

**Dehydration-Induced Weakening at Sub-Seismic and Seismic
Strain-Rates and Experimental Constraints on the Rheology of
Serpentine and Olivine at Mantle Pressures**

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Introduction

Serpentine ($\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$) is a geodynamically significant mineral that forms from hydrothermal alteration of ultramafic rocks [e.g., *Spear*, 1993; *Evans*, 2013]. Serpentinite (a rock composed of mostly serpentine) commonly occurs within oceanic transform faults [e.g., *Gregg et al.*, 2007], mid-ocean ridge normal faults [e.g., *Cannat et al.*, 1992], oceanic detachment faults [*Escartin et al.*, 2003], the upper corner of the mantle wedge in subduction zones [e.g., *Hyndman and Peacock*, 2003; *Reynard*, 2013], subducting mantle in association with slab-bending faulting [*Ranero et al.*, 2003], and continental fault zones such as the San Andreas [*Moore and Rymer*, 2007; *Lockner et al.*, 2011]. Serpentine has a unique combination of mineral properties; it is stable to high pressures (~ 6 GPa in cold subduction zones) [e.g., *Perrilliat et al.*, 2005], has a significantly lower strength (2-4 order of magnitude) than the ultramafic minerals it replaces [e.g., *Hilaliret et al.*, 2007], and contains up to 13 wt % water. At temperatures greater than ~ 600 °C, serpentine reacts to either olivine, talc and water or olivine, pyroxene and water (depending on pressure) [*Spear*, 1993]. These unique properties make serpentine a fundamental component in many global and tectonic-scale processes including: the global water cycle by transporting water into the mantle [e.g., *Reynard* 2013], exhuming high-pressure metamorphic rocks in active subduction zones [e.g., *Gerya et al.*, 2002], decoupling the subducting slab from the mantle wedge [e.g., *Wada et al.*, 2008], initiating continental rifting [e.g., *Hirth and Guillot*, 2013], and possibly initiating plate tectonics [e.g., *Amiguet et al.*, 2012]. Furthermore, because serpentine occurs in many active fault zones it is an important material for understanding the seismic cycle (or lack thereof in some faults). To date the link between the properties of

serpentine and seismicity remains unclear, particularly within subduction zones [e.g., *Chernak and Hirth*, 2010], oceanic transform faults [e.g., *Boettcher and Jordan*, 2004; *McGuire et al.*, 2012], and normal faults near mid ocean-ridges [e.g., *Escartin et al.*, 2008].

To fully assess how serpentine affects the strength and slip stability of a fault zone at a given depth and temperature its important to understand: (1) the rheology of serpentine over its range of stability conditions at slip velocities (or strain-rates) spanning the seismic cycle (< 1 nm/s to > 1 m/s), (2) the transient rheologic changes that occur during serpentine dehydration as well as any feedbacks that maybe associated with fluid release and nucleation of reaction products, and (3) the rheology of reactions products (olivine + talc) that are generated during dehydration. To provide new insight on these issues I conducted rock deformation experiments on serpentine and serpentine reaction products. This work is separated into four chapters: in the first chapter I explored the frictional properties of serpentine at seismic slip rates and the feedbacks associated with metamorphism during shear heating, in the second chapter the rheology of serpentinite undergoing thermal decomposition, in the third chapter the rheology of stable serpentine at mantle conditions, and in the fourth chapter I explore the rheology of fully dehydrated serpentine. Finally, because one overarching goal of this work is to better understand the links between serpentine and seismicity, in the following sections I provide a brief overview of the distribution of seismicity in geologic regions known to contain serpentine.

Seismicity along mid-ocean ridges

Earthquakes near mid-ocean ridges primarily occur within high angle normal faults, strike-slip and shallow thrust faults that flank the topographic highs near the center of the ridge, [e.g., *Escartin et al.*, 2008; *Bergman and Solomon*, 1984]. The (centroid) depths of these earthquakes are shallow adjacent to the ridge ($< 6\text{ km}$) due to the high geothermal gradient and become progressively deeper away from the ridge at depths as high as 20 km. The deepest earthquakes occur at temperature no greater than $800\text{ }^{\circ}\text{C}$ [e.g., *Bergman and Solomon*, 1984]. Serpentine is thought to occur within these faults, particularly faults that transect the mantle. This is evidenced by cored and dredged samples from exposed oceanic mantle in the axial valley of the Mid-Atlantic Ridge [*Cannat et al.*, 1992] as well as within the footwall of oceanic detachment faults adjacent to the ridge [*Escartin et al.*, 2003].

Seismicity within mid-ocean ridge transform faults

Along mid-ocean ridge transform faults the majority of fault motion appears to occur aseismically [e.g., *Boettcher and Jordan*, 2004]. The earthquakes that do occur typically occur at depths ranging from 3-20 km, many of which extending well into the mantle. The maximum depth for seismicity is highly dependent on the thermal gradient: colder (and older/slower) lithosphere host deeper earthquakes than warmer lithosphere. The $\sim 600\text{ }^{\circ}\text{C}$ isotherm is generally thought to be a thermal maximum for seismicity (assuming a half-space cooling model) [e.g., *Abercrombie and Ekstrom*, 2001], however, this conclusion is strongly model dependent and earthquakes may occur at even higher temperatures [*Behn et al.*, 2007]. *McGuire et al.*, 2012 suggested the relative lack of large earthquakes was caused largely by the material properties of the fault, which is

presumably serpentine-rich material for temperatures $< 600^{\circ}\text{C}$ and olivine-rich material at higher temperatures.

Intermediate-depth seismicity within subduction zones

Earthquakes within subduction zones are observed from very shallow depths near the trench down to depths as deep as 700 km [e.g., Kirby *et al.*, 1991]. Of these only the intermediate depth events (50-300 km) occur in rocks that contain serpentine [e.g., Hacker *et al.*, 2003], which include the mantle wedge and subducting lithospheric mantle. In general, there is a strong correlation between the location of intermediate-depth earthquakes and the predicted stability of hydrous minerals (e.g., serpentine, lawsonite, amphibole and chlorite), suggesting a link between dehydration reactions and seismicity [e.g., Hacker *et al.*, 2003; Abers *et al.*, 2013]. Earthquake hypocenters are typically observed along parallel trending planes within the subducting crust and mantle giving rise to the double Benioff zone [e.g., Brudzinski *et al.*, 2007]. The overall distribution of these earthquakes is highly dependent on the thermal structure; in older (cold) lithosphere, earthquakes primarily occur within the subducting crust and deep within the mantle, whereas in younger (hot) lithosphere, earthquakes primarily occur within the mantle near the slab Moho [Abers *et al.*, 2013]. Within the serpentinized corner of the mantle wedge large earthquakes are not common, however in warmer subduction zones ‘slow’ earthquakes (e.g., slow slip events, very low frequency earthquakes and non-volcanic tremor) are observed [e.g., Obara, 2002; Rogers and Dragert, 2003].

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serpentinization. *Journal of Geophysical Research: Solid Earth*, 113, B04402.

CHAPTER 1

Dynamic weakening of serpentinite gouges and bare-surfaces at seismic slip rates

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Abstract

To investigate differences in the frictional behavior between initially bare-rock surfaces of serpentinite and powdered serpentinite ('gouge') at sub-seismic to seismic-slip rates, we conducted single velocity-step and multiple velocity-step friction experiments on an antigorite-rich and lizardite-rich serpentinite at slip rates (V) from 0.003 m/s to 6.5 m/s, sliding displacements up to 1.6 m, and normal stresses (σ_n) up to 22 MPa for gouge and 97 MPa for bare surfaces. Nominal steady-state friction values (μ_{ss}) in gouge at $V = 1$ m/s are larger than in bare surfaces for all σ_n tested and demonstrate a strong σ_n dependence; μ_{ss} decreased from 0.51 at 4.0 MPa to 0.39 at 22.4 MPa. Conversely, μ_{ss} values for bare surfaces remained ~ 0.1 with increasing σ_n and V . Additionally, the velocity at the onset of frictional weakening and the amount of slip prior to weakening were orders of magnitude larger in gouge than in bare surfaces. Extrapolation of the normal stress dependence for μ_{ss} suggests that the behavior of antigorite gouge approaches that of bare surfaces at $\sigma_n \geq 60$ MPa. X-ray diffraction revealed dehydration reaction products in samples that frictionally weakened. Microstructural analysis revealed highly localized slip zones with melt-like textures in some cases gouge experiments and in all bare-surfaces experiments for $V \geq 1$ m/s. 1-D thermal modeling indicates that flash heating causes frictional weakening in both bare surfaces and gouge. Friction values for gouge decrease at higher velocities and after longer displacements than bare surfaces because strain is more distributed.

1. Introduction

Our understanding of the frictional behavior of faults at seismic slip velocities (>0.1 m/s) has significantly improved over the last 15 years with experiments performed on initially bare rock surfaces [e.g., *Di Toro et al.*, 2004; *Goldsby and Tullis*, 2011; *Han et al.*, 2007; *Hirose and Shimamoto*, 2003; *Tsutsumi and Shimamoto*, 1997] and gouges [e.g., *Brantut et al.*, 2008; *Han et al.*, 2010; *Kitajima et al.*, 2010; *Mizoguchi et al.*, 2009; *Reches and Lockner*, 2010]. In general, these studies of high-velocity friction (HVF) demonstrate that rock friction coefficients decrease dramatically from ~ 0.7 to as low as 0.1 as slip velocities approach seismic rates and (in most cases) increase rapidly as velocities decelerate; this general behavior is nominally independent of rock composition [*Di Toro et al.*, 2011; *Goldsby and Tullis*, 2011]. Such dynamic fault-weakening behavior revealed in laboratory experiments is consistent with several earthquake-related observations retrieved from:

1) seismology: e.g., the large stress drops constrained from analysis of seismic radiation patterns of some earthquakes [*Imanishi and Ellsworth*, 2006; *Malagnini et al.*, 2010; *Viegas et al.*, 2010], or the (debated) breakdown of the scaling between radiated energy and seismic moment [*Abercrombie*, 1995; *Kanamori and Heaton*, 2000];

2) geophysics: e.g., the lack of a pronounced heat flow anomaly along major fault zones [*Lachenbruch and Sass*, 1992; *Fulton et al.*, 2013] or the large seismic slip accommodated in fault patches in the Sumatra 2004 of moment magnitude (M_w) 9.3 [15 m of max slip, *Stein and Okal*, 2005] and the Tohoku 2011 M_w 9.0 [50 m of max slip, *Fujiwara et al.*, 2011] events;

3) geology: e.g., estimates of coseismic frictional strength obtained from ancient exhumed faults [e.g., *Di Toro et al.*, 2006; *Griffith et al.*, 2009] or active deep-drilled seismic faults [*Chester et al.*, 2013; *Hirono et al.*, 2007].

