Small-Particle Plasma Spray (SPPS) Thermal Barrier Coatings

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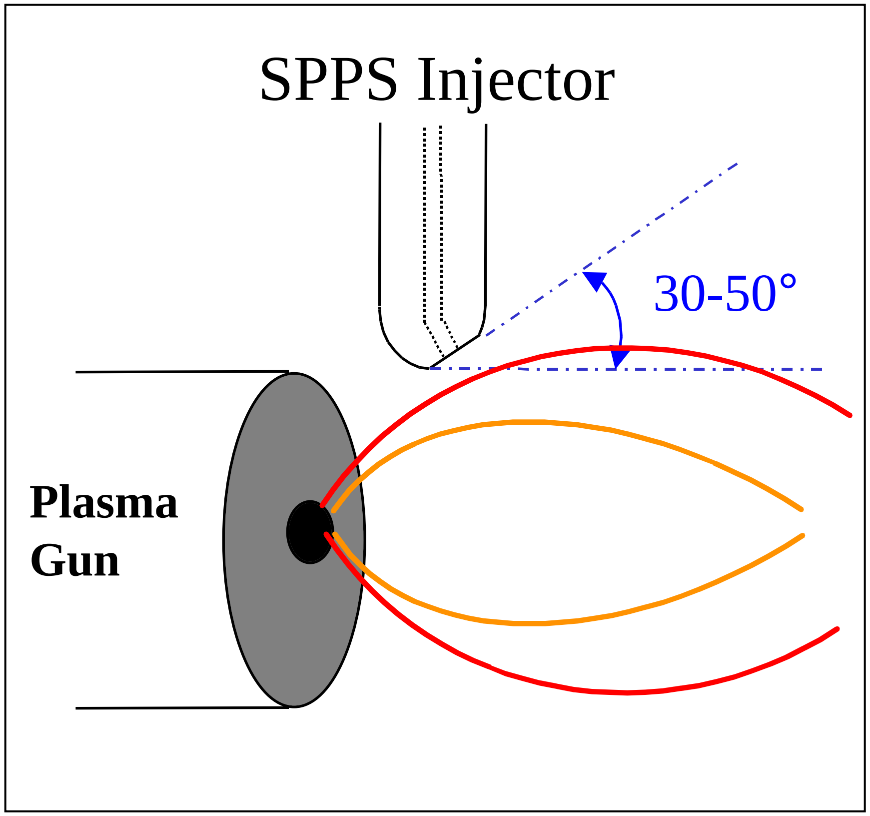
Sample Fabrication

- Used Small Particle Plasma-Spray (SPPS) powder injection technology:

  Allows small particles to be placed into the plasma in a more controlled manner
  - Reduces powder vaporization
  - Less open porosity in coatings

- U.S. Patent No. 5,744,777



TBC Studies Using SPPS

AGTSR

Northwestern U.

Steering committee: ORNL, GE, AlliedSignal, Allison, P&W, & Siemens/Westinghouse

Multi-Layer

- Micrometer thick layers
- Enhanced toughness from delamination
- Enhanced lifetimes due to microstructural stability

Graded Porosity

- More pores near the TBC surface
- Creates desired elastic modulus anisotropy
- Lower thermal conductivity from pores

Materials Engineering

- Coated powders
- Control grain growth and sintering
- New materials

The Current Work:

Understand the properties of SPPS coatings through:

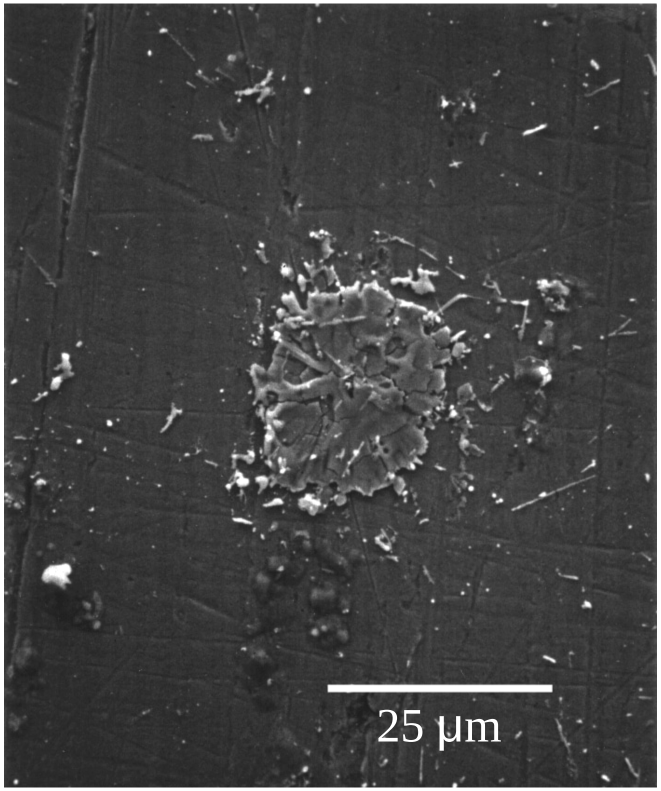
I. Characterization of coatings as a function of spraying conditions.

II. Development of a mechanical test to assess elastic properties and damage mechanisms.

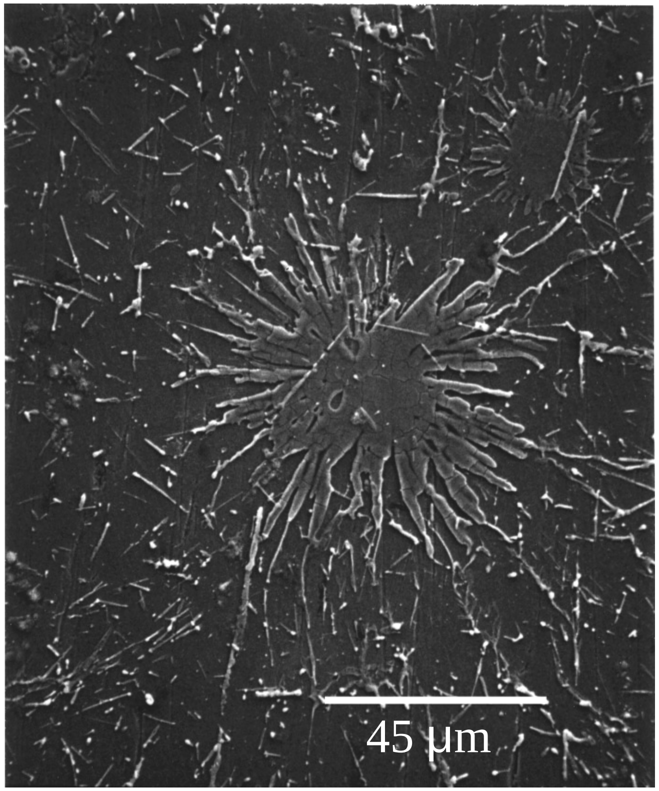
I. Comparison of Microstructure, Thermal Conductivity and Damage Tolerance for Two Spraying Conditions

