

# **Photo-Stimulated Luminescence Spectroscopy Stress Sensor for In-Situ Stress and Behind Casing Cement Integrity Measurement**

**Y. Polsky, M.J. Lance and C. Mattus – ORNL**

**R.J. Daniels – UTK**

**Barry Freifeld – LBNL**

**Barbara Kutchko – NETL**

**Doug Blankenship and Adam Foris – SNL**

---

U.S. Department of Energy

National Energy Technology Laboratory

Mastering the Subsurface Through Technology, Innovation and Collaboration:

Carbon Storage and Oil and Natural Gas Technologies Review Meeting

August 16-18, 2016

# Presentation Outline

---

- Project background
- Technical approach
- Results to date
- Summary and potential path forward

# Project Overview: Goals and Objectives

---

## In Situ Stress Measurement

- Existing *in situ* stress measurement methods are either complex to implement or overly interpretive
  - Minifracs
  - Borehole imaging (breakouts)
  - Overcoring
  - Sleeve fracturing

## Cement Stress Measurement

- No direct measurements of cement stress behind casing made in field today
  - Permanent state of health monitoring needed
  - Field research tool for better understanding of cement loading

**Goal** - Adapt previously demonstrated method for measuring stress in ceramic materials to develop:

- 1) Borehole in situ stress sensor and
- 2) Cement stress condition sensor

# Benefit to the Program



## Subsurface Control for a Safe and Effective Energy Future

### Adaptive Control of Subsurface Fractures and Fluid Flow

Intelligent Wellbore  
Systems

Subsurface Stress &  
Induced Seismicity

Permeability  
Manipulation

New Subsurface  
Signals

Energy Field Observatories

### Benefits –

- 1) Characterize in situ stress magnitude and direction
  - Simpler implementation
  - Better directional resolution
- 2) Directly measure cement stress
  - Utilize standard fiber behind casing methods
  - Does not depend on mechanical coupling of sensor to cement

# Background - Issues with Borehole in situ Stress Measurements

- Hydrofrac methods
  - Conventional interpretation of test possible only if borehole axis aligned with one of major principal stress axes
  - Borehole axis must be in induced fracture plane (best for vertical wellbores)
  - Typically assumes linear elastic, homogeneous rock properties to determine in-plane principal stresses
  - Pre-existing fractures near wellbore can produce erroneous interpretation of test results

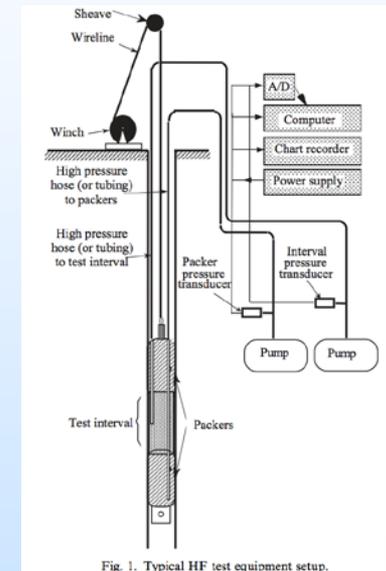
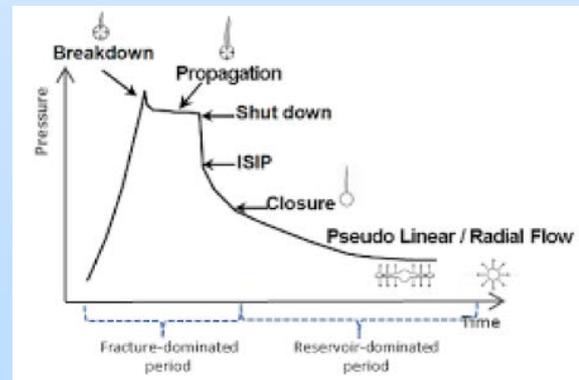
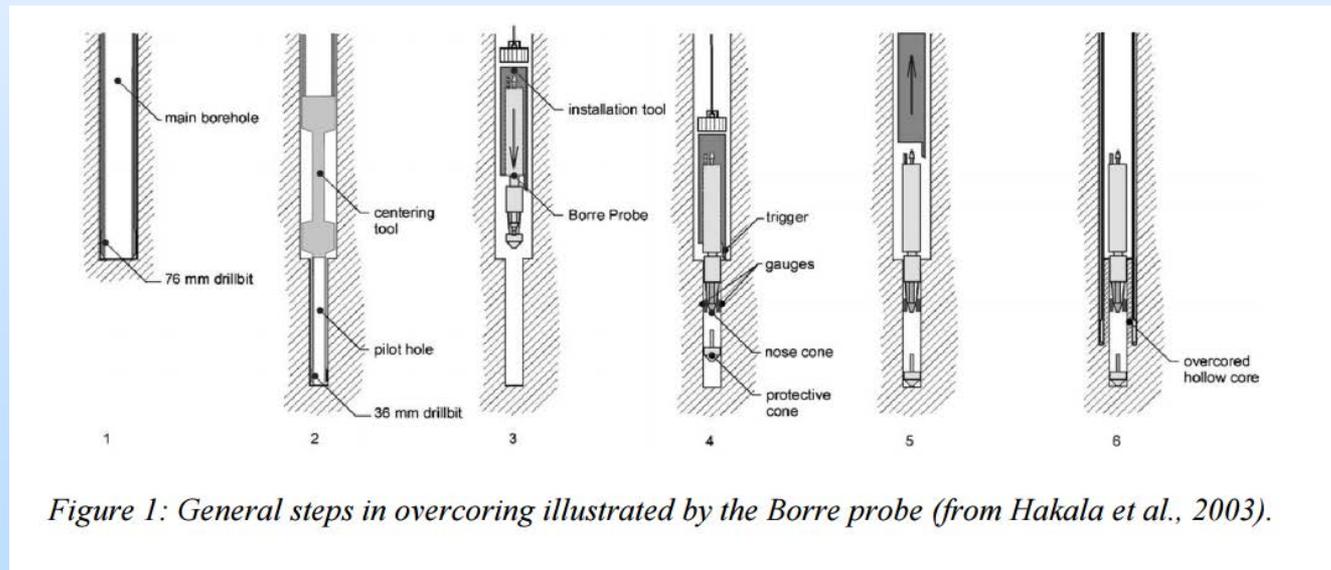


