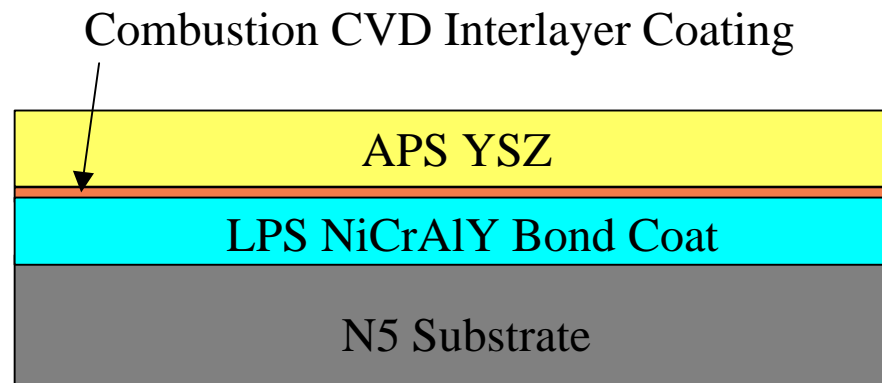


# Combustion CVD Coatings for TBC Improvement

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# Motivation

- Toughen the interface between air plasma sprayed YSZ and the nickel-chromium-aluminum-yttrium bond coat of thermal barrier coatings (TBC's) used on hot section components of gas turbines.



# Procedure

- Bond coated substrates supplied by GEPS (Schenectady, NY)
- Combustion CVD interlayer coatings applied by Georgia Tech
- APS YSZ applied by GEPS
- Furnace Cycle Testing to be performed at Georgia Tech

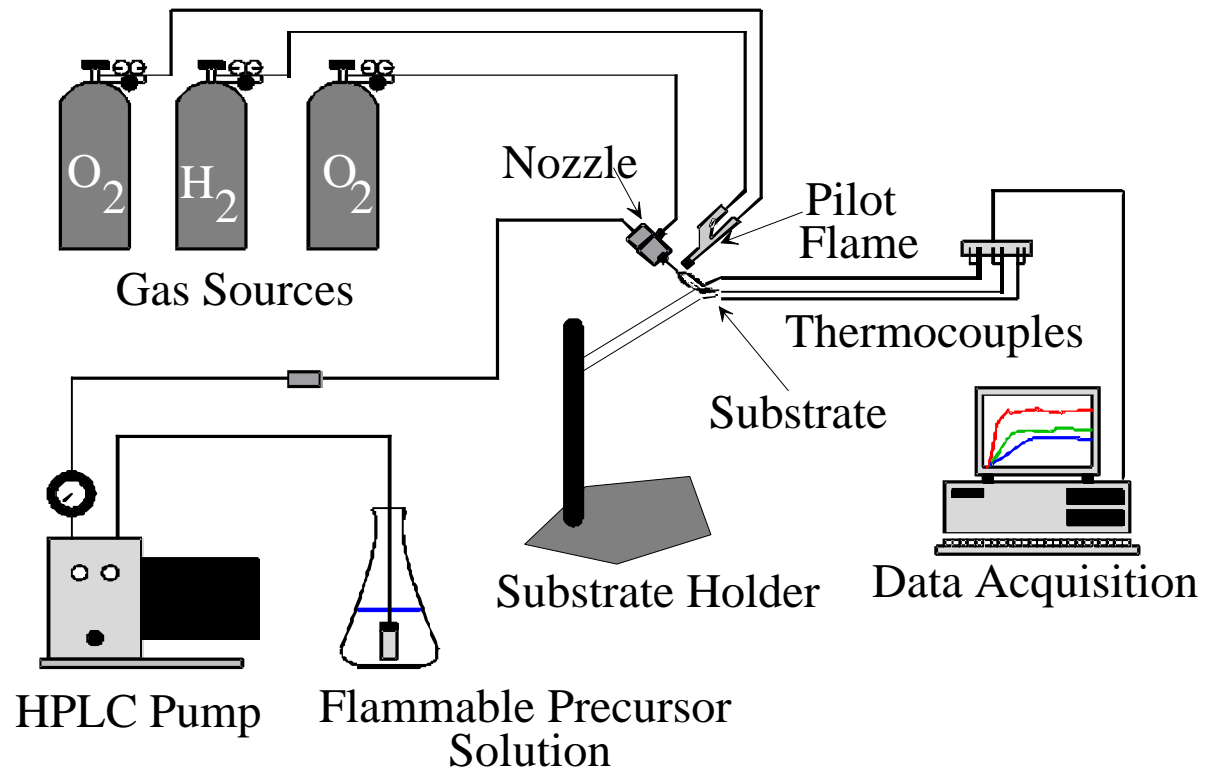
# Interlayer Coatings Under Investigation

- YSZ/Alumina Composite
- YSZ/Alumina Graded Coatings
- Alpha Alumina Coatings

# Combustion Chemical Vapor Deposition

- A Novel Approach for Depositing Ceramic Thin Films
- A Reaction Chamber Is Not Necessary
- Combustion Provides Most of the Heat for:
  - Chemical Reactions
  - Diffusion
  - Nucleation
  - Film Growth
- The Substrate Is Located in or near a Flame

# Combustion CVD System



# Process Parameters for YSZ/Alumina Composite Coatings

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Solvent	Toluene
Precursors:	Yttrium 2-EH* Zirconium 2-EH Aluminum acac**
Solution Flow Rate	4 ml/min
Precursor Concentration	0.002 M
Oxidizer	Oxygen
Oxidizer Flow Rate	6 - 12 lpm
Deposition Rate	1.8 - 2.5 $\mu\text{m/hr}$
Flame Temperature	1350 - 1550°C
Substrate	a-plane sapphire