	"COOLER" Conditions	**"HOTTER" Conditions**
Power	25 kW	40 kW
% Hydrogen	10%	20%
Total Gas Flow	50 SLM	40 SLM
Spray Distance	6 cm	6 cm
Injector Offset	11 mm	11 mm
Injector Angle	30º	30°

Splat Morphology

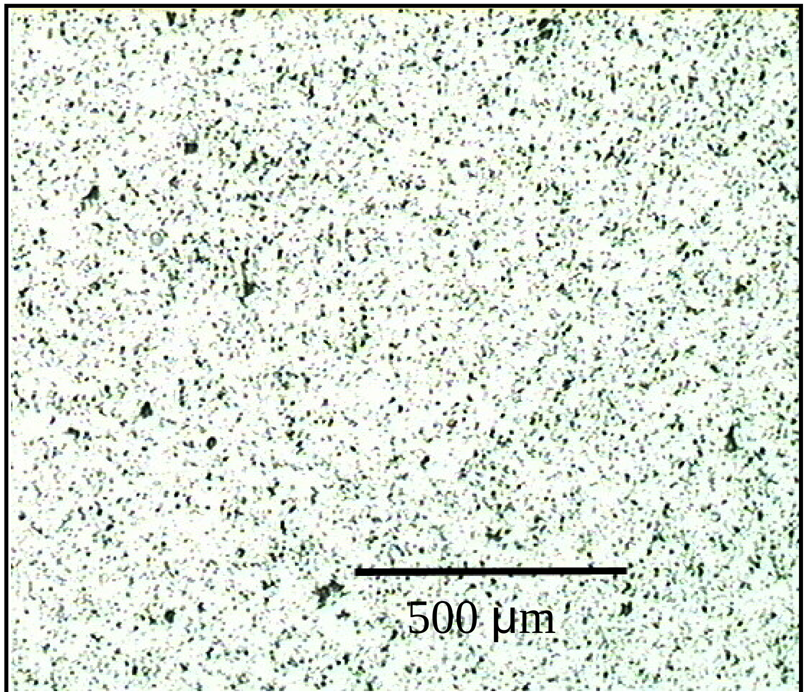


COOLER Conditions



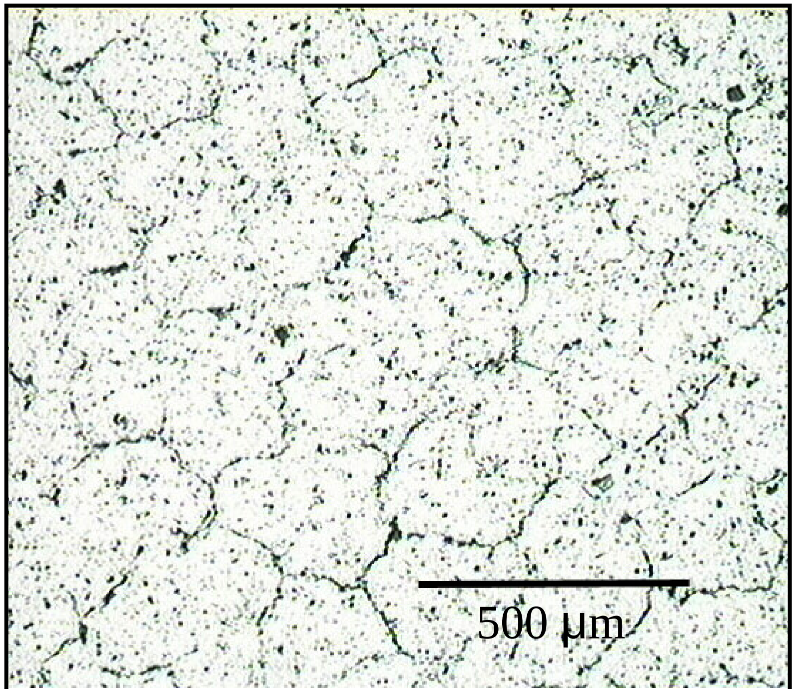
HOTTER Conditions

Coating Microstructure: Polished Top Surfaces



Cooler Conditions

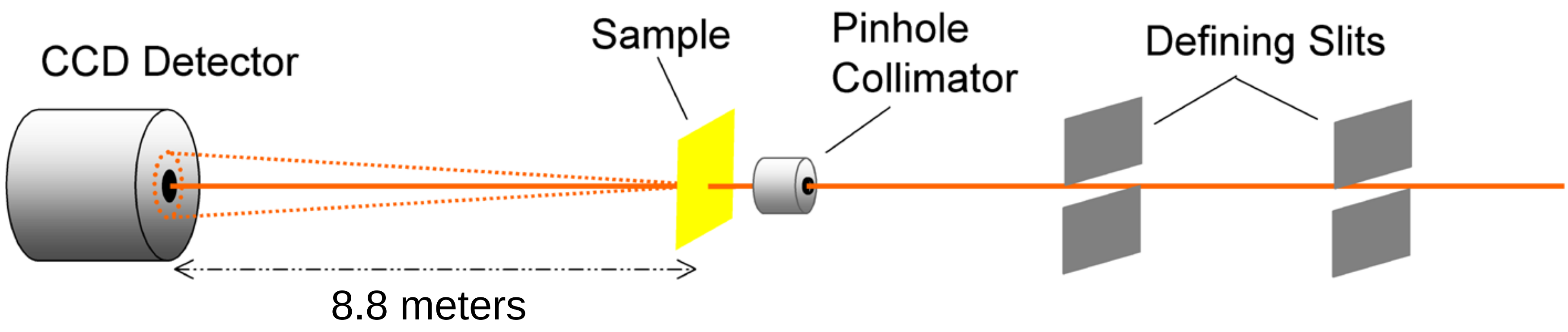
91% dense

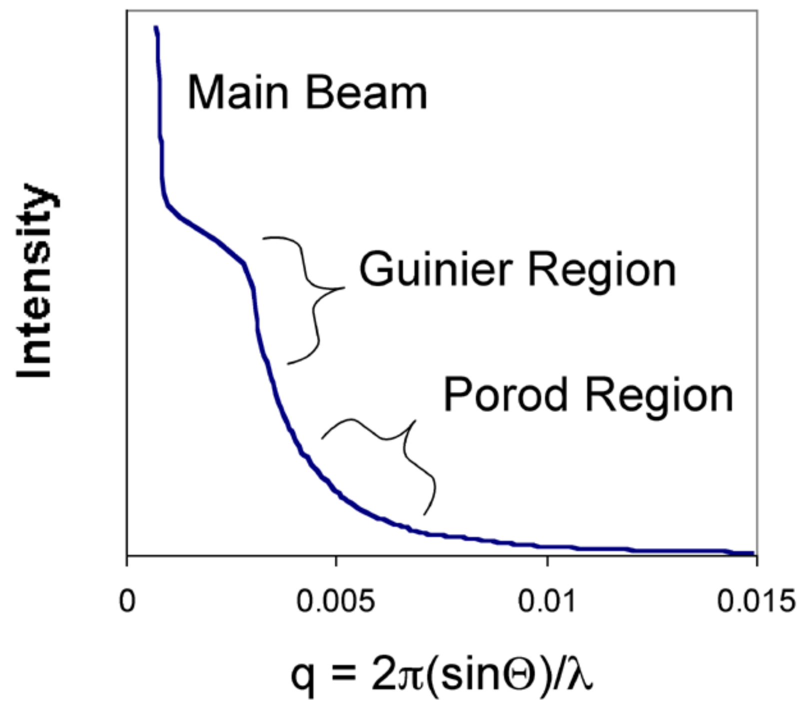


Hotter Conditions

92.2% dense

Small-Angle X-Ray Scattering



