

# PHASE VOLUME FRACTION COMPARED BETWEEN PS-PVD & EB-PVD **AFTER THERMAL CYCLING VIA X-RAY DIFFRACTION** MAXWELL SMITH<sup>1</sup>, ZACHARY STEIN<sup>1</sup>, MATTHEW NORTHAM<sup>1</sup>, LIN ROSSMANN<sup>1</sup>, BROOKE SARLEY<sup>1</sup>, JUN-SANG PARK<sup>2</sup>, PETER KENESEI<sup>2</sup>, JONATHAN ALMER<sup>2</sup>, VAISHAK VISWANATHAN<sup>4</sup>, BRYAN HARDER<sup>3</sup>,

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# Motivation

Thermal barrier coatings (TBCs) are applied to turbine blades to protect them from operating temperatures up to 1200 °C. The standard application method is electron beam physical vapor deposition (EB-PVD). While effective, this technique is expensive and has a slow deposition rate. Plasma-spray physical vapor deposition (PS-PVD) is a promising technique that offers several advantages over EB-PVD[1], primarily lower cost, faster deposition rate, and non-line of sight deposition. This study investigates the rate of formation of monoclinic zirconia during thermal cycling, which negatively impacts the coating lifetime. If PS-PVD can be shown to have sufficient durability, it will replace EB-PVD for a fraction of the cost.



# Objectives

- Determine phase volume fraction of tetragonal (t), monoclinic (m), and cubic (c) phases present in uncycled, 300, and 600 thermal cycles samples of EB-PVD and PS-PVD.
- Compare rate of monoclinic phase growth between EB-PVD and PS-PVD.

# Background

- X-rays diffract off of lattice planes of crystals based on Braggs law.[2]
- From XRD data, the phases present in a identified and be quantified. Based on both intensity and peaks. interplanar spacina of transmission XRD Was Synchrotron utilized.
- (m) phase has greater volume than the c and (t) phases, causing cracking upon transformation.
- zirconia (YSZ) of a TBC.



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Tetragonal (t) Monoclinic (m)

Phase

Phase



- Samples in an infrared heater were thermally cycled (see diagram below) during XRD data collection using a  $30 \times 300 \mu m$  slit size.
- Scans were taken across two samples during a thermal cycle, passing through all layers of the TBC system (see diagram) about once every second).
- Uncycled PS-PVD samples had a 1 hour heat treatment.
- Samples had been thermally cycled for either 0, 300, or 600 cycles, with 20 min hot time at 1100 °C and 10 min of cooling per cycle.
- Phase volume fraction was calculated using XRD intensities; equation for **(m)** phase shown below.

 $x_m = \frac{I_m(\overline{1}11) + I_m(111)}{I_m(\overline{1}11) + I_m(111) + I_t(101)}$ 



- Results and Discussion
- 100% (t) and (c) PVF was detected in EB-PVD as expected.[4]
- The (c) and (t) phases have similar unit cell dimensions, causing overlap in the spectral pattern. Their PVF results have been combined pending deconvolution.
- Overall the PS-PVD showed a decrease(-0.26%) in (m) phase formation from 1 to 300 cycles and increase (0.04%) from 300 to 600 cycles.





# EB-PVD steps.

- The failure.[5]
- Initial
- could relatively during 800°C).
- transforms to (t) phase.
- phase.[6]

separate their effects.





[1] Harder, Bryan J., and Dongming Zhu. "Plasma spray-physical vapor deposition (PS-PVD) of ceramics for protective coatings." (2011). [2] Speakman, Scott A. "Introduction to X-ray powder diffraction data analysis." Massachusetts Institute of Technology (2013).

[3] Seetha Raghavan et. all. "Mechanical properties of coatings by Electron-Beam Physical Vapor Deposition and Plasma-Spray Physical Vapor Deposition compared using Synchrotron X-ray diffraction" (2017)

[4] Kakuda, Tyler R., et al. "Evolution of thermal properties of EB-PVD 7YSZ thermal barrier coatings with thermal cycling." (2009)

[5] Lance, M. J., et al. "Monoclinic zirconia distributions in plasma-sprayed thermal barrier coatings." Journal of Thermal Spray Technology 9.1 (2000)

[6] Witz, Grégoire, et al. "Phase evolution in yttria-stabilized zirconia thermal barrier coatings studied by rietveld refinement of X-ray powder diffraction patterns." Journal of the American Ceramic Society 90.9 (2007)

Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357. This work is also supported by the Florida Space Grant Consortium.

SEM Imaging courtesy of Marion Bartsch and Liudmila Chernova of the German Aerospace Center (DLR).



Thermocouple



Thermal Cycles



### Conclusions

samples had negligible or no **(m)** phase and PS-PVD has on average 2% more (m) phase in all samples at all thermal cycling

associated volume increase contributes to stress that can lead to cracking and

All PS-PVD samples had m phase, in contrast to EB-PVD. high fraction of **(m)** be a result of the low temperature deposition (650

After cycling with 1100°C hold, the (m) PVF reduces as it

At 600 cycles extended exposure to high temperature caused formation of the (m)



Monoclinic Phase PS-PVD

## Future Work

• Deconvolution of the (t) and (c) phases should be done to

• Another important area of comparison between PS-PVD and EB-PVD is their response to infiltration of CMAS, a labmade mix of silicates that simulate the dust that enters aircraft engines and causes coating damage.

## References and Acknowledgements