



# Tantalum Oxide-Based Environmental Barrier Coatings

J. Guiheen, C.-W. Li – Honeywell, Inc.

H. Chan, M. Harmer, S. Wu – Lehigh U

K. Faber, M. Moldovan, C. Weyant –  
Northwestern U

M. Ferber, K. Moore – ORNL

DOE/Energy Efficiency Sciences Program under Cooperative

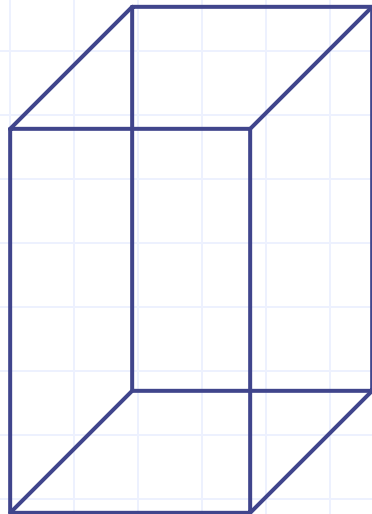
Agreement No. DE-FC20-01CH11086-A000

# Requirements for Environmental Barriers

- ◆ Thermal match with the substrate (AS800)
- ◆ Corrosion resistance
- ◆ Microstructural stability
- ◆ Phase stability
- ◆ Chemical compatibility with the substrate

⇒  $\text{Ta}_2\text{O}_5$  is a possible candidate

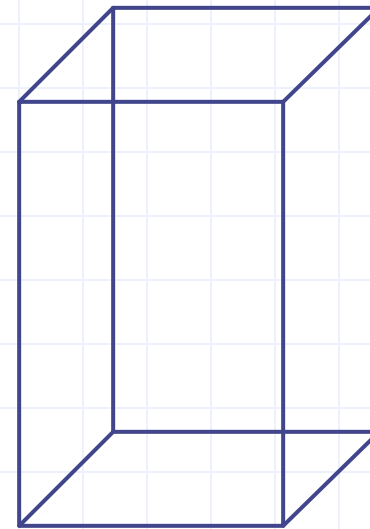
# Phase Stability of Ta<sub>2</sub>O<sub>3</sub>



$\beta$ -Ta<sub>2</sub>O<sub>5</sub>  
(orthorhombic)



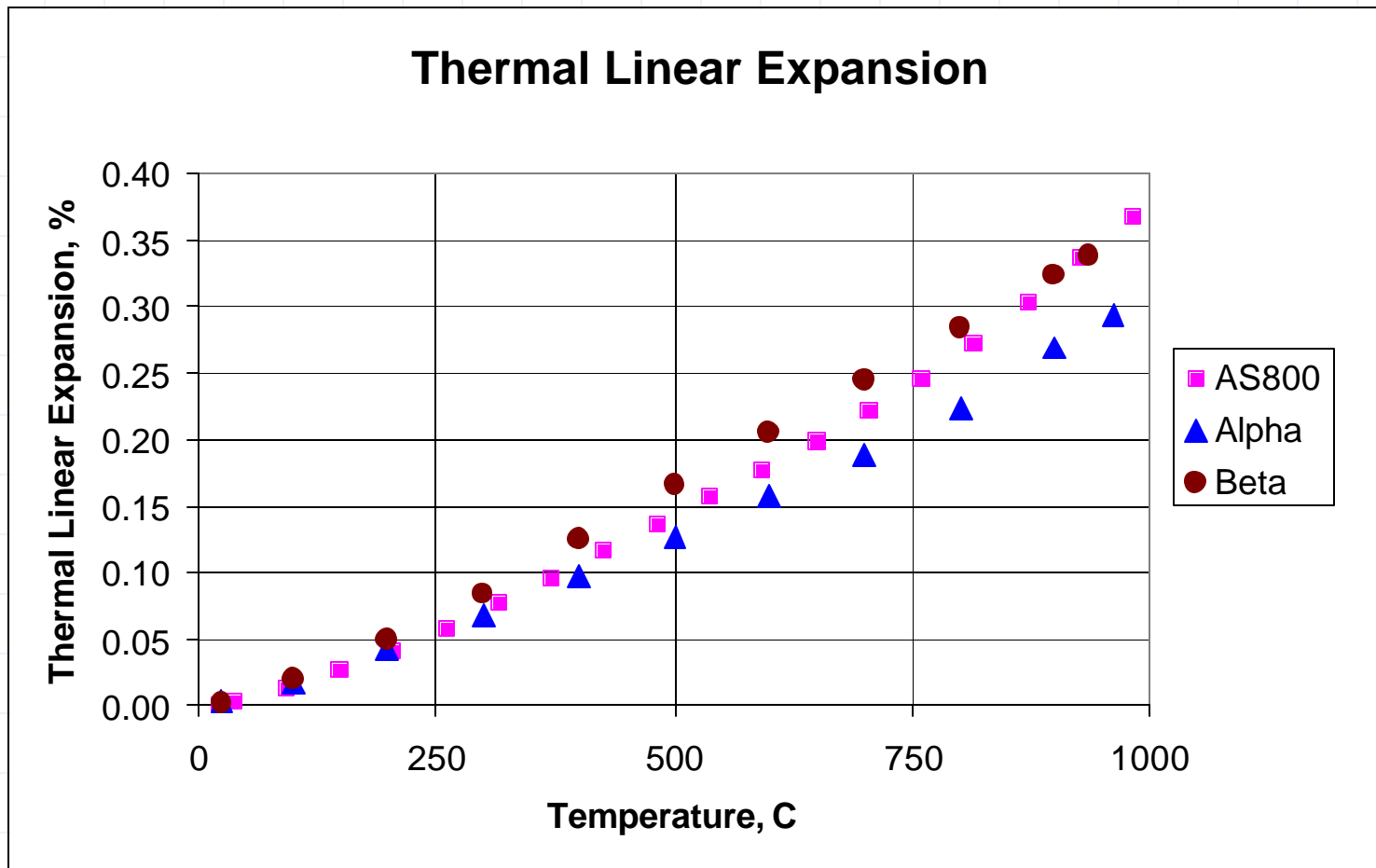
1360°C



$\alpha$ -Ta<sub>2</sub>O<sub>5</sub>  
(tetragonal)

