Technical Report – SCEC award 21149 - Clay chemistry at earthquake timescales

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Abstract

Clay structure can be radically altered by heating during earthquakes, which could have a significant impact on fault strength, as well as a potential for recognizing structures that have experienced earthquake slip in the rock record. In this project, we aimed to quantify clay reaction kinetics when heated over a variety times and temperatures. Our initial results show that clays react at earthquake timescales and significant changes occur above temperatures of about 300 °C.

Technical Report

Project Goals

The stability and strength of faults in the brittle crust are determined by a variety of physical and chemical factors operating over widely varying spatial and temporal scales. Among these, the production and/or localization of frictionally weak phases, such as phyllosilicates, within fault gouge are particularly important. Previous research has examined the origin of these phases (hereafter, "clays") during cataclasis and fluid-rock interaction across a range of depths in the crust. Friction experiments have documented the profound effect clay exerts on frictional strength and stability of faults (Byerlee 1978; Ikari et al., 2009). Collectively these studies outline that variations in the composition and mechanical properties of fault gouge occur over geologic time scales, likely encompassing a preponderance of discrete slip events that produce the mechanically weak materials observed in active fault zones today. However, earthquakes cause a significant change of the in-situ conditions over very short timescales, which may also promote mineral alteration. Specifically, temperature rise from frictional heating commonly generates temperatures in excess of 500 °C (Coffey et al., 2019). Thus, it seems reasonable that at least some component of mineral alteration and variations in gouge strength must be attributable to geochemical processes occurring coseismically. Although a variety of experimental work has examined the physical effects of shear heating on dynamic strength of gouge at seismic slip rates (Di Toro et al., 2011), comparatively few studies have conducted detailed analysis of changes in gouge mineralogy associated with such pronounced (but transient) variations in temperature.

Beyond dictating fault strength and stability, fault zone chemistry also leaves clues to the spatial distribution of earthquakes within a fault zone. For example, biomarker thermal maturity, carbonate dissociation, and short-lived isotope diffusion all provide information about where and when earthquakes happen in faults zones (Savage et al., 2014; Ault et al., 2015; Rowe and Griffith, 2015). As we will discuss in more detail below, clay mineral alteration during rapid heating is a possible paleoseismic indicator in the rock record (Schleicher et al., 2015) but the potential for structural recrystallization/rehydration and erasure of that signature has not been fully explored.

For this research we conducted a series of controlled heating experiments utilizing in situ X-ray diffraction (XRD) analyses to address the following questions: 1) How does the crystal structure of clay phases commonly observed in fault gouge (e.g. smectite, illite, chlorite, kaolinite) change during shear heating in the crust?; and 2) How do the rates of crystal-structure change (mineral kinetics) vary as a function of temperature rise? Ultimately, these results will

lay the foundation for later exploration of how shear-heating induced changes in clay-mineral structure affect fault strength over time scales of seconds to days, and whether the signatures produced during experimental heating may be used to infer ancient, coseismic slip on faults.

Project Outcomes

We started our experiments with a Na-Montmorillonite standard from the Clay Mineral Society, SWy-1. We isolated the smallest grain sizes via centrifugation, targeting the same sub-2-μm fraction used in the Clay Mineral Society analysis (Chipera and Bish, 2001)

Technique Development

Examination of clay structures with x-ray diffractometry is facilitated by the use of oriented-aggregate mounts. This type of mount aligns the basal planes of the clay mineral particles along the surface of the mount, defining a preferred orientation with respect to the x-ray beam. There are several different methods to accomplish this. The Millipore filtration method is the best technique to use for quantification purposes (Moore and Reynolds, 1997), as it reduces the potential for grain-size stratification in the sample. This technique outlined in detail in the USGS open file report on clay analysis (Poppe et al., 2001).

