Coated Particle Fuel Characterization Lab
for the
Advanced Gas Reactor Fuel Development and Qualification Program

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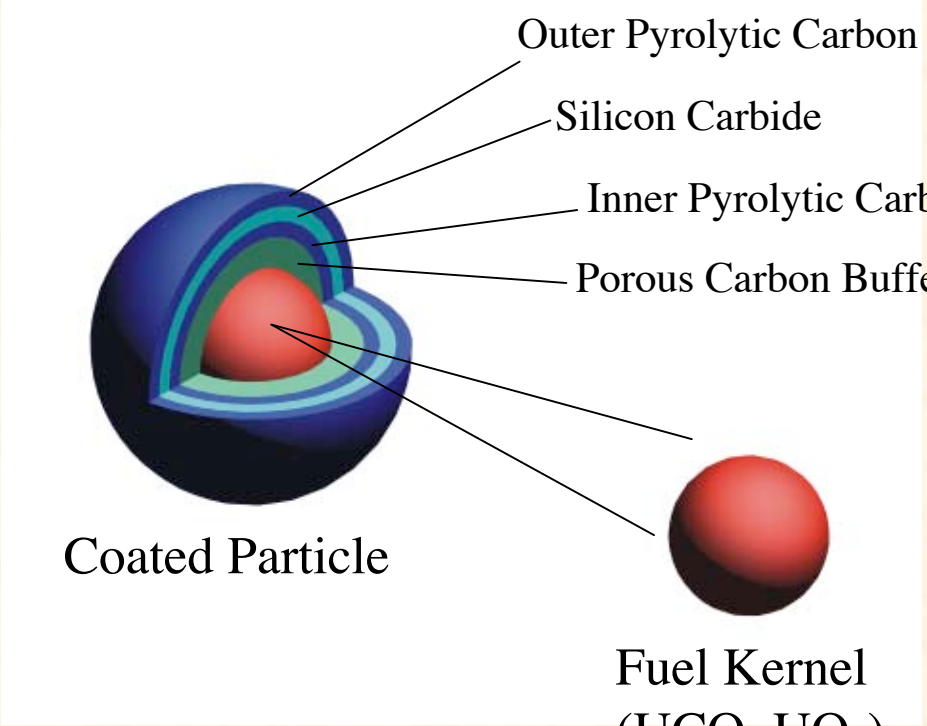
OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

UT-BATTELLE

Purpose of Presentation

- Introduction to ORNL's new coated particle fuel characterization effort
- Description of current capabilities
- Demonstration of some of the measurements

TRISO-Coated Particle Fuel



- **Kernels to be used at ORNL in AGR Program**
  - **500 μm depleted UO2**
  - **350 μm depleted UO2**
  - **350 μm natural UCO**
  - **350 μm LE UCO**

Properties to Measure

KERNELS
Size
Shape
Density
Composition

SiC
Thickness
Uniformity
Density
Grain Size
Microstructure
Failure fraction

IPyC
Thickness
Uniformity
Density
Porosity
Microstructure
Permeability
Anisotropy

BUFFER
Thickness
Uniformity
Density
Porosity

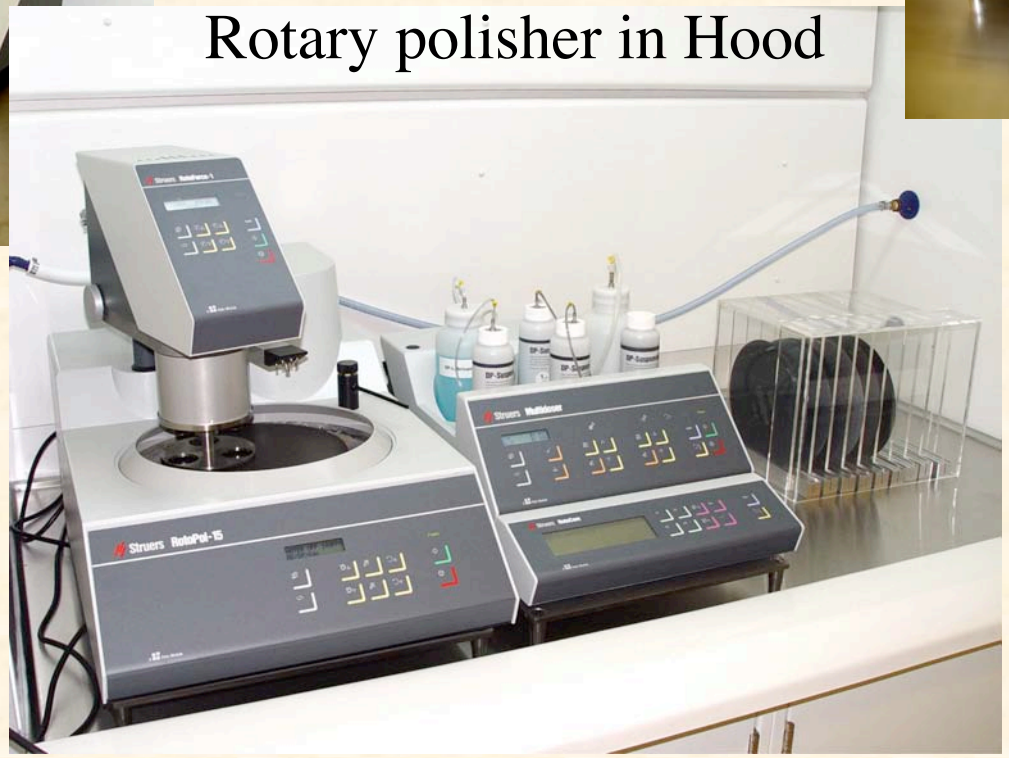
OPyC
Thickness
Uniformity
Density
Porosity
Microstructure
Anisotropy