?V = 8.6%

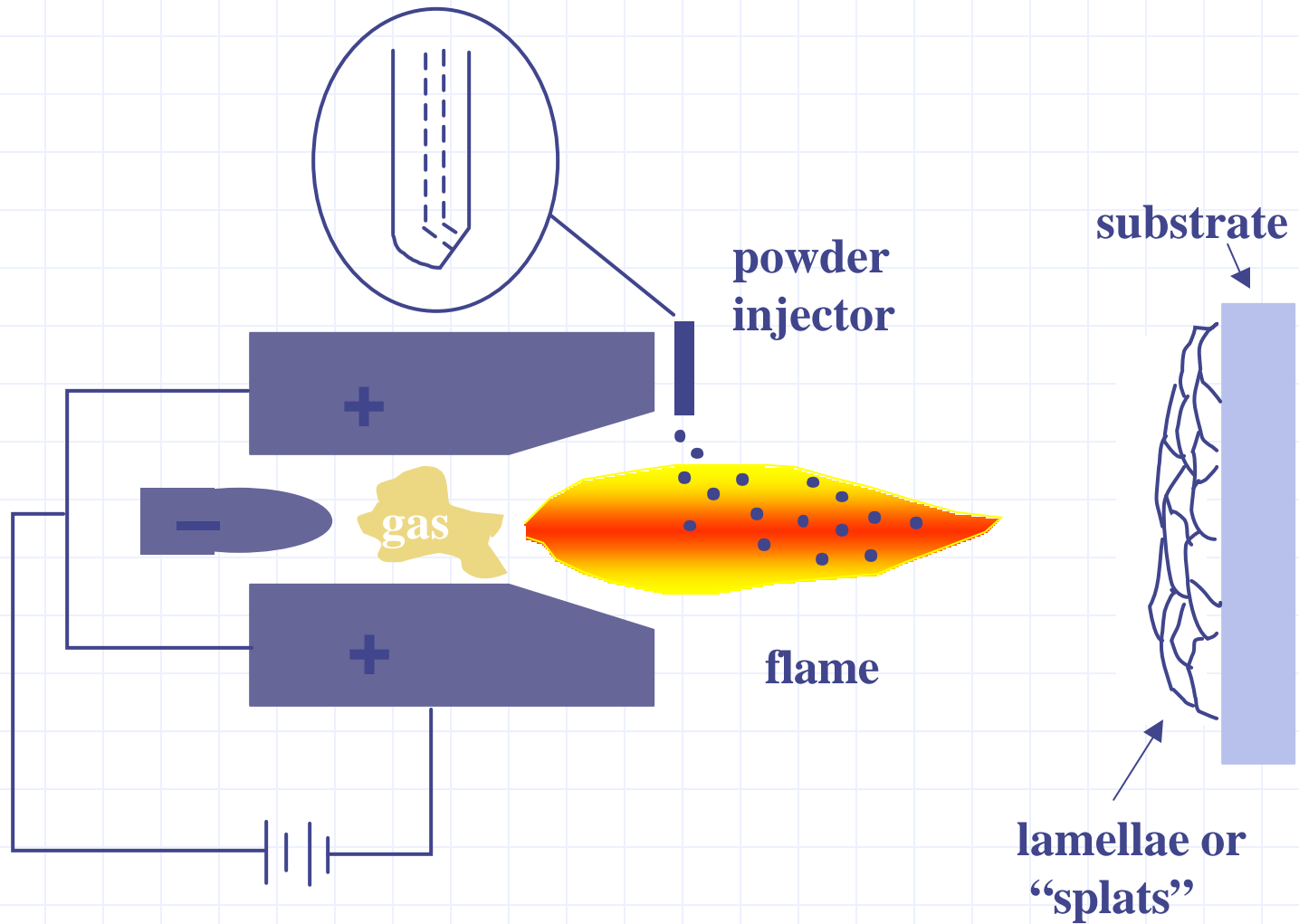
# Thermal Expansion of $Ta_2O_5$ and AS800



# Honeywell/NU/Lehigh/ORNL Program on EBC's

- ◆ Processing Methods for  $Ta_2O_5$  and  $Ta_2O_5$  Alloy Coatings/Plasma Spray Optimization
- ◆ Compositional Tailoring of a Family of  $Ta_2O_5$  Coatings
- ◆ Life Limiting Phenomena:
  - Oxidation/Recession
  - Residual Stress
  - Thermal Cycling

# Air Plasma Spraying

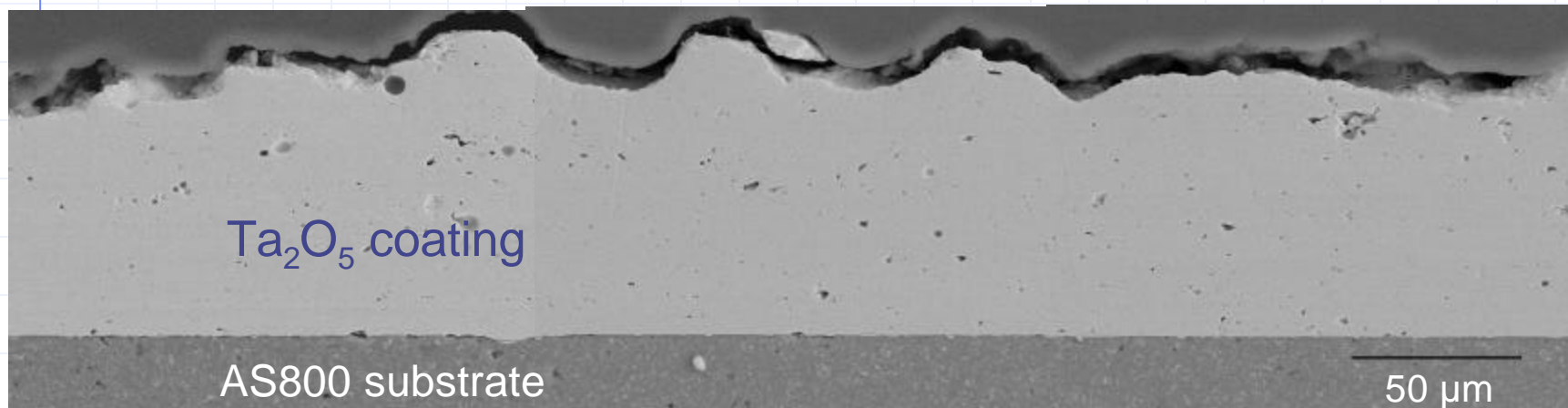


# Optimized Coating

Used Design of Experiments methodology to optimize coating.