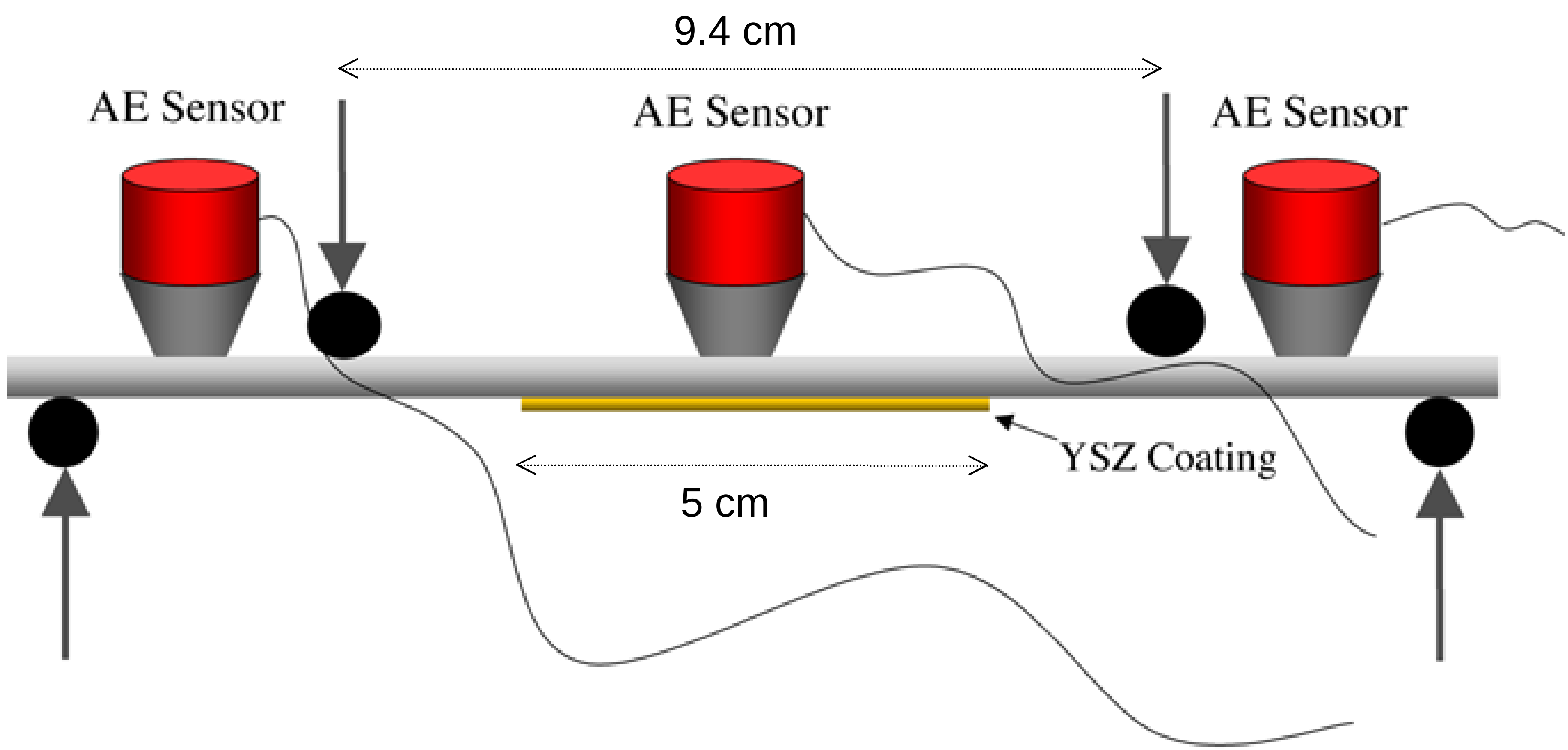


- SAXS is sensitive to changes in electron density which can be caused by porosity.
- With this setup, pores in the range of 20 - 400 nm are detected.
- Pattern can be analyzed to get scattering area to volume ratio.

SAXS, Conductivity and Hardness

	Hotter Conditions (92.2% dense)	**Cooler Conditions** (91% dense)
As Sprayed: S/V Conductivity Hardnesss	 $62 \pm 8\ cm^2/cm^3$ $1.7 \pm 0.1 W/m\ K$ $6.5 \pm 0.2\ GPa$	 $122 \pm 21 cm^2/cm^3$ $2.1 \pm 0.1\ W/mK$ $7.5 \pm 0.3\ GPa$
1200ºC/600 hr: S/V	 $17 \pm 1\ cm^2/cm^3$	 $23 \pm 3\ cm^2/cm^3$