A number of physical mechanisms have been proposed to explain the dynamic weakening behavior observed in experiments and postulated to occur on faults [see *Di Toro et al.*, 2011; *Niemeijer et al.*, 2012; *Rice and Cocco*, 2007 for a summary]. In particular, mechanical data and microstructural investigations of experimentally deformed bare rocks are consistent with flash heating of asperities [*Goldsby and Tullis*, 2011; *Violay et al.*, 2014], frictional melting [*Di Toro et al.*, 2006; *Hirose and Shimamoto*, 2005; *Niemeijer et al.*, 2011; *Spray*, 2005], silica gel weakening [*Di Toro et al.*, 2004; *Goldsby and Tullis*, 2002] and superplasticity (grain boundary sliding accommodated by dislocation motion or diffusion) [*Green et al.*, 2010; *Holdsworth et al.*, 2013; *Schubnel et al.*, 2013]. However, all faults generate a mm to cm thick layer of gouge during rupture and seismic slip [*Reches and Dewers*, 2005], even within their deeper roots [6-15 km, e.g., *Sibson*, 1977; *Snoke et al.*, 1999]. This raises the questions: Which dynamic-weakening mechanisms occur in gouge-bearing faults? How might the presence of gouge modify the occurrence and/or efficacy of these weakening processes at seismic slip rates? Lubrication due to the presence of powders [i.e., powder lubrication: *Han et al.*, 2010; *Reches and Lockner*, 2010; *Tisato et al.*, 2012] is inconsistent with the rapid recovery of frictional strength at the end of sliding. Moreover, in exposed fault zones it is commonly observed that slip tends to be localized along very thin surfaces within gouge [e.g., *Chester and Chester*, 1998; *Fondriest et al.*, 2013; *Sibson*, 2003], leading some workers to suggest that once strain is localized within gouge the system will

emulate bare surface slip behavior [e.g., *Smith et al.*, 2012; T. Tullis, personal comm.]. But is it appropriate to extrapolate rock friction behavior obtained in rock-on-rock friction experiments to natural gouge-bearing faults? Furthermore, how does the effective normal stress affect this behavior? Interestingly, the results from *Smith et al.* [2013] on calcite gouge and *Han et al.* [2007] on (cohesive) calcite-bearing marble suggest that the shear stress or strength of calcite gouge is a factor of 2 or greater than marble bare surfaces at seismic slip velocities despite having localized strain.

Serpentinite is a common rock type in the oceanic lithosphere, and earthquakes may propagate into serpentinized mantle along mid-oceanic ridges, transform faults and subduction zones; the latter alone release about 85-90% of the global seismic moment [*Scholz*, 2002]. For this reason, the frictional behavior of serpentinite has been studied over a wide range of slip rates from plate rates to seismic slip rates [e.g., *Hirose and Bystricky*, 2007; *Kohli et al.*, 2011; *Reinen et al.*, 1992]. Moreover, serpentine group minerals are expected to react to talc, olivine and enstatite due to frictional heating during rapid slip. These minerals are thought to be stable in the geologic record, and could therefore provide evidence for seismic slip [e.g., *Kohli et al.*, 2011]. Currently, the only widely accepted evidence for ancient seismic faulting is the presence of pseudotachylytes [*Sibson*, 1975]. Other proposed geologic evidence for seismic slip includes thermally altered biomarkers in sedimentary rocks [*Polissar et al.*, 2011], peculiar crystal-plastic features [*Bestmann et al.*, 2012; *Smith et al.*, 2013], injection of fluidized gouge [*Fondriest et al.*, 2012; *Lin*, 2011; *Rowe*, 2013] and the combination of mirror-like surfaces with truncated and exploded grains [*Fondriest et al.*, 2013; *Siman-Tov et al.*, 2013]. As a consequence, the occurrence of serpentine breakdown minerals in slipping

zones could be indicative of ancient seismicity in faults exhumed from seismogenic depths, outlining the importance for further field studies of exhumed fault zones hosted in oceanic rocks.

Employing a rotary-shear apparatus, we extend the study of the frictional behavior of serpentinite rocks to higher normal stresses (up to 96.6 MPa for bare surfaces and 22.4 MPa for gouges) and slip velocities (up to 4.3 m/s for bare surfaces and 6.5 m/s for gouges) than investigated previously. We also explore differences in dynamic frictional-weakening behavior observed on serpentine gouge and during tests on initially bare surfaces of serpentine by conducting relatively short displacement, high-velocity experiments while varying the normal stress between tests. Following each experiment the slip surfaces and wear material were analyzed with X-ray powder diffraction (XRPD) and several microstructural analysis techniques. These analyses, coupled with 1-D thermal modeling, allow us to constrain the effects of velocity, normal stress, shear heating, strain localization and dehydration reactions on dynamic frictional weakening of serpentine and, by extension, other materials.

2. Experimental Procedures

2.1 Sample preparation and data acquisition

Twenty-seven frictional sliding experiments on initially bare-surfaces and powdered rock samples were performed with SHIVA (Slow- to High-Velocity rotary-shear friction Apparatus) at the Istituto Nazionale di Geofisica e Vulcanologia (INGV) in Rome, Italy [for a description of SHIVA see *Di Toro et al.*, 2010; *Niemeijer et al.*, 2011]. Two materials were tested: an antigorite-rich serpentinite (ATG) and a lizardite-rich serpentinite (LIZ). ATG samples contain ~90% antigorite with minor magnesite and

magnetite; LIZ samples contain ~80% lizardite, ~14% clinochlore (chlorite) and minor magnetite and trace amounts of olivine and enstatite, as determined by petrographic and XRPD analyses (Table 1).

Bare surface samples were first cored into two ~50 mm long solid cylinders. The cylinders were pressed inside precut aluminum rings with an inner and outer diameter of 50 mm and 55 mm respectively and cemented within the rings with epoxy. Next, using a lathe, a depression was machined into the end face of each cylinder to yield an annulus with 50 and 30 mm outer and inner diameters, respectively. The sliding surface was ground with 320 grit sandpaper and samples were loaded into SHIVA using holders described in *Niemeijer et al.* [2011] (Figures 1a and 1b). Powdered ('gouge') samples were crushed and sieved between 37 and 105 μm , although some finer-grained material was also present in the starting material. The powder was evenly packed into a steel gouge holder (55 and 35 mm outer and inner diameters) and sheared between two roughened steel discs (Figures 1c, 1d, and 1e)(for a description of the steel gouge holder and its calibration, see *Smith et al.*, 2013). Normal stress was applied from the non-rotary end with an air-actuated pneumatic piston cylinder and servo-controlled via an electro-valve in parallel with a digital pneumatic regulator. The regulator has a resolution of 0.02 bars and a response time of 0.2 s for a step of 50% of full scale [*Di Toro et al.*, 2010].

In all gouge experiments, 4 g of powder was used, yielding a ~1.7 mm thick layer of compacted gouge. Slight variations in thickness occurred after initial loading of the gouge. This produced small fluctuations in the shear stress data with a wavelength of ~150 mm, consistent with the average circumference of the gouge holder (outer and inner

circumferences of 173 and 111 mm). The normal stress also fluctuated in response to variations in thickness. These fluctuations were in some cases accentuated by the delayed servo-response of the regulator such that the stress was either over-or under-corrected causing variations as high as 5%; we report the average imposed normal stress during deformation in Table 1. In most experiments the sample holder prevented extrusion of the gouge, but in Runs 705, 818, 825 and 826, as much as ~0.2 g (~85 μ m of thickness) of the gouge leaked from the holder during deformation. In Runs 727 and 733 sliding displacement was not recorded; however we still report mechanical data assuming that the imposed velocity profile was similar to that in other experiments with identical experimental parameters. Mechanical data (axial load, torque, axial displacement, and angular rotation) were acquired at a frequency of 25 Hz for samples deformed at velocities less than 0.1 m/s and 25 kHz for higher slip velocities. The total slip, slip rate, and shear stress were determined following methods discussed in *Di Toro et al.* [2010]. High-frequency noise in the data was reduced with a Fast-Fourier-Transform (FFT) smoothing filter (Appendix A).

Following each gouge experiment, a portion of the slip surface was collected for XRPD analysis. Similarly, following each bare surface experiment a portion of the wear material was collected for XRPD analysis. All but one XRPD analyses was conducted at Geosciences Dept. Padua, Italy (see Appendix B for XRPD analysis details).

Micro-imaging was conducted with an Olympus SZX16 optical microscope fit with a digital camera, a JEOL 6500 and LEO 1500VP field-emission scanning electron microscope (SEM) with an accelerating voltage of 10-20 kV and a Cameca SX100 electron microprobe.

2.2 Multiple velocity-step experiments

In each multiple velocity-step experiments we imposed the identical preset velocity function. Samples were first deformed at a sliding velocity of ~ 3 mm/s to a displacement of 60 mm, then the velocity was stepped to ~ 4.5 m/s then decelerated back to rest, resulting in a total displacement of ~ 0.5 m. We imposed an acceleration and deceleration of ~ 40 m/s², however, the servo-controlled response of the motor tends to overshoot this value and we observe initial peaks as high as 65 m/s² during acceleration and 70 m/s² during deceleration (see Table 1). In these experiments a constant normal stress (σ_n) was imposed, ranging from 4.9 to 19.5 MPa in bare-surface experiments (Runs 734, 727, 735, and 733), and 7 to 8.5 MPa in ATG gouge experiments (Runs 824b and 818), and 8.5 MPa in one LIZ gouge experiment (Run 705).

2.3 Single velocity-step experiments

In single velocity-step experiments, samples were accelerated from rest to a set velocity then decelerated back to rest after a preset amount of displacement. In one suite of experiments the samples were slid at 1 m/s for ~ 1 m of displacement with an imposed acceleration and deceleration of ~ 20 m/s²; actual accelerations and decelerations peaked as high as 65 m/s² (Table 1). In bare-surface experiments (Run 736) a constant σ_n was imposed, ranging from 5 to ~ 97 MPa, which is the highest normal stress ever applied in high-velocity friction experiments on natural rocks: four times higher than in the study of *Hirose and Bystricky* [2007] (see Appendix C for normal stress calculation of these runs). In ATG gouge experiments (Runs 820, 745, 821, 822, and 823), a constant σ_n was imposed, ranging from 4 to 22.4 MPa and in LIZ gouge experiments (Runs 747, 746, 748, 825, and 826) from 3.8 to 18.7 MPa. We were not able to deform gouge at higher

normal stresses due to the torque limit of SHIVA. We also deformed ATG gouge at 0.1 m/s and 13.3 MPa for 1 m of slip (Run 835) and ~6.5 m/s and 10.9 MPa for 1.6 m of displacement (Run 834), respectively.

