DEVELOPMENT OF NDE METHODS FOR CERAMIC COATINGS AND MEMBRANES

J. G. Sun

Nuclear Engineering Division
Argonne National Laboratory
Argonne, IL 60439

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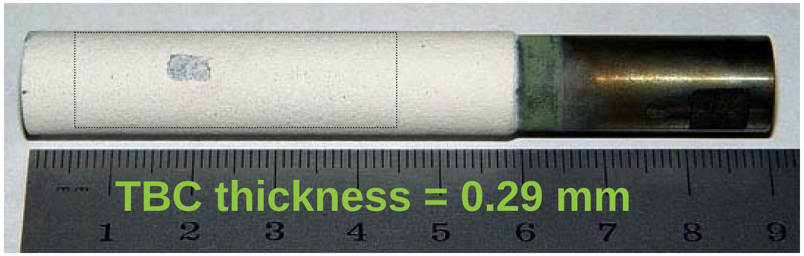
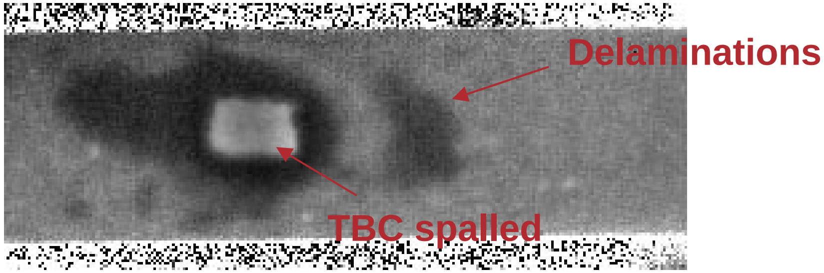
Outline

- Background
- Thermal imaging methods for TBCs
  - Multilayer thermal modeling method
    - *Determine TBC thermal properties (e.g., conductivity)*
  - Thermal tomography
- X-ray microCT for ceramic gas-separation membranes
- Summary
- Planned future efforts

Background

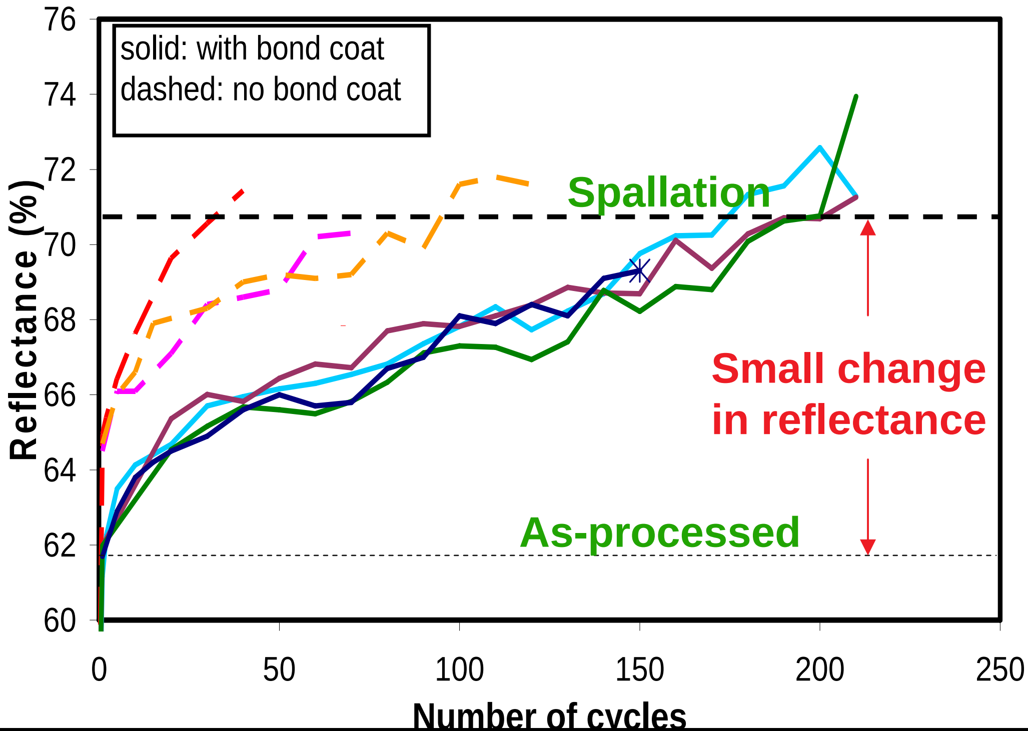
- Thermal barrier coatings (TBCs) are “prime reliant” material for turbine engine components – evaluation of their conditions by NDE is important
- NDEs may be used to:
  - Assess TBC process reliability and product quality (specs, defects, etc)
    - *Current TBC processing relies on robust process conditions*
  - Monitor TBC degradation and predict TBC lifetime
    - *As-processed TBC → TBC sintering → TBC degradation (internal cracking) → TBC delamination → TBC spallation (failure)*
  - Detect FOD (foreign object damage)
- NDE detection of delamination is relatively easy: thermal imaging, optical, etc





Background - continued

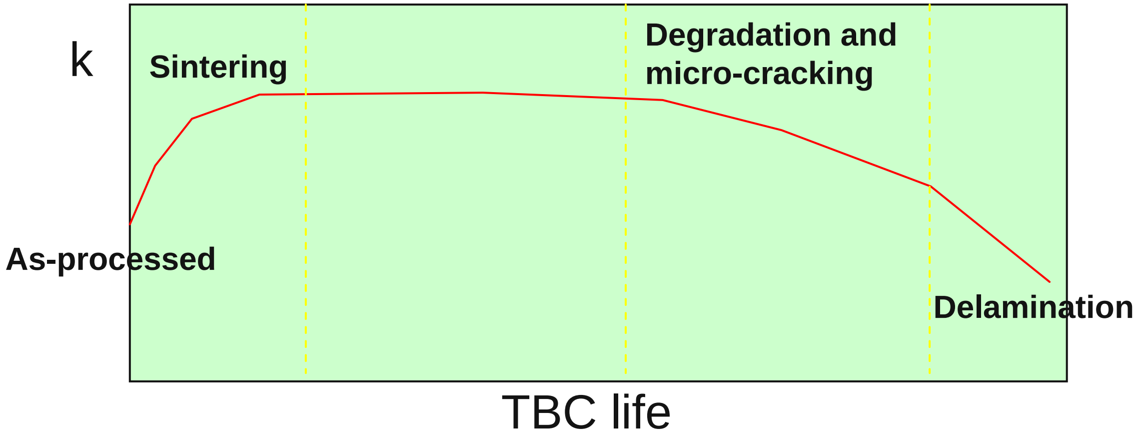
- Few NDEs were reported to monitor TBC condition and predict TBC lifetime
  - Mid-IR reflectance (J. Eldridge of NASA)
    - *Reflectance increases with damage*
  - Electrical impedance spectroscopy (P. Xiao of Univ. Manchester)
    - *Resistance (cracking), modulus (TGO growth), capacitance (delamination)*
- Current methods are not quantitative and difficult to be generalized!



From: J. Eldridge and C. Spuckler, *Int. J. Appl. Ceram. Technol.*, Vol. 3, pp. 94-104, 2006

A Quantitative Approach for TBC Lifetime Prediction

- Based on NDE measurement of TBC conductivity k
  - TBC conductivity is the most important thermophysical parameter
    - *Measured for all coatings*
    - *Used in component design*
  - TBC conductivity evolution has characteristic features (many studies)
    - *As-processed TBC: baseline conductivity*
    - *→ TBC sintering: conductivity increase*
    - *→ TBC degradation (internal cracking): conductivity decrease*
    - *→ TBC delamination: significant conductivity drop*
    - *→ TBC spallation (failure)*



