Progressive Production Techniques for Industrial Carbon Fiber

JEC Europe, March 2017

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Harper International

Introduction

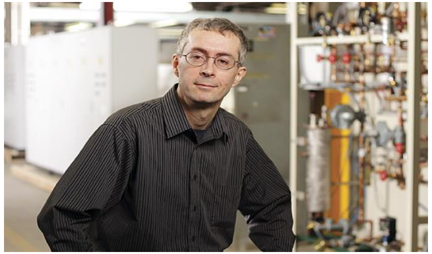
Challenge: Continuously progress state-of-the-art Carbon Fiber processing to support market advancement

Agenda

- About Harper
1. Computational Techniques
2. Microwave Processing
3. On-Line Measurements
- Summary

About Harper

- Headquartered near Buffalo, NY
- An employee-owned company
- Onsite Technology Center
- Multi-disciplined engineering talent
  - Chemical
  - Ceramic
  - Mechanical
  - Electrical
  - Industrial
  - Process & Integration



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Carbon Fiber Carbonization Process – Scales of Operation

Scale	Size Range (Tow-Band Width)	Capacity
Commercial Production Line	1000 – 4200 mm	500 - 4000 ton/year
Pilot Line	300 -1000 mm	20 - 100 ton/year
Microline	≤100 mm	Less than 10 ton/year
Scientific Line	Fractional tows (<1k or less than 1,000 filaments)	Less than 1 ton/year

Courtesy of Oak Ridge National Laboratory

Courtesy of Georgia Institute of Technology

*Georgia tech has produced the highest tensile strength PAN based carbon fiber ever reported, and highest combination of strength and modulus ever reported, on their Harper Scientific Line.

http://www.news.gatech.edu/2015/07/22/innovative-method-improves-strength-and-modulus-carbon-fibers

Advancing the State of the Art

Five Important Technology Developments in Carbon Fiber Production

1. Sealed, Precision Flow Oxidation Oven Systems
2. Closed Pipe Treatment of Exhausts , Energy Recovery, Integration
3. Plants Configured for Natural Gas Energy Source
4. Next Gen Carbonization Systems
5. Utilization % Increases

↑ Production, ↓ Cost

Harper 1800 T/yr carbonization HT system

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1. Computational Techniques

High Performance Computing for Manufacturing (HPC4Mfg)

This research was supported by the High-Performance Computing for Manufacturing Project Program (HPC4Mfg), managed by the U.S. Department of Energy Advanced Manufacturing Office within the Energy Efficiency and Renewable Energy Office. It was performed using resources at Oak Ridge National Laboratory, which is supported by the Office of Science of the U.S. Department of Energy under Contract No. DE-AC0500OR22725.

HPC4Mfg – Why?

Scaling Up Carbon Fiber Production

- Increasing furnace capacity, efficiency
  - Increasing impact of off-gas on fiber properties.

↓

- Developing kinetic model implementing full scale 3D CFD

↓

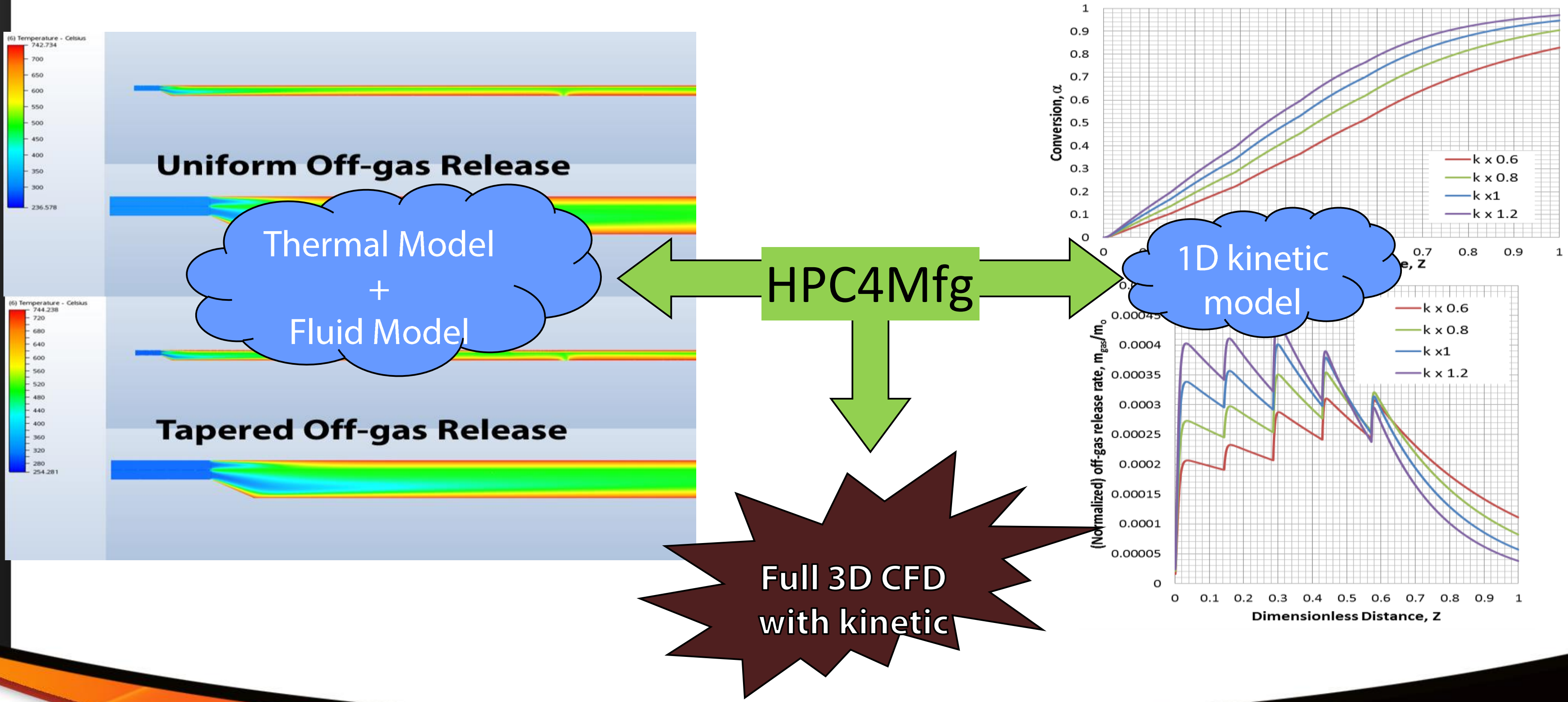
- Implementation of model for uniformity of process conditions

