# Exploring Nonlinear Mechanical Behaviour of Rocks at LANSCE

Los Alamos Neutron Science Center

Los Alamos NATIONAL LABORATORY

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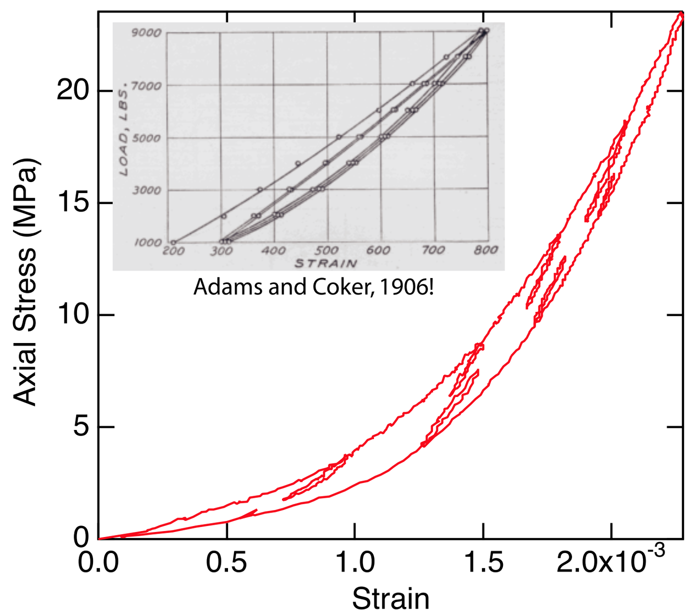
## MOTIVATION

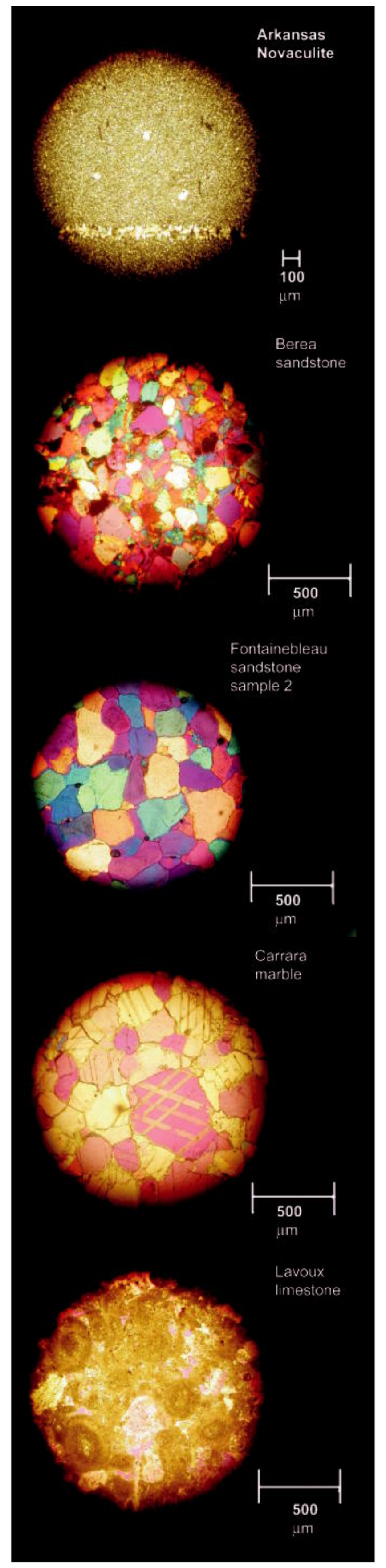
* Although rocks have been studied by macroscopic mechanical experiments for a long time, the complexity, inhomogeneities, multiple phases, fluid content, interfaces, and lack of transparency have defied most experiments to resolve the corresponding microscopic behavior associated with peculiar nonlinear macroscopic observations.
* *Neutron diffraction* is one of a few experiments that can lead to an understanding of the atomic or microscopic effects that govern the properties of complex geomaterials. Neutrons can easily penetrate rocks and reveal properties of the bulk interior material (rather than the near-surface regions measurable by X-rays).
* We are performing experiments with rocks on three beamlines at the Lujan neutron facility where the scattering data will be correlated with the large body of nonlinear acoustic data which exists, to determine *which atomic-plane level constituents of the rocks are active* in nonlinear processes.

