New Characterization Techniques for Coated Particle Fuel

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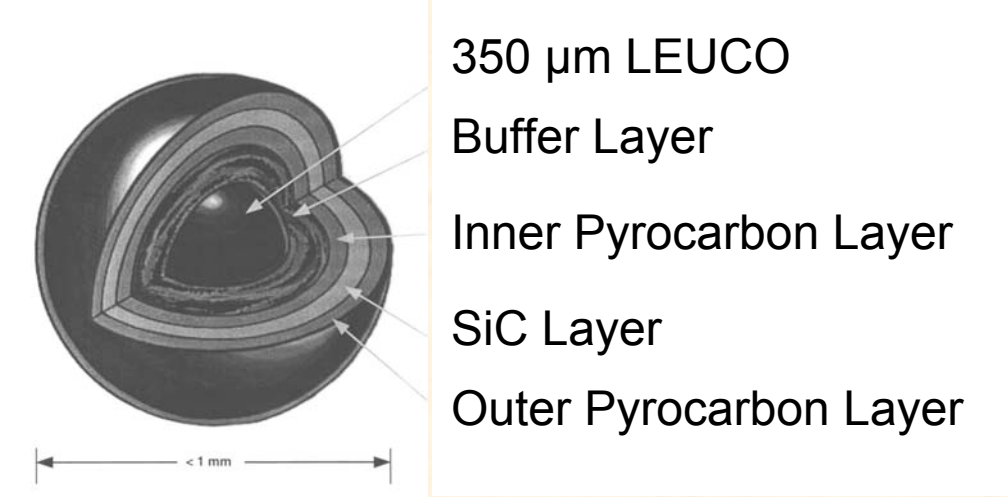
Purpose of Presentation

- Provide overview of coated particle fuel development effort at ORNL.
- Describe new methods developed at ORNL for analyzing coated particle fuel using automated optical microscopy and image analysis.
- Describe new method developed for measuring optical anisotropy in pyrocarbon coatings using an advanced ellipsometry system developed by ORNL called the Two-Modulator Generalized Ellipsometry Microscope (2-MGEM).

Coated Particle Research at ORNL

- Current emphasis is on developing a baseline fuel fabrication process that will be used to provide compacts of TRISO-coated 19.8% U-235 enriched uranium oxide/uranium carbide (UCO) for in-reactor testing in 2007.



Coated Particle Research at ORNL

Research is ongoing in the areas of kernel production, coating development, and compacting methodology. Characterization methods are being developed for all stages of manufacture.

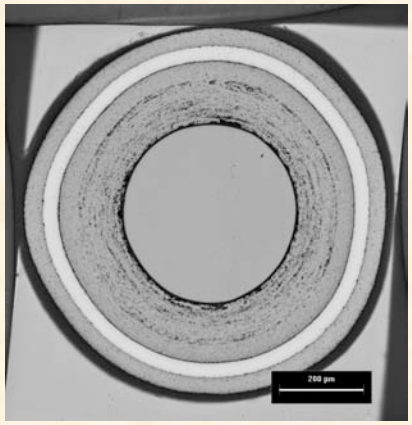
Kernels are fabricated by internal gelation

Coatings are applied in a fluidized bed CVD furnace

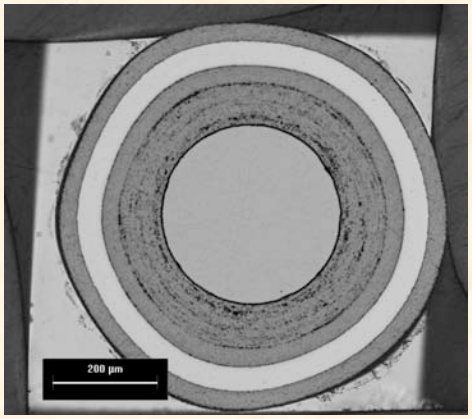
Compacting process uses a thermosetting resin/graphite blend

Coated Particle Research at ORNL

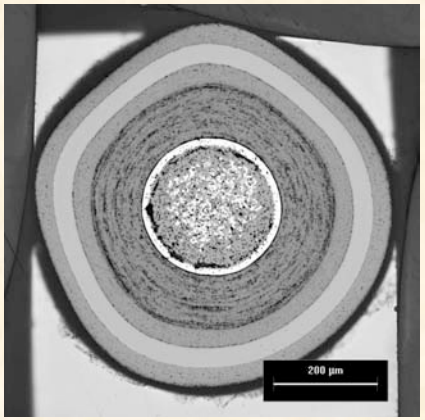
- Coating development is being done using kernels of ZrO_2, depleted UO_2, natural UCO.



