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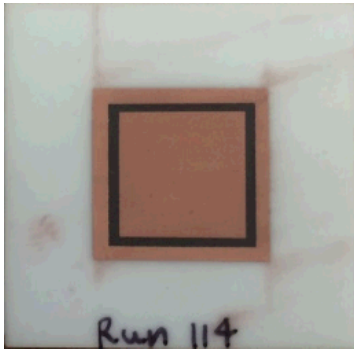

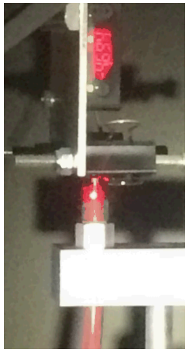

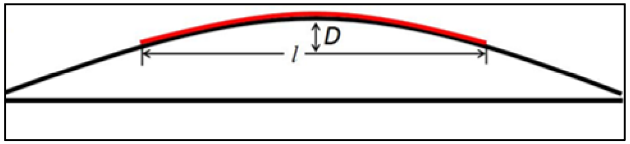

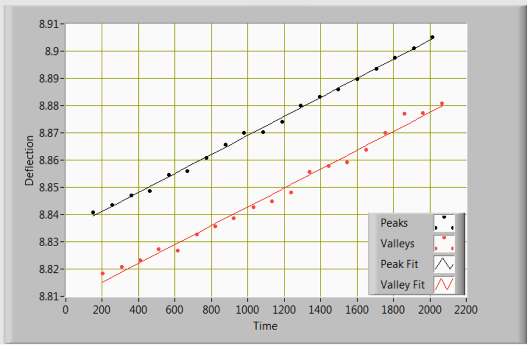

Residual Stress measurement of Aerosol Deposited Films

Andrew Vackel, Jesse Adamczyk, Tom Holmes, Andy Mayer, Pylin Sarobol

U.S. DEPARTMENT OF ENERGY
NNSA National Nuclear Security Administration

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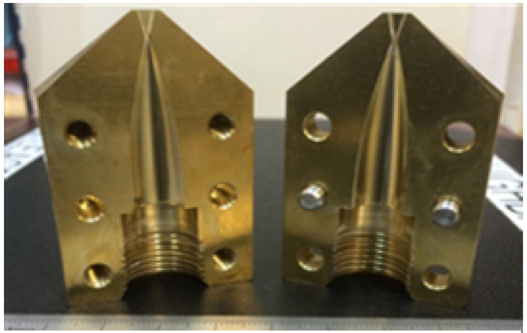
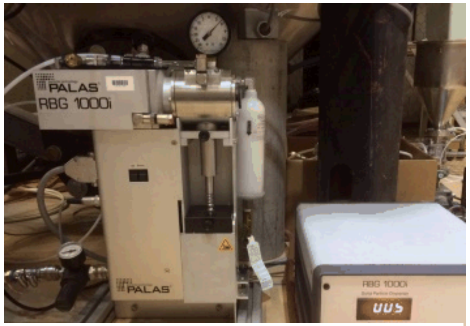
Aerosol Deposition (AD)

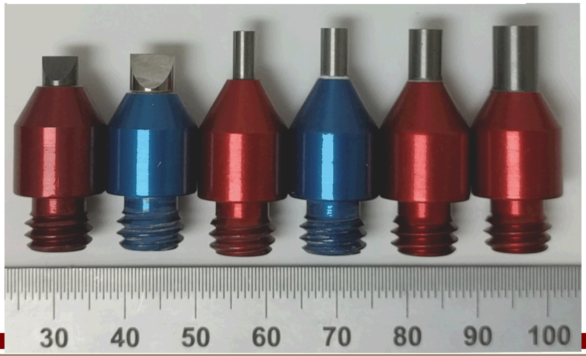
- Room temperature, solid state deposition process for thick films (>1µm)
- Relies on aerosolized fine powder accelerated to high speeds (>300m/s) within a vacuum environment for coating consolidation
- Capable of depositing ceramics, carbides, and metals with no feedstock melting or substrate heating
- Enabling integrations of high melting temperature and/or thermally sensitive materials

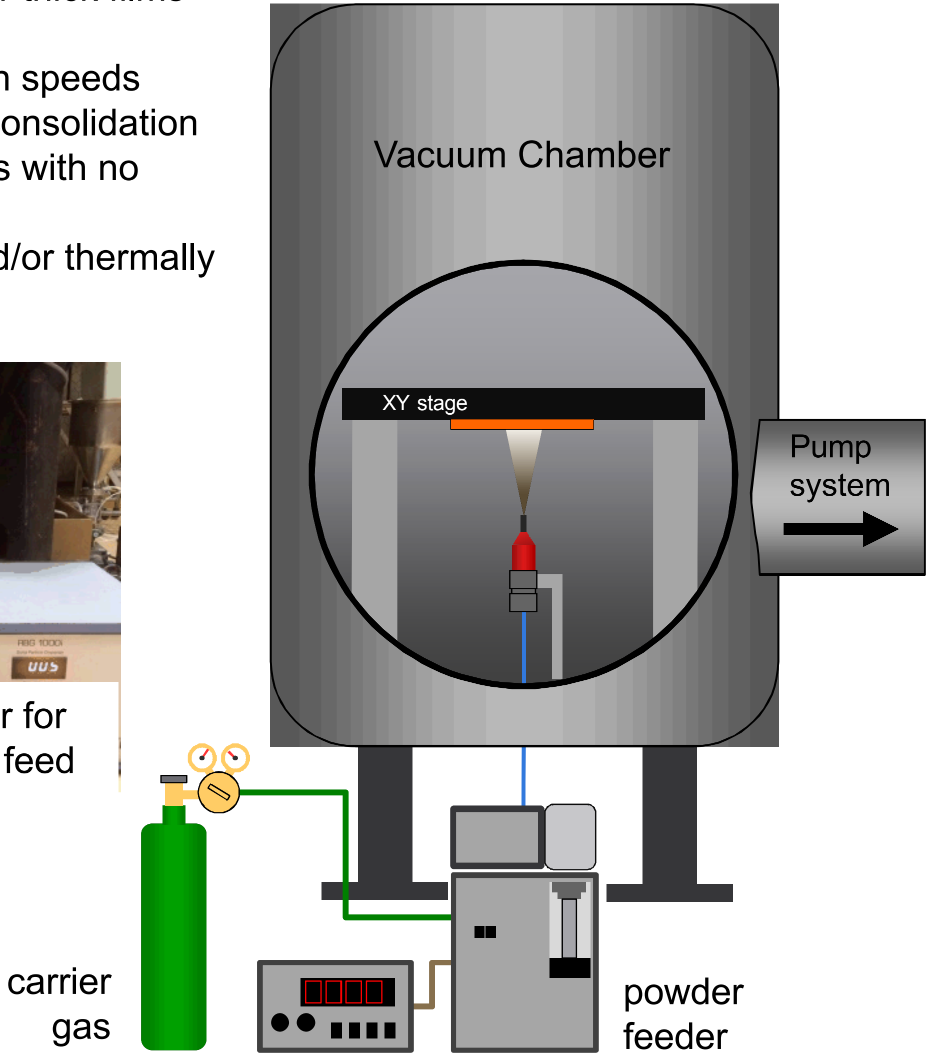


Nozzles for achieving supersonic particle flow



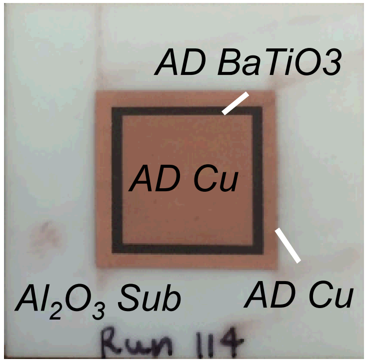
Aerosol Generator for controlled powder feed



