

## High-Temperature Laboratory Experiments to Support EGS Stimulations: Permeability Response in Fractured Crystalline Samples

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### ABSTRACT

Enhanced or engineered geothermal systems (EGS) have the potential to provide increased renewable energy resources through created or improved fluid permeability within hot reservoir rocks. The EGS Collab project is focused on intermediate-scale fracture modeling, creation, and monitoring, in support of enhanced permeability in crystalline rocks. Here we propose a series of laboratory-scale experiments to investigate fracture permeability response to chemical reactions at target geothermal temperatures and stresses. The planned experiments utilize samples from crystalline basement at the two proposed FORGE sites (Fallon, Nevada; and Milford, Utah) as well as phyllite from the rock formation hosting the eight boreholes drilled as part of the EGS Collab project. Several experimental samples have been produced by subsampling the original core in plugs of desirable orientations, subjecting these plugs to fracturing techniques, and subcoring. Early experiments will be conducted at variable temperature (~100, 200°C) and fixed confining stress, while later-stage experiments will investigate the effect of varying confining pressure conditions (10-20 MPa). Each experiment utilizes pre- and post-reaction X-ray computed microtomography imaging, as well as changes in fluid pressure and solution chemistry, to evaluate changes to fracture flowpath resulting from high-temperature reaction of mildly saline fluids with common mineral phases (e.g., feldspars, quartz, biotite, muscovite, calcite). The experimental datasets generated through this series of smaller-scale laboratory experiments will be useful for validating reactive transport models for better prediction of fracture conductivity, and will complement the larger datasets being generated in the EGS Collab project in support of the U.S. DOE's Frontier Observatory for Research in Geothermal Energy (FORGE) mission.

### 1. INTRODUCTION

To better understand the mechanisms that impact rock fracturing and fracture conductivity at depth, the U.S. Department of Energy's Geothermal Technologies Office established the EGS Collab Project (<https://www.energy.gov/eere/geothermal/egs-collab>, last accessed January 2018). This multi-institutional project brings together geothermal reservoir modelers and experimentalists to predict, stimulate, characterize, and monitor intermediate-scale (i.e., 10's of meters) fractures at reservoir stress conditions represented at the 4850' level within the Sanford Underground Research Facility (SURF). The EGS Collab Team of authors will present several papers at this year's conference and the interested reader is referred to the full 2018 proceedings for further project details.

Within geothermal reservoirs targeted for permeability enhancement, fractures (naturally occurring, reactivated, or newly induced) are expected to carry the bulk of the heat-transmission fluids. Given the continuous cycling of fluids through the subsurface in geothermal power operations, and the prevalence of highly reactive minerals such as calcite and moderately reactive phases such as micas and other sheet silicates in natural fracture zones (e.g., Lutz et al., Vidal et al., 2017), stimulated zones should be expected to be susceptible to fluid-rock interaction. While geomechanical processes certainly dominate the response of fluid flow networks at the immediate timescale, chemical dissolution and re-precipitation reactions can also affect flow pathway distribution and evolving permeability over variable length- and time-scales (e.g., U.S. DOE, 2015).

In this paper, we focus on proposed experiments to examine the impact of fluid-rock reaction, under representative geothermal temperature and stress conditions, on fracture surfaces, apertures, and overall conductivity at the smaller laboratory scale (i.e., 10's of millimeters). This work is intended to be complementary to the intermediate-scale experiments conducted at the SURF site for the EGS Collab Project (Kneafsey et al., 2018). Previous research, conducted on isolated fractured samples from a variety of rock types, has demonstrated both decreasing and increasing conductivity responses during periods of fluid-rock reaction generated by flowing chemically or thermally dis-equilibrated fluids through individual samples. A range of responses to chemical perturbations have been demonstrated, ranging from increased flow due to widening of average fracture apertures, the development of fast flowpaths or channels associated with fractures, enhanced wallrock porosity and diffusive transport into the matrix, as well as permeability decreases due to the dissolution of fracture-propping asperities (e.g., Smith et al., 2013; Ellis et al., 2013; Fauro et al., 2006; Yasuhara et al., 2006; Yasuhara and Elsworth, 2004; Polak et al., 2004; Detwiler et al., 2003; Polak et al., 2003; Durham et al., 2001; Morrow et al., 2001; Moore et al., 1994). It is likely that the overall impact to reservoir sustainability will depend upon a combination of geochemical and geomechanical factors; thus we propose to examine the impact of both temperature and confining stress on permeability response in our smaller-scale experiments.