Materialography

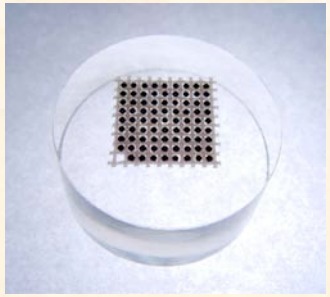
Hot Press

Back Pot

Rotary polisher in Hood

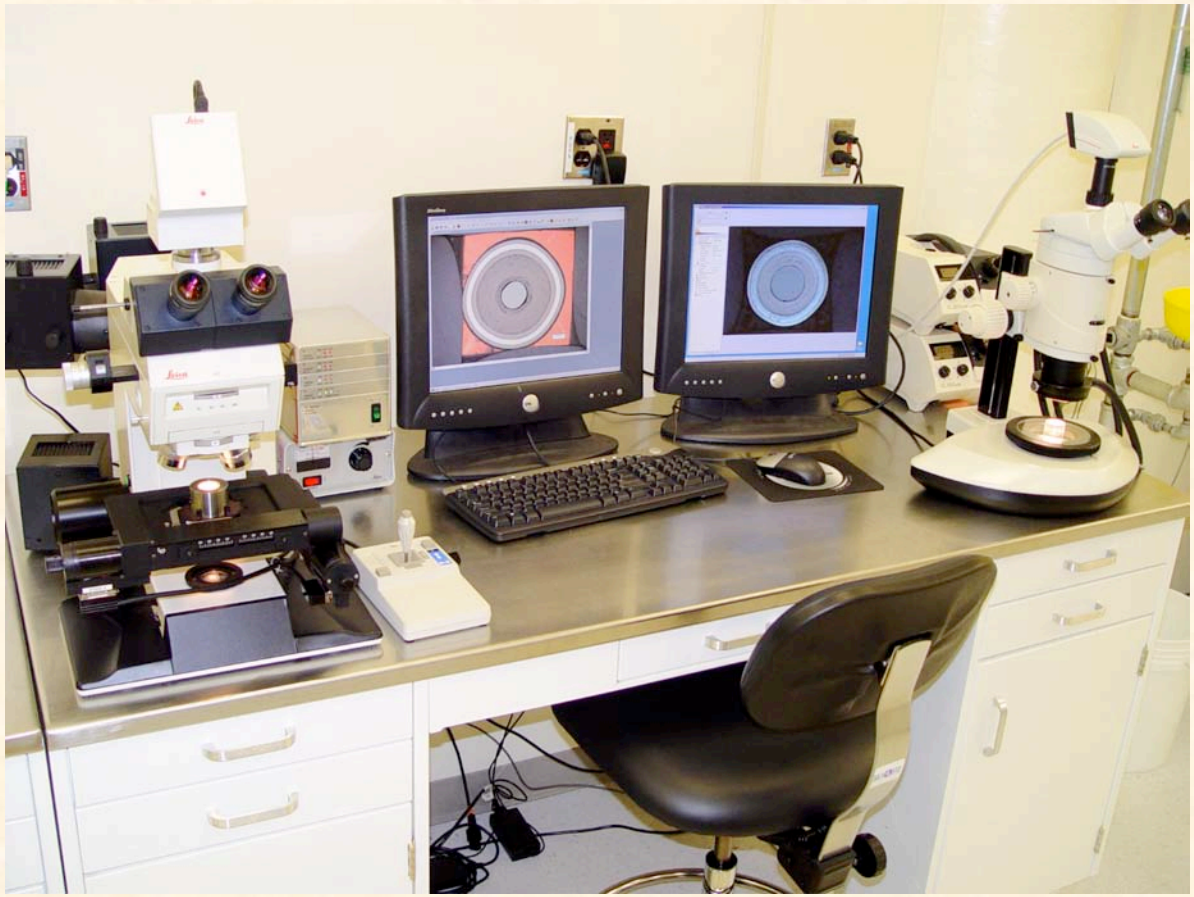


Mount



Microscopy

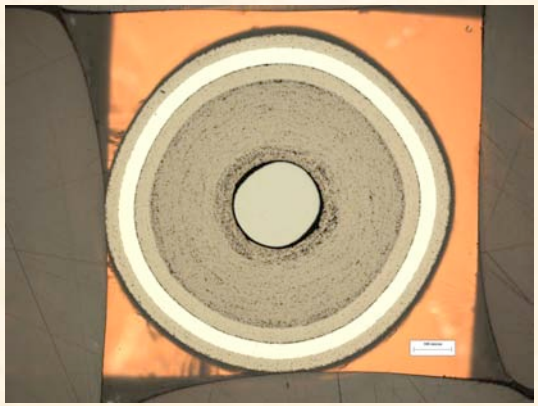
Analytic Scope

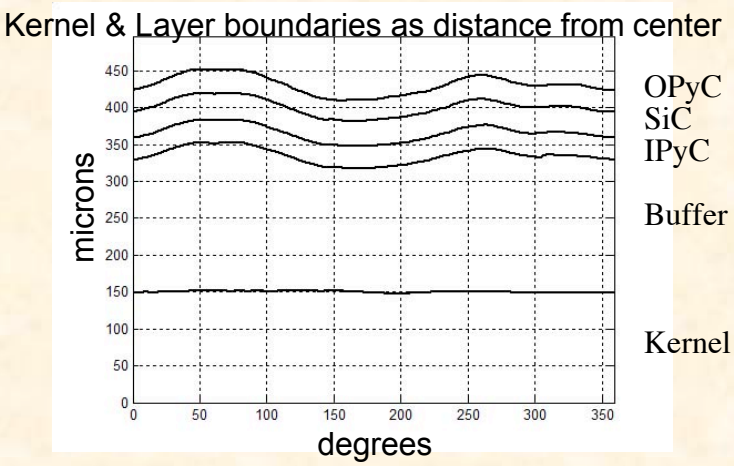


Stereo Scope

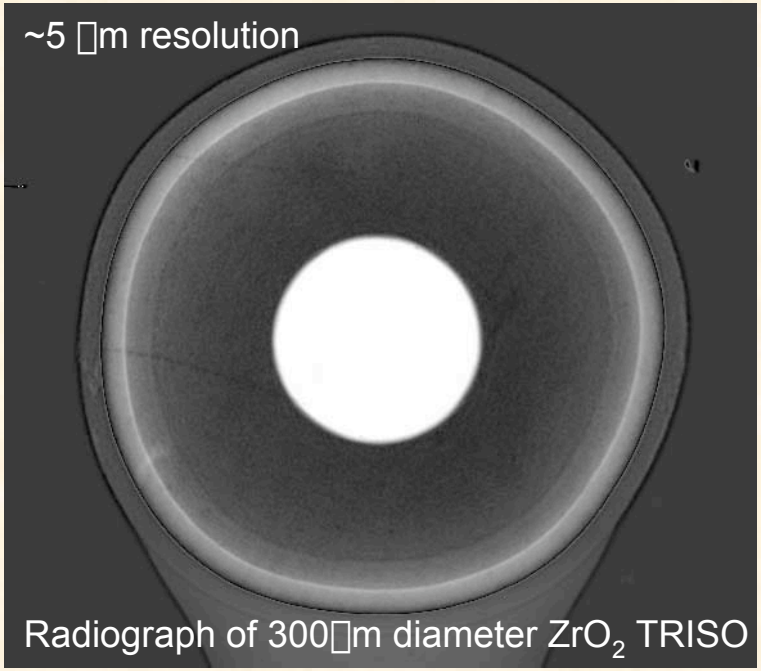
Computer-controlled stage for automatic imaging

Measurement	**Buffer/IPyC/SiC/OPyC** thickness
Technique	Optical inspection of polished cross sections
Equipment	Hot mount press RotoSystem for grinding and polishing Cold mount vacuum impregnator Optical microscope with reflected and transmitted light, digital camera, goniometer stage
Method	Using clear acrylic, mount 99 coated particles in a grid Fine grind with 9 µm Allegro until kernels exposed Backpot with fluorescent epoxy to immobilize kernels Fine grind with 9 µm Allegro to near mid-plane Polish with 3 µm Dac then 1 µm Dac Image at 12.5x polished cross-section with reflected bright field illumination and use transmitted light to illuminate mid-plane Capture 99 images using grid scan Extract info on coating thicknesses with Mat Lab