Round 1: Seven factors; two levels

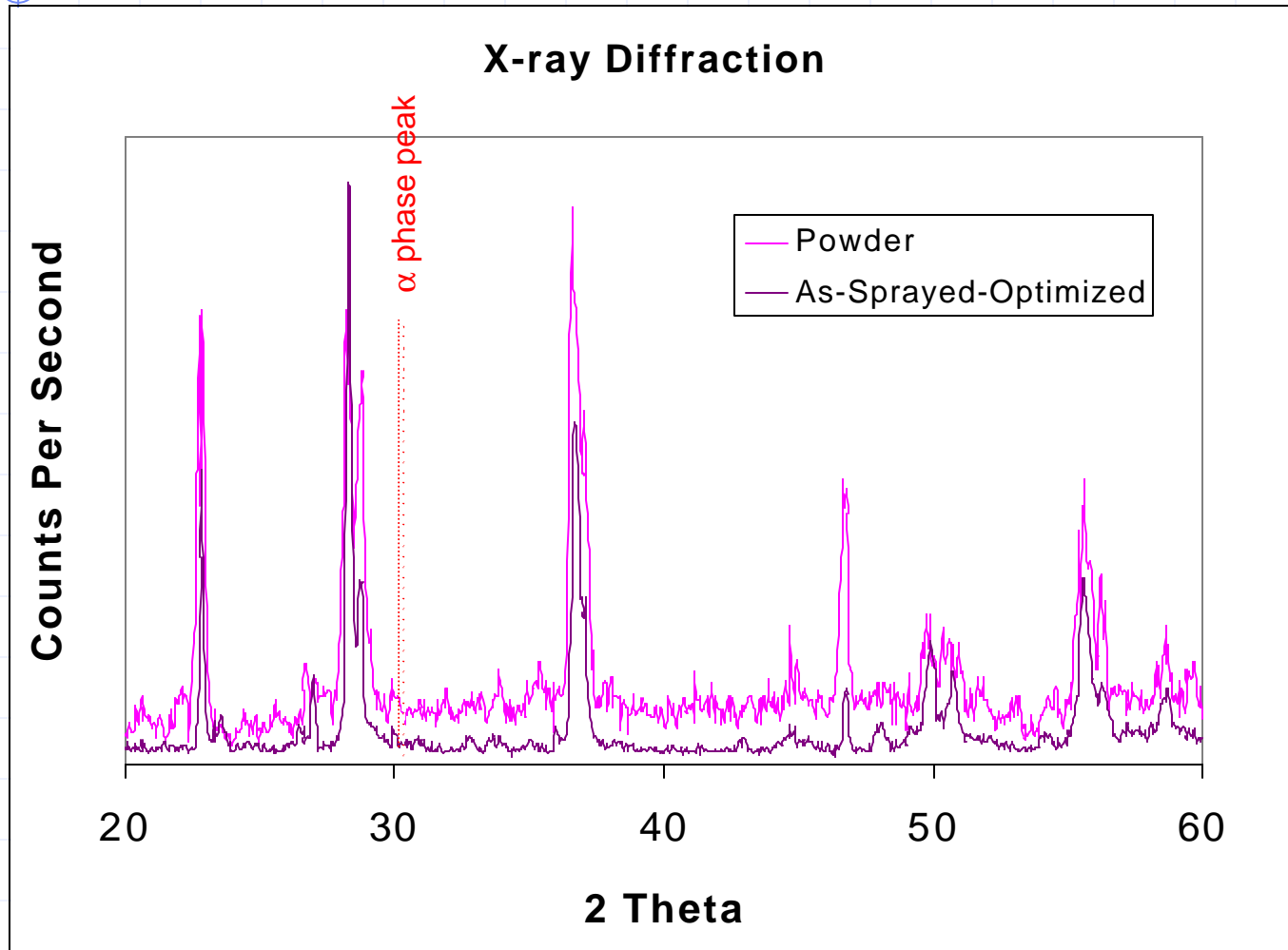
Round 2: Five factors; two levels and one factor; three levels



Minimize offset, injector angle, distance, carrier gas flow.

Maximize power, total gas flow and % hydrogen.

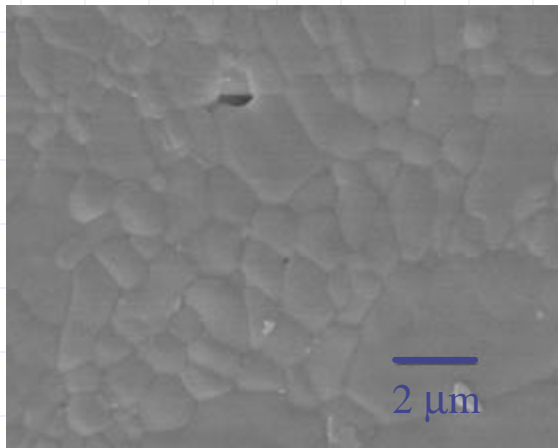
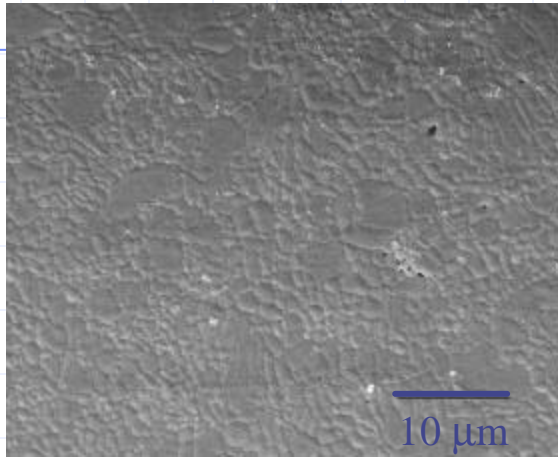
# Phase Stability of Ta<sub>2</sub>O<sub>5</sub>