ZrO_2



UO_2



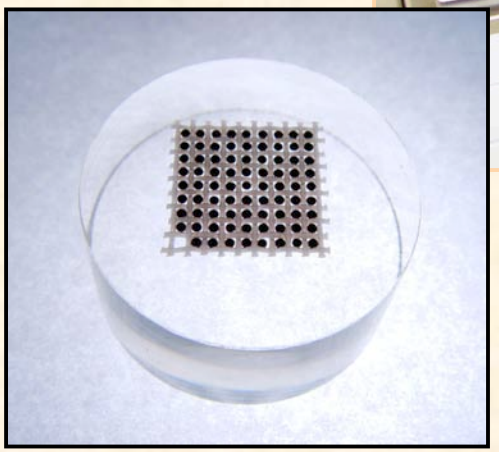
UCO

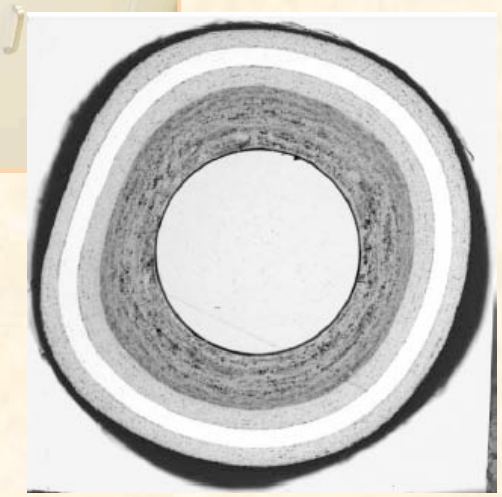
- 19.8% enriched UCO will be coated for use in irradiation testing after coating properties have been optimized.

Computer-Automated Optical Analysis

- Uses computer-controlled sample positioning and digital imaging plus ORNL-developed image analysis software
- Capable of quickly and easily analyzing 1000's of particles for size and shape with 2 μm resolution
- Capable of quickly and easily analyzing 100's of particle cross sections with 1 μm resolution
- Benefits over manual measurements:
  - Data acquisition is easier and more consistent
  - Measurements are typically more accurate
  - More particles are analyzed and more measurements are made on each particle to yield improved statistical analysis of the sample.