Kinetic Model

Project Approach

- Develop process kinetic and gas flow models
- Develop coupled kinetic CFD models for carbonization
- Use developed models to scale-up process and equipment

HPC4Mfg

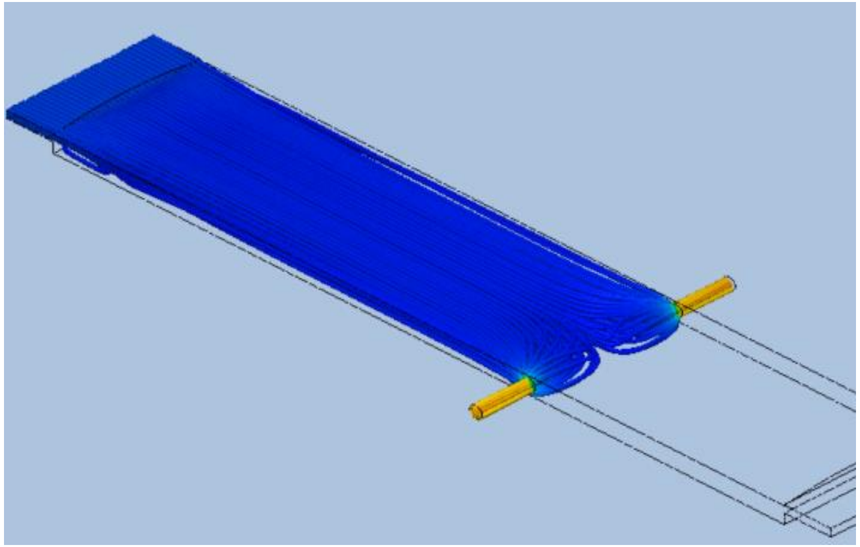

Uniform Off-gas Release
Tapered Off-gas Release
Thermal Model
+
Fluid Model
HPC4Mfg
1D kinetic model
Full 3D CFD with kinetic
Conversion, α
k x 0.6
k x 0.8
k x1
k x 1.2
(Normalized) off-gas release rate, m_{gas}/m_o
Dimensionless Distance, Z

Benefits

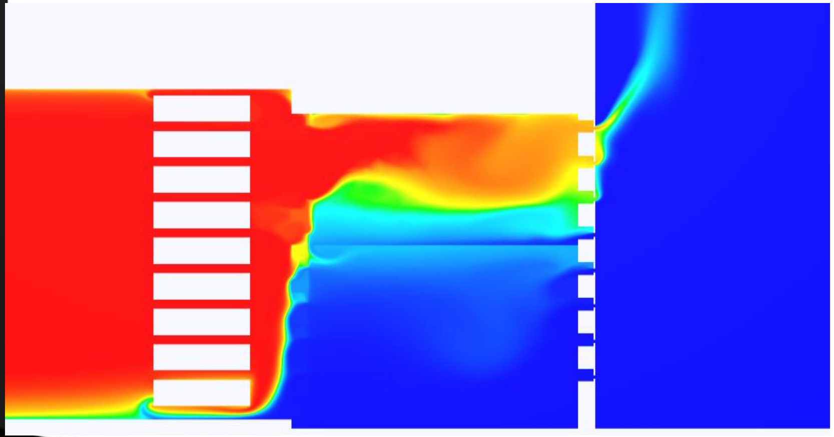
- Improved understanding of the physical and chemical processes
- Increased ability to control the process conditions in the furnace
- Thoughtful evaluation of various scale-up approaches for
  - High capacity
  - Improved process uniformity and conversion

