

DEVELOPMENT OF A NEUTRON DIFFRACTION BASED EXPERIMENTAL CAPABILITY FOR INVESTIGATING HYDRAULIC FRACTURING FOR EGS-LIKE CONDITIONS

Yarom Polsky, Luc Dessieux, Ke An, Lawrence M. Anovitz, Philip Bingham and Justin Carmichael

Oak Ridge National Laboratory
1 Bethel Valley Rd
Oak Ridge, TN, 37831 U.S.A
e-mail: polskyy@ornl.gov

ABSTRACT

Understanding the relationship between stress state, strain state and fracture initiation and propagation is critical to the improvement of fracture simulation capability if it is to be used as a tool for guiding hydraulic fracturing operations. The development of fracture prediction tools is especially critical for geothermal applications such as EGS because the opportunities to build understanding empirically will be limited due to the high costs associated with field trials. There is a significant body of experimental work associated with hydraulic fracture investigation, but past efforts are typically hampered by an inability to accurately and comprehensively measure strains within the sample mass near critical regions of interest.

This work aims to develop non-destructive neutron diffraction based strain measurement techniques that can be used to interrogate the internal volume of geological specimens subjected to tri-axial stress states resembling geothermal application conditions. Demonstrating the ability of the technique to generate useful quantitative data is the primary focus at this stage of the effort. Details of the experimental setup and diffraction technique will be presented in this communication, including the description of a custom designed high-pressure, neutron scattering and imaging compatible triaxial flow cell.

INTRODUCTION

Hydraulic fracturing to enhance formation permeability is an established practice in the Oil & Gas (O&G) industry and is expected to be an enabler for EGS. However, it is rarely employed in conventional geothermal systems and there are significant questions regarding the translation of practice from O&G to both conventional geothermal and EGS applications. Lithological differences

(sedimentary versus crystalline or otherwise low permeability rocks), significantly greater formation temperatures and different desired fracture characteristics are among a number of factors that are likely to result in a gap of understanding of how to manage hydraulic fracturing practice for geothermal operations.

Whereas the O&G community has had both the capital and the opportunity to develop its understanding of hydraulic fracturing operations empirically in the field, as well as through extensive R&D efforts, field testing opportunities for EGS are likely to be minimal due to the high expense of hydraulic fracturing field trials. A significant portion of the knowledge needed to guide the management of geothermal/EGS hydraulic fracturing operations will therefore likely have to come from experimental efforts and simulation.

Prior theoretical, numerical and experimental work performed to improve the understanding of the fracture initiation and propagation process is extensive. Concise and thorough reviews of both modeling and experimental validation efforts have been reported in the literature (Mendelsohn, 1984 and Guo *et al*, 1993), but the volume of theoretical and numerical work by far exceeds that of experimental work. While the sophistication of theoretical and numerical studies has accelerated as improved theory, multiphysics code development and high-performance computing have been incorporated into simulation capability, experimental work has progressed less spectacularly with generally minor advances in characterization and measurement capabilities.

Most experimental efforts focus on developing tests that produce data in support of model validation. These typically seek to relate fluid parameters such as breakdown pressure, shut-in pressure, injection rate and leak-off rate to fracture initiation and propagation (Guo *et al*, 1993, Heuze *et al*, 1990 and Blair *et al*, 1990). They often work with artificial or

engineered rocks, such as gypsum cement or “gypstone”, which have relatively homogeneous and consistent properties. This property uniformity enables more reliable comparison of results between experiments performed with different samples of the same type and also permits more accurate comparison of experimental behavior to simulation predictions of stress/strain states near fracture initiation sites and fracture propagation characteristics. Fracture propagation has been measured using a variety of techniques including embedded wire meshes and acoustic emissions detection while stress states are usually inferred from less direct methods incorporating embedded or boundary-located load cells and strain gauges (Matsunaga *et al.*, 1993 and Heuze *et al.*, 1990).