This technique was used as a starting point for mounting the samples, but changes were required to accommodate the significant swelling that smectites experience when mixed with water. Our new procedure mixes 9 mL of 99.9% isopropyl alcohol with 2 mL of a 40 mg/mL concentration clay solution, which prevents the filter from clogging and detrimentally decreasing the flow rate. Crucially, it enables clean transfer from the filter to the sample holder. To accommodate both the high temperatures used during the experiments as well as the sample geometry of the heating stage, custom samples holders were designed and fabricated. Our resulting holders are 23-mm-diameter discs cut from 1-mm-thick Inconel sheet metal. The other deviation from the USGS preparation technique is the introduction of nano-corundum into the sample solution to serve as a standard against which changes to the smectite can be analyzed. The data from several sample mounts were compared with the oriented mount data from the Clay Mineral Society report (Chipera and Bish, 2001) to ensure that our methodology is sound.

All XRD analysis was performed in a Rigaku Smartlab Powder Diffractometer in a parallel beam geometry. All scans were performed with a step size of 0.1° at a rate of 2° per minute. They all used a fixed incident angle of 4°, to eliminate the effects of decreased beam footprint and increased penetration depth associated with a more typical rotating incident beam. The heating experiments were performed in an Anton Paar DHS 1100 with the TCU 200 control unit. The heating stage sat on the diffractometer's goniometer and was covered by a graphite dome. All experiments were run with a vacuum pulled inside the dome. The sample mounts were attached firmly to the heating stage with Inconel clips, to ensure good heat conduction to the sample. Multiple clip-sample geometries were tested beneath the dome to maximize the signal intensity.

Results

To date, we have run a total of four heating experiments, which are show clear crystallographic changes in smectite over short heating durations at high temperature. We detail these experiments below.

The first heating experiment was performed on a sample with 15% corundum by weight (experiment 15A). The heating rate was set to 50 °C/minute, and scans were conducted while the temperature was held at 25, 100, 300, 500, and 700 °C (Figure 1). The maximum 2θ value is 9 degrees, corresponding to a 1-nm d-spacing. The largest change is seen at 300 °C, and it is observed in the other visible smectite peaks (003 and 005) as well.

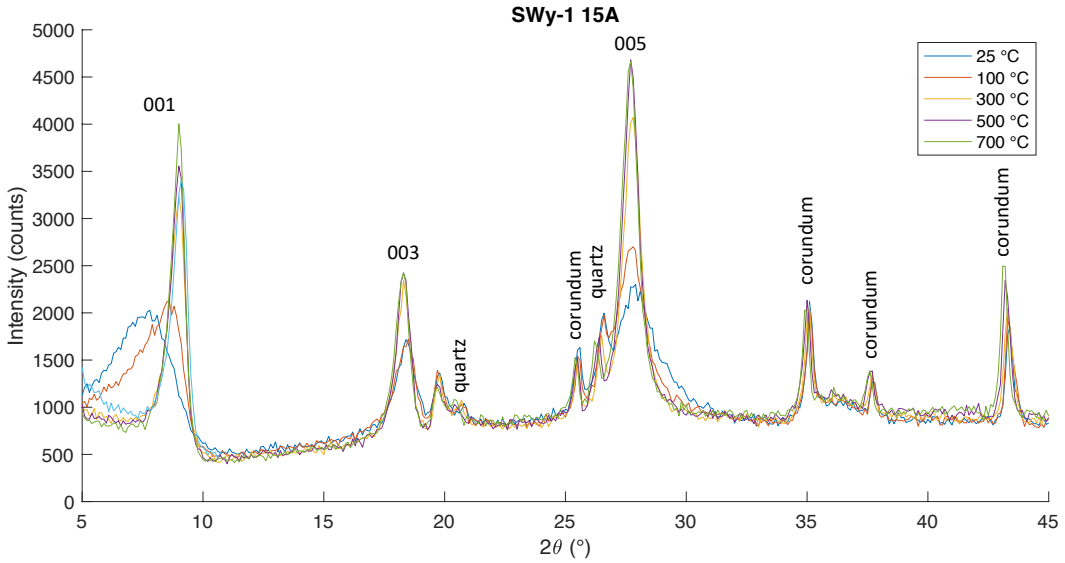

Figure 1: Experiment 15A. The montmorillonite 001 (basal) peak narrows and increases in both 2θ and intensity.

