OPTICAL NONDESTRUCTIVE EVALUATION TECHNIQUES FOR ENVIRONMENTAL BARRIER COATINGS

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OUTLINE

- Description of Optical NDE methods
- Results of application to Slurry Cast EBC

  -Green state

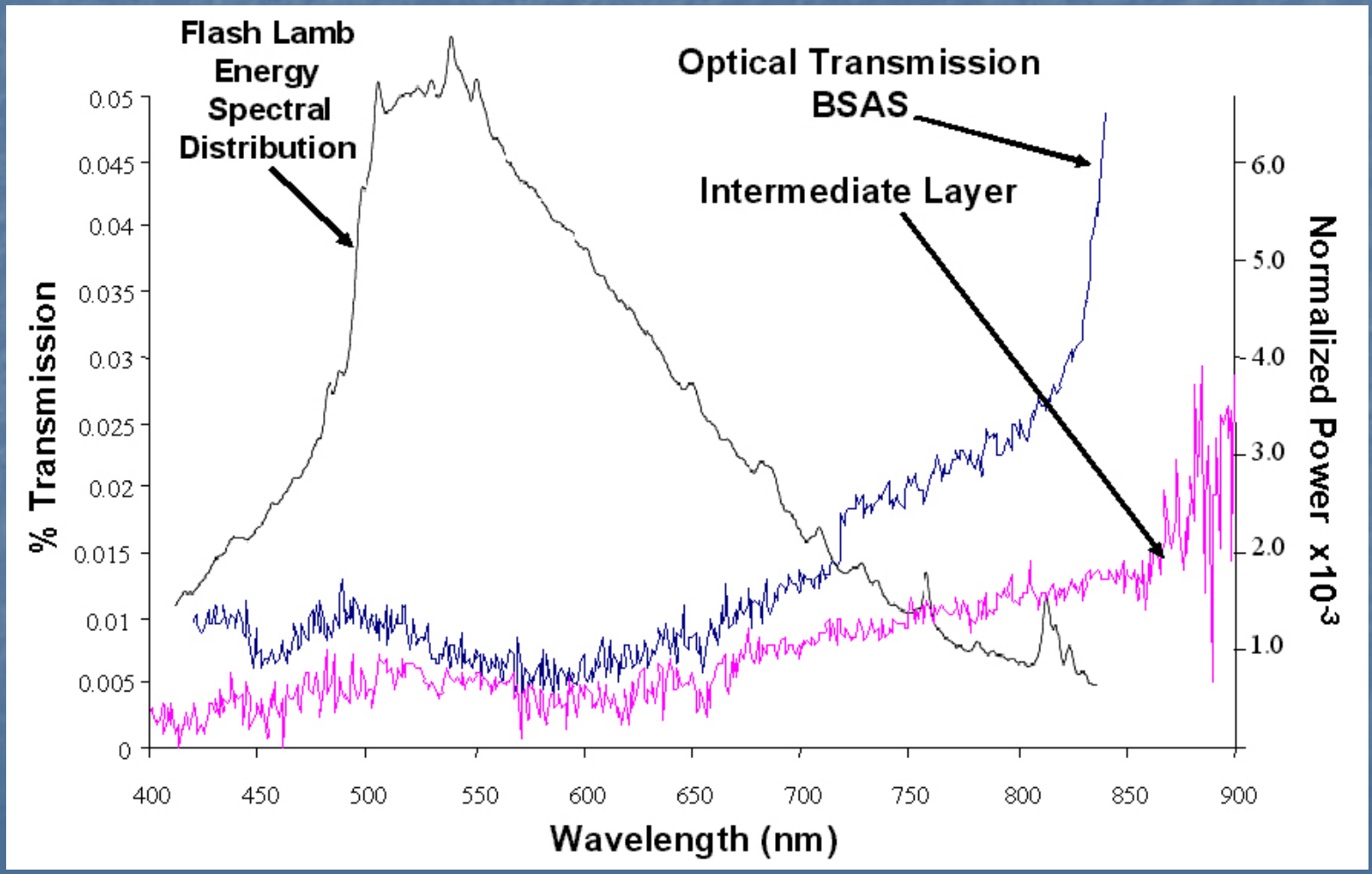
  -Sintered
- Results of application to BSAS and Yb_2SiO_5
- Summary/Conclusions

COOPERATIVE EFFORTS

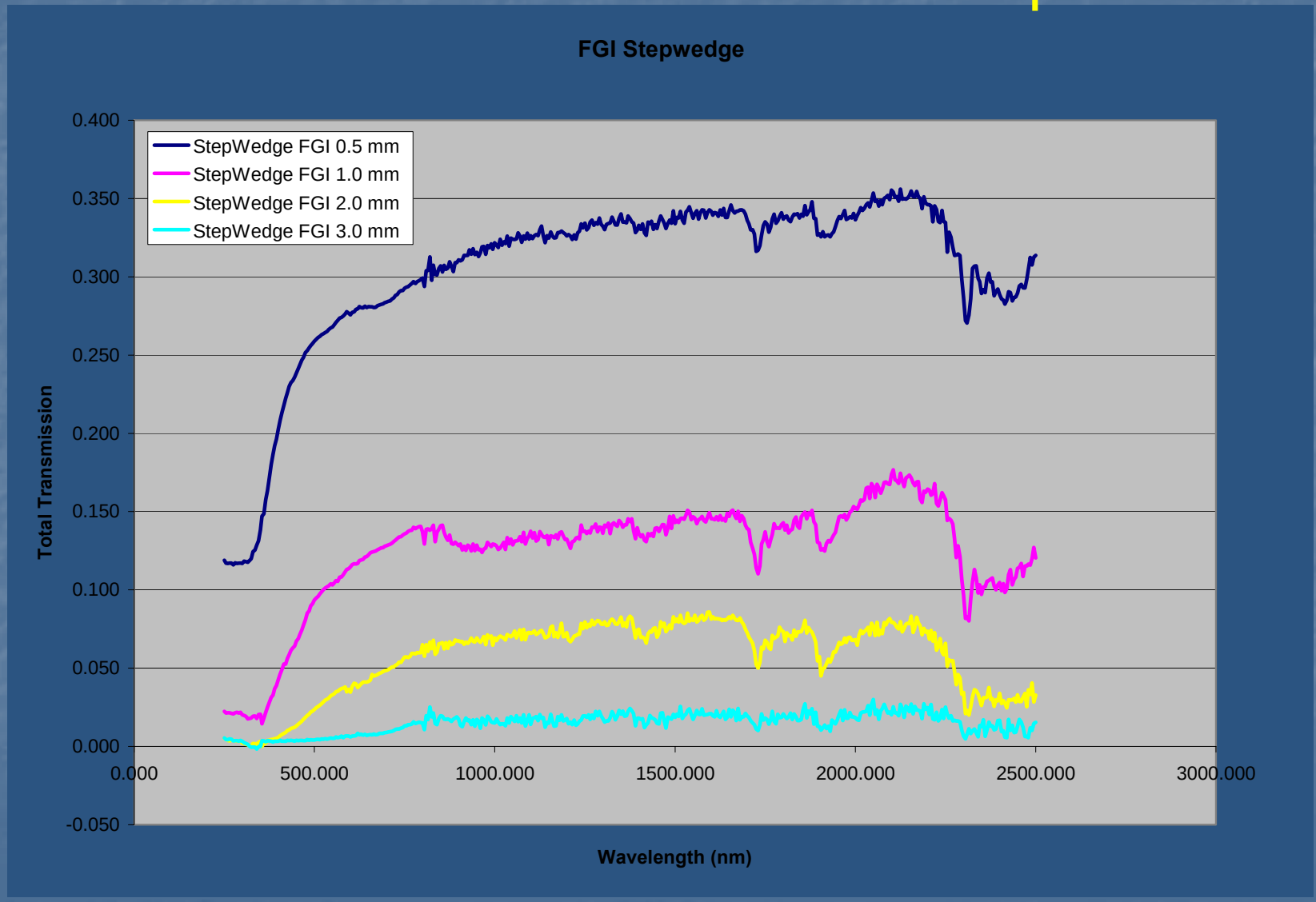
- Ceramatec—
  --oxide-based slurry deposited coatings
- NASA-Glenn Research Center
  Alternative EBC compositions for SiC/SiC
- P&W/United Technology Research Center
  EBCs on SiC/SiC
- GE Global Research Center
  EBCs on SiC/SiC
- Siemens-Westinghouse
  EBCs for oxide/oxide CMCs

PARAMETERS FOR APPLICATION OF OPTICAL METHODS

Coatings MUST have optical translucency



Optical transmission characteristics of one EBC for Oxide/Oxide composite



Optical Techniques Under Development at Argonne

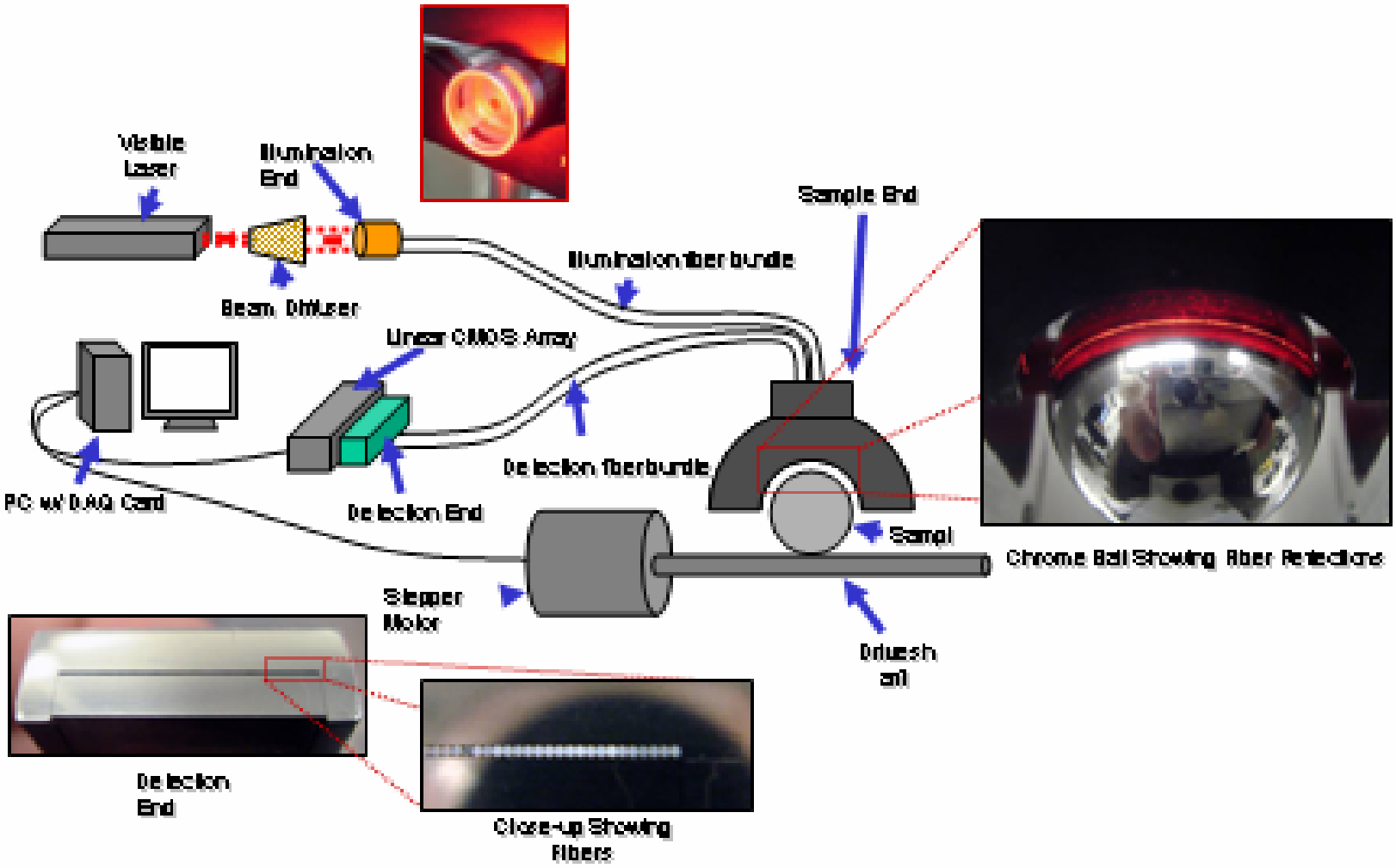
Elastic Optical Scattering (EOS)