The difficulty of measuring actual strains within geological materials is a particular shortcoming of available experimental techniques. Injection pressure histories and load cell or strain gauge data alone are inadequate for providing a detailed view of the deformation behavior of the rock. For example, previous experimental hydraulic fracture studies of actual geological materials (Westerly granite) performed in the interest of geothermal applications displayed incipient failure behavior that could not be definitively attributed to either imminent shear failure, imminent tensile failure or merely pore volume expansion because a complete strain state could not be determined for the sample in the region of interest (Solberg *et al.*, 1980). The ability to acquire detailed localized strain state information is therefore critical to informing the fracture mechanism description and propagation models that underlie simulation codes.

This paper describes ongoing efforts at Oak Ridge National Laboratory (ORNL) to develop an experimental capability to map the internal strains in core samples subjected to triaxial stress states and temperatures representative of EGS-like conditions using neutron diffraction based strain measurement techniques. There has been previous work reported in which neutron diffraction techniques were successfully used to measure microscopic strains associated with residual stress and uniaxial loading in sandstone materials (Frischbutter *et al.*, 2000 and Pintschovius *et al.*, 2000). However, the technique is admittedly immature for relatively heterogeneous geological materials for a number of reasons including: the measured microscopic strains typically take the form of lattice strains and their relationship to the more conventional macroscopic strains used in engineering applications requires a translation model; the possible sources of error associated with the measurements have not been fully characterized; and the low fluxes associated with many neutrons sources

require excessively long exposures to obtain useful data.

The capability being developed at ORNL will utilize the Spallation Neutron Source, the world's most powerful pulsed neutron source, will attempt to address the core material applicability concerns associated with this technique, and is still in a proof of concept phase. A specialized pressure cell has been developed that permits independent radial and axial fluid pressurization of core samples, with axial flow through capability and a temperature rating up to 350°C. This cell will ultimately be used to hydraulically pressurize EGS-representative core samples to conditions of imminent fracture and map the associated internal strain states of the sample. This will hopefully enable a more precise mapping of the rock material failure envelope, facilitate a more refined understanding of the mechanism of hydraulically induced rock fracture, particularly in crystalline rocks, and serve as a platform for validating and improving fracture simulation codes. The elements of the research program and preliminary strain measurement results for sandstone limestone and marble samples will be discussed in this paper.

EXPERIMENTAL SETUP

Geothermal Pressure Cell

The centerpiece of the experimental setup is a custom designed pressure cell capable of holding core samples with a diameter of 38.1 mm and length of up to 152.4 mm (Figure 1). Samples are placed inside a thin Kapton sleeve that fits over the stem pieces at the ends of the vessel through which fluid is injected or discharged. A compression cylinder and seal ring are used to seal the inner and outer surfaces of the sleeve against the stem piece and inner wall of the pressure vessel respectively. This sealing arrangement permits pressurization of the annular space between the sleeve and pressure vessel wall through a tapped through hole on the outside of the vessel. The pressure applied in this space compresses the sleeve producing a confining pressure that acts on the sample's radial face. The seal on the opposite face of the sleeve permits independent pressurization of the axial face of the sample through the stem piece thus producing a triaxial stress state.

Operating specifications for the cell are:

- Max confining/radial pressure – 10,000 psi
- Max axial/flow pressure – 5,000 psi
- Max temperature – 350°C

The titanium cell body and Kapton sleeve are relatively transparent to neutrons to mitigate

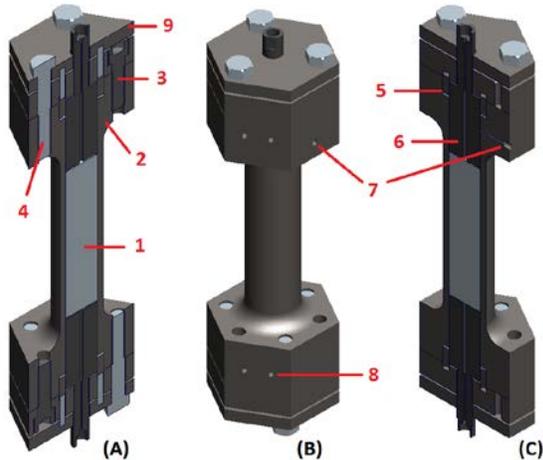


Figure 1 - The geothermal fluid flow cell. An isometric view of the cell is shown in (B), while (A) and (C) are both cross-sectional views. View (A) shows the internal fasteners which bear the load of the internal pressure. Labels: (1) core sample, (2) vessel body, (3) internal retaining fasteners, (4) seal forming fasteners, (5) seal area, (6) fluid flow stem, (7) liner pressure port, (8) mounting holes, and (9) seal forming plate assembly.