Micron-resolution x-ray imaging test results

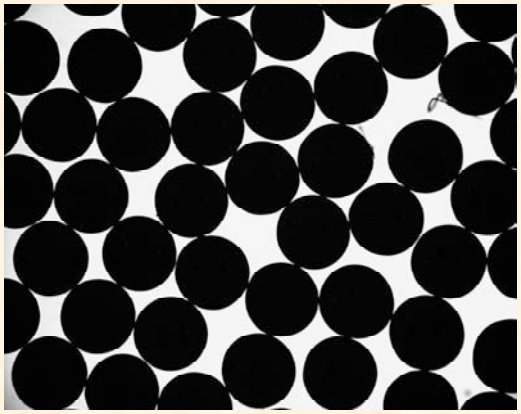


1-2 μm resolution

Imaging of this quality would be adequate for non-destructive measurement of coating thickness and identifying coating defects

Measurement	**Kernel** diameter and sphericity - Option 1	**Kernel** diameter and sphericity - Option 2
Technique	Optical inspection of a random sample	Measure each kernel with laser obscuration
Equipment	Optical microscope with transmitted light, digital camera, goniometer stage	Particle size analyzer with pneumatic particle transport system - under development
Method	Pour single layer of kernels in tray and cover with glass Shadow image at 5x with transmitted light Capture images using frame scan Extract radius R(N) for N= 1-360° Report mean, s.d., min, max, sphericity for each kernel Report mean, s.d., min, max of individual means Report mean, s.d., min, max of sphericities	Suck up kernels into a 1.5x diameter ID tube Pass kernels through a laser slit Measure amount of light blocked by each kernel Disentrain kernels from air flow in a cyclone separator

Sieved ZrO_2



132 kernels measured

Diameter	Sphericity
509±4 μm	1.03±.01
502-522 μm	1.01-1.08

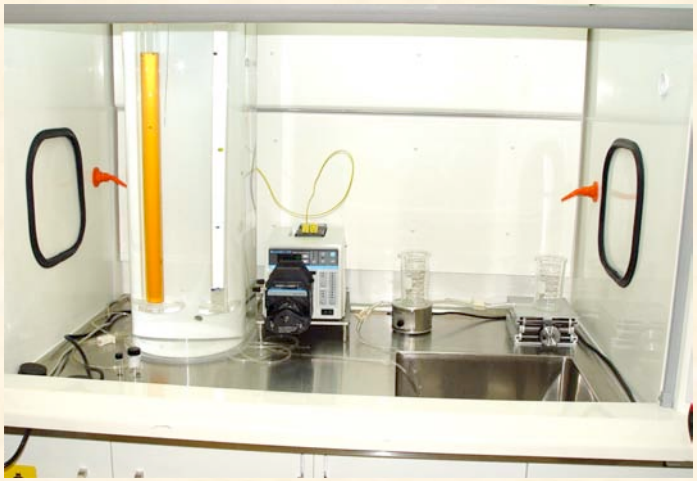
Measurement	**Kernel** density
Technique	Gas pycnometry and Hg porosimetry
Equipment	He pycnometer Analytical balance Hg porosimeter
Method	Run standard measurement using pycnometer Need to check if kernel porosity is significant

Measurement	**Buffer** density
Technique	Measure buffer coated kernel and subtract out contribution of kernel
Equipment	Hg porosimeter Particle counter
Method	Count representative sample of kernels Measure weight and volume of kernels Count representative sample of buffer coated kernels Measure weight and volume of buffer coated kernels Density of buffer = Diff. in av. weight / Diff. in av. Volume

