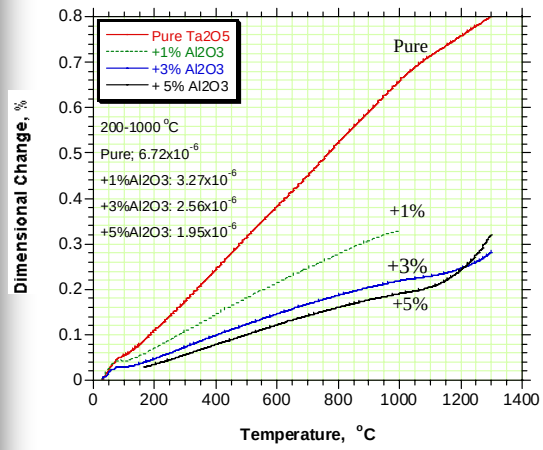
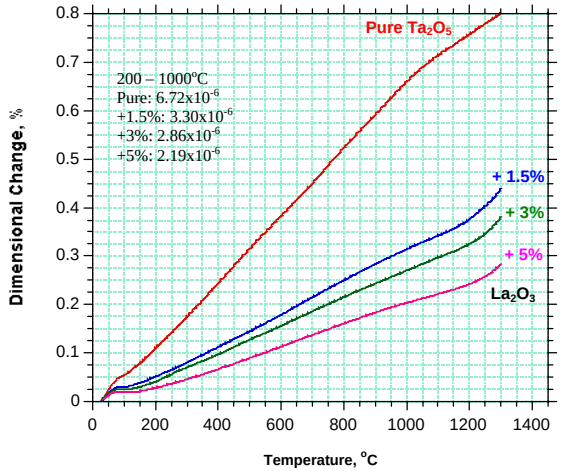
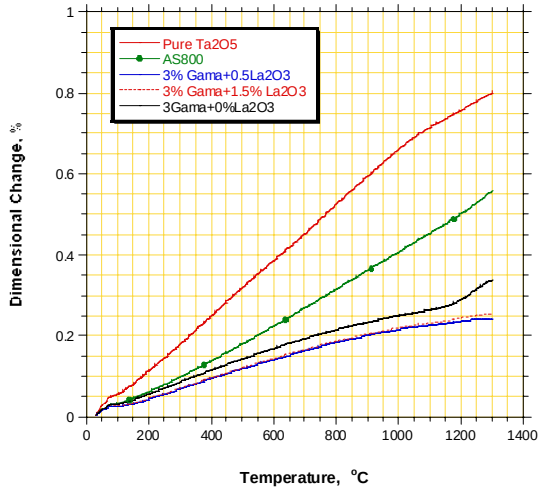
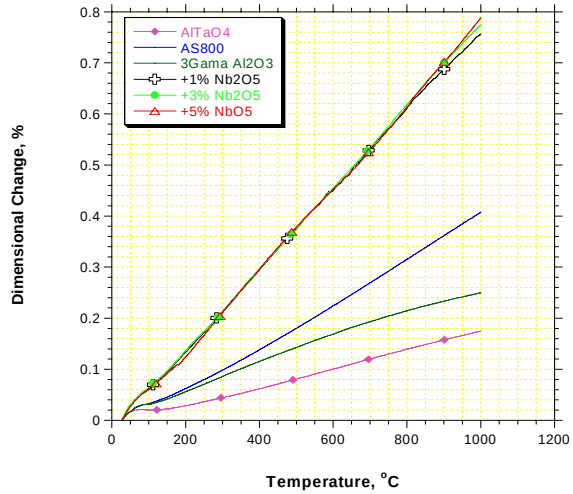
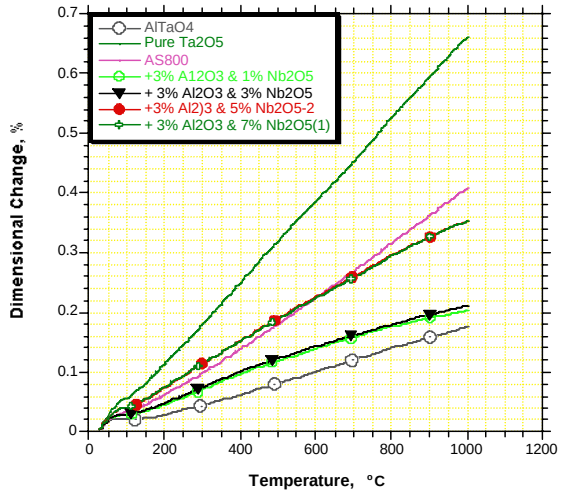
Alloy Additions Change the Thermal Expansion











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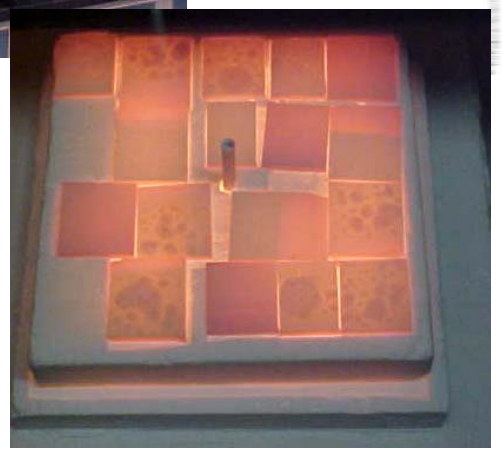
Life Limiting Phenomena: Thermal Cycling



Thermal cycling of SPPS'd coatings show certain coatings to be robust (and some not).