Fig. 1. Typical HF test equipment setup.

Haimson et al, 2003

# Issue with Overcoring Stress Measurements

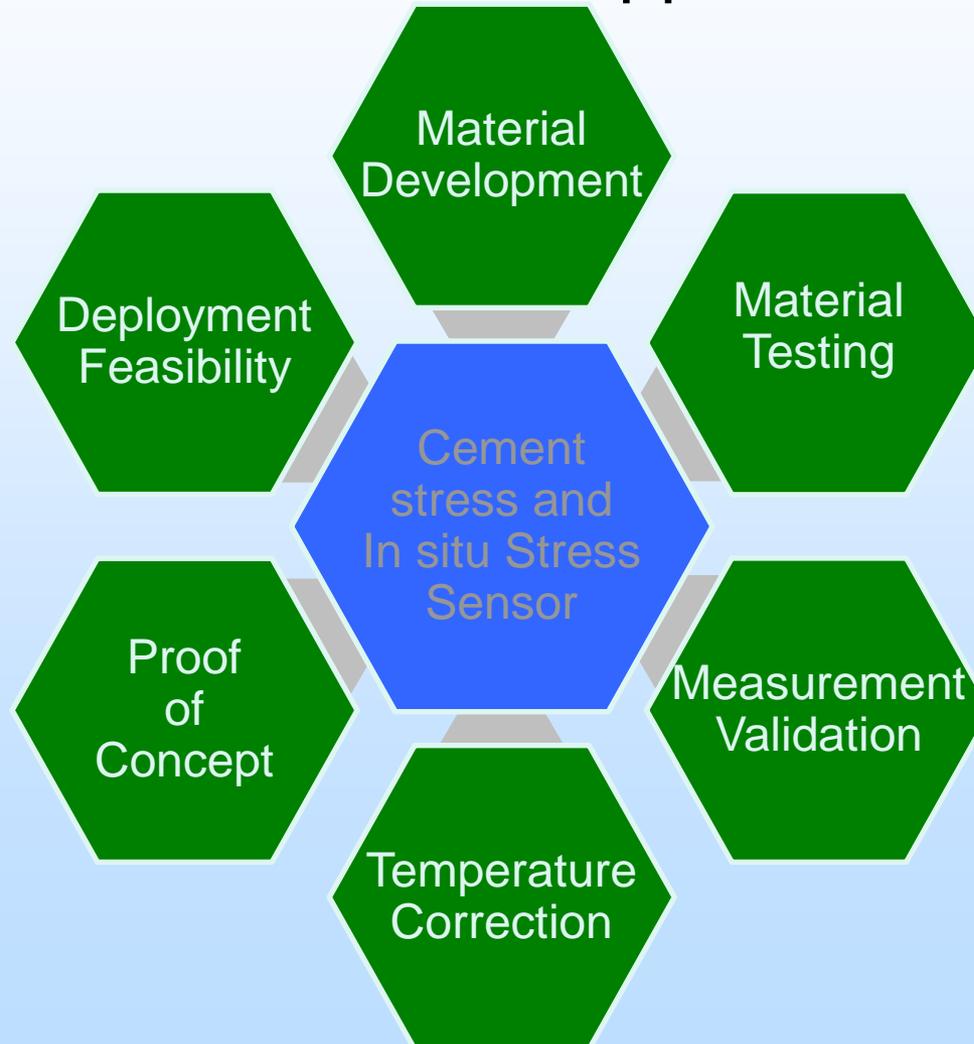
- Operation can be complex to implement and involves mechanically complex equipment
- Requires precise mechanical engagement with pilot hole wall
- Depth limited
  - Claims that can be done up to 2 km, but only done in field up to 1 km