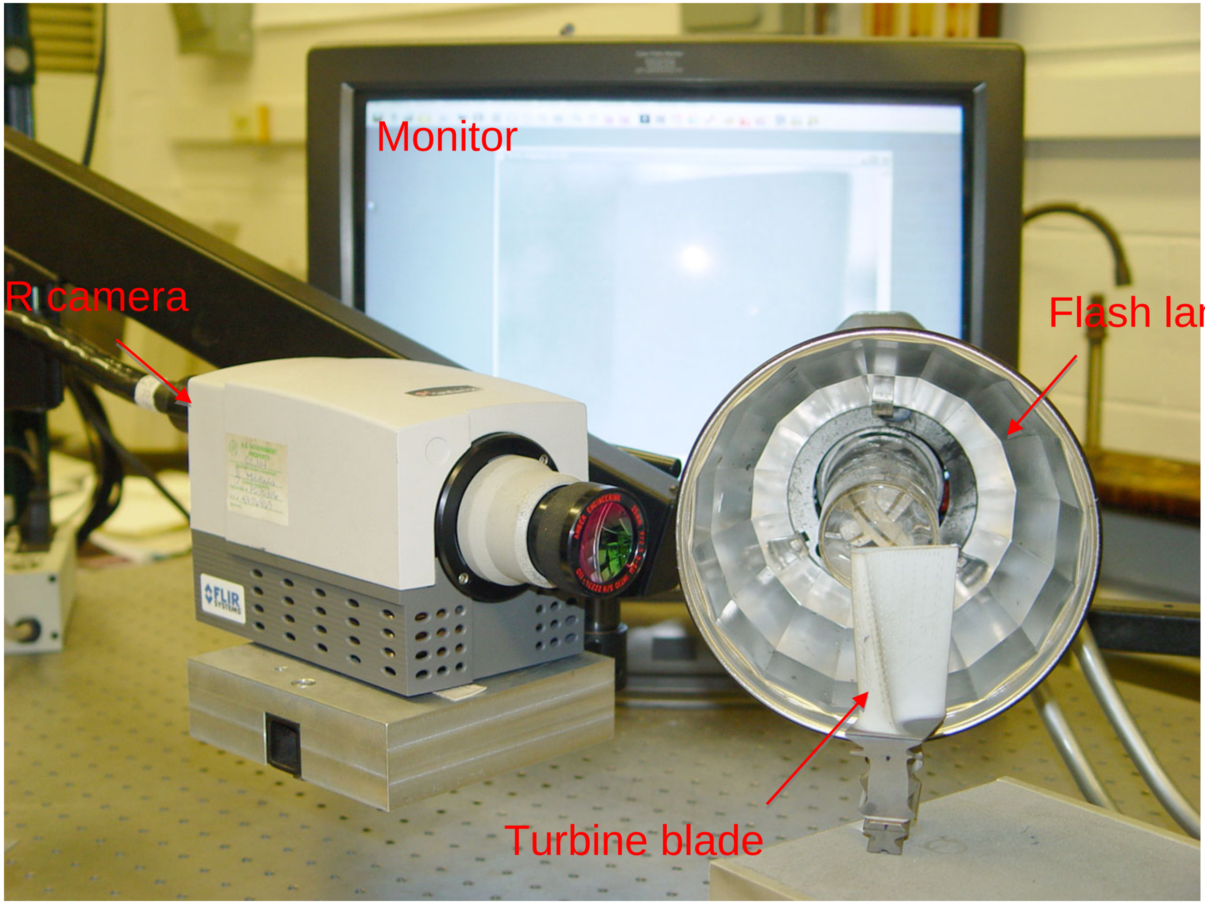
NDE Development for TBCs

- NDE development at ANL is focused on thermal imaging methods
  - For quantitative measurement of TBC thermal properties, e.g., conductivity
    - *TBC conductivity is measured mostly by laser flash method which is a two-sided thermal method and not suitable for NDE of real component*
  - For evaluation of TBC degradation and prediction of TBC lifetime
  - Applicable to TBCs of all types (APS or EBPVD) and conditions (e.g., thickness)

- Multilayer thermal modeling method (2D imaging):
  - TBC thermal conductivity and thickness distribution
    - ***Accuracy is most important!***
  - TBC cracking and delamination

- Thermal tomography method:
  - 3D imaging of TBC structure and property distribution
  - Determination of TBC thickness and damage depth

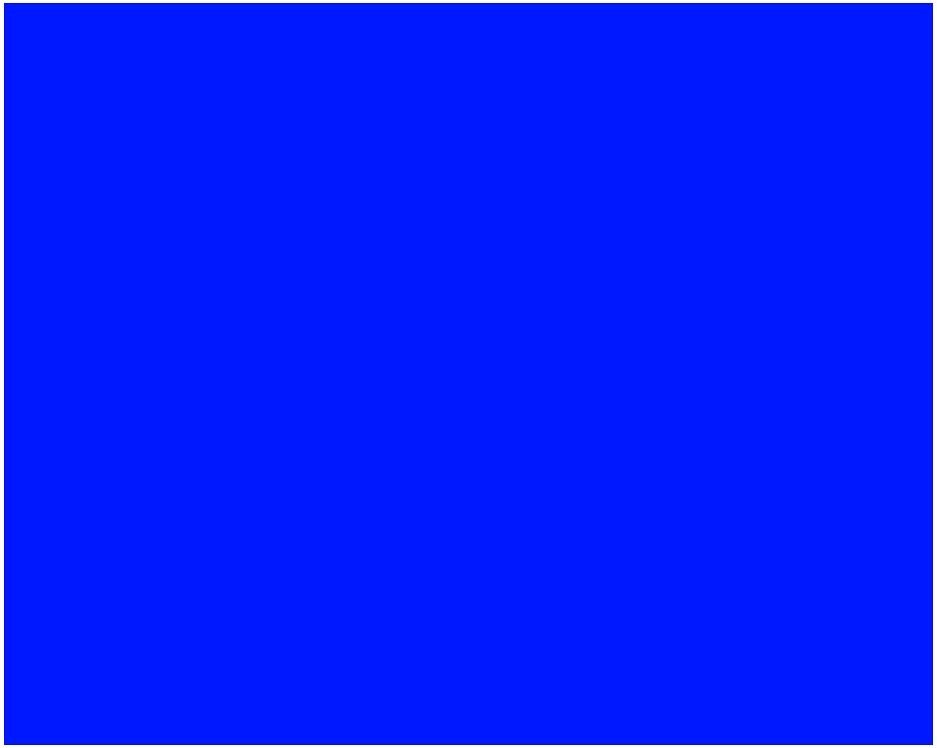
One-Sided Flash Thermal Imaging Setup for Testing of a TBC Coated Turbine Blade



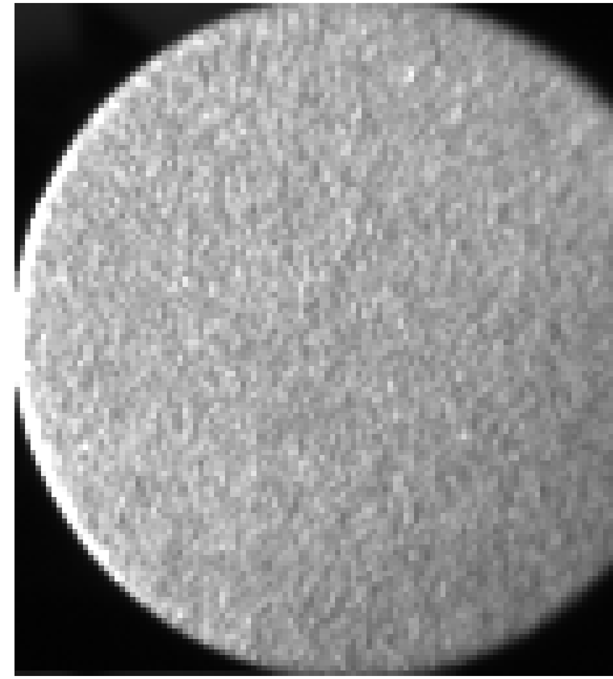
- Image entire surface (100% inspection)
- Fast (a few seconds for testing, up to a few minutes for data processing)
- Data processing is completely automated (no operator adjustment)

Typical Raw Thermal Imaging Data

Flash lamp
at left side



A typical image

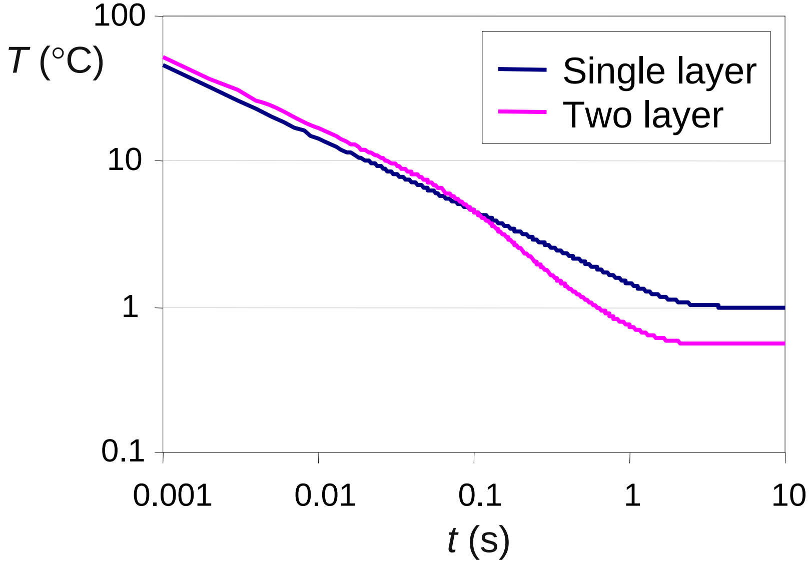


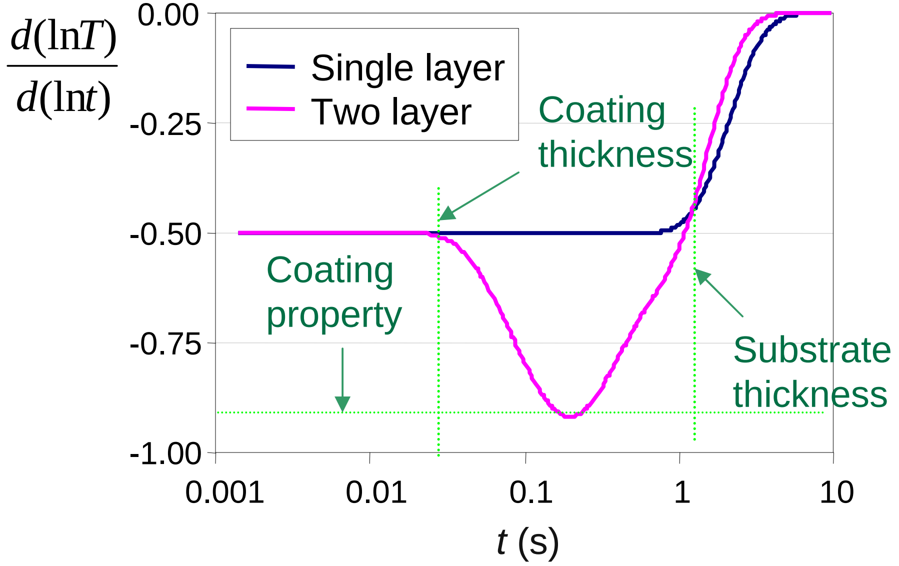
Low temperature  High temperature

- Total time period is ~0.1 s
- APS TBC of 1” diameter

Characteristics of Thermal Imaging Data (1- and 2-layer)