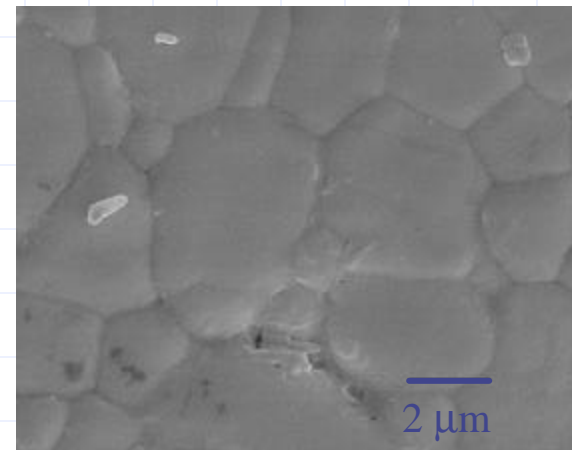
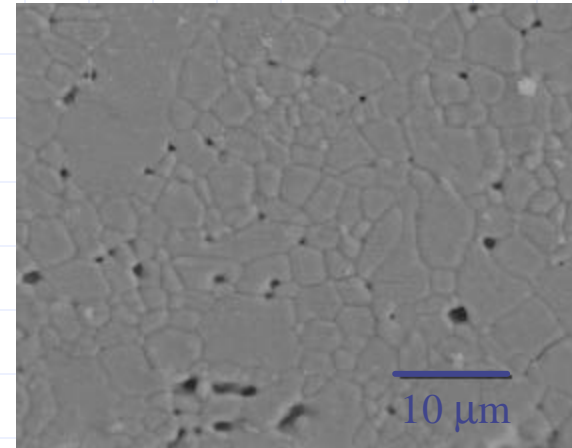
Trace  
 $\alpha$ -Ta<sub>2</sub>O<sub>5</sub>  
detected.