The second heating experiment was performed on a sample with 12% corundum by weight (experiment 12A). The heating rate was set to 100 °C/minute, and scans were conducted while the temperature was held at 25, 100, 300, 500, 700, and 900 °C (Figure 2).

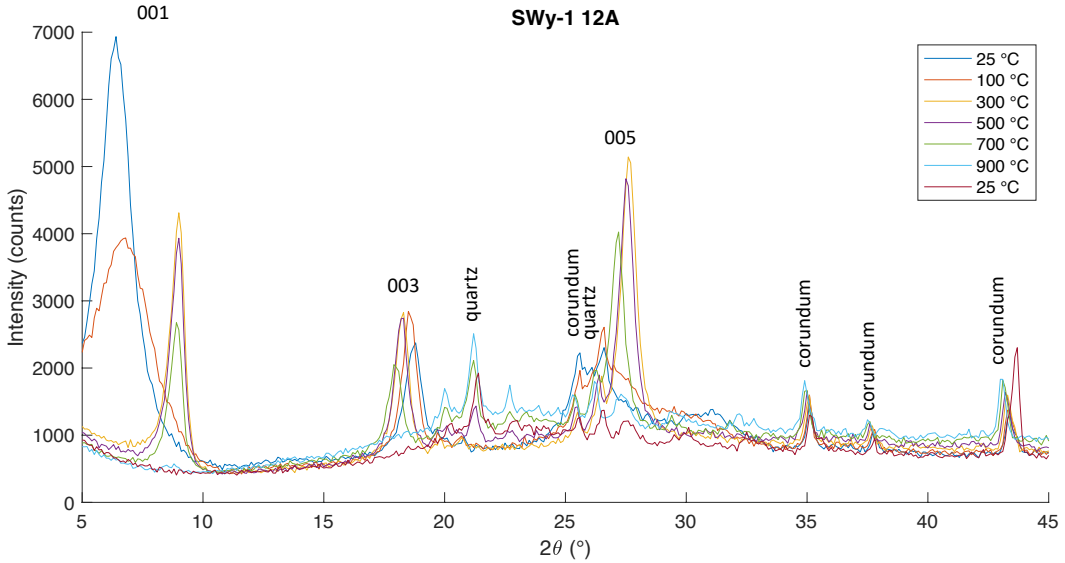

Figure 2: Experiment 12A.

The initial conditions of this sample are clearly different than experiment 15A; the 001 peak of unheated sample has a lower 2θ, a narrower peak, and a much higher intensity than the previous experiment. As it is heated, this peak trends downward in intensity, with only a slight increase between 100 and 300 °C. However, like the previous experiment, it narrows at higher temperatures, and it arrives at the same upper location of 9 degrees. It also similarly experiences a striking transition at 300 °C. At 900 °C the peak intensity decreases dramatically and is barely visible; it can no longer be seen by the time it has cooled.

The third heating experiment was performed on another sample with 12% corundum by weight (experiment 12B). The heating rate was again set to 100 °C/minute, and scans were taken while the temperature was held at 25, 100, 300, 500, 700, and 900 °C (Figure 3).