Deformation Gauge

# Technical Status

## Technical Approach



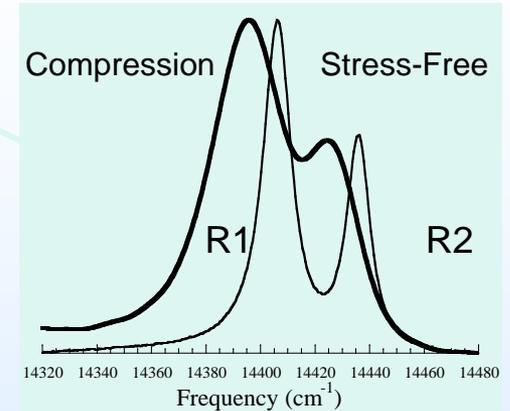
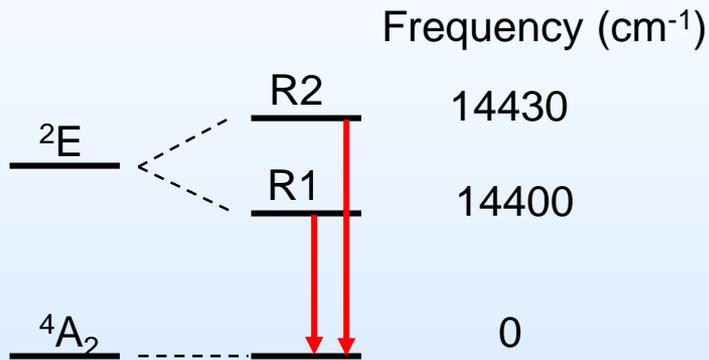
# Project Organization

---

- ORNL - Lead
  - Develop and characterize stress sensing material
  - Develop temperature compensation approach
- NETL
  - Evaluate market and performance impacts of alumina doping of API Cements
- SNL
  - Evaluate feasibility of deploying stress sensing cement
- LBNL
  - Evaluate feasibility of measurement through fiber and deployment
  - Compare PS measurement to fiber interferometric measurement
- Reno Refractories – Industry Participant

# Basis for PSLS Stress Sensing

## Luminescence of Cr<sup>3+</sup> in Al<sub>2</sub>O<sub>3</sub>



The peak shift gives the mean hydrostatic stress in randomly-oriented alumina.

Stress changes the distance between the ion and the surrounding crystal which causes the energy levels to shift:

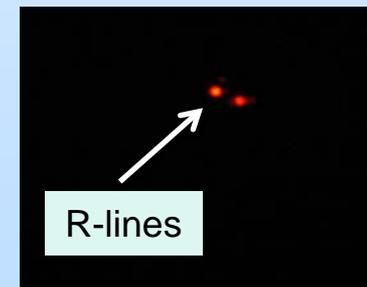
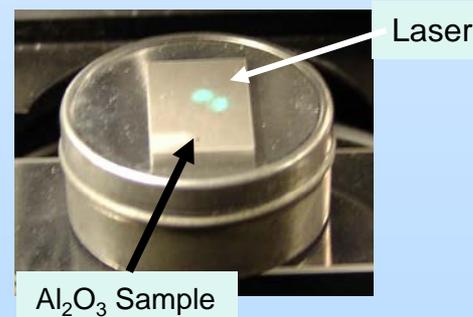
$$\Delta\nu = \Pi_{ij} a_{ik} a_{jl} \sigma_{kl}$$