Uses polarized laser light to probe the subsurface characteristics of optically translucent ceramic materials

Optical Coherence Tomography (OCT)

Uses a time-gated reflectometer to obtain cross-sectional images of subsurface features of optically translucent ceramic materials in axial and transverse planes

Automated System Schematic



Experimental Setup

- Issue with diameter of fibers in current fiber array
- Compensating for spatial resolution of current fiber array
  - C-cracks were oriented with largest diameter perpendicular fiber array in center of scan
  - Scan speed reduced four times slower than initial scans
  - Increased acquisition frequency
  - Increased signal to noise ratio
  - 2 minutes per ball
- New design will give close to an order of magnitude better spatial resolution

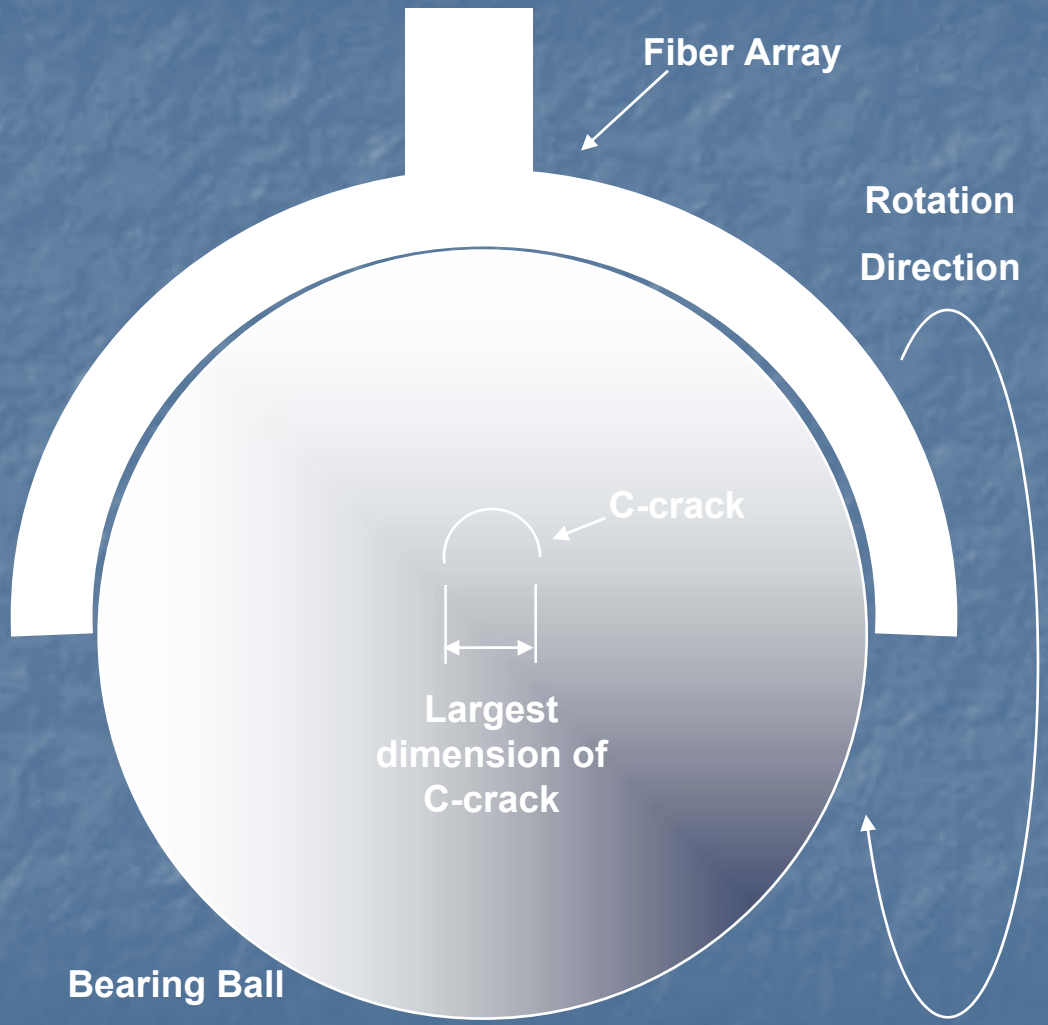
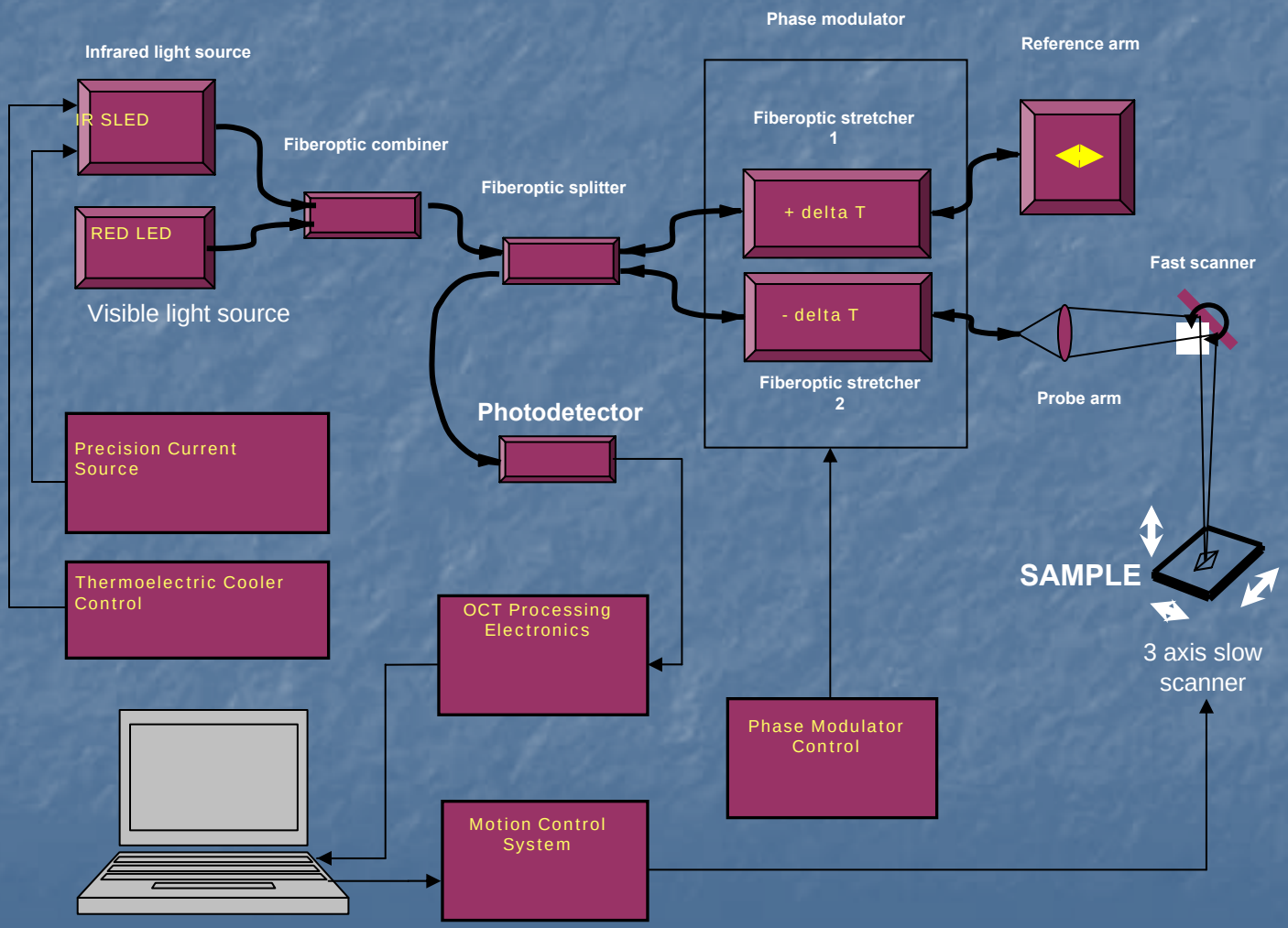


