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Aerosol Deposition and characterization of $BaTiO_3$ films

PRESENTED BY

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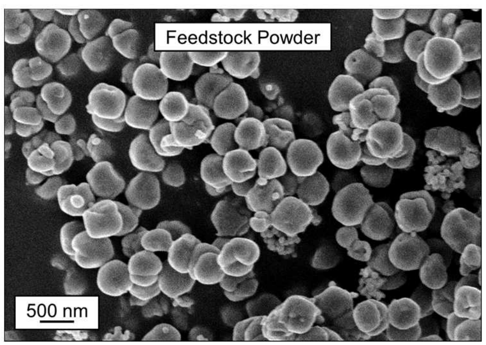
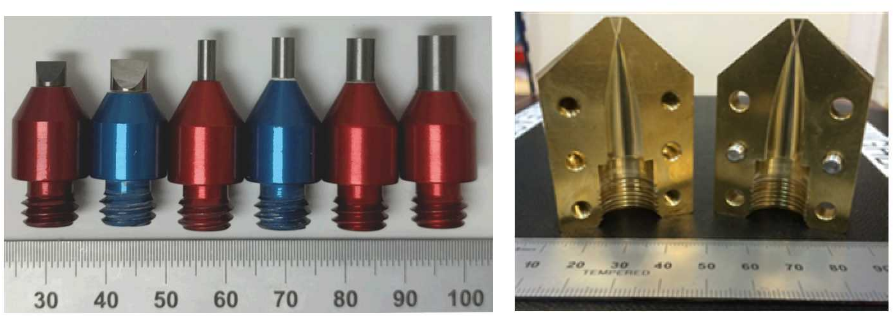
Aerosol Deposition (AD)

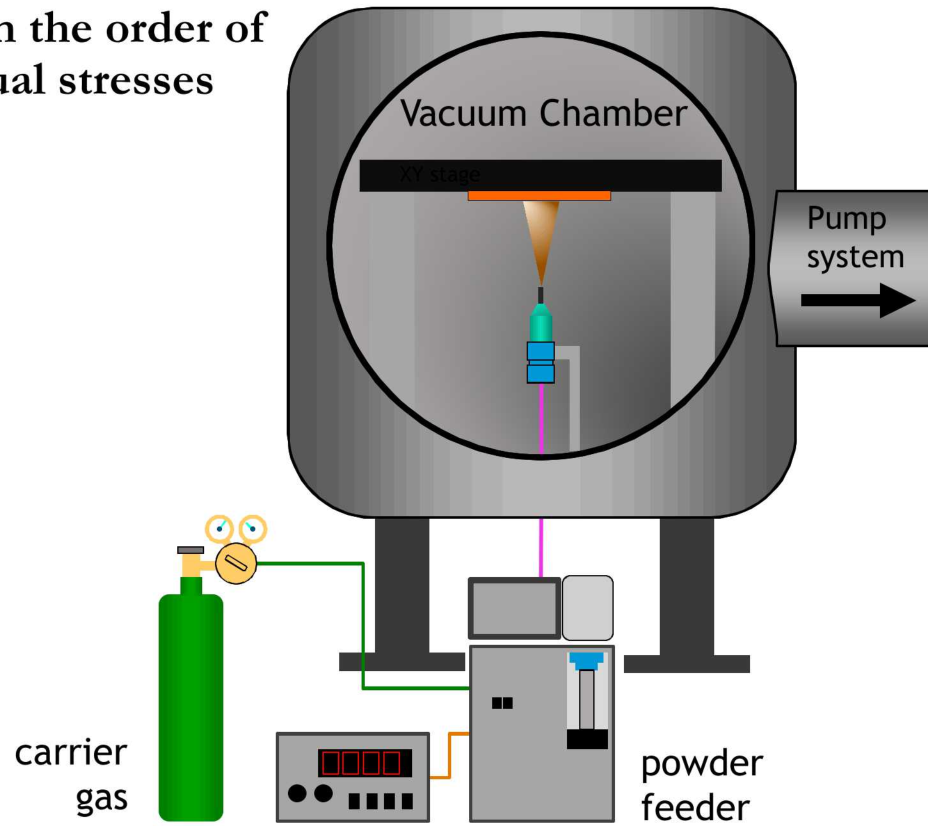
- Uses kinetic energy for impact consolidation of dry, fine-size powder feedstock at room temperature with no binders or heating
- Capable of depositing otherwise brittle materials (oxides, carbides, nitrides etc.) by relying on the plasticity exhibited by fine sized feedstock
- Deposits dense, nanocrystalline films on the order of <1μm to 10s of μms, compressive residual stresses