Mercury Porosimeter In Hood

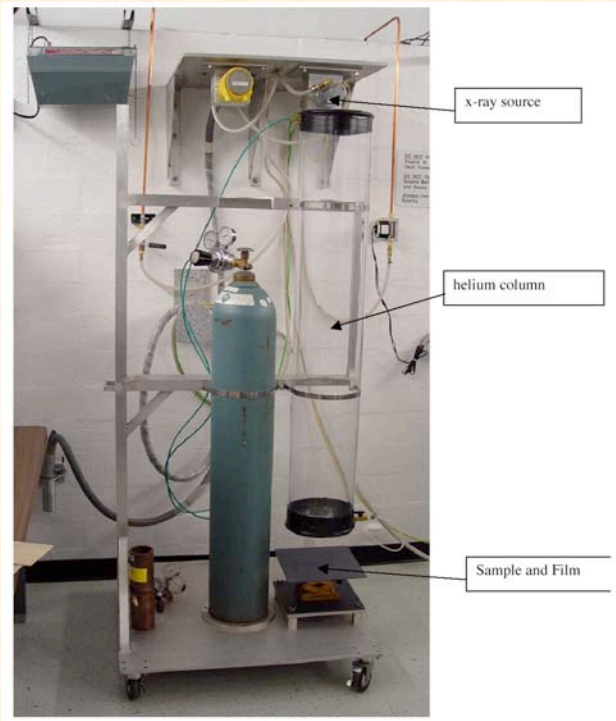
Measurement	**SiC** density	**OPyC** density
Technique	Measure float density and check porosity	Same
Equipment	Liquid gradient density column Temperature controlled water bath Peristaltic pump with fill system Calibrated reference floats Hg porosimeter	Same
Method	Build 3.1- 3.3 g/cc column with Bromoform and Methylene iodide	Build 1.7-2.1 g/cc column with Tetrachloroethylene and Dibromoethylene

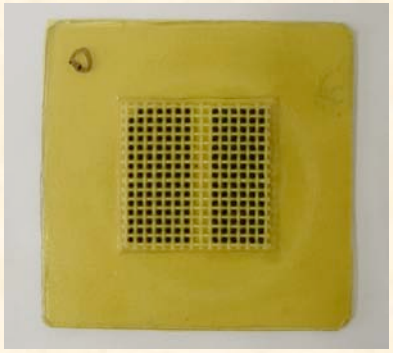
SiC

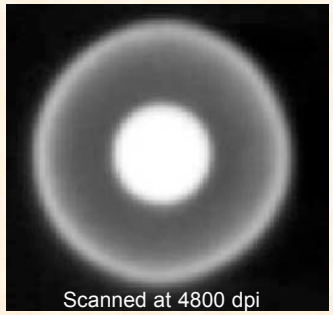


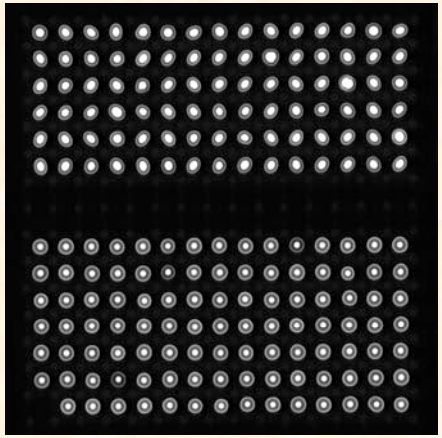
PyC

Measurement	**IPyC** permeability	**SiC** Defects
Technique	U leaching by HCl	Mercury intrusion
Equipment	HCl ready tube furnace 20 µm resolution x-ray system	Muffle furnace Hg porosimeter 20 µm resolution x-ray system
Method	Heat particles in HCl Mount particles in grid and seal with Kapton tape X-ray particles and look for U in buffer	Burn back to SiC in air at 750-850°C Expose particles to Hg under pressure Mount particles in grid and seal with Kapton tape X-ray particles and look for Hg inside of SiC