Diagram of Optical Coherence Tomography System



THEORETICAL SPATIAL RESOLUTION LIMITS FOR OCT SYSTEM

In-Plane resolution

$$\Delta x = \frac{4\lambda}{\pi}\left(\frac{f}{d}\right)$$

Where

λ=laser wavelength

f=focal length of the focusing lens

d=diameter of the focusing lens

Vertical plane resolution

$$\Delta z = \frac{2\ln(2)}{\pi}\left(\frac{\lambda^2}{\Delta\lambda}\right)$$

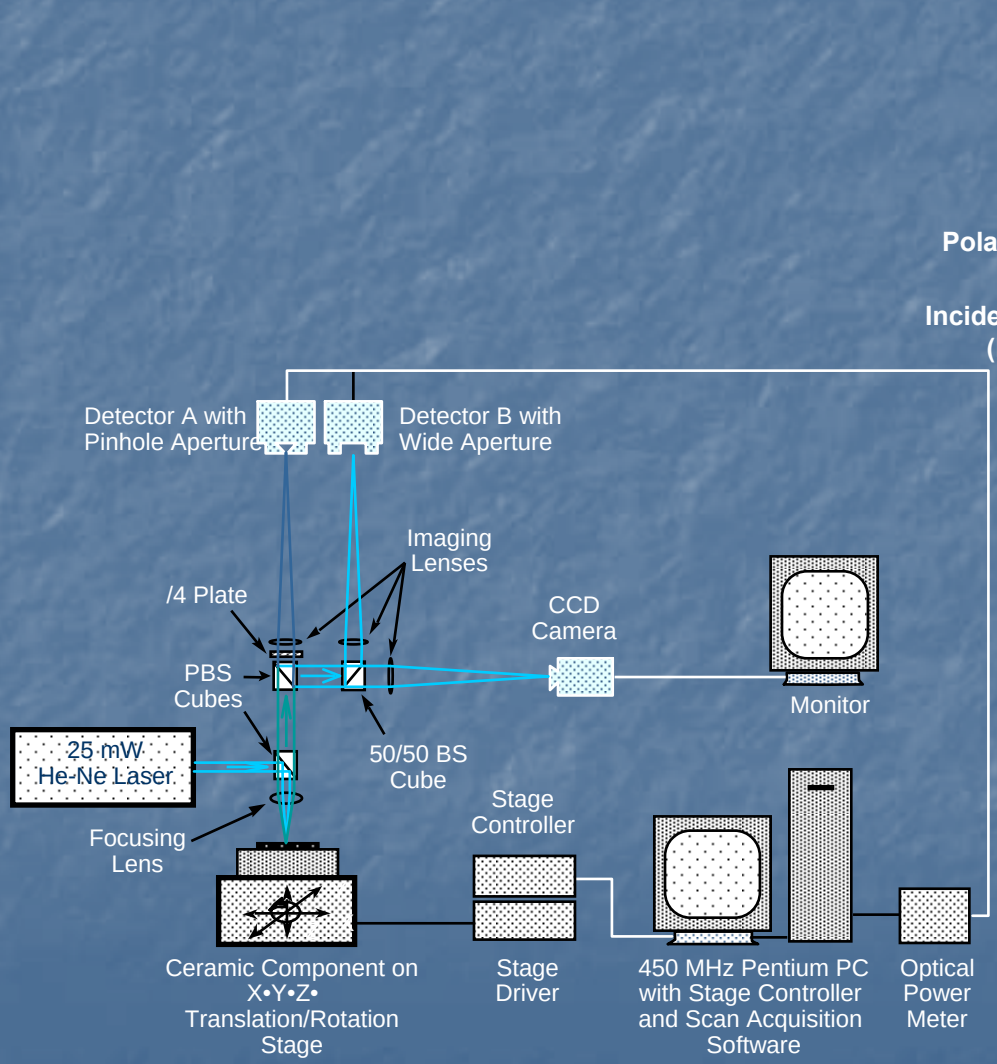
For our system

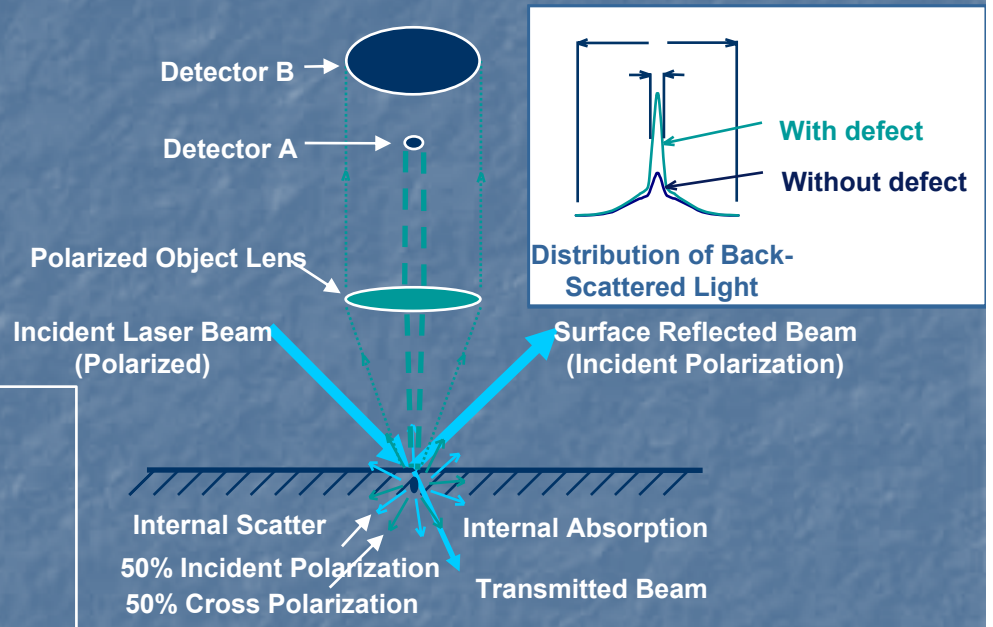
Δx=~10 um(theory suggests ~2-5 um

ΔZ=~18 um(theory suggests ~10 um

Note that for ΔZ, if Δλ is larger, then ΔZ is smaller. Recent work in Austria, using femtosecond laser, has reduced ΔZ to ~4 um and Δx to ~ 2 um over regions of size ~ 5 mm square.

SCHEMATIC OF ELASTIC OPTICAL BACKSCATTER NDE EXPERIMENTAL TEST SETUP





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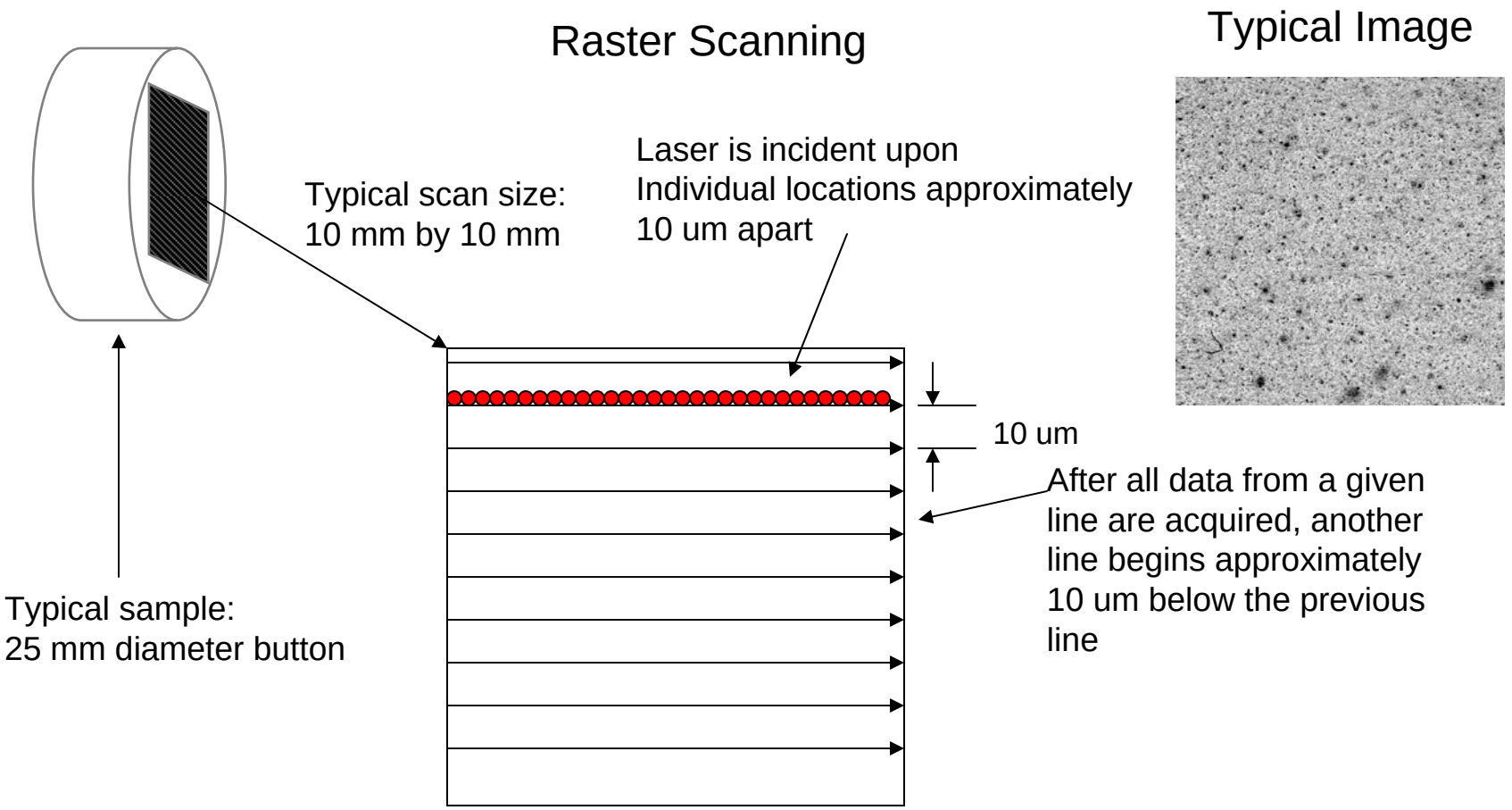
ELASTIC OPTICAL BACKSCATTER DATA ACQUISITION PROCESS



The backscattered light from each point is represented by one pixel in a gray scale image.

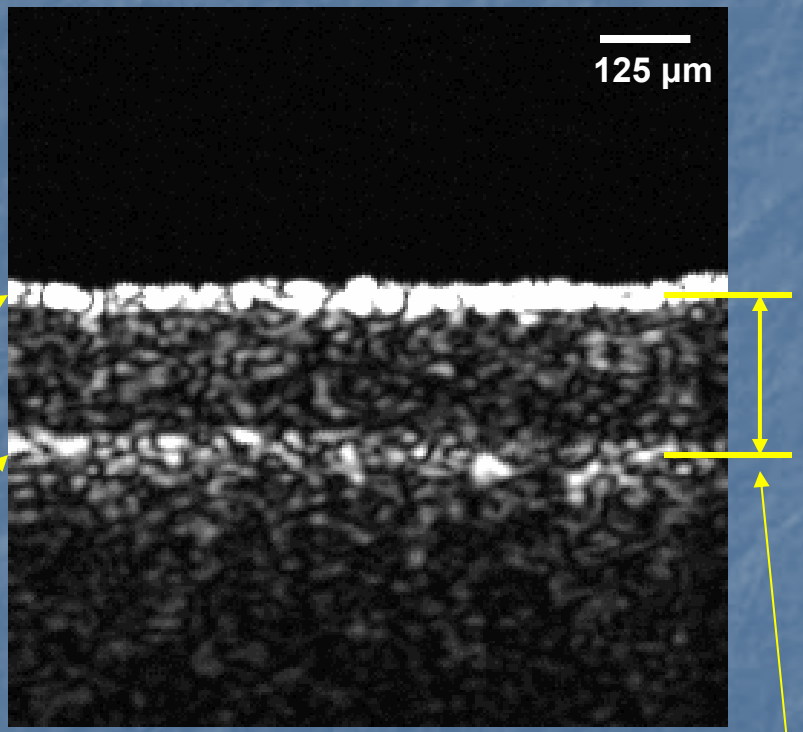
- **CONDTIONS TO BE DETECTED/CHARACTERIZED :**
  - **COATING THICKNESS**
  - **EXISTENCE OF SUBSURFACE CRACKING**
  - **EXTENT OF DELAMINATION/PRE-SPALL**