3. Results

3.1 Multiple velocity-step tests

Results from all multiple velocity-step tests are reported in Table 1. Three representative experiments (Runs 705, 824b and 735) are shown in Figure 2a. These tests illustrate differences between the frictional behavior of serpentine bare surfaces and serpentine gouge at sub-seismic and seismic slip velocities.

During slip at 3 mm/s, ATG bare surfaces and LIZ and ATG gouge had similar nominal steady-state values of the friction coefficient (μ_{nss}) but differed in their frictional stability. μ_{nss} is the average of the relatively constant friction values generated during slip at these velocities. We use the term “nominal” to acknowledge that friction may evolve with continued displacement due to changes in the shear zone microstructure and/or thermal structure. Values of μ_{nss} for ATG were ~0.67 for bare surfaces (although slightly higher at lower normal stresses), ~0.7 for ATG gouge, and ~0.65 for LIZ gouge (Table 1, labeled *Slow* μ_{nss}). Both LIZ and ATG gouge displayed stable sliding and slight strain hardening. Conversely, stick-slip instabilities occurred in all bare surface experiments at 3 mm/s. These events were audible and produced 0.1-0.25 MPa stress drops, as reflected in the friction record (Figure 2b).

After the velocity increased, friction of both bare surface and gouge samples first decreased (dynamically weakened) over a finite sliding displacement, reaching a minimum value. During deceleration, friction increased (recovered), resulting in a U-

shaped friction versus displacement profile (e.g., Figure 2a). In all runs there were sustained flat regions defining the minimum of the U-shaped profiles in which friction was relatively constant with changes in velocity. Similar to sliding at lower velocities, we define the average friction over these flat regions as μ_{nss} (labeled as *Fast* μ_{nss} in Table 1). The μ_{nss} values for ATG bare surfaces were ~ 0.1 while those for ATG and LIZ gouge ranged from 0.35 to 0.38 (Table 1).

To characterize the dynamic weakening, we report the amount of slip required to reach the new nominal steady-state friction value after the velocity step. Previous studies have defined this length-scale as either the slip-weakening distance (D_{w}), the distance over which the shear stress (or friction) drops by 95% of the total stress drop [*Hirose and Shimamoto, 2005; Mizoguchi et al., 2009*], or the thermal-weakening distance (D_{th}), the distance over which stress falls to $1/e$ of the total stress drop [*Di Toro et al., 2011*]. We report both distances in Table 1 for comparison with previous studies, however we discuss only D_{th} in this study. Values of D_{th} in bare surface experiments on ATG ranged from 3 to 8 mm. In contrast, for the same acceleration and normal stresses, D_{th} values in both the ATG and LIZ gouge experiments were over an order of magnitude larger, ranging from 40 to 180 mm.

To further quantify dynamic weakening, we report the velocity at the onset of frictional weakening, which we refer to as the fall-off velocity (V_{f}). Values of V_{f} were determined by first noting the displacement at which friction rapidly decreases (green and gray star in Figure 2a), then finding the corresponding velocity for the given displacement (Figure 2c). Values of V_{f} thus determined are simply a first-order observation dependent upon the imposed experimental conditions. For otherwise

equivalent experimental conditions, V_f in gouge was as much as an order of magnitude larger than that in bare surface experiments (Table 1).

3.2 Single velocity-step tests at 1 m/s

The results of all single velocity-step experiments are reported in Table 1 and illustrated in Figure 3. In general, these experiments demonstrated similar differences between the behavior of bare surfaces and gouge observed in the multiple velocity-step experiments. In addition, because the velocity was held constant and normal stress was varied we were able to explore the normal stress dependence on μ_{nss} , D_{th} and V_f (compiled in Figure 4).

Values of μ_{nss} decreased with increasing normal stress in both gouge and bare surface experiments (Figures 3a, 3b and 3c); the normal stress dependence is larger for gouge than for bare surfaces (Figure 4a). Values of μ_{nss} for bare surfaces decreased from 0.14 to 0.11 with an increase in σ_n from 5 to ~ 30 MPa and remained at a value of ~ 0.11 with additional increase in σ_n from 30 to ~ 97 MPa (Figure 4a). The friction data for gouge are more scattered than for bare surfaces. Interpretation of these experiments is complicated by (1) variations in sample thickness which caused sinusoidal oscillations in shear and normal stress, and (2) sample leaks, which caused spikes in the data. As such, the reported μ_{nss} values have larger standard deviations (Table 1). Values of μ_{nss} for ATG gouge decreased from 0.51 to 0.39 with an increase in σ_n from 4 to 22.4 MPa (Figure 4a). Friction for LIZ gouge did not appear to reach a steady-state value in any single-velocity experiment (Figure 4a); for these experiments we report only a minimum friction value (μ_{min}). Values of μ_{min} for LIZ gouge decreased from 0.50 to 0.31 with an increase in σ_n from 3.8 to 18.7 MPa. The ATG and LIZ gouge samples deformed at $\sigma_n \sim 12$ MPa (Runs

821 and 748) deviate somewhat from this general trend. Values for μ_{ns} from multiple velocity-step experiments (with maximum slip rates of ~ 4.5 m/s) are generally consistent with single velocity-step results (with maximum slip rates of ~ 1 m/s) (Figure 4a).

Values of D_{th} for both ATG bare surfaces and gouge decreased with increasing normal stress, though there is scatter for the gouge samples (Figure 4b). We did not calculate D_{th} values for LIZ gouge because steady-state friction was not reached. Values of D_{th} for ATG bare surfaces decreased from 0.46 to 0.15 mm with an increase in σ_n from ~ 11 to 97 MPa. Values of D_{th} for ATG gouge are more than an order of magnitude greater than for ATG bare surfaces and decreased from 130 to 57 mm with an increase in σ_n from 4 to 22.4 MPa. Values of D_{th} from multiple velocity-step experiments are similar to those observed from single velocity-step experiments on both bare surface and gouge samples (Figure 4b).

Values of V_f are independent of normal stress in bare-surface experiments and decrease modestly with increasing normal stress in gouge experiments; V_f is much smaller for bare surface samples than gouge samples deformed at the same conditions. Figure 4c shows that values of V_f for all ATG bare surface experiments were ~ 0.2 m/s. In contrast, for approximately the same velocity profile, V_f decreased from 1 to 0.76 m/s for LIZ gouge and from 1 to 0.87 m/s for ATG gouge with increasing normal stress. These trends in V_f for gouge also reflect a decrease in the amount of slip that occurred prior to frictional weakening with increasing normal stress; at low σ_n , the gouge experiments reached the peak velocity (1 m/s) and continued to slip for tens of millimeters before frictional weakening occurred, while at higher σ_n , the gouge weakened during acceleration before the peak velocity was attained. For bare surfaces, values of V_f in

multiple velocity-step experiments are similar to those observed from single velocity-step experiments (Figure 4c). In contrast, for gouge samples, values for V_f are significantly higher in the multiple velocity-step experiments (Figure 4c).

3.3 Additional single velocity-step tests

To better constrain the velocity and displacement dependence of friction for the ATG gouge samples, we conducted two single velocity-step experiments at 0.1 m/s and ~ 6.5 m/s (Runs 835 and 834, Figure 5). These experiments were conducted at normal stresses of 13.3 and 10.9 MPa, respectively. No frictional weakening was observed at 0.1 m/s (Run 835, blue trace in Figure 5); the value of μ_{nss} remained ~ 0.65 throughout the duration of the 0.9 m slip cycle. In contrast, the sample deformed at a peak velocity of 6.5 m/s (Run 834, red trace in Figure 5) yielded a value of the friction coefficient of 0.31. This sample was also deformed to a larger amount of slip, ~ 1.6 m, than in the other single-velocity tests (slip ~ 1 m) and velocity-stepping tests (slip ~ 0.45 m). As is shown in Figure 5, the friction coefficient was independent of velocity and achieved a steady value (~ 0.3) during the acceleration stage for $V > 4$ m/s (after 0.2 m of slip) and began to increase during the deceleration stage for $V < 2.5$ m/s (after 1.5 m of slip).

3.4 Velocity dependence and frictional hysteresis

In all of the high-velocity experiments, we observe hysteresis in plots of friction versus velocity, with greater velocity dependence and higher friction values during acceleration than deceleration (e.g., Figures 2a, 3a, 3b, 3c and 5). In Figure 6a we plot unfiltered data for two multiple velocity-step experiments on ATG bare surfaces (Runs 734 and 735; blue and green traces). In this figure the value of friction traces a clockwise ‘path’ in log velocity space. During acceleration from 3 mm/s to ~ 0.1 m/s, the friction

remains nominally constant; friction then begins to decrease at a velocity of ~ 0.1 m/s, and decreases more rapidly at velocities above ~ 0.3 m/s. In Figure 6b we plot the friction data for ATG gouge during one single velocity-step experiment (Run 834, red trace in Figure 6b). In this run, there was no initial period of slow slip at 3 mm/s; the sample was accelerated from rest to the target slip rate of 6.5 m/s. We also plot the data from a multiple velocity-step gouge experiment (Run 824b, black trace in Figure 6b); in this test, the sample was slid for 60 mm at 3 mm/s and then accelerated to 4.5 m/s.

During acceleration, the gouge experiments show behavior similar to that for the bare surface experiments, except that for the gouge, the onset of weakening is shifted to higher velocities (~ 1 m/s) producing a larger hysteresis (Figure 6b). During deceleration, friction during both ATG bare surface and ATG gouge experiments increases to values somewhat lower than those determined during the initial low-velocity portion of the experiments; friction for the gouge recovers at a higher velocity (~ 0.2 m/s) than for the bare surfaces (< 0.02 m/s) (Figures 6a and 6b).

In Figure 6 we superimpose μ_{nss} data on the friction path data for experiments on bare surfaces (green circles) and gouge (green squares). In both diagrams, black numbers correspond to the run number. For velocities below ~ 0.1 m/s, values of μ_{nss} are similar to the transient values observed during acceleration. In contrast, at higher velocities, values of μ_{nss} are similar to the transient values observed during deceleration.