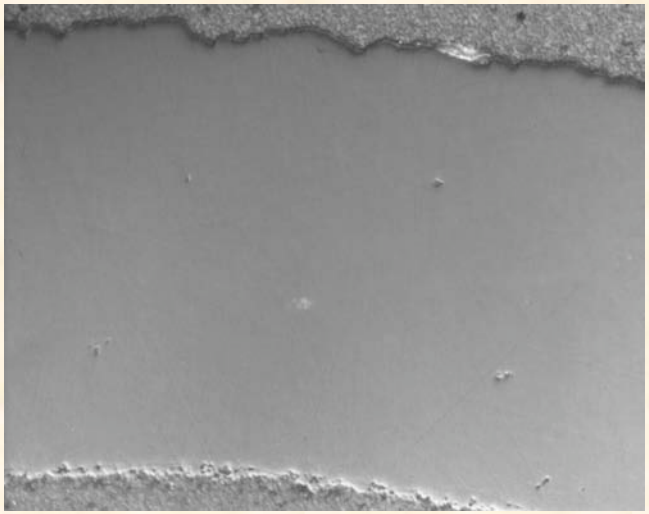




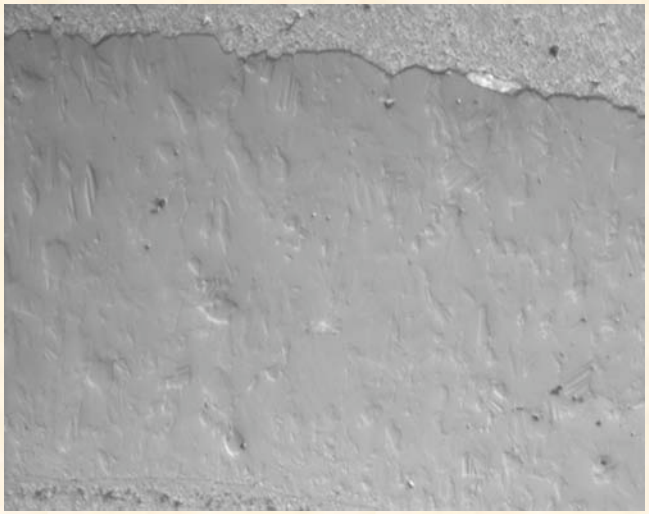


Measurement	**SiC** grain size	**SiC** grain size
Technique	Relief polishing	Wet chemical etch
Equipment	Vibratory polisher	Glassware Hotplate/Stirrer Voltage supply
Method	Prepare polished cross sections as for coating thickness measurement Relief polish with .05 µm silica Image with optical microscope	Electrolytic oxidation in potassium dichromate and phosphoramide solution Observe different grain orientations by color HF etch to remove oxide Electrolytic grain boundary etch in oxalic acid Observe grain size

Before relief polish



After relief polish



New Ellipsometry Method for Measuring the Crystallographic Anisotropy of Pyrocarbon Layer to Replace OPTAF

- More complete measurement of anisotropy
- Higher resolution
- Less sensitive to experimental error from specular reflection
- Able to measure anisotropy over the entire cross-section

Mueller-Stokes Description of Polarization

Stokes Vector:

$$\mathbf{S} = \begin{bmatrix} I_o \\ Q \\ U \\ V \end{bmatrix} = \begin{bmatrix} I_o \\ I_0 - I_{90} \\ I_{45} - I_{-45} \\ I_{rc} - I_{lc} \end{bmatrix}$$

Mueller Matrix:

$$\mathbf{M}_{normal} = \begin{bmatrix} 1 & m_{12} & m_{13} & 0 \\ m_{12} & m_{22} & m_{23} & m_{24} \\ -m_{13} & -m_{23} & m_{33} & m_{34} \\ 0 & m_{24} & -m_{34} & m_{44} \end{bmatrix}$$

OPTAF only measured m_{12} => incomplete measurement

$$OPTAF = \frac{R_s}{R_p} = \frac{1+N}{1-N} \qquad N = \frac{R_s - R_p}{R_s + R_p} = \frac{1-(R_p/R_s)}{1+(R_p/R_s)} = -m_{12}$$

Preliminary results with coated particles

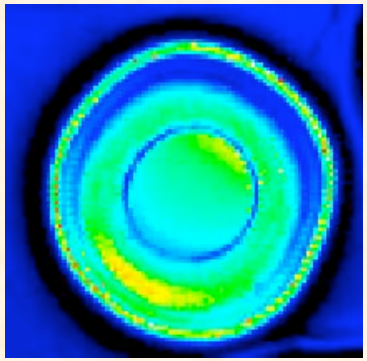
C_2H_2:C_3H_6 ~1:1

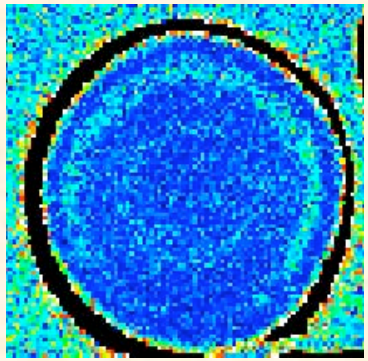
Intensity

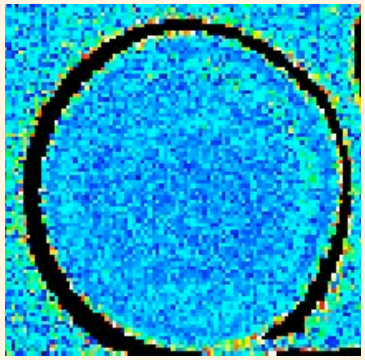
Diattn |N|
(0.00 to 0.04)

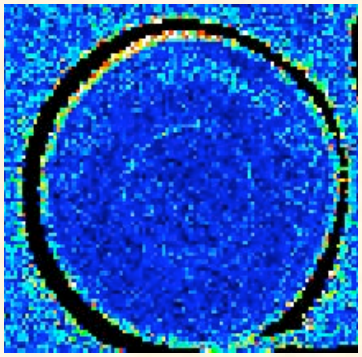
Retard |_|
(0 to 0.1)

Circ. Diattn
(0 to 0.05)