Typical surface temperature and its slope at a single pixel





- Thermal imaging data, i.e., surface temperature and its slope at each surface pixel, are significantly different for 1- and 2-layer materials
- Characteristics in thermal data allow for direct calculation of coating thickness and thermal properties, as well as substrate thickness
- High detection sensitivity for TBC due to large thermal-property disparity between coating and substrate

Recent Thermal Imaging NDE Development for TBCs

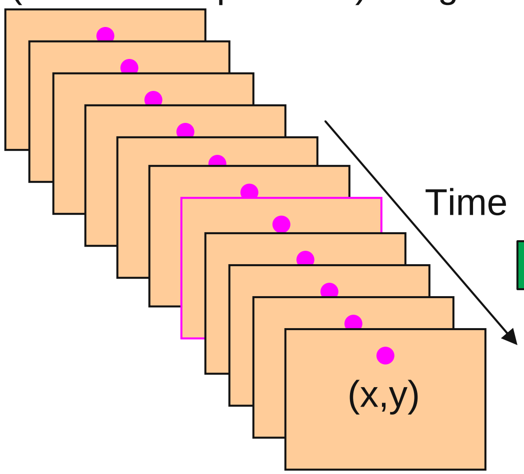
- 2D multilayer thermal modeling method
  - Two issues identified last year were resolved:
    - *(1) nonlinear temperature reading*
    - *(2) two independent parameters in solution*
  - Automated data-processing software was established
    - *Speed was not optimized*
    - *Coating optical property was not yet accounted for*
  - Calibration tests are underway for both APS and EBPVD TBCs

- 3D Thermal tomography method
  - Data-processing software was improved

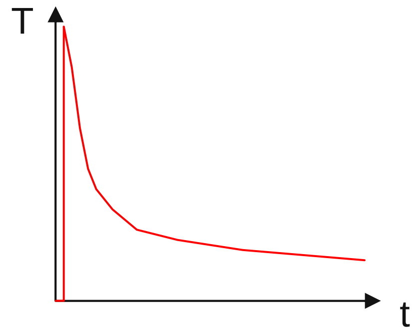
2D Multilayer Thermal Modeling for TBC Systems

Measured data T(x,y,t):

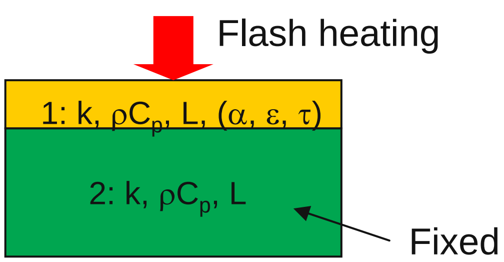
Time series of 2D thermal (surface temperature) images



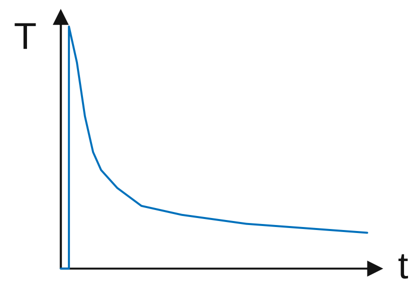
Surface temperature variation at pixel (x,y)



Multilayer TBC model



Numerical solution of surface temperature variation



- Fitting numerical result with exp. data to derive correct TBC parameters:

  Coating 1:
  k, (ρC_p,) L, (α, ε, τ)

  Substrate 2:
  fixed properties

- Repeat this process for every pixel

Formulation for Multilayer Thermal Modeling Method

- Governing heat transfer equation (1D):

$$\rho c \frac{\partial T}{\partial t} = k \frac{\partial^2 T}{\partial z^2}$$

- Analytical solution (2 layers, no interface resistance):

$$T(t) = T_\infty \left[1 + 2 \frac{x_1 \omega_1 + x_2 \omega_2}{x_1 + x_2} \sum_{K=1}^{\infty} \frac{x_1 \cos(\omega_1 \gamma_K) + x_2 \cos(\omega_2 \gamma_K)}{x_1 \omega_1 \cos(\omega_1 \gamma_K) + x_2 \omega_2 \cos(\omega_2 \gamma_K)} \exp\left(-\frac{\gamma_K^2 t}{\eta_2^2} \right) \right]$$

where γ_K is the *K*-th positive root of the following equation,

$$x_1 \sin(\omega_1 \gamma) + x_2 \sin(\omega_2 \gamma) = 0$$

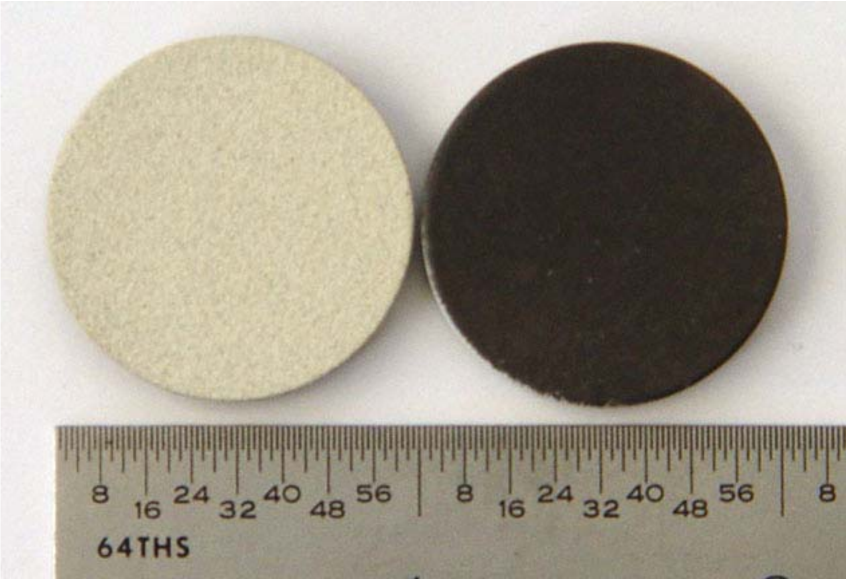
and $x_i = e_{12} - (-1)^i \qquad e_i = \sqrt{k_i \rho_i c_i} \qquad i = 1, 2$

$$\omega_i = \eta_{12} - (-1)^i \qquad \eta_i = L_i / \sqrt{\alpha_i} \qquad i = 1, 2$$

$$e_{12} = e_1 / e_2 \qquad \eta_{12} = \eta_1 / \eta_2$$

- Two independent parameters for coating: *e* (effusivity) and $\eta = L/\alpha^{1/2}$, which can derive coating conductivity *k* and heat capacity ρc (or thickness *L*)

TBC Sample Preparation for Thermal Imaging Test

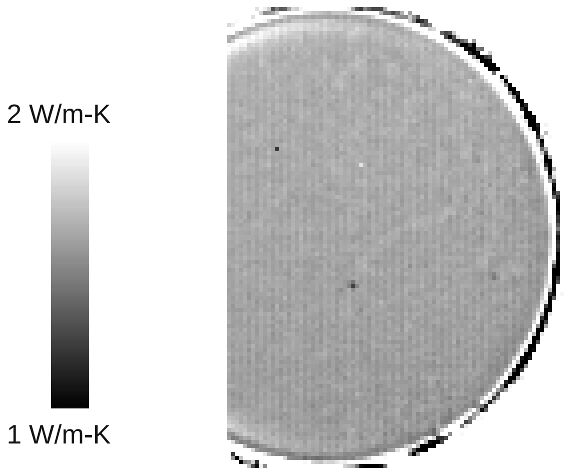


- For thermal imaging, TBC surface needs to be coated black
  - Same requirement as for laser flash, the current standard method for TBC conductivity measurement
  - This requirement can be removed if an optical model for TBC is developed

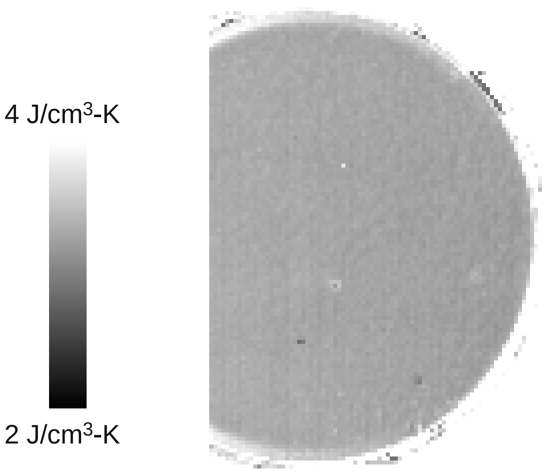
- Tested EBPVD TBC sample (black coated)
  - 1” diameter, coating thickness = 0.127mm
  - Sample curtsey of Dr. A. Feuerstein, Praxair Surface Technologies, Inc.
  - Image acquisition rate was at 1068 frames/second

Multilayer Modeling Prediction for TBC Thermal Properties

TBC conductivity k
(W/m-K)