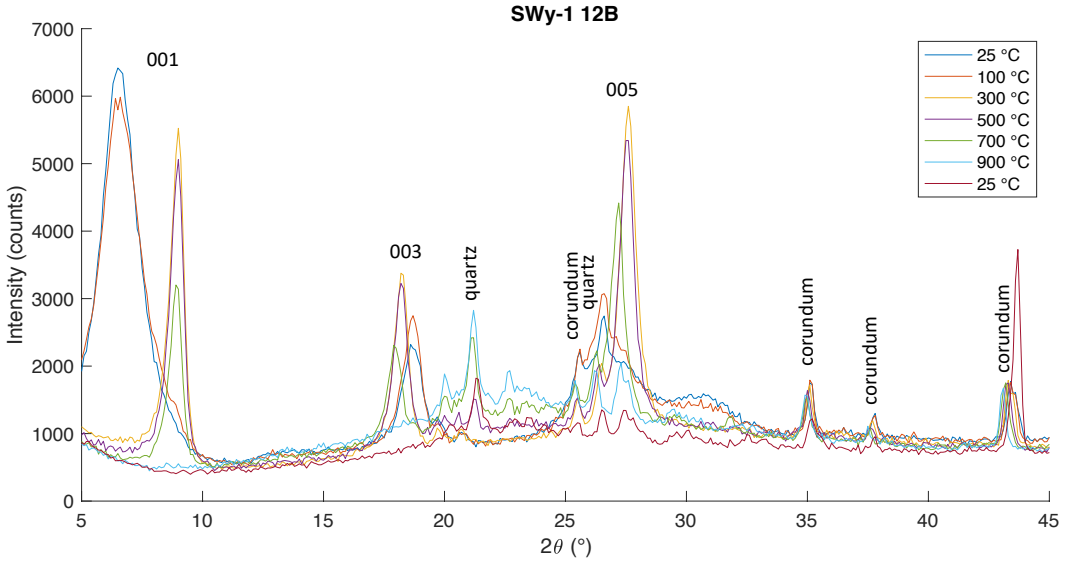

Figure 3: Experiment 12B. The initial scan of this sample is extremely similar to 12A, and it experiences nearly identical changes throughout.

The fourth heating experiment was performed on a sample with 15% corundum by weight (experiment 15B). The heating rate was set to a constant 5 °C/min, and short scans were conducted continuously as the temperature increased. Two scans were conducted at the maximum temperature of 900 °C, and then continuous scans were resumed once the cooling rate was set to -15 C/min (Figure 5). Scans of the full 5-45 degree range were taken before and after the experiment (Figure 4).

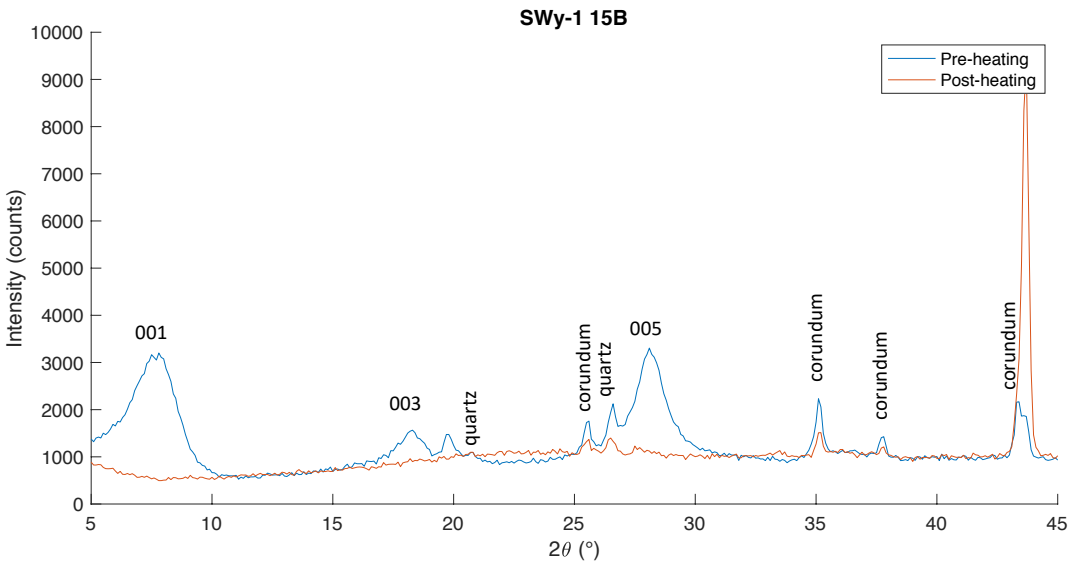

Figure 4: Full scans pre- and post-heating for experiment 15B. The post-heating scan is dominated by a large peak at approximately 44 degrees.

All of the 001 peak positions leveled out at 9 degrees, corresponding to a d-spacing of 1 nm. This likely indicates that interlayer water was driven out of the clay, leading to progressive collapse of the structure collapsed, until the sample was entirely dry. This is the same location of illite's 001 peak, however, the 003 and 005 peaks distinguish these collapsed smectites as separate phase.