Here we describe the preliminary work done to support a series of high-temperature, reactive-flow experiments designed to more systematically examine the chemical response of fractured crystalline rocks to variations in temperature and stress. The experiments will provide an indication of the magnitude and impact of high-temperature (i.e.,  $\geq 100^\circ\text{C}$ ) reactions on fluid transport through fracture pathways. The experimental datasets generated through this matrix of laboratory-scale work will be useful for validating reactive transport and/or mechanical models for better prediction of fracture conductivity relevant to the U.S. DOE's Frontier Observatory for Research in Geothermal Energy (FORGE) mission.

## 2. SAMPLE PREPARATION & CHARACTERIZATION

Our experimental plan entails mineralogical characterization and preparation of fractured core samples from relevant reservoirs, including the two proposed FORGE sites (granitic samples from the proposed Fallon, Nevada, FORGE site, <https://www.fallonforge.org>; and granite/granodiorite from the Milford, Utah site, <http://www.forgeutah.com/>) as well as Poorman Formation phyllite from the previous KISMET project (Oldenburg et al., 2016) and host formation of the current EGS Collab experiments at the SURF site (Figure 1).



**Figure 1: Map of western United States, showing locations of experimental samples from the two proposed FORGE sites as well as the site of previous KISMET and current EGS Collab projects. Map from [www.google.com](http://www.google.com).**

Two methods of fractured sample preparation were tested with the phyllite samples: subcoring around existing (non-mechanical) fractures; and inducing fractures in oriented plugs followed by additional subcoring. Sampling around existing fractures resulted in single-fracture samples with larger apertures and abundant gaps along sample cylinder walls, caused by missing rock chips. Fractures induced using a hydraulic press and guard shields to prevent crushing resulted in smaller-aperture fractures with multiple splays in similar orientations. Due to the scarcity of pre-existing fractures and limited core availability from the two proposed FORGE sites, the induced fracture method was selected, resulting in a suite of 15-mm diameter sample cores with lengths ranging from 32-38 mm, possessing fractures bounded by fresh mineral surfaces.

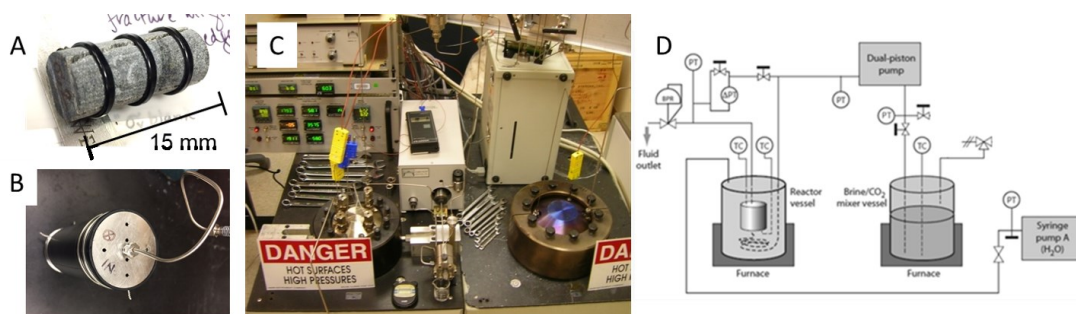
While the Poorman phyllite at SURF lacks the high temperature gradients needed for geothermal power production, it is important to include samples from this formation in order to contribute to the cohesive dataset obtained as part of the larger, ongoing characterization

efforts taking place in support of the field experiments for the EGS Collab Project. Optical microscopy, scanning electron microscopy (SEM), and X-ray diffraction (XRD) techniques have been utilized to initially characterize samples of the Poorman phyllite taken from KISMET core k-002, depths 118.4-120 feet. These samples are primarily composed of plagioclase and quartz with large volumes of calcite (no dolomite identified), biotite, muscovite, and an abundant X-ray bright sulfide mineral (pyrrhotite or pyrite); this mineralogy is in keeping with the earlier characterization efforts of Hart et al. (2014) and Roggenthen (2017). Prominent foliation is evident in both optical view and higher-resolution SEM imaging, as well as in the X-ray computed tomography (XRCT) datasets discussed below, and induced fractures (unsurprisingly) tended to follow the direction of foliation. Fracture pathways were observed to travel both between and across large clusters of intergrown silicate mineral grains.

Similar characterization is underway for samples from the remaining two sites. Previous work by Simmons et al. (2016) as well as communication with C. Jones (EGI: Univ. of Utah) indicate that the granodiorite samples from well 58-32 (depths ~6800-6804 feet) are comprised of plagioclase, quartz, potassium feldspar, and biotite, with accessory hornblende, illite, and chlorite. Samples from the Fallon, Nevada site are described as primarily felsic in composition, encompassing metavolcanics, metaquartzites, and granitic intrusives of Cretaceous to Triassic age (e.g., Hinz et al., 2016). Specific sample selection from these two sites was coordinated with project members to ensure use of more representative reservoir material from proposed high-temperature regions. Higher-resolution imaging and microprobe analysis of identified mineral grains will be conducted to verify correlated mineralogy observed in XRCT gray-scale images and to assess contributions to changes in solution chemistry (see section 3).