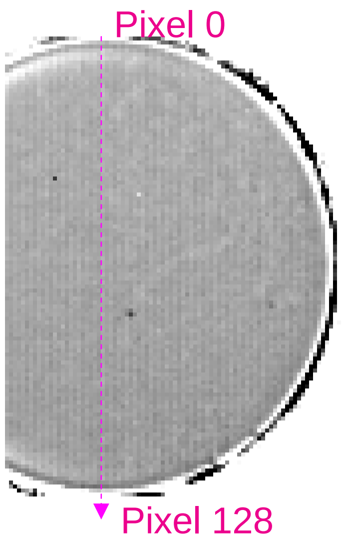


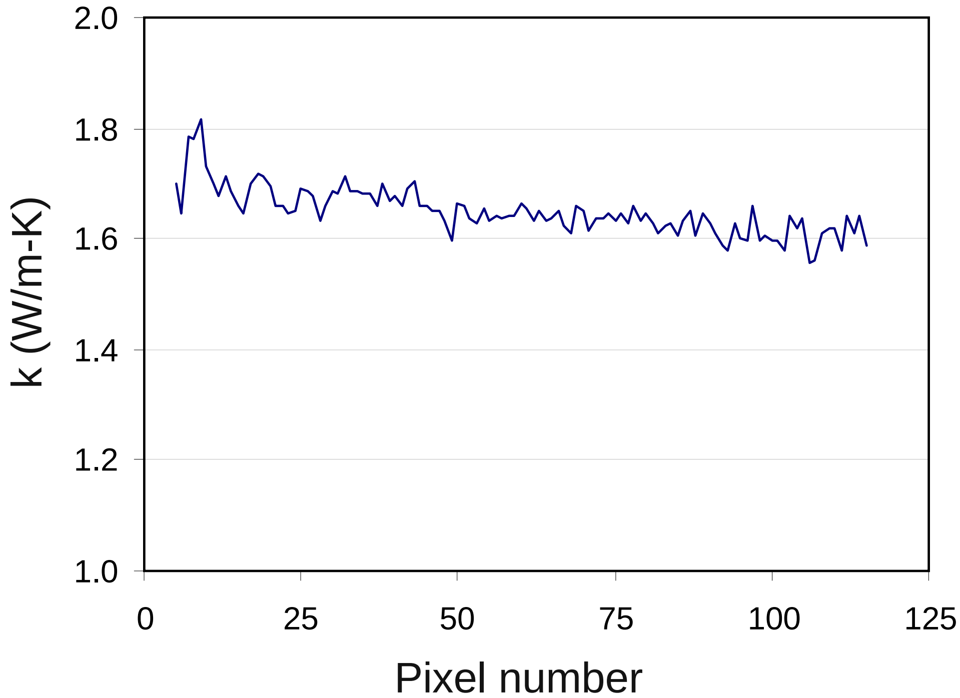
TBC heat capacity ρC_p
(J/cm^3-K)



- Average TBC conductivity: 1.65 W/m-K (1.71)
- Average TBC heat capacity: 3.3 J/cm^3-K (high)
  - Higher prediction in heat capacity data is likely due to uncertainty in TBC thickness and other parameters discussed later

Typical Line Plot of Predicted TBC Thermal Conductivity

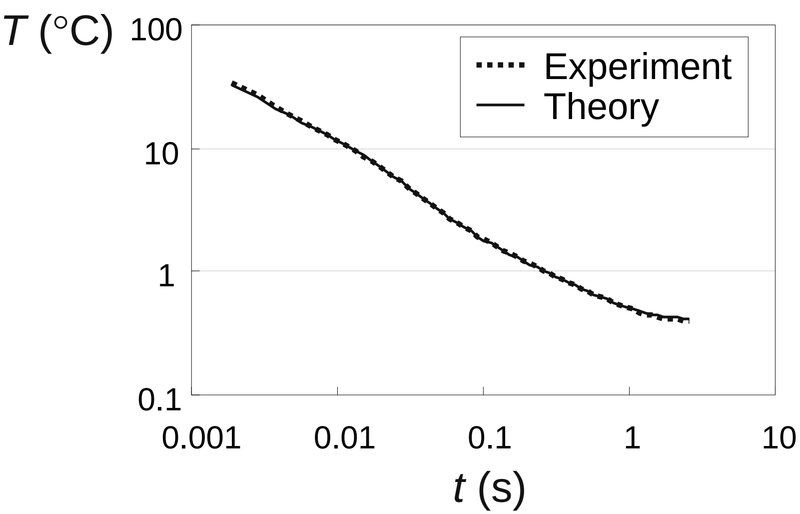




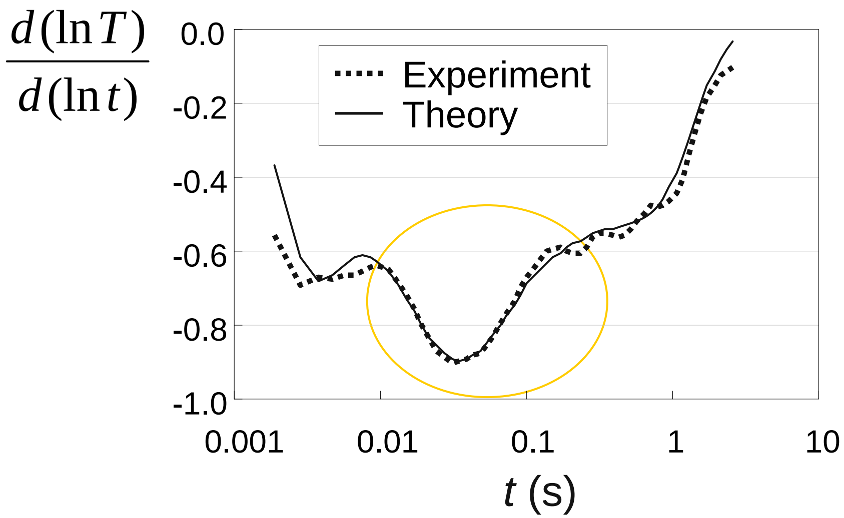
- Gross conductivity change is likely due to thickness variation