For isotropic polycrystalline  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>:

$$\Delta\nu = \frac{1}{3}(\Pi_{11} + \Pi_{22} + \Pi_{33})(\sigma_{11} + \sigma_{22} + \sigma_{33}) = \Pi_{ii} \sigma_h$$

Incident Laser Wavelength: 515 nm

With Filter: Emission at ~690 nm



# Material Development Overview

Characterize  
alumina  
powders

Develop  
cement mixing  
procedures

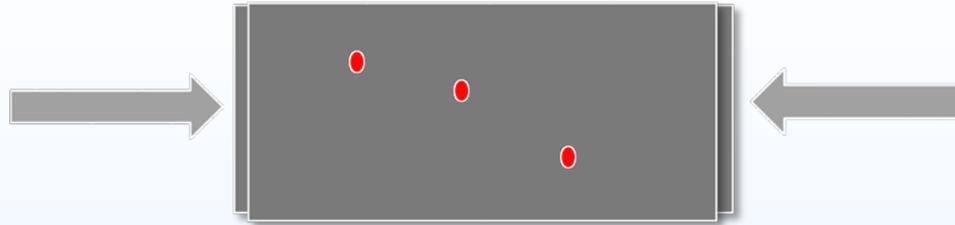
Fabricate  
cement  
samples

Characterize  
cement PS  
uniformity

## Stress sensing material performance criteria

1. R1 and R2 peaks should be within  $0.01 \text{ cm}^{-1}$  for uniform stress state
2. Stress transfer between cement matrix and alumina must be adequate to capture minimum stress level
3. Grout must be slightly expanding to react displacement of borehole wall
4. Intensity of R-lines should be adequate to perform measurement in  $< 1$  minute

# Measurement Uniformity Criterion



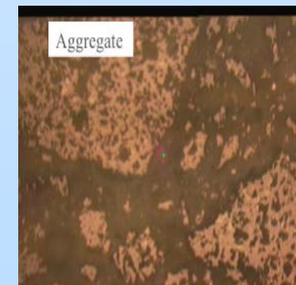
## Factors affecting measurement uniformity

- Distribution of alumina in matrix
- Cementing of alumina to matrix
- Microscopic features
- Residual stresses in alumina



$$\Pi_{ii} = 7.62 \frac{cm^{-1}}{GPa} \text{ for polycrystalline Alumina}$$

Target stress resolution is 1 MPa



# 1<sup>st</sup> Powder Measurements – Small Spot Diameter

Standard deviation and intensities of commercially available alumina powders for 10  $\mu\text{m}$  spot size

Powder Description	Particle Size ( $\mu\text{m}$ )	Spot Size	STD ( $\text{cm}^{-1}$ )	Peak Width ( $\text{cm}^{-1}$ )	Intensity (counts/s)
Alcoa A2 Unfired	150	10 $\mu\text{m}$	0.0416	9.7270	27212.90
Inframat 200 nm @ 1200°C	0.2	10 $\mu\text{m}$	0.0528	9.4101	45291.20
Inframat 200 nm @ 1400°C	0.2	10 $\mu\text{m}$	0.0574	9.4329	29933.80
Alcoa A2 fired @ 1400°C	150	10 $\mu\text{m}$	0.0911	9.8466	39840.60
Inframat 35 $\mu\text{m}$	35	10 $\mu\text{m}$	0.0930	9.4645	744.30
Type DX 0.3 $\mu\text{m}$	0.3	10 $\mu\text{m}$	0.0965	9.0934	185.13
Inframat 20 $\mu\text{m}$	20	10 $\mu\text{m}$	0.0977	9.5480	4733.30
Inframat 200 nm @ 800°C	0.2	10 $\mu\text{m}$	0.1008	10.1625	4046.38
Type N 0.3 $\mu\text{m}$	0.3	10 $\mu\text{m}$	0.1025	9.3237	122.29
Inframat 200 nm	0.2	10 $\mu\text{m}$	0.1348	12.6365	8336.12
SigAld 10 $\mu\text{m}$	10	10 $\mu\text{m}$	0.1434	10.3200	13770.60
Inframat 12 $\mu\text{m}$	12	10 $\mu\text{m}$	0.1501	11.4908	3073.11
Type DX 1 $\mu\text{m}$	1	10 $\mu\text{m}$	0.1543	9.5016	993.38
Inframat 150 nm	0.15	10 $\mu\text{m}$	0.1547	12.9824	9759.59
Inframat 15 $\mu\text{m}$	15	10 $\mu\text{m}$	0.1600	10.4740	1052.95
US Research Nanomaterials 80 nm	0.08	10 $\mu\text{m}$	0.2062		
Inframat 5 $\mu\text{m}$	5	10 $\mu\text{m}$	0.2072	12.6365	4707.27
Inframat 25 $\mu\text{m}$	25	10 $\mu\text{m}$	0.2654	9.9683	1508.66
Inframat 10 $\mu\text{m}$	10	10 $\mu\text{m}$	0.3162	11.7383	5864.28
Inframat 200 nm @ 600°C	0.2	10 $\mu\text{m}$	0.3560	11.4219	6339.77
Inframat_1 to 1_4 $\mu\text{m}$		10 $\mu\text{m}$	0.4203	12.8563	4452.89
Inframat 200 nm @ 1000°C	0.2	10 $\mu\text{m}$	0.5726	10.2499	3069.41
Inframat 3 $\mu\text{m}$	3	10 $\mu\text{m}$	0.7222	12.9111	5015.05
Alfa Aesar 1 $\mu\text{m}$	1	10 $\mu\text{m}$	0.8741	10.5240	2175.00

