

# Materials Testing in Hydrogen Gas at NIST, Boulder

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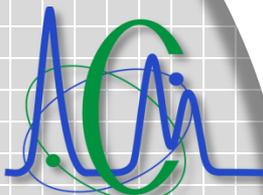
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DS LAURIA, JR FEKETE, K EASON

Sandia National Laboratories, Livermore, CA, April 9-10, 2013

Material Measurement Laboratory

NIST

# Outline



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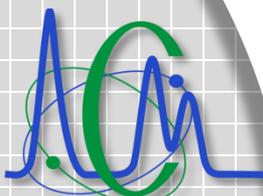
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- Capabilities
- Design
- Transducers
- Test Procedures
- Safety
- Issues



# Objective

- Provide critical data and measurement methods that enable safe and economical transport of hydrogen fuel.
  - Our focus has been on fatigue crack growth testing
  - We may start some fracture testing this year
    - Have not done fracture testing in the past (in H2)

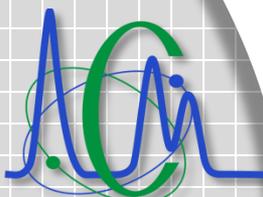


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# Design Approach

- Followed the lead of Sandia
- New laboratory built for hydrogen purpose
- Custom test vessels (2)
- Servo-hydraulic load frames (2)
- Support Equipment for Remote Location
  - Used to have a chiller
  - Power electronics
  - Hydraulic power unit
  - Air compressor
  - Hydrogen sensors

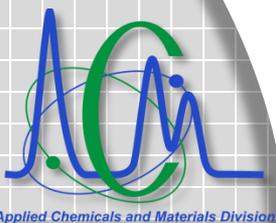


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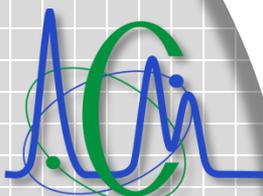
# Laboratory

- Class I, Division II, Group B
- 925 square feet, 12,000 cubic feet
- 350 cubic meters
- Maximum hydrogen gas volume = 6.4 cubic meters



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# Test Capabilities



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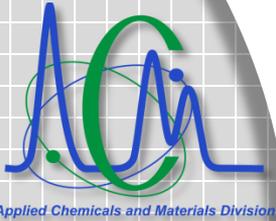
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Load Frame 1	100 kN, 22 kip		
Load Frame 2	250 kN, 55 kip		
Pressure Vessel 1	138 MPa, 20,000 psi	0.55 ft <sup>3</sup> , 1.5 liters	4" x 7.5"
Pressure Vessel 2	34 MPa, 5000 psi	0.341 ft <sup>3</sup> , 9.6 liters	5" x 32"
Room Temperature			
Test control	tensile, 10 <sup>-6</sup>	fatigue, 0.01 - 10 Hz	