scattering effects that may adversely influence radiographic or diffraction measurements. They also have low activation cross-sections enabling the cell to be handled, reloaded, transported and reused immediately after neutron irradiation experiments because induced radioactivity is minimal. A neutron radiograph of a sandstone sample inside the cell is shown below in figure 2. The end stem piece is removed and a plastic tie wrap is instead inserted into the assembly for referenced and contrast. Note that the thickness of the Kapton sleeve is clearly discernible in the image.

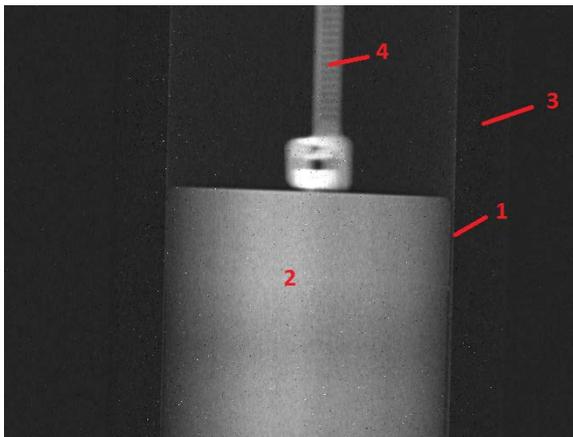


Figure 2 - Neutron radiograph of sandstone sample within pressure cell without end stem piece and zip tie inserted for reference. Labels: (1) Kapton sleeve, (2) Sandstone core, (3) Cell wall, (4) zip tie

Lattice Strain Measurement by Neutron Diffraction

Experiments were performed using the VULCAN instrument at the Spallation Neutron Source. The VULCAN instrument is a neutron diffractometer designed to study deformations, residual stresses and structural features such as phases and textures within materials (An *et al*, 2011). A layout of the instrument is shown below in figure 3 (now equipped with +/- 90° banks). For in-situ deformation measurement, samples can be held and loaded by the VULCAN MTS load frame built into the experimental configuration. Two +/- 90° fixed detectors measure the two orthogonal extensional strain components in the horizontal plane. Alternatively, samples can be characterized independent of the load frame by mounting them on a translation stage for strain scanning at different locations. The pressure cell was mounted with the axis of the cylindrical sample oriented vertically to permit measurement of radial and circumferential strains within the sample.

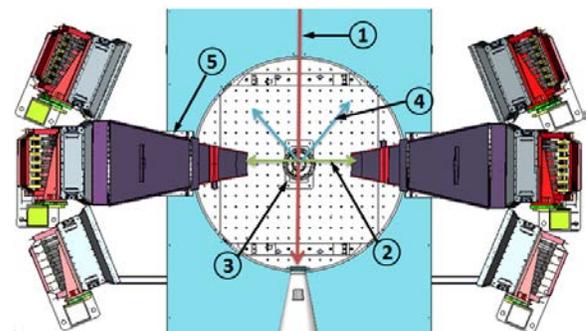
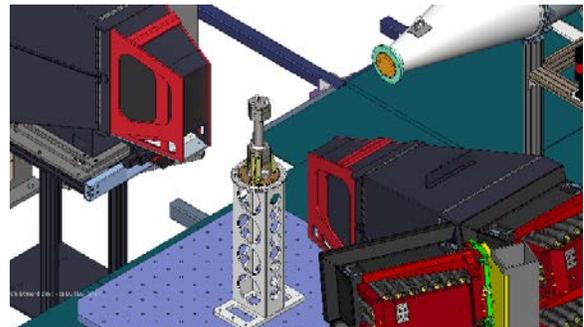


Figure3- VULCAN instrument layout. Labels: (1) incident neutron beam, (2) diffracted beams, (3) sample, (4) measured strain component directions, (5) +/- 90° neutron detectors.

