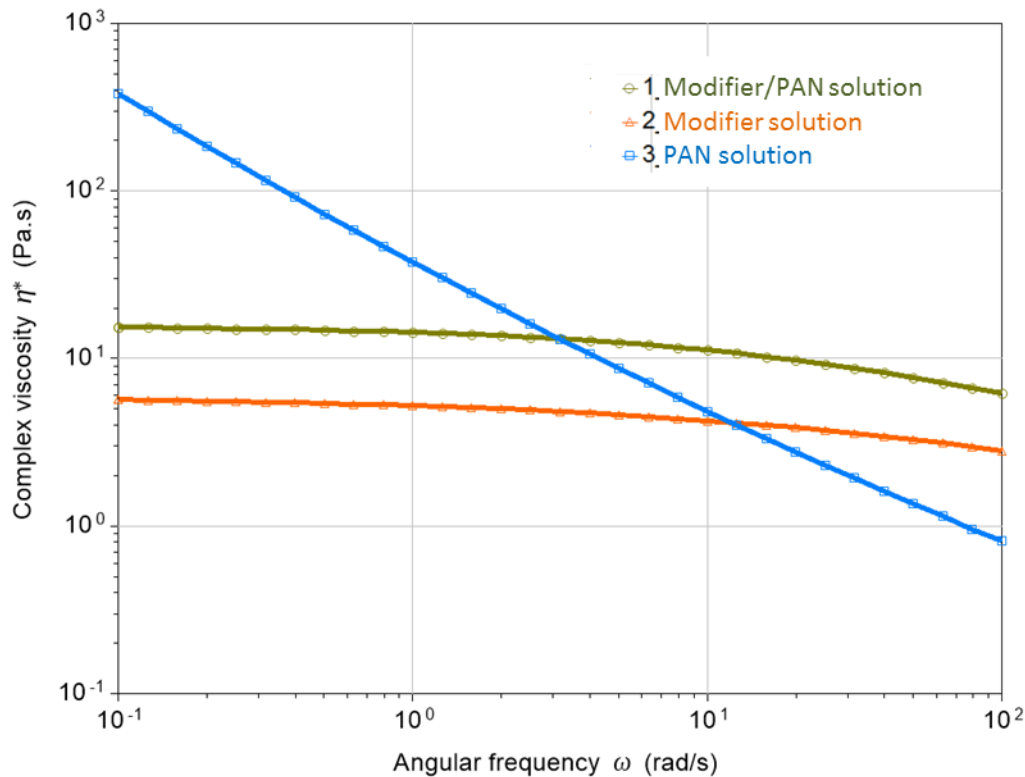
# Modifier: A carbon precursor



Sample	Mn (kDa)	PDI
Example precursor	136	1.9
Commodity Precursor	120	1.8
Rheology Modifier	153	2.0

# PAN precursor in presence of modifier exhibit lower degree of shear thinning behavior

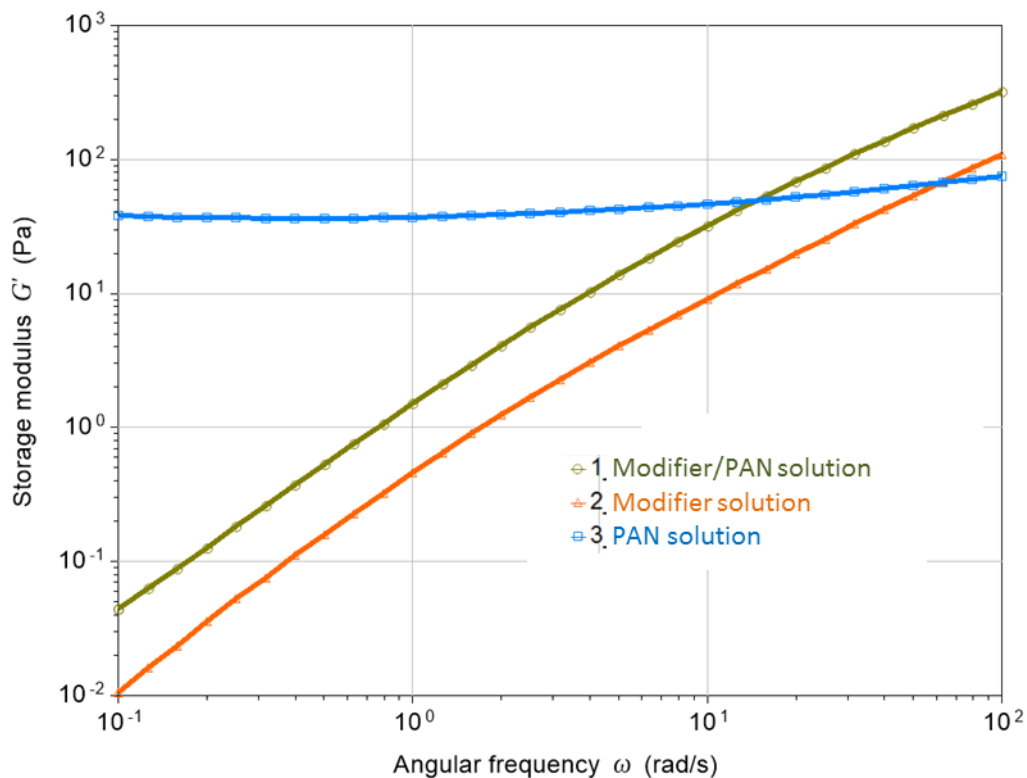
Better flow property of modifier in solution