Images acquired by an automated microscope





Data extracted by image analysis software

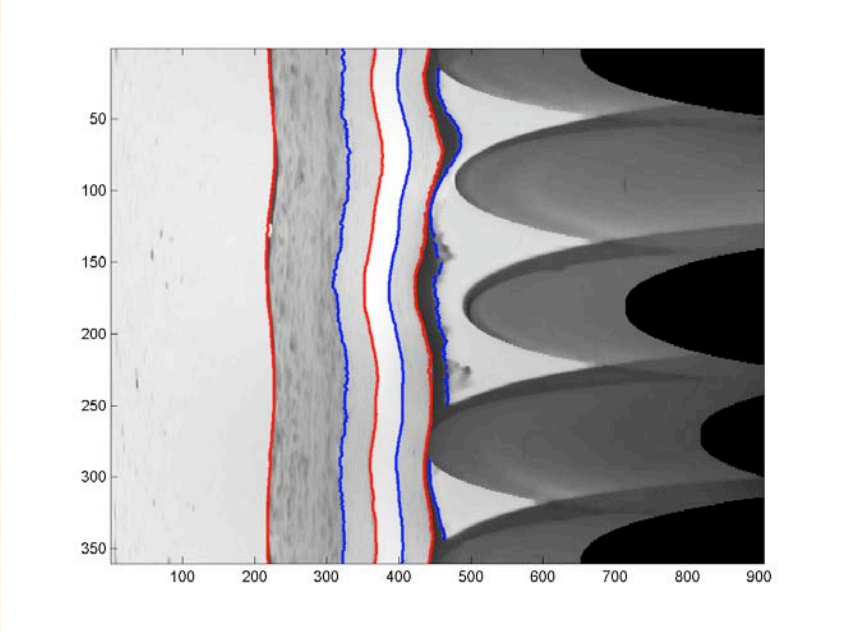
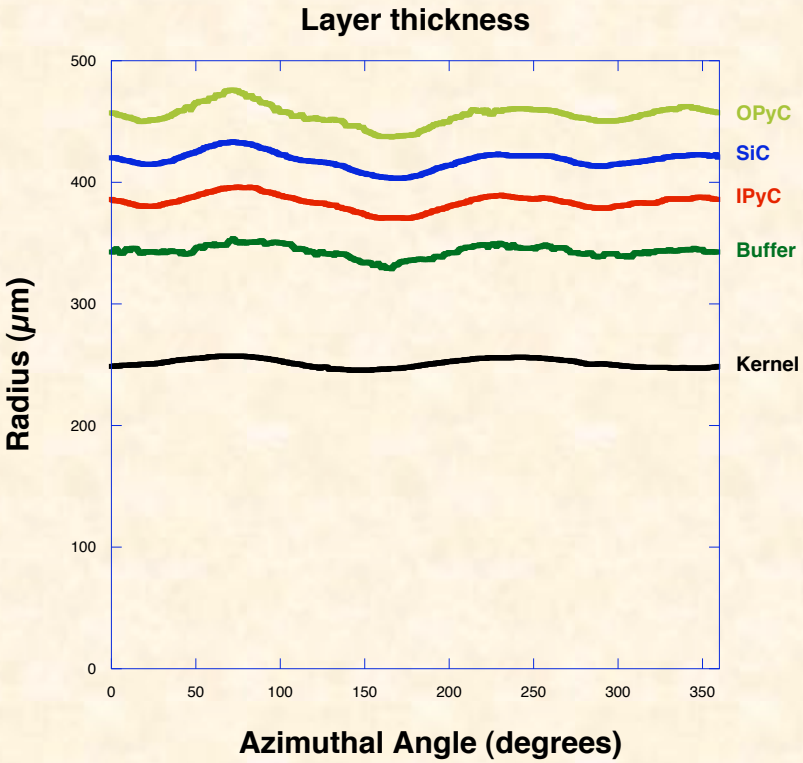


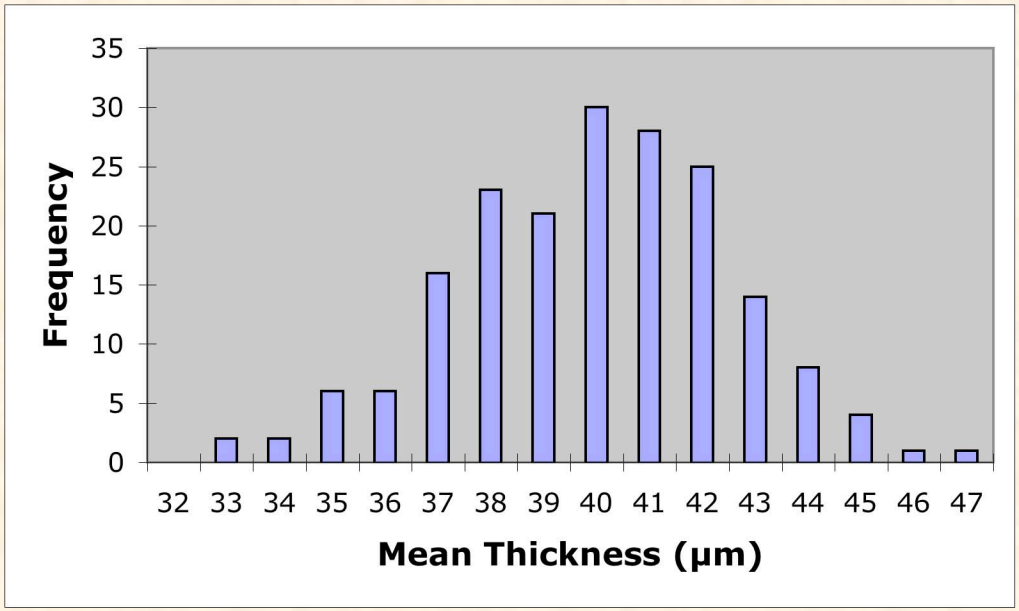
Image is unwrapped and interfaces are detected



360 coordinates are extracted for each interface

Data compiled for statistical analysis

Example:
IPyC layer in German reference fuel
(187 particles were measured)