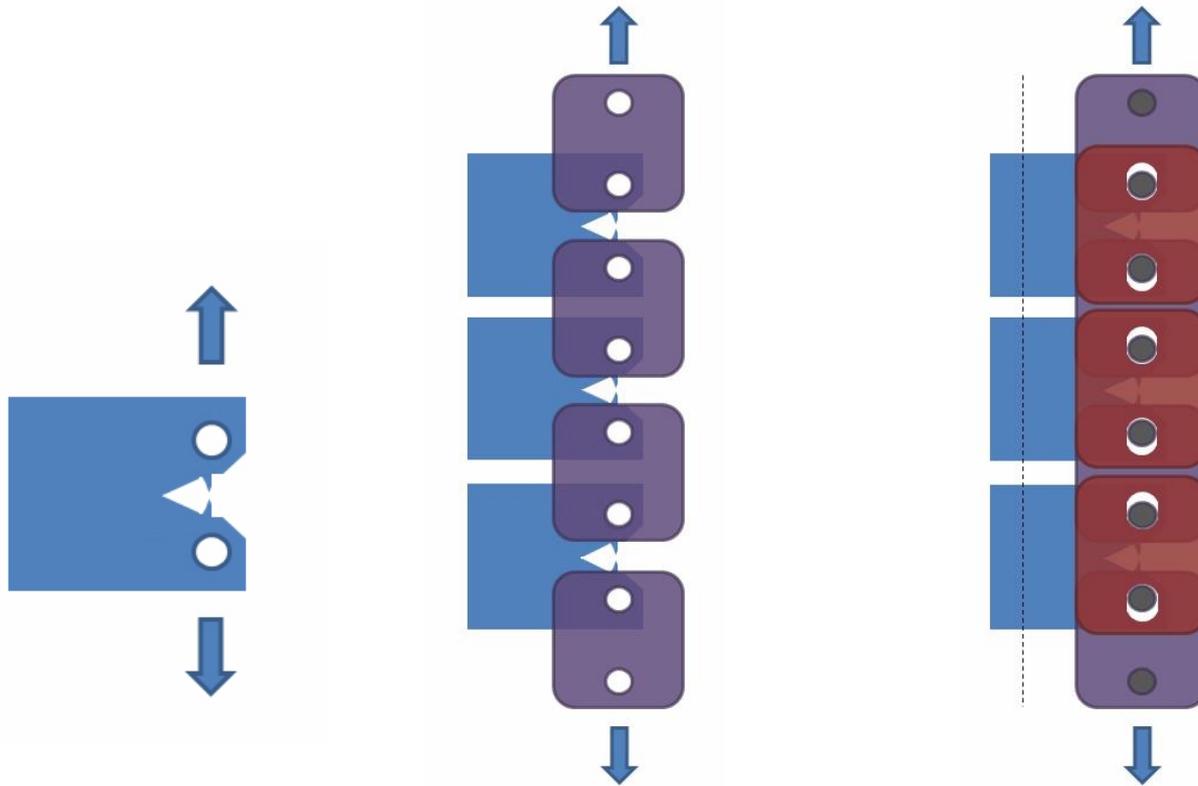


# Test Capabilities

- Multiple-specimen fatigue

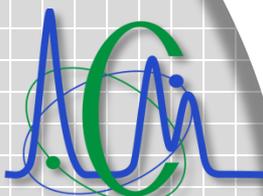
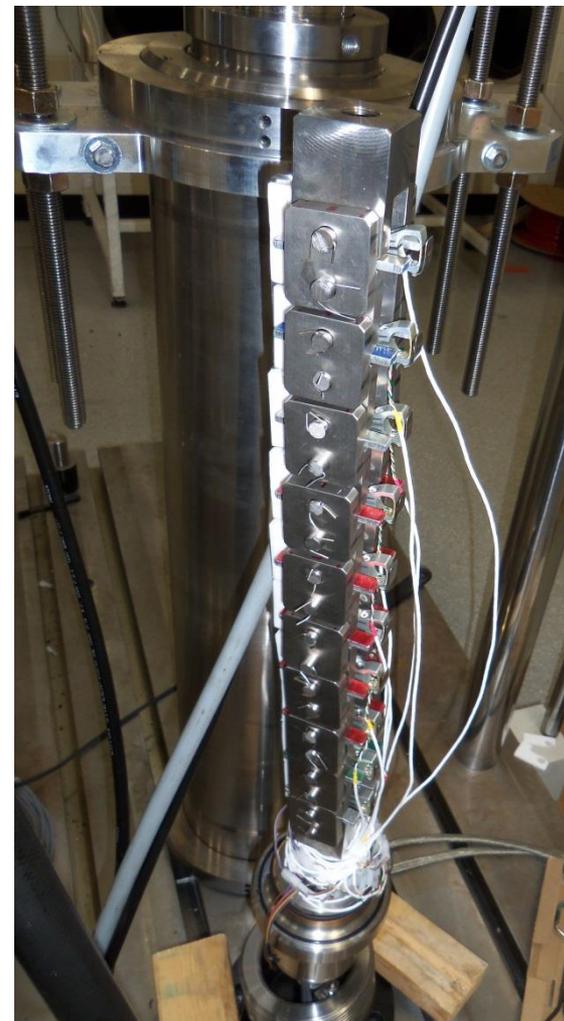


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# Test Vessels

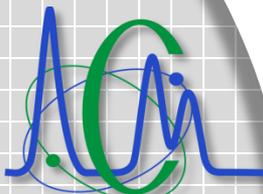
- Small vessel same as Sandia's
- Large vessel has inner length of 813 mm (32 in.) and inner diameter of 127 mm (5 in.), 316 SS, same end caps