OCT Measurement of Thickness of 7 % YSZ, EB-PVD

OCT Scan taken along red line perpendicular to the surface

Pressure Side

OCT Vertical Cross-Sectional Image
Λ=1310 nm



TBC thickness ~175 um

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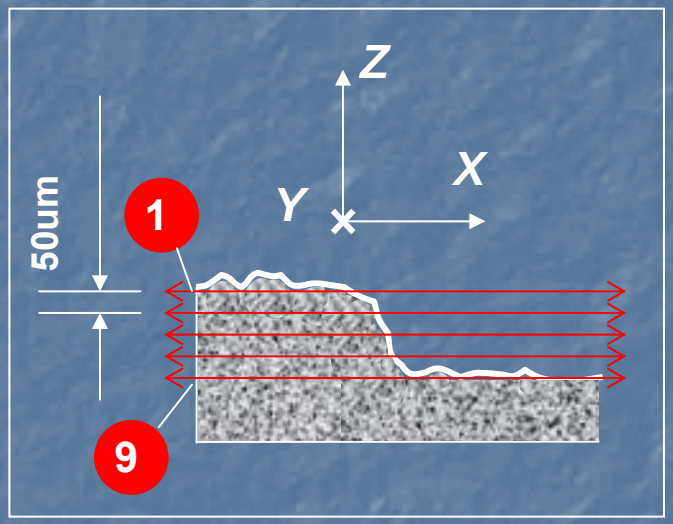
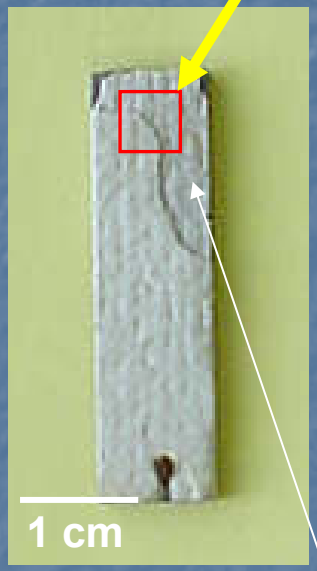
OCT Investigation of Si/Mullite/BSAS EBCs on SiC/SiC Composites

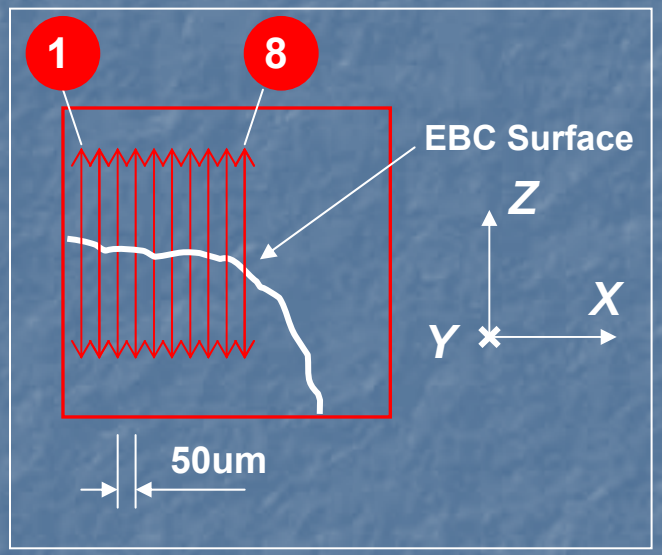
HORIZONTAL CROSS-SECTION ORIENTATION



Scanned area



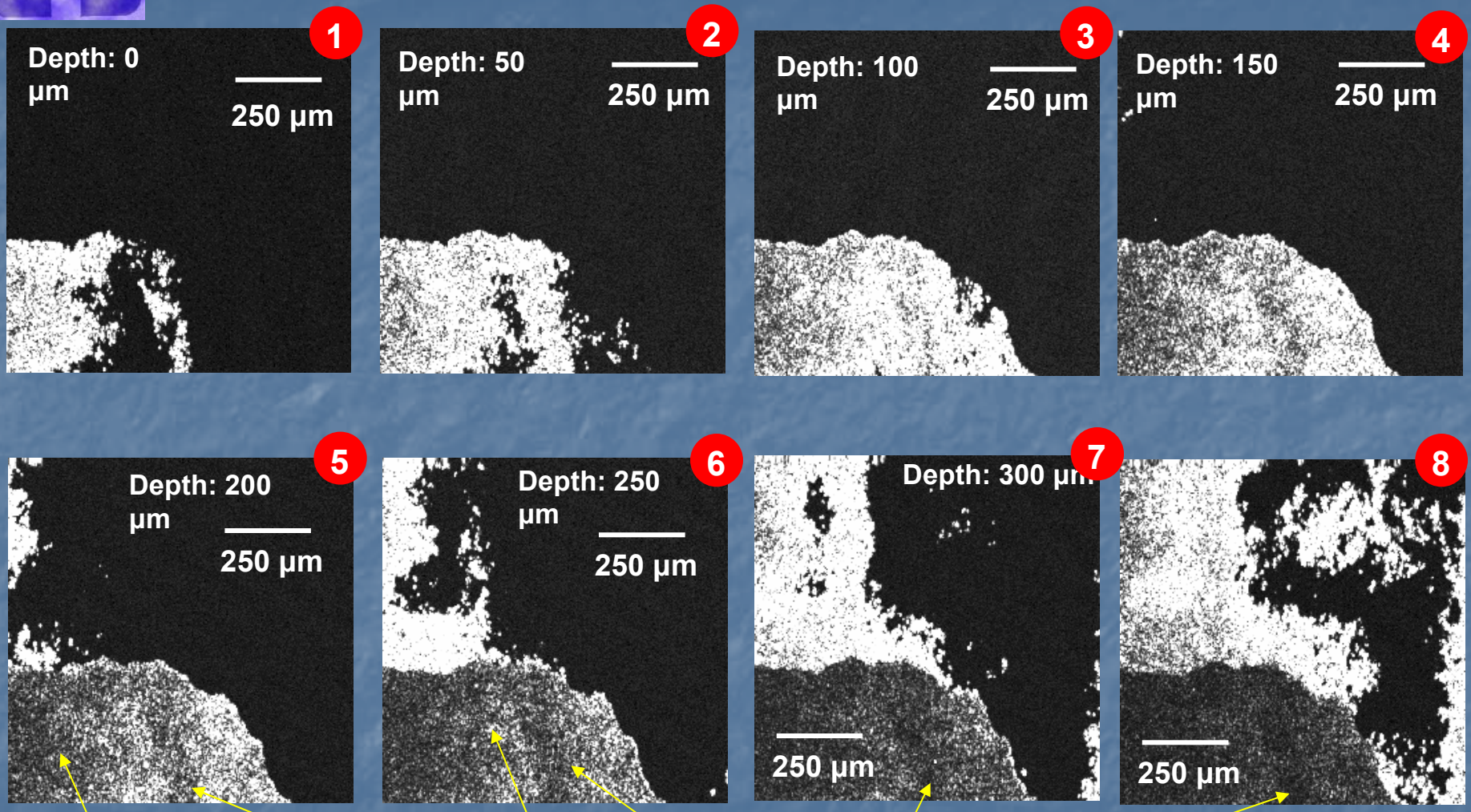
VERTICAL CROSS-SECTION ORIENTATION



EBC Spalled

OCT Investigation of EBCs on Composites

Horizontal Cross-Section Images



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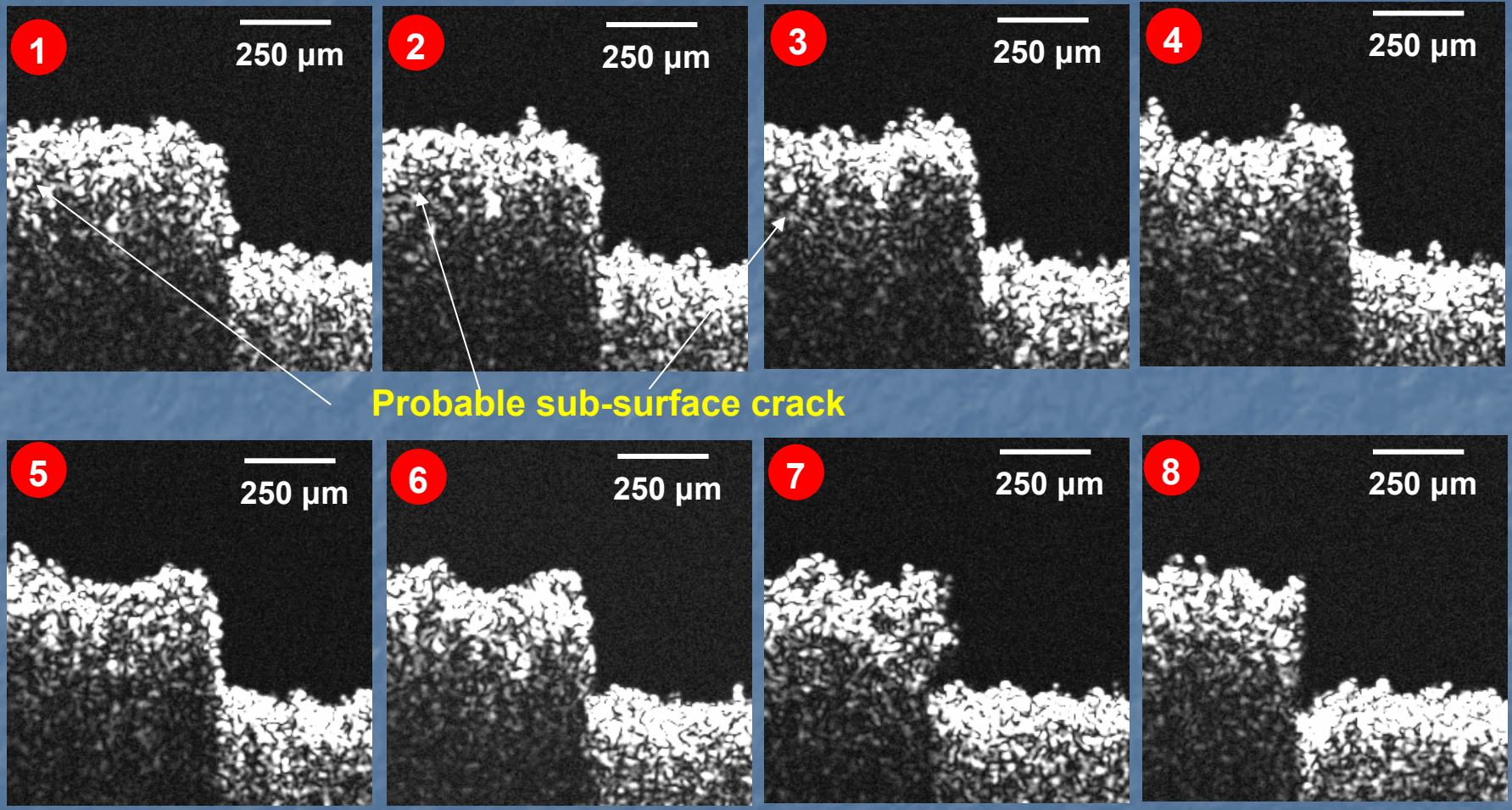
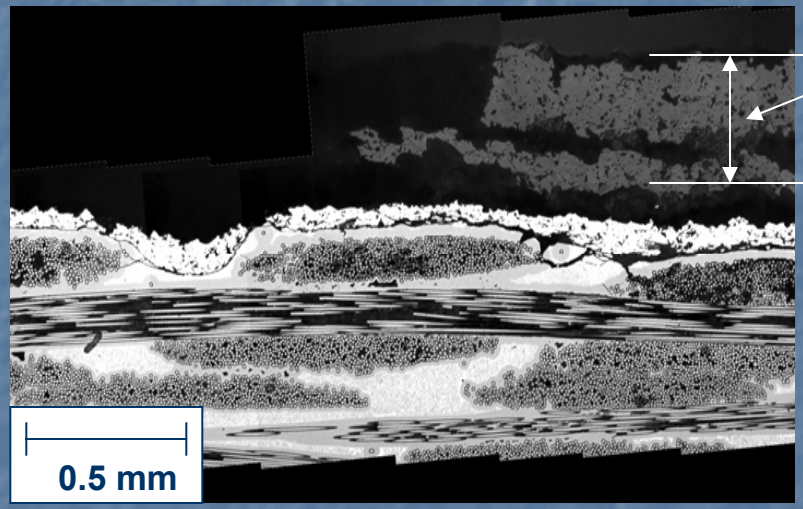
OCT Investigation of EBCs on Composites