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\*2-ethylhexanoate

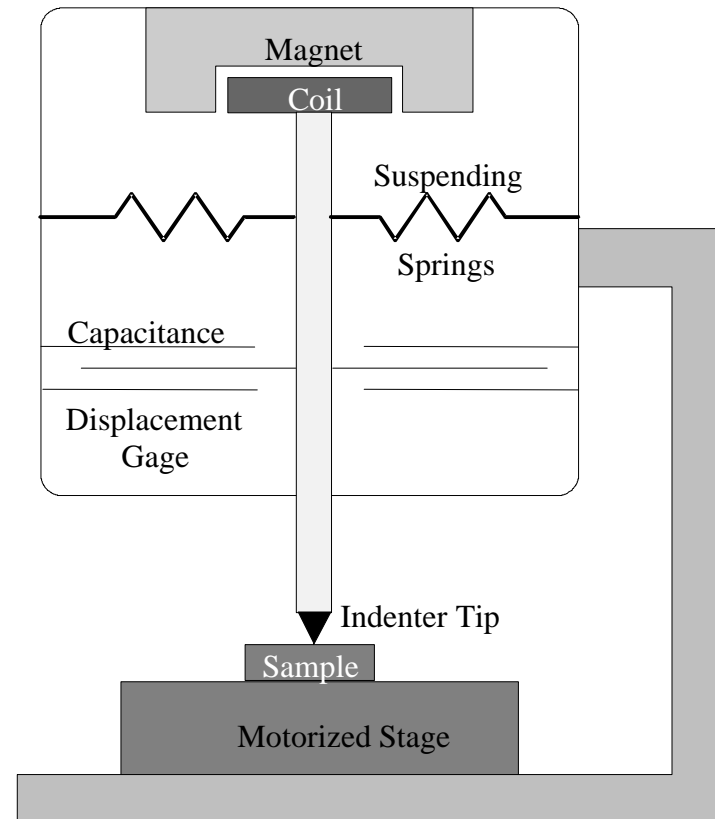
\*\*acetylacetonate

# YSZ/Alumina Composite Coatings

- Alumina is a Desirable Additive
  - Higher Elastic Modulus and Hardness than Zirconia
  - Lower Cost than Zirconia
- Alumina Additions to YSZ (in Bulk Sintered Powders) Shown to Improve Mechanical Properties
  - Reduced Amount of Monoclinic Phase
  - Increased Elastic Modulus
  - Increased Hardness
  - Increased Fracture Toughness



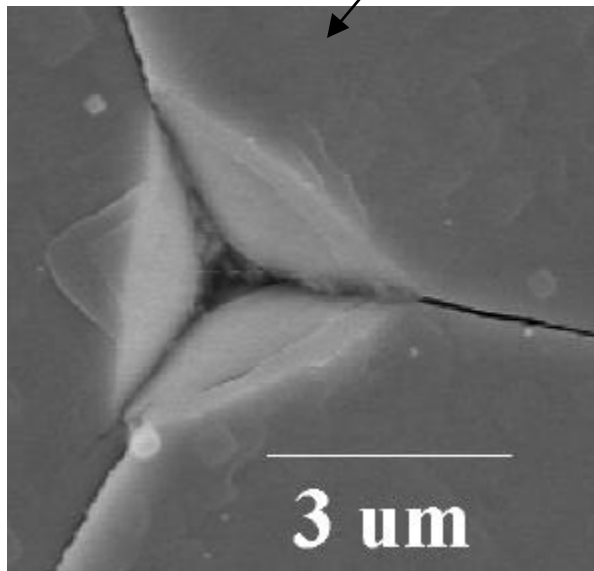
# Nanoindenter7



- Schematic of the Nanoindenter7
  - Load Resolution  $\pm 75.0$  nN
  - Depth Resolution  $\pm 0.04$  nm

# Nanoindenter7

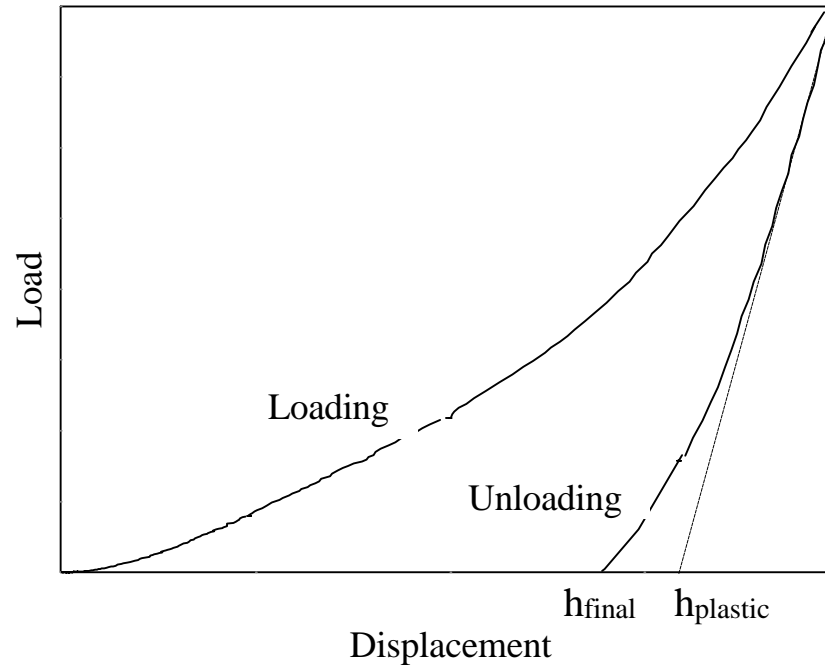
Typical Indentation Left in a Film after Loading by the Nanoindenter7