Neutron diffraction methods measure average strains within volumes as opposed to the areas or linear segments typical of more conventional methods. The gauge volume is defined by adjustable incident beam

slits and the receiving collimators in front of the detector. VULCAN has a variety of aperture and collimator settings and can currently achieve sampling volumes down to 6 mm^3 ($1.5 \text{ mm} \times 2 \text{ mm} \times 2 \text{ mm}$). A smaller gauge volume comes at the expense of a longer measurement time needed to accumulate sufficient neutron count statistics that can be correlated to lattice spacing. The gauge volumes used for the experiments described in this paper were 125 mm^3 ($5 \text{ mm} \times 5 \text{ mm} \times 5 \text{ mm}$) and 200 mm^3 ($5 \text{ mm} \times 8 \text{ mm} \times 5 \text{ mm}$) for uniaxial loading and pressure cell strain mapping experiments respectively.

Three different neutron chopper settings (20 Hz, 30 Hz and 60 Hz) are available to define incident beam wavelengths ($\sim 4.32 \text{ \AA}$, $\sim 2.88 \text{ \AA}$ and $\sim 1.44 \text{ \AA}$). Chopper settings of 20 Hz and 30 Hz were used depending on the specimen material. Measurement time for each evaluated point within the sample varied between 5 and 30 minutes depending on the statistics required and neutron scattering characteristics of the materials tested.

TESTS PERFORMED

Two types of tests were performed, each with three different materials. The first set of tests were uniaxial compression tests performed utilizing the VULCAN MTS load frame. Uniaxial load tests are typically required to relate diffraction measured lattice strains to macroscopic stresses using so-called diffraction elastic constants (Hutchings *et al*, 2005). They are also performed to evaluate whether or not crystalline inhomogeneity precludes relating lattice strains to macroscopic properties.

The samples tested were Scioto Sandstone, Carthage Marble and Sierra White Granite. The specimens were cylindrical in shape with diameters of 8.85 mm or 10 mm and lengths of 30 mm. Samples were incrementally loaded up to a force value corresponding to approximately 75% of unconfined compressive strength. The lattice strain measurement gage volume was located roughly in the center of the sample.

The second set of tests was performed to confirm that strains could be measured through the titanium pressure cell. 38.1 mm diameter by 152.4 mm long core samples of Scioto Sandstone, Indiana Limestone and Sierra White Granite were inserted and strains were measured for confining pressure values of 0 psi, 1500 psi and 2500 psi. Two rows of nine measurement points were evaluated across the diameter of each for a total of eighteen points per pressure level (see Figure 4.). The two rows were

slightly above and below the centroid of the sample and were selected to compare the consistency of strain measurements as the load condition was expected to produce an identical strain state across each row.



Figure 4- Center of specimen gage volume measurement points.

RESULTS

X-ray powder diffraction (XRD) measurements were made for each sample type in order to identify the crystallographic phases present in the measured materials. XRD patterns for the granite, sandstone and marble specimens used in the experiments are shown below in Figure 5. Exact crystal structures were defined for the sandstone and marble samples, but not for the granite sample at the time of this publication. Test results for granite will therefore not be included in the discussion because strain calculations for the data set have yet to be undertaken.

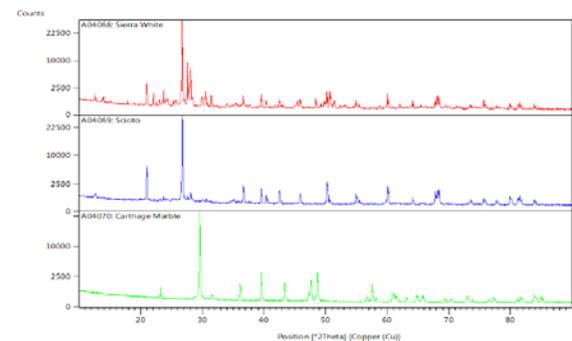


Figure 5- X-ray diffraction pattern for Sierra White Granite (top), Scioto Sandstone (middle), and Carthage Marble (bottom).