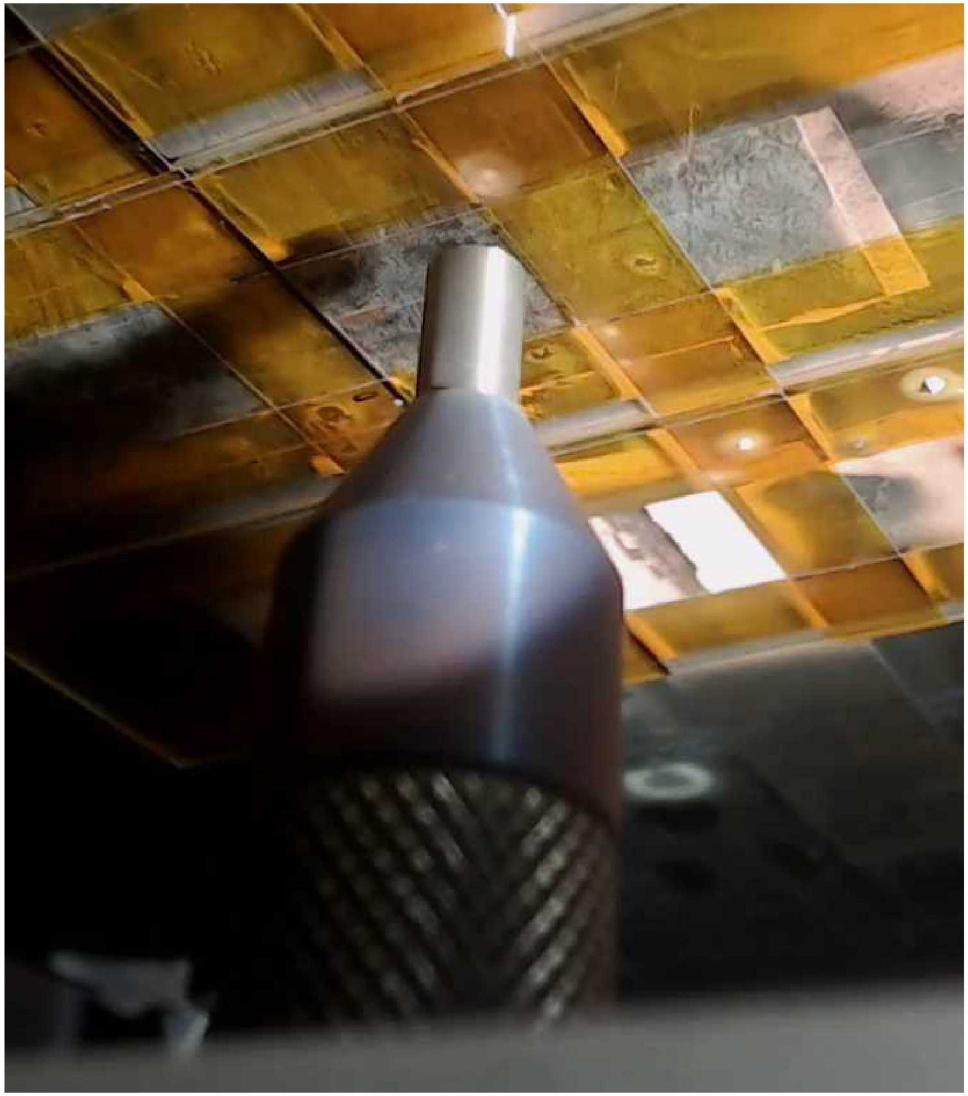
Aerosol Generator for controlled powder feed