- Berkovich Indenter Tip
  - Triangular Pyramid
  - Area-to-Depth Function Equal to Vickers Indenter
  - Hardness and Elastic Modulus Measurements

- Cube-corner Indenter Tip
  - Much Sharper than Berkovich
  - Displaces More Material with same Load
  - Fracture Toughness Measurements

# Nanoindenter7



- Typical Load vs. Displacement Curve for a Nanoindenter7 Indentation

# Microhardness

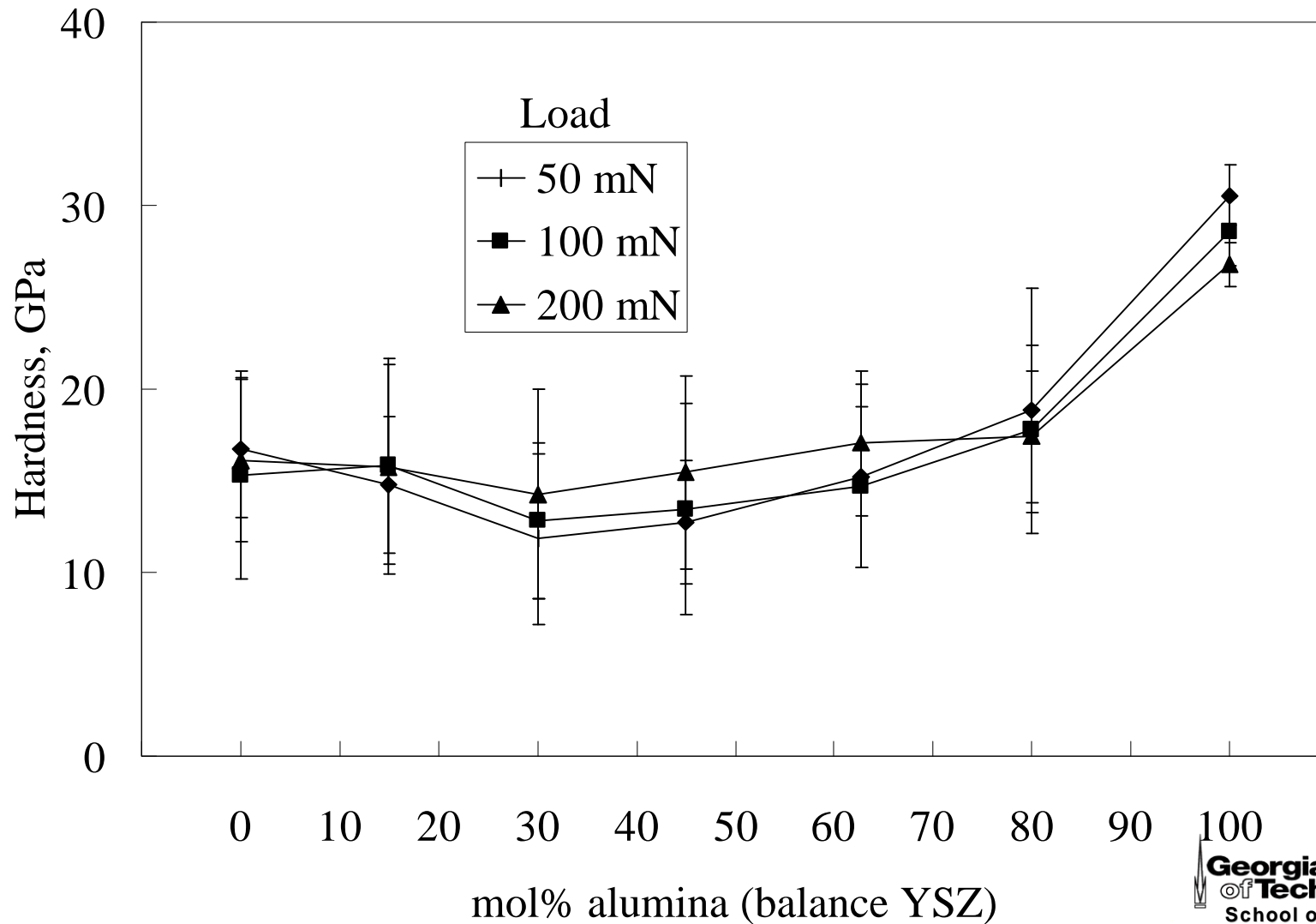
## ■ $H = P/A$

- P: Applied Load
- A: Projected Area of the Indentation after Removal of the Load ( $A=24.5h_p^2$  for Berkovich Indenter Tip)
- Same for Bulk Materials and Thin Films