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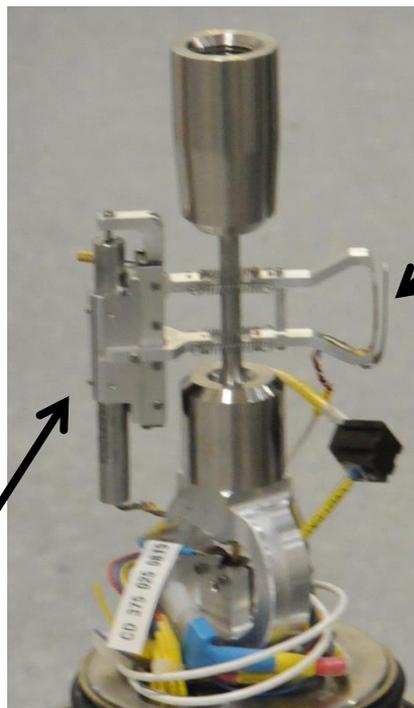
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# Transducers



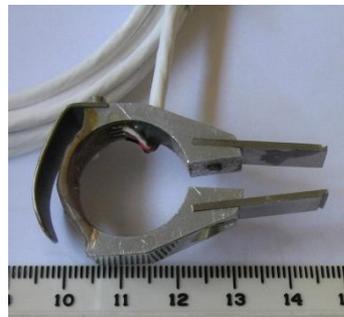
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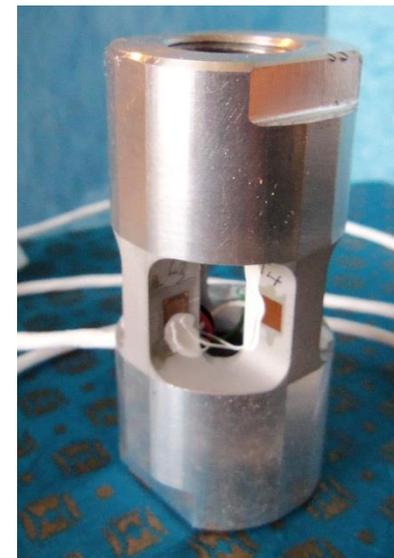


Vented-type  
LVDT  
extensometer

Strain-gauged  
extensometer



Strain-gauged  
clip gauge

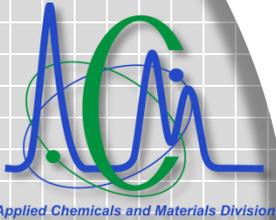


Strain-gauged  
22 kN (5 kip)  
Load cell



44 kN (10 kip)  
proving ring  
load cell (vented  
LVDT)

# Hydrogen Compressors



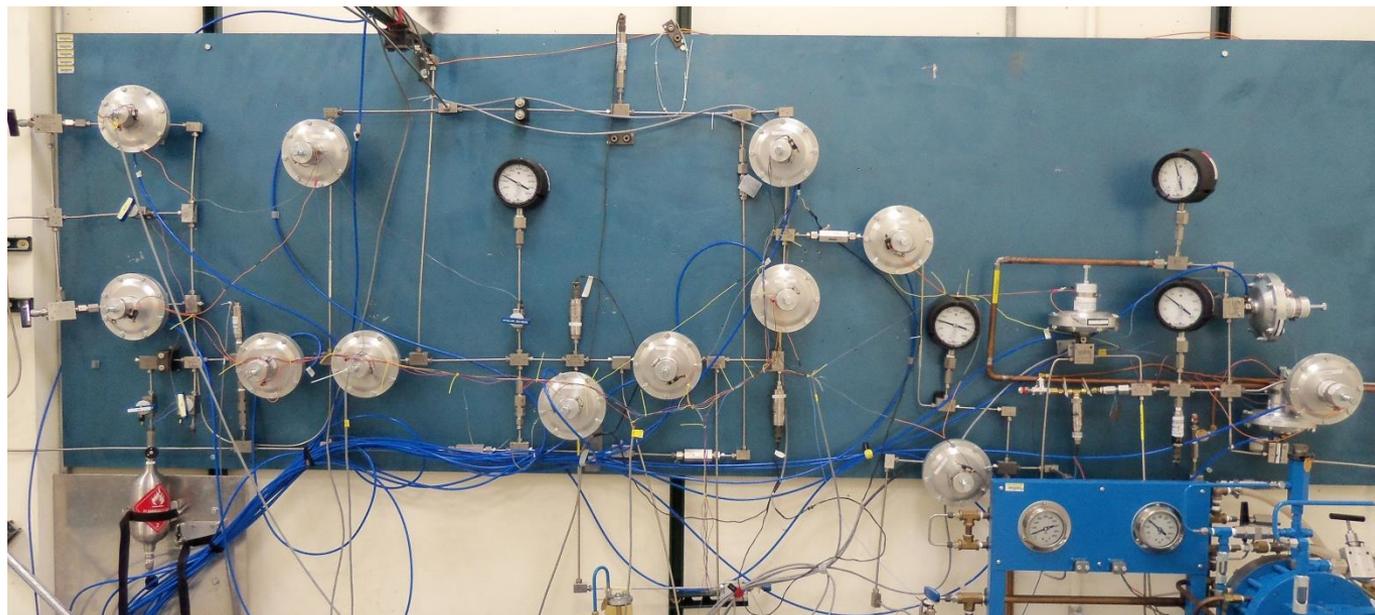
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- 41 MPa (6000 psi)
- 150 MPa (22,000 psi)
- Both are diaphragm-type