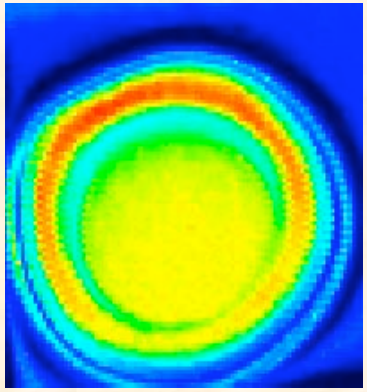


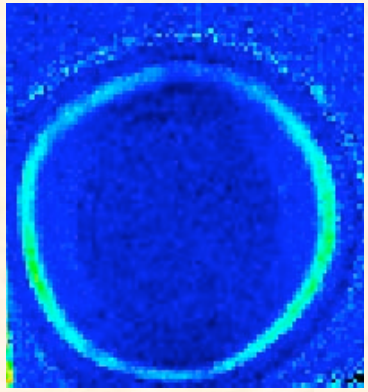
C_3H_6

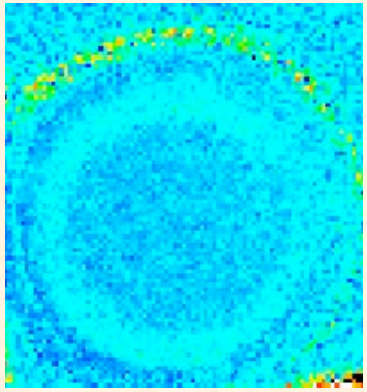
(0.00 to 0.08)

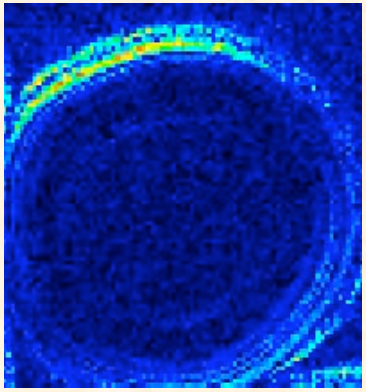
(0.00 to 0.08)

(0.00 to 0.05)





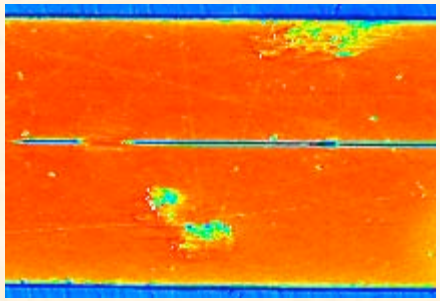




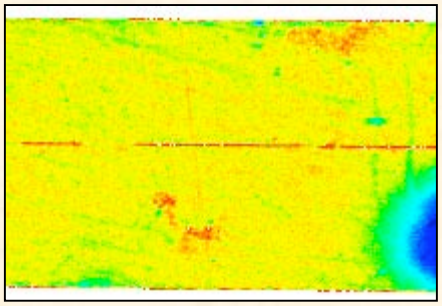
Extra Slides

Highly Oriented Pyrolitic Graphite (10/03/03)

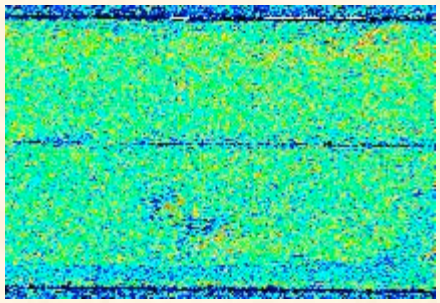
Intensity (0 – 1)



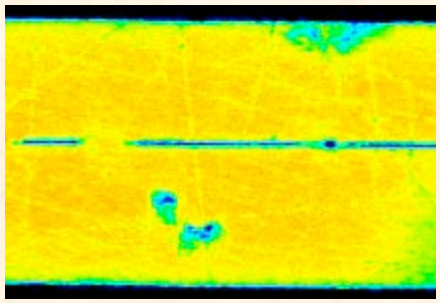
Retardation (-0.25 - -0.50)



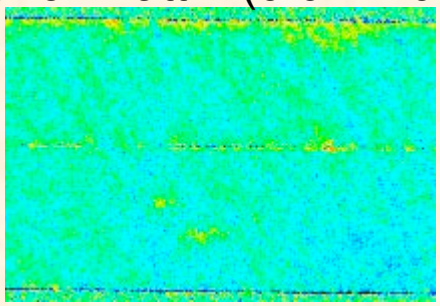
Beta (0.98 – 0.95)