The strains associated with lattice deformations are calculated in the standard manner by evaluating peak shifts associated with particular crystallographic planes using the relation

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0}$$

where d_{hkl} is the measured lattice plane spacing at a load measurement point and d_{hkl}^0 is the lattice plane spacing with no external loading. Lattice values at the 0 psi load state were taken for the d_{hkl}^0 of each measured location for the strain mapping measurement in the pressure cell experiments.

Relating these microscopic strains to more conventionally used macroscopic stresses requires the definition of diffraction-specific elastic constants. For relatively isotropic, texture free materials (materials without preferred grain orientations), macroscopic stresses can be related to lattice plane family strains using the constitutive relation:

$$\sigma_{ij} = \frac{E_{hkl}}{(1 + \nu_{hkl})} \left[\varepsilon_{ij}^{hkl} + \frac{\nu_{hkl}}{(1 - 2\nu_{hkl})} (\varepsilon_{11}^{hkl} + \varepsilon_{22}^{hkl} + \varepsilon_{33}^{hkl}) \right]$$

The diffraction-specific elastic constants are determined by a set of uniaxial load calibration experiments. Analysis of the data for this purpose was not completed by the time of this publication and will therefore not be presented. It is also worth mentioning that in the case of the Indiana Limestone, uniaxial compression tests were not undertaken because suitable samples were not available. The primary focus of this work is to determine whether or not adequate peaks could be identified for strain measurement within the geological materials of interest in the desired testing configuration and to confirm that the measured strain variation with loading appears to be in agreement with expected physical behavior.

Uniaxial compression test results

Macroscopic stress versus lattice strain measurements for Scioto Sandstone and Carthage Marble specimens are shown in Figures 6 and 7 respectively. Selected sandstone peak measurements are associated with quartz crystals while those for the marble are associated with calcite crystals. Macroscopic stress was calculated as the applied load divided by the total cross-sectional area of the sample perpendicular to the load direction. Bank 1 and bank 2 refer to the loading and perpendicular to loading directions respectively.

The trends in successive load measurements are generally linear indicating elastic behavior of the rock and the stress versus strain slope differences

between the lattice plane measurements are indicative of expected crystalline anisotropy. Estimated diffraction elastic constants for the sandstone quartz lattice planes (65 GPa – 90 GPa) are reasonably close to others reported from lattice strain measurements (Frischbutter *et al.*, 2000).

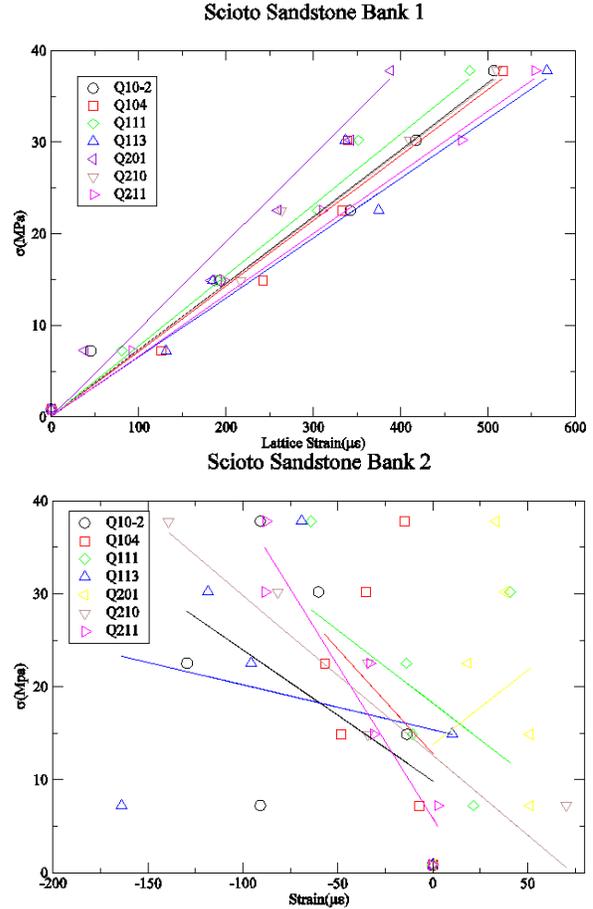


Figure 6 – Macroscopic stress vs microscopic strain in direction of loading (Bank 1) and perpendicular to loading (Bank 2) for Scioto Sandstone.