It is likely that some aspect of the sample preparation process is the primary cause of the distinct differences between the 12A-B and 15A-B experiments. The elevated peak positions of 15A and 15B indicate they began the experiments in a more dehydrated state. Since the observed differences are not isolated to the low temperatures but instead propagate throughout the heating process, this has implications for the hydration state of in situ smectites that experience earthquake heating. Further experiments are planned to identify the cause of the differing initial hydration states and determine the scope of the effects once high temperatures have been reached.

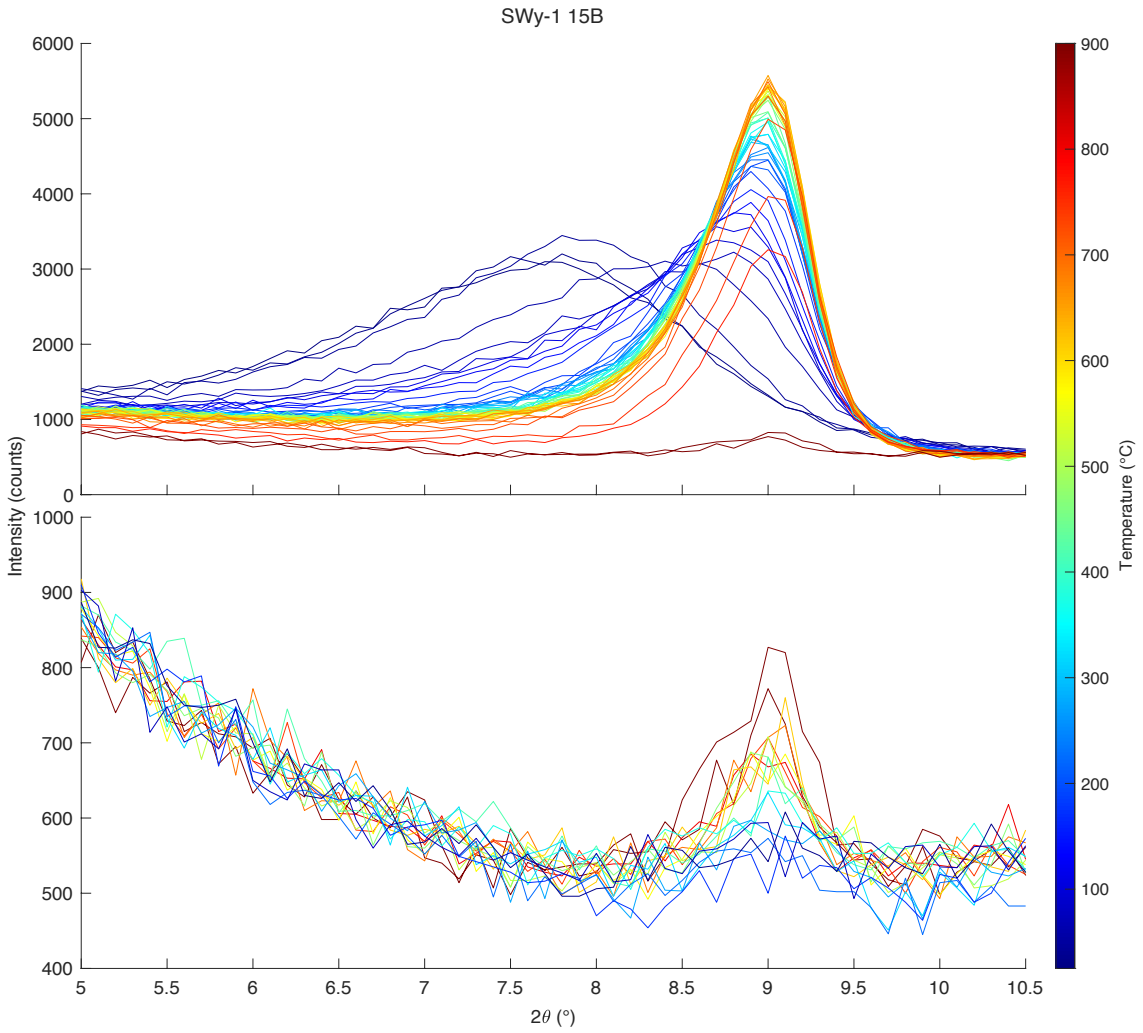

Figure 5: Experiment 15B, containing only the 001 peak for smectite.