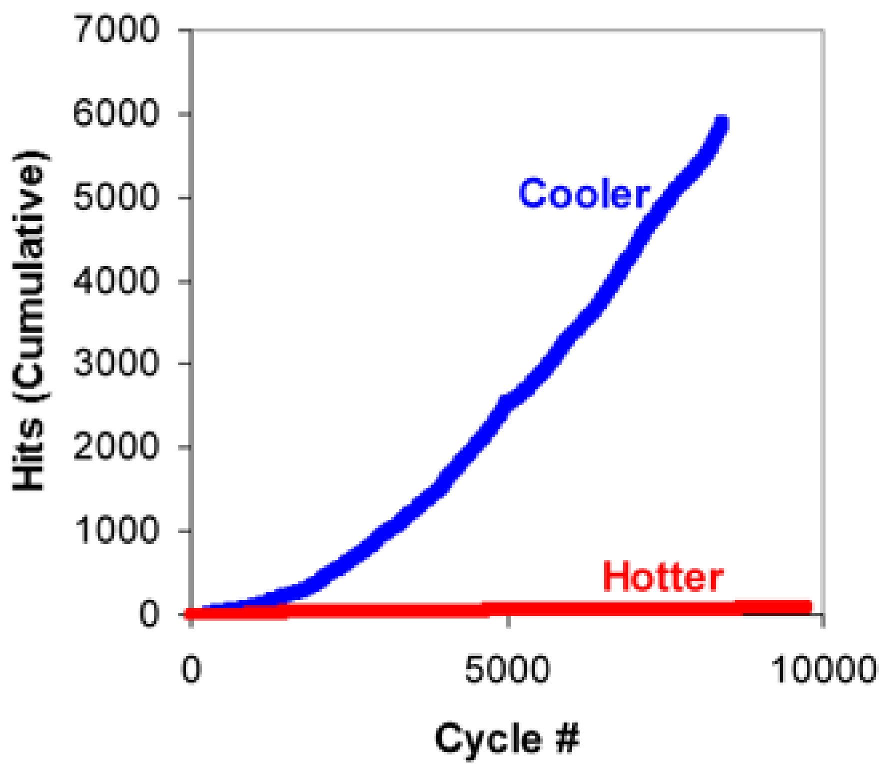
Four-Point Bend Testing Setup

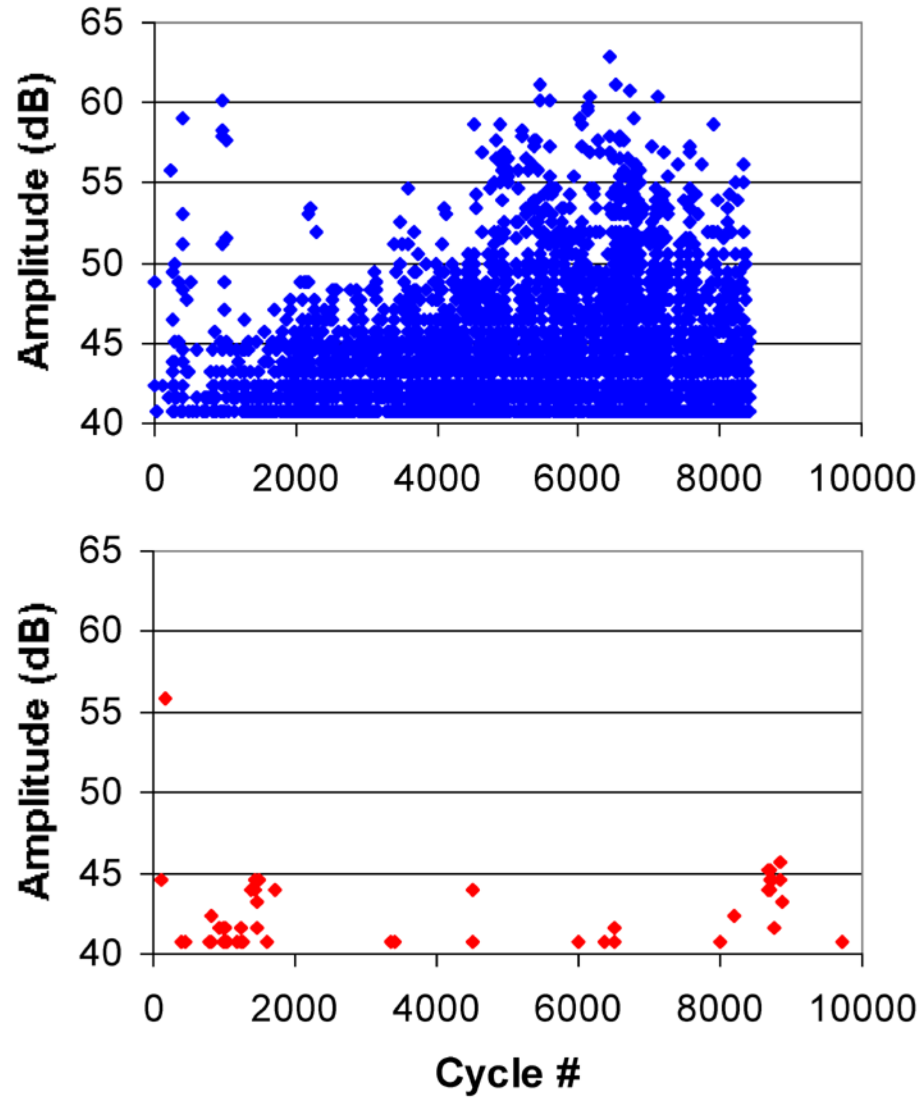


- *Sensors: DECI model SE 150-m, 7 cm apart*
- *Monitoring System: Vallen AMS-3*
- *Coating Thickness: 100 μm*