- Strong gel like behavior of PAN precursor solution at lower frequencies.
- Incorporation of modifier changes the degree of interaction among PAN molecules; it also reinforces the PAN solution at higher frequencies.

# PAN precursor in solution exhibits extensive entanglement and dipolar interaction to cause gel like behavior

Large frequency dependence modulus of PAN solution  
(reference temperature: 25 °C)



Incorporation of viscosity modifier reduces the modulus of the solution at lower frequencies but increases the same at higher frequencies due to rigidity of the structures.

# ORNL has established a solution spinning laboratory for generation of renewable PAN precursors and other high performance fibers



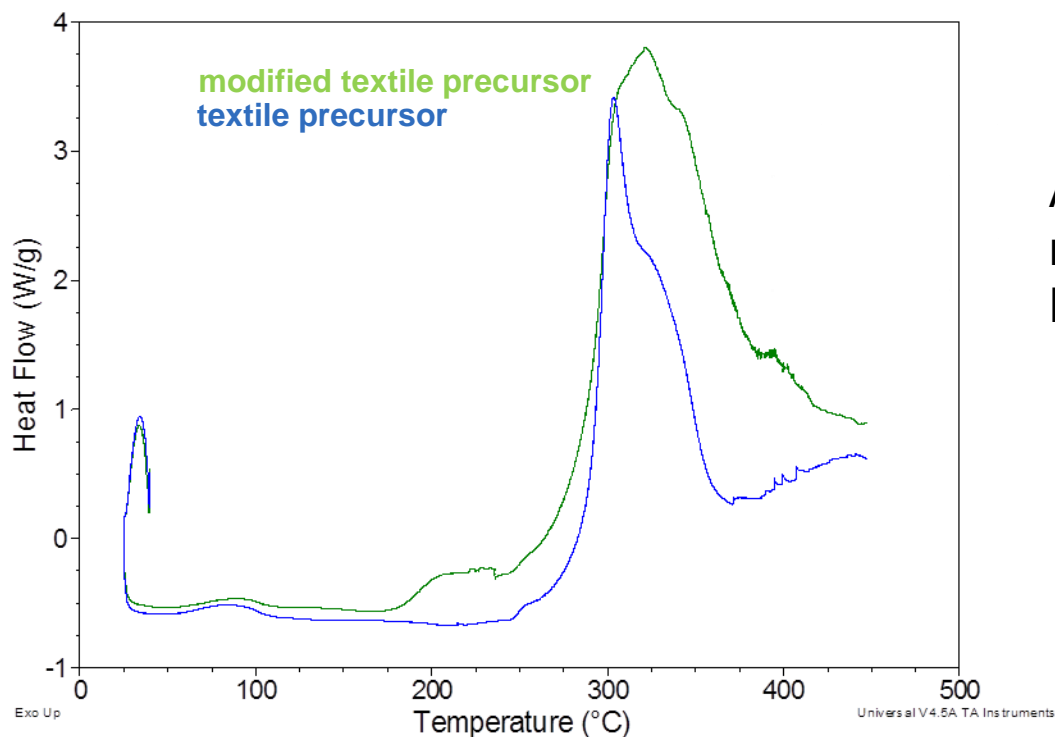
First batches of precursor fibers are made. Modified precursors are spun for future conversion work.