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Life Limiting Phenomena: Thermal Cycling

Summary of results:

- Approximately 4000 cycles logged on 30 coated & uncoated samples from limited # of thermal spray trials:
~2000 cycles at 1200C. ~2000 cycles at 1315C

- Spallation on the following samples:

  Pure Ta_2O_5

  Ta_2O_5+ 2w%Al_2O_3,

  Ta_2O_5+ 3w%Al_2O_3,

  Ta_2O_5+5w%Al_2O_3 samples

- No Spallation seen on any of the following samples:

  Ta_2O_5+ 1.5w%Al_2O_3

  Ta_2O_5+ 1.5w%Al_2O_3 + 1.5w%La_2O_3

  Ta_2O_5+ 3w%Al_2O_3 + 3w%La_2O_3

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Life Limiting Phenomena: Keiser Rig Testing

Studies conducted to date indicate:

Pure-Ta_2O_5 is not an effective barrier for oxygen or water vapor transport @ 1200 or 1315C.

Initial SPPS Pure-Ta_2O_5 is not thermally stable at 1200C or 1315C. Changes in microstructure with exposure time were seen.

Results indicate that stand-alone SPPS pure Ta_2O_5 will have limited value as a EBC for Si_3N_4.

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Life Limiting Phenomena & Performance Testing
• Residual Stress Evaluation

X-ray techniques used to assess the changes in the coating residual stress stated before and after thermal cycling.

- Residual stresses present due to:
  - CTE mismatch between substrate and coating
  - Temperature differences between plasma stream and substrate
- Residual stress alters D-spacings,

  (and the Debye-Scherrer pattern ring shape)

Determine the D-Spacings from the ring pattern shape & determine the stress state assuming Hooke's law.

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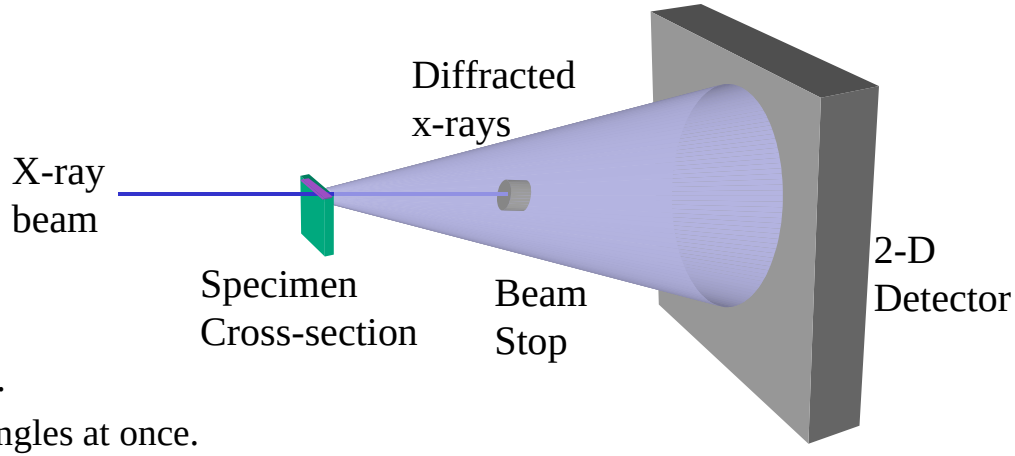
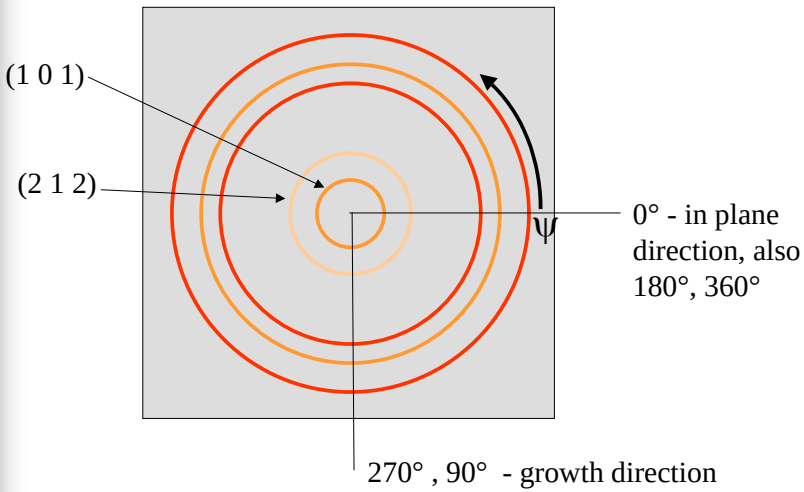
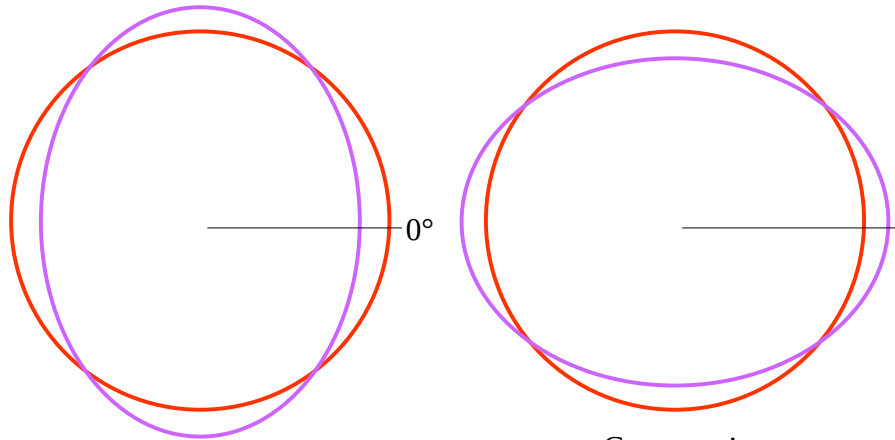
EXPERIMENTAL PROCEDURE - APS