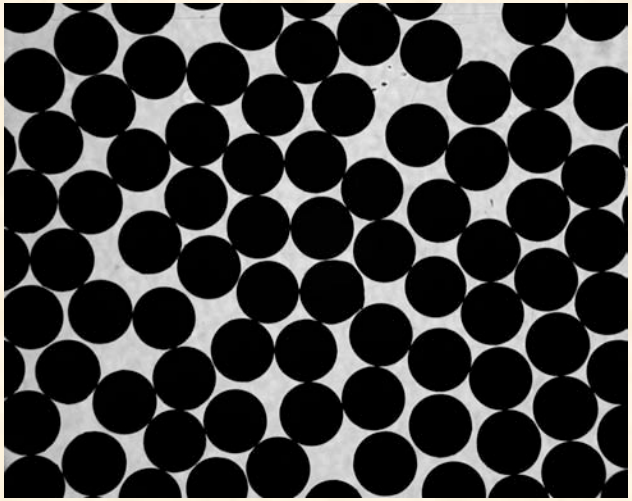


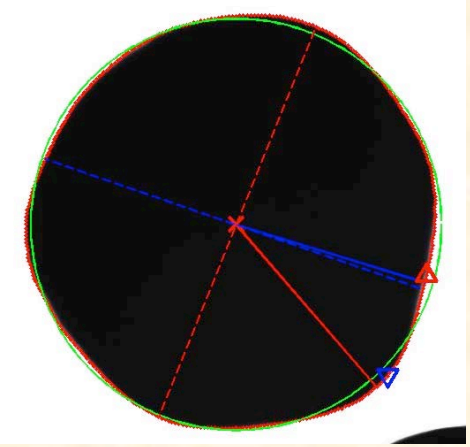
Average Mean IPyC Thickness

39 ± 3 µm

Particle size and shape can also be measured

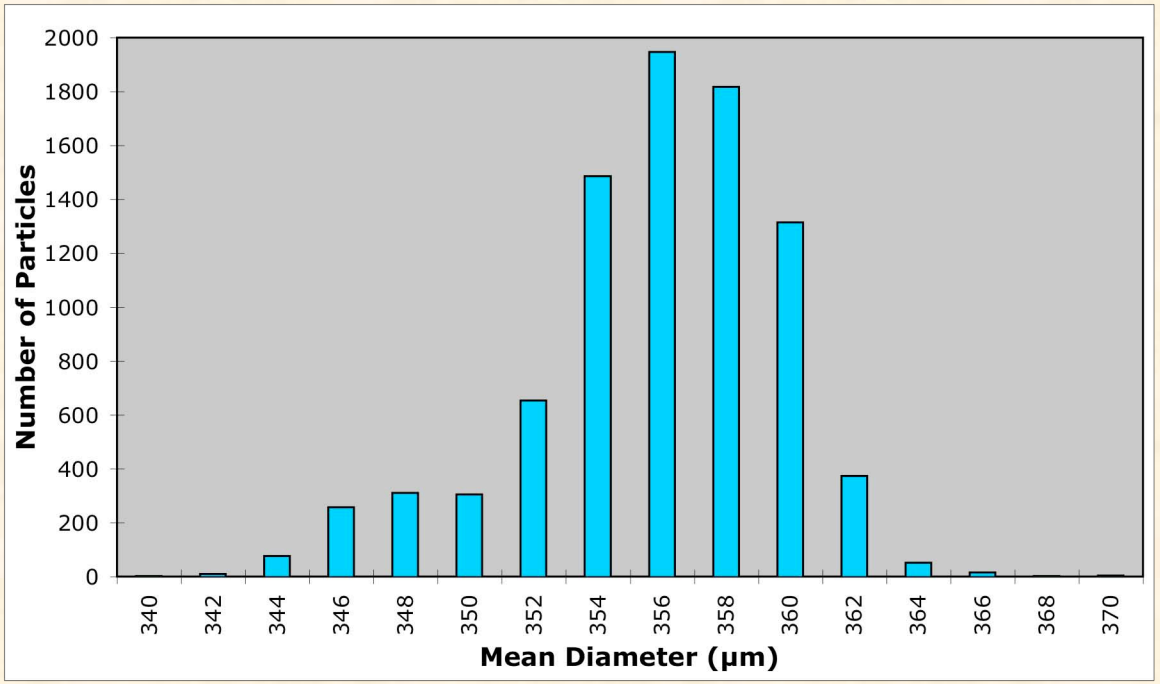
- Typically thousands of particles are imaged using transmitted light to detect the perimeter.