Nozzles for achieving supersonic particle flow

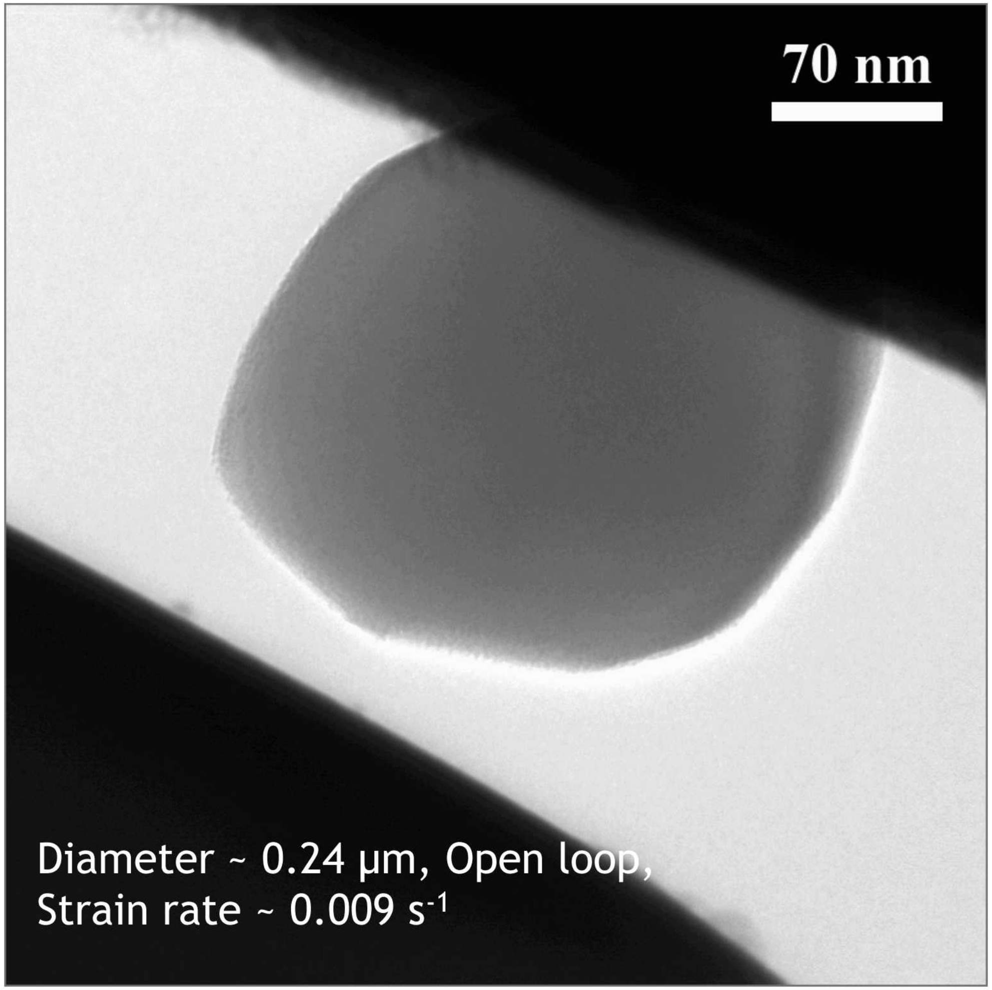


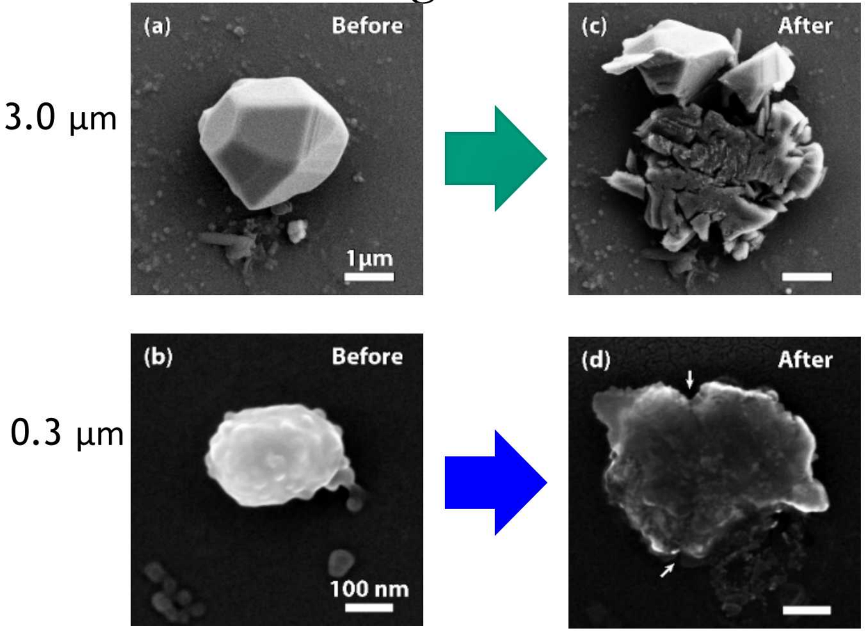
Aerosol Deposition (AD)



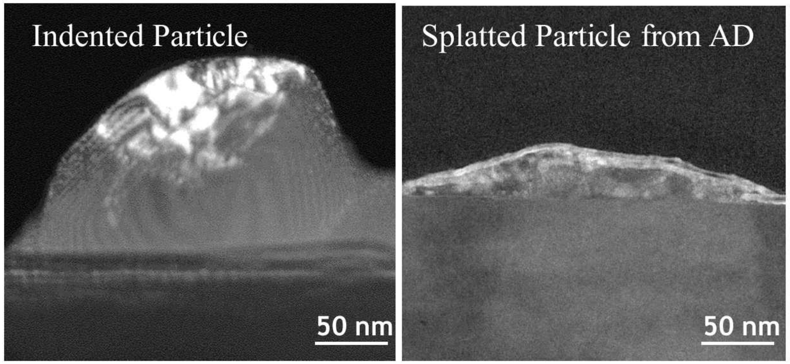
Plasticity in Ceramics

Particle compression tests reveal plasticity in Alumina with fine grain sizes





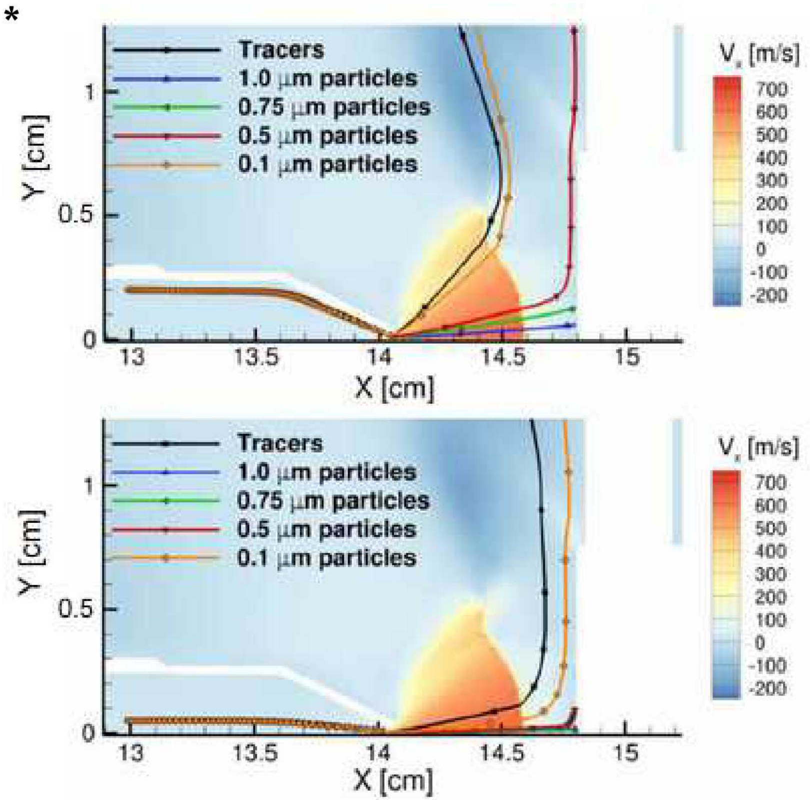
Sarobol, Pylin, et al. "Room temperature deformation mechanisms of alumina particles observed from in situ micro-compression and atomistic simulations." Journal of Thermal Spray Technology 25.1-2 (2016): 82-93.

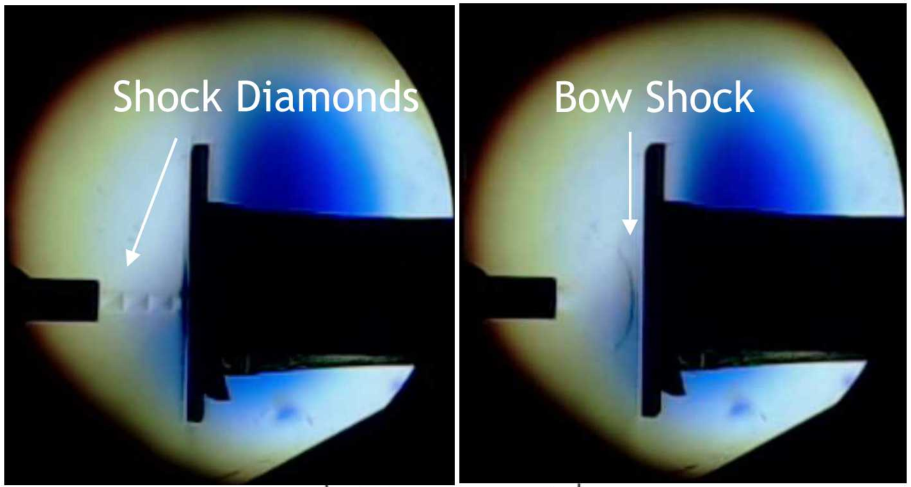


Processing limitations and constraints

- AD has a notoriously low deposition rate compared to other powder based spray coating techniques (cold spray, thermal spray etc.) - Why?
  - Gas Dynamics – Bow shock limits what particles are able to impact the substrate with adequate kinetic energy for particle deformation