3.5 Microstructural and mineralogical analysis

3.5.1 Bare surface experiments

All bare-surface samples deformed at velocities of 1 m/s and higher developed grooved striations and exhibit glass-like material on the slip surface. The glass-like

material was observed in two textural forms. The first was a dark-green veneer with glassy sheen that occurred in bands along topographically higher ridges on the slip surface (Figure 7a). This material was observed in all bare-surface experiments. An SEM micrograph of one of these surfaces shows finger-like tendrils and bulbous structures (Figure 7b), similar to glass fibers described in *Friedman et al.*, (1974). The second textural form of glass-like material was 10-20 μm thick glassy patches of light-green and translucent material observed on the slip surface (Figures 7c and 7d) of samples deformed at $\sigma_n > 20$ MPa. The glassy material is composed of a nominally homogenous ultra-fine grained matrix (i.e., the matrix grain-size is either too small to resolve with an SEM (<10 nm) or amorphous) with larger magnetite inclusions as suggested by backscattered electron SEM micrographs (Figure 7e and 7f) and exploratory micro-chemical analyses. These glassy patches occurred as bands oriented parallel to striations on the slip surface [similar to the "melt welts" described by *Brown and Fialko*, 2012] and are more numerous in samples deformed higher normal stresses. At the highest normal stress the entire slip surface appeared to be covered with the glass-like material.

X-ray powder diffraction analysis of wear material collected after bare surface experiments showed evidence of serpentine dehydration products, olivine and enstatite (Table 1), in all samples tested (all of which were deformed at high enough slip velocity to cause frictional weakening). In several ATG bare surface experiments (e.g., Runs 733, 734, and 735) magnesite was detected, but never in the LIZ experiments where carbonates were not present in the starting material. A comparison of XRPD profiles shows a systematic increase in the peak heights of olivine with increasing applied normal

stress (Figure 8), suggesting a relative increase in the mass of the reaction products.

None of the bare surface samples showed XRPD evidence for talc.

3.5.2 Gouge experiments

In all gouge samples some degree of strain localization and grain-size reduction was observed. The ATG gouge sample tested at $V=0.1$ m/s was poorly consolidated after deformation and developed a 400-500 μm -thick zone of reduced grain size where strain was apparently localized (Figure 9a). The localized zone was composed of a matrix of 50-500 nm rounded antigorite grains (Figure 9b). This sample did not develop a well-defined slip surface or slickenlines.

ATG and LIZ gouge samples deformed at $V \geq 1$ m/s developed 100-200 μm thick fine-grained localized zones consisting of an ultra-fine-grained matrix hosting clasts ranging from < 1 to 20 μm in diameter (Figures 9c and 9e). The fine-grained zones tend to be welded in all samples (e.g., Figures 9d & 9f). In some cases, microstructures indicate that the welded zones were brecciated after welding. The inspection of the localized zone from Run 746 (LIZ) illustrates clasts that are welded aggregates of fine-grained material (Figure 9f), suggesting a welded zone formed first then became brecciated and comminuted. In instances where the localized slip surface was exposed during sample recovery, we observed dark glassy striated patches (Figure 9g). These thin zones appeared translucent and became more abundant in samples deformed at higher normal stresses. An SEM image shows the cross-sectional profile of the dark glassy material (Figure 9h). At higher normal stresses and at the highest slip velocities (Run 834) we observed whitish (e.g., Figure 9g) and brownish streaks along the slip surface in addition to the dark glassy material. Brownish streaks are composed of micron- to

submicron-in size granular clasts; spot XRPD analysis of one of these streaks revealed strong peaks for olivine and weak peaks for enstatite (Run 834, Table 1). A cross-section through a whitish streak in an ATG sample (Run 823, Figure 9i and 9j) shows an ultra-fine-grained matrix with amoeboid-shaped vesicles. The vesicles are dispersed in a glass-like matrix probably resulting from quenching of the friction melt. The vesicles become smaller with increasing distance from the slip surface; next to the latter they become interconnected defining a shear-parallel fabric (Figure 9j). Similar glass-like material and degassing related textures were observed in LIZ samples. Figures 9k and 9l show an ultra-fine-grained zone that appears to have been injected into the brecciated zone. In this sample we note a high density of vesicles near the top of this apparently once-molten layer.

In both LIZ and ATG gouge samples strain localized within the gouge layer near the interface between the gouge holder and the stationary side of the shearing rotary disk (dashed line, Figure 1e). In all ATG samples deformed at $V \geq 1$ m/s we observed little textural evidence for deformation outside of the 100-200 μm thick localized zone. This is also true of LIZ gouge samples at lower normal stresses (<10 MPa), however, in samples from Runs 825 and 826 deformed at a normal stress of 18 and 19 MPa, respectively, we observed localized zones containing ultra-fine-grained (glassy) material adjacent to both the stationary and rotary-side of the sheared layer. Further microstructural inspection of these samples revealed a varying degree of deformation throughout the shearing gouge layer.

X-ray powder diffraction revealed a general trend that gouge samples that underwent frictional weakening showed evidence for serpentine dehydration products

(Table 1). In contrast, Run 835 (which was conducted at 0.1 m/s and showed no frictional weakening) did not show XRPD peaks for olivine or enstatite (Table 1). Two samples did not fit this trend. One LIZ sample (Run 747) did not demonstrate frictional weakening, yet contained trace amounts of olivine and enstatite; we suspect these phases were inherited from the starting material. One ATG sample (Run 820) demonstrated frictional weakening, yet contained no XRPD evidence for dehydration; since this sample was deformed at 4 MPa we suspect the amount of reaction products may have been too small to resolve with XRPD. Diffraction peaks for talc were not found in any samples.

4. Discussion

Our experiments show that serpentine bare surfaces and gouge undergo frictional weakening during high-velocity shear experiments. However, friction evolves differently in each material with changes in slip displacement, velocity and normal stress. These differences can be reconciled by exploring the underlying processes that promote dynamic weakening: strain localization and shear heating. In the following discussion we first compare our bare surface data to previous high-velocity friction studies on similar materials. This comparison allows us to develop a conceptual model to better understand experimental observations on serpentine bare surfaces. We then use 1-D thermal models to explore how measured friction values are affected by frictional heating at the surface-scale (mm) and asperity-scale (μm) in both bare-surface and gouge experiments.

4.1. Displacement- and velocity-dependent hysteresis of friction on bare surfaces

Values of macroscopic friction for a given sliding surface are a manifestation of frictional heating and temperature evolution on both the macro- (slip-surface) scale and

the asperity-scale (μm). These relationships are qualitatively demonstrated via variations in hysteresis loops between short and long displacement experiments shown in Figure 10a. In Figure 10a we plot results from our ATG bare-surface experiments together with results of 50 experiments from *Kohli et al.* [2011] on ATG bare surfaces conducted at a normal stress of 5 MPa, a maximum velocity of 0.3 m/s, and a maximum acceleration of 10 m/s^2 . This data set can be subdivided into two friction paths shown schematically in Figure 10b. Path 1 is modeled after the friction data from small displacement experiments ($<0.05 \text{ m}$) from *Kohli et al.* [2011] shown in Figure 10a. *Kohli et al.* [2011] concluded that weakening occurred via flash heating of asperities above a critical weakening velocity (V_w) of $\sim 0.1 \text{ m/s}$ for samples deformed at room temperature, based on the coincidence of the steady-state friction data (grey diamonds in Figure 10a) with the deceleration-path data, the observation of talc in XRPD analyses from the wear material of high velocity samples, and the general agreement between the data and theoretical descriptions for flash weakening [*Beeler et al.*, 2008; *Rice*, 2006].

Flash weakening theory predicts that values of friction should decrease when the asperity velocity exceeds the weakening velocity V_w [*Rice*, 2006]. However in all experiments in which the slip rate was accelerated above V_w friction remained transiently high before decreasing to steady-state values (black ovals, Figure 10a) resulting in a modest hysteresis. This velocity overstepping represents a deviation from theoretical predictions and is characterized using the difference between the fall-off velocity (V_f) and the critical weakening velocity (V_w), defining an overstep velocity ($V_f - V_w$) (Figure 10b). Following *Kohli et al.* [2011] and *Goldsby and Tullis* [2011], we posit that the overstepping results from the displacement required for strain localization and subsequent

shear heating to occur within the thin gouge layer generated by wear between the bare surfaces. The displacement that occurs during acceleration between V_f and V_w is defined as an overstep displacement (δ) following *Kohli et al.* [2011] (labeled δ_w therein). All δ values are reported in Table 1 assuming $V_w = 0.1$ m/s. In this study δ was ~ 0.4 mm for single step velocity-step tests and ranged from 0.5 to 1 mm in multiple-velocity step tests, consistent with δ values observed by *Kohli et al.* [2011].

Path 2 is defined by experiments conducted in this study (green and blue traces in Figure 10a) that have higher total displacement, peak velocities and normal stresses than the *Kohli et al.* [2011] experiments. In our experiments the friction during acceleration is similar to that observed in the short displacement (Path 1) experiments of *Kohli et al.* [2011]; the somewhat higher value for V_f in the Path 2 experiments arises from greater acceleration (50 m/s^2 compared to 10 m/s^2). Nonetheless, δ remains approximately the same (Table 1), indicating that the initial weakening results from flash heating in both suites of experiments. At peak velocities, the Path 2 steady-state friction values are lower than the Path 1 values, but are consistent with the overall weakening trend and the results of *Hirose and Bystricky* [2007] on serpentine bare surfaces slid at 1 m/s. During deceleration frictional recovery is delayed to lower velocities than observed in Path 1 experiments, resulting in a larger hysteresis loop (Figure 10b). We suggest that the difference between Path 1 and Path 2 experiments is in part due to the sensitive dependence of V_w on the fault surface temperature (T_{surf}), where $V_w \propto (T_w - T_{surf})^2$; if the difference between the surface temperature and the weakening temperature (T_w) is reduced by a factor of two, a typical value seen in our thermal models, then V_w decreases by a factor of 4 [*Rice*, 2006]. The higher displacements, slip rates and normal stresses

used in Path 2 experiments generate higher surface temperatures, leading to lower values of V_w during deceleration than seen in Path 1 experiments. This is consistent with the observation that in Run 734 performed at 4.9 MPa normal stress, the recovery of the friction coefficient occurs at a higher velocity than in Run 735 performed at 14.9 MPa (Figure 10a). Small patches of melt produced on the slip surface may further delay Path 2 recovery. Conversely, in Path 1 experiments there is not enough time for the surface temperature to significantly increase, thus V_w remains constant, resulting in rapid friction recovery at velocities higher than in Path 2 experiments. In the following section we use 1-D thermal modeling to further explore this hypothesis.