# Microstructural Stability of Ta<sub>2</sub>O<sub>5</sub>



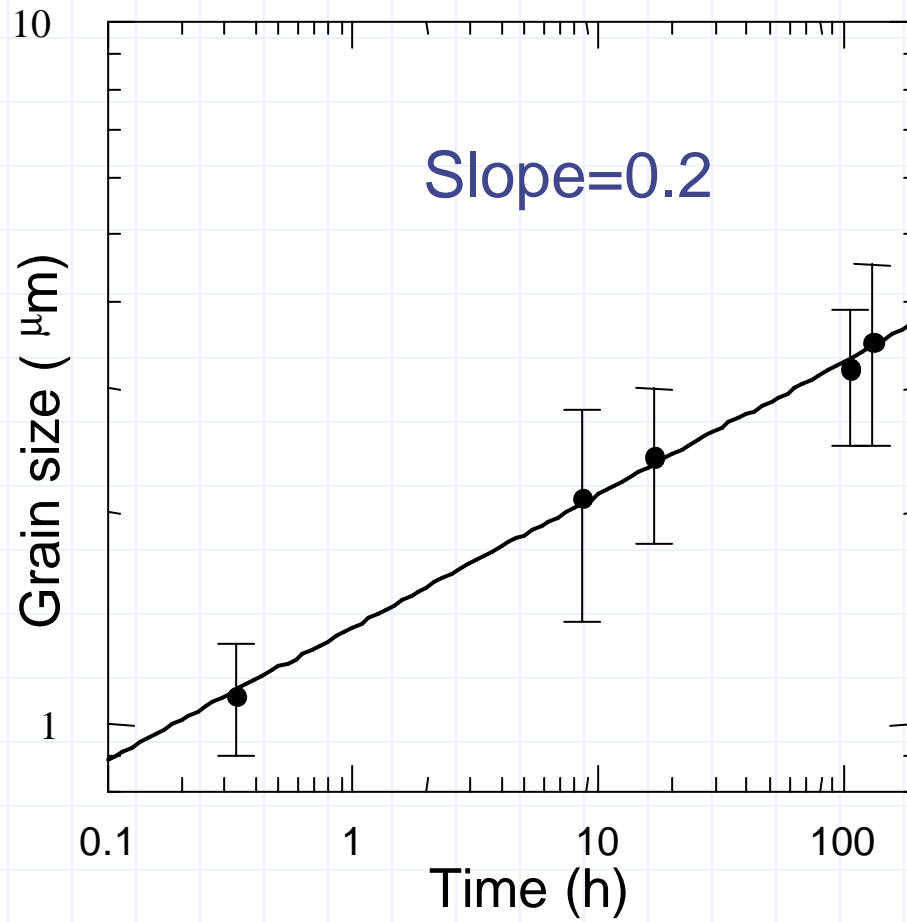
Ta<sub>2</sub>O<sub>5</sub> as-sprayed



After 105 hours at  
1200°C

# Microstructural Stability

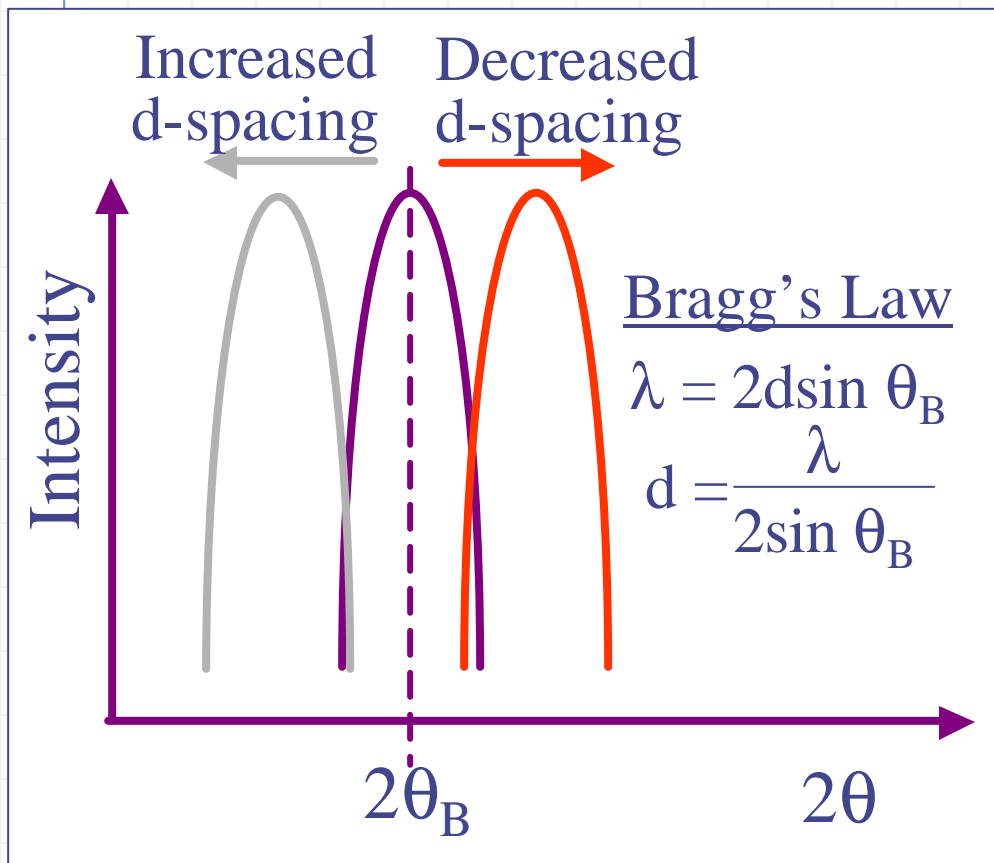
Grain Growth at 1200°C



$$d-d_0 = (2k)^{1/2}t^{1/2}$$

# X-ray Residual Stress Analysis

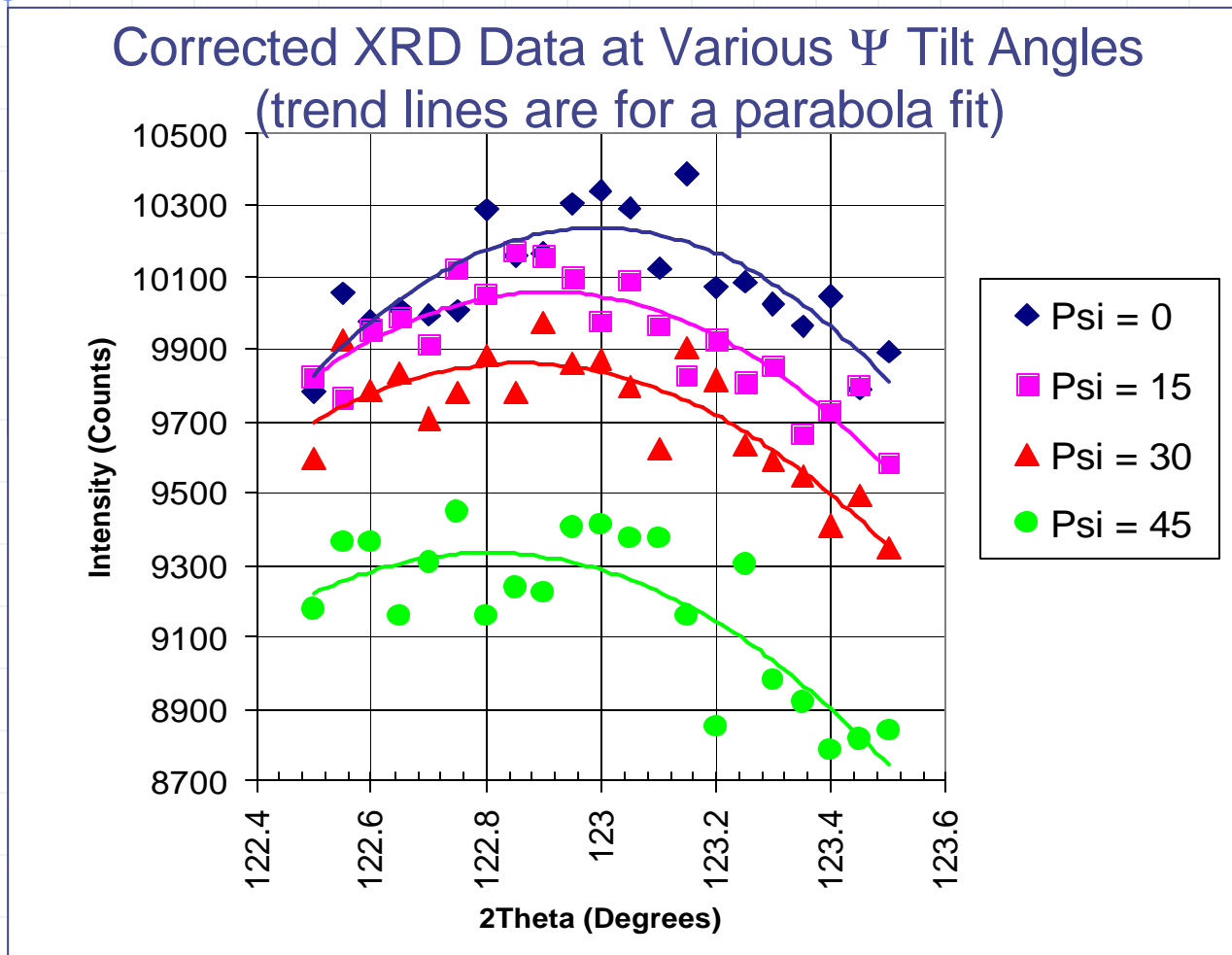
$$\frac{d_{f\Psi} - d_o}{d_o} = \frac{1+\nu}{E} \mathbf{s}_f \sin^2 \Psi - \frac{\nu}{E} (\mathbf{s}_{11} + \mathbf{s}_{22})$$



$d_f$  = stressed lattice spacing  
 $d_o$  = unstressed lattice spacing  
 $\mathbf{s}_f$  = stress component in  $f$  direction  
 $\Psi$  = tilt angle  
 $E$  = elastic modulus  
 $\nu$  = Poisson's ratio