*





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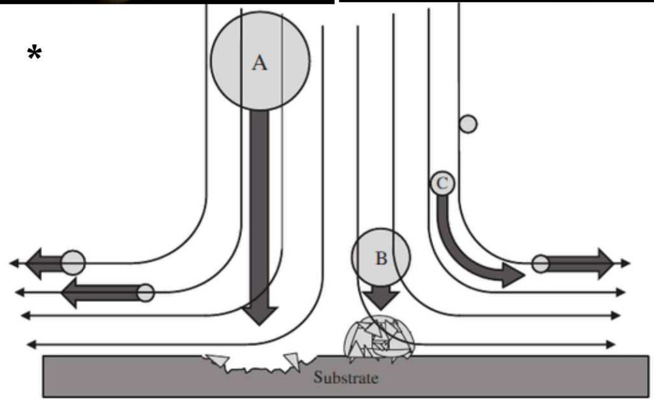
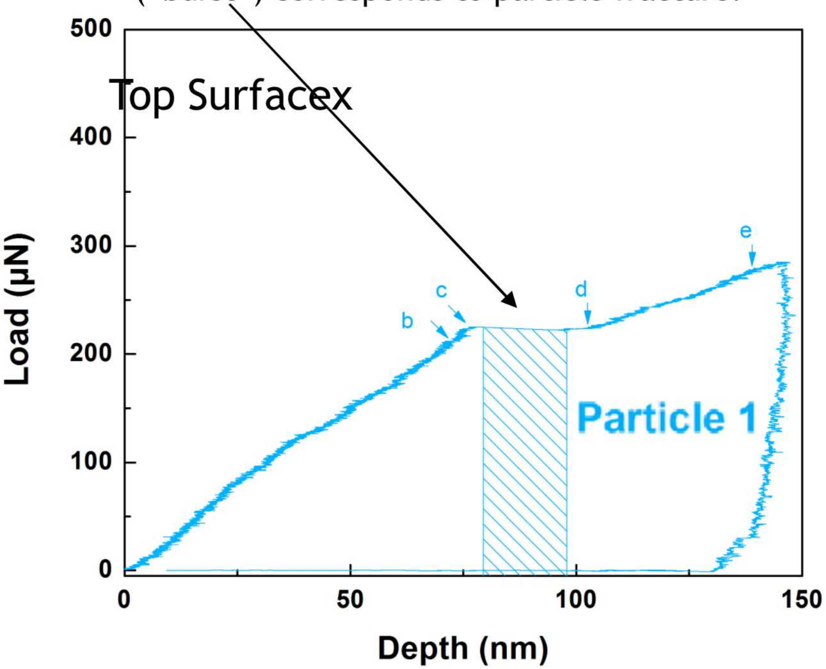


* Johnson, Scooter D., et al. "Deposition efficiency of barium hexaferrite by aerosol deposition." *Surface and Coatings Technology* 332 (2017): 542-549.

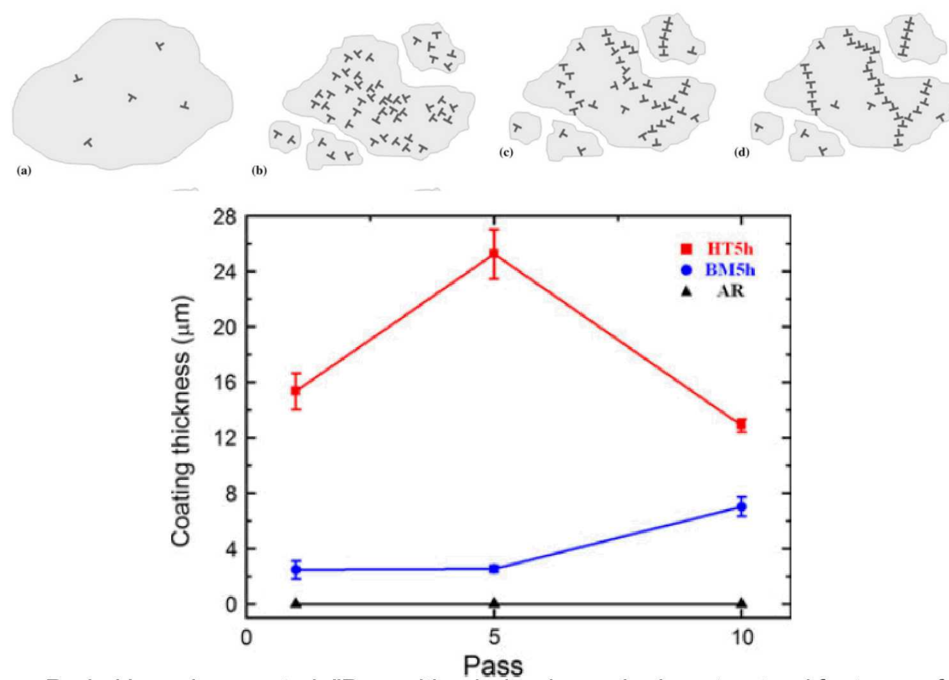
Processing limitations and constraints

- **AD has a notoriously low deposition rate compared to other powder based spray coating techniques (cold spray, thermal spray etc.) - Why?**
  - Gas Dynamics
  - **Particle Deformation – Energy is used to generate dislocations and slip**

Large displacement gain at a constant load ("burst") corresponds to particle fracture.