Vertical Cross-Section Images



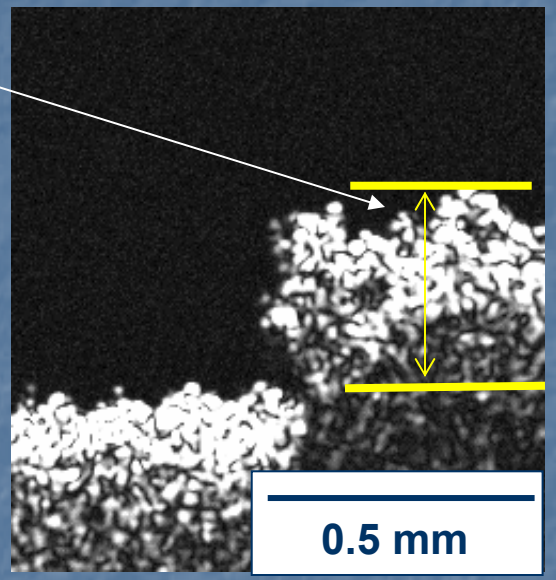
OCT Measurement of EBC Coating Thickness BSAS on MI SiC/SiC

5x Cross Sectional Optical Micrograph



EBC layer

OCT Cross-Sectional Image



EBC Thickness Measurements	
Micrograph	0.29 mm
OCT	0.30 mm

All measurements +/- 10 µm

Cooperative efforts with Ceramatec

TAILORABLE ENVIRONMENTAL BARRIER COATINGS FOR SUPER ALLOY TURBINE ENGINE COMPONENTS IN SYNGAS

P.I. Shekar Balagopal

DOE contract # DE-FG 02-03ER83620

Applications: Graded Ceramic oxide coating on Alloy substrates for protection from corrosion gases up to 1500°C:

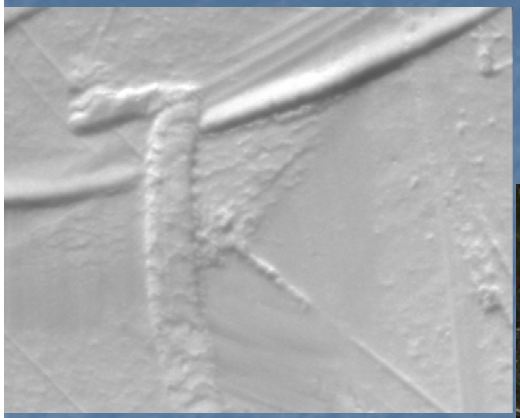
SLURRY CAST OXIDE EBCS
From Ceramatec

- Two as-cast samples deposited on quartz plates
  --One ~40 um thick
  --One ~8-10 um thick

- Two dense samples on quartz plates
  --One ~40 um thick
  --One ~8-10 um thick

Photos of slurry-cast coating in green-state on quartz plate

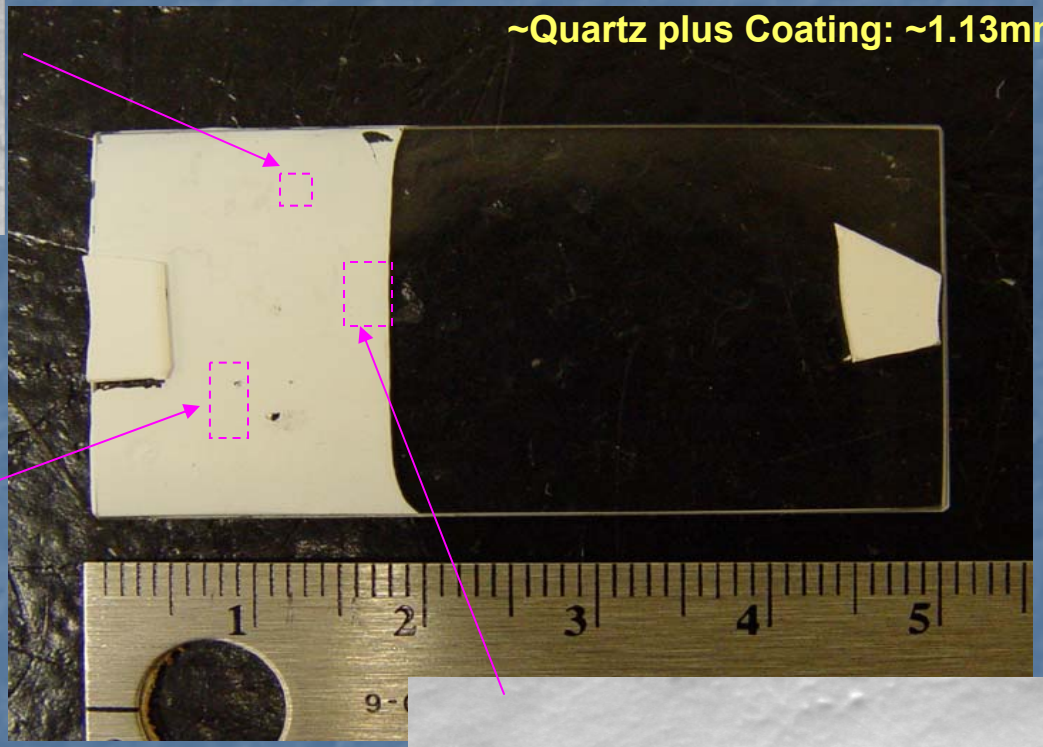
Nonuniform surface

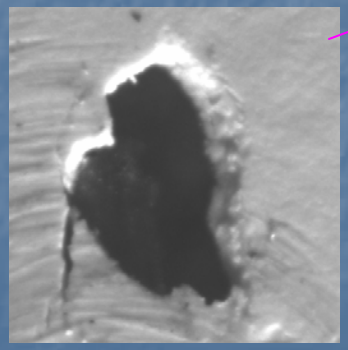


Direct measurements

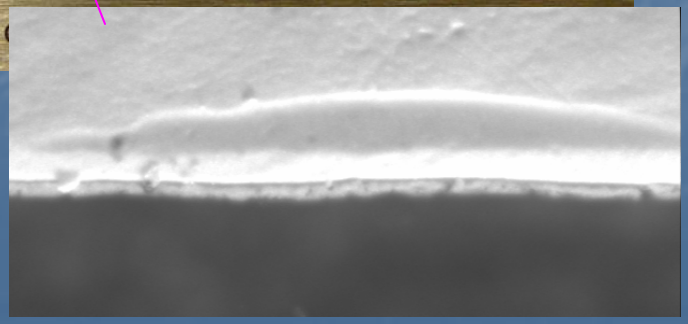
Quartz slide thickness : ~ 1.08mm

~Quartz plus Coating: ~1.13mm



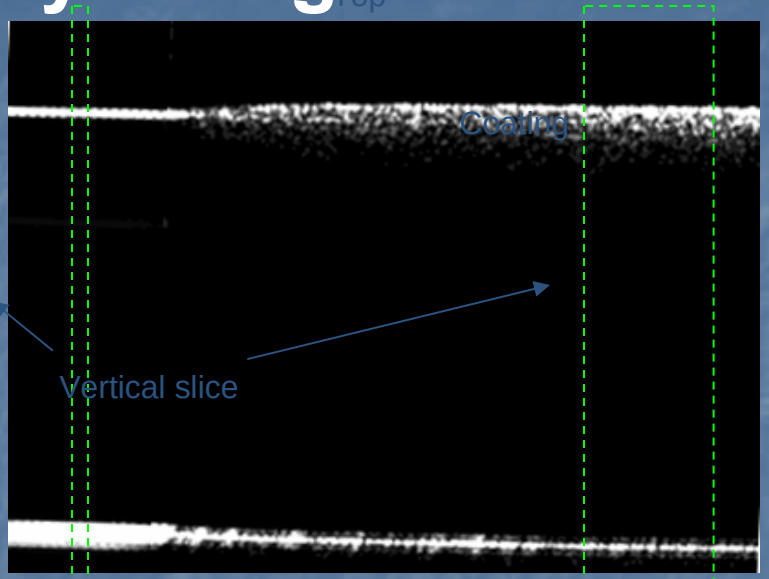


Visible hole



Optical Coherence Tomography data for thick* slurry cast green-state oxide EBC

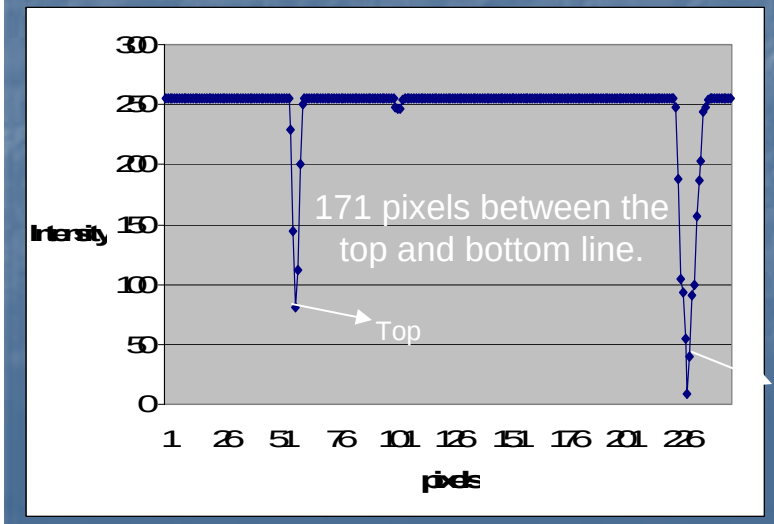
* ~40 um



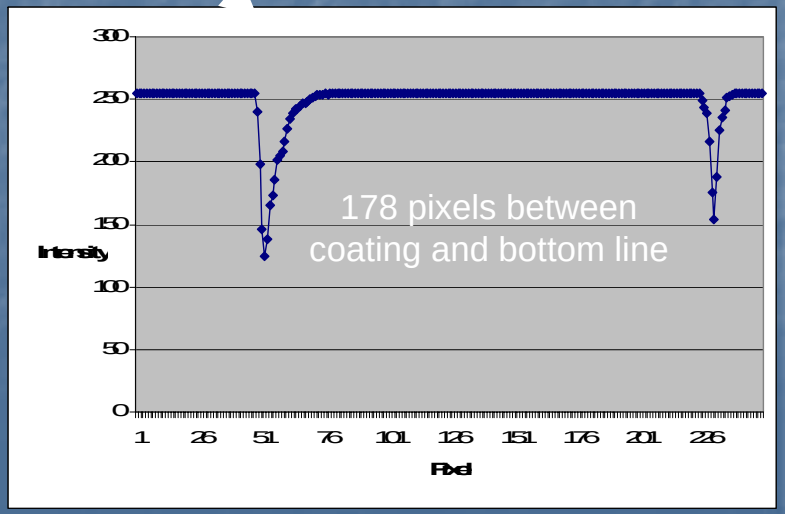
It was suggested that optical band pass for slurry cast would NOT allow He-Ne. OCT uses 1310 nm or near IR

6.3 um/pixel for a scan set at 2.8mm.