4.2. 1-D Thermal Modeling: Bare Surfaces

To quantitatively explore differences in temperature evolution between the slip surface and asperity contacts in bare surface experiments we employ 1-D thermal models. These models do not consider the latent heat of reaction; nonetheless they allow for a first-order exploration of the processes governing dynamic weakening and frictional recovery. For the bare surface experiments we calculate the surface temperature using the following model (see Appendix D for derivation):

$$T_{surf} = T_0 + \int_0^t \frac{\tau(t')V(t')}{\rho c} \frac{1}{\sqrt{4\pi\alpha_{th}(t-t')}} dt' \quad (1)$$

where T_0 is the initial temperature (21 °C), $\tau(t')$ is time dependent shear stress, $V(t')$ is time dependent velocity, ρc is the effective heat capacity per unit volume, and α_{th} is the thermal diffusivity. The heat capacity and thermal diffusivity for antigorite are 2700 kJ (K m³)⁻¹ and 0.90 mm² s⁻¹ respectively [Osako *et al.*, 2010]. For a given experiment the integral is evaluated numerically using measured values for τ and V . In Figure 11a we plot T_{surf} for three single velocity-step tests and one multiple velocity-step test together

with a projection of the MgO-SiO₂-H₂O phase diagram [Perrillat *et al.*, 2005] and the wet solidus for ultramafic rocks [Till *et al.*, 2012] (see discussion below). Figure 11a shows that peak surface temperatures increase with increasing normal stress from ~200 °C at 5 MPa to > 1600 °C at 97 MPa which is generally consistent with the microstructural observation of glassy material on samples deformed at the highest normal stresses. However, recall that olivine and enstatite were observed in the wear material in all bare-surface experiments (Table 1), even those at the lowest normal stress (e.g., Run 734). The presence of these dehydration products is inconsistent with the 1-D thermal model for the surface temperature. To reconcile this observation with the thermal model, we consider the temperature evolution at the asperity contacts (T_{asp}) with the following flash-heating model (see Appendix D):

$$T_{asp} = T_{surf} + \frac{\tau_c}{\rho c} \left(\frac{vd}{\pi a_{th}} \right)^{1/2} \quad (2)$$

where τ_c is the contact shear stress and d is the contact diameter. Here we have assumed that a contact ceases to exist after it has slipped a distance equal to the contact diameter, and thus the contact lifetime is equal to d/V . Following Kohli *et al.* [2011] τ_c is approximated as 3 GPa (based on indentation hardness measurements [Goldsby and Hirth, 2006] and plasticity data for antigorite [Hilairet *et al.*, 2006]) and d is estimated to be 5 μ m (based on the critical slip distance observed during velocity changes at low slip velocities on identical samples of similar roughness [Reinen *et al.*, 1991]). Figure 11b shows that T_{asp} in all experiments quickly increased above antigorite thermal stability over slip displacements < 1 mm. The total displacements at which the onset of weakening was observed (stars in Figure 11b) is coincident with predicted antigorite dehydration reactions and generally consistent with the flash weakening temperature

inferred by *Kohli et al.* [2011] (~ 600 °C). Recall that δ is the displacement that occurs during acceleration between V_f and V_w , where V_w is 0.1 m/s. So the total displacement prior to weakening is simply δ + slip that occurs from the onset of acceleration to 0.1 m/s which is on the order of 200 to 300 μm . Furthermore, using experimental parameters from *Kohli et al.* [2011], equation 2 predicts temperatures consistent with their observation of talc in the wear material. In contrast, our experiments have over an order of magnitude larger velocity, which promotes asperity temperatures significantly above the talc stability field, into the olivine and enstatite field, and eventually above the wet solidus, consistent with the observation of olivine and enstatite in the wear material and the glass-like textures and materials on the slip surface (e.g., Figure 7b). The modeled asperity temperature is only valid before the onset of weakening, since we have neglected changes to the contact shear stress and possible latent heat effects that accompany the onset of weakening.

These models also suggest that flash heating is likely causing the initial weakening observed in Path 2 experiments, similar to the Path 1 (Figure 10b); moreover, the fact that both samples from Runs 734 and 735 (performed at normal stresses of 4.9 MPa and 14.9 MPa, respectively), weaken at about the same slip rate of 0.1 m/s (Figure 6a), suggest that the initial weakening is independent of normal stress, consistent with the flash heating mechanism [*Rice*, 2006]. However, with increasing displacement/deformation time the T_{surf} increases at a rate dependent on the shear stress and velocity. For high enough slip and velocity, weakening due to flash heating will be associated with asperity melting, which will lead to the generation of melt patches with increasing power dissipation and eventually lead to bulk melting of the entire fault

surface [Tsutsumi and Shimamoto, 1997; Hirose and Shimamoto, 2005]. A first order fit of the friction data from Run 735 (ATG) with the flash weakening model given in Rice [2006], such that T_{surf} is allowed to increase using equation 1 (Figure 10c), demonstrates that an increase in surface temperature alone cannot explain the delayed recovery during deceleration. We therefore conclude that the delayed frictional recovery shown in Path 2 is in part a manifestation of this transition from flash weakening to bulk melting.

4.3. 1-D Thermal Modeling: Gouge

To quantitatively explore differences in temperature evolution between a uniformly shearing gouge zone and asperity contacts between clasts within the zone we modify the 1-D thermal modeling to account for the thickness of the deforming zone. Similar to bare surface models we do not consider the latent heat of reaction; nonetheless these models allow for a first-order exploration of the processes governing dynamic weakening in gouge. To calculate the temperature within the gouge samples (T_{goug}) we set $y = 0$ in equation (D5) to find:

$$T_{goug} = T_0 + \int_0^t \frac{\tau(t')v(t')}{\rho c \sqrt{2\pi}} \frac{1}{\sqrt{W^2 + 2\alpha_{th}(t-t')}} dt', \quad (3)$$

where W is the half width of the deforming zone. T_{goug} is the maximum temperature in the deforming zone (at $y = 0$), where shearing is most intense; repeating the analysis using the average temperature in the deforming zone did not significantly alter our results. Figure 11c shows the predicted temperatures for three single velocity-step experiments with a peak velocity of 1 m/s (Runs 820, 745 and 823), one single velocity-step experiment with a peak velocity of 0.1 m/s (Run 835) and one multiple velocity-step experiment with a peak velocity of 4 m/s (Run 824b). We use a deforming zone thickness of 150 μm (i.e., $W = 75 \mu\text{m}$), based on our microstructural observations (see

Figure 9). Repeating the analysis for thicknesses ranging from 50 to 500 μm resulted in negligible differences ($\sim 25^\circ\text{C}$). The model predicts that gouge temperatures may become high enough to dehydrate serpentine for all experiments at $\sigma_n > 5\text{ MPa}$, which is consistent with XRPD data, except for Run 835 that did not contain dehydration products. Additionally, the model predicts temperatures high enough to produce melt in Run 823, consistent with the melt textures along the slip surface. However, if dynamic weakening occurs for $T > 600^\circ\text{C}$, the model is inconsistent with the observed total weakening distances in the all experiments. For example, Runs 820 and 745 dynamically weakened after $\sim 200\text{ mm}$ and 5 mm of slip respectively whereas the model predicts that the temperatures in both runs should be $\sim 200^\circ\text{C}$ for the given slip, far below the critical temperature.

To reconcile this inconsistency we explore the role of flash heating within the gouge zone using the relationship suggested by *Beeler et al.* [2008], which accounts for the width of the deforming zone. In this case, the flash-heating model (D7) is modified to:

$$T_{asp} = T_{goug} + \frac{\tau_c}{\rho c} \left(\frac{V(g/w)d}{\pi \alpha_{th}} \right)^{1/2}, \quad (4)$$

where g is the grain size and w is the thickness of the deforming gouge layer. Again, we assume the deforming gouge layer thickness is $\sim 150\text{ }\mu\text{m}$ (i.e., $w=2W=150\text{ }\mu\text{m}$) based on the width of ultra-fine grained zones in our experiments. The initial grain size (g) within this deforming zone is less obvious. The grain size prior to deformation (i.e., the grain size of the starting material) will undoubtedly be somewhat comminuted during initial slip as strain becomes more concentrated into the $\sim 150\text{ }\mu\text{m}$ shear zone. As demonstrated by *Di Toro et al.* [2013], once strain becomes highly localized very little additional

deformation occurs outside the localized zone, thus the material adjacent to the localized zone should preserve the grain size at the onset of localization. In our experiments the grain size of the material adjacent to the ultra-fine grained zones was $\sim 15 \mu\text{m}$ suggesting a g/w ratio of ~ 0.1 . In equation 4 we assume $(g/w) = 0.1$ while all other physical constants remain the same as previously assumed in equation 2. Figure 11d shows that the asperity-scale temperature increase varies dramatically with varying normal stress and velocity, and that the magnitude of the temperature rise for a given displacement is significantly lower than in equivalent bare-surface experiments (Figure 11b). Similar to bare surface experiments, there is a strong correlation between the onset of dynamic weakening (stars, Figure 11b) and model temperatures $> 600^\circ\text{C}$, suggesting that dynamic weakening is caused by flash heating in gouge. However the dynamic weakening in gouge occurs after larger displacements and at higher velocities than bare surfaces because strain is more distributed. Remarkably, the gouge flash heating model predicts that Run 824b and 823 should have similar asperity-scale temperatures with slip despite having different imposed normal stresses and velocity profiles, consistent with the total weakening distances observed in both runs (Figure 11d). Our conclusion that dynamic weakening in the gouge is promoted by flash heating is also supported by comparison of the friction data (during acceleration) with the model illustrated in Figure 10d.

Flash heating has also been observed in HVF experiments on carbonate gouge. In experiments conducted at $\leq 2 \text{ MPa}$ *De Paola et al.* [2011] and more recently *Mitchell et al.* [2013] showed CO_2 degassing after only a few hundred microns of slip suggesting that flash heating was occurring. In higher normal-stress experiments (8.5 MPa), *Smith et al.*

[2013] demonstrate that dynamic weakening in carbonate gouge initiates at sliding displacements of (100-150 mm), similar to the behavior seen in our experiments.