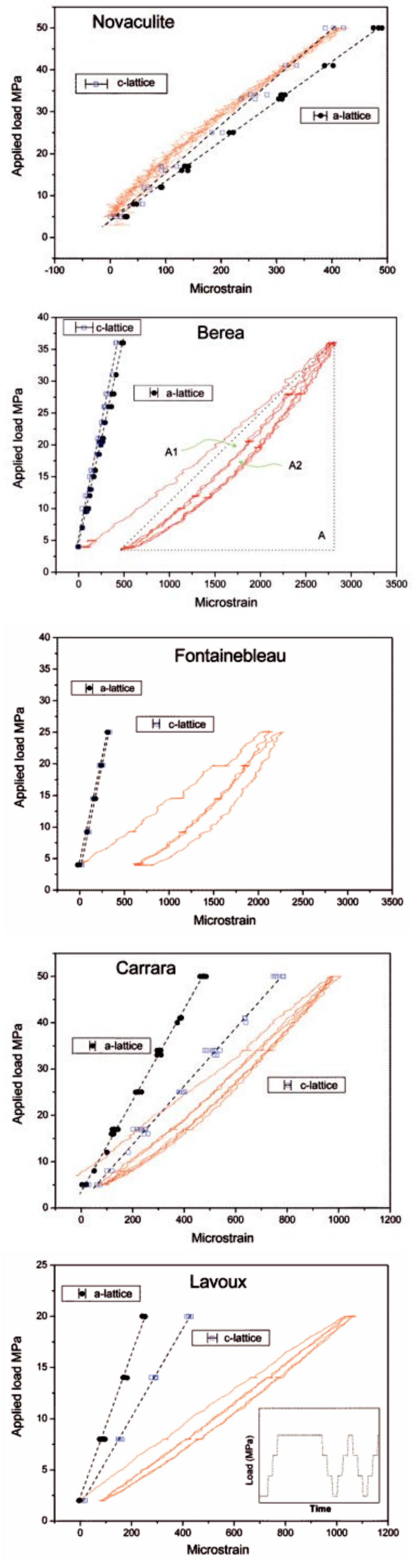
## SMARTS

Spectrometer for Materials Research at Temperature and Stress



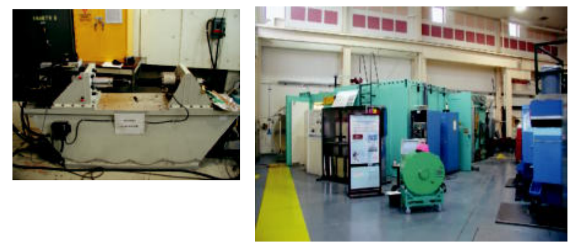

Macroscopic Stress-Strain curves for Berea sandstone and granite (inset). Note hysteresis, inner loops, and end-point memory.



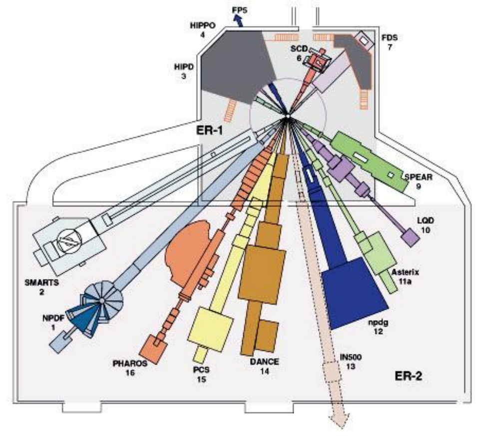




Atomic-scale stress-strain information obtained from the neutron Rietveld data indicate that the strain experienced by the crystalline quartz is ~1/5 of the macroscopic strain (the rest taken up by the grain contacts and bonds in the rock). No hints of nonlinearity whatsoever are evident in the neutron data. Conclusion? The grain bond system (a small fraction of the total rock) is responsible for all the peculiar quasi-static nonlinearity we see.

### SMARTS



Special horizontal Instron Load Frame (left) which goes in the SMARTS "cave" at the end of the beam (right)



### NPDF

A photo of the newly-upgraded Neutron Powder Diffractometer

### HIPPO

Data collection racks, HIPPO cover, our data collection equipment

*Beamlines at the LANSCE (Los Alamos Neutron Science Center)/Lujan Center.*

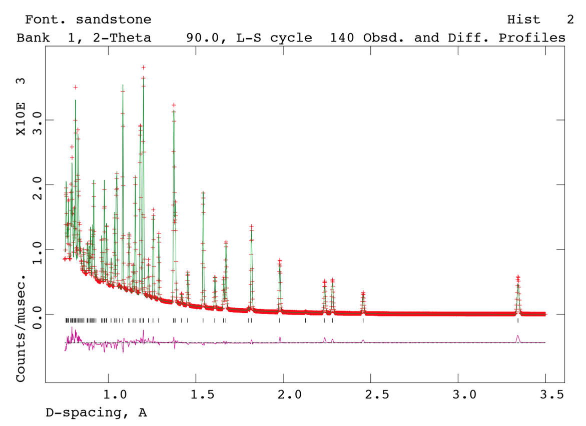
*LANSCE produces intense sources of pulsed protons and spallation neutrons from a tungsten target.*