It is worth mentioning that lattice strain measurements in the direction orthogonal to loading do not exhibit a well-defined trend for the sandstone. This is expected because these strains are associated with Poisson ratio effects, are relatively small and are mostly below the 100 microstrain resolution of the VULCAN instrument. They are presented to illustrate the limitations of diffraction based strain measurement techniques. It is expected that when the technique is applied to actual fracture initiation experiments this will not be an issue because the measured strains at incipient fracture conditions will be considerably greater.

Pressure cell test

Strain measurements across the diameters of the Scioto Sandstone and Indiana Limestone samples as a function of confining pressure are shown below in Figures 8 and 9. The 1,500 psi data points for the Indiana Limestone were lost due to a failure of the data acquisition system. The trends in the strain are in good agreement with expected behavior. Bank 1 and bank 2 strains in this configuration represent radial

are on average 85 GPa and 0.31 and can be used to provide an order of magnitude estimate of the measured stress level (Zhao *et al*, 2009). This calculation is only done for illustrative purposes because single crystal and poly-crystal diffraction elastic constants usually differ due to a variety of different intergranular effects. It would be expected that actual measured diffraction elastic constants for the poly-crystal would be less than those of the single crystal for the reason stated above and as reported by others (Frischbutter *et al*, 2000).

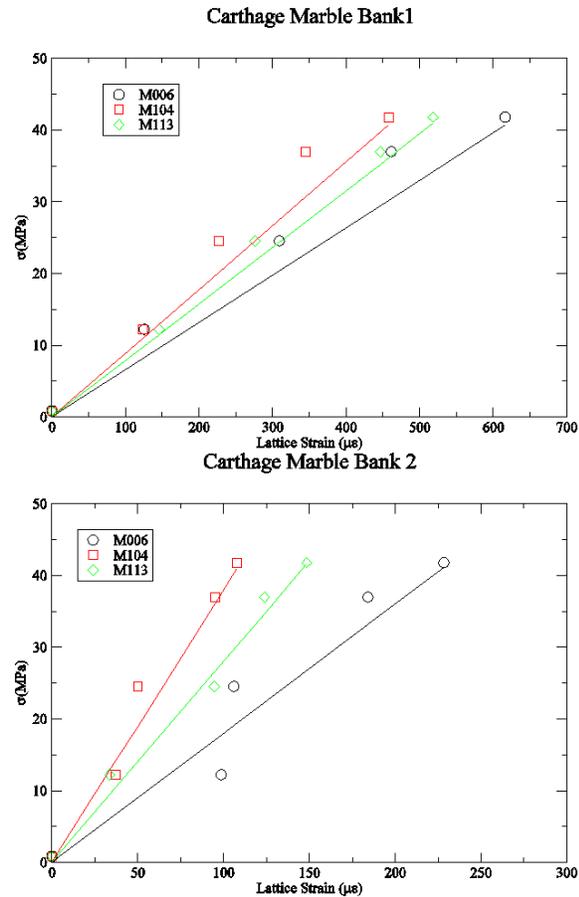


Figure 7 – Macroscopic stress vs microscopic strain in direction of loading (Bank 1) and perpendicular to loading (Bank 2) for Carthage Marble.

and circumferential strains using radial coordinates to represent the axisymmetric loading of the sample.

The circumferential and radial stresses are expected to be equal to the pressure for this arrangement based on the geometry and load symmetry. This expected stress level can be compared to the calculated stress level using a constitutive equation, as defined in the preceding section, provided that the diffraction based lattice constants are known. Uniaxial load diffraction measurement tests were not performed for the limestone used in the confining pressure tests. However, literature reported values for Young's modulus and Poisson's ratio of single-crystal calcite