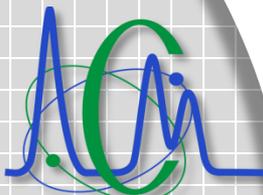
# Gas Supply System

- Labview automated control
- Accumulator for each test chamber to lessen the load on the compressors
- Micro-needle valves used for de-pressurization at  $<69$  kPa (10 psi) per minute
- Gas sampling of 1 liter, 6 MPa (900 psi) samples
- Supply interlocked to ventilation systems and hydrogen sensor alarm



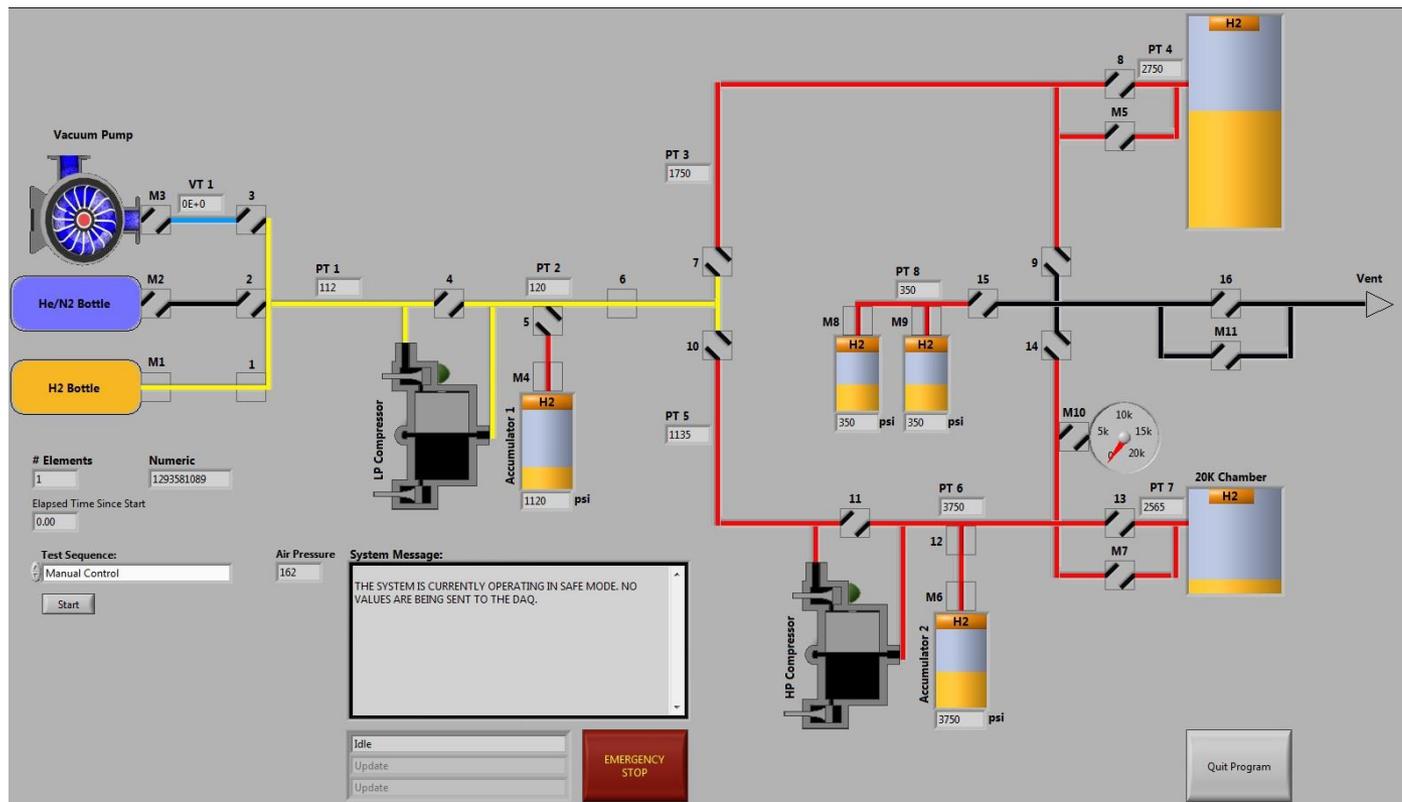
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# Gas Supply System