Off-Gas Treatment Considerations

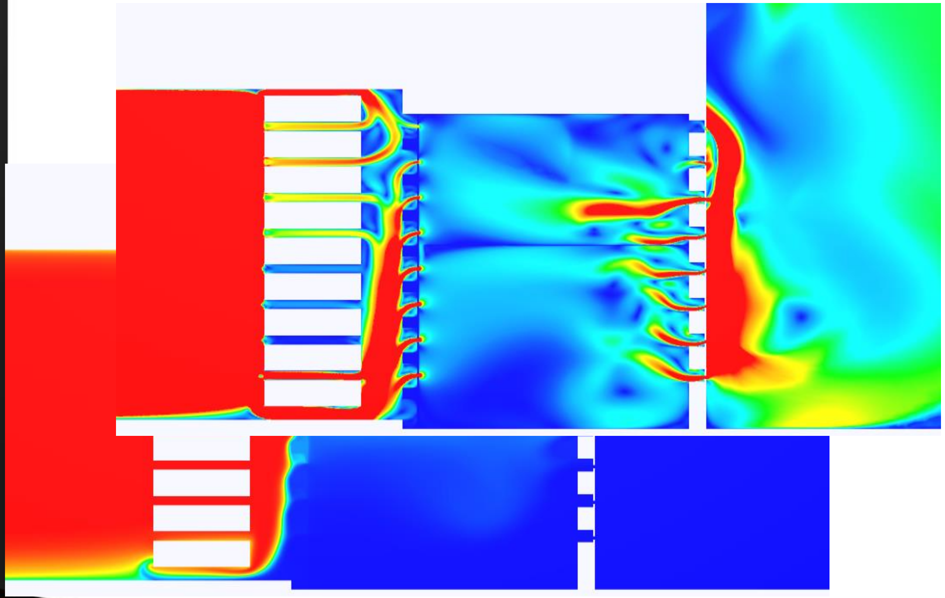
- 2500 TONS Carbon Fiber → 2500 TONS of Off-Gas
  - Venting consistency is important!