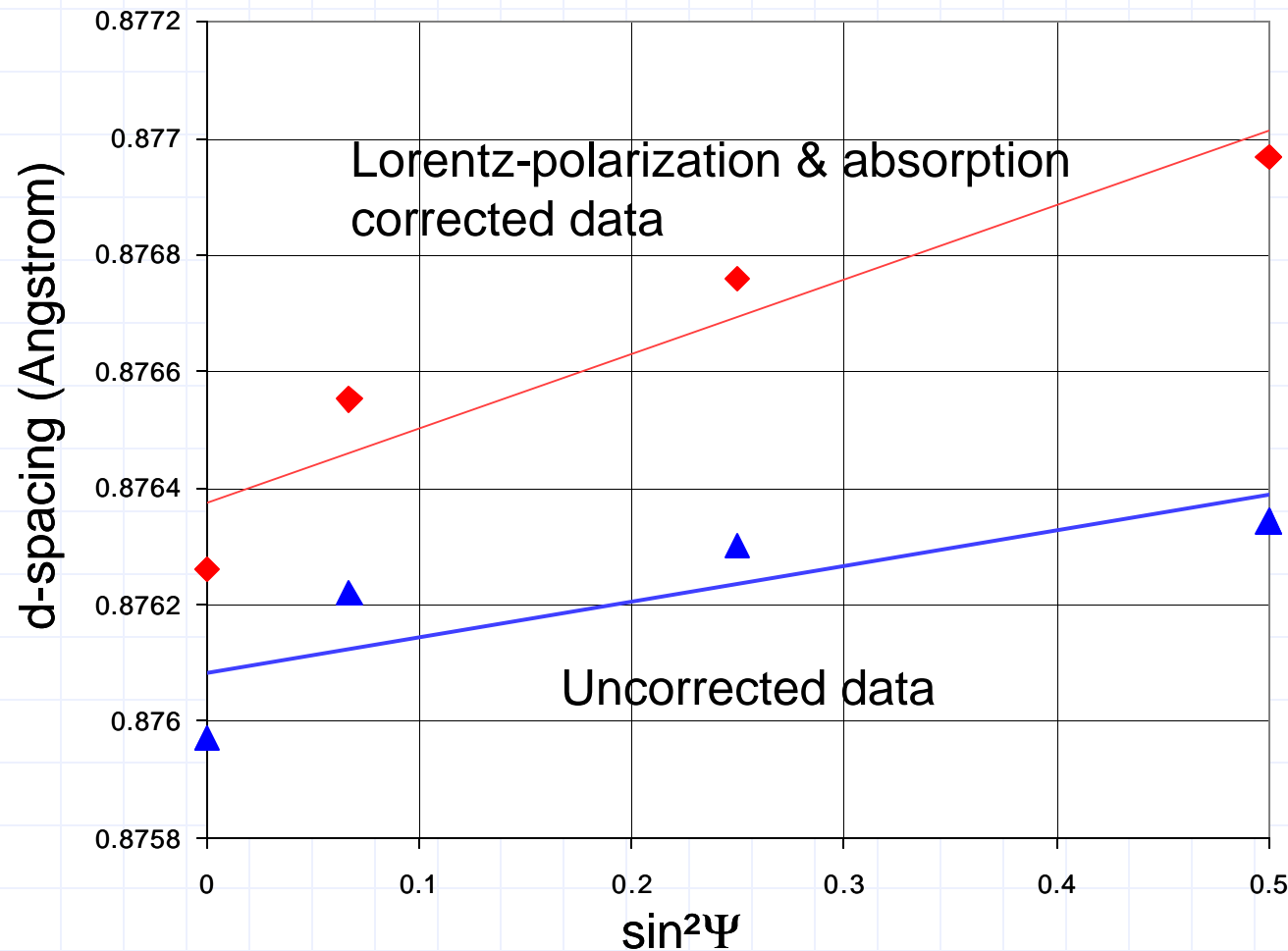
Measure  $d$  for a series of  $\Psi$  tilts to determine stress.

# X-Ray Residual Stress Results



- CuK $\alpha$  Radiation
- Peak shift to left

# Residual Stress in Ta<sub>2</sub>O<sub>5</sub> on AS 800



- Calculated residual stress: -39 MPa

- Measured residual stress:  $215 \pm 16$  MPa

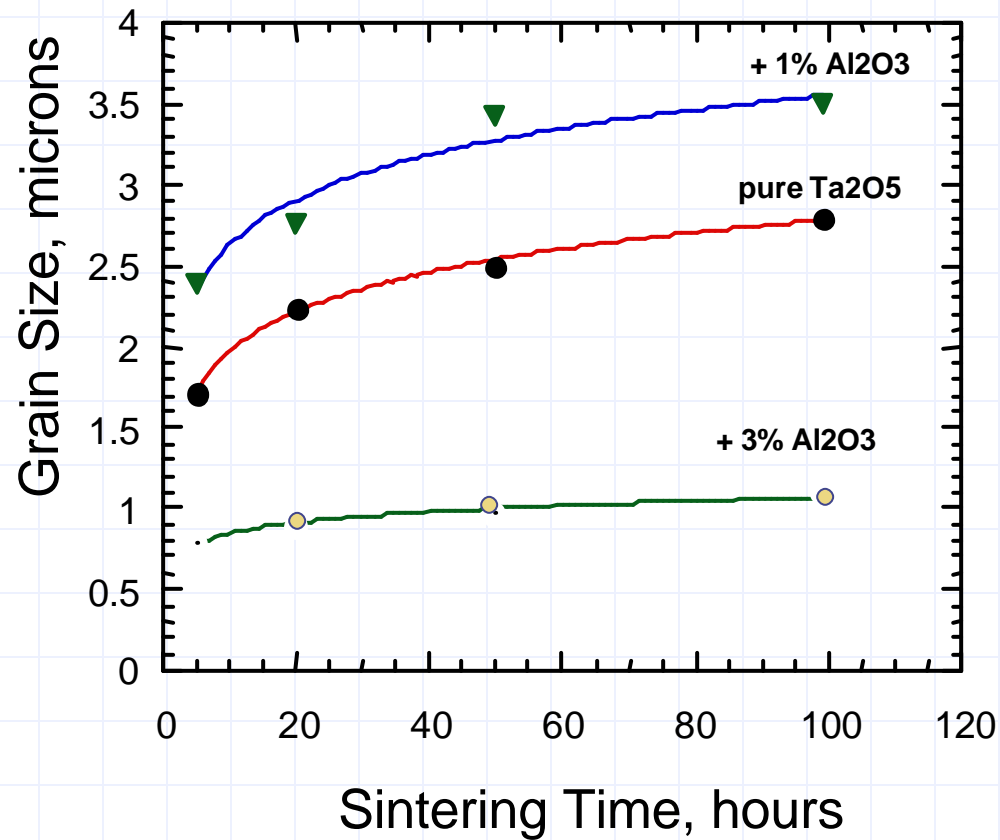
- Re-measure after Keiser Rig exposure.

# Ta<sub>2</sub>O<sub>5</sub> Alloys and Composites

- ◆ Use oxide additions for
  - limiting grain growth
  - stabilizing β-Ta<sub>2</sub>O<sub>5</sub>
- ◆ Size mismatch is critical
- ◆ Choices: Al<sub>2</sub>O<sub>3</sub> and La<sub>2</sub>O<sub>3</sub>
  - monitor solid solubility
  - monitor second phase formation