The application of phase equilibria to high-velocity frictional processes remains a challenge due to short reaction times, high energy input rates and dynamic nature of the physical parameters under which reactions occur. Based on the identification of crystalline reaction products in wear material, in conjunction with the thermal models, we conclude that reaction kinetics must be rapid enough for phase transformations to occur within the lifetime of an asperity (few milliseconds). In the short-displacement experiments of *Kohli et al.* [2011], the consistency between the modeled asperity temperatures, XRPD observation of talc and the predicted reaction temperature suggests near-equilibrium phase relationships at pressures similar to the asperity normal stress are applicable in HVF systems. For material not in contact at asperities, the reaction pressure will be set by the pore fluid pressure (which is lower than the asperity pressure), thus generally reducing the reaction temperature in these zones. Following this logic, in Figure 11 we projected phase boundaries through ambient pressures for T_{surf} and T_{goug} plots and through 5-6 GPa for asperity temperature plots (based on our assumption that the contact shear stress is 3 GPa and friction is ~ 0.6 prior to weakening). Nonetheless, we acknowledge that considerable uncertainty remains regarding the magnitude, spatiotemporal variability of pressure at the asperity scale.

Other textural observations suggest the application of equilibrium phase boundaries is more complicated in HVF systems. Recall that in antigorite bare surface and gouge experiments we observed melt-like textures directly on the slip surfaces (e.g., Figures 7b, 7d and 9j). These textures suggest the reaction $Atg \rightarrow \text{melt} + H_2O$, which is

not consistent with equilibrium thermodynamics. Thus, rapid heating apparently promotes reaction overstepping.

4.4 Comparisons of gouge and bare surfaces at higher normal stress

With increasing normal stress the high-velocity friction behavior of gouge approaches that of bare surfaces. This trend is demonstrated by both δ and μ_{nss} values. As discussed above, values of δ characterize the onset of dynamic weakening. In gouge, values of δ decrease with increasing normal stress while values for bare surfaces remain approximately constant (e.g., Figure 11d and Table 1). Similarly, values of μ_{nss} for gouge decrease with increasing normal stress while values for bare surfaces remain approximately constant (e.g., Figure 4a). A linear approximation to the μ_{nss} data for gouge predicts that both gouge and bare surfaces would have the same values of μ_{nss} at normal stresses ~ 60 MPa or larger (as shown by dashed line in Figure 4a) and data for both would be nominally independent of normal stress. A normal stress of 60 MPa corresponds to a depth of ~ 5 km along an Andersonian normal fault at hydrostatic conditions [e.g., *Cowie et al.*, 1993]. It remains unclear if bare-surface samples would actually strengthen if wear material was allowed to accumulate during slip; in our experimental assembly, wear products are flung out of the assembly, leaving a minimal thickness of wear debris.

4.5 Geophysical Implications

The results of this study have potential implication for earthquakes in geologic locations that contain serpentine, such as within oceanic transform faults [e.g., *Francis*, 1981], oceanic detachment faults [e.g., *MacLeod et al.*, 2002] and some locations along the San Andreas fault [e.g., *Moore and Rymer*, 2007]. However, all natural faults contain

a layer of gouge material produced during a seismic rupture propagation even in initially cohesive fault rocks [Reches and Dewar, 2005]. Thus, some of the first-order observations found in this study may be applicable for all nominally dry faults as those reproduced in our experiments (room-humidity).

For serpentine-rich fault zones at low normal stresses (<60 MPa) under nominally dry conditions, dynamic frictional-weakening will be delayed to longer slip by the presence of unconsolidated material possibly resulting in smaller slips during earthquakes. For ruptures that propagate into shallow unconsolidated fault-patches, less seismic energy will be emitted due to smaller static- and dynamic-stress drops. These interpretations complement hypotheses derived from slow velocity experiments on gouge, which have been interpreted to promote the lack of shallow seismicity in some fault zones [e.g., Marone and Scholz, 1988]. For large slip events (>5 m) gouge likely has no ability to subdue radiated seismic energy; previous studies have demonstrated for long displacements (5-25 m) under room-humidity conditions, friction values will decay to ~ 0.1 for phyllosilicate-rich gouges [e.g., Kitajima *et al.*, 2010; Mizoguchi *et al.*, 2007]. We suspect these friction trends are also applicable in highly permeable fluid-saturated faults, with the added complication that water cools the asperity contacts rendering the flash-heating weakening mechanism less efficient than in room-humidity [Violay *et al.*, 2014]. In water saturated faults with low permeability, thermal pressurization may occur adding further complication [e.g., Noda *et al.*, 2009]. In such cases it is not clear how the presence of gouge may affect dynamic weakening during seismic slip; no machine to date can simulate these conditions. At normal stresses > 60 MPa the presence of

unconsolidated serpentine within the fault may have no direct effect on the onset of dynamic weakening or steady state friction values.

5. Conclusions

Our work finds significant frictional differences between the serpentinite bare surfaces and gouge at low normal stresses (<22 MPa). We demonstrate that the frictional behavior is strongly normal stress dependent in gouge while relatively normal stress invariant in bare surfaces. Extrapolation of our data suggests that the behavior of antigorite gouge will approach that of bare surfaces at normal stresses ≥ 60 MPa (~ 5 km depth). We thus infer the presence of gouge will alter the weakening properties of shallow sections of serpentine-rich faults, but have little effect at greater depths. Using 1-D thermal modeling, X-ray diffraction and microstructural analysis, we constrain the effects of velocity, normal stress, shear heating, strain localization and dehydration reactions on frictional weakening. We show that the evolution of friction for a given slip event on either bare surfaces or gouge zones is dependent on the evolution of temperature at both the asperity-scale and surface-scale and we show that both scales need to be modeled in order to reconcile experimental data. We conclude that flash heating is the primary process causing initial weakening in bare surfaces. Flash heating also occurs in gouge, however because strain is more distributed, dynamic weakening occurs at higher velocities and after larger displacements than in bare surfaces experiments. We find values of friction in LIZ gouge have longer weakening distances than ATG gouge but generally have similar dynamic weakening trends. Finally, we observe slip-generated dehydration products and melt textures in both bare-surface and gouge samples. These

mineral and textural signatures likely form in natural serpentine-rich faults, the presence of which would indicate seismic slip.

Appendix A: Smoothing data

High frequency noise was removed from all data using a fast-fourier-transform (FFT) smoothing filter. Figure A1 shows filtered or smoothed friction data from three representative experiments (black, green and blue lines) and corresponding unsmoothed (raw) data (gray lines).

Appendix B: X-ray powder diffraction methods

Twenty-six X-ray powder diffraction analyses were conducted at the Geosciences Department at the University of Padua used a Panalytical θ - θ diffractometer (Cu radiation) equipped with a long, fine-focus Cu X-ray tube (operating at 40kV and 40mA), sample spinner, Ni filter and a solid-state detector (X'Celerator). The system optics consist of a fixed $\frac{1}{2}^\circ$ divergent slit and 1° antiscatter slit on the incident beam path and soller slits (0.04 rad) on incident and diffracted beam paths. The powders were mounted on a 32-mm (internal diameter) circular sample holder. Scans were performed over the 2θ range 3 - 80° with a virtual step size of 0.017° in 2θ and a counting time of 100 s/step. Phase identification and semi-quantitative analysis were performed using the software package X'Pert HighScore Plus; the phase identification was confirmed by comparison with the reference pattern database Panalytical-ICSD (Inorganic Crystal Structure Database). One additional XRPD analysis was conducted at Brown University on sample 834 using a Bruker D-8 Advance diffractometer with DaVinci system, a Cu X-ray tube operating at 40 kV and 40 mA and a Bruker Vantec-500 (Xe-CO₂ gas filled) detector with a 13.5 cm diameter window set at 20 cm from the goniometer center and Ni filter.

The scans were performed over the 2θ range 25-80° with a virtual step size of 0.25° in 2θ and a counting time of 60s /step. The phase identification was performed using the software package Diffrac.Eva by Bruker and confirmed by comparison with the ICDD (The International Centre for Diffraction Data).

Appendix C: Area correction for single velocity-step bare-surface experiments

In the suite of 1 m/s experiments on ATG bare surfaces (Runs 736a to 736h), one sample was used for eight consecutive slip cycles, with 1 m of slip per cycle. The applied normal force was increased after each cycle from 6.2 kN during the first cycle up to 53 kN during the last cycle. This force correlates with an increase in nominal normal stress from ~5 to 40 MPa assuming the initial nominal area of the slip surface (~12.5 cm²) remained constant. However, during each slip cycle the area progressively decreased because the sample on the stationary side became progressively wedge shaped due to wear during sliding (Figure C1a). Wear of the sample on the opposing rotary side resulted in significant loss of material normal to the fault, but the surface area remained approximately constant. The final slip surface area (dashed orange region in Figure C1c) was determined via imaging software to be 5.38 cm², indicating that the actual normal stress was 96.6 MPa during the last cycle.

We constructed a simple model to estimate the area of the slip surface after each slip cycle. The model is constrained by the final slip surface outline in plane view, the initial and final area of the slip surface, the mass of wear material collected after each slip cycle and the axial shortening. The axial shortening was monitored with a linear variable differential transformer (LVDT) and the wear material was collected in a foil tray located under the sample. In Figure C1d the cumulative mass of collected wear material and

cumulative axial shortening are plotted for all eight slip cycles; both data sets show a similar non-linear increase with increasing normal force. The final shape of the slip surface is modeled as a wedge-shaped quadrilateral (in cross-section) on one side (Figure C1e) and as a migrating flat surface on the other side (Figure C1e). The effective annular width of the slip surface (y) is calculated from the final measured surface area (Figure C1c). The thickness of the stationary sample (Z_1) remained approximately constant, while the thickness of the rotary sample (Z_2) decreased in accord with axial shortening. The evolution of the cross-sectional area of the top sample is calculated using mass balance and the change in the annular width (λ); values of λ are estimated by assuming that the eroded angle remains constant. The total volume of wear calculated from this model is $\sim 6.5 \text{ cm}^3$ (purple circle in Figure C1f). The total volume of collected wear (collected mass/density of antigorite) was somewhat less than the value determined by mass balance (blue diamond in Figure C1f), indicating that $\sim 60 \%$ of the total wear material was recovered. We ignore changes in mass due to frictional-heating-induced metamorphism. The black line in Figure C1f shows cumulative wear by assuming that 60% of the total wear material was collected after each slip pulse. This assumption is reasonable because the collection tray did not capture all particles expelled from the sample. We then use the cumulative collected wear in conjunction with the LVDT displacement to calculate the value of λ that is consistent with the estimated volume of wear material produced after each slip cycle (green triangles in Figure C1f). Finally, we use λ to calculate the surface area. The modeled surface area values were used to correct the recorded normal stress and shear stress values for each slip cycle (Figure C1g).