Typical Fitting Results for One Pixel

Temperature vs. time



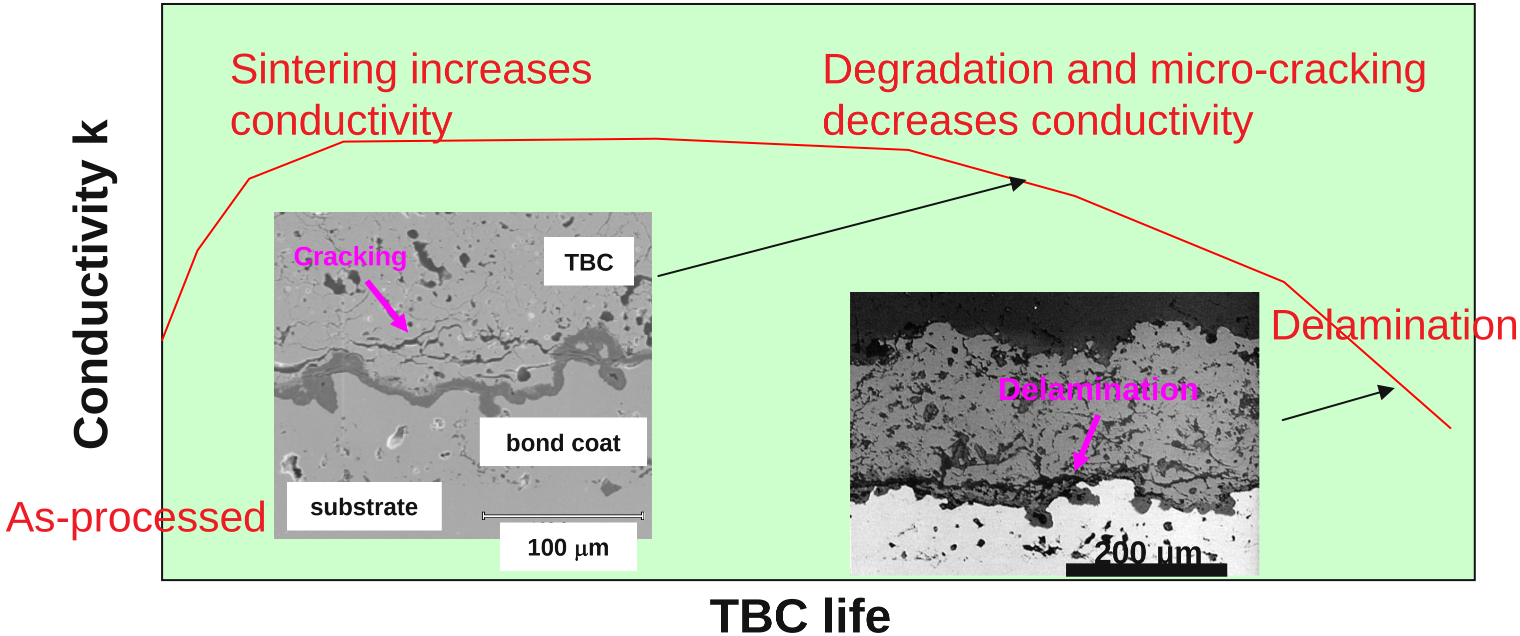
Temperature slope vs. time



- **Good fitting agreement!**

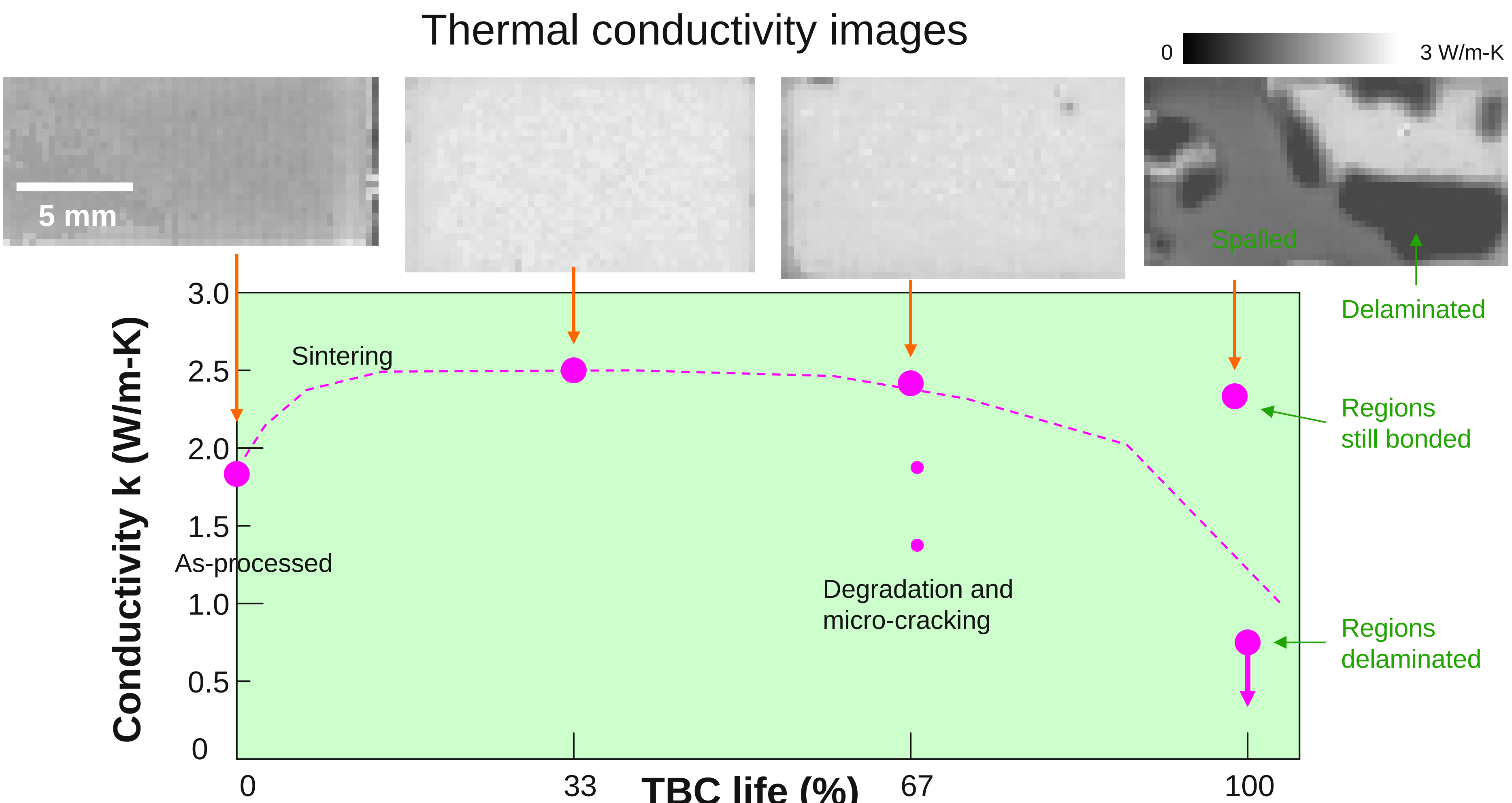
Thermal Conductivity Based TBC Lifetime Prediction

- Initial TBC conductivity (baseline)
- Conductivity increase in early life – due to sintering
  - Many studies (NASA, SUNY, DLR, etc); e.g., the increase may be modeled by a Larson-Miller parameter (LMP)
- Conductivity decrease in mid-to-late TBC life – due to micro-cracking
  - NASA, etc
- Significant conductivity decrease in late TBC life – due to delamination

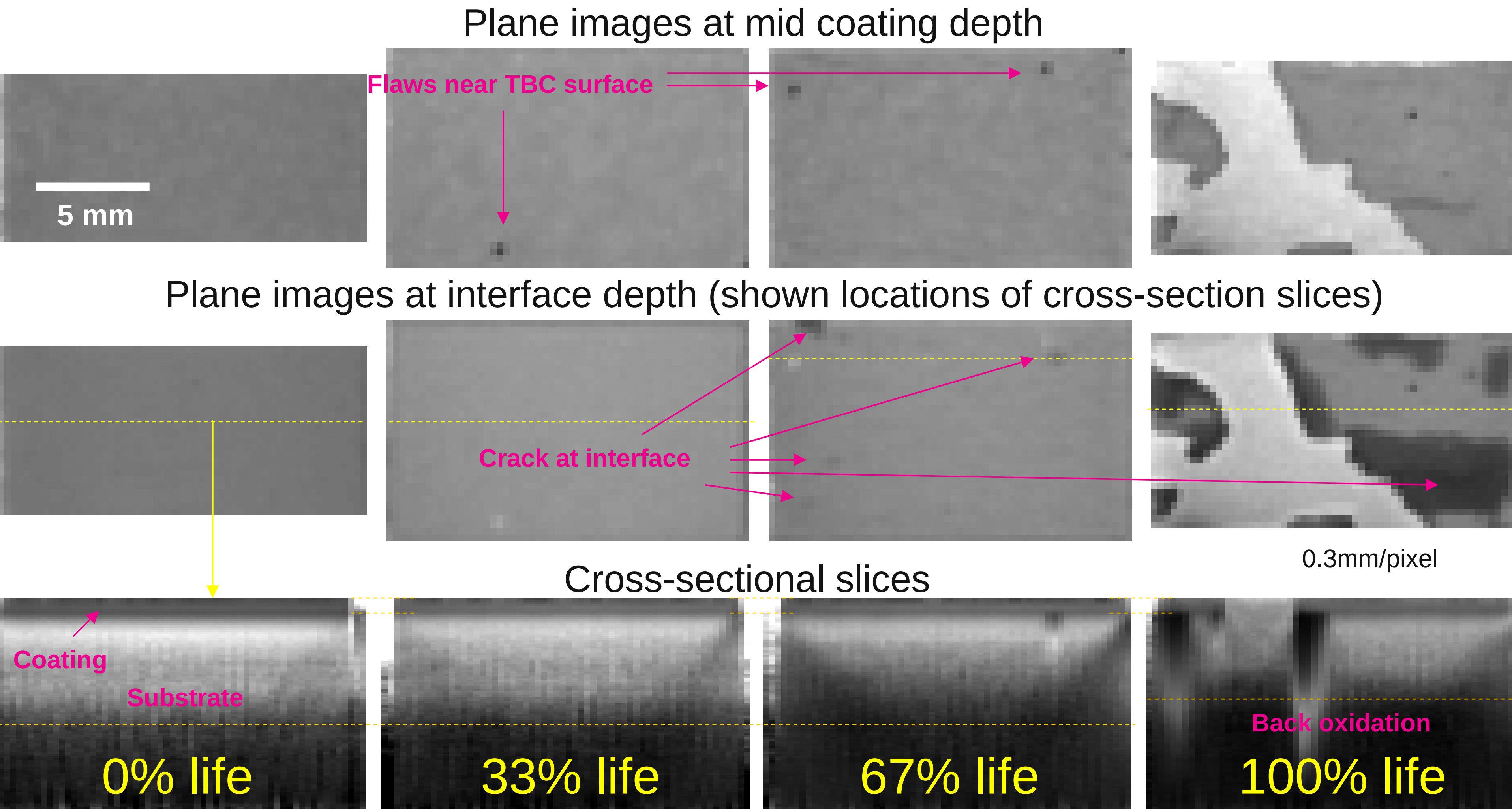


Prediction of TBC Property for Thermally-Cycled Samples

- Based on quantitative measurement of TBC conductivity values
- Sample curtsey of Mr. A. Luz, Imperial College London



Thermal Tomography Images of 3D TBC Structure



- Thermal tomography algorithm was improved to enhance image quality
- However, major development is needed to eliminate artifacts

Current Effort: Calibration of Multilayer Modeling Method

- Current computer algorithm is robust with consistent convergence in predicted TBC thermal properties (complete automated prediction)

- Current effort is focused on calibrating predicted TBC results
  - With literature values
  - With laser flash measurements

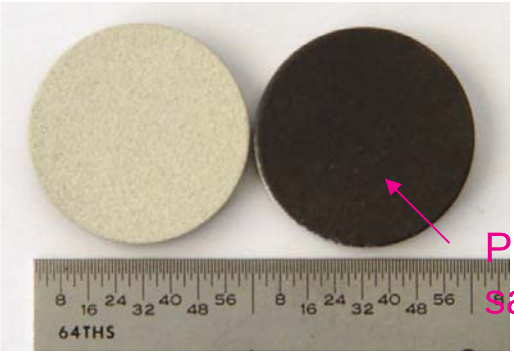
- Broad collaborations were established with industry and academics
  - Siemens, Praxair, Rolls Royce, DLR, SUNY, NASA, UCSB, etc