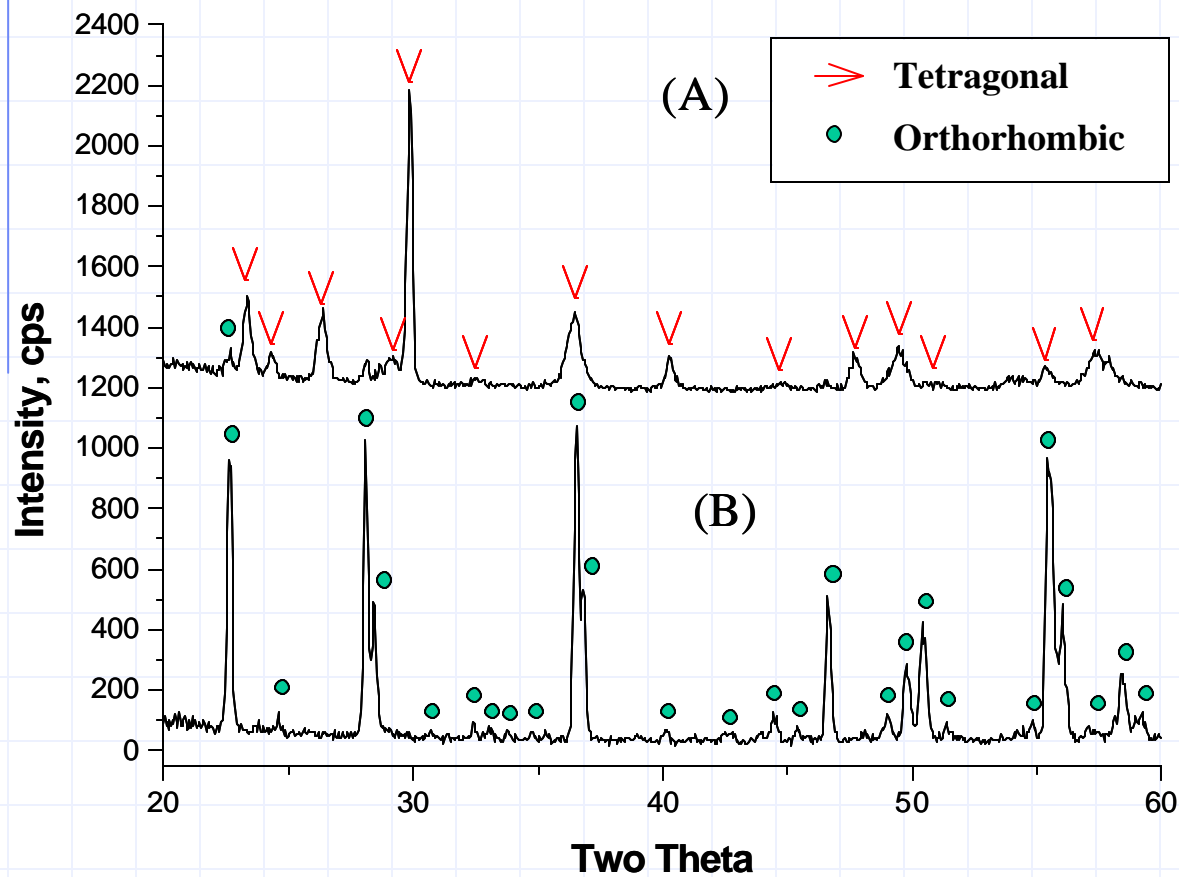
Ref: C.-W. Li, D. Raybould, L. Xue (Honeywell Inc.) Patent Pending

# $\text{Al}_2\text{O}_3$ Stabilizes Grain Size



Sintered at 1300°C in air.

# $\text{Al}_2\text{O}_3$ Stabilizes $\beta\text{-Ta}_2\text{O}_5$

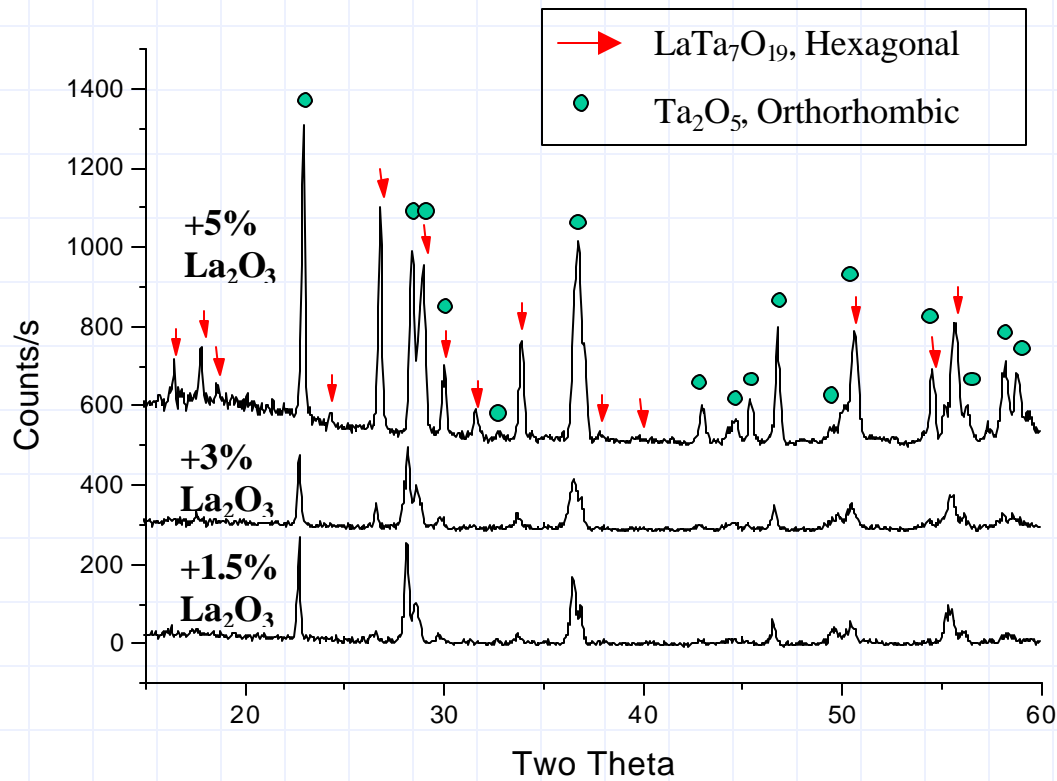


(A) Pure  $\text{Ta}_2\text{O}_5$ ,  
1360°, 5 h.

(B)  $\text{Ta}_2\text{O}_5$  with  
3%  $\text{Al}_2\text{O}_3$ ,  
1400°C, 5 h.