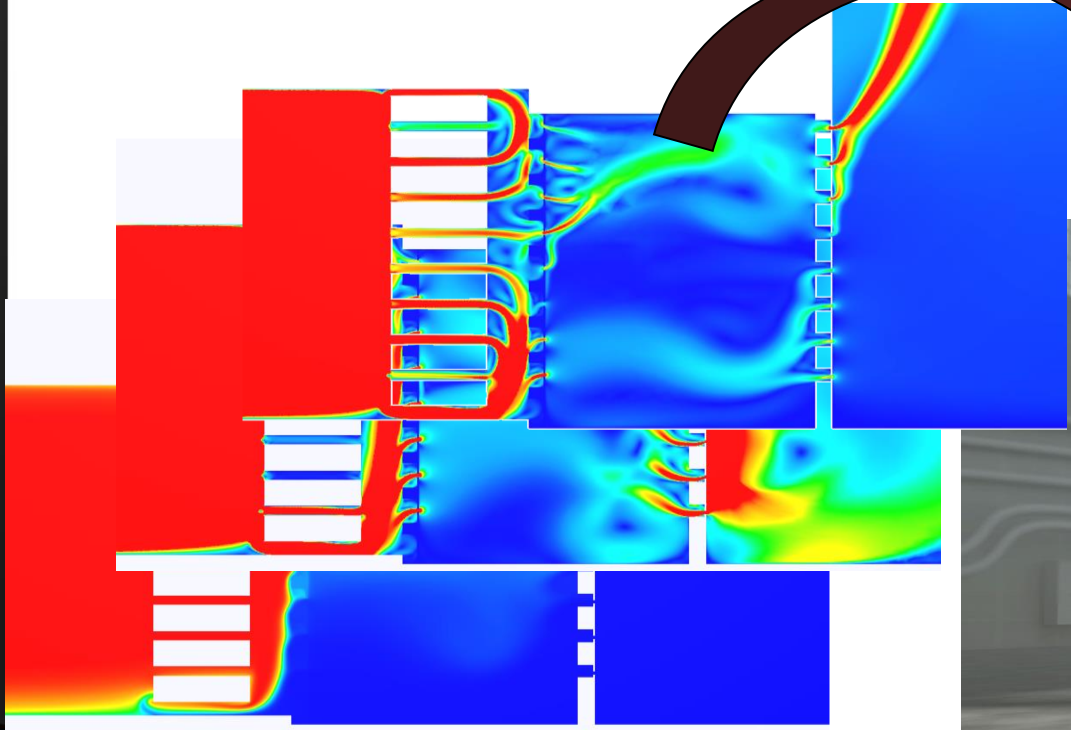
Iterative Design



Iterative Design



Iterative Design



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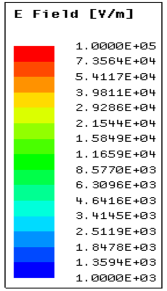
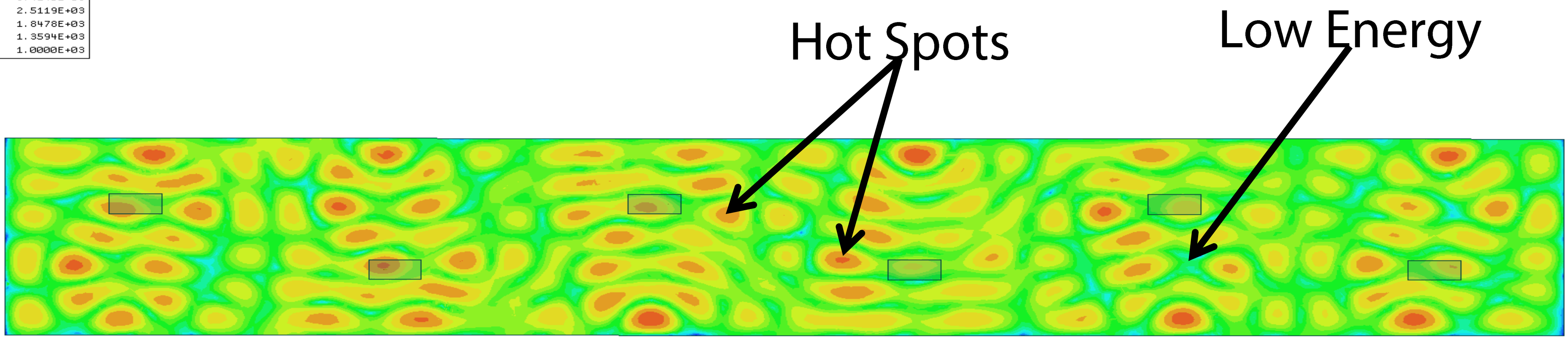
Microwave Heating Potential for Carbon Fiber

- Direct heating of carbon fiber by microwaves
  - Change in heating modes from convection / radiation to direct heating
  - Changes the thermal profile – Carbon fiber is at highest temperature
  - Changes in heating rates / energy efficiencies
- Challenge: Uniform energy distribution

Field Distribution in a Tunnel Applicator
Plan view along the length

- Energy field - Linear wave guide
- Result – Non uniform processing



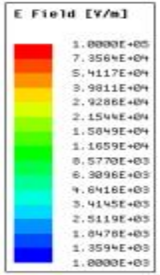


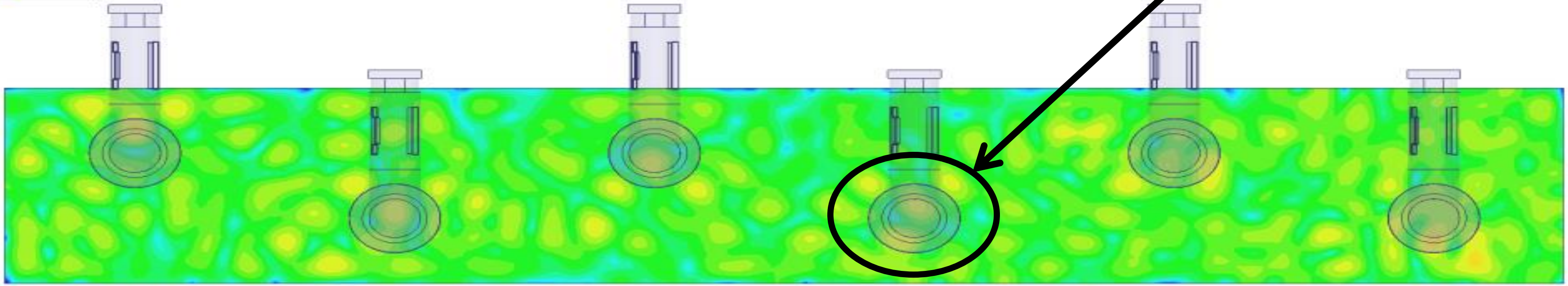
Courtesy of Ferrite Microwave Technologies

Field Distribution in a Tunnel Applicator
Plan view along the length

- Energy Field - Circular polarizing wave guide
- Result - more uniform processing
  - Even distribution of energy → Even heating