In experiment 15B, the initial scan is similar to 15A, and quite different from the previous two experiments. The evolution of the peak is also similar to 15A, narrowing and increasing in intensity and 2θ as it is heated, until it begins rapidly decreasing in intensity at around 715 °C. Unfortunately, there was a data collection error, so the data between 775 °C and 900 °C is missing, but a dramatic decrease in peak intensity can be seen at 900 °C. Figure 5b shows scans throughout the cooldown process; the intensity of the peak is seen decreasing as the sample cools.

The large peak visible upon cooling at around 44 degrees during experiment 15 B corresponds to Inconel (Figure 4). Combined with overall intensity reduction upon cooling and the reduced quantity of sample still remaining on the mount upon removal, this indicates that more sample holder and less sample itself are being detected upon cooling. Thus, the reduction in intensity of the 001 peak seen during cooling for 15B (Figure 5b) is at least partially explained by physical damage to the sample. This reduced sample signal affected 12A and 12B as well; they also

experienced visible Inconel spikes upon cooling and poor condition of the samples upon retrieval (Figure 6). The cause of the sample damage has not yet been determined, but cracking due to rapid cooling is the most likely cause we have identified. Further experiments will attempt to reduce this damage to ascertain how much smectite structure can actually be preserved after reaching very high temperatures, as well as to retain samples for further analysis of rehydration potential.

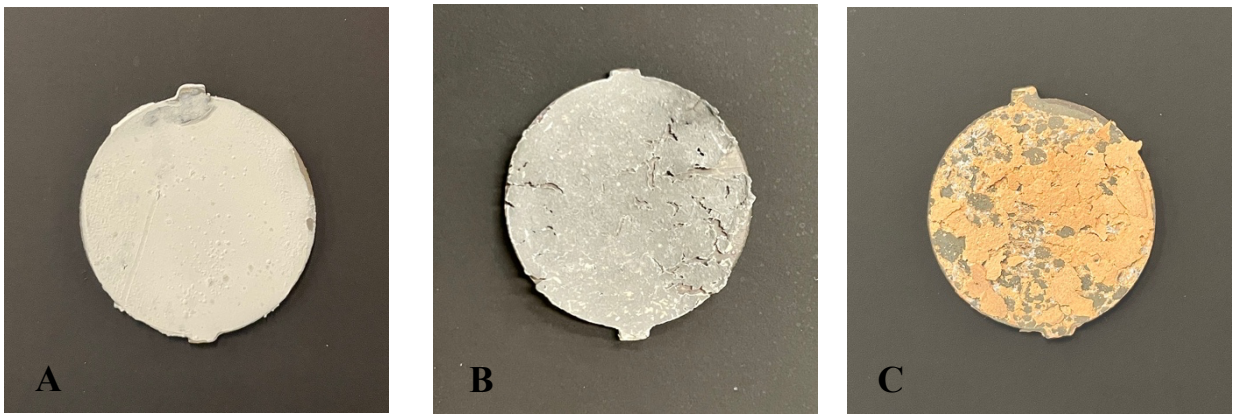

Figure 6: A) An unheated sample. B) The sample recovered from 15A that was heated to 700 °C. C) The sample recovered from 12B that was heated to 900 °C.

Conclusions and Further Work

All experiments indicate a prominent change in the smectite structure between 100 °C and 300 °C, and again between around 700 °C and 900 °C, even with the significant variability observed across initial hydration states and heating rates. Further experiments not exceeding these temperatures are needed to determine if these changes are preserved upon cooling and if these observed patterns can be applied to other samples to identify approximate temperature states. Future experiments will include testing illite and glauconite clays.

Broader Impacts

This grant has supported UCSC grad student Julia Krogh. Studies of fault zone temperature rise during earthquakes has led to better understanding of what controls earthquake propagation and arrest.

Publications

None at this time.

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