- Each particle is processed to extract perimeter coordinates at up to 360 positions. From this data, size and shape information is calculated.

A typical measurement of size is based on over 1 million data points



Example:

Diameter measurement

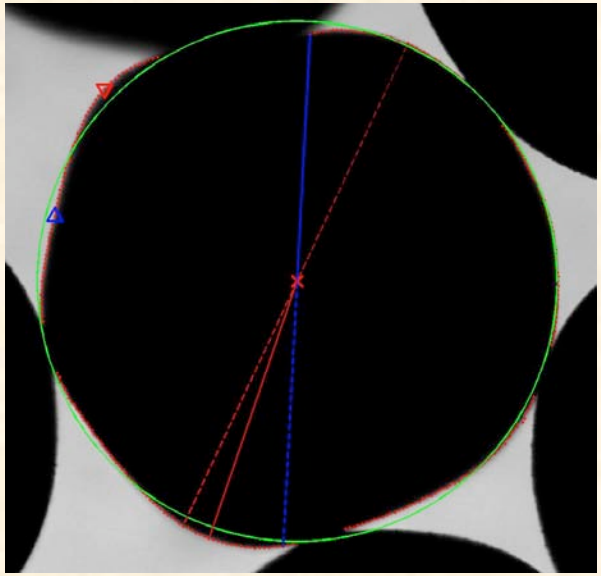
ORNL UO_2 kernels

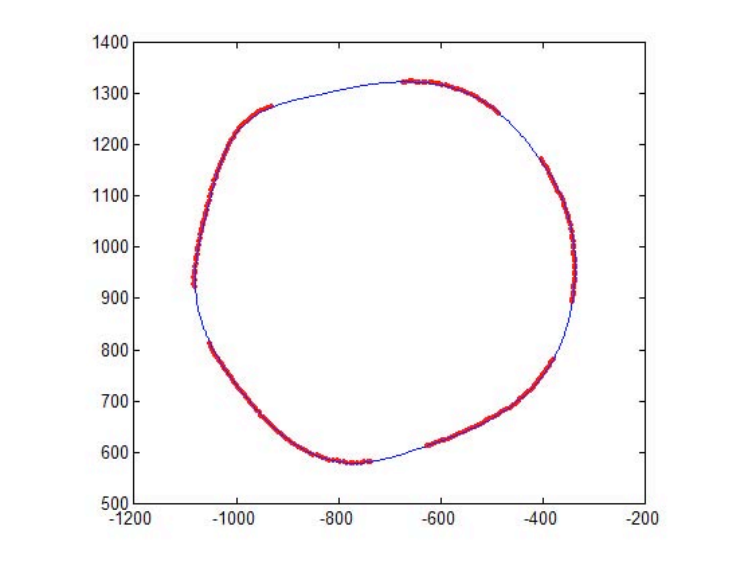
8630 kernels measured

Mean Diameter: 355 ± 4 µm