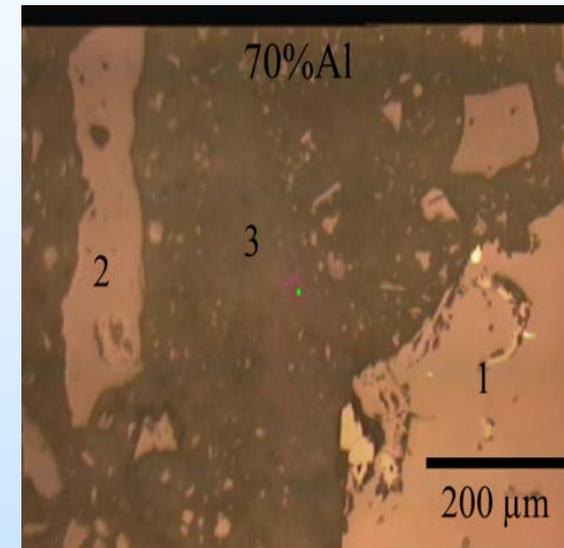
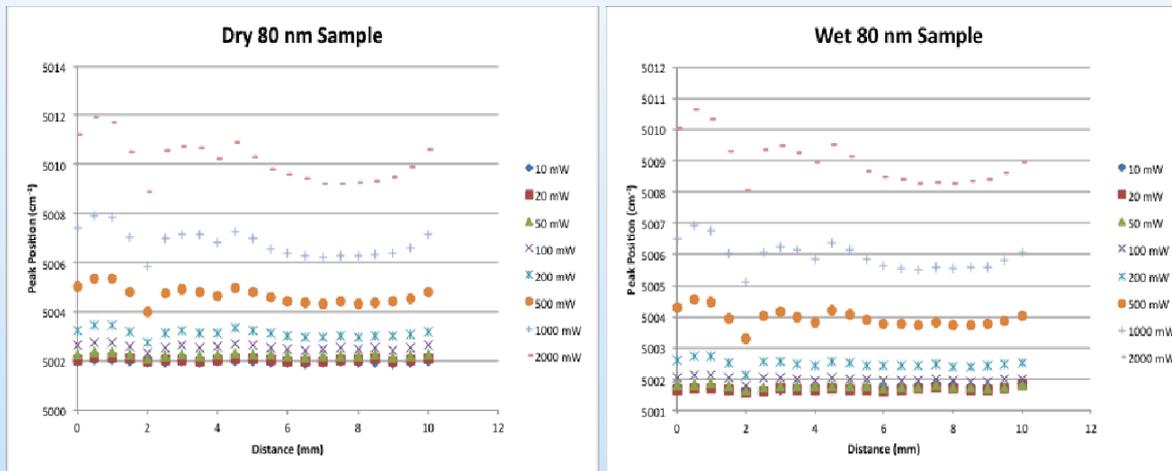
# 2<sup>nd</sup> Powder Measurements – 1 mm Spot Diameter

Standard deviation and intensities of commercially available alumina powders for 1 mm spot size