### 3. REACTIVE CORE-FLOOD EXPERIMENTS

Within the limitations of our experimental infrastructure, core-flooding experiments will be conducted to span as wide a range as possible of representative geothermal temperatures and stress (in this case, limiting to confining pressure) conditions. The first sets of experiments will consist of reaction at two representative temperatures with a fixed confining pressure condition, and the second round of experiments will vary the confining pressure at a fixed temperature. The final round of experiments will duplicate one set of temperature/pressure conditions with sample cores from the other two sites. Given the limitation of our experimental materials, we plan to conduct core-flooding experiments at conditions of 100 and 200 °C, and 10 to 20 MPa of confining pressure. In all cases, fluid composition (low salinity NaCl “brine”), flowrate (anticipated value of 0.05 mL min<sup>-1</sup>) and outlet fluid pressure will be kept constant.



**Figure 2: Core-flooding samples and experimental set-up. Representative fractured phyllite sample (A), shown with polymer jacketing and fluid deliver endcaps (B), for use in core-flooding apparatus shown in (C), component diagram (D).**

The experimental core-flooding apparatus shown in Figures 2c,d consists of a reaction vessel, pressurized by water driven by a dedicated pump, containing the vertically-oriented isolated fracture sample; a dual-cylinder pump to deliver continuous pulse-free fluid to the sample; a back-pressure regulator to control outlet fluid pressure; multiple absolute and one interchangeable differential pressure transducers; an optional pair of pressure vessels to pre-heat or otherwise isolate flowing “brine;” and a data-logging system to record system and sensor outputs at selected time frequencies. All wetted surfaces are composed of chemically inert titanium or C-276 alloy material. Core-flooding experiments, based on previous work (e.g., Smith et al., 2013) and preliminary kinetic modeling, are planned to span 4-6 weeks to ensure sufficient reaction at the lower (100 °C) temperature condition.

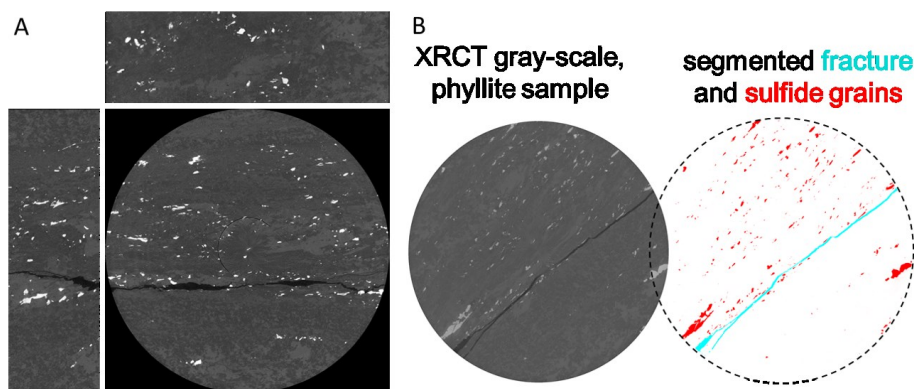
Samples are triply jacketed to ensure not bypass fluid flow along the sample walls and to ensure a snug fit between two fritted endcaps that deliver fluid to each sample cylinder face. The system is first pressurized to the desired confining pressure at room temperature, saturated, and then pore/confining pressures varied to “seat” the fractured sample. A short tracer test is carried out and initial fracture permeability is measured while still at room temperature. Sample and fluid are then brought to experimental temperature as quickly as possible at the constant experimental flowrate. At the end of each experiment, samples will be cooled quickly to room temperature, purged of remaining fluid with a nitrogen gas stream, and removed for post-reaction XRCT imaging.

Changes to bulk permeability are assessed through variation in differential pressure along the length of the core. A rough measure of sample compression can be gained by monitoring changes to the delivered volume of compression fluid in the reactor vessel, although smaller sample/vessel volume ratios decrease the utility of this measure. Solution chemistry is monitored by collecting fluid samples at the back-pressure regulator output. Aliquots are retained for analysis of major and trace elements and solution pH, and *in situ* solution pH at temperature is modeled along with mineral-specific saturation indices using the EQ3/6 geochemical speciation code (Wolery,

1992). It is anticipated that the contribution of individual elements can be initially assessed through assignment of marker major elements (e.g., dissolved bicarbonate for calcite, sodium for plagioclase) and the use of relevant high-temperature kinetic mineral reaction rate equations (e.g., Smith and Carroll, 2016) in future reactive transport simulations.