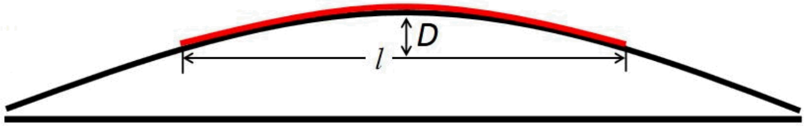


Residual Stress in AD films

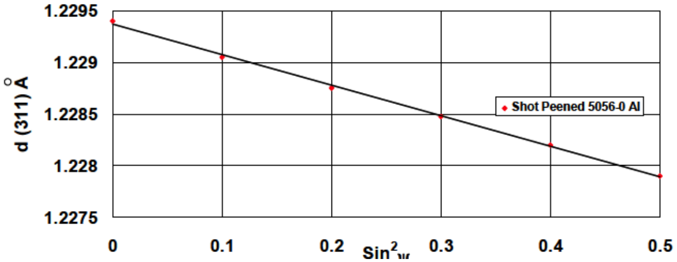
- Motivation for study
  - Multi-layer integration, device building utilizing AD films
  - Understand process parameter influence on stress, coating formation dynamics, and resultant properties
  - Additional method for accessing repeatability of process



- Common measurement techniques for thin/thick films
  - X-Ray diffraction (Ψ^2 method)
  - Neutron diffraction
  - Substrate Curvature – Stoney Formula
  - Layer removal



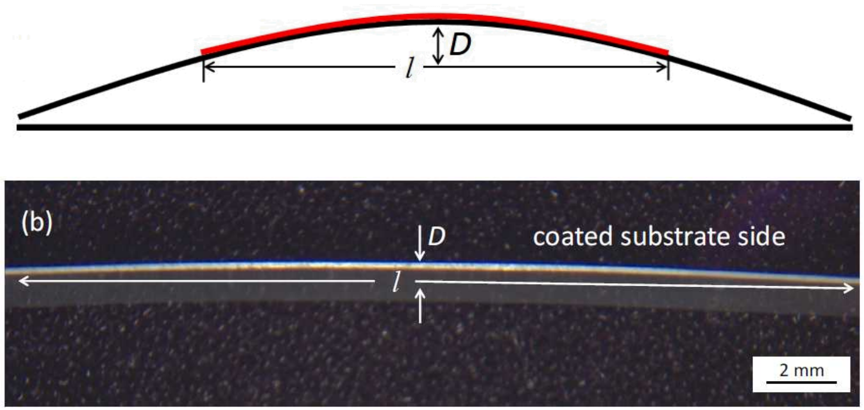
Schubert, Michael, Jörg Exner, and Ralf Moos. "Influence of carrier gas composition on the stress of Al2O3 coatings prepared by the aerosol deposition method." Materials 7.8 (2014): 5633-5642.



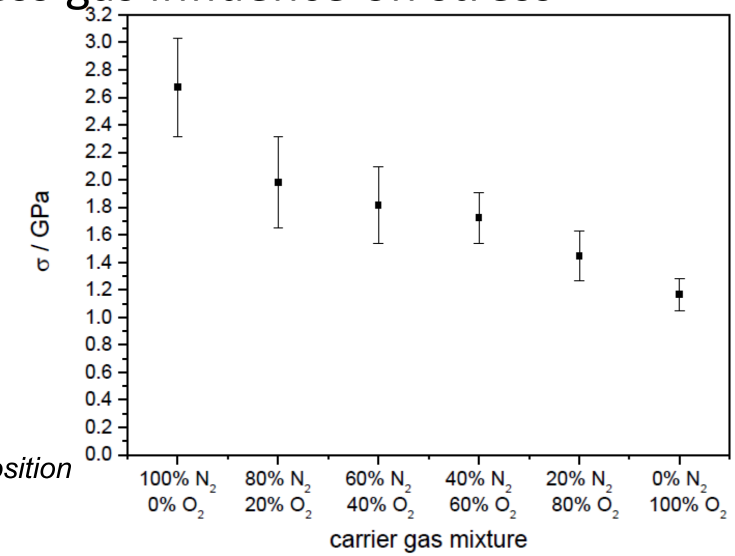
Fitzpatrick, M., T. Fry, and P. Holdway. "NPL good practice guide no. 52: determination of residual stresses by X-ray diffraction—Issue 2." *NPL, Great Britain* (2005).

Residual Stress in AD films: Literature Survey

- Curvature based measurements – Schubert et al.
  - Use of curvature to determine process gas influence on stress



Schubert, Michael, Jörg Exner, and Ralf Moos. "Influence of carrier gas composition on the stress of Al2O3 coatings prepared by the aerosol deposition method." Materials 7.8 (2014): 5633-5642.

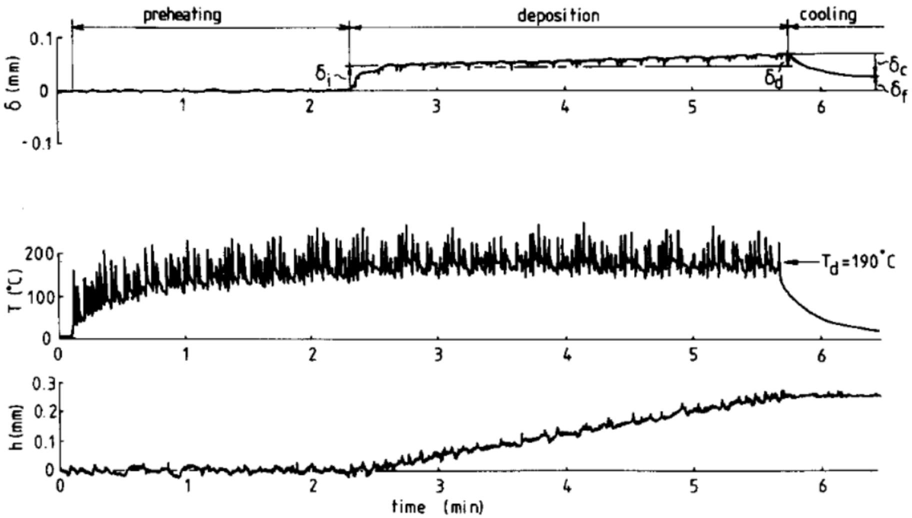


- XRD based measurements - Hoshina et al.
  - Determined 180-250 MPa of residual stress in BaTiO3 films

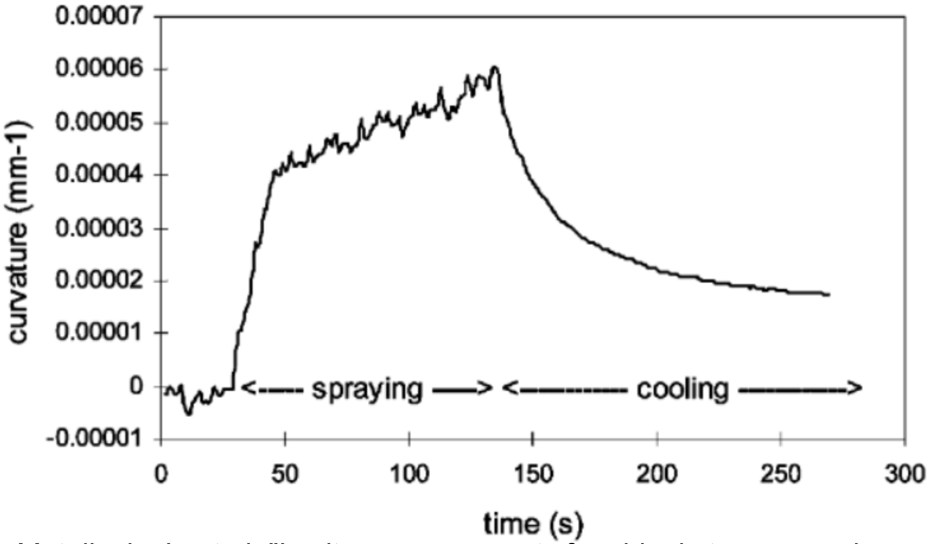
Hoshina, Takuya, et al. "Size effect of nanograined BaTiO3 ceramics fabricated by aerosol deposition method." Japanese Journal of Applied Physics 49.9S (2010): 09MC02.