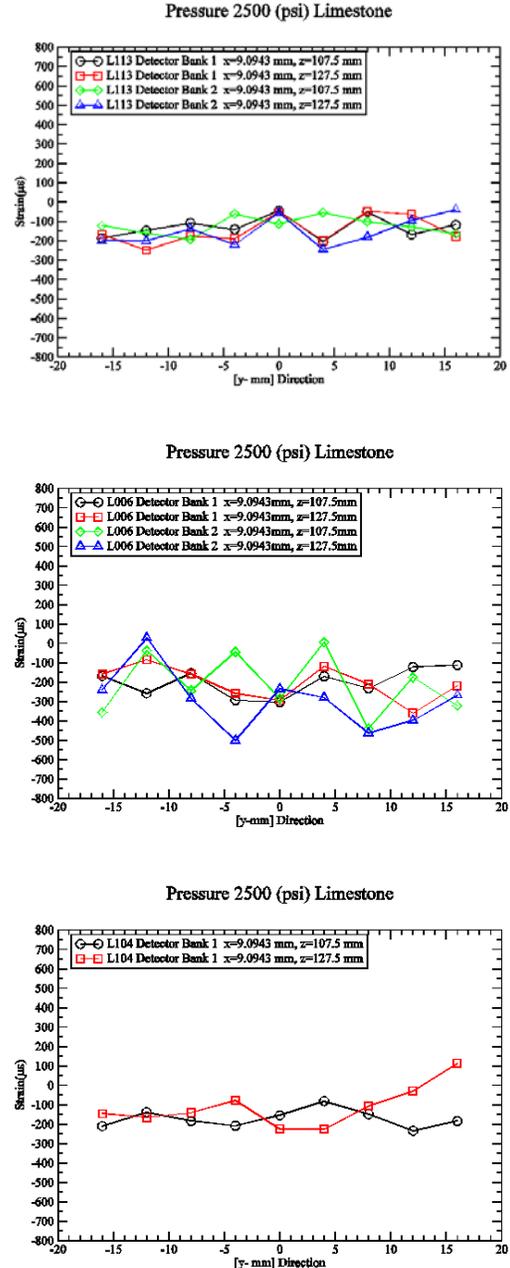


Figure 8 – Indiana Limestone lattice strain measurements across the diameter of the core at 2500 psi confining pressure.

The calculated strains for single crystal calcite at the 2500 psi load level are 140 microstrain while the observed strains are in the range of 100 – 150 microstrain for the points with higher accuracy as will be explained below. These are reasonably close and indicative of the expected trend. It is reiterated that at these relatively low load levels strain levels are at the resolution limit of the instrument. It is also emphasized that the counting statistics for many of the points are poor as this was a scoping exercise. The data is evaluated primarily in qualitative terms in order to assess the potential utility of neutron based strain measurements for this experimental configuration.

Expected strain levels for the sandstone based on the average measured diffraction elastic constant (70 GPa) and assumed Poisson's ratio (0.15) are approximately 125 microstrain and 210 microstrain at the 1500 psi and 2500 psi load levels respectively. This trend is somewhat reflected in the data with the disclaimers mentioned above in mind.

There are a number of factors that contribute to the scatter in the data. As already mentioned, the strain levels induced in the pressure experiments are approaching the resolution limits of the instrument. Counting statistics are another critical contributor to measurement errors. It is clear that future experiments involving the pressure cell and large samples will require longer counting periods than those used in these experiments to obtain more accurate results due to effects associated with longer penetration paths as compared to the tests performed with the smaller uniaxially loaded samples. This is also pertinent to the point to point variability of the data seen for some of the peaks through the pressure cell. Measurement points in the positive direction measured by bank 1, for example, involved longer neutron path lengths through the sample (because of the sample position relative to the beam and detector banks). The signal is more attenuated with a longer path length through materials and the points to the right in the bank 1 plots clearly exhibit more scatter because of the longer path length taken. It is also noted that we have no explanation for the erratic 135 mm position, 2500 psi, bank 2 data for the sandstone. We assume there was a detector issue that produced the odd behavior.