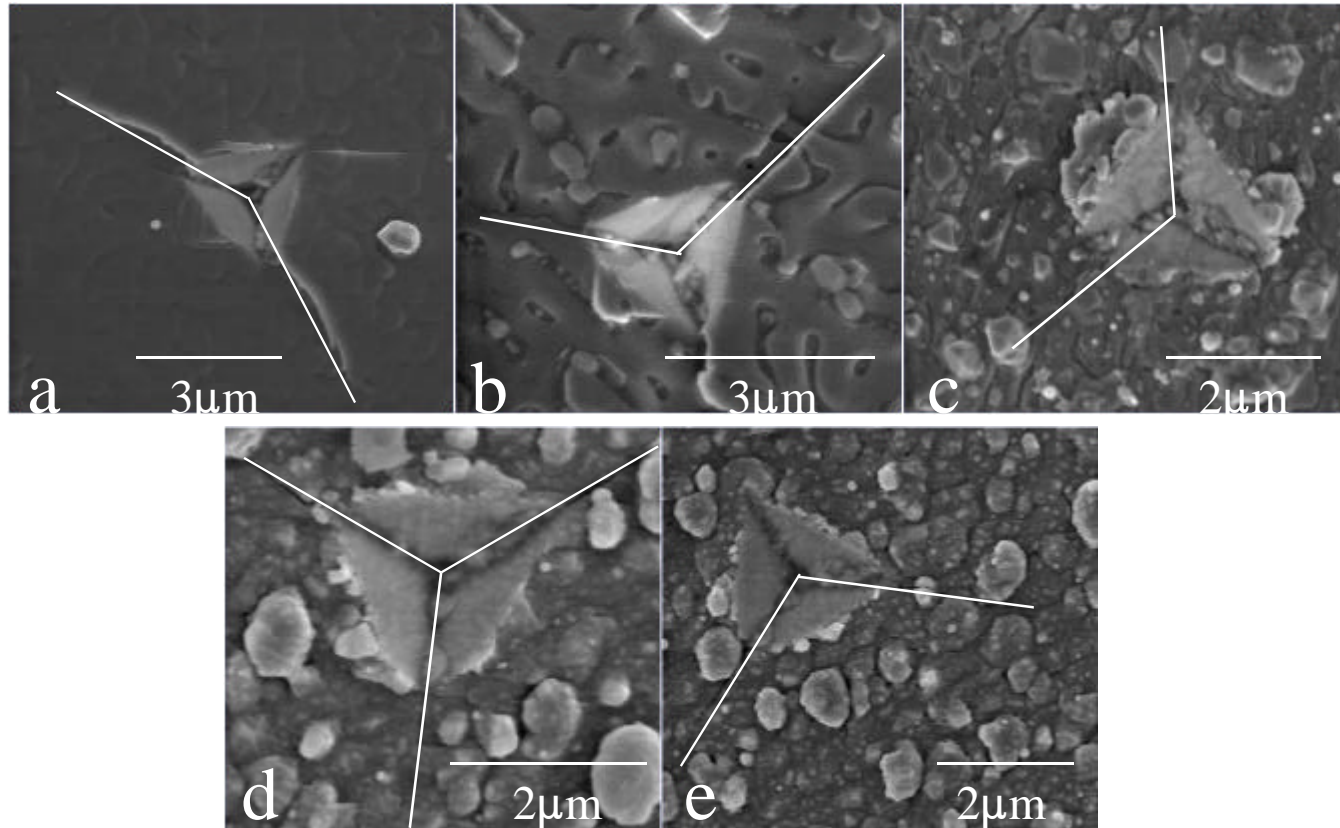
## ■ Hardness of Thin Films (Microhardness)

- Small Indenters (Radius of Curvature  $<100\text{nm}$ )
- Shallow Indentation Depths (5-10 times less than the Film Thickness)
- Light Loads ( $\sim\text{mN}$  down to  $\mu\text{N}$ )

# Hardness as a Function of Alumina Amount



# Crack Measurement



- Cube-corner indentations via Nanoindenter7 for each of the YSZ-alumina films; a) 100% YSZ, b) 15 mol% Al<sub>2</sub>O<sub>3</sub>, c) 30 mol% Al<sub>2</sub>O<sub>3</sub>, d) 45 mol% Al<sub>2</sub>O<sub>3</sub> and e) 62.8 mol% Al<sub>2</sub>O<sub>3</sub>.