Patented field distribution system





Courtesy of Ferrite Microwave Technologies

Potential Benefits & Challenges of Microwave Heating

✓ Potential Benefits:

- Improved energy utilization
- Smaller equipment footprint
- Reduced utilities requirement
- Possible to combine multiple heat treatment stages
- Reduce oven residence times

❖ Potential Challenges:

- Design of vents for removing volatiles

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3. On-Line Measurements

- Continuous measurement of CF properties
- Feed back to equipment to modify properties

iacmi

The Institute for Advanced Composites Manufacturing Innovation

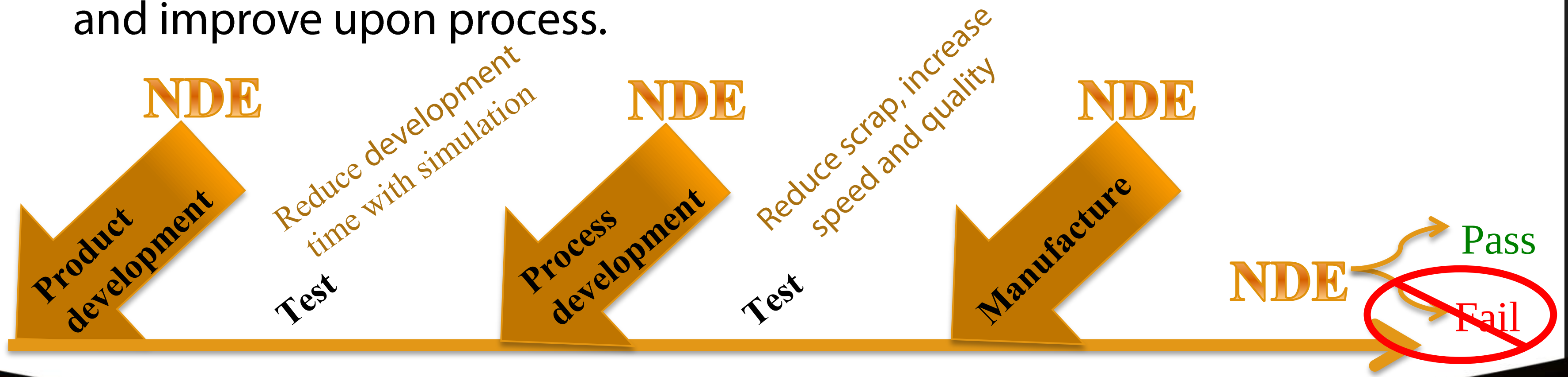
Selective Feedback for Intelligent Process Control

- Selecting measurable properties that influence final quality
- Selecting controls that can be manipulated to influence quality

- This is being done now – manual inspection – manual adjustment
  - Fuzzy fiber coming out → Adjust system parameters based on experience
  - Linear density wrong → Adjust system parameters based on experience

Expanding the Role of Non Destructive Evaluation

- Current – NDE at end for pass/fail of product
- What if:
  - Diagnose the material state in-line to support process decisions in order to reach speed & quality targets.
  - Characterize the material state to support simulation in order to innovate and improve upon process.