Tension Bending Fatigue with AE

- Fatigue cycle: 0.3 Hz 125-310 MPa

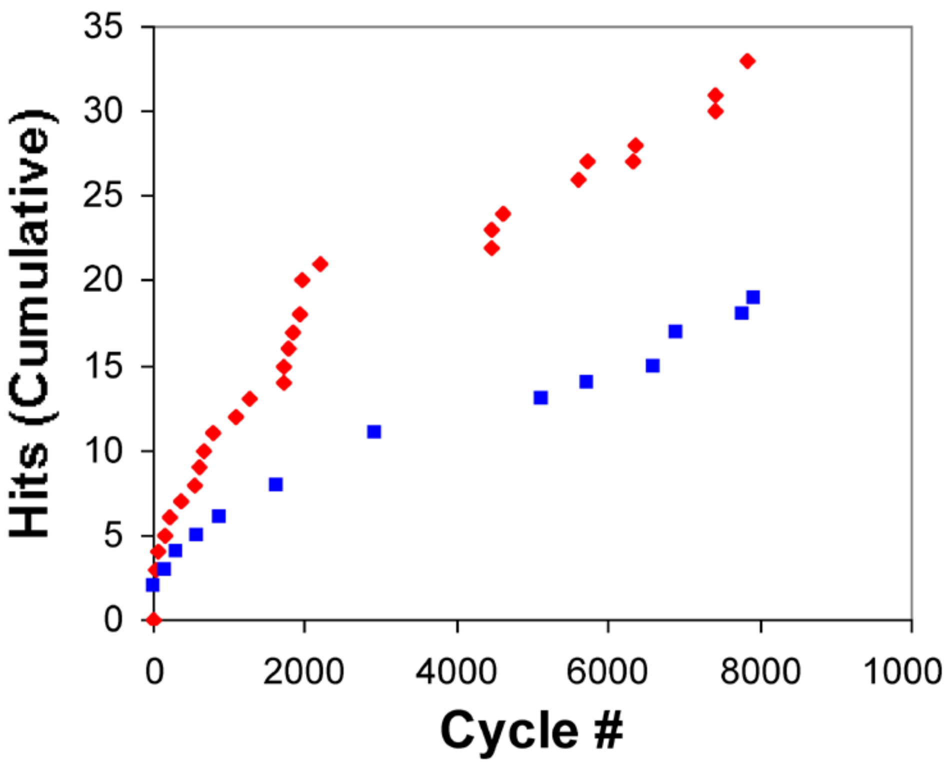




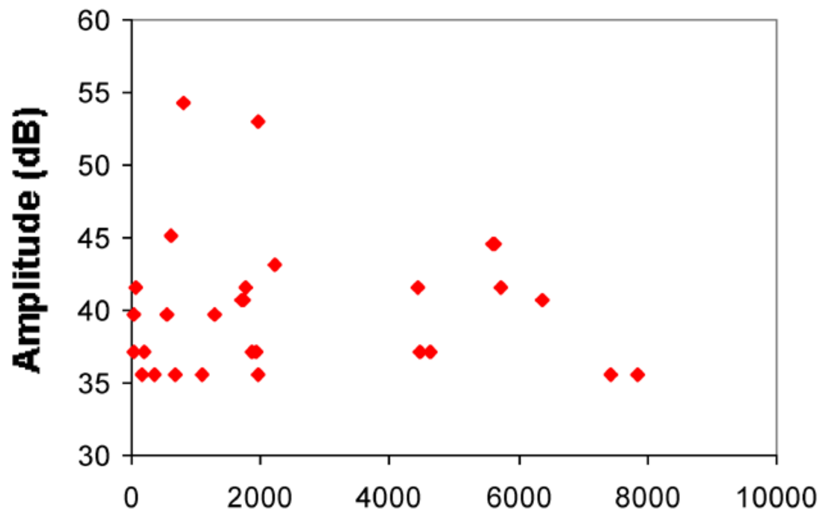
- Waveforms showed that hits >55 dB were very short duration
  - Characteristic of through-crack formation

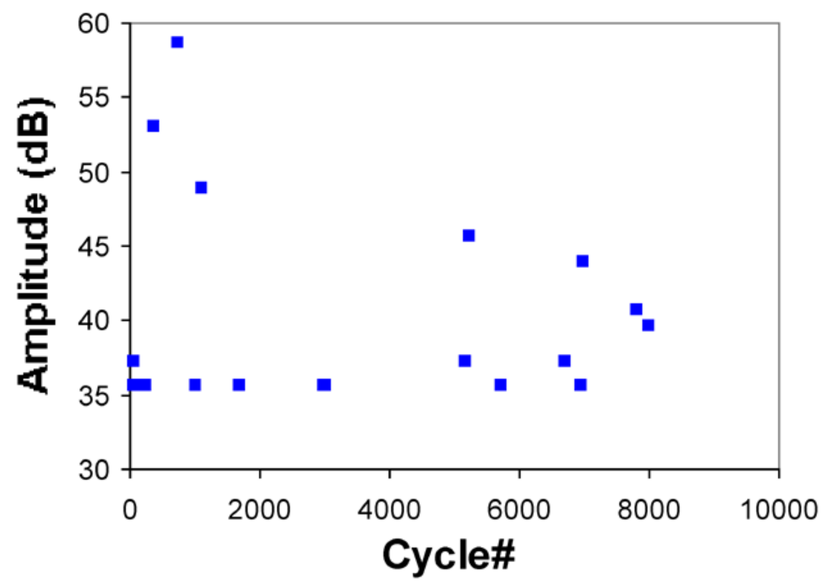
Compression Bending Fatigue with AE

- Fatigue cycle: 0.3 Hz 125-310 MPa

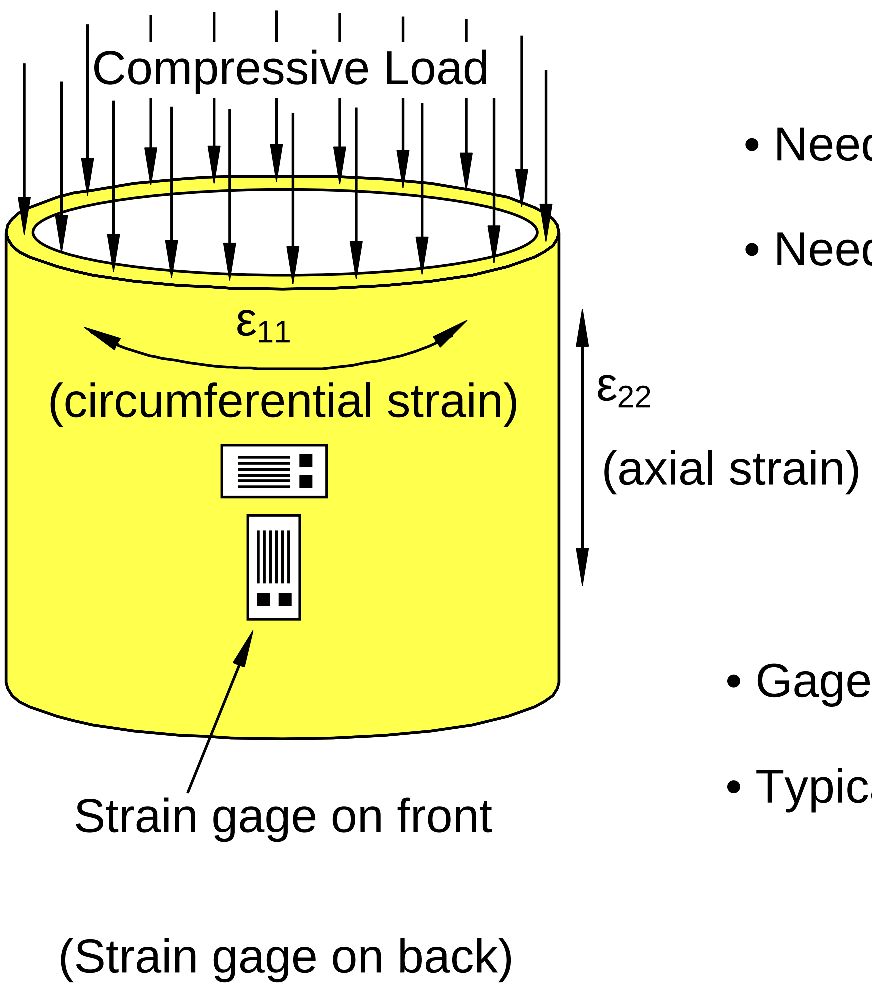


- Very good results, no visible damage
- Overall, samples indistinguishable from one another