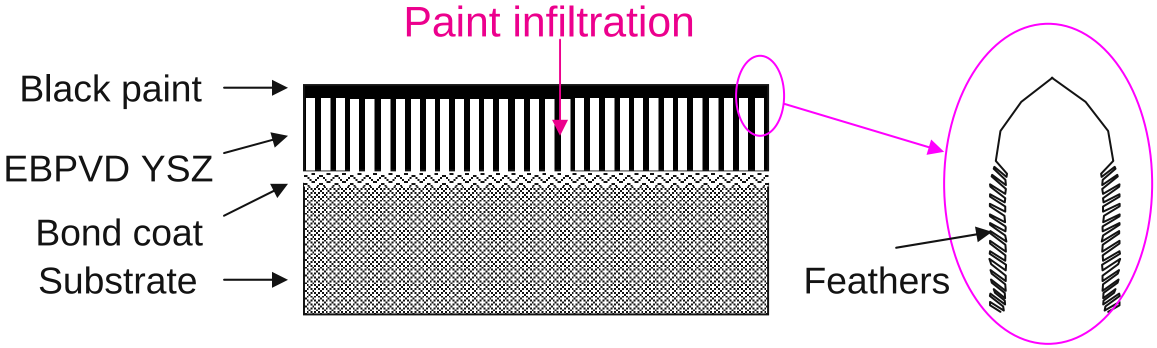
- When successful, this new technology may be widely used for both fundamental thermal property measurement and for NDE
  - Only laser flash method is currently available
  - Other reported methods are not expected to be widely adopted

Preliminary Calibration Findings

- For APS TBCs, good agreements in both predicted thermal properties were obtained – work ongoing
  - TBC coatings from 0.2 to ~1mm thickness
  - Different substrates
  - Also for metal coatings (with high conductivity)

- For EBPVD TBCs, predicted thermal properties were higher
  - Possible cause: infiltration of black paint inside feathers and TBC columns
    - *This effect is being assessed*
  - No painting is possible if a long-IR camera is used?

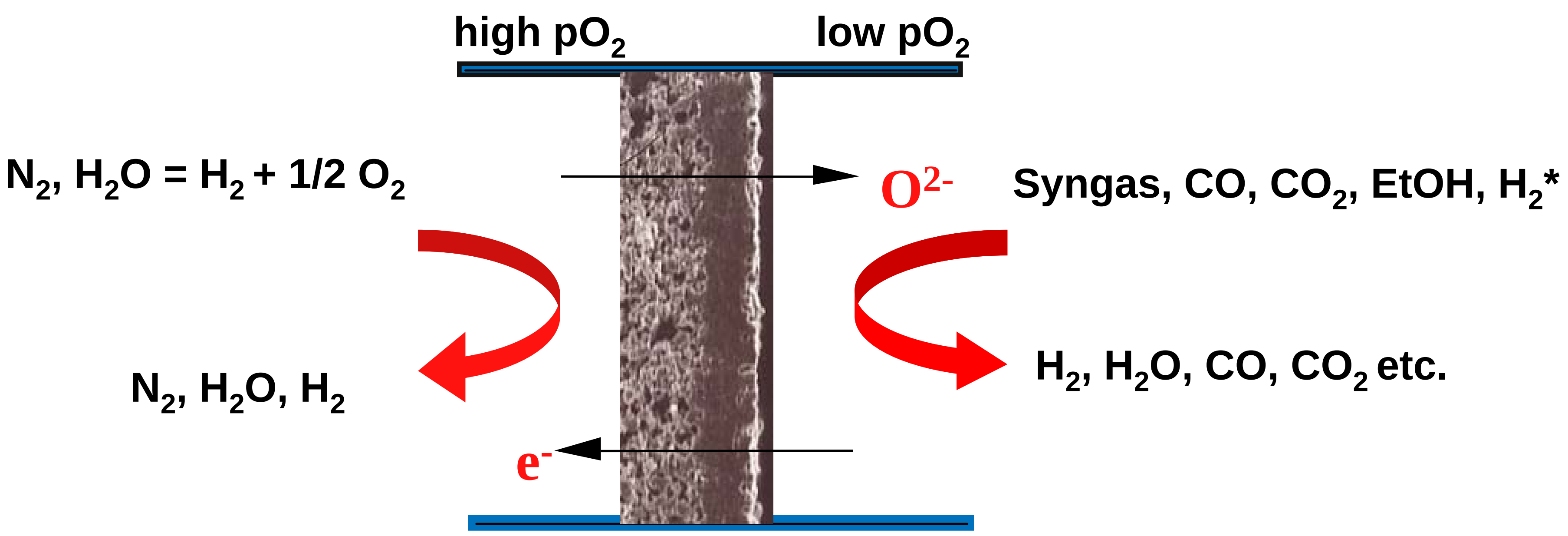




NDE Investigation for Gas-Separation Membranes

- Gas-separation membranes are being developed for FE applications
  - For example, hydrogen and oxygen membranes
- NDE opportunities are investigated for ceramic membranes
  - For materials
  - For components

Gas-Separation Membranes



*** Hydrogen is used as a model gas to maintain high pO_2 gradient.**

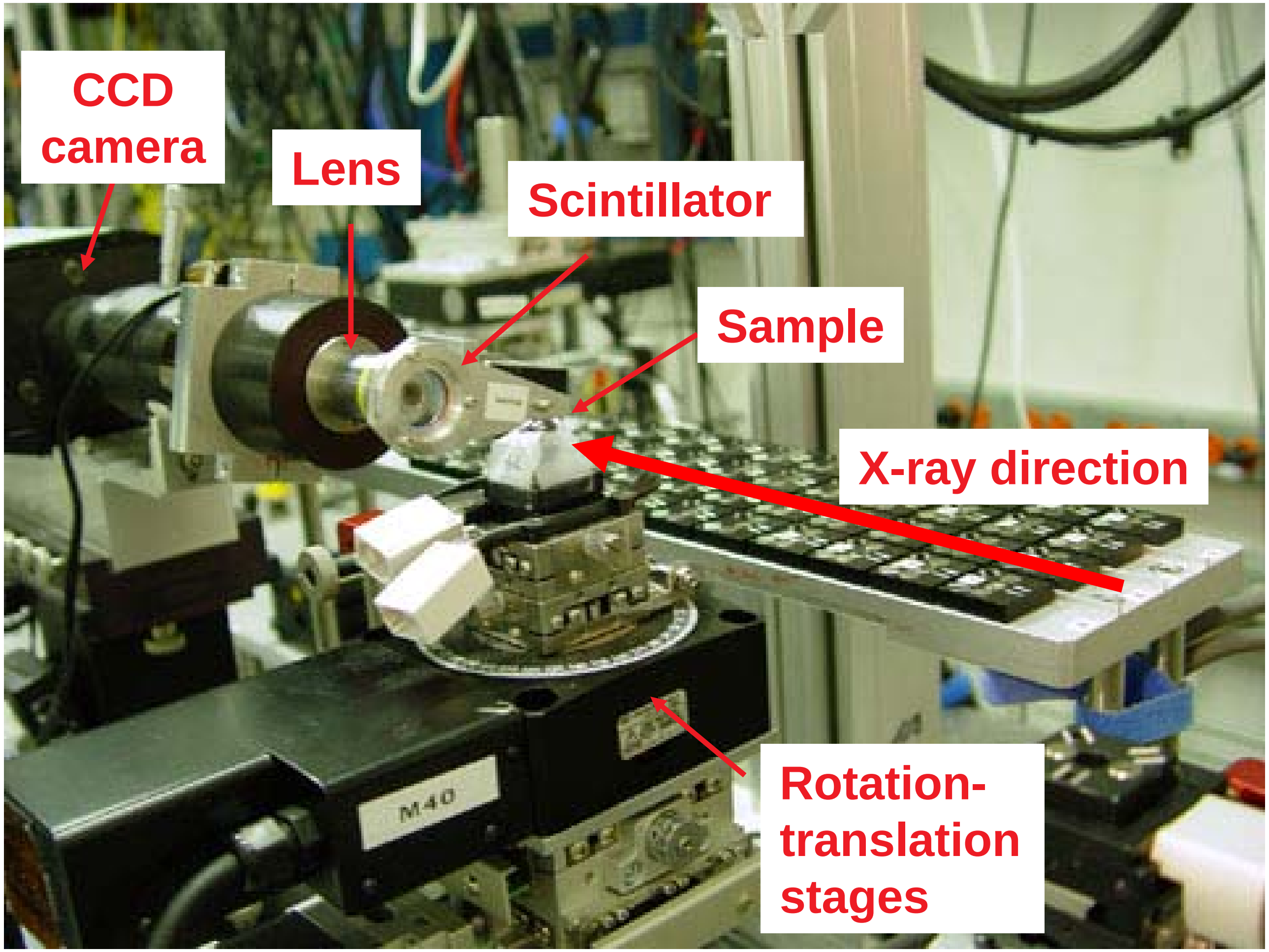
- Gas-separation membranes are being developed to produce hydrogen
  - E.g., ceramic oxygen-transport membrane via water splitting. Hydrogen is produced when the membrane removes oxygen from the water dissociation zone
  - Membrane structure: a thin film supported by a porous ceramic substrate

NDE Investigation for Ceramic Membranes

- NDE method: 3D synchrotron microtomography (microCT) at ANL's Advanced Photon Source (APS)