*Proton beam currents during all the experiments varied from 100 to 110 $\mu$A.*

## NPDF

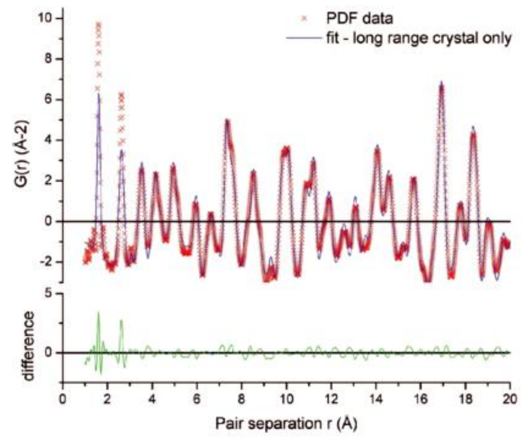
The Neutron Powder Diffractometer has the unique capability of simultaneous high-Q Rietveld and pair-density function analyses, enabling determination of the average and local structures of complex materials with high accuracy. The questions these experiments are designed to answer are (1) can neutrons "see" the grain bond system and if so, (2) can neutrons help to ascertain the role(s) of intergranular bonds vs. the bulk crystalline volume in the nonlinear behaviour of rocks? Results below show evidence of non-crystalline silica in a pure quartz sandstone.

1.



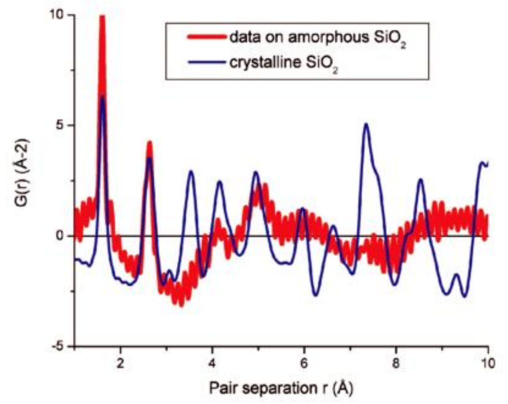
Rietveld analysis shows an excellent match with crystalline quartz—there are no other crystal phases in Fontainebleau sandstone.

2.



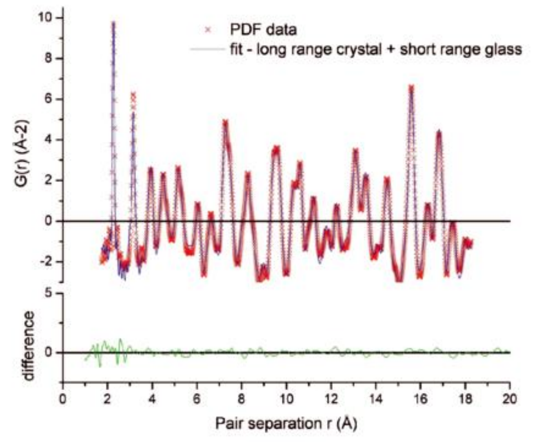

When the PDF data (red crosses) is compared to a perfect quartz model, there is a large discrepancy in the nearest neighbor peaks.

3.




PDF data (red) of amorphous silica shows that only the nearest neighbor peaks are sharp—and correspond with those of the crystal (blue).

4.

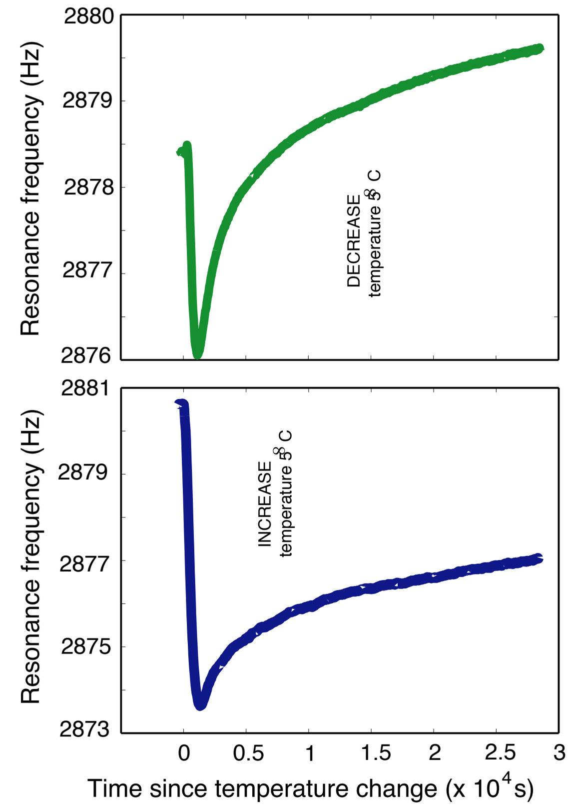



A revised model adding ~7% amorphous silica to the crystal model makes a greatly improved fit.