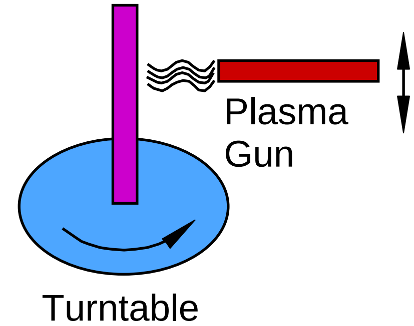


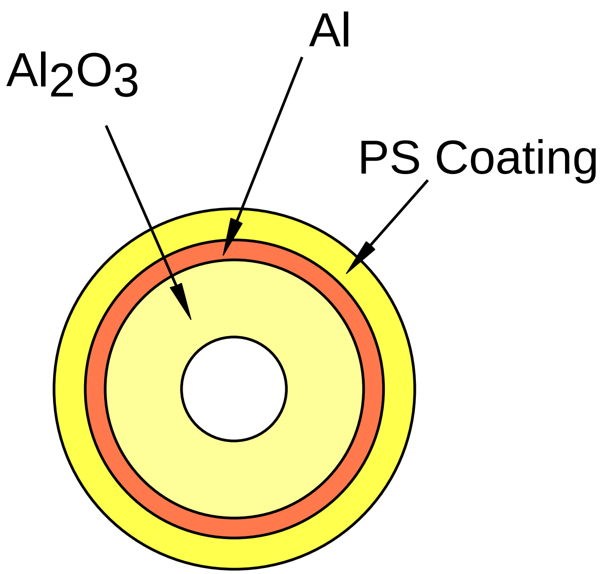
II. New Mechanical Test for Coatings to Establish Coating Properties: Stand Alone Coating Test (SACT)



- Need to measure strain very accurately
- Need to balance the load distribution
- Gages bonded with epoxy
- Typical 0-5% difference between gage responses

Preparing SACT Samples





Side View

1. Plasma–spray alumina on aluminum coated tubes of alumina

2. Cut up 12” tube into 1’’ tubes and machine ends

3. Soak tubes in Water/HCl for 3 days to dissolve aluminum

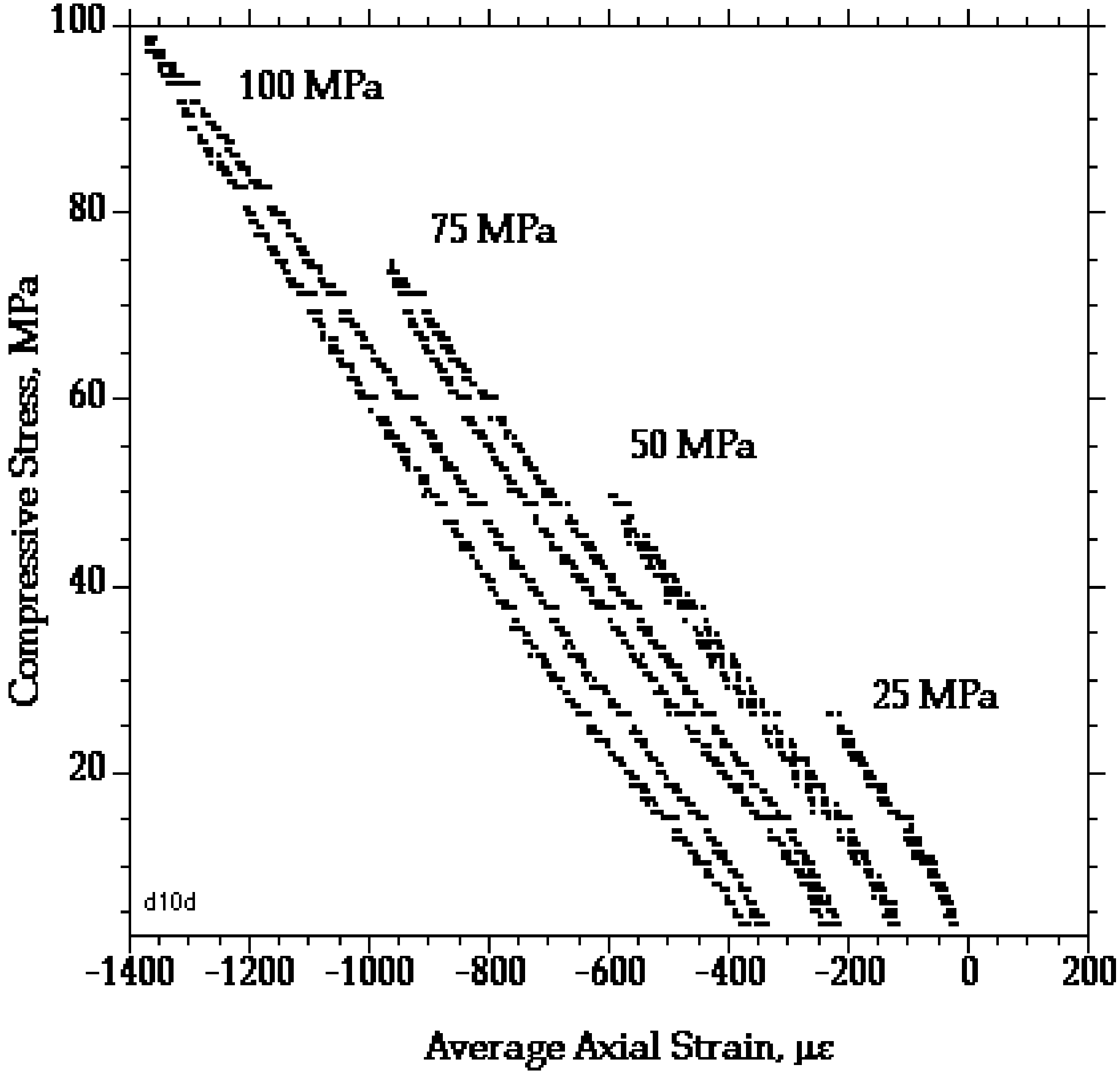
4. Wash coatings/dry/physical measurements