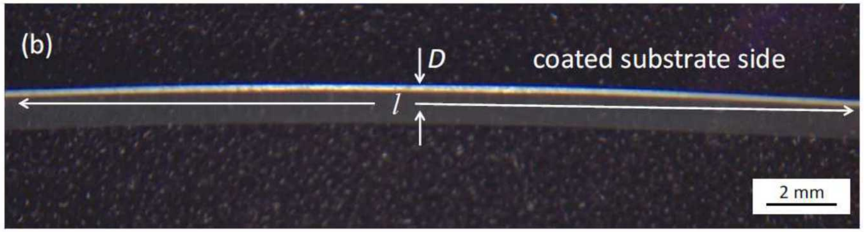
Ball milling and heat treatment to preform nanocrystalline grains increases deposition efficiency

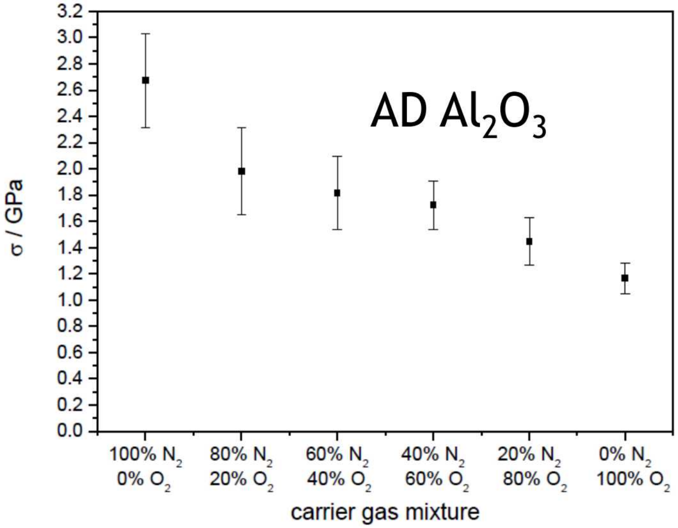


Park, Hyungkwon, et al. "Deposition behavior and microstructural features of vacuum kinetic sprayed aluminum nitride." *Journal of thermal spray technology* 22.6 (2013): 882-891.

Processing limitations and constraints

- **AD has a notoriously low deposition rate compared to other powder based spray coating techniques (cold spray, thermal spray etc.) - Why?**
  - Gas Dynamics
  - Particle Deformation
  - **Residual Stresses – Impact consolidation leads to compressive residual stresses**



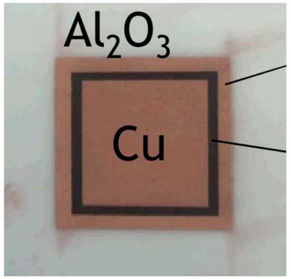


Schubert, Michael, Jörg Exner, and Ralf Moos. "Influence of carrier gas composition on the stress of Al2O3 coatings prepared by the aerosol deposition method." Materials 7.8 (2014): 5633-5642.

$BaTiO_3$ by AD

Why $BaTiO_3$?

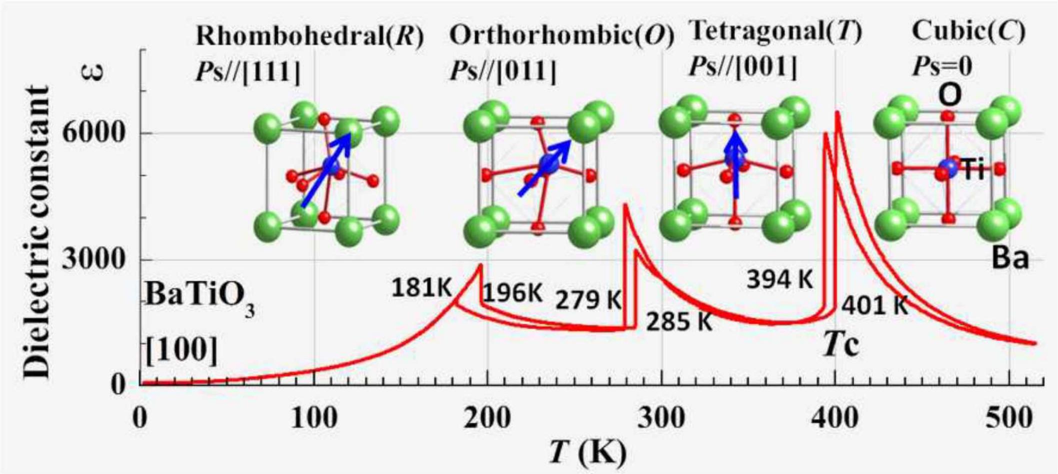
- **Ferroelectric with high dielectric constant (up to7,000) with spontaneous polarization**
- **Well studied and used material**



Why AD?