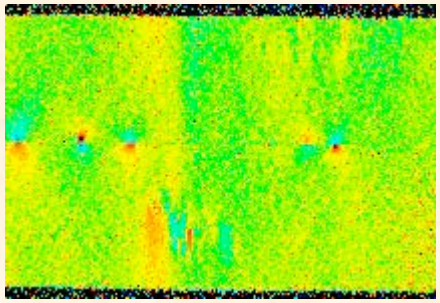
Diattn. (0.40 – 0.65)

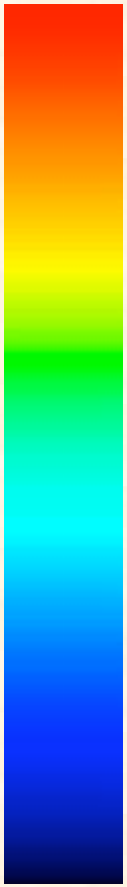


Circ. Diattn. (0.02 - -0.02)



Princ. Angle (170° – 180°)





Mueller matrix elements of HOPG

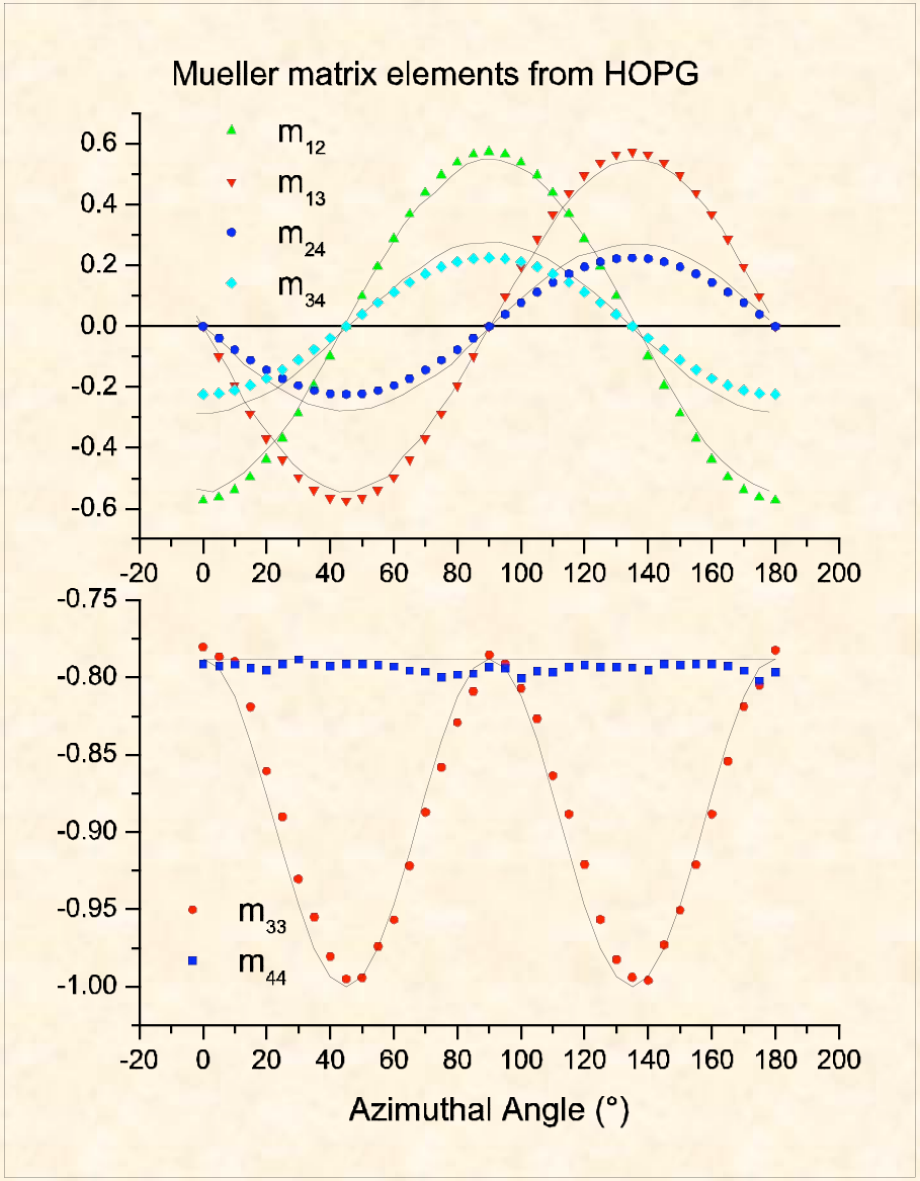
_ = 633 nm

Solid lines calculated using

$n_o = 1.79$

$k_o = 1.20$

$n_e = 1.60$

$k_e = 0.25$

From 2-MGE measurements