<u>Advantages of this Tube Geometry</u>

Generate 9-10 specimens per 12” tube

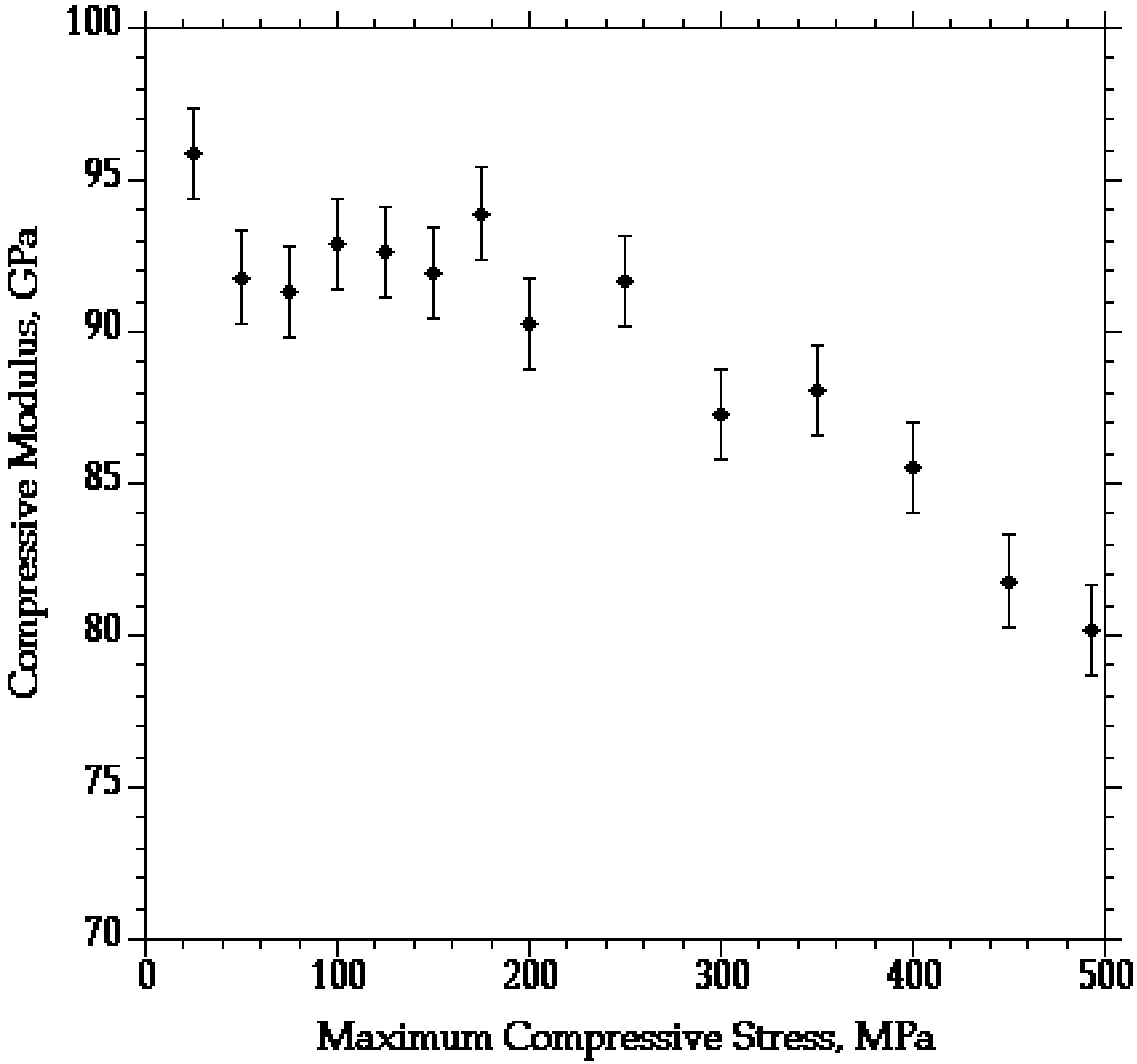
Typical coating is only 200 µm thick*

Common Mechanical Response: Hysteresis



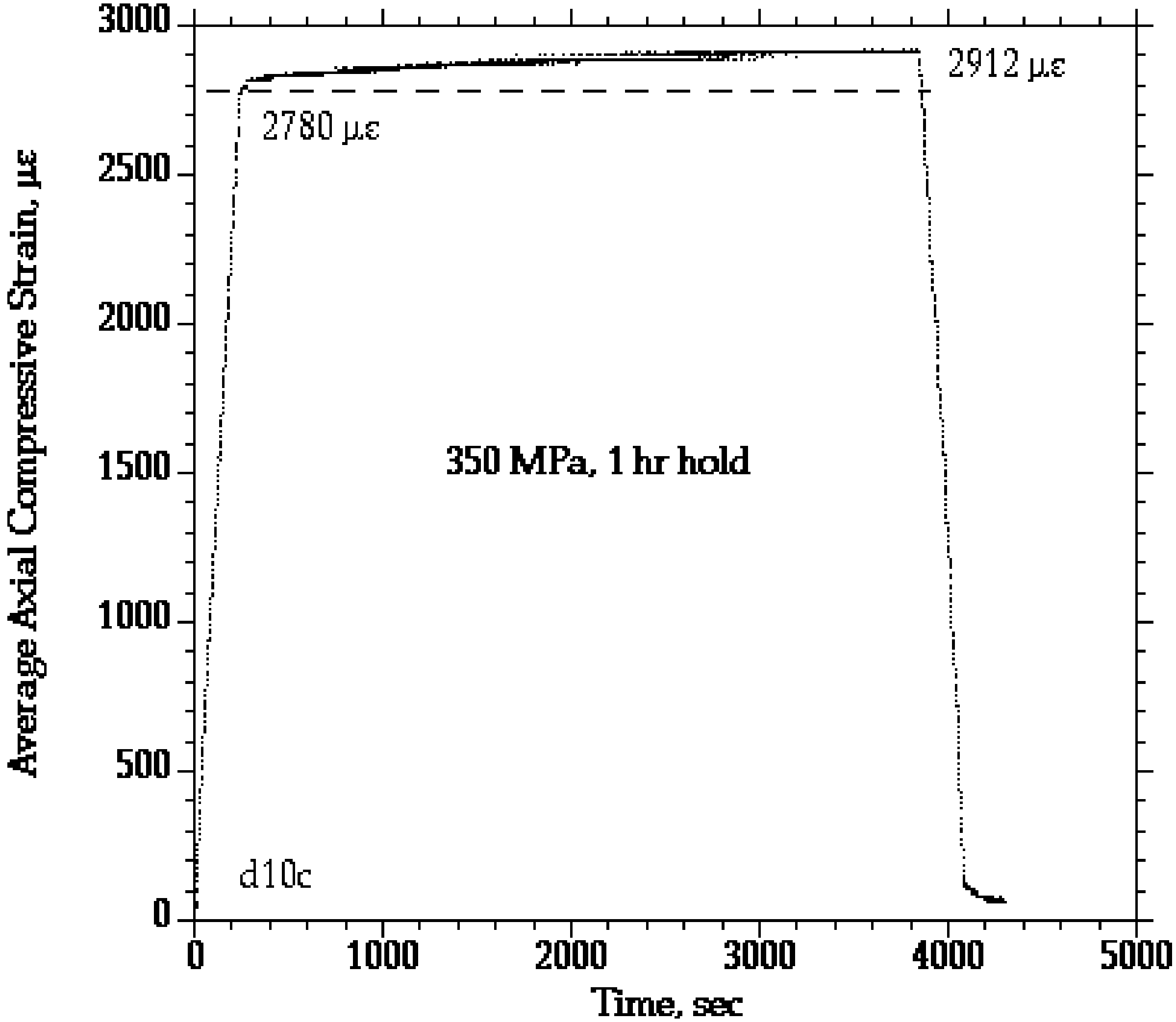
- Hysteresis loops beyond 25 MPa
- Hysteresis loops widened with increasing stress

Common Mechanical Response: Decreasing Modulus with Cyclic Loading



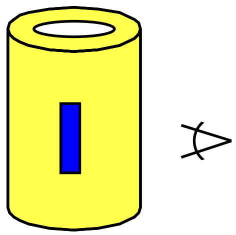
- Modulus decreases with increased stress in cyclic loading

Failure Mechanism: Damage Accumulation via Crack Growth?