# Fracture Toughness

■ 
$$K_C = a \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}}$$

–  $\alpha$  = empirical constant, ~0.032 for a cube-corner

–  $E$  = elastic modulus

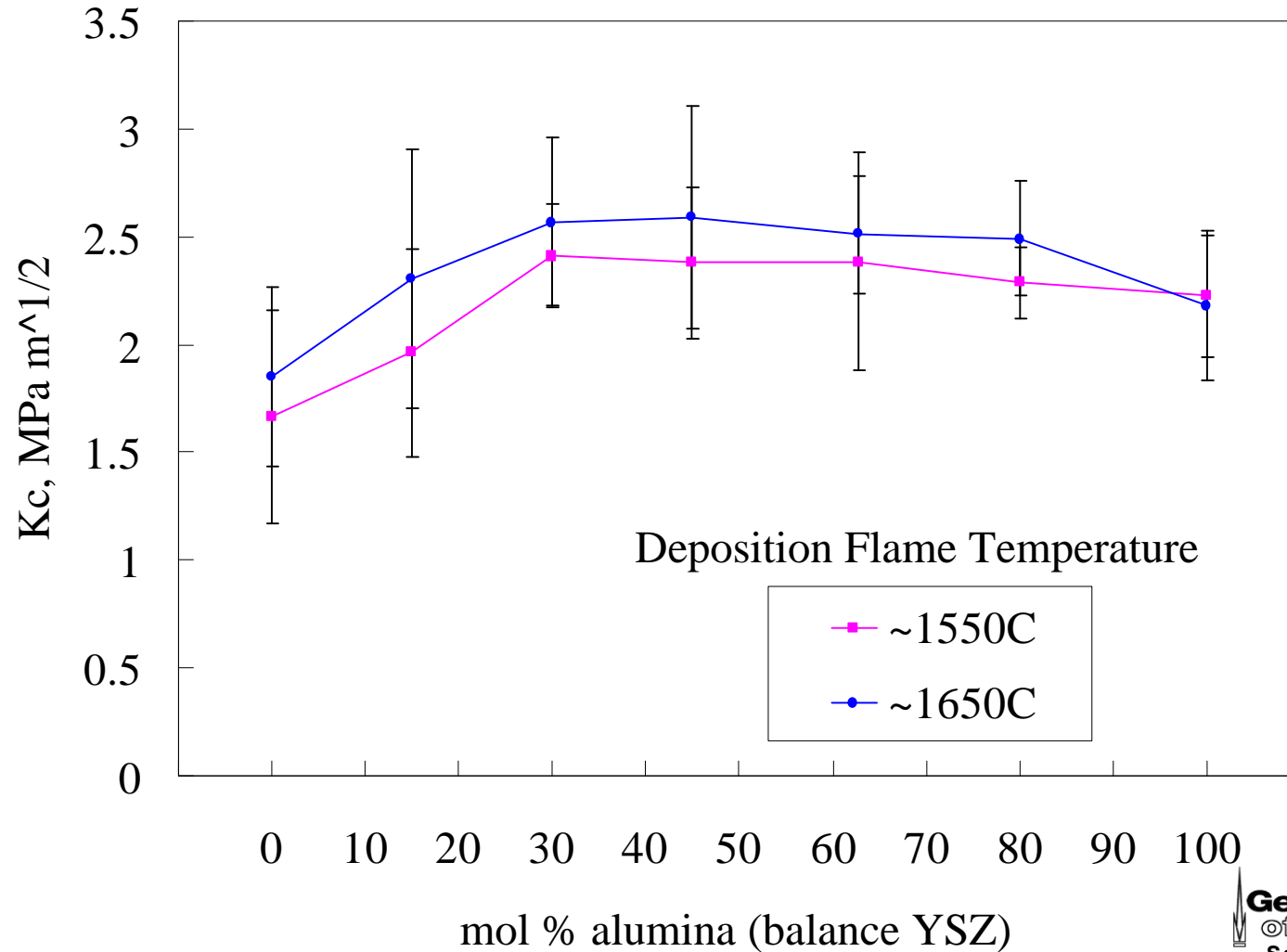
–  $H$  = hardness

–  $P$  = applied load

–  $c$  = crack length

■ Crack Length is Determined from Scanning Electron Microscope Image of Indentation