An analytical equation is fit to each particle perimeter

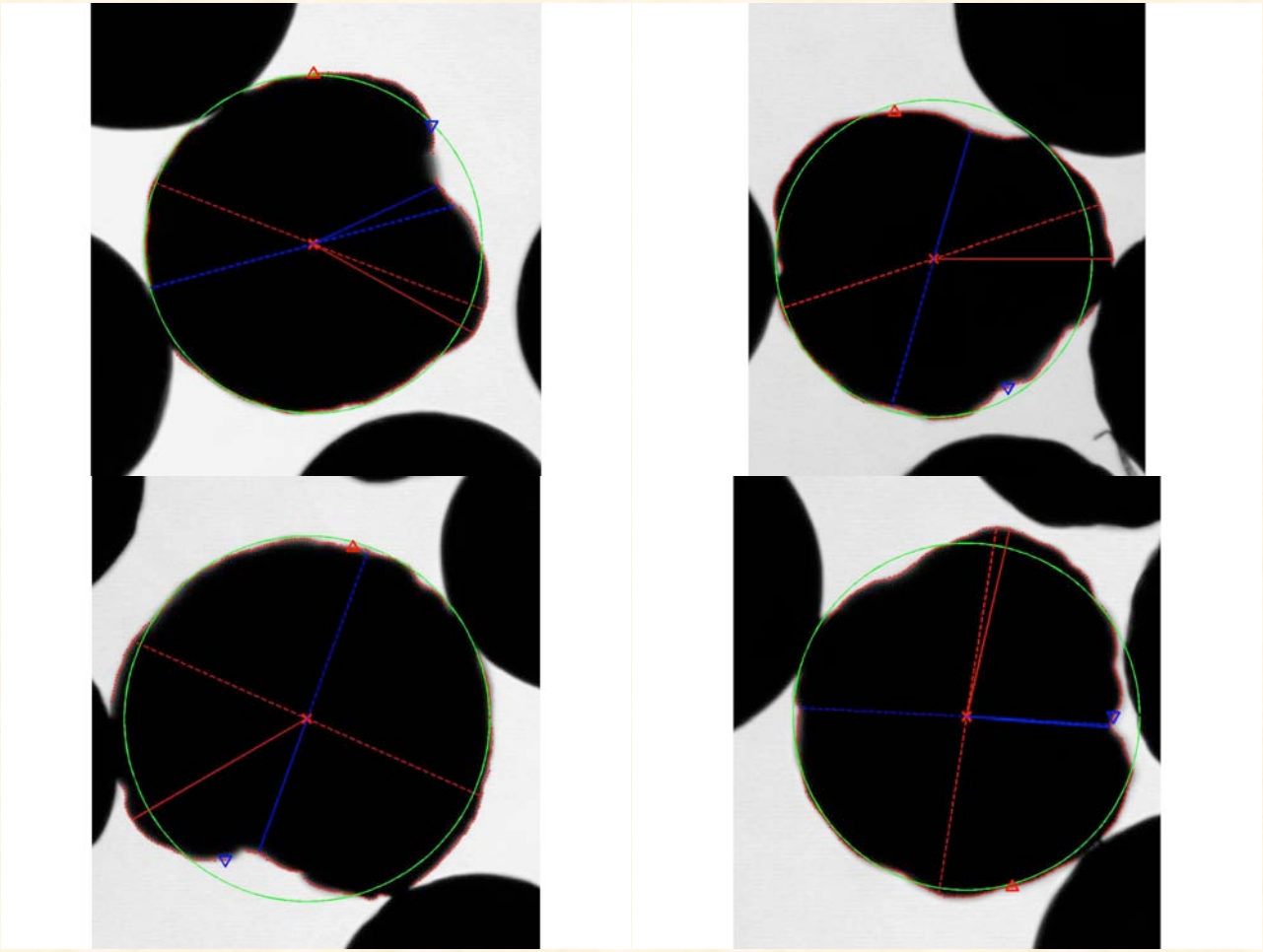
- A ten harmonic Fast-Fourier-Transform is performed to obtain an analytical solution of the image boundary.





- From this fit, particle shape parameters can be extracted.
- Shape can be reported in terms of aspect ratio or curvature.

Size and shape analysis can be used to identify and extract images of abnormal particles