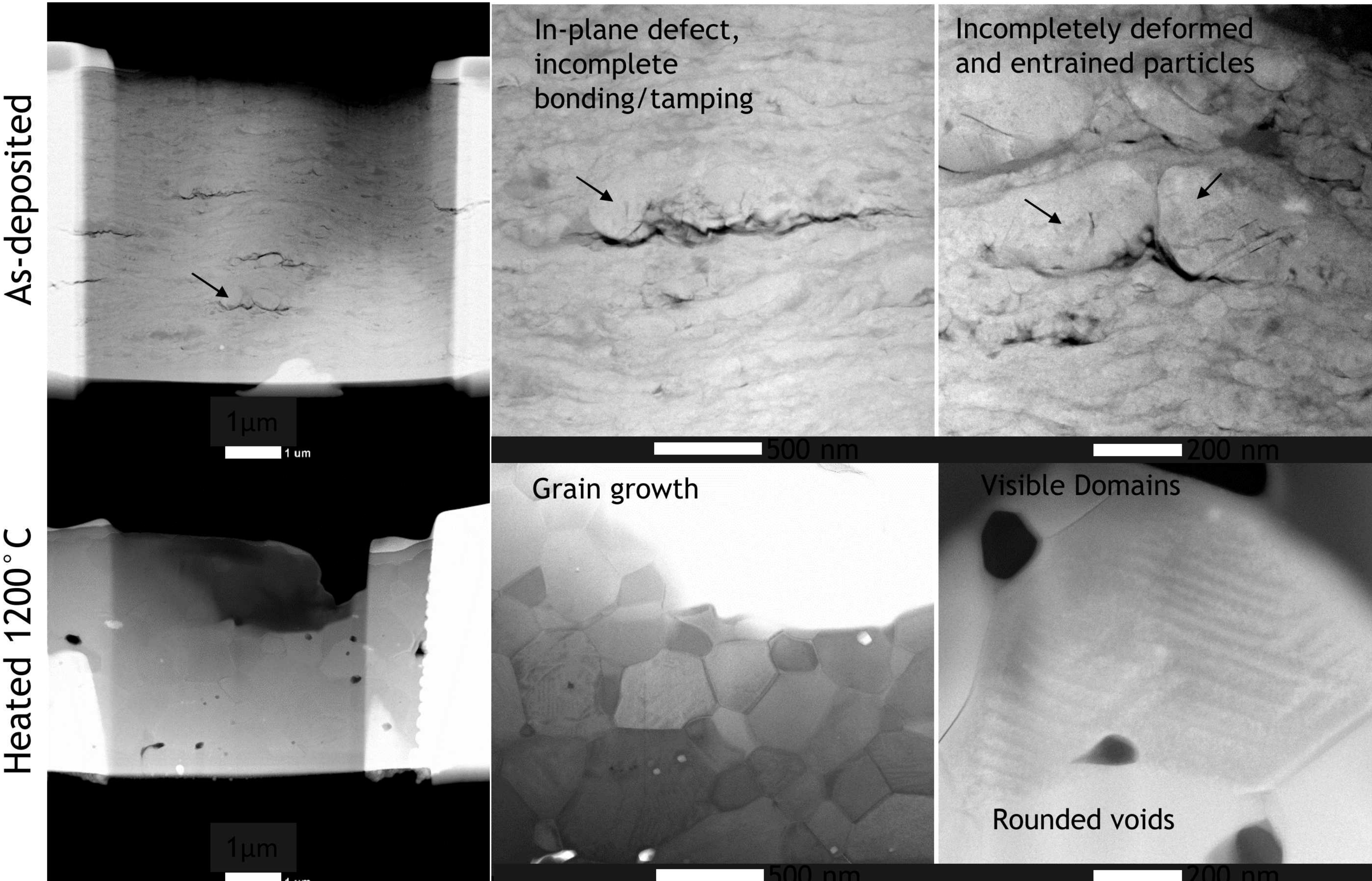
- **No feedstock heating -> *Preservation of material stoichiometry and phase***
- **Room temperature process -> *Integration with low melting temperature materials***

$BaTiO_3$ case study:
- Phase and grain size from deposition and heat treatment
- Residual stress by XRD and substrate curvature
- Electrical properties



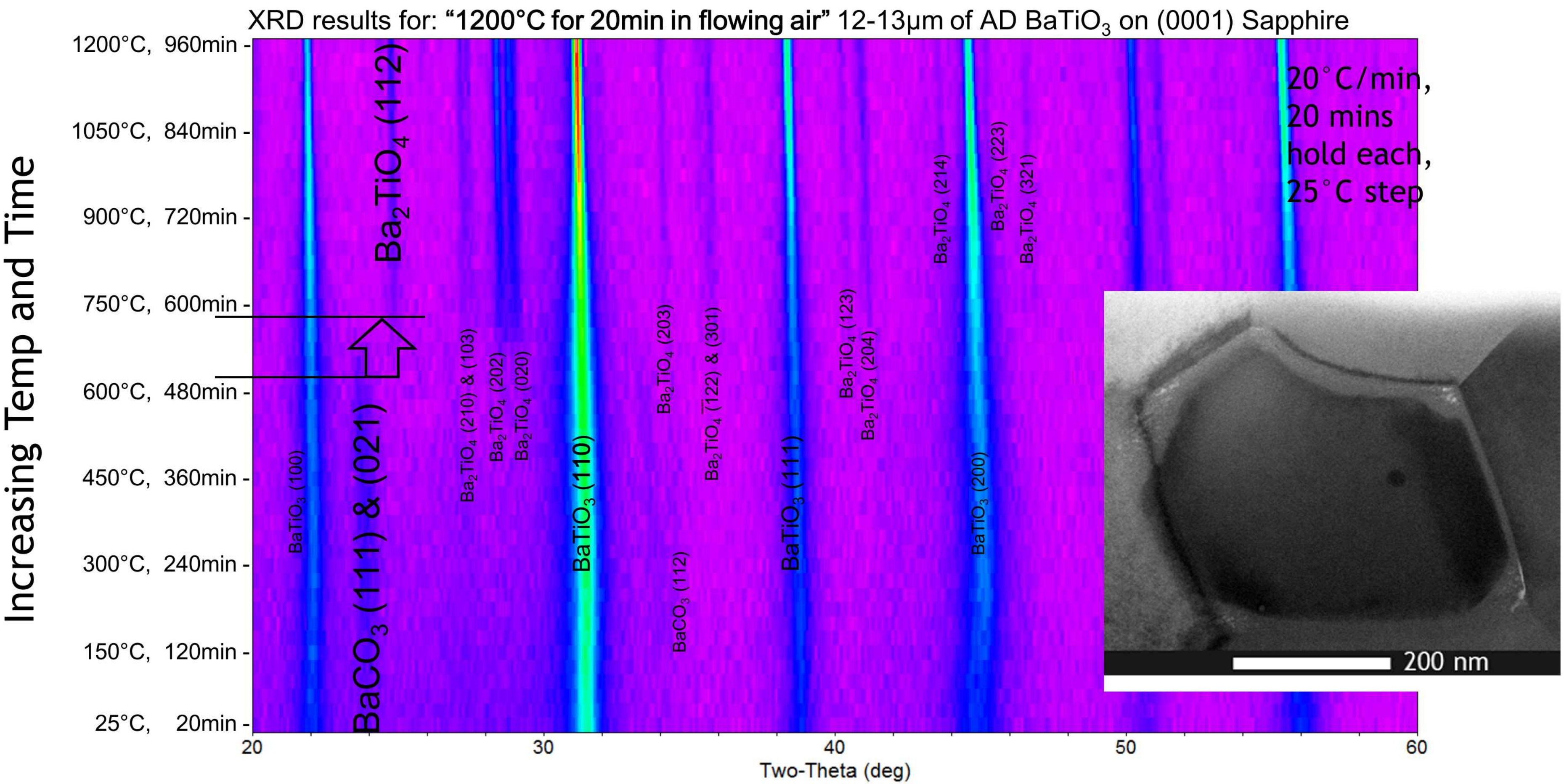
Harlan Brown, Mark Rodriguez, James Griego