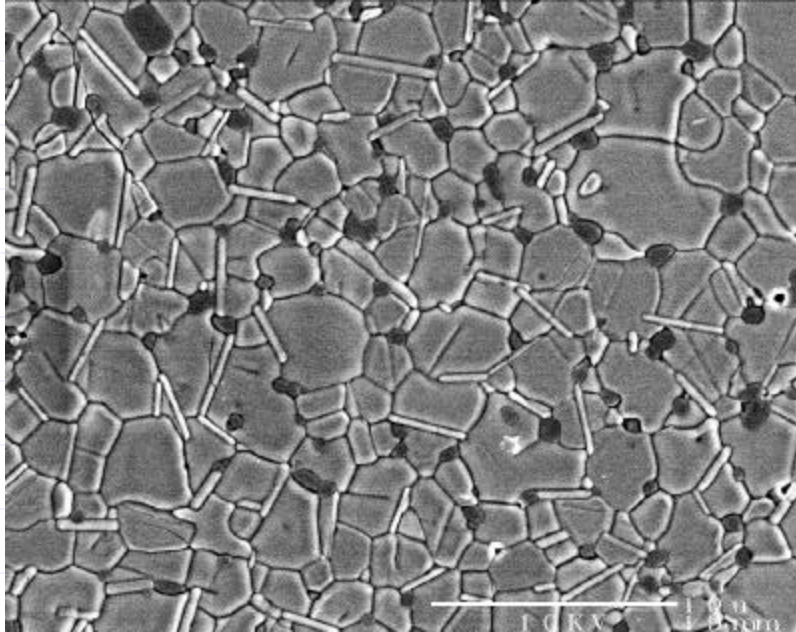
# La<sub>2</sub>O<sub>3</sub> Stabilizes β-Ta<sub>2</sub>O<sub>5</sub>



1400°C  
5 hours

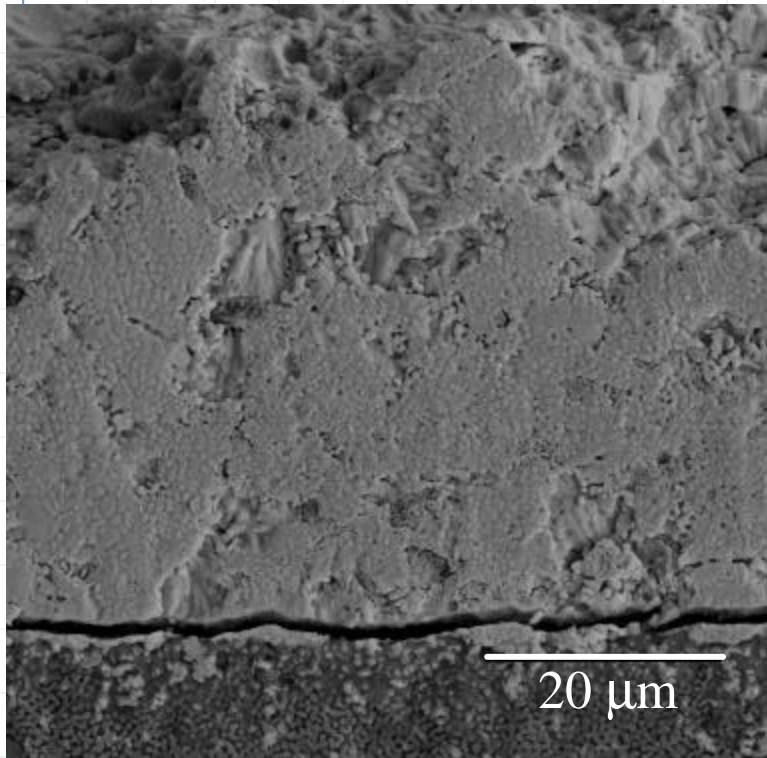
No traces of α-Ta<sub>2</sub>O<sub>5</sub> at concentrations > 3% dopant.

# Co-doping of $Ta_2O_5$ with $Al_2O_3$ and $La_2O_3$

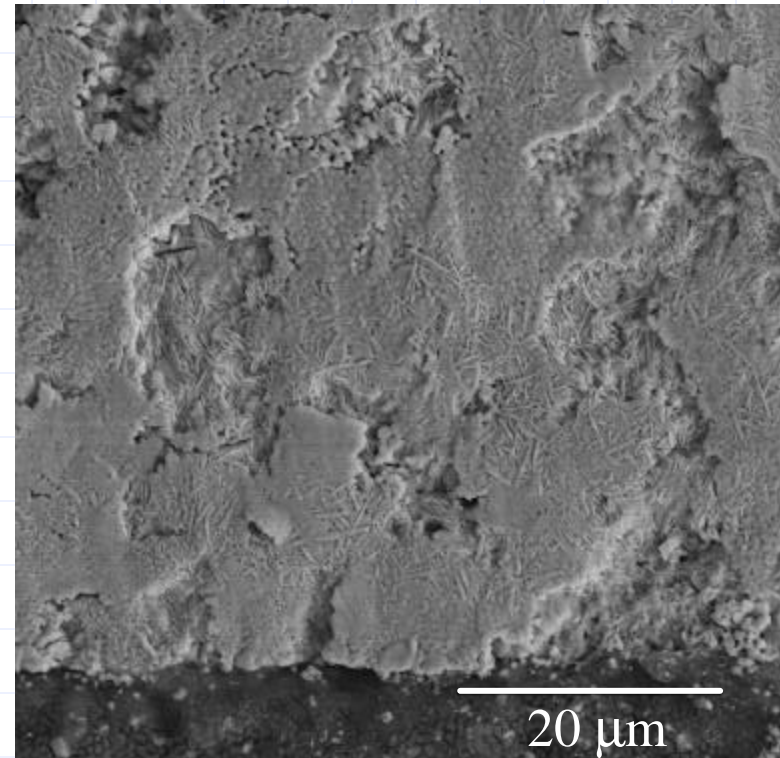


- ◆ 2%  $\gamma$ - $Al_2O_3$  and 1%  $La_2O_3$
- ◆ 95% Dense
- ◆ Needle-like grains:  $LaTa_7O_{19}$
- ◆ Dark, equiaxed grains:  $AlTaO_4$

# Alloys/Composites Translated to Plasma-Sprayed Coatings



3%  $\text{Al}_2\text{O}_3$  Addition



3%  $\text{La}_2\text{O}_3$  Addition

# Summary

- ◆  $\text{Ta}_2\text{O}_5$  shows promise as an interlayer in Honeywell's next generation EBC system for AS800 from thermal mismatch and compatibility considerations.
- ◆ Plasma-sprayed  $\text{Ta}_2\text{O}_5$  has been optimized for density for EBC applications.
- ◆ Alloys of  $\text{Ta}_2\text{O}_5$  shown promise for stabilizing the  $\beta$  polymorph and grain size.