Appendix D: Thermal Model

To model the temperature evolution within the deforming zone we use the one-dimensional heat equation,

$$\frac{\partial T}{\partial t} = \frac{\tau \dot{\gamma}}{\rho c} + \alpha_{th} \frac{\partial^2 T}{\partial y^2}, \quad (D1)$$

where t is the time since slip began, y is the distance from the center of the deforming zone, τ is the shear stress, $\dot{\gamma}$ is the strain rate, α_{th} is the thermal diffusivity, and ρc is the effective heat capacity per unit volume. The initial conditions are $T = T_0$ at $t = 0$, which models the ambient laboratory conditions before deformation begins. For the boundary conditions we choose $T \rightarrow T_0$ as $y \rightarrow \pm\infty$. These boundary conditions ignore the presence of the metal gouge holder, which will have a higher thermal diffusivity than the gouge. However, for the typical experiment durations of about one second it is likely that thermal diffusion through the gouge holder has a small effect, justifying our choice of boundary conditions.

The frictional heating in the deforming zone is controlled by the shear stress and strain rate. *Rice* [2006] argued that unrealistically high accelerations are required to make inertial effects important within the gouge layer, and thus the shear stress should be constant throughout the deforming zone. *Platt et al.* [2014] used numerical simulations to show that this assumption is valid for the normal stresses considered here. We assume that the deforming zone has a gaussian shape

$$\dot{\gamma} = \frac{V}{W\sqrt{2\pi}} \exp\left(-\frac{y^2}{2W^2}\right) \quad (D2)$$

where W is the half-width of the deforming zone. A better approach would be to model the physical processes driving strain localization within the gouge, but this is beyond the scope of this paper.

For the shear stress and strain rate profiles justified in the previous paragraph we can solve equation (D1) using a Green's function approach, leading to the solution,

$$T(y, t) = T_0 + \int_0^t \int_{-\infty}^{+\infty} \frac{\tau(t')v(t')}{\rho c W \sqrt{2\pi}} \exp\left(-\frac{y'^2}{2W^2}\right) G(y - y', t - t'; \alpha_{th}) dy' dt', \quad (D3)$$

where the Green's function is,

$$G(y - y', t - t'; \alpha_{th}) = \frac{1}{\sqrt{4\pi\alpha_{th}(t-t')}} \exp\left(-\frac{(y-y')^2}{4\alpha_{th}(t-t')}\right). \quad (D4)$$

The integral over y' can be done exactly leading to,

$$T(y, t) = T_0 + \int_0^t \frac{\tau(t')v(t')}{\rho c \sqrt{2\pi}} \frac{1}{\sqrt{W^2 + 2\alpha_{th}(t-t')}} \exp\left(-\frac{y^2}{2W^2 + 4\alpha_{th}(t-t')}\right) dt'. \quad (D5)$$

This expression was previously derived in *Andrews* [2002] and was used to model thermal pressurization during seismic shear.

To find the temperature evolution of the sliding surface for the bare surface experiments we set the deforming zone thickness equal to zero in equation (D5). This is equivalent to solving using a Green's function for a half-space heated by a flux at the boundary, and leads to the solution for the surface temperature,

$$T_{surf} = T_0 + \int_0^t \frac{\tau(t')v(t')}{\rho c} \frac{1}{\sqrt{4\pi\alpha_{th}(t-t')}} dt'. \quad (D6)$$

Equation (D6) can be repurposed to calculate the asperity temperature using the typical flash heating model by setting the shear stress equal to the contact shear stress τ_c , the slip velocity equal to the current value from the experiment, and the initial temperature T_0 equal to the current temperature on the sliding surface. The assumptions of constant velocity and shear stress allow us to evaluate the integral in equation (D6) to find the maximum asperity temperature;

$$T_{asp} = T_{surf} + \frac{\tau_c}{\rho c} \left(\frac{vd}{\pi\alpha_{th}}\right)^{1/2}, \quad (D7)$$

where d is the contact (i.e., asperity) diameter.

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Table 1. Experimental conditions and mechanical data from 27 high-velocity friction experiments. Materials: ATG, antigorite-rich serpentinite; LIZ, lizardite-rich serpentinite; bare surface (BS) and gouge (G). Experimental conditions: SS_X, single velocity-step test at X m/s; MS_X_Y, multiple velocity-step test with initial velocity X m/s and stepped velocity Y m/s. NA, not applicable-indicates data was not recorded or measured. NW, experiment had no frictional weakening. Parentheses indicate standard deviation. X-ray powder diffraction (XRPD) data is reported in alphabetical order for observed mineral phases, antigorite (Atg), augite (Aug), clinochlore (chlorite, Chl), enstatite (En), lizardite (Liz), magnesite (Mgs), magnetite (Mt) and olivine (Ol).

Figure 1. Sample assembly for bare-surface and gouge experiments. (a) Photograph of rotary side of bare-surface sample holder with serpentinite sample prior to deformation. (b) Schematic of bare surface experimental assembly. (c) Photograph of gouge holder (rotary side) loaded with serpentinite powder. (d) Schematic of gouge holder [modified after *Smith et al.*, 2013]. (e) Enlargement of 1d showing gouge sample compartment (green) and lubricated metal-metal rotary contacts (red). Dashed line indicates typical location of strain localization within the gouge.

Figure 2. (a) Results from three multiple velocity-step experiments; 3 mm/s for ~6 cm of displacement, acceleration to ~4.5 m/s and deceleration to rest. The approximate thermal-weakening distance (D_{th}) and slip-weakening distance (D_w) are indicated for Run 735 (see text for details). Stars indicate the displacement at which the friction rapidly decreases; the corresponding velocity (fall-off velocity) is shown in 2b. (b) Frictional stick-slip instabilities observed in Run 735 at 3 mm/s. (c) The imposed velocity profiles for experiments shown in 2a. Note that the acceleration is very similar in both gouge and bare surface experiments.

Figure 3. Results from single velocity-step experiments with peak velocities of 1 m/s on (a) antigorite bare-surfaces, (b) ATG gouge, and (c) LIZ gouge. A representative velocity profile is shown for each suite of experiments (grey line). Large wavelength oscillations in gouge friction are caused by inconsistencies in gouge thickness (see Methods section).

Figure 4. Normal stress dependence on (a) nominal steady-state and minimum friction values (LIZ gouge), (b) thermal weakening distance, and (c) the fall-off velocity for multiple velocity-step and single velocity-step experiments. Run numbers are indicated in parentheses.

Figure 5. Velocity dependence of antigorite gouge; inset shows velocity profile for corresponding experiments. Run numbers are indicated in parentheses.

Figure 6. Hysteresis and velocity dependence of (a) ATG bare surfaces and (b) ATG gouge. Lines trace the evolution of friction from the onset of acceleration to the completion of deceleration. Run numbers are indicated in parentheses. In Runs 734, 735, and 824b, the velocity was accelerated from 3 mm/s from which they were sheared for 6 cm. Experiment 834 was accelerated from rest with no prior deformation. Symbols indicate nominal steady state values (μ_{nss}); corresponding Run number is noted in black.

Figure 7. Microstructures of antigorite bare-surfaces. (a) Photomicrograph showing dark striations on slip surface at low normal stress; Run 727, $\sigma_n=8.8$ MPa. (b) SEM micrograph of dark band from sample from Run 727 showing melt-like tendrils and bulbous features on the slip surface. (c) Photomicrograph showing glass-like band on cut section of the slip surface at higher normal stress; Run 733, $\sigma_n=19.5$ MPa. (d) Magnified region from 6c showing glassy luster and transparent nature of the material. (e) SEM micrograph from polished glassy surface shown in 7d. (f) Enlargement from 7e showing gray ultra-fine grained matrix with bands of nm-scale iron-rich minerals that are too fine-grained to resolve.

Figure 8. Comparison of XRPD results from 3 single velocity-step experiments (a) and 2 multiple velocity-step experiments (b); the applied normal stress for each experiment and run number is noted in the key.

Figure 9. Microstructures of antigorite and lizardite gouges (all BSE-SEM micrographs with exception of Fig. 9g). The SEM micrographs show cross-sectional profiles; samples are oriented such that top surface was adjacent to non-rotary disk, however in all cases the section of the gouge zone closest to the stationary side was not recovered. (a) Run 835 (ATG) deformed at 0.1 m/s; orientation of shearing is not known, $\sigma_n=13.3$ MPa. (b) Enlargement of granular fine-grained material within localized zone from 9a (black arrow). (c) Run 821 (ATG) deformed at 1 m/s; sample is cut perpendicular to shearing direction, $\sigma_n=11.8$ MPa. (d) Enlargement of ultra fine-grained material in 9c (black frame box). (e) Run 746 (LIZ) deformed at 1 m/s; sample is cut parallel to shearing direction, $\sigma_n=8.5$ MPa. (f) Enlargement of brecciated clast within localized zone in 9e. (g) Photomicrograph of slip surface from Run 823 deformed at 1 m/s showing vitreous and striated dark material and whitish streaks; rotary direction is indicated with black arrow, $\sigma_n=22.4$ MPa. (h) Enlargement of dark glassy material shown in 9g; sample is cut perpendicular to shearing direction. (i) Enlargement of whitish streak in 8g showing vesicular ultra-fine-grained material along the slip surface; sample is cut along black dashed line. (j) Enlargement of 9i showing irregular-shaped vesicles. (k) Run 826 deformed at 1 m/s showing an ultra-fine-grained zone ‘flowing’ into the brecciated zone; sample is cut parallel to shearing direction, $\sigma_n=19$ MPa. (l) Enlargement of 9k (dashed box) showing flow structure.

Figure 10. Interpretation of friction hysteresis in high speed experiments on serpentinites. (a) Comparison of ATG bare-surface friction data from this study with results from short displacement experiments from *Kohli et al.* [2011] (see text for

discussion). Parentheses indicate the Run number. (b) Schematic model illustrating friction hysteresis during an earthquake slip event (EQ) for serpentinite bar-surfaces (solid lines) and dry gouge (dashed lines) (see text for discussion). (c) Predicted friction profile (black curve) using flash weakening model [Rice, 2006] for bare surface sample (Run 735); equation (1) is used to model surface temperature increase during slip, which is then inserted into the formula for V_w from Rice (2006). Model shows that the deceleration friction path will be offset to lower velocities and the measured friction data (green trace) only partially fits the model during deceleration. (d) Predicted friction profile (black curve) during acceleration using flash weakening model [Rice, 2006] for gouge sample (Run 834); the temperature of the deforming zone is assumed constant.