# Fracture Toughness as a Function of Alumina Amount





# Elastic Modulus

## ■ Determined from Slope of the Unloading Curve

$$- dP/dh = \beta E^* \sqrt{A}$$

$$- \frac{1}{E^*} = \frac{1 - \nu_0^2}{E_0} + \frac{1 - \nu^2}{E}$$

–  $E^*$  = effective modulus of the system

–  $E_0$  = elastic modulus of indenter

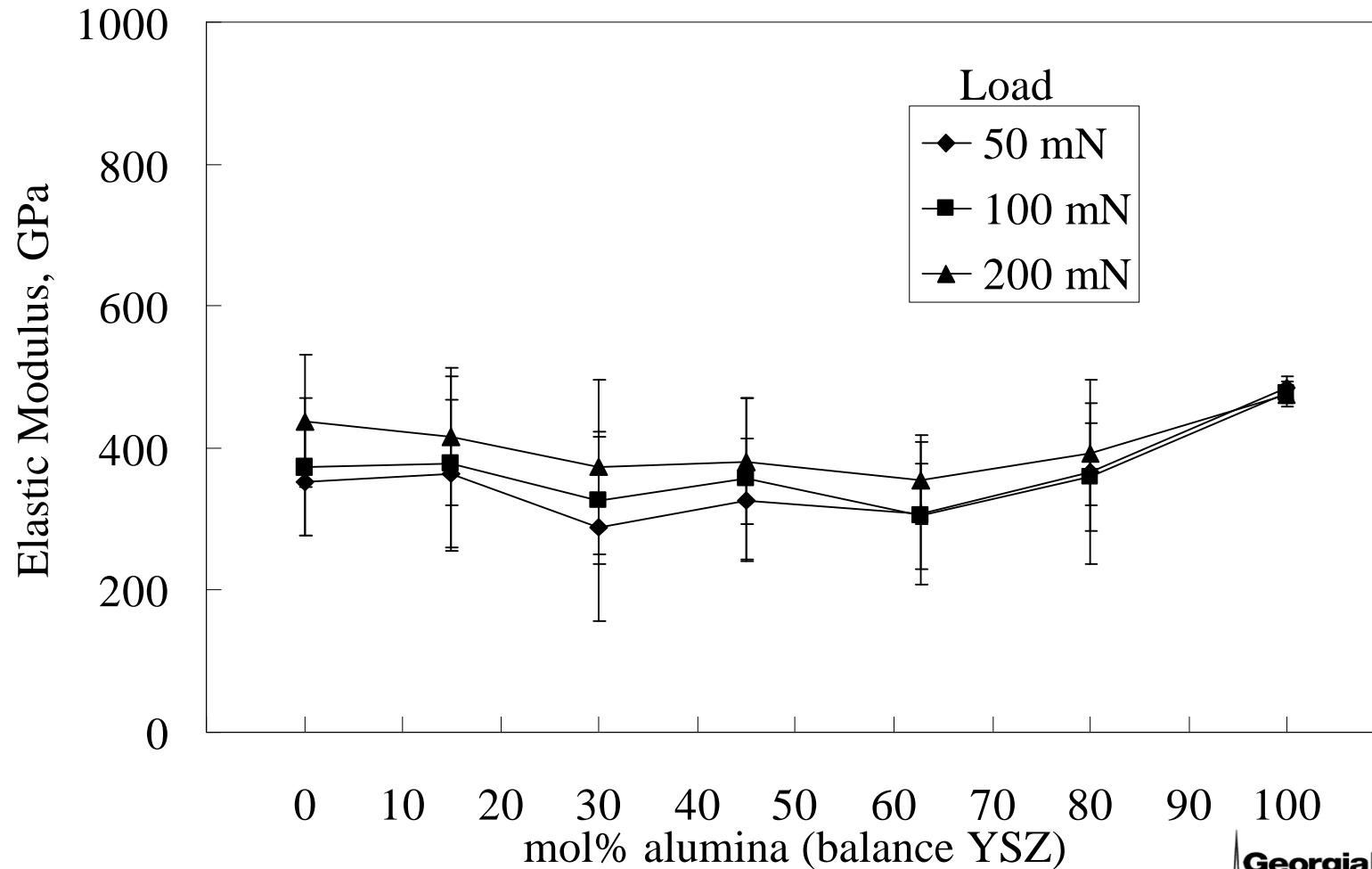
–  $E$  = elastic modulus of film

–  $\nu_0$  = Poisson's ratio of indenter

–  $\nu$  = Poisson's ratio of film

–  $\beta$  = constant dependent on indenter shape

# Elastic Modulus as a Function of Alumina Amount



# Process Parameters for YSZ/Alumina Graded Coatings

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Solvent	Isopropanol
Precursors:	Yttrium 2-EH* Zirconium 2-EH Aluminum acac**
Solution Flow Rate	2 ml/min
Precursor Concentration	0.002 M
Oxidizer	Oxygen
Oxidizer Flow Rate	0.6-0.8 liters/min
Deposition Rate	1.8 - 3.0 $\mu\text{m/hr}$
Flame Temperature	1250 - 1450°C
Substrate	Ni, Cr Superalloy