7 pixels=~ 42 um



Plot profile of quartz slide.



Plot profile of coating

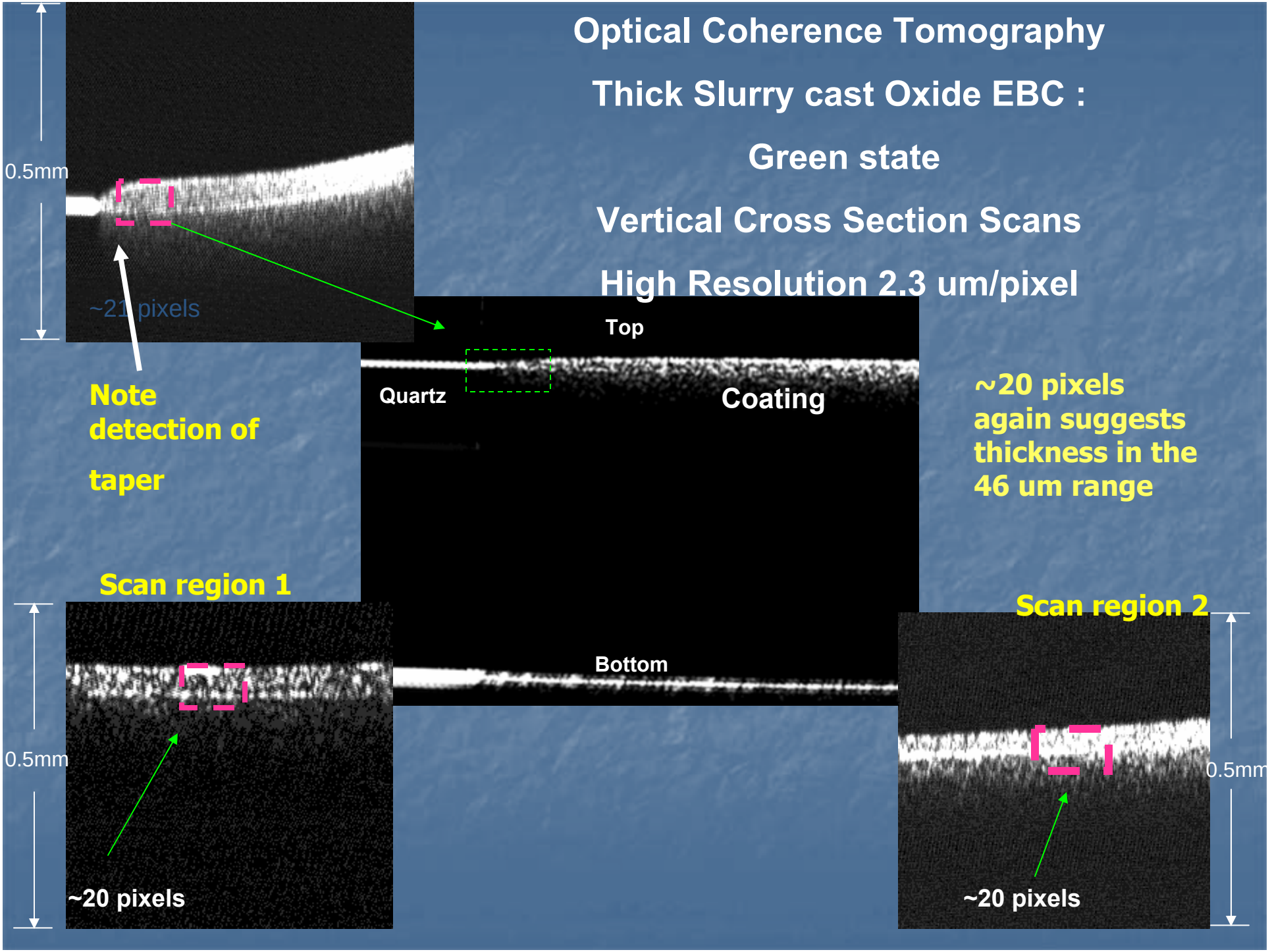
Optical Coherence Tomography

Thick Slurry cast Oxide EBC :

Green state

Vertical Cross Section Scans

High Resolution 2.3 um/pixel

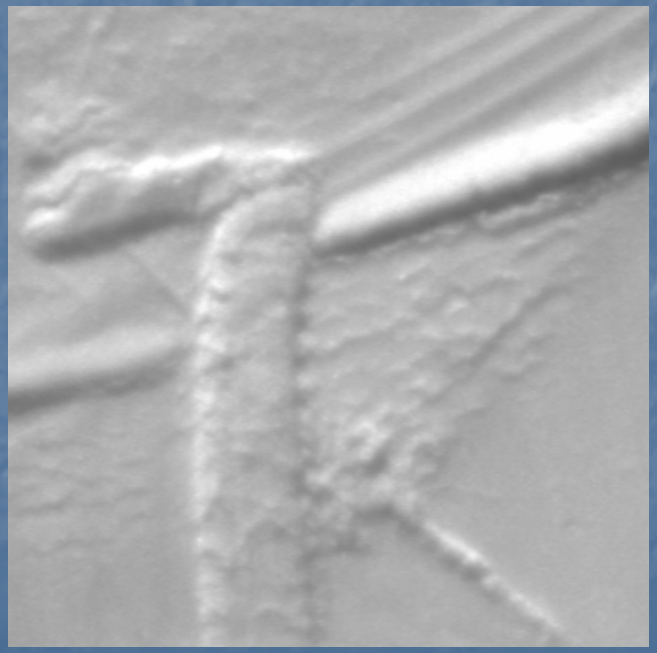


OCT data of Green slurry-cast oxide EBC

Thick EBC sample

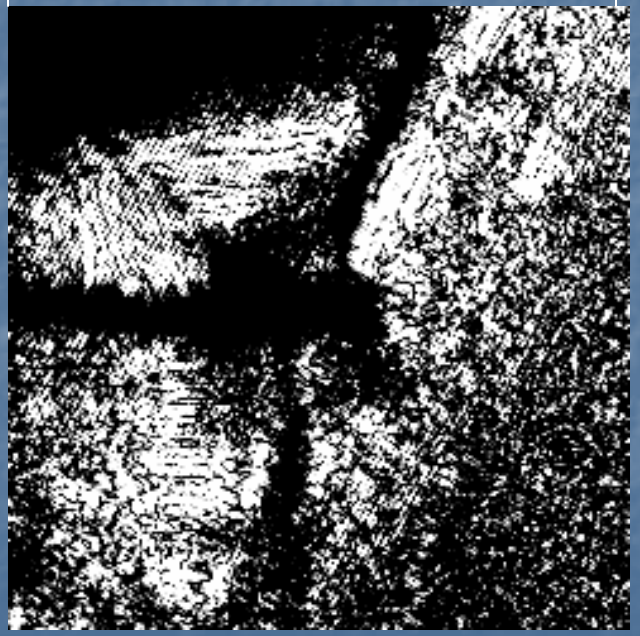
1mm

Microscope Image of surface



OCT Horizontal Image
~25-30 um distance
below surface

1mm

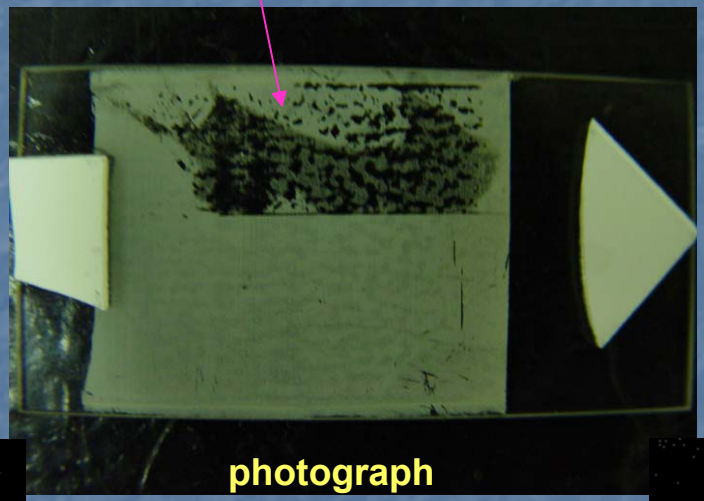


Black region

Suggests

Crack or delaminated region

OCT data for Slurry-Cast green-state thin <10 um sample BC1

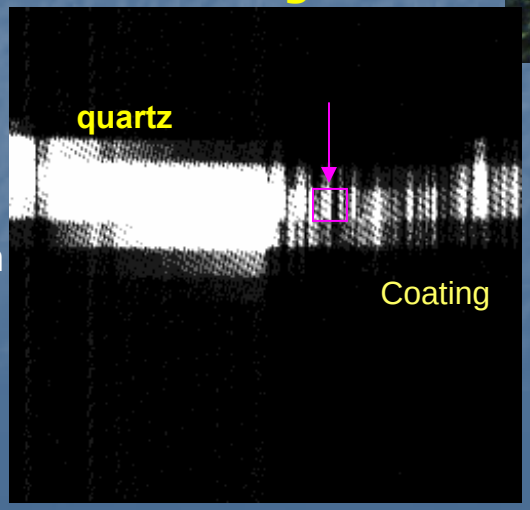


photograph

Vertical OCT scans

2.3 um/pixel

Scan region 1



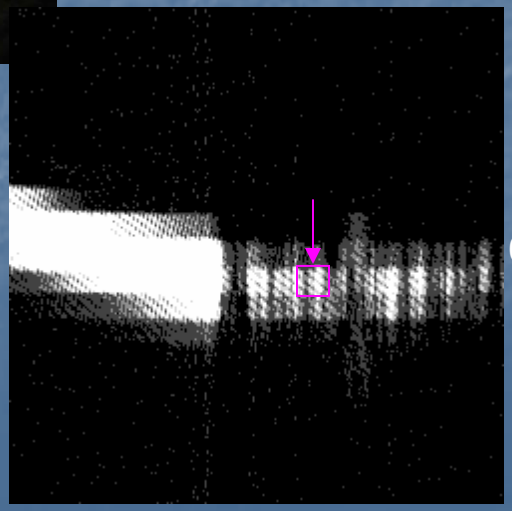
0.1mm

OCT Vertical Scan

Results:

Beyond the present Vertical resolution capability

Scan region 2

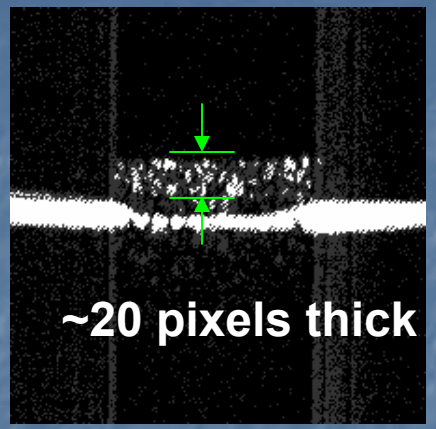


0.1mm

OCT Vertical Scan

OCT data for sintered slurry cast oxide EBC Thick sample (Vertical scans)

The coating on this slide chipped off during transportation, therefore only a piece of the coating was scanned for thickness values.