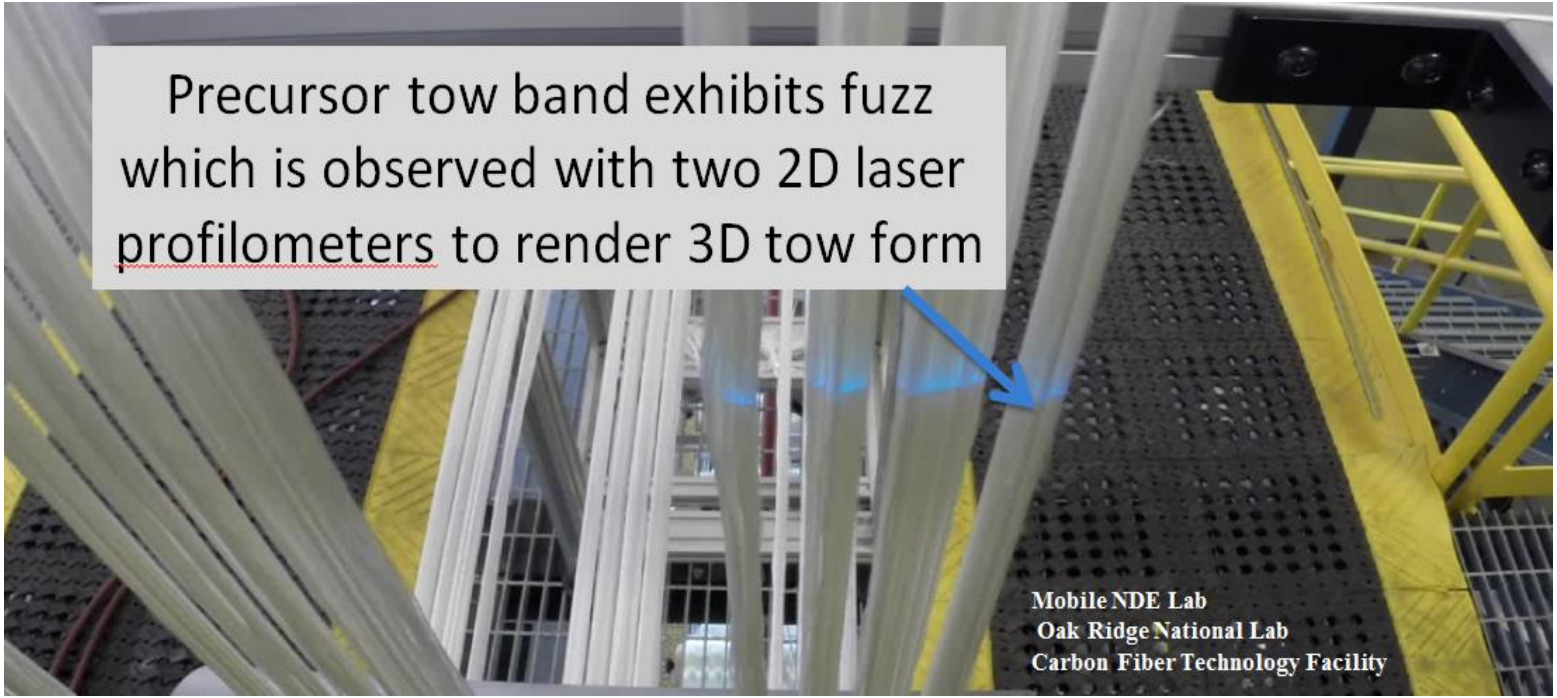


Online Measurements

1. Tow Characterization (fuzz, width)
2. Linear Density
3. State of Oxidation

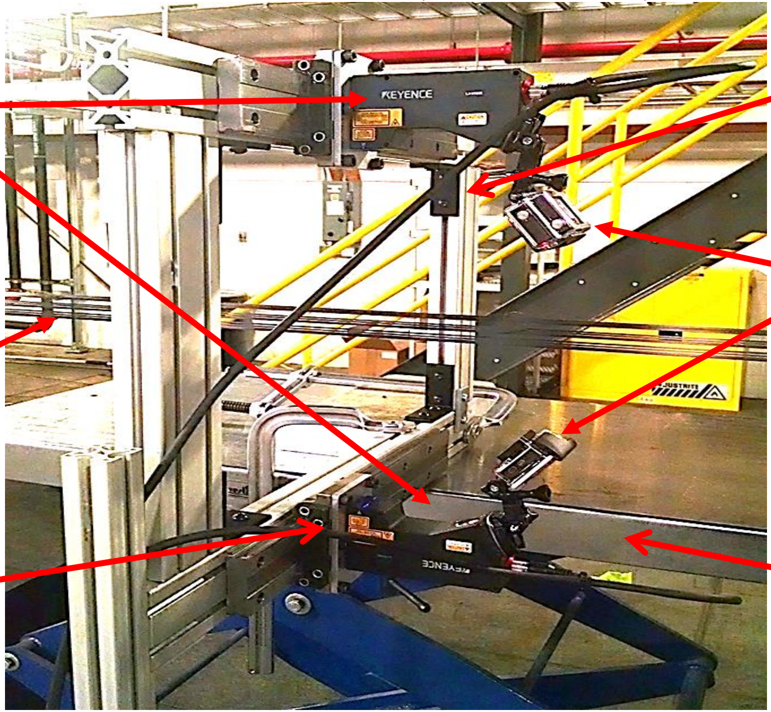
Courtesy: Cliff Eberle, Oak Ridge National Laboratory