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\*2-ethylhexanoate

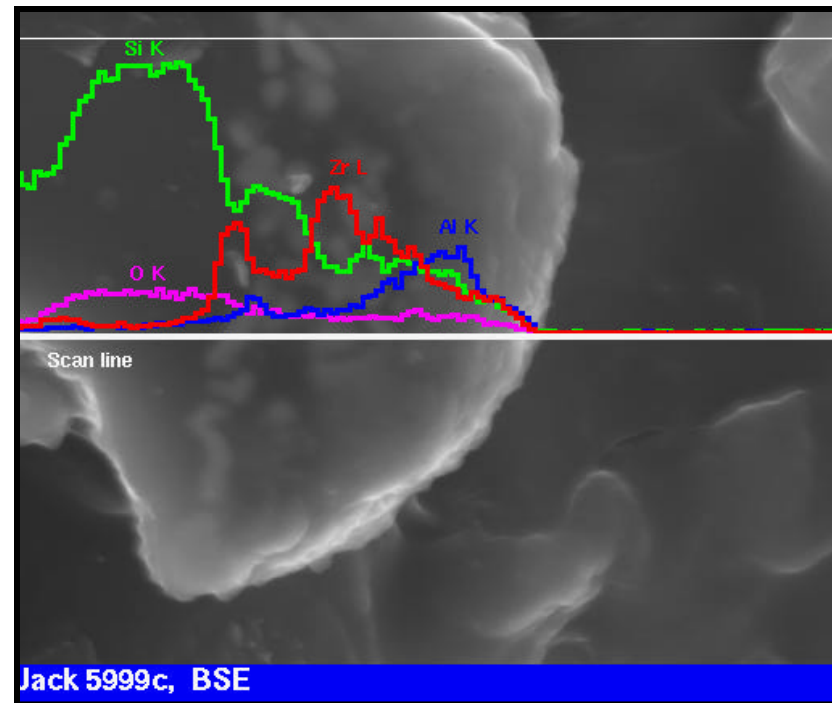
\*\*acetylacetonate

# Alumina/YSZ Graded Coating

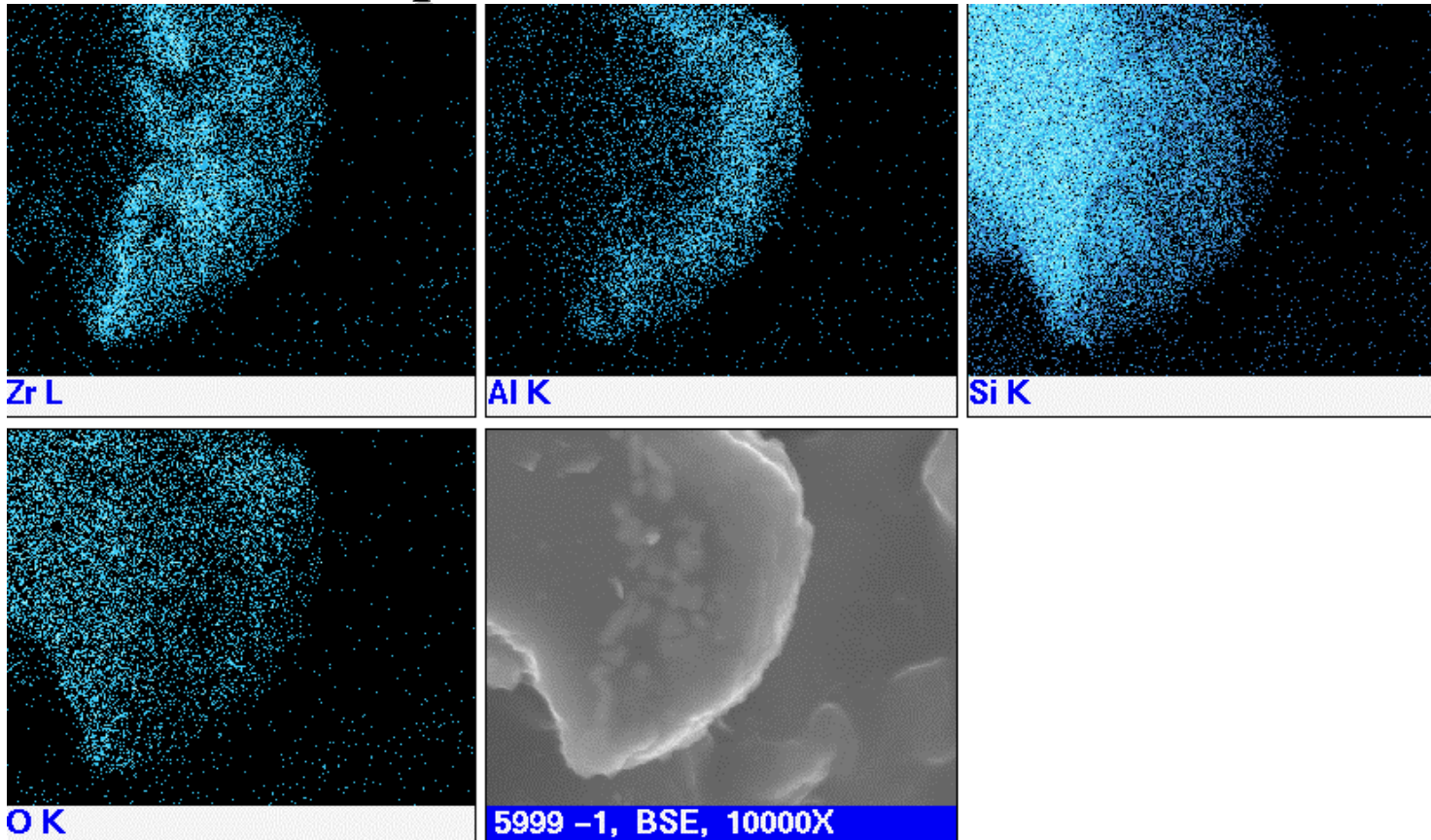
Deposited on Fused Silica

## Line Profiles

- Silicon Profile
- Zirconium Profile
- Aluminum Profile
- Oxygen Profile



# X-Ray Dot Maps of Alumina/YSZ Graded Coating Deposited on Fused Silica



# Process Parameters for $\alpha$ -Alumina Coatings

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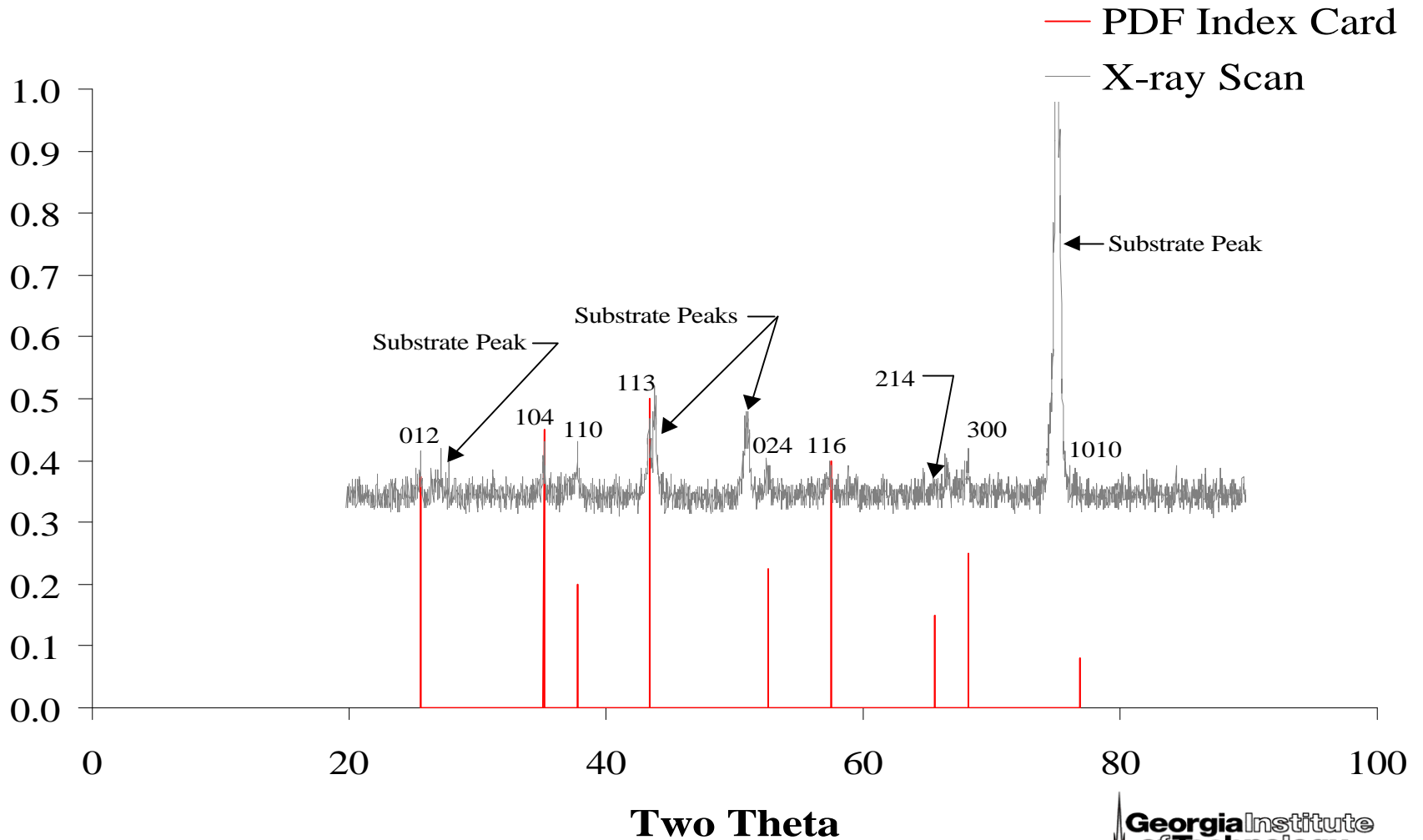
Solvent	Isopropanol
Precursors:	Aluminum acac**
Solution Flow Rate	2 ml/min
Precursor Concentration	0.002 M
Oxidizer	Oxygen
Oxidizer Flow Rate	0.6 - 0.8 liters/min
Deposition Rate	1.8 - 3.0 $\mu\text{m/hr}$
Flame Temperature	1250 - 1450°C
Substrate	Fused Silica Ni, Cr Superalloy