#### 4. SAMPLE IMAGING

Individual samples were jacketed using heat-shrinkable synthetic polymer material before X-ray computed tomography (XRCT) imaging at the 8.3.2 beamline at the Advanced Light Source (Lawrence Berkeley National Laboratory). No effort was made to offset the induced fractures. White light was used to collect 1025 angles per tile, and images were reconstructed using TomoPy software framework (Gursoy et al., 2014), resulting in a resolution of  $\sim 7.1$  microns per pixel. Representative XRCT images of fractured phyllite are shown in Figure 3. Prior to reaction, 3-D reconstructions are used to estimate fracture surface area. In the Poorman phyllite samples already imaged, induced fracture apertures range from 10's to 100's of microns, with a primary fracture roughly parallel to the long axis of the sample, and up to five splay fractures that terminate in wallrock rather than extending the full length.



**Figure 3: Representative XRCT images showing texture and fracture geometry from phyllite sample with induced fracture. 2-D gray-scale core image with orthogonal sections (A); core image with segmented fracture region and sulfide grains potentially useful as tracking particles (B). Note that in XRCT images, low-density phases (i.e., void space) is represented as a low gray-scale value (i.e., black, 0).**

After core-flooding reaction at variable temperature and stress conditions, each sample will be re-imaged under the same conditions and the two image datasets will be analyzed by difference to quantify changes to fracture aperture, matrix or wall-rock porosity, and to quantify extent of chemical reaction correlated to composition according to the methods outlined by Mason et al. (2014). While the determination of overall volume change is straightforward, the determination of the responsible process (i.e., chemical dissolution, secondary precipitation, physical grain removal and movement, wallrock deformation) requires additional segmentation and analysis of each full 3-D image data. The XRCT data also has the potential to provide an assessment of overall sample deformation resulting from reaction-induced changes in mineral volumes, as described below.

#### 5. PROPOSED APPLICATION OF DIGITAL VOLUME CORRELATION TECHNIQUE

Particle image velocimetry (PIV), or the broader term digital volume correlation (DVC) refer to well-established techniques used to assess motion by tracking bulk movement of tracer particles or points (Adrian, 2005). Here, assuming a suitable concentration of X-bright mineral “tracers” are present in each rock sample, we propose to employ the same technique to the pre- and post-reaction XRCT datasets to non-destructively assess relative displacement in one fracture “half” relative to the other. It has been previously demonstrated that volume correlation analysis can accurately differentiate effects of chemical dissolution or imaging artifacts and noise from deformation, as well as provide a robust measure of three-dimensional local mechanical deformation (Walsh et al., 2013).

Our proposed application was derived from traditional PIV techniques with accommodation for 3-D rather than 2-D application and using imaged sulfide particles within XRCT datasets as tracker particles: 1) pre- and post-reaction tomography images are aligned and segmented into corresponding 3-D subregions; 2) cross-correlation between subregions is calculated to obtain a coarse displacement shift value; 3) steps 1 and 2 are repeated for remaining subvolumes to produce a full displacement field; 4) steps 1-3 are repeated at higher resolutions to obtain finer measures of displacement. A more detailed description is provided in Walsh et al. (2016), with particular emphasis to their Figure 3. The application of this technique is highly dependent on an even distribution of suitable X-ray bright mineral grains (see bright/white grains in Figure 3); the mineralogy of the phyllite samples is amenable to this application and pre-reaction imaging of the Milford, Utah, and Fallon, Nevada samples should determine these samples’ suitability for the technique.

While we expect to find a net movement of fracture walls toward each other as a result of the confining pressures applied during experimental reaction, we are most interested in localized deformation that may occur at fracture asperities. In addition, this technique should be able to identify any mechanical transport of intact mineral grains, a phenomenon sometimes referred to as “mineral clogging” that has been invoked in permeability reductions associated with extensive calcite dissolution at lower temperature (e.g., Luquot et al., 2014). Correlation of regions of enhanced displacement with mineral reaction could provide useful datasets for investigating effects of coupled geochemical-geomechanical processes such as these on fracture permeability.

## 5. CONCLUSION

Fractured subcore preparation and non-destructive imaging has been completed for Poorman phyllite samples from the EGS Collab site. Subcore preparation and characterization is still ongoing for samples from the two proposed FORGE sites, with XRCT imaging to proceed immediately. Experiments will commence after imaging of the suite of fractured subcores has been completed.

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