Powder Description	Particle Size (um)	Spot Diameter	STD (cm <sup>-1</sup> )	Peak Width (cm <sup>-1</sup> )	Intensity (counts/s)
Sigma Aldrich 10 um	10	1mm	0.0055	10.2788	2852.63
Inframat 200 nm @ 1000°C	0.2	1mm	0.0124	9.7918	1151.45
Type N 0.3 um	0.3	1mm	0.0127	9.4725	102.43
Inframat 35 um	35	1mm	0.0130	11.6449	263.16
Inframat 200 nm @ 1400°C	0.2	1mm	0.0173	9.2901	12329.90
Inframat 200 nm @ 600°C	0.2	1mm	0.0178	10.5327	1046.06
Inframat 25 um	25	1mm	0.0182	9.9849	654.75
Inframat 200 nm @ 800°C	0.2	1mm	0.0198	9.9146	1160.60
Inframat 15 um	15	1mm	0.0252	10.1887	185.54
Inframat 40 nm	0.04	1mm	0.0322	12.5234	1641.16
Alfa Aesar 1 um	1	1mm	0.0329	10.2526	672.90
Inframat 200 nm @ 1200°C	0.2	1mm	0.0370	10.2630	1353.84
Inframat 10 um	10	1mm	0.0410	12.0871	1538.6399
Inframat 12 um	12	1mm	0.0421	11.0832	624.99
Inframat 20 um	20	1mm	0.0524	10.1162	723.68
Research Nanomaterials 80nm	0.08	1mm	0.0530	11.1269	12146.36
Inframat 200 nm Control	0.2	1mm	0.0542	11.7894	758.22
Inframat 3 um	3	1mm	0.0664	12.0871	745.35
Type DX 0.3 um	0.3	1mm	0.1496	9.4013	110.12
Type DX 1 um	1	1mm	0.1601	9.5419	215.09
Inframat 5 um	5	1mm	0.1648	12.2630	874.08
Inframat 100 nm	0.1	1mm	0.1921	12.9499	653.57

# Laser Power Effect on Piezospectroscopic Response

- Higher laser power levels have larger measurement standard deviations
- Likely due to material heterogeneity and thermal conductivity variation away from illumination point

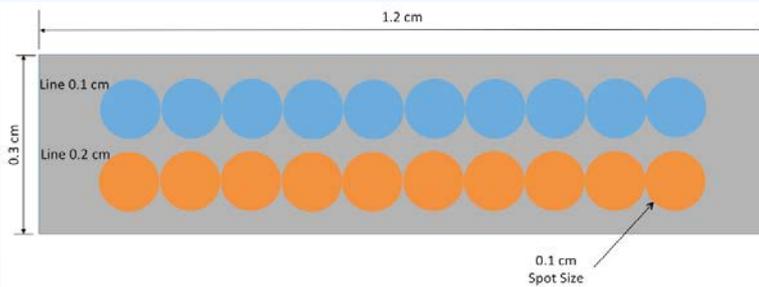


R2 peak position measured for 1 mm spot size at different locations along a 10 mm line segment on dry (left) and wet (right) API class H cement sample with 33.3% 80 nm alumina solid volume fraction for different laser power magnitudes