# Oriented PAN precursors exhibit reduced oxidation (stabilization) kinetics

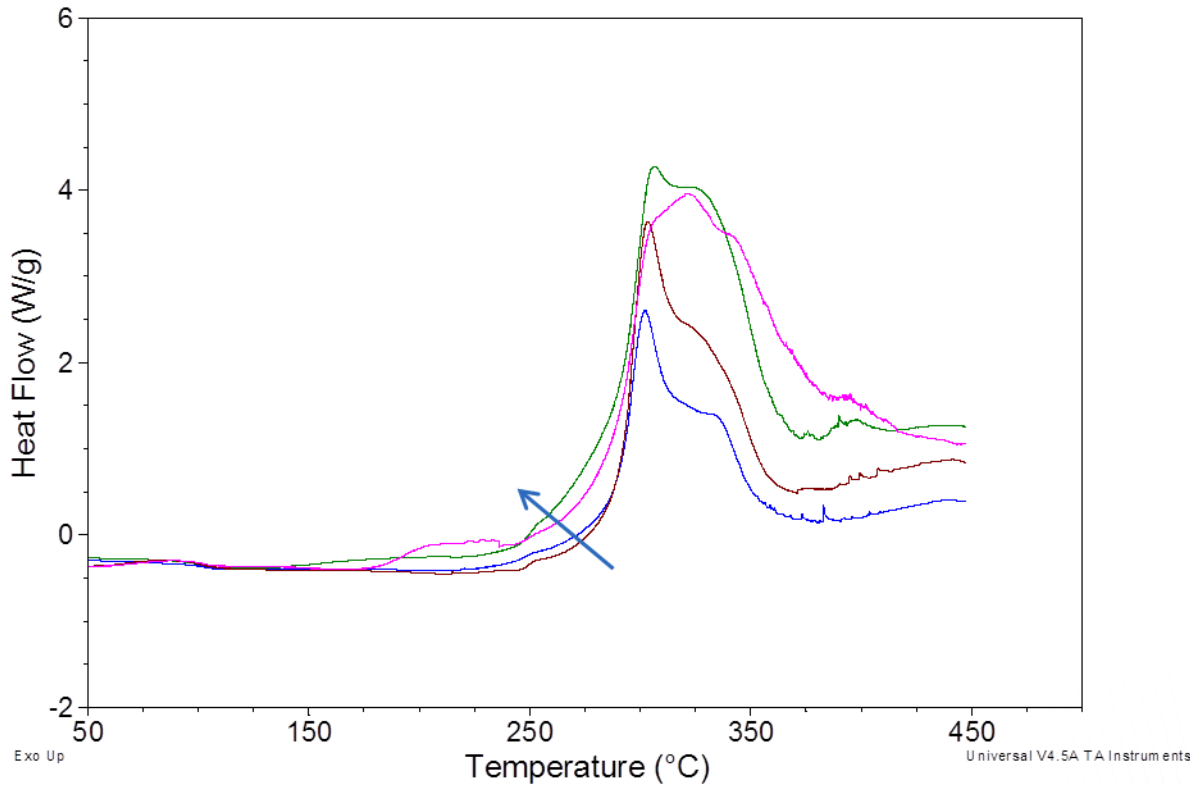
- Conversion kinetic parameters depend on precursor orientation factors.
  - Higher degree of orientation is desired for superior performance of carbon fibers.
  - However, slower kinetic parameter is detrimental for manufacturing throughput.
- We devised an approach that addresses slower kinetic parameters of textile-PAN precursors' conversion to infusible fibers.