In-situ Curvature Measurements in Thermal Spray

- Allows observation of coating dynamics in real time
- Discrete measurements of stress during deposition and cooling
- Now available as commercial tool

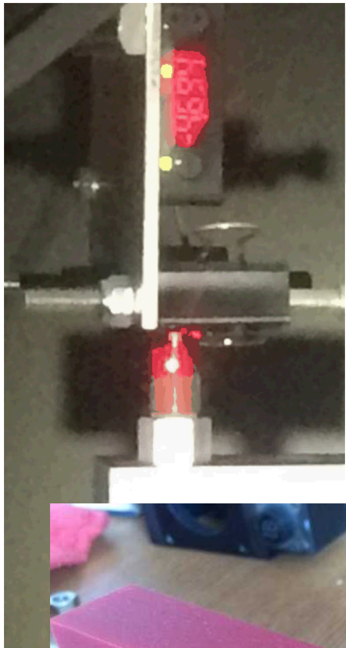


Kuroda, S., T. Fukushima, and S. Kitahara. "Simultaneous measurement of coating thickness and deposition stress during thermal spraying." Thin solid films 164 (1988): 157-163.



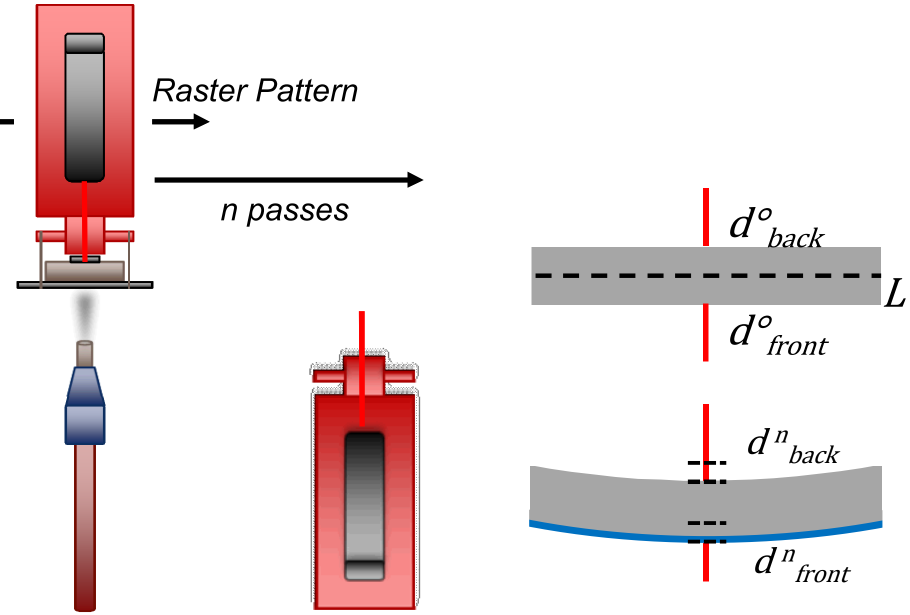
Matejicek, J., et al. "In situ measurement of residual stresses and elastic moduli in thermal sprayed coatings: Part 2: processing effects on properties of Mo coatings." Acta Materialia 51.3 (2003): 873-885.

Experimental setup for substrate deflection from AD films



Center Point : 30mm
Range : ± 4mm
Resolution: ± 1μm

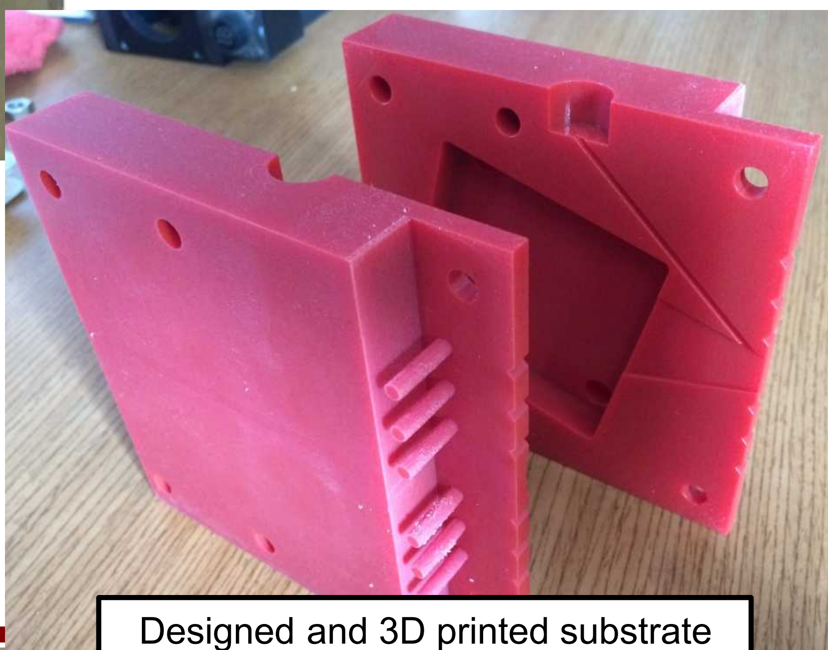
Preliminary measurements and feasibility



$$t^{n}_{film} = d^{o}_{front} - d^{n}_{front} - d^{n}_{back}$$

$$K^{n} = \frac{2\, dnfront}{{d^{n}_{front}}^{2} + \frac{L^{2}}{4}}$$

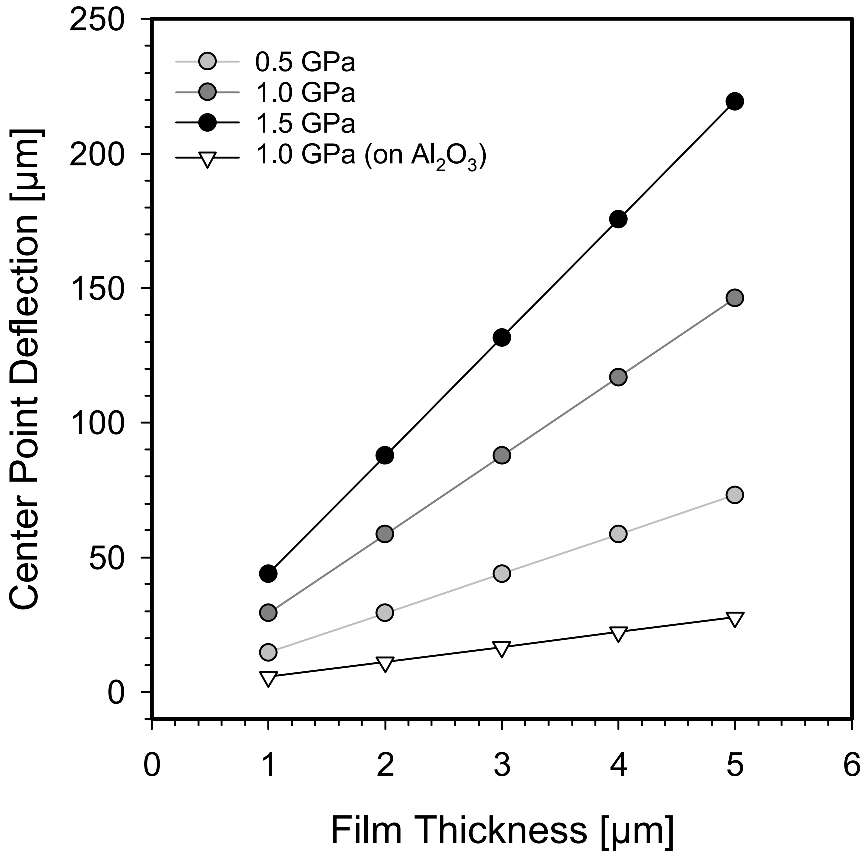
$$Stoney\ \sigma_{Film} = \frac{E'_{sub}\, {t_{sub}}^{2}\, (Kn - K^{\circ})}{6\, tnfilm}$$