Figure 11. Results from 1-D thermal modeling with projected antigorite phase diagram (see text details). (a) Sliding surface temperatures in four ATG bare-surface experiments; the applied normal stress and run number are noted by the corresponding temperature profile. (b) Asperity contact temperatures for bare surface samples modeled in 11a; line colors correspond with 11a. (c) Shear zone temperatures in five ATG gouge experiments assuming a 150 μm -wide shear zones. (d) Asperity contact temperatures within deforming gouge; line colors correspond with 11c.

Appendix A1. Results from high-frequency noise filtering in three characteristic friction experiments. Parentheses indicate experimental Run number.

Appendix C1. Photographs of deformed bare-surface sample (Run 736) after eight consecutive deformation cycles: (a) the stationary surface and (b) rotary surface. (c) Sequential mechanical erosion reduced the surface area of the non-rotary side by ~57%; yellow lines indicate initial surface area and orange dashed lines outline final slip surface area. (d) Mass of wear material collected after each deformation cycle with the corresponding applied normal force and axial shortening for the given slip cycle. (e) Cross-sectional diagram modeling the final sample shape (green) and material lost to erosion (white). The initial shape is indicated with bold black lines (see text for description of model). (f) Cumulative collected wear (blue diamonds) and the estimated total mass of wear material (black line), assuming 60% of wear material was captured. The modeled volume of wear material (purple circles) was fit to the estimated wear and the corresponding surface area parameter (λ , green triangles). (g) Modeled surface area after each deformation cycle (Runs 736a to 736h) and the calculated normal stress (applied normal force/surface area). The measured initial and final surface areas are indicated with black stars.

Exp. #	Material	Exp. Conditions	Total Disp. (m)	Peak Vel (m s ⁻¹)	Peak Accel., Decel. (m s ⁻²)	Peak τ (MPa)	Avg σ_n (MPa)	μ_{max} ; μ_{min}	D_w (mm)	D_{th} (mm)	δ (mm)	V_f (m s ⁻¹)	XRPD
736a	ATG_BS	SS_1	1.01	NA	41, 31	NA	5.4	0.14	NA	NA	NA	NA	Atg, En, Ol
736b	ATG_BS	SS_1	1.01	1.09	49, 30	5	12.4	0.14	27	4.6	0.4	0.2	Atg, En, Ol
736c	ATG_BS	SS_1	1.01	1.09	50, 30	8.1	20	0.12	82	4.8	0.4	0.2	Atg, En, Ol
736d	ATG_BS	SS_1	1.01	1.09	48, 29	11.4	32.1	0.11	15	2.3	0.4	0.2	Atg, En, Ol
736e	ATG_BS	SS_1	1.01	1.09	47, 30	16	49.8	0.11	21	1.9	0.4	0.2	Atg, En, Ol
736f	ATG_BS	SS_1	1.01	1.09	45, 29	26.2	65.1	0.11	24	1.6	0.4	0.2	Atg, En, Ol
736g	ATG_BS	SS_1	1.01	1.09	49, 30	31.1	75.7	0.11	22	1.6	0.3	0.2	Atg, En, Ol
736h	ATG_BS	SS_1	1.01	1.09	50, 29	35.2	96.6	0.11	9.3	1.5	0.3	0.22	Atg, En, Ol
820	ATG_G	SS_1	0.92	1	47, 49	2.5	4.0 (0.2)	0.51 (0.03)	200	130	NA	NA	Atg, Mgs, Mt
745	ATG_G	SS_1	0.92	1.2	60, 60	5.5	8.3 (0.03)	0.48 (0.02)	240	120	55	1	Atg, En, Mt, Ol
821	ATG_G	SS_1	0.91	1	41, 50	9	11.8 (0.04)	0.52 (0.01)	280	200	60	1	Atg, Mgs, Ol
822	ATG_G	SS_1	0.9	1	35, 46	12.4	17.8 (0.08)	0.4 (0.02)	175	67	40	0.85	Atg, Mgs, Ol
823	ATG_G	SS_1	0.9	1.1	24, 49	16.1	22.4 (0.07)	0.39 (0.02)	162	57	30	0.76	Atg, Mgs, Ol
747	LIZ_G	SS_1	0.92	1.2	65, 63	NA	3.8 (0.06)	*0.50	NW	NW	NW	NW	Chl, En, Liz, Mt, Ol
746	LIZ_G	SS_1	0.92	1.2	60, 62	5.45	8.5 (0.09)	*0.46	NA	NA	150	1	Chl, En, Liz, Mt, Ol
748	LIZ_G	SS_1	0.92	1.2	51, 60	8.3	11.5 (0.6)	*0.52	NA	NA	100	1	Chl, En, Liz, Mt, Ol
825	LIZ_G	SS_1	0.9	1	40, 52	11.7	17.8 (0.3)	*0.34	NA	NA	90	0.92	Aug, Chl, En, Liz, Mt, Ol
826	LIZ_G	SS_1	0.9	1.1	29, 48	14.3	18.7 (0.4)	*0.31	NA	NA	70	0.87	Aug, Chl, En, Liz, Mt, Ol
834	ATG_G	SS_6.5	1.65	6.5	59, 30	8.9	10.9 (0.08)	0.31 (0.02)	180	70	20	1.4	Ol, Pys
835	ATG_G	SS_0.1	0.89	0.14	4, 9	NA	13.3 (0.07)	0.65 (0.02)	NW	NW	NW	NW	Atg, Mgs, Mt
734	ATG_BS	MS_0.003_4.5	0.46	4.3	60, 70	4.4	4.9 (0.04)	0.83 (0.06); 0.12 (0.03)	25	2.5	1	0.3	Atg, En, Mgs, Mt, Ol
727	ATG_BS	MS_0.003_4.5	0.46	4.3	NA	6.6	8.8 (0.01)	0.67 (0.01); 0.09 (0.02)	NA	NA	NA	NA	Atg, En, Mgs, Mt, Ol
735	ATG_BS	MS_0.003_4.5	0.46	4.3	62, 70	10	14.9 (0.4)	0.66 (0.005); 0.09 (0.01)	38	5.9	0.5	0.3	Atg, En, Mgs, Mt, Ol
733	ATG_BS	MS_0.003_4.5	0.46	4.3	NA	16	19.5 (0.3)	0.68 (0.05); 0.09 (0.02)	NA	NA	NA	NA	Atg, En, Mgs, Mt, Ol
824b	ATG_G	MS_0.003_4.5	0.51	4	65, 50	6.9	8.5 (0.2)	0.72 (0.002); 0.35 (0.03)	68	44	33	2	Atg, Mgs, Ol
818	ATG_G	MS_0.003_4.5	0.51	4	63, 49	7.1	7 (0.2)	0.73 (0.002); 0.38 (0.05)	197	183	70	2.6	NA
705	LIZ_G	MS_0.003_5	0.51	4.8	65, 75	5.9	8.5 (0.2)	0.65 (0.003); 0.38 (0.02)	163	131	20	1.6	Chl, Liz, Mt ($\pm$ Ol, En)
Composition	LIZ												Atg, Mgs, Mt
Composition	ATG												

Table 1.

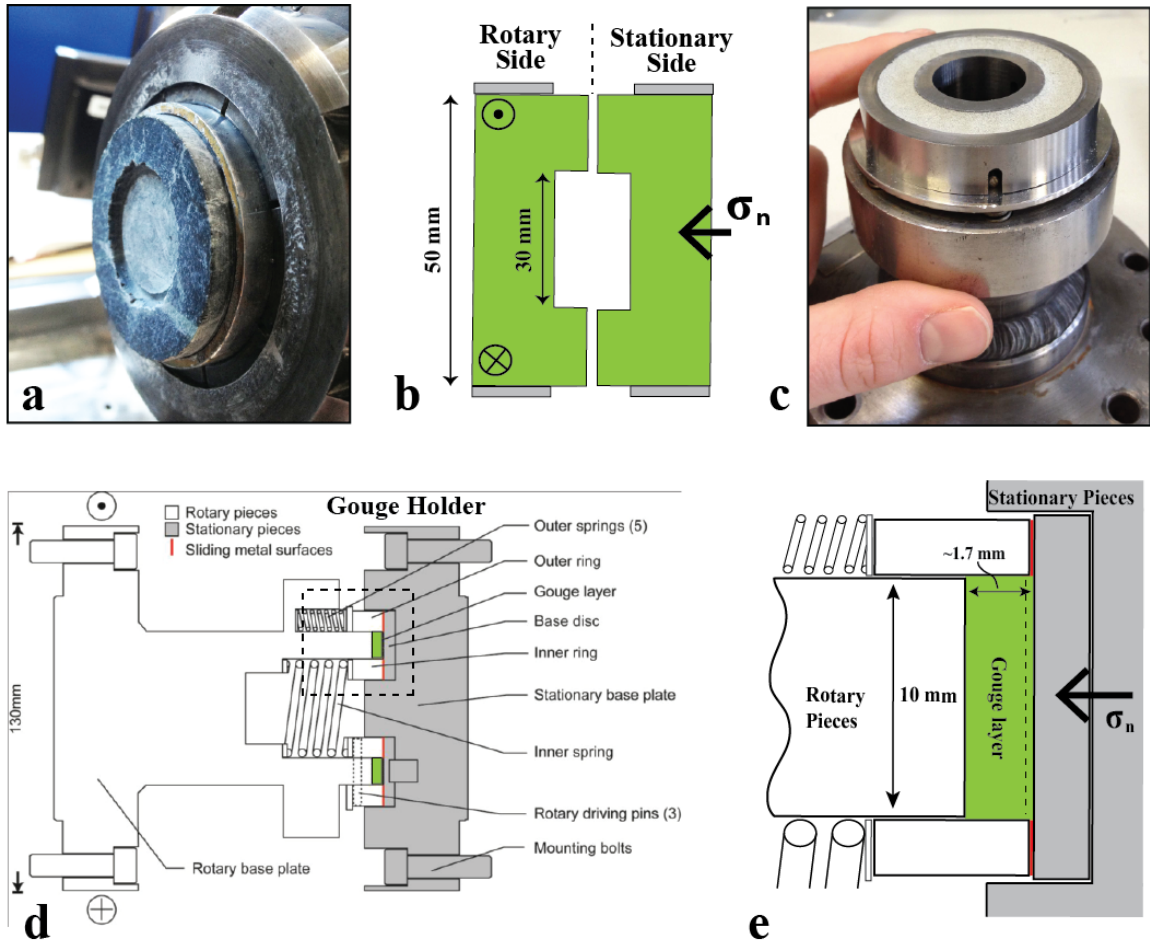


Figure 1.