1. Fuzz detection



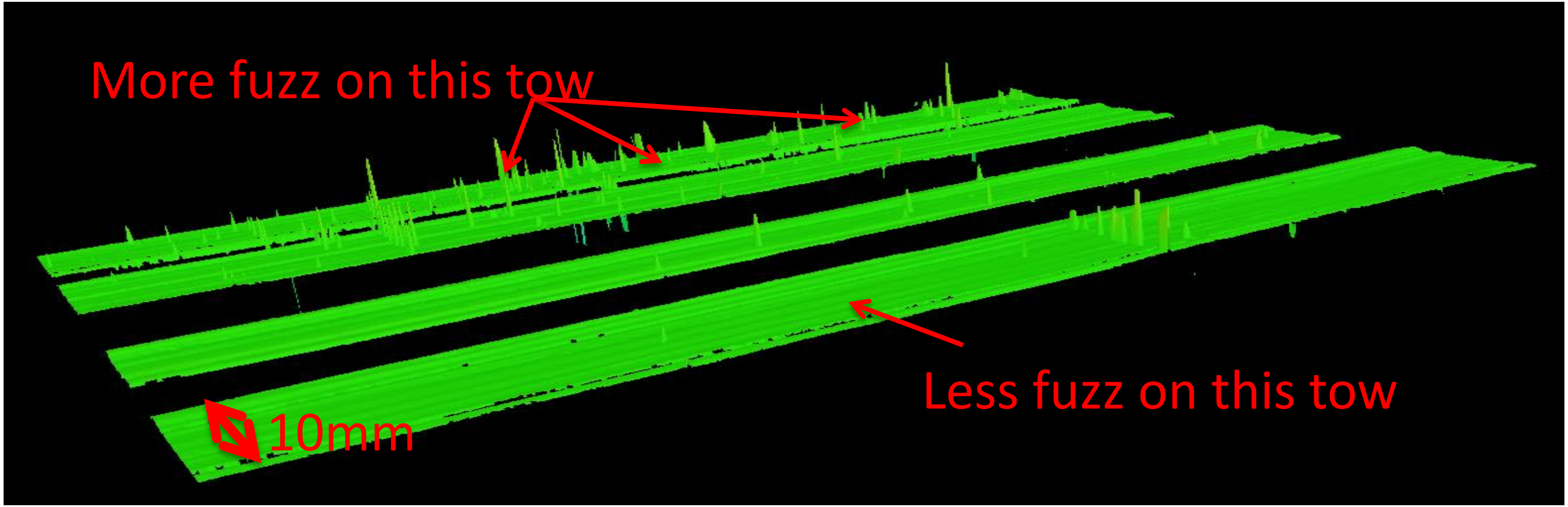
12 tows total of 24k precursor (4 tows shown)

Setup of Real-Time, Non-Contact, Profilometry NDE System at CFTF



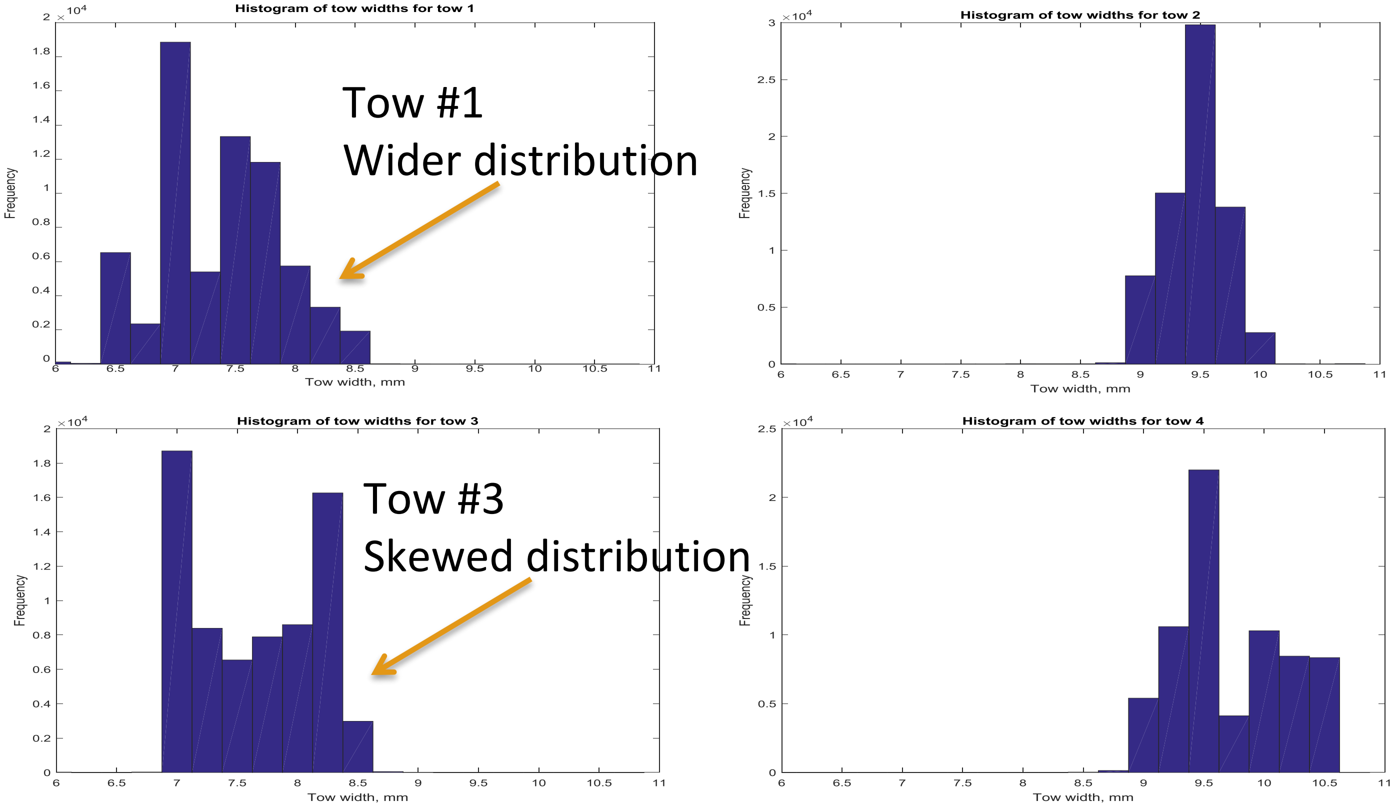
2D laser specs: Z Range: +/- 48 mm, X Beam Width: 62 mm, Sample Rate (max.) : 62500 Samples/sec. , Resolution: X: 100 µm, Z: 1 µm