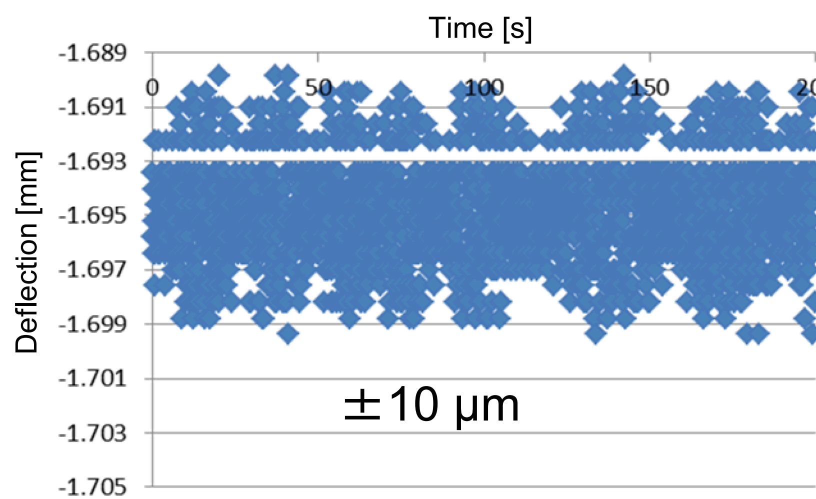
Designed and 3D printed substrate holder/laser measurement fixture

Expected substrate deflection in AD

- Back calculations of expected deflection based on expected film stress, substrate dimensions and stiffness, and film thickness

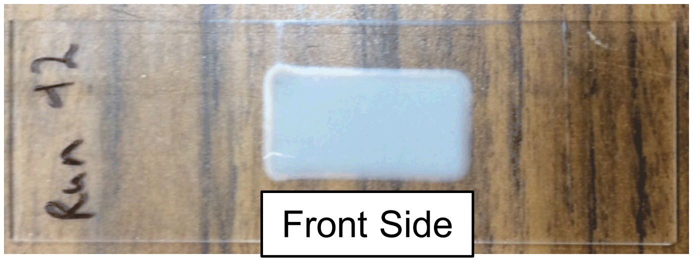


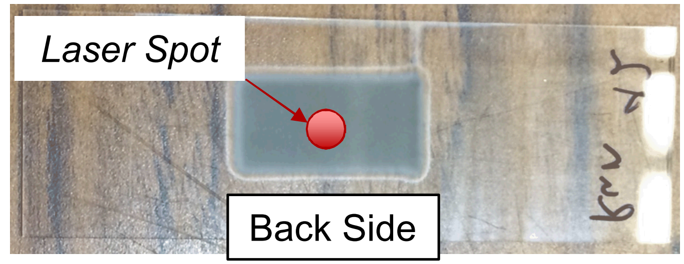
Laser measurement background noise



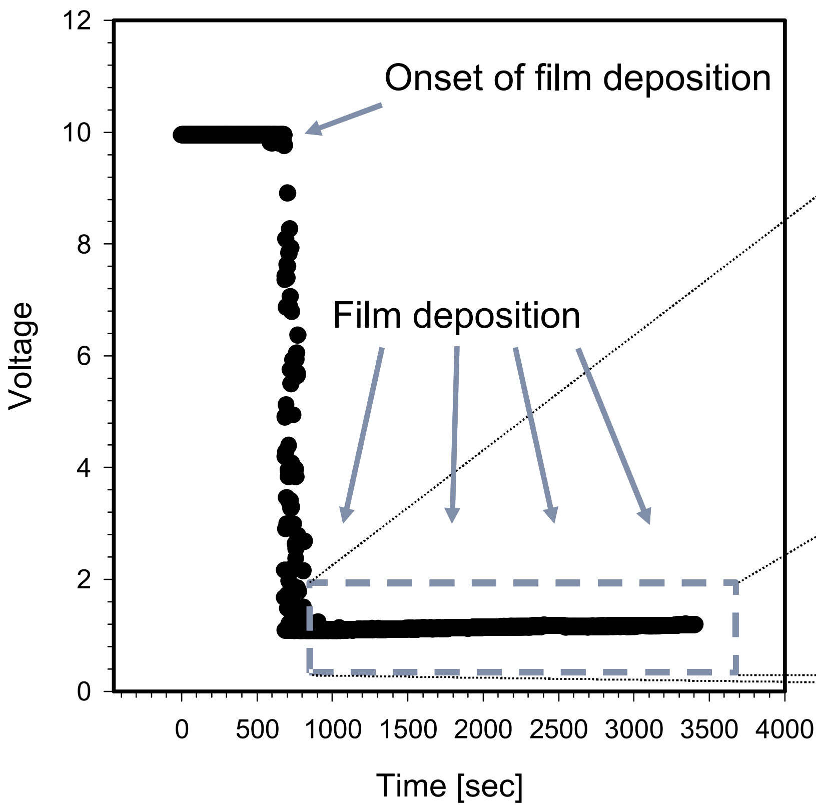
Based on 1mm thick soda-lime glass substrates

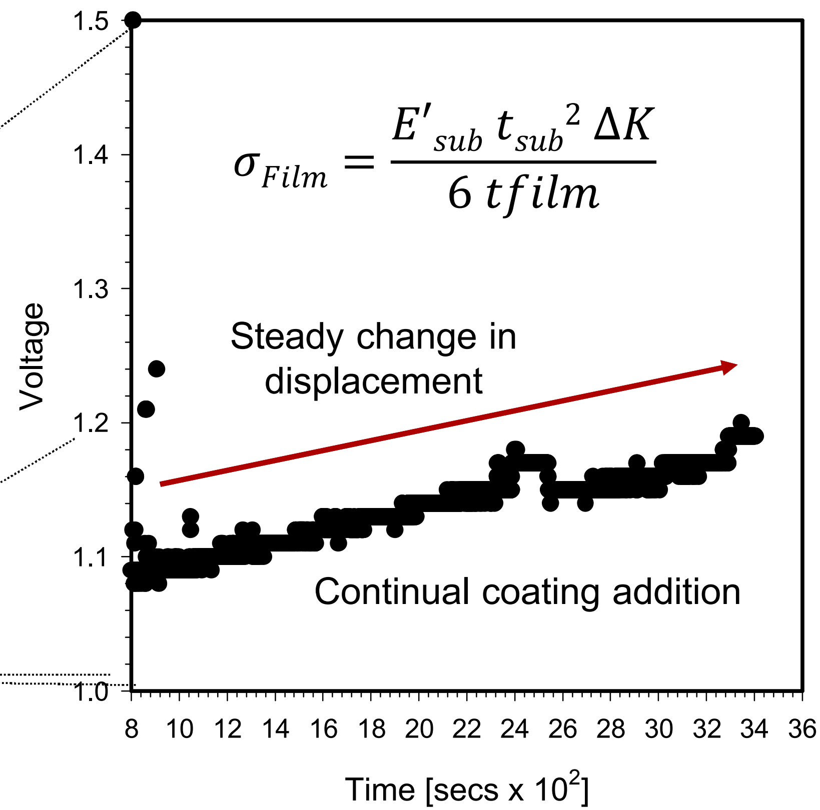
Preliminary Experimental Results – Deposition