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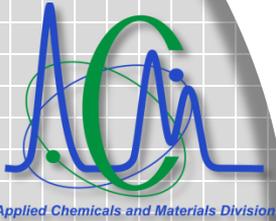
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# Safety

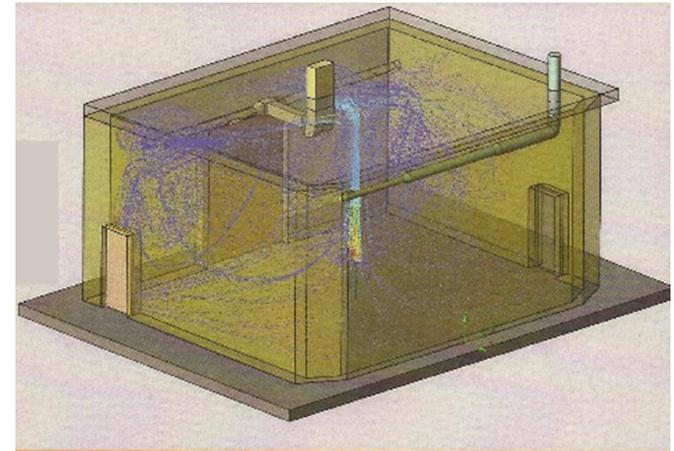
- Small Volume of Gas
- Large Building
- Ventilation
- Hydrogen Sensors
- “Fail-Safe” Design
- Minimize Spark Sources
- Explosion-Proof Camera
- Explosion-Proof Intercom between H2 lab and control room
- ASME rated vessels
- Automatic, remote operation from control room, with manual overrides
- Hydrogen lab on far side of blast wall



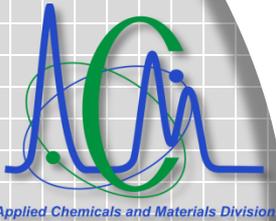
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# Safety

- Ventilation: 7 air changes per hour, 100 % outside air



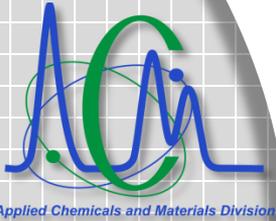
- Hydrogen Sensors
  - Catalytic bead
  - Palladium thin-film



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# Safety

- “Fail-safe” design
  - Normally-closed air-operated valves
    - Backup manual valves
  - Explosion-proof or intrinsically-safe electronics in top 2/3 of lab height
  - System locks down upon ventilation loss, power loss, compressed air loss, or hydrogen alarm
  - Hydrogen-compatible materials



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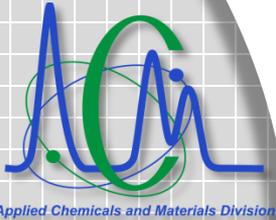
# Issues

- Slow decompression
  - Sandia informed us of it
  - Initially used automatic valves and automated procedure to slowly decompress
    - Failures of automatic valves (stuck closed)
  - Currently using micro-metering valves and simpler automated procedure
  - Recalibrate clip gauges for each run
- High leak rate
  - Changed assembly procedure and increased torque on end-cap seal
- Pull Rods
  - Galling between pull rod and “slip washer”
  - Changed assembly procedure, see if it works



# Issues

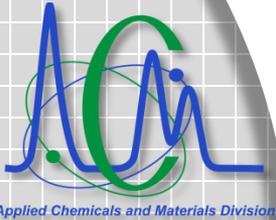
- Strain-gauge-based load cell
  - ~1% uncertainty
    - Mass of specimen string 180 to 220 N (40 to 50 lbs) and stiction of seals
    - Measure compliance for crack growth and load difference for stress intensity factor range
      - Both differential, relative measurements
- Fluitron Gas Compressors
  - Require frequent diaphragm changes
  - Difficult to re-prime



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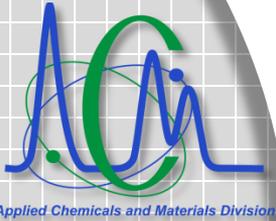
# Issues