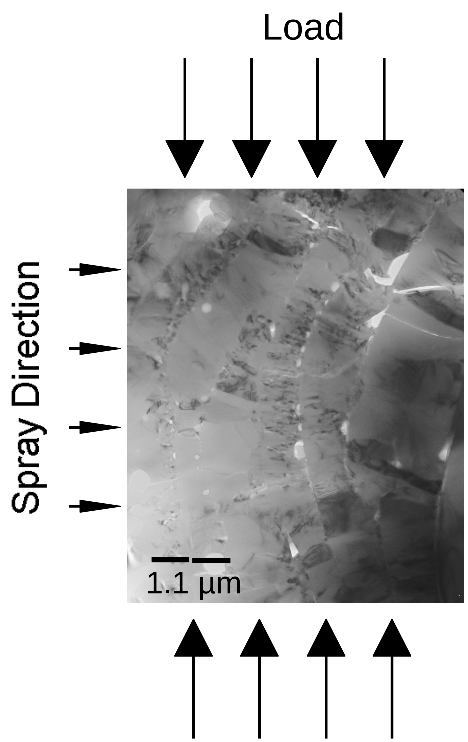


- Strain over time with constant stress
- Real change in strain response over time

Coating Deformation Mechanisms



Cross–Sectional View



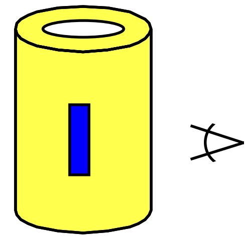
Brightfield TEM

- Extremely complex microstructure (lamella are not planar)

- Extremely defective microstructure
  - bimodal porosity distribution
  - large porosity distribution at the interface
  - unmelted particles
  - quench cracks

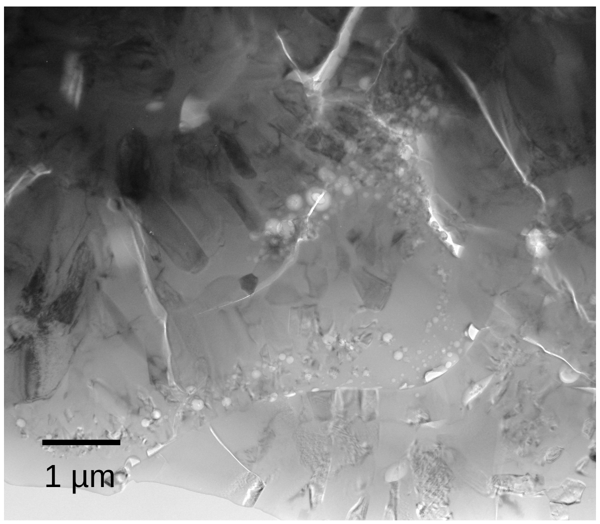
- Sliding of the adjacent lamellae has been suggested

TEM Investigation of Deformation Mechanisms

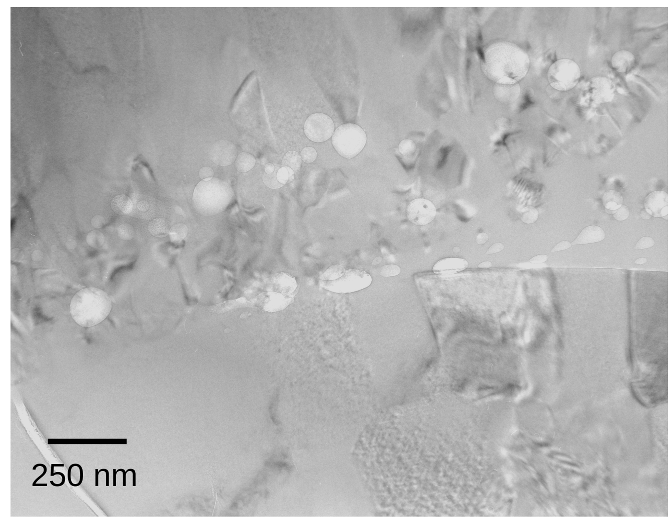


Cross–Sectional View

Fractured Sample (400 MPa)



Brightfield TEM



• No evidence of lamella slipping in any sample

Summary of Deformation Process

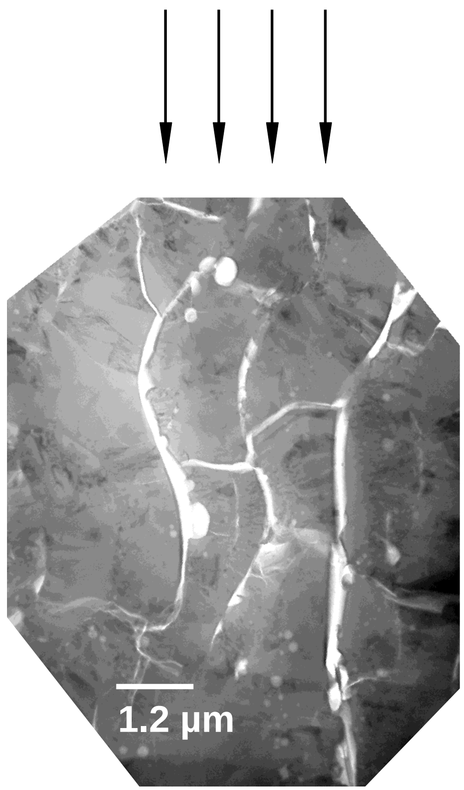
• Intrinsic defects (quench cracks and porosity) in the microstructure react with compressive stress

• Cracks eminate from these defects, turning parallel with the compressive stress

• Likely that cracks follow lamella boundaries

• Ultimately, small cracks grow to long axial cracks and split

• Crack growth mechanism observed in fatigue*



Sample fractured at 400 MPa

*E.F. Rejda, Fat. Fract. Engng Mater. Struct. 20 [7] 1043-50 1991)