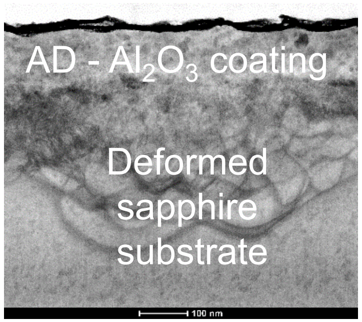


$BaTiO_3$ on 25 x 75mm Glass Slide



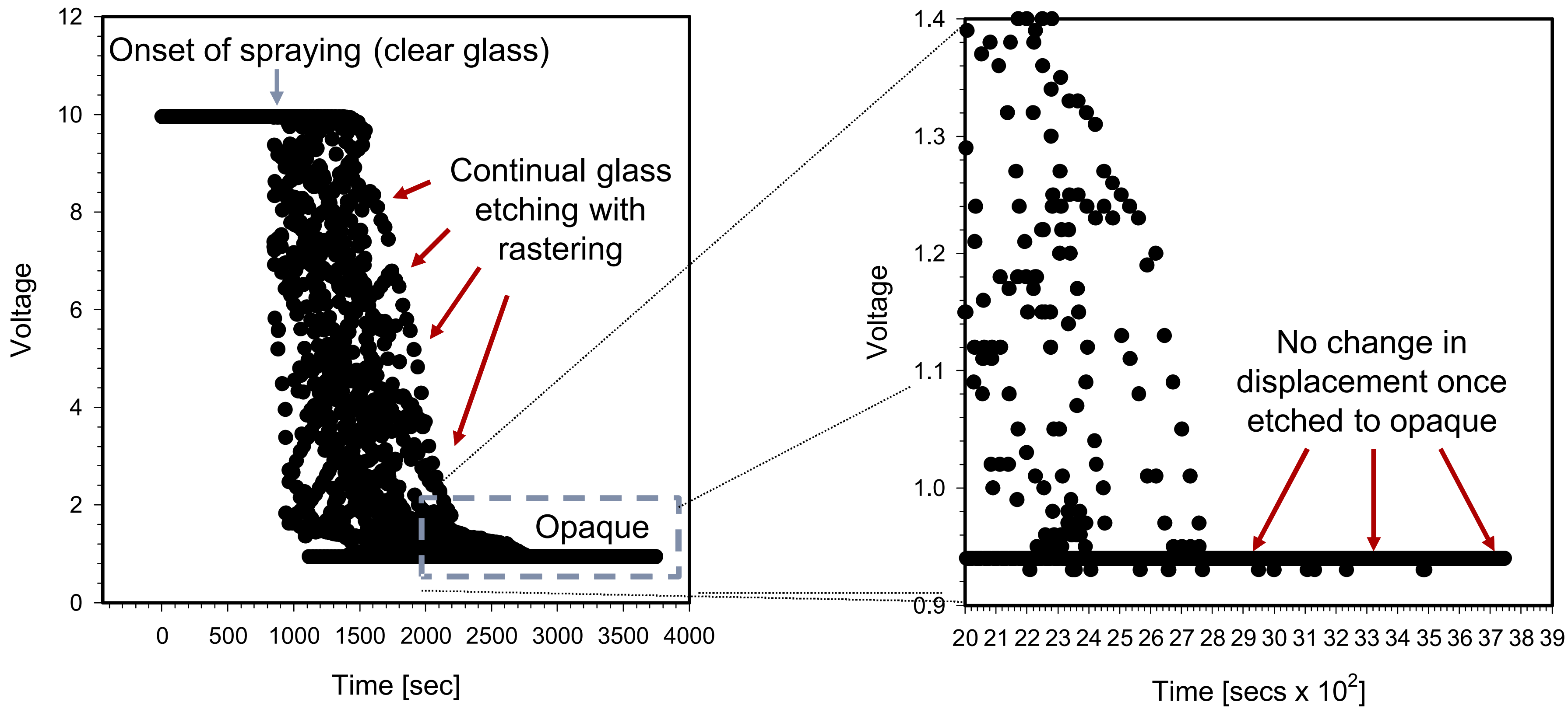


Preliminary Experimental Results – Etching

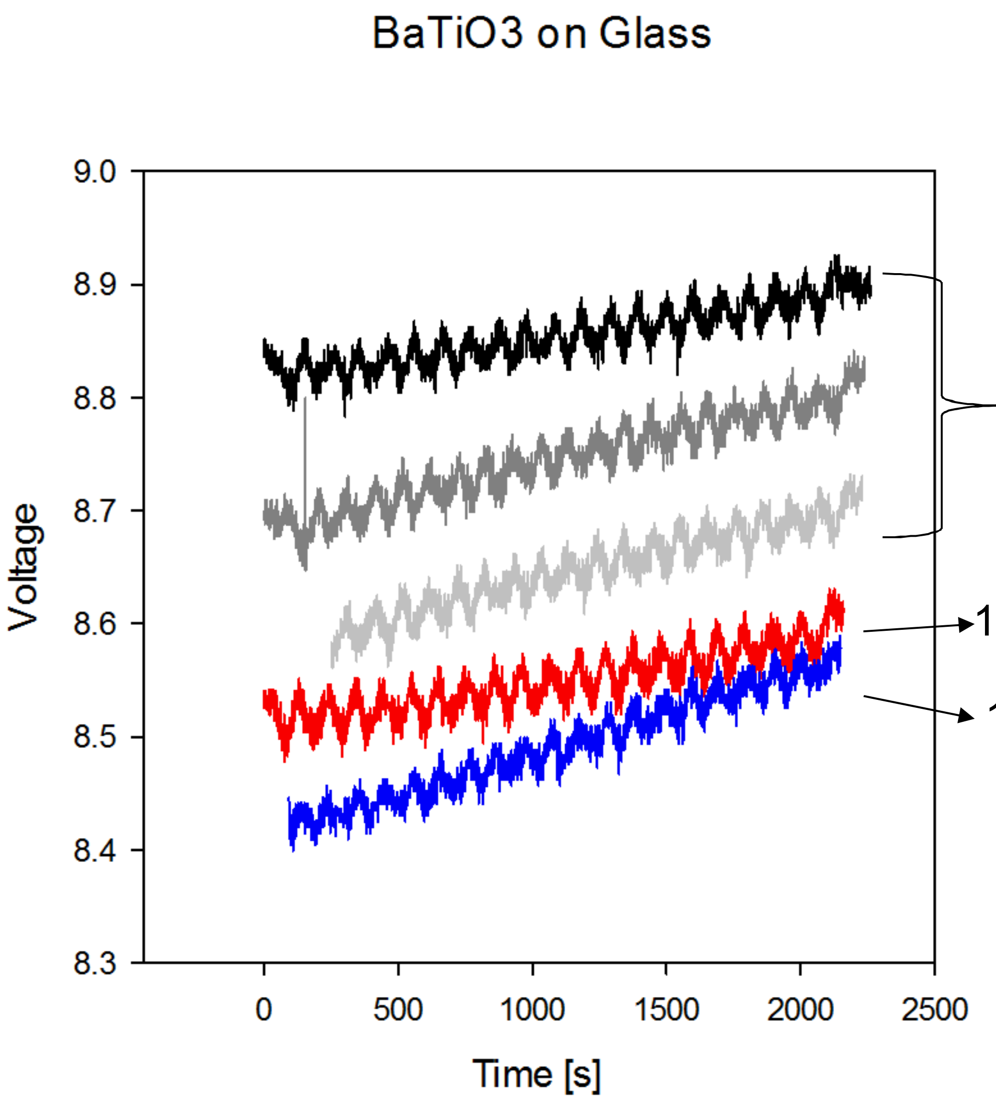


Al_2O_3 on Glass

- Highly damaging to substrate – Etches glass
- Self limiting film growth – Requires harder substrate for deposition