**Mixing of material is important for producing uniform response!**

# Spatial Variation of Peak Position for Cast Samples

2D spatial scan locations

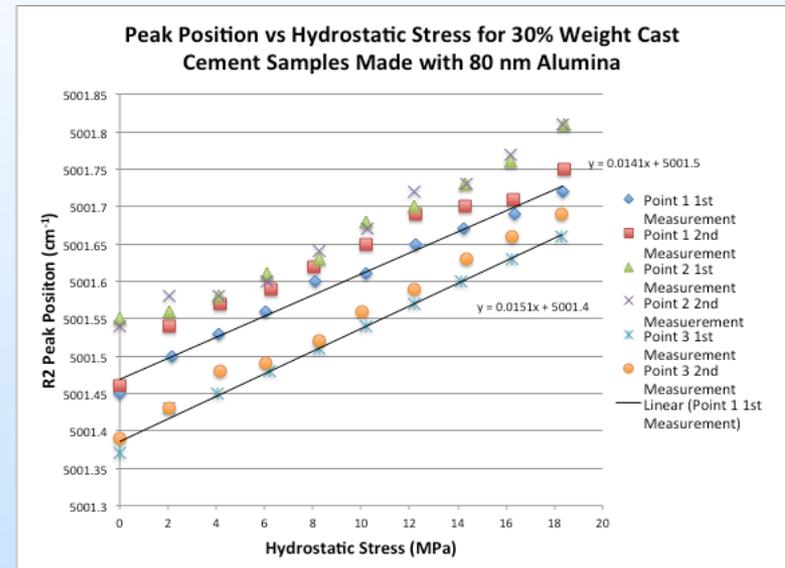
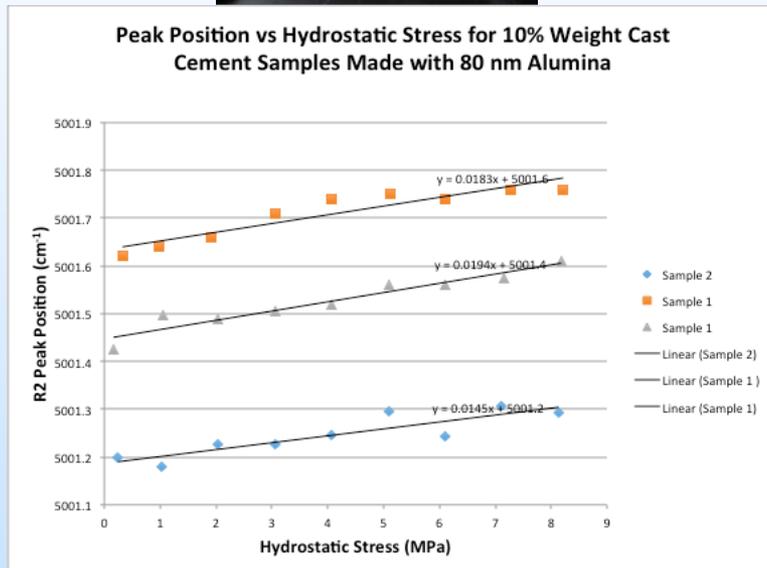
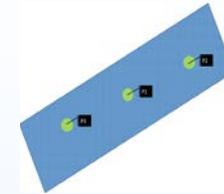
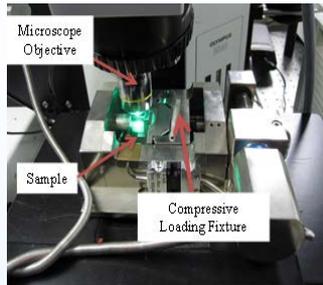


Average R2 Peak Position and Standard Deviation for Samples Cast with Different Alumina Powders

Composition	Avg. R2 PP (cm <sup>-1</sup> )	STD PP Position (cm <sup>-1</sup> )
<b>300 nm @ 30 wt%</b>	14434.7653	0.027286038
<b>80 nm @ 30 wt%</b>	14434.28727	0.03178992
<b>Secar71</b>	14434.701	0.037983206
<b>80 nm @ 5 wt%</b>	14434.23765	0.055967721
<b>35000 nm @ 5 wt%</b>	14433.84281	0.088187525
<b>ThermaLock</b>	14434.19393	0.241981507
<b>5000 nm @ 5 wt%</b>	14433.92162	0.272124763

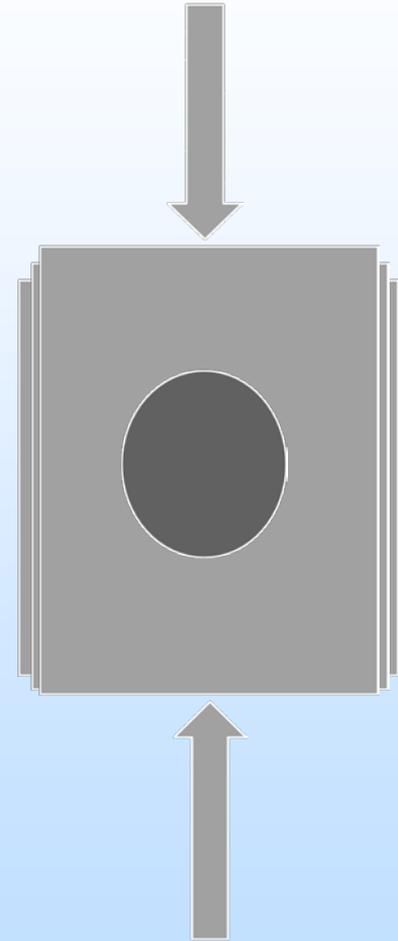
- Spatial variability of peak position for cast samples comparable to powder values for small mean particle size
- There appears to be larger variation for larger mean particle sizes