Profile along Tow for In-Line Fuzz Detection of Precursor



Combining the 2D data through time after correlating top and bottom lasers, we can create a 3D profile.

Tow Width Distributions

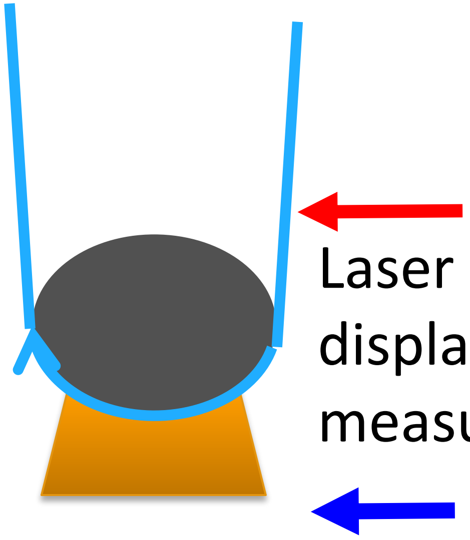


2. Measuring Linear Density

Oscillations of tow are a "fingerprint" with which to diagnose state of fiber

$$m\ddot{x} + kx = f(t)$$

Presentation contains video.
Contact Harper at
info@harperintl.com
for more info.

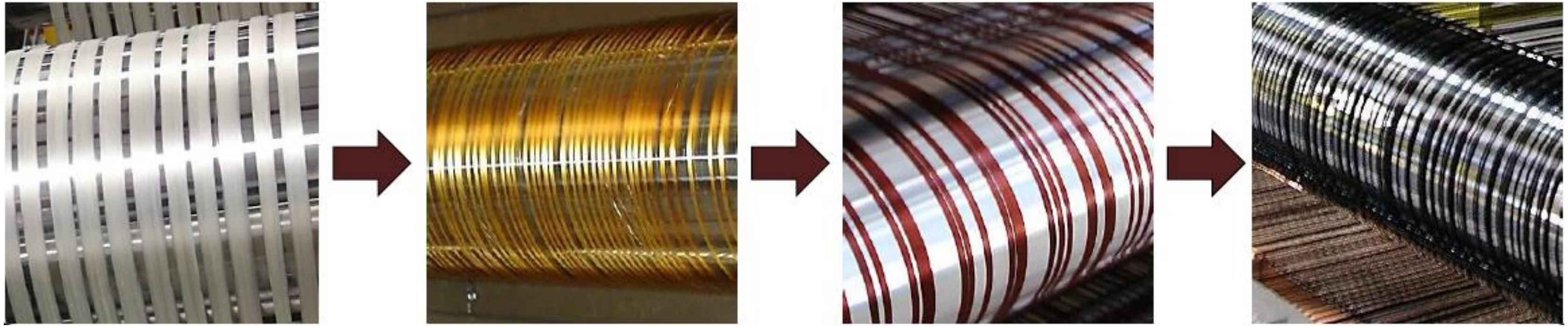


Laser displacement measurement

Excite the machinery or tow fibers to make a coherent measurement

3. Oxidation Measurement

- Oxidation process: longest and most expensive conversion step
- Detection of oxidation state → streamline the manufacturing
- Relationships fiber's color and Oxidation State



Diagnosing Oxidation of Fiber

Red Intensity → Oxidation State

Impact of Non Destructive Evaluation

- Feedback on fiber properties prior to final inspection
- More control and consistency of fiber quality
- Better understanding of process conditions and fiber properties

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Summary

Addressing the challenge to continuously advance state-of-the-art of carbon fiber processing with Progressive Techniques

- High Performance Computing 4 Manufacturing
  - Thermal + Fluid + Kinetic
- Microwave Techniques and potential impacts
  - Heat the fiber and nothing but the fiber
- On-line measurements and advanced control systems
  - Enhanced predictive models
  - Multi-dimensional process control

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Thank you!

Spark the future.™

Please visit Harper at Hall 6, Booth C61

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