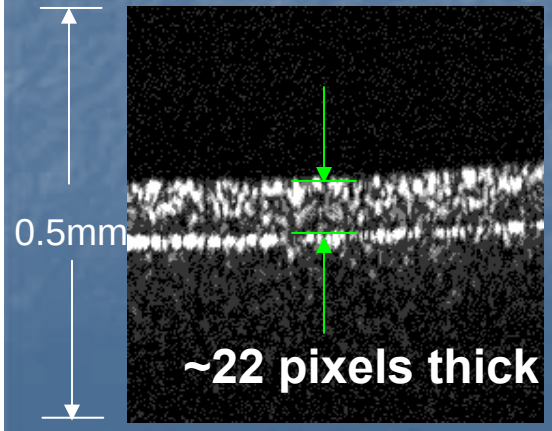


Scan region 2

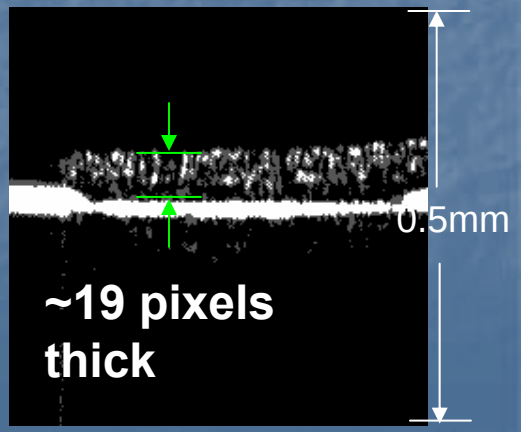
~2.3 um/pixel

Result ~40 um thick

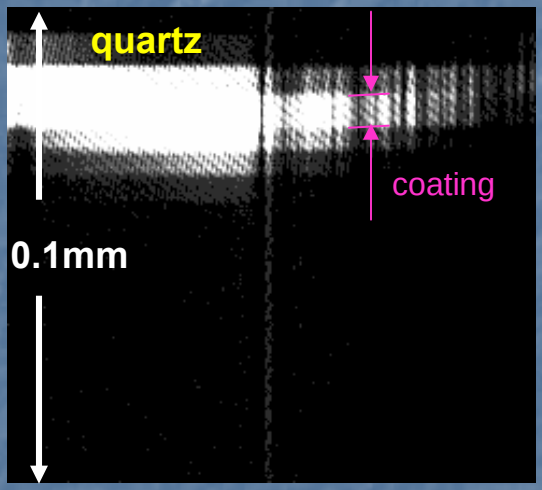
Scan region 1

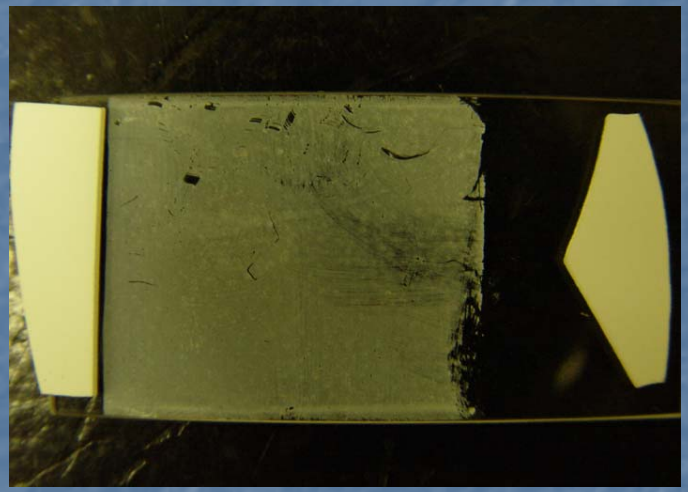


Scan region 3

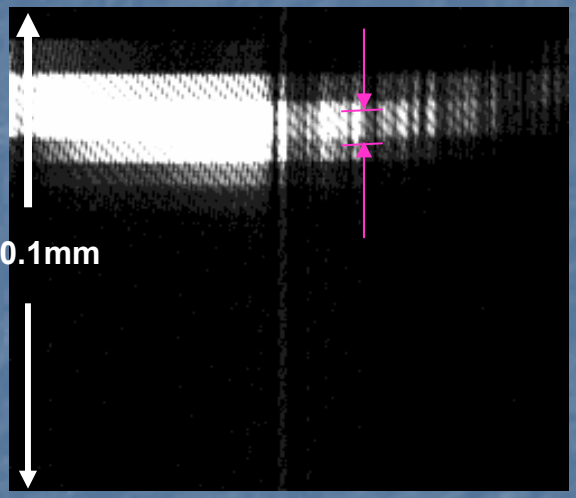


OCT data for sintered slurry-cast oxide EBC Thin sample <10 um





Photograph of sintered thin EBC sample



Results demonstrate that present OCT system cannot achieve this level of spatial resolution

NASA EBCs on MI SiC/SiC

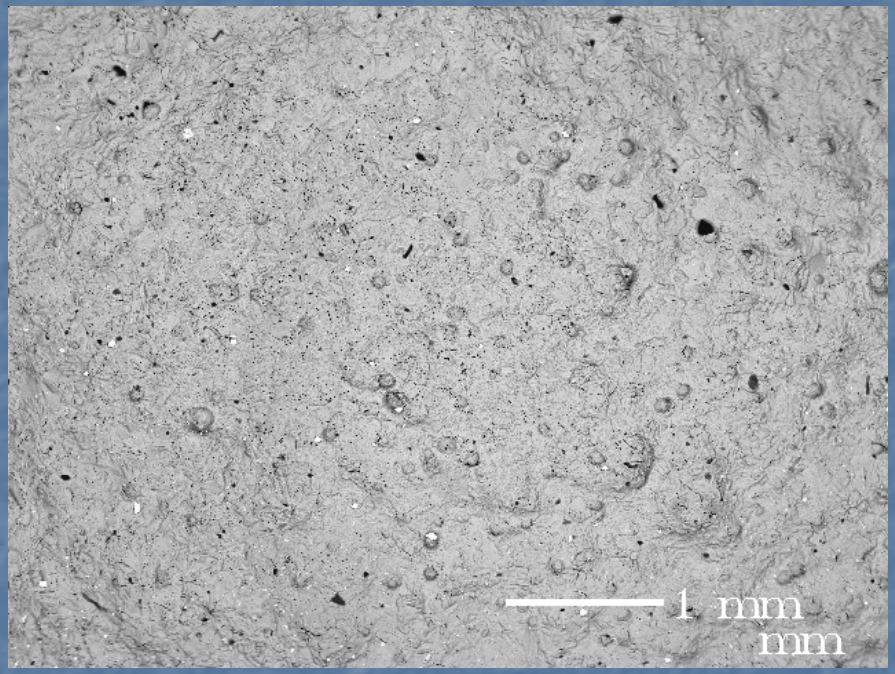
K. Lee*

* (Now at Rolls-Royce)

Si/Mullite/BSAS (5 mil/8 mil/8mil)

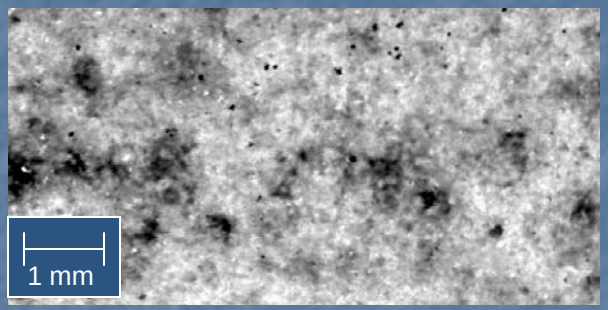
On MI SiC/SiC (sample 1a)

1000 h, 1380C-1h cycles, 90H_2O- Bal O_2

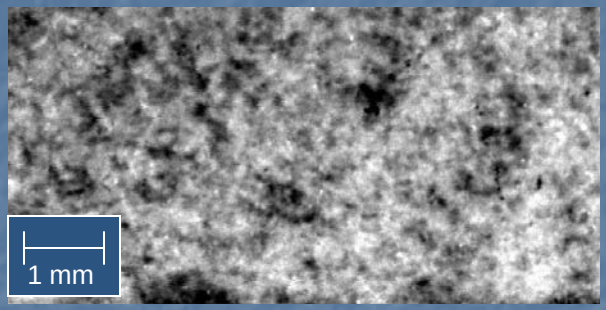


LASER BACKSCATTER INVESTIGATION OF EBCs
EBC: BSAS(8 mil) On MI SiC/SiC

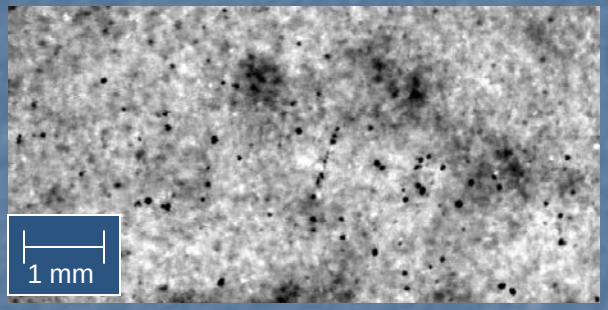
Substrate: MI SiC/SiC(1a)
Si (5 mil)/Mullite(8mil)/BSAS (8 mil)



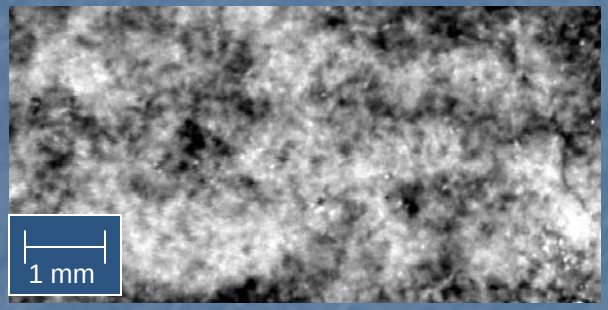
Substrate: MI SiC/SiC(3a)
Si (5 mil)/ Mod Mullite(8mil)/BSAS (8 mil)



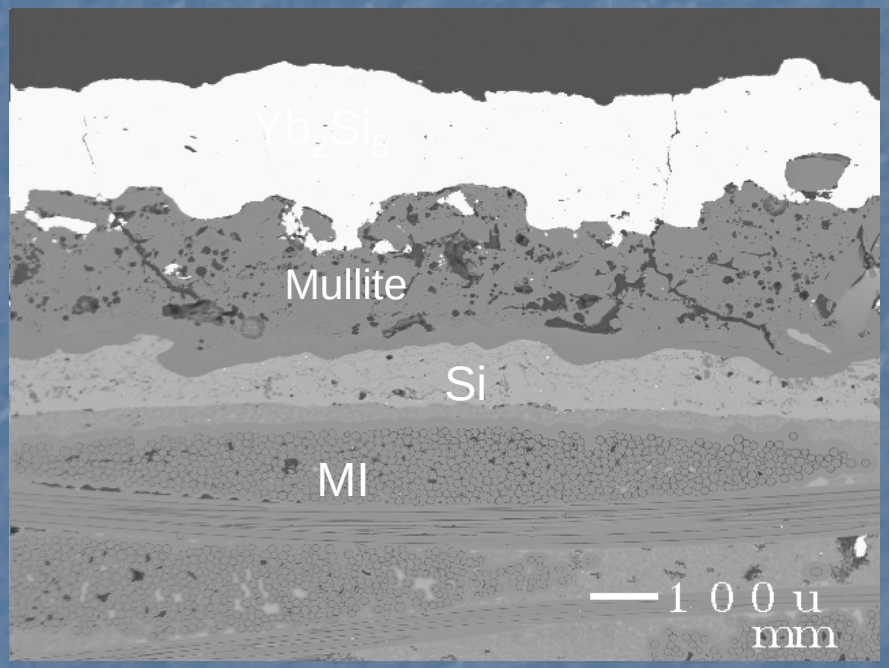
Substrate: MI SiC/SiC(2a)
Si (5 mil)/Mullite + SAS(8mil)/BSAS (8 mil)