- Remote Site
  - Power outages
    - At least 3 per year
    - Not enough power: add a load frame (pre-cracking)
  - Chiller
    - Too large, so it freezes
    - Use water, dump 6000 gallons per day on the ground
  - Air Compressor
    - Screw-type, high-reliability
    - Oil leaks, control switch failure

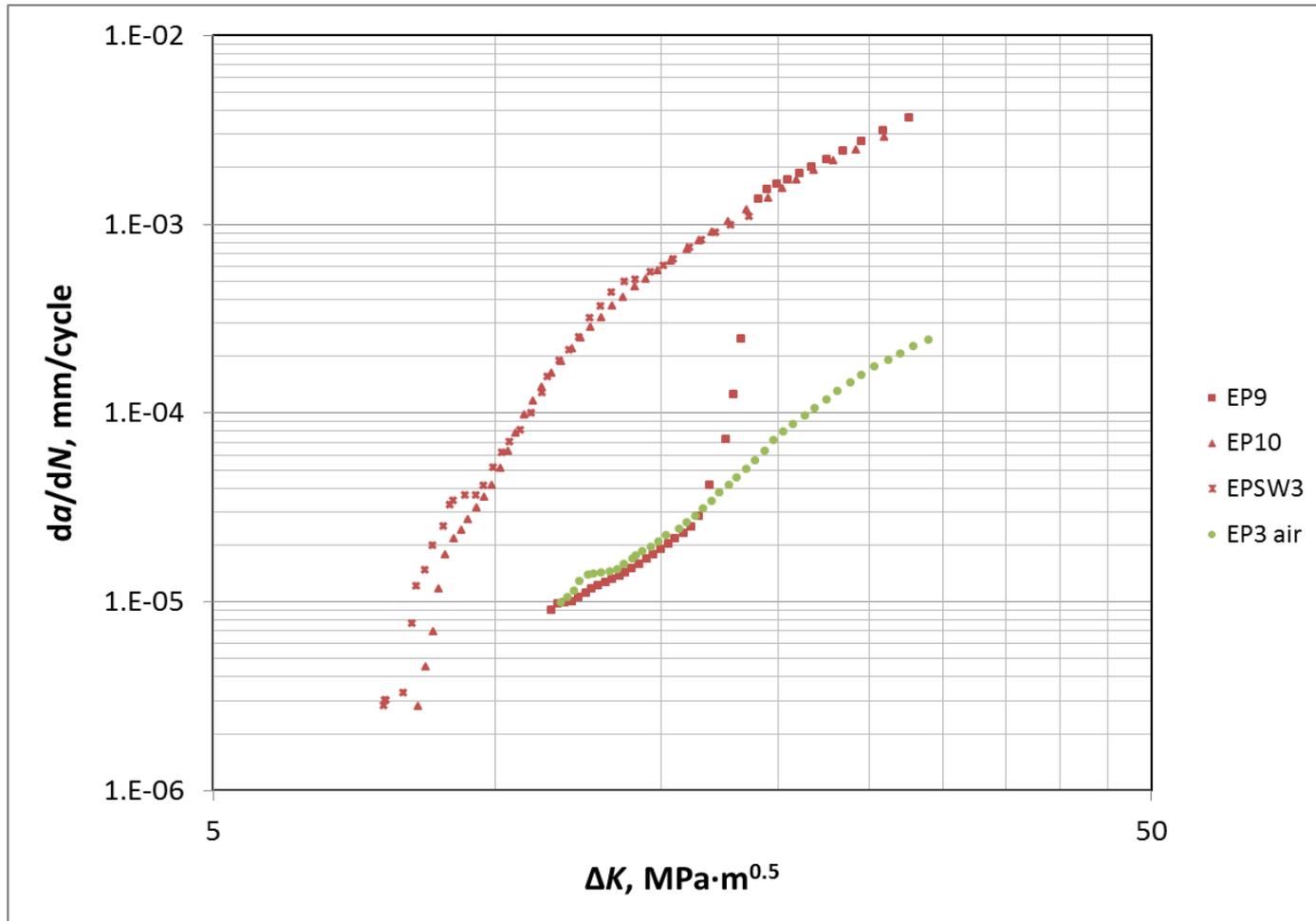


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# Gas Contamination

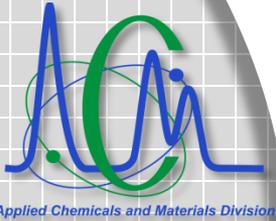


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# Acknowledgements

- SNL Team: Brian Somerday, Chris San Marchi, Ken Lee, David Zanini, Kevin Nibur



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