Microstructure – FIB and TEM



Phase Analysis

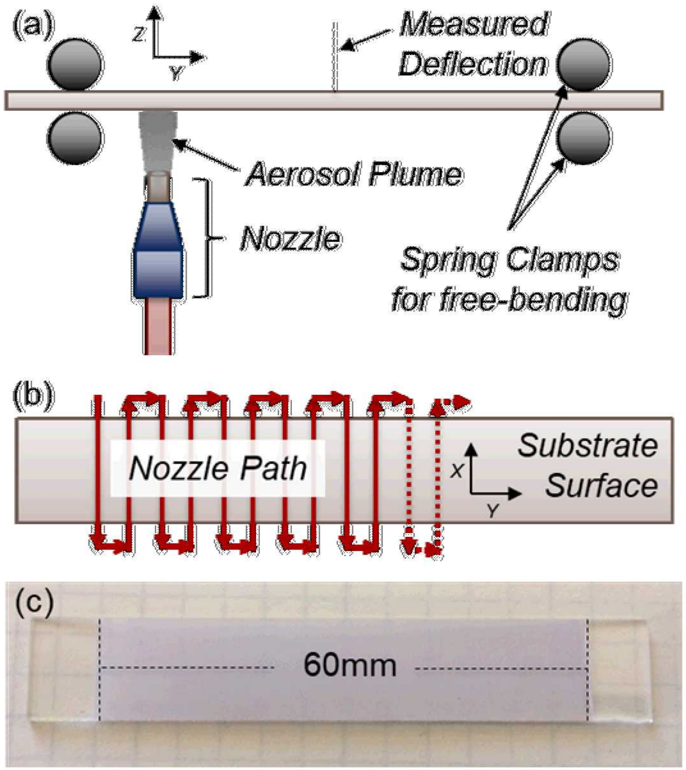
- $BaTiO_3 + BaCO_3 \rightarrow Ba_2TiO_4 + CO_2$ occurs ~680°C[1]

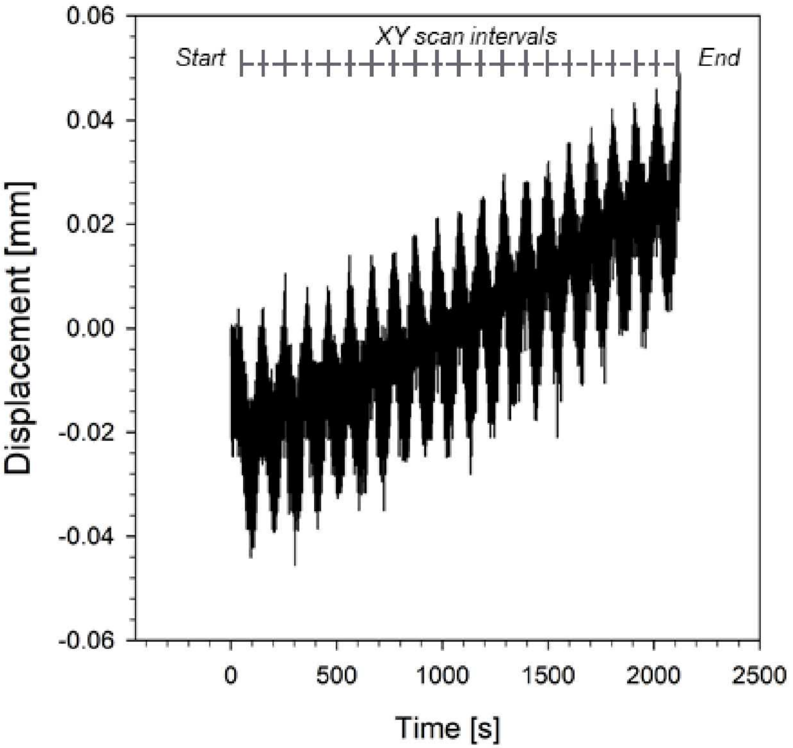


1. TEMPLETON, LIESELOTTE K., and Joseph A. Pask. "Formation of BaTiO3 from BaCO3 and TiO2 in air and in CO2." Journal of the American Ceramic Society 42.5 (1959): 212-216.