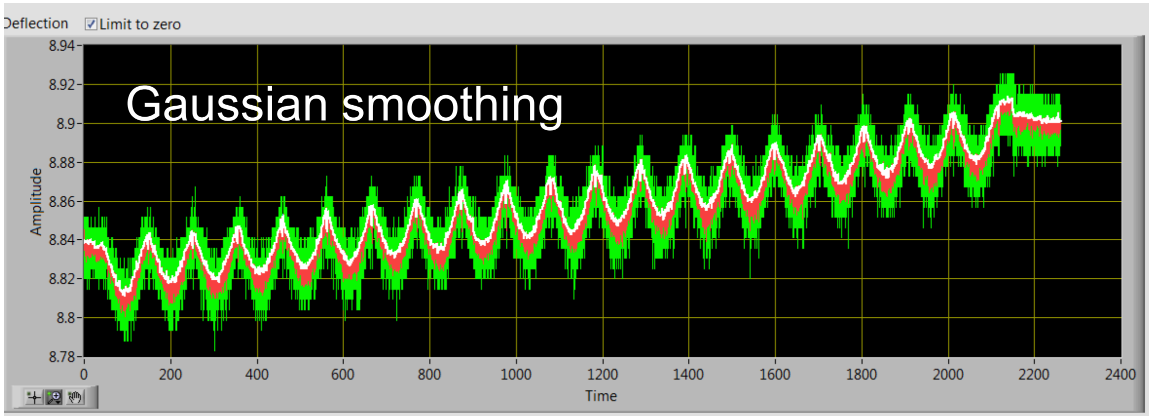


Substrate Deflection Measurements of AD Deposition

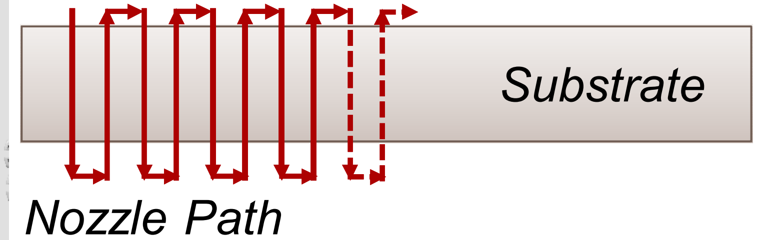


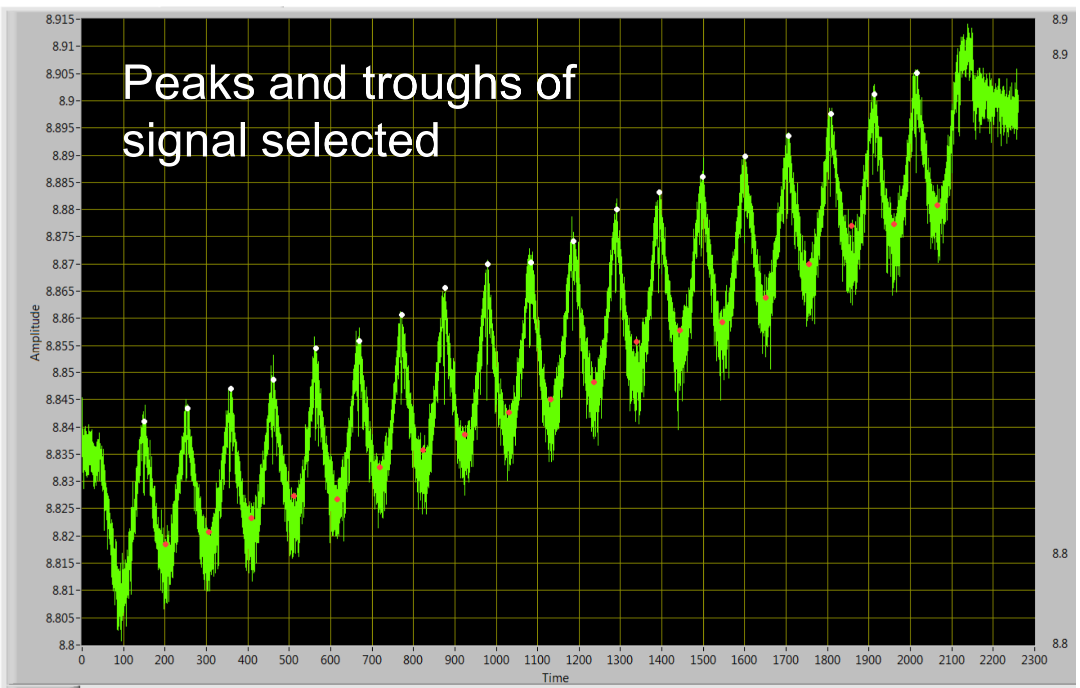
- 1 Torr Chamber pressure
- ~3-4 μm Coating thickness
- Glass Slide Substrates (60mm x 12.5mm)