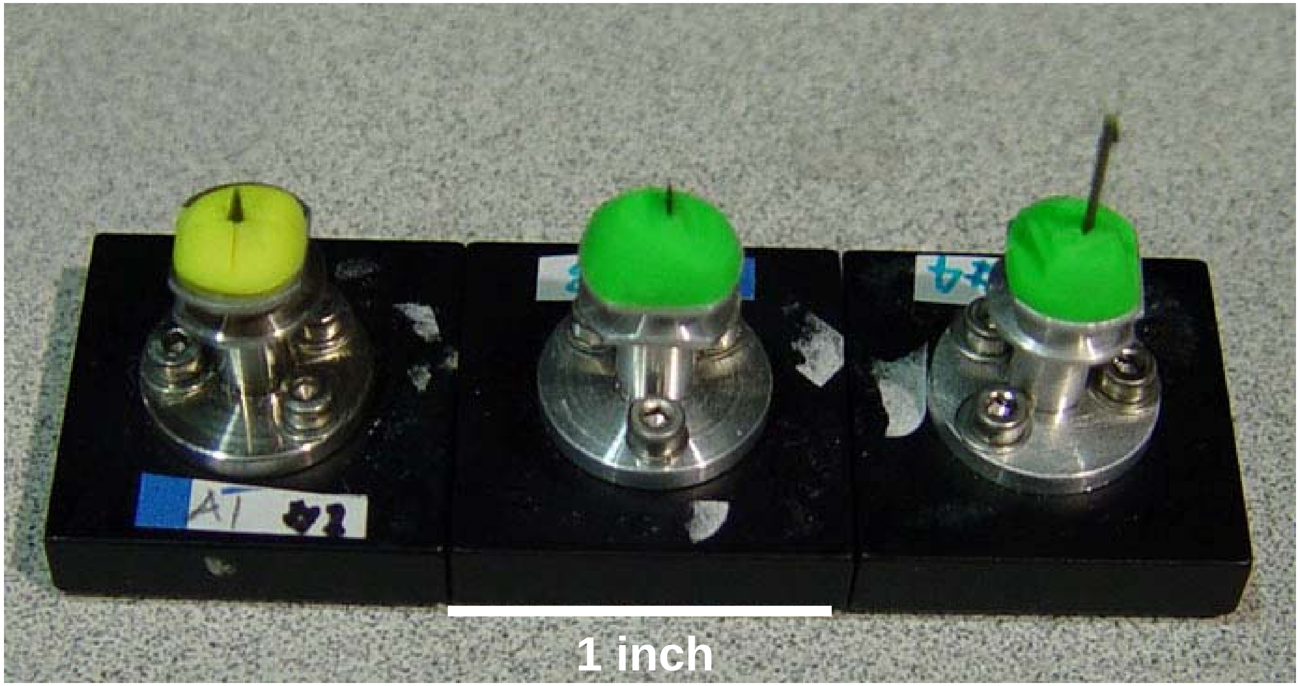
- NDE possibilities for materials and components:
  - dimension,
  - film/substrate microstructure,
  - defects (leaking)

3D MicroCT System at APS Synchrotron Beamline 2-BM



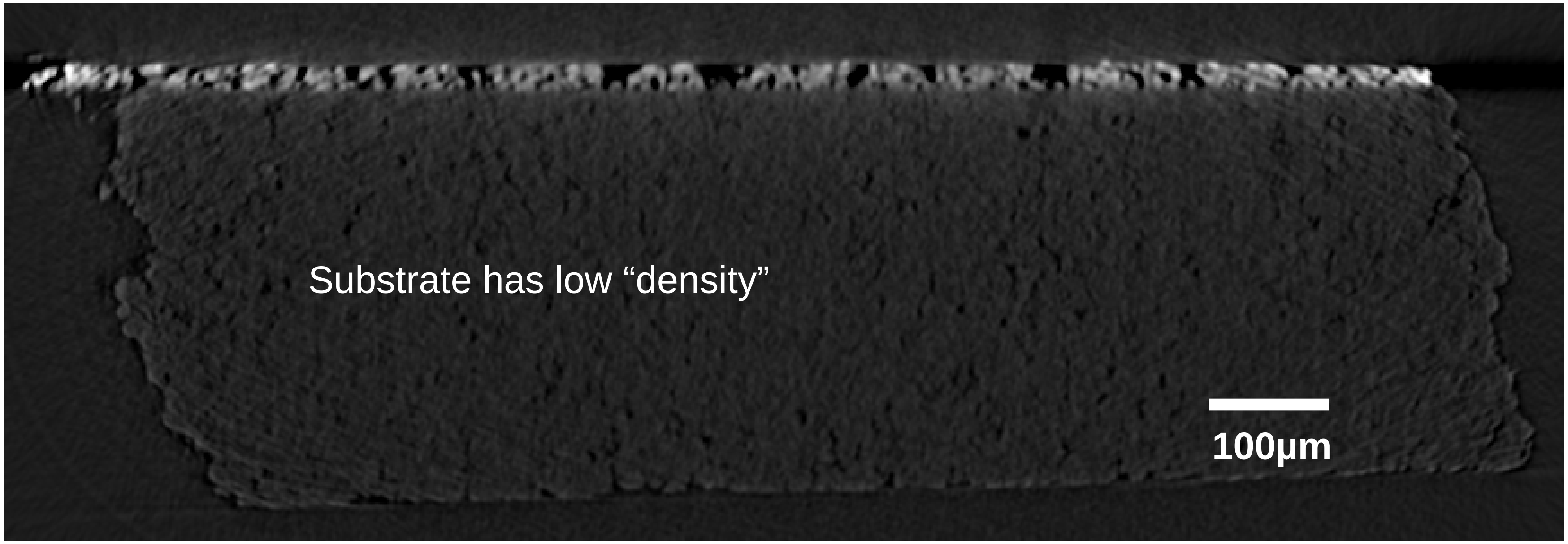
- X-ray source: monochromatic energy (~25keV),
- Detector: consists of a scintillation plate, a microscope lens (eg, 10X) and a digital camera of size 2048x2048 pixels

Typical Membrane Samples



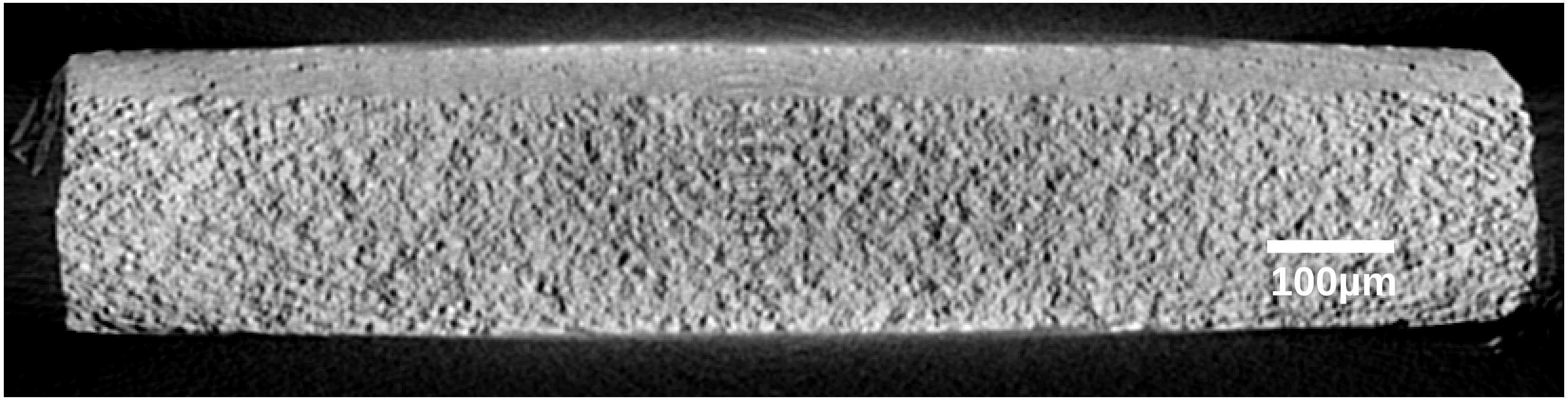
- Three small membrane samples (~2mm in dimension)
- MicroCT voxel dimension was 1.48 µm/pixel

Typical Cross-Sectional Slices for Sample 1



- Pd(60vol%)/CeO_2(40vol%) composite thin film on porous alumina (Al_2O_3) substrate
- CT image shows that the film is porous and at ~22 µm thickness

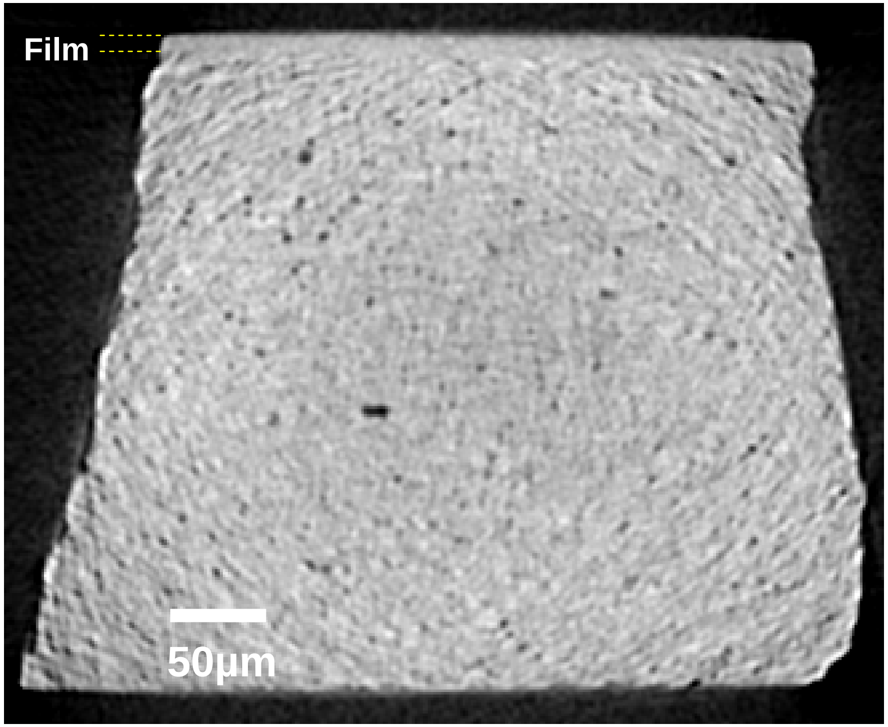
Typical Cross-Sectional Slices for Sample 2