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\*\*acetylacetonate

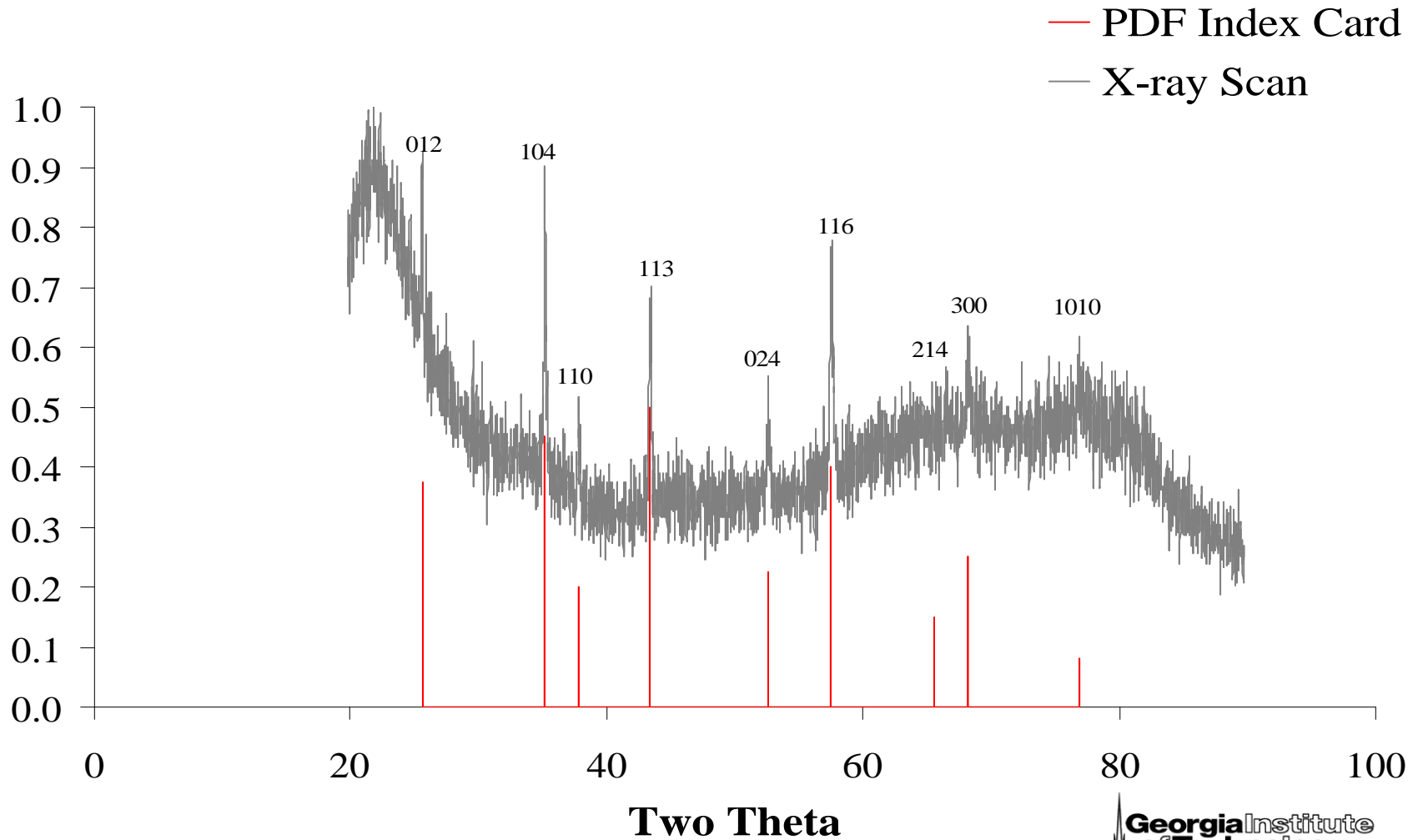
# $\alpha$ -Alumina on Super-Alloy

## X-Ray Diffraction Data



# $\alpha$ -Alumina on Fused Silica

## X-Ray Diffraction Data





# Conclusions

- Liquid fuel Combustion CVD can be used to deposit YSZ/alumina composite, YSZ/alumina graded, and alpha alumina coatings.
- Fracture toughness of YSZ/alumina composite coatings increases with increasing amounts of alumina (up to 30 mole percent).

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