Substrate Deflection Measurements of AD Deposition – Analysis



Spray path over substrate creates undulating deflection signal

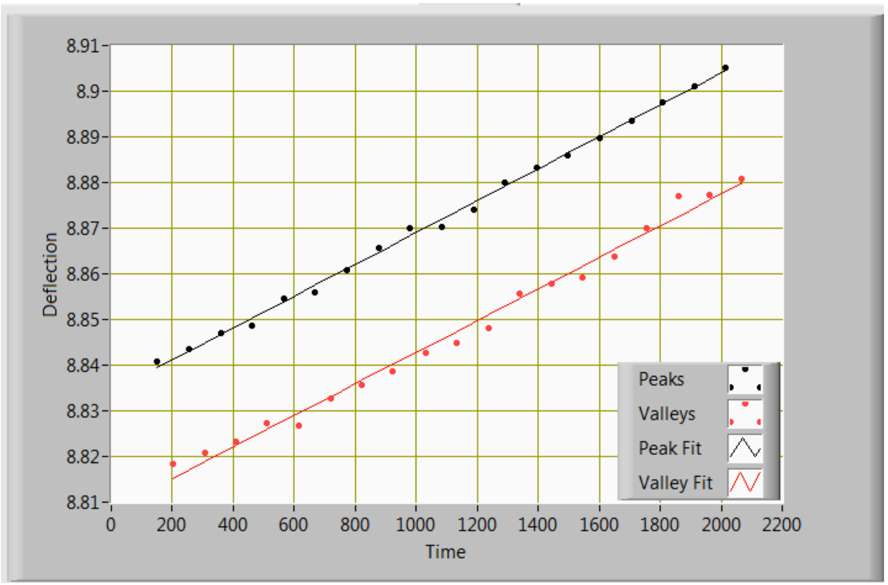


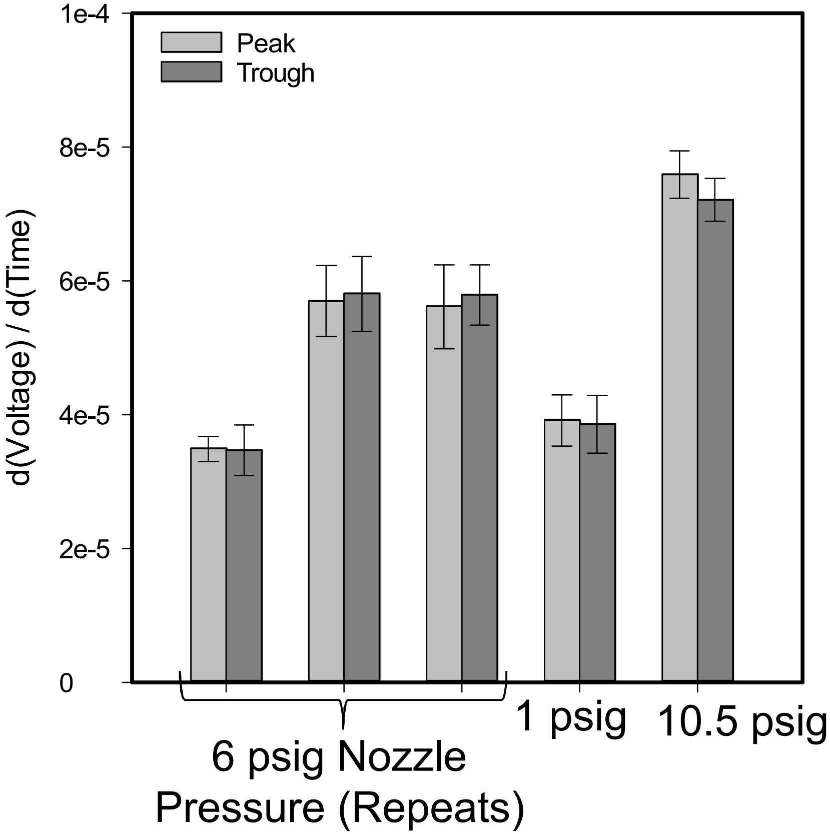


Substrate Deflection Measurements of AD Deposition – Analysis

Plot of Peak Heights and Troughs

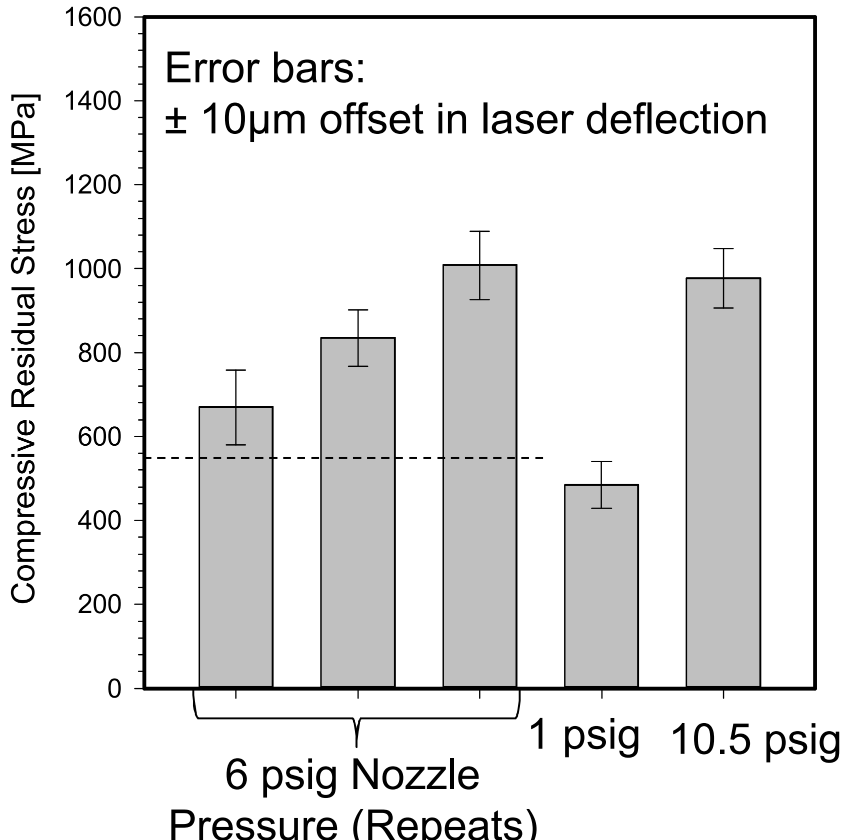
- Linear Accumulation of Deflection with Film Growth
- Little divergence between peak height and trough

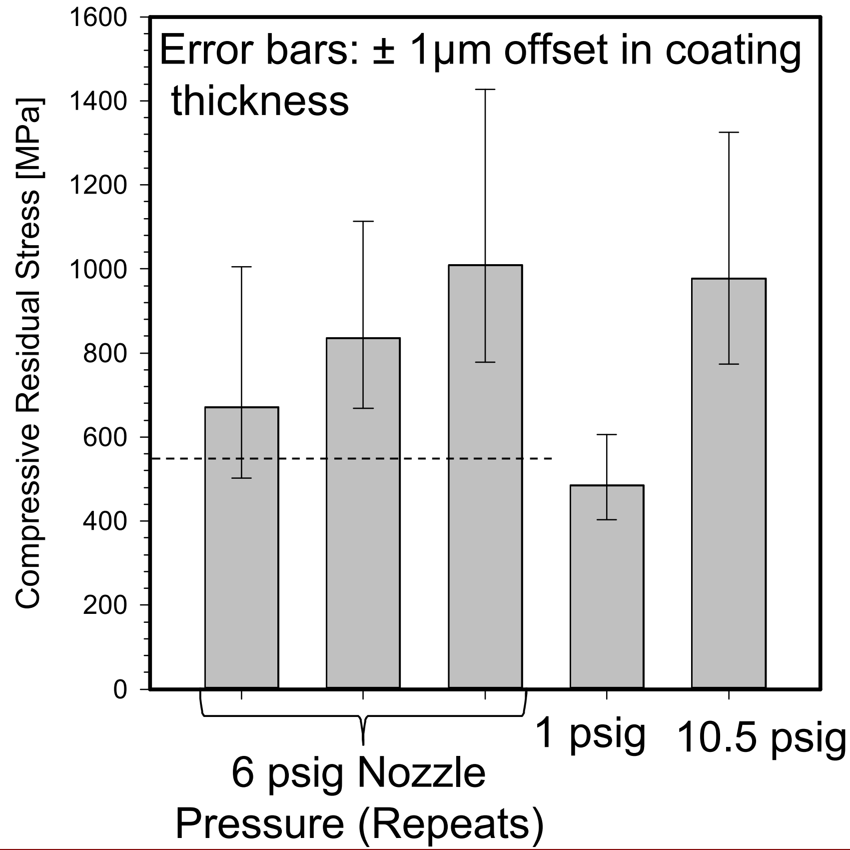




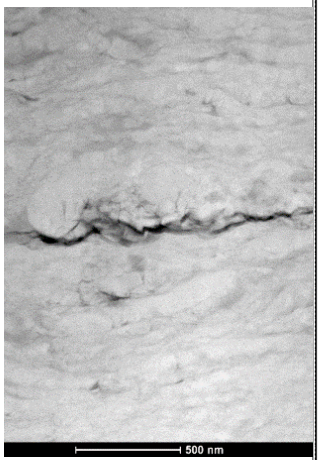
Stress Calculations

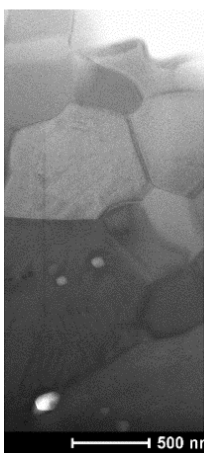
- Prone to error – laser measurement and coating thickness
- Limited repeatability