- Specimens sectioned to 0.5-2 mm thick.
- Transmission experiment with 80 keV x-rays.
- 2-D detector - allows for collection of all ψ angles at once.
- Each specimen was probed through the thickness of the coating and into the substrate.



Shift of rings from circles to ellipses indicates lattice strain



Tensile
residual stress

Compressive
residual stress

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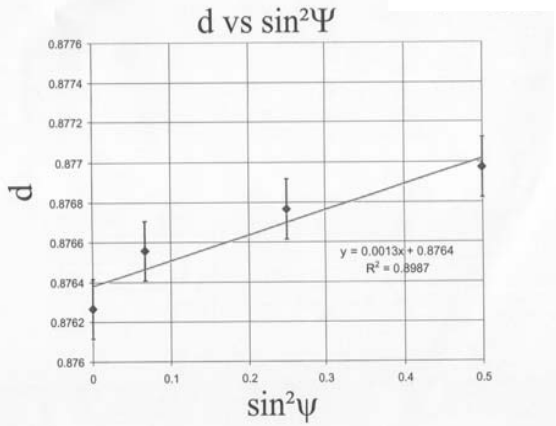
For Biaxial Stress State:

$$\frac{d_{\phi\Psi} - d_O}{d_O} = \frac{1+\nu}{E}\sigma_\phi \sin^2\Psi - \frac{\nu}{E}(\sigma_{11} + \sigma_{22})$$



Solving for σ gives Residual stress

Stress State seen to change as a function of Exposure conditions.

Composition	Preheat	Heat Treat	Stress (MPa)	+/- Error
Pure	900	As-Sprayed	187.22	16.38
Pure	900	139 Cycles	-258.11	32.31
Pure	900	489 Cycles	-121.1	28.79
Pure	900	986 Cycles	-245.61	34.09
Pure	900	1074 Cycles	-22.83	33.2
Pure	450	139 Cycles	-208.3	28.4
Pure	450	489 Cycles	-240.79	31.73
Pure	900	72 Hours Static	71.58	18.92
Pure	900	168 Hours Static	-152.96	18.92
2% Al2O3	900	As-Sprayed	251.57	13.96
2% Al2O3	900	145 Cycles	372.14	22.35
2% Al2O3	900	489 Cycles	-156.49	127.83
2% Al2O3	900	72 Hours Static	349.3	22.65
2% Al2O3	900	168 Hours Static	-171.32	46.12
3% Al2O3	900	As-Sprayed	227.75	10.26
3% Al2O3	450	As-Sprayed	-18.4	13.17
3% Al2O3	900	72 Hours Static	351.97	39.51
3% Al2O3	900	168 Hours Static	190.01	15.69

Table II: Residual stresses in various tantalum oxide based EBCs. Heat treatments were at 1200°C in air with cycles of 25 minutes at temperature and 5 minutes fan cooling.

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Summary of Current Program Findings

- Methods to successfully plasma spray Ta_2O_5 and Ta_2O_5-based alloys were developed. SPPS gives dense, adherent coatings.
- Ta_2O_5alloy compositions that stabilize β-Ta_2O_5 up to 1450C, limit grain growth, and match the CTE of Silicon-based ceramics were developed.
- SPPS coatings are capable of extended thermal cycling to 1200C and 1315C on AS800.
- Residual Stress Changes are seen to occur as a function of exposure time for SPPS'd Ta_2O_5-based coatings. Additional work is ongoing to understand and explain these observations.
- Keiser Rig testing has shown that stand-alone SPPS Pure-Ta_2O_5 coatings undergo changes during exposure and allow substrate changes to occur. The evaluation of the performance of Ta2O5-based alloys showed similar results under a separate DOE program. Therefore, it seems likely that use of Ta2O5 and Ta2O5-based alloys for EBCs will only be as part of a multi-layer coating system.

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