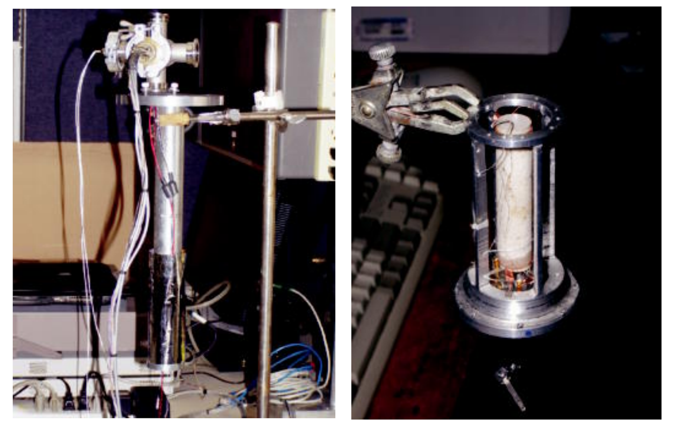
## HIPPO

High-Pressure Preferred Orientation Diffractometer

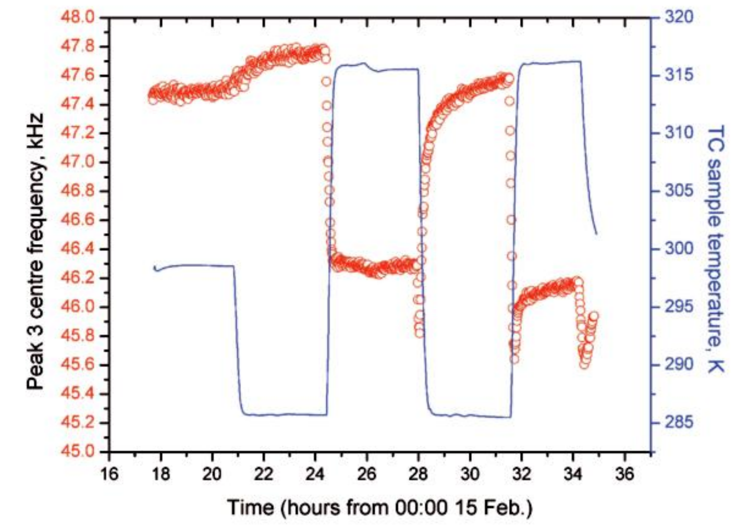
History: Modulus drop observed after a temperature change IN EITHER DIRECTION for a sample of Berea sandstone



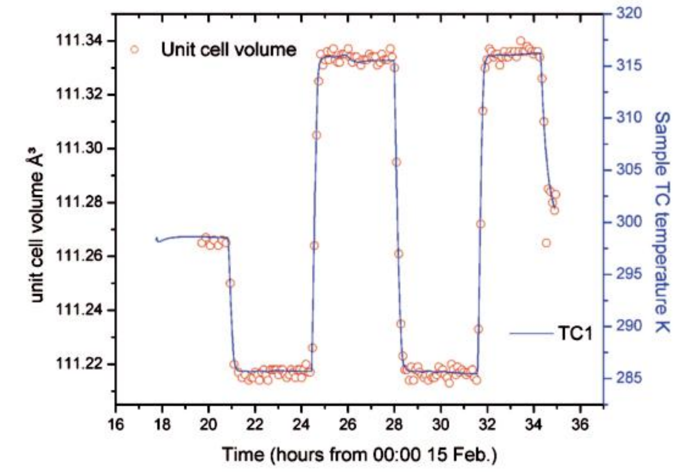

HIPPO's proximity to the neutron spallation source and its numerous detectors mean it can watch atomic plane structures change in real time. Counting for 1 minute or less is sufficient for a Rietveld analysis of the scattering data. Scattering experiments were performed to observe the crystalline structure of sandstone samples undergoing periodic temperature changes. Modulus (resonance frequency) and temperature was tracked as a function of time. Neutron results—unit cell volume—show none of the peculiar macroscopic nonlinear behavior.



Sandstone sample in holder, thermocouples, and a piezoelectric source and receiver all mounted in an isothermal temperature chamber and mylar thermal radiation shielding.




Corresponding shift of frequency as temperature changed.




Plot of temperature and unit cell volume during the experiment

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