As a general conclusion, the results are very encouraging because it appears that distinct lattice peaks can be identified and used for strain measurement in representative materials and there is strong evidence that the technique can be applied even within a pressure cell permitting strain mapping

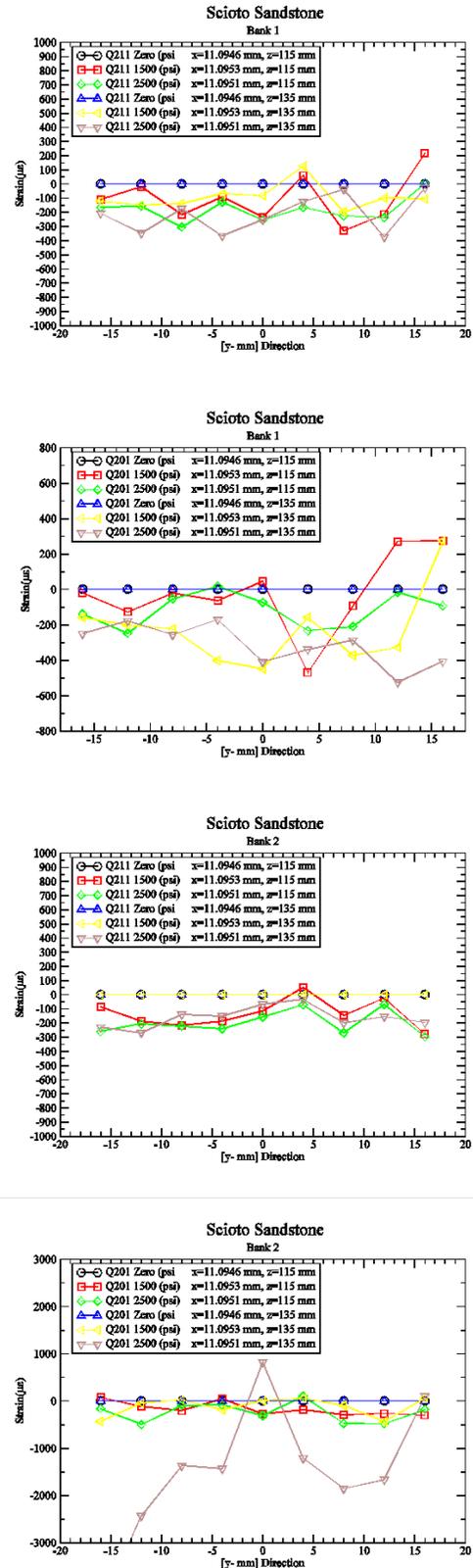


Figure 9 – Scioto Sandstone lattice strain measurements across the diameter of the core at 0 psi and 1500 psi confining pressure.

measurements to be taken for triaxial stress conditions. It is believed that many of the resolution issues can be improved with longer measurement periods. Additionally, there are data processing techniques that combine multiple lattice measurements to evaluate volumetric crystal deformations that can improve accuracy. They have not yet been undertaken in this work but are planned.

CONCLUSION

Lattice strain measurements were made for sandstone, limestone and marble geological core samples subjected to two different load configurations: conventional uniaxial load testing performed on unconfined samples; and uniform confining pressure applied within a custom-designed titanium pressure vessel. While neutron diffraction based strain measurements have been previously reported for geological materials, demonstration of the potential for utilizing lattice deformations as “microscopic strain gauges” to quantify and map stresses inside geological materials within a pressure vessel capable of producing a triaxial stress state is believed to be unique. If further refined and calibrated, this capability represents a powerful tool for better understanding a variety of deformation related behaviors in rock mechanics application.

The current application of interest for this research group is improved understanding of hydraulically induced fracture. Future experiments will generate incipient fracture conditions and use this technique to characterize the regional strain state near the fracture initiation site in order to validate and guide the development of hydraulic fracture simulation capabilities. However, the penetrative and interrogative capabilities of neutron diffraction based strain measurement along with its ability to provide indication of stresses associated with microstructural phenomenon such as grain interactions and texture may provide the opportunity to study more diverse and fundamental rock mechanics issues such as bedding, plastic deformation and failure of geological materials.

Use of the technique for any applications associated with geological materials will require further refinement and improvement of sample characterization, strain calibration procedures and data reduction in order to improve the accuracy and interpretation of measurements. Nonetheless, the obtained data does represent a proof-of-principle that otherwise inaccessible internal material regions can be probed, even through surrounding structural materials.

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