AD Film Residual Stress

- XRD (for (211) peak of the films on sapphire substrates were -0.44% (-510 to -560 MPa) and 0.06% (37 to 43 MPa) for the as-deposited film and heat-treated film, respectively
- In-situ substrate curvature measurements on soda lime glass calculated to be -430, -404, and -507 MPa for three repeat trials



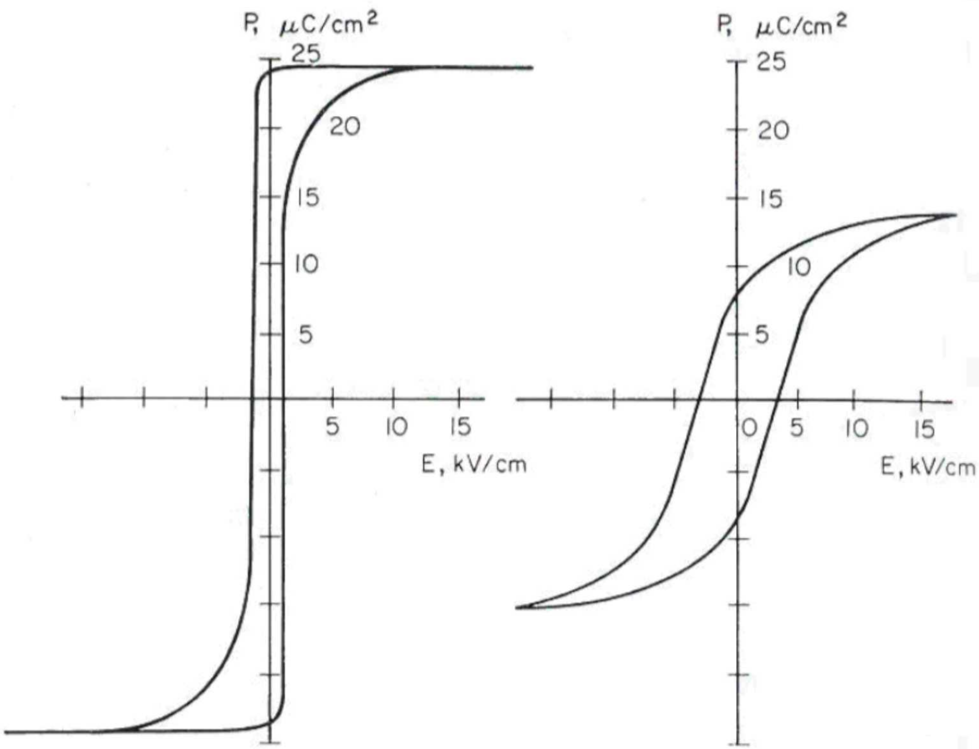


Electrical Performance

- **Pseudocubic, disordered, stressed film generates <u>linear dielectric response</u>**
  - **Stress relief through heat treatment or film delamination in literature shows return of hysteresis and saturation point**
- **Voltage withstanding suggests no thru cracks are present**

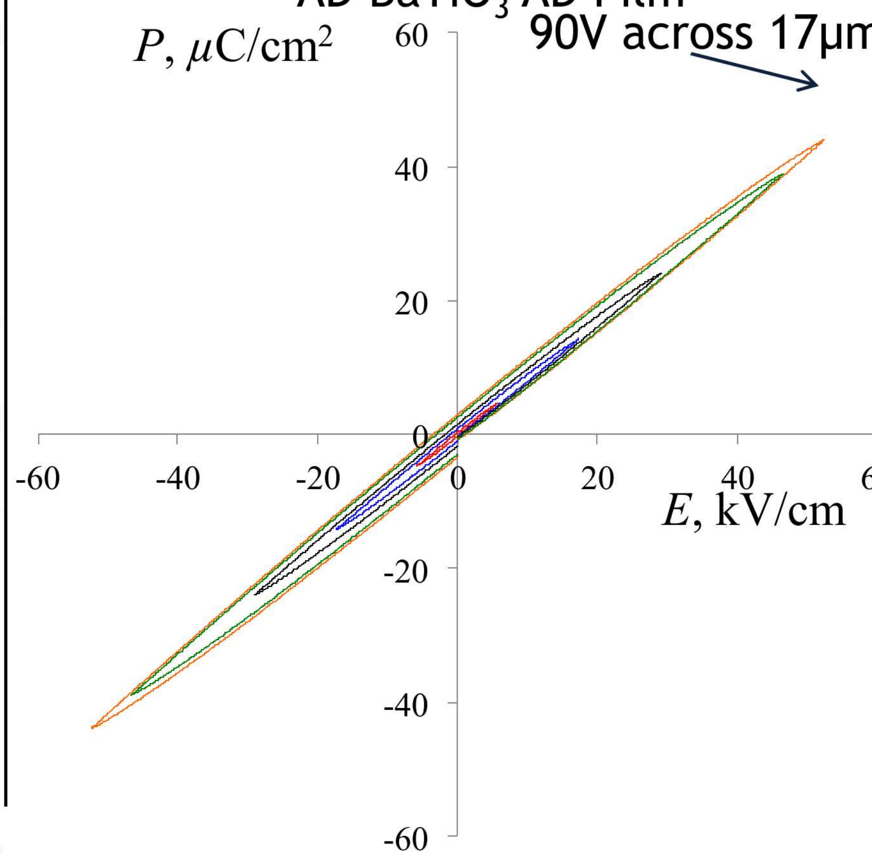
$BaTiO_3$ (100) Crystal

$BaTiO_3$ Ceramic



Jaffe, Cook and Jaffe, *Piezoelectric Ceramics* pp. 78 (1971).

AD $BaTiO_3$ AD Film



Increasing processability and Functionality of AD $BaTiO_3$ films

- **Increase in deposition efficiency**
  - **Pre-Processing of powder by ball milling, heat treatment, size control**
  - **Nozzle design and gas flow dynamics for minimized bow shock**
- **Recover polycrystalline electrical behavior**
  - **Heat treatment for residual stress relief, grain growth**
    - **Thermal strain considerations**
  - **Increase feedstock purity or heat treatment in CO_2 atmosphere**

Acknowledgements

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  - Joe Fonseca, Mike Clearwater, Tom Holmes, Andrew Miller, Joe Padilla, Carlos Silva, Andy Mayer, Sam Siska
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- Mark Rodriguez, James Griego
  - XRD phase and residual stress analysis
- Paul Kotula
  - FIB and TEM
- Center for Integrated Nanotechnologies
  - Particle compression testing

Thank You!
Questions?