# Uniaxial Compression Test Piezospectroscopic Response

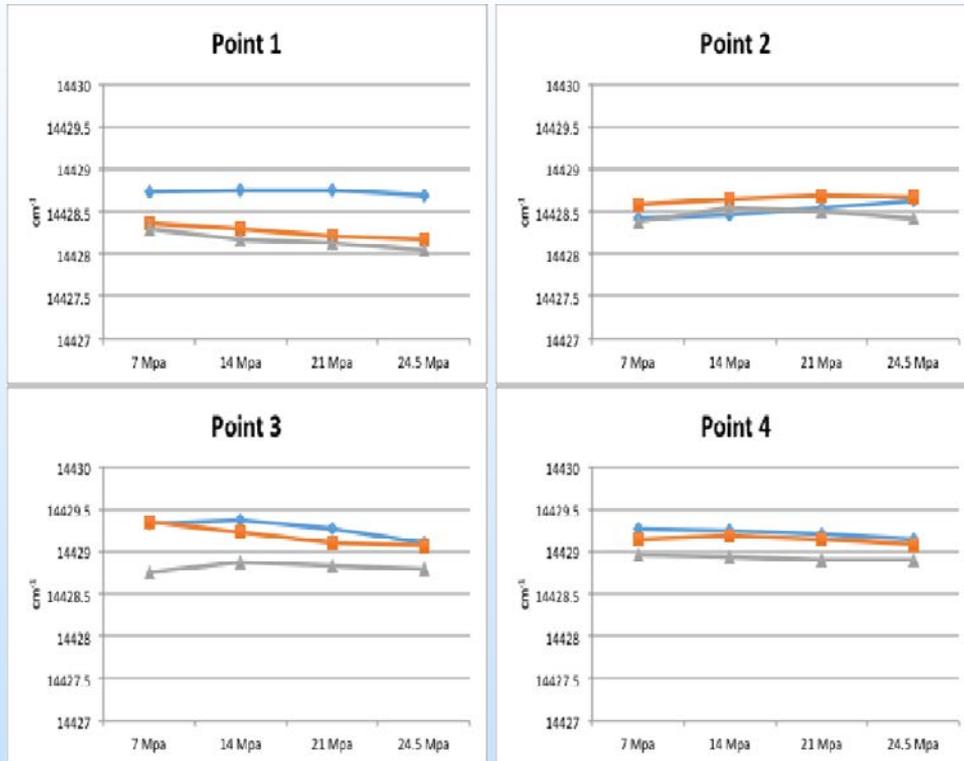
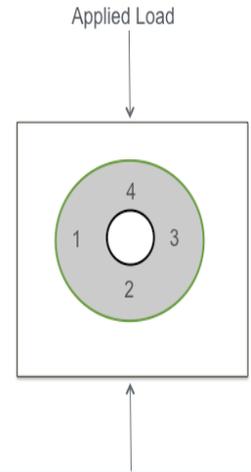


- Slopes of curves (PS coefficient) are reasonably consistent
- Good response even in lower alumina concentration cement
- PS coefficient indicates that there is a stress concentration effect with nanoparticles

# In Situ Stress Sensor Proof of Concept Laboratory Test Setup



# Proof-of-concept experiment results



R2 peak position vs hydraulic press pressure at different measurement locations

- Decreasing peak position corresponds to increased compressive stress
- Expect compressive stress of points 1 and 3 to increase with increasing load and be 3x larger than change of points 2 and 4
- Trends not consistent
- Temperature effects not accounted for

Results inconclusive!

# Accomplishments to Date

---

- Characterized suitability of large number of  $\alpha$ -Alumina powders for use as dopants
- Identified numerous factors that affect piezospectroscopic behavior of doped cements
- Developed API Class H and Portland Cement formulations that exhibit reasonably consistent PS response
- Have evaluated deployment feasibility of ‘smart’ cement

# Synergy Opportunities

---

- ‘Smart’ cement could help understand actual loads experienced by wells during operation
- Field application of stress measurements using developed material could be associated with a number of recently created field demonstration sites

# Summary

---

- Progress has been made developing a novel stress sensing material for borehole applications
- Alumina powder can exhibit PS variability that limits use as a sensor so powder selection and preparation is important
- Mixing of cement/alumina important for producing homogeneous composite
- Power level of laser stimulation can affect measurement
- Preliminary stress sensing cement samples exhibit initial load transfer issues but excellent linearity when stress transfer begins
- Results to date are encouraging and indicate that material can be used for stress measurement applications with further development

# Appendix

---

# Gantt Chart



# Bibliography

---

- Y. Polsky, R.J. Daniels, M. Lance, and C. Mattus, “Development of a Novel Stress Sensor for In Situ Stress Measurement”, *Proc. 41st Workshop on Geothermal Reservoir Engineering*, Stanford Univ., Stanford, CA, February 22-24, 2016.
- Other manuscripts currently in preparation