- $SrFeCo_{0.5}O_x$ (CFC2) thin film on porous CFC2 substrate
- CT image shows that the film is dense and between 32-38 µm thickness

Typical Cross-Sectional Slices for Sample 3

CT image



SEM image

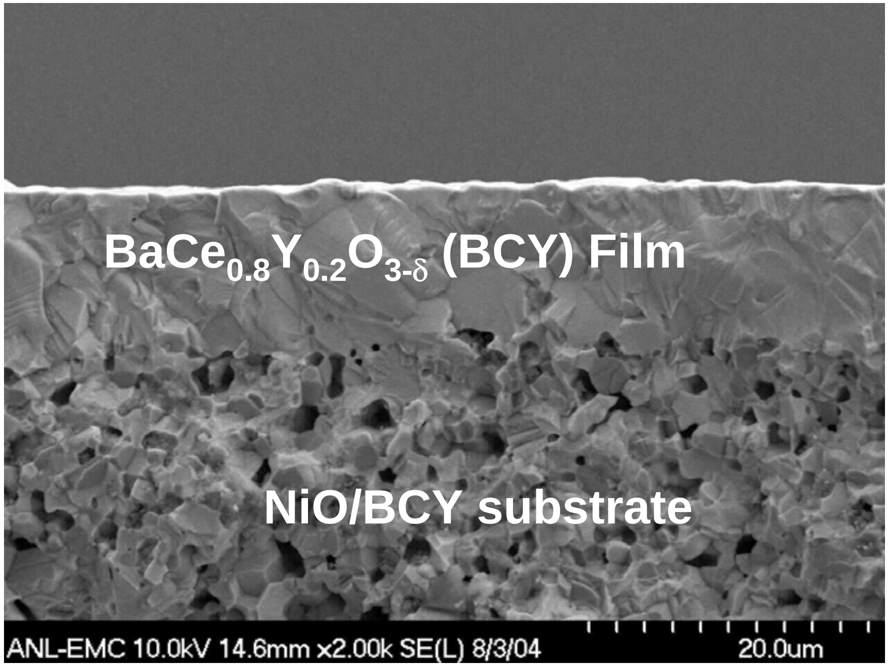


Image curtsey of Dr. T. Lee, ANL

- $BaCe_{0.8}Y_{0.2}O_3$ (BCY) film on BCY/NiO substrate (porous)
- CT image shows that the BCY film is dense and at ~10 µm thickness
- CT resolution is not sufficient to determine the pore size distributions

MicroCT Imaging of Cracks in Silicon Nitride Subsurface

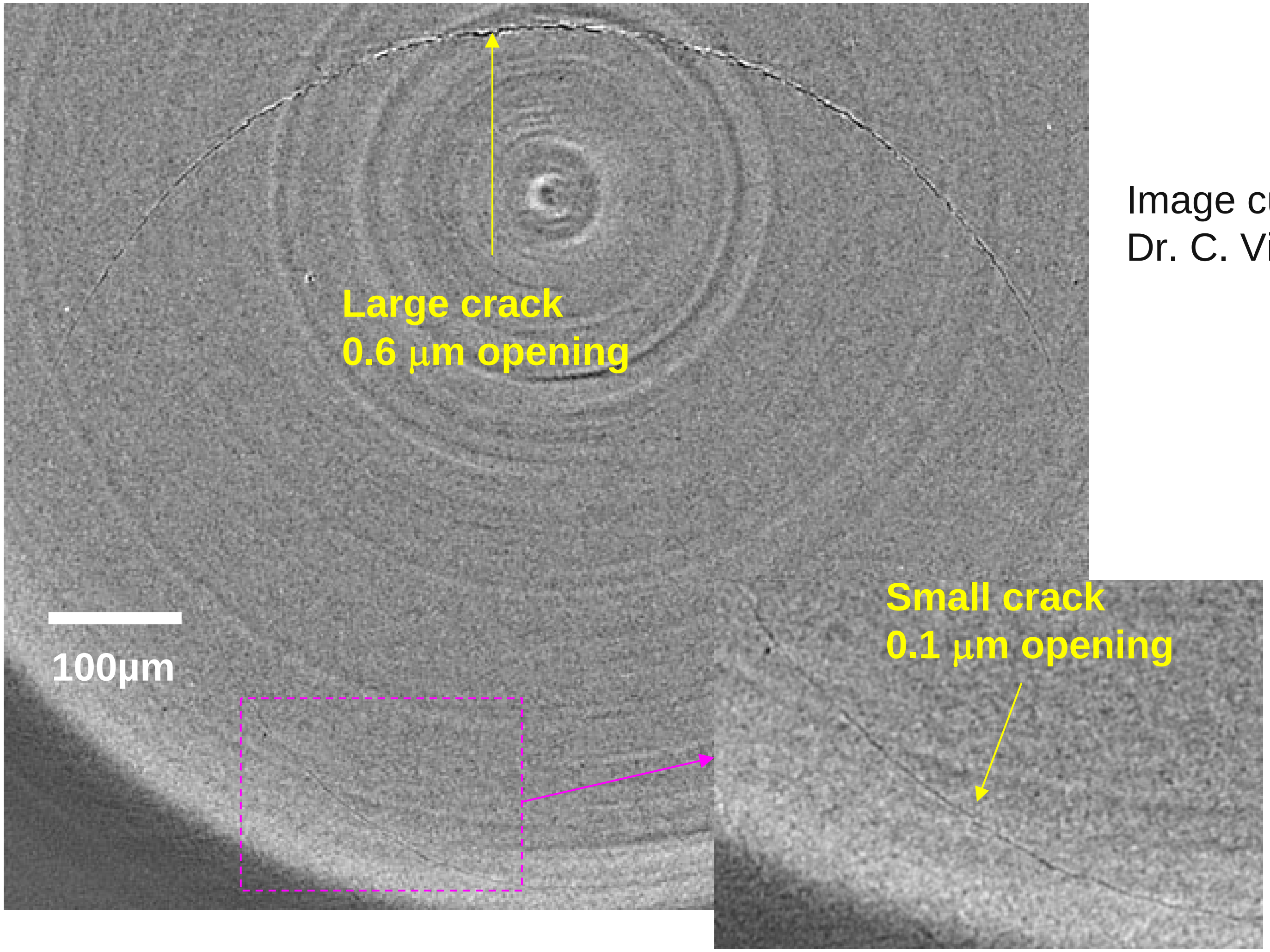


Image curtsey of
Dr. C. Vieillard, SKF

- MicroCT may detect very thin cracks (@ ~1/10 of the 0.74 µm pixel size)

Summary for NDE Developments for TBCs

- Multilayer modeling method was developed for quantitative imaging of TBC parameters
  - Current software is robust to predict TBC thermal properties
  - Capable to predict evolution of TBC conductivity for entire TBC life cycle
    - *Conductivity increase due to sintering*
    - *Conductivity decrease due to cracking/delamination*
  - Calibration of predicted thermal properties is ongoing
    - *Predicted values for APS TBCs are accurate (comparable to laser flash)*
    - *Predicted values for EBPVD TBCs are higher*
      - Possible effects were identified and being investigated
    - *Prediction of conductivity variation along coating depth is being verified*

- Thermal tomography method was improved to show enhanced 3D structural image

- Collaborations were established with industry and academics for relevant technology development and potential applications

Summary for NDE Investigation for Ceramic Membranes

- Synchrotron x-ray microCT was used to study ceramic membrane materials
  - High resolution, fast data acquisition an processing (<20min each)
  - It can resolve micron-sized volumetric features
    - *May determine membrane film and substrate dimension and structure*
      - However, not sufficient resolution for fundamental membrane study
    - *May detect film defect and leaking*
  - Can detect crack-type defect with gap-opening in the order of 1/10 of the voxel dimension (i.e., <0.1µm)

- For NDE of membrane components, other NDE methods may be needed
  - E.g., thermal imaging for leaking and cracking detection

Planned Future Efforts

- Further development of thermal imaging methods for TBCs
  - Continue calibration of predicted TBC properties with laser flash results
  - Apply to wider TBC parameter range: thin/thick, graded/layered, etc
  - For EBPVD TBCs, investigate effects that affect prediction accuracy
  - Develop additional models, such as for 2-layer coating system and coating transparency
  - Establish an accurate and reliable instrumentation for TBC characterization
- Validation of NDE model for TBC lifetime prediction
  - In collaboration with partners, perform TBC life-cycle tests and collect NDE data
  - Correlate TBC parameter change with TBC degradation state
  - Compare with destructive examination results
- Correlation between different NDE methods
  - Work with collaborators who are developing other methods