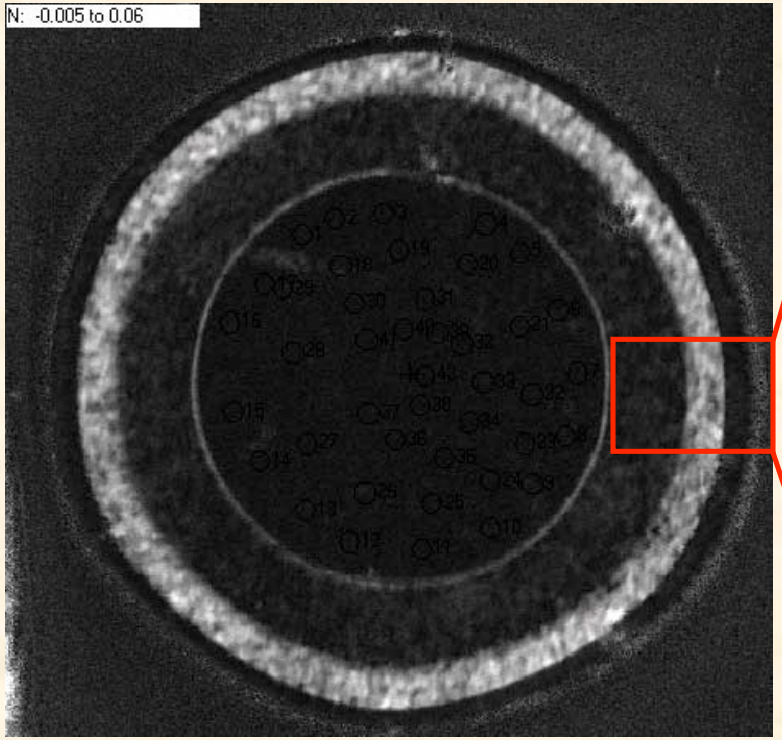


A new Ellipsometry microscope for measuring the crystallographic anisotropy of pyrocarbon has been developed to replace the optical polarimeter

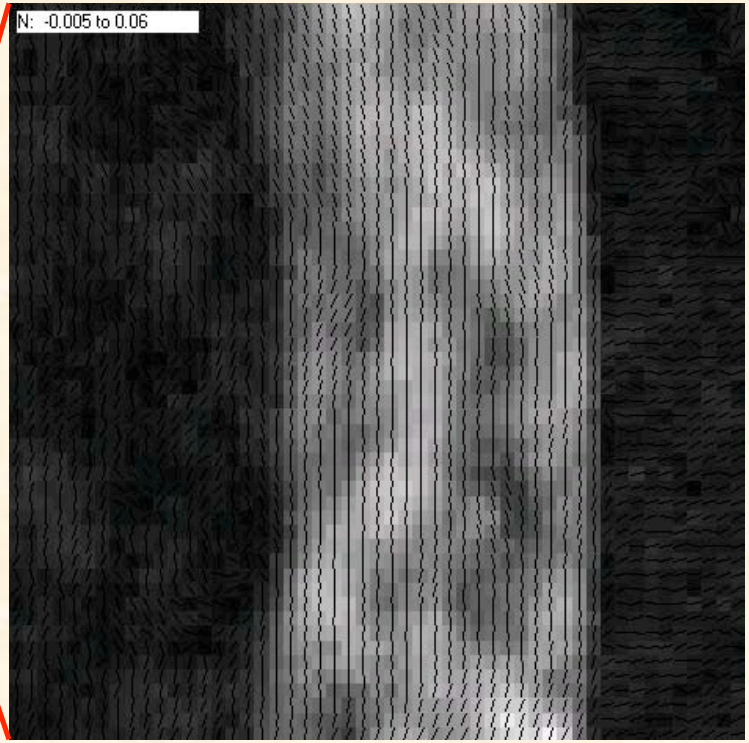
Advantages of 2-MGEM over old system

- **More accurate:** measures the entire Mueller matrix describing polarization
- **More robust:** non-specular reflection doesn't affect the accuracy
- **More sensitive:** can measure anisotropies that are 10 times smaller
- **More complete:** measures up to 250,000 points across the particle surface

Diattenuation and preferred orientation can be measured over the entire cross section. High resolution surface scans show variation in the diattenuation through the IPyC layer