# Precursor could be modified to induce a slight degree of stabilization at early stage of oxidation with our rapid release of exothermic heat



Approach involves inducing reactivity to a segment in the PAN precursor

# In line modification of textile precursors offer a feasible path to enhance rate of conversion



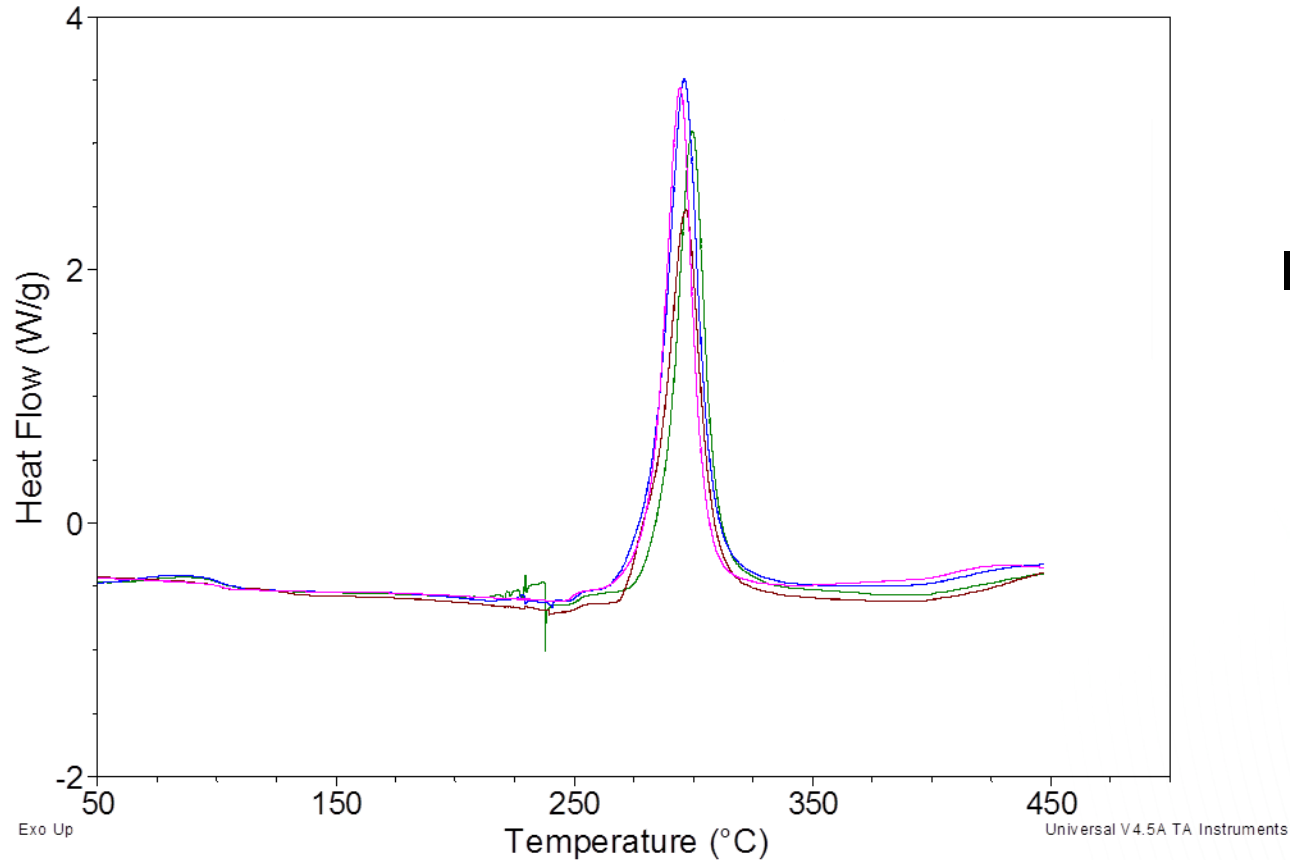
Oxidative environment

# A method that is applicable for rapid conversion for other nonconventional PAN precursors



Carbon fibers  
(properties meet DOE-EERE  
Vehicle Technologies Program  
Goal)

# In line modification of textile precursors that is effective for oxidative reaction only



Inert environment

# Summary

- Successfully developed an approach to induce better precursors from traditional textiles using slightly modified approach during fiber spinning operation
- Higher degree of orientation causes retarded conversion kinetics with textile precursors
- An approach with in-line modification of precursor addresses that challenge
- Method is applicable for all PAN precursors

# Acknowledgements

- Research is sponsored by ORNL's Technology Innovation Program
- Author acknowledges assistance from colleagues at Carbon and Composites group and Carbon Fiber Technologies Facility.