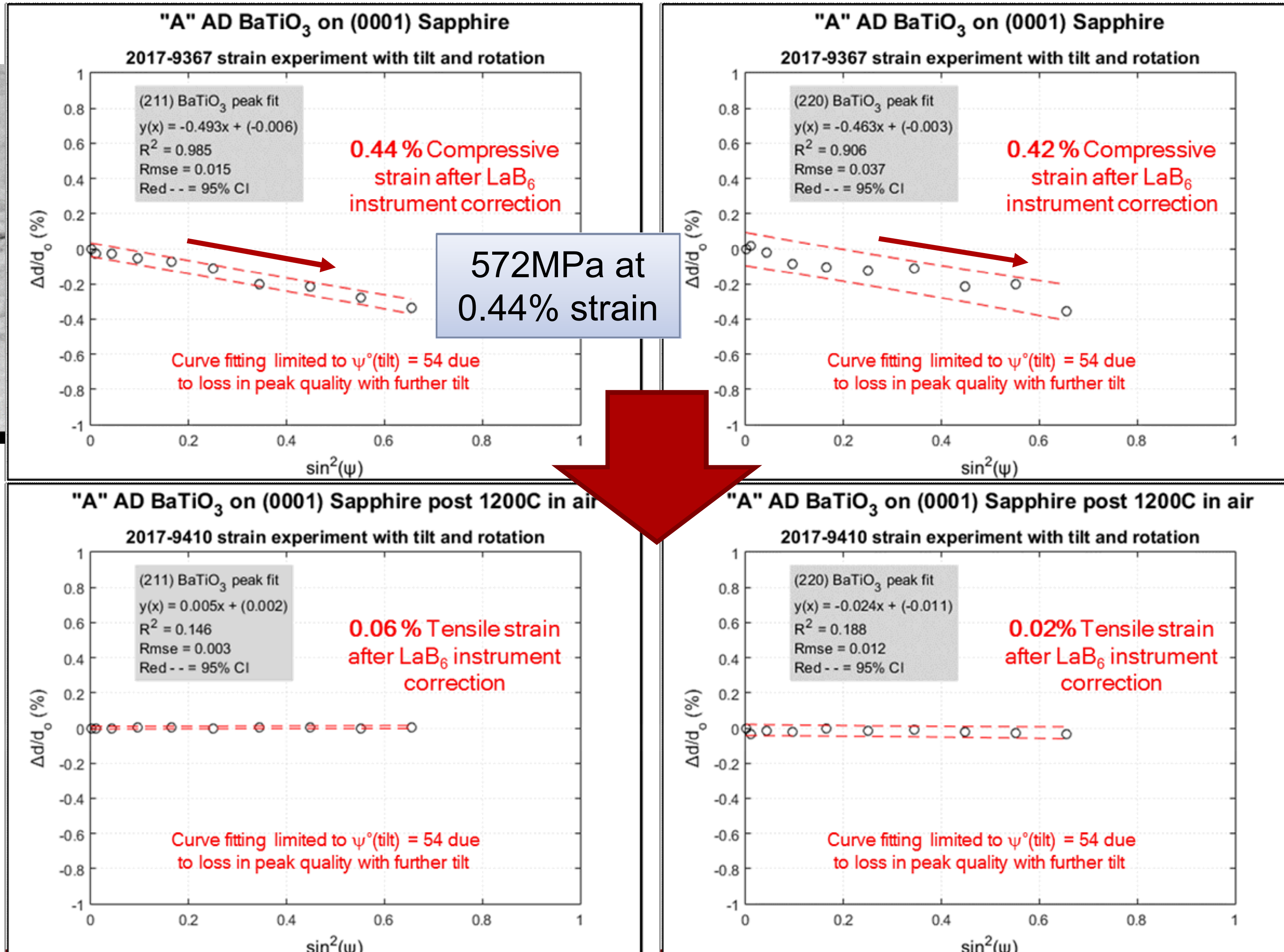




Stress Calculations



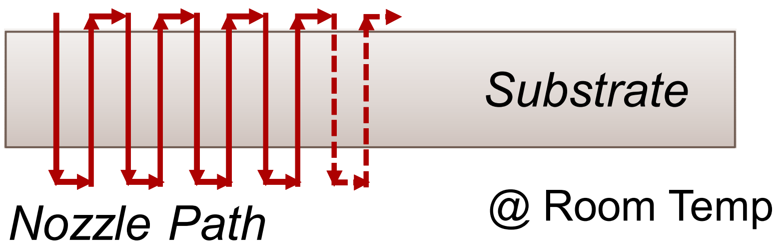




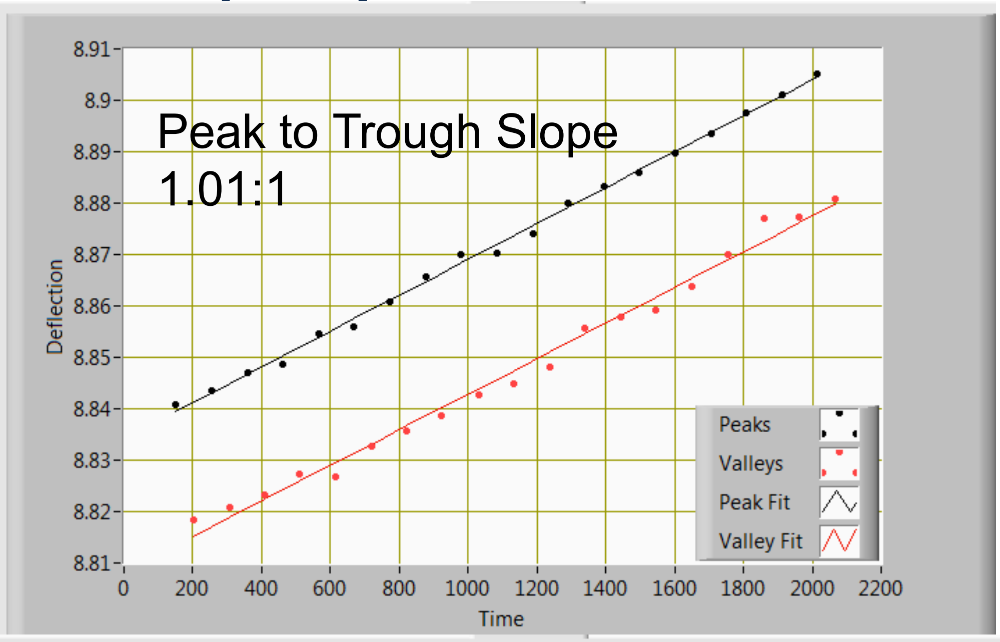
(211) (220)

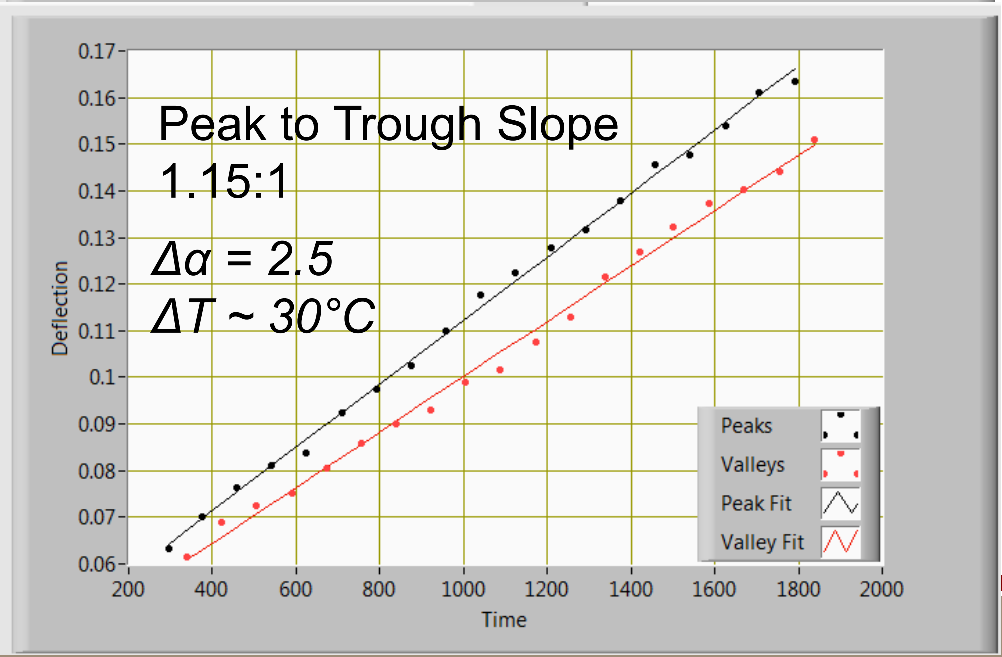
Comparison to Thermal Spray

- AD has linear deflection accumulation with film growth
- Peak and Trough of deflection data correspond to raster pattern



- TS has linear deflection accumulation with film growth
- Peak and Trough of deflection data diverge with film growth due to CTE mismatch and temperature undulations with each layer addition





Summary

- AD Films under considerable compressive stress
- In-situ substrate deflection measurement possible
  - Linear deflection accumulation with film growth
  - No fluctuations from temperature observed
  - Somewhat responsive to parameter differences
- With better measurement resolution, possibly a more rapid way to measure film stress
- Enables further in-situ investigation of films based on process parameters (gas pressures, nozzle geometries, stand off distance etc.)
- Enables in-situ detection of film growth, delamination

Thank you - Question?

extras

$$\sigma = \frac{4Ed^2}{3(1-\nu)l^2} \cdot \frac{D}{t}$$