2 µm spatial resolution



Lines show fast axis orientation

Benefits of 2-MGEM

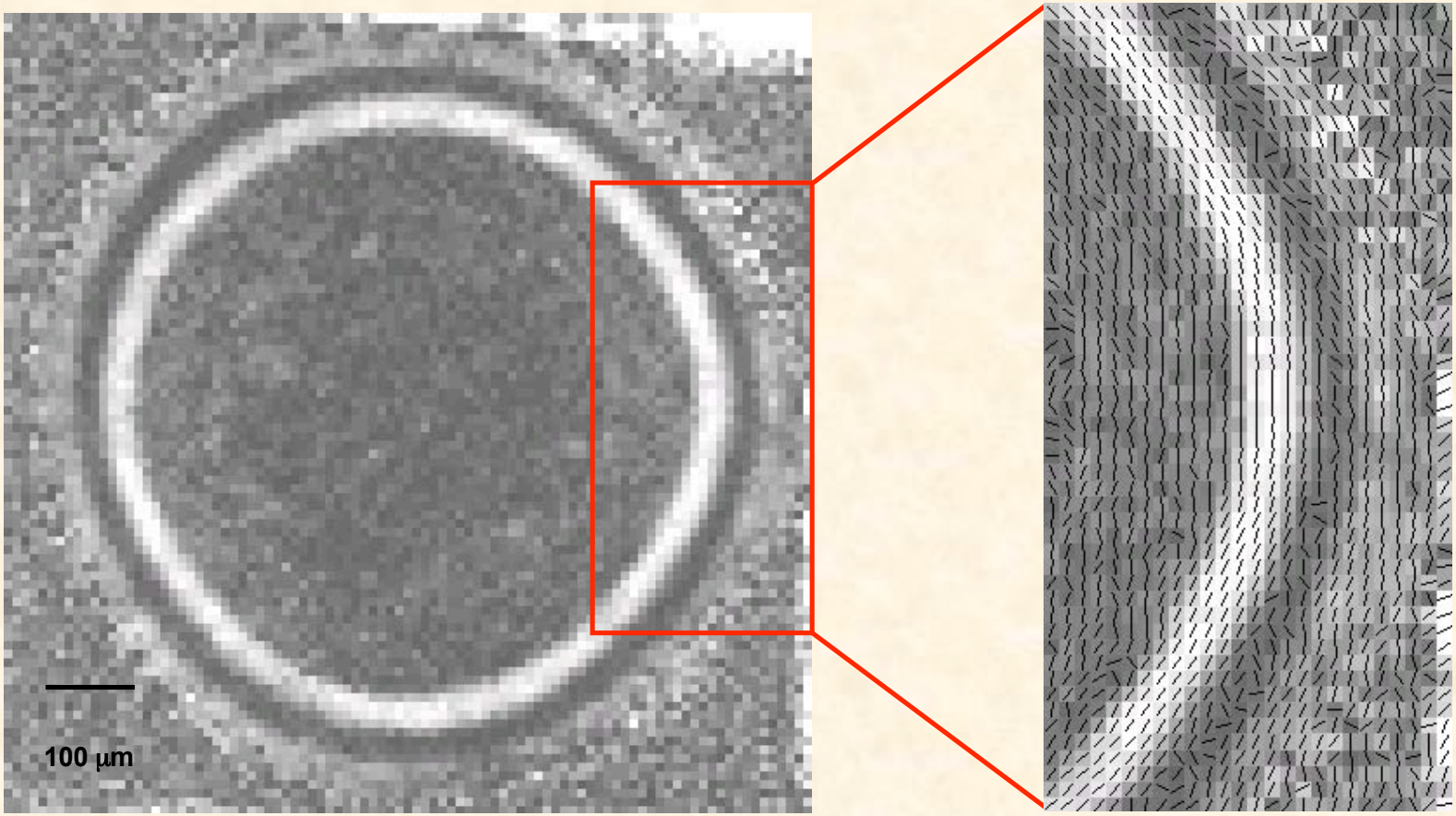
- Accurate measurement of anisotropy for satisfaction of a fuel product specification.
- Less effort required to collect statistically sufficient data.
- Repeatable high resolution measurement of diattenuation for parametric studies of anisotropy versus coating conditions and other coating properties such as density, porosity, and permeability.
- High spatial resolution mapping of anisotropy within the pyrocarbon layers for better understanding of the phenomenon.

Summary

- Coated particle fuel research is alive and well at ORNL.
- New characterization methods are being developed to better understand the process and to make analysis more accurate and efficient.
- Automated microscopy and image analysis software play a key role in a modern approach to fuel development and quality control.
- Advanced ellipsometry provides capability to better study relationship between pyrocarbon anisotropy and fuel performance.

End Show

Diattenuation and preferred orientation can be measured over entire cross section



Lines show fast axis orientation

Computer-controlled stage allows automated data acquisition similar to optical analysis system

Example:

Anisotropy of German reference fuel

12 particles measured

Diattenuation

	IPyC	SiC	OPyC
Average	0.0140	0.0016	0.0080
Standard Deviation	0.0016	0.0003	0.0010
Maximum	0.0174	0.0024	0.0097
Minimum	0.0122	0.0011	0.0062

IPyC Diattenuation	*Frequency*
0.012	0
0.013	4
0.014	5
0.015	0
0.016	2
0.017	0
0.018	1
0.019	0
More	0

IPyC OAF = 1.028±0.004

OPyC Diattenuation	*Frequency*
0.006	0
0.007	1
0.008	6
0.009	4
0.01	1
More	0

OPyC OAF = 1.016±0.002