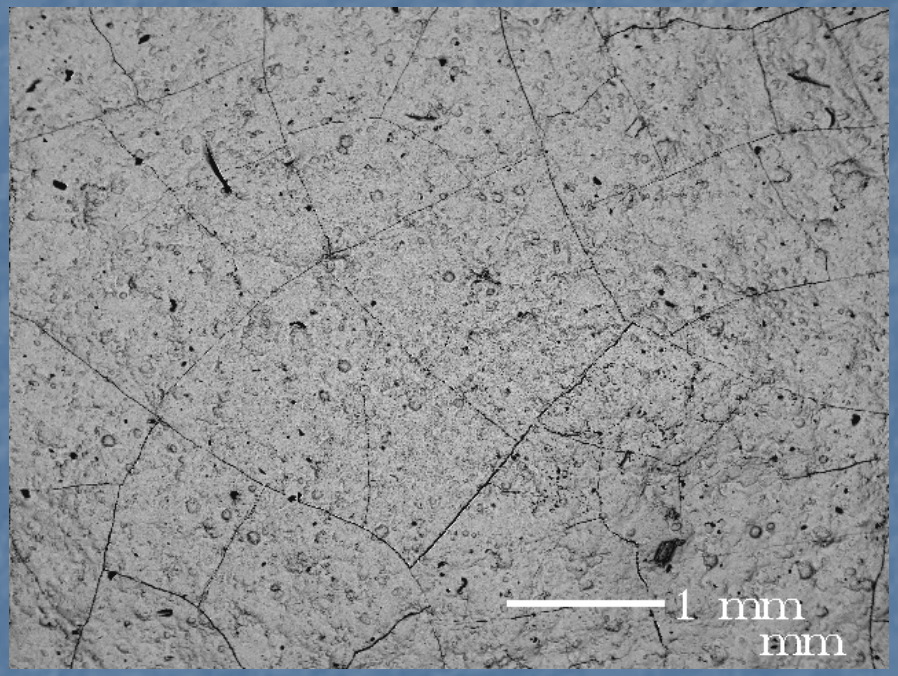


Substrate: MI SiC/SiC(4a)
Si (5 mil)/Mod Mullite + SAS(8mil)/BSAS (8 mil)



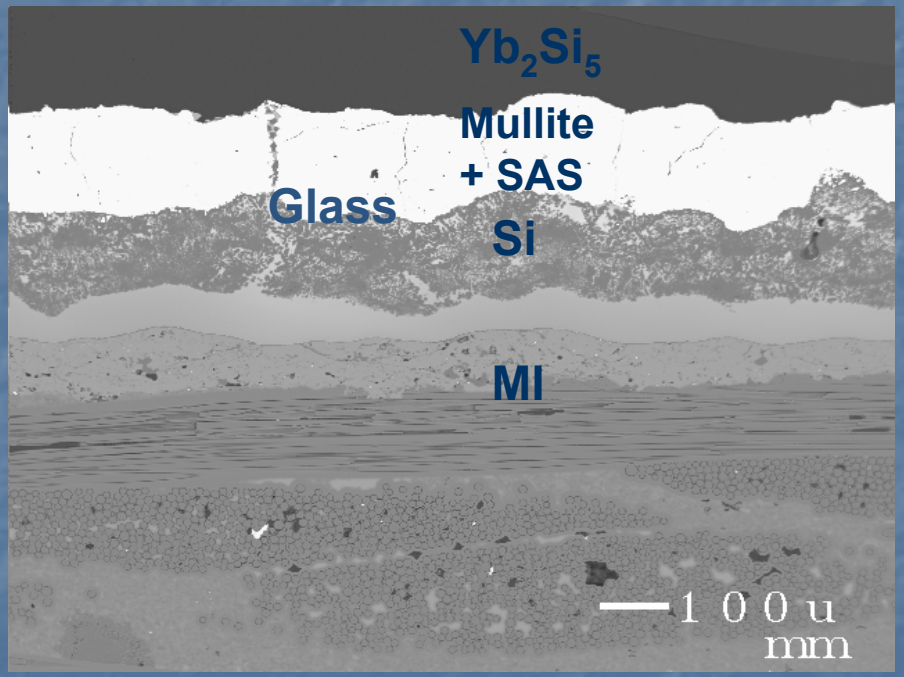
Si/Mullite/Yb_2SiO_5 (5mil/8mil/8mil) On MI SiC/SiC (sample 1b) 1000 h, 1380C-1h cycles, 90H_2O- Bal O_2

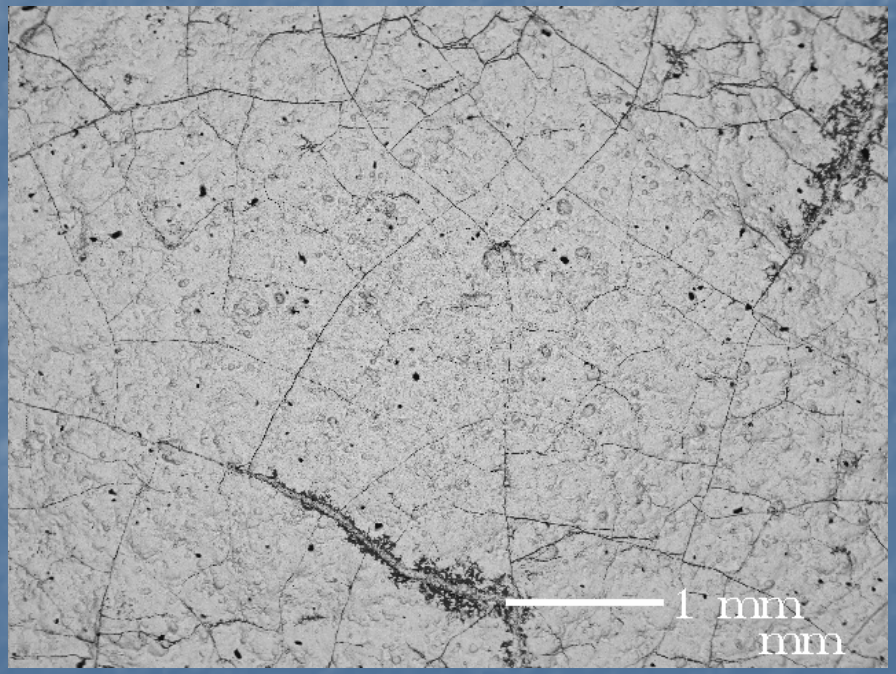




Si/Mullite+SAS/Yb_2SiO(5 mil/8mil/8mil)) On MI SiC/SiC (sample 2b)

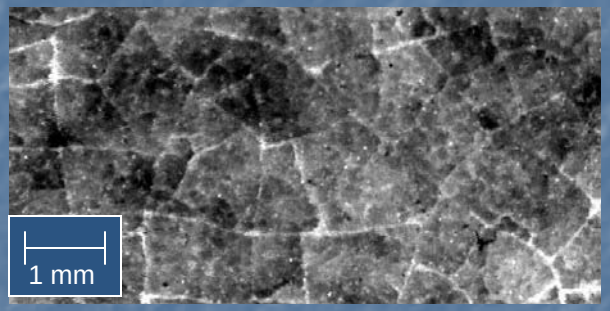
1000 h, 1380C-1h cycles, 90H_2O- Bal O_2



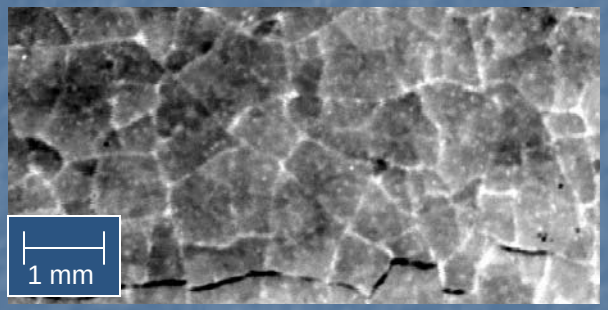


LASER BACKSCATTER INVESTIGATION OF EBCs
EBC: Yb_2SiO_5 (8 mil) On MI SiC/SiC

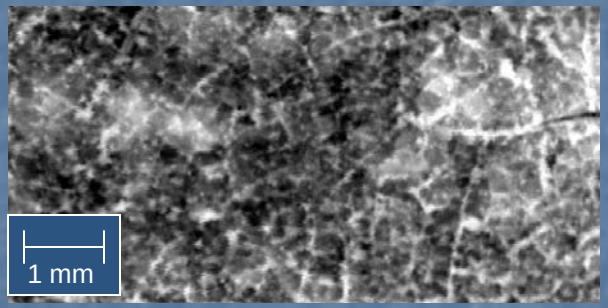
Substrate: MI SiC/SiC(1b)
Si (5 mil)/Mullite(8mil)/ Yb_2SiO_5(8 mil)



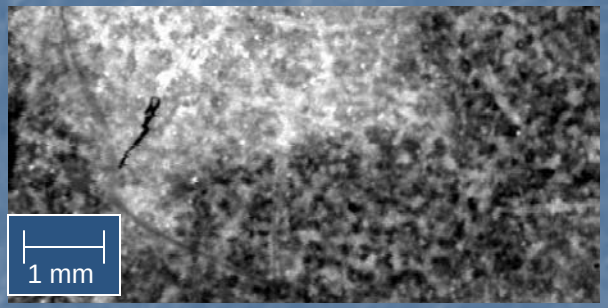
Substrate: MI SiC/SiC(3b)
Si (5 mil)/ Mod Mullite(8mil)/ Yb_2SiO_5 (8 mil)



Substrate: MI SiC/SiC(2b)
Si (5 mil)/Mullite + SAS(8mil) / Yb_2SiO_5(8 mil)



Substrate: MI SiC/SiC(4b)
Si (5 mil)/Mod Mullite + SAS(8mil)/Yb_2SiO_5 (8 mil)



SUMMARY/CONCLUSIONS

- Two optical NDE methods, OCT and EOS, have been demonstrated to detect features/characteristics of interest for EBCs
  - -mapping thickness variations
  - -Detecting delaminations
- Use of OCT has been demonstrated for application to as-deposited (green-state) slurry-cast EBCs for thickness mapping
- EOS data have been shown to correlate with cracking observed for various EBCs deposited on CMCs

- Higher spatial resolution of OCT can be obtained by installing new femtosecond lasers with greater optical band width

- While correlations between TBC spallation and laser scatter data have been demonstrated , this has NOT been demonstrated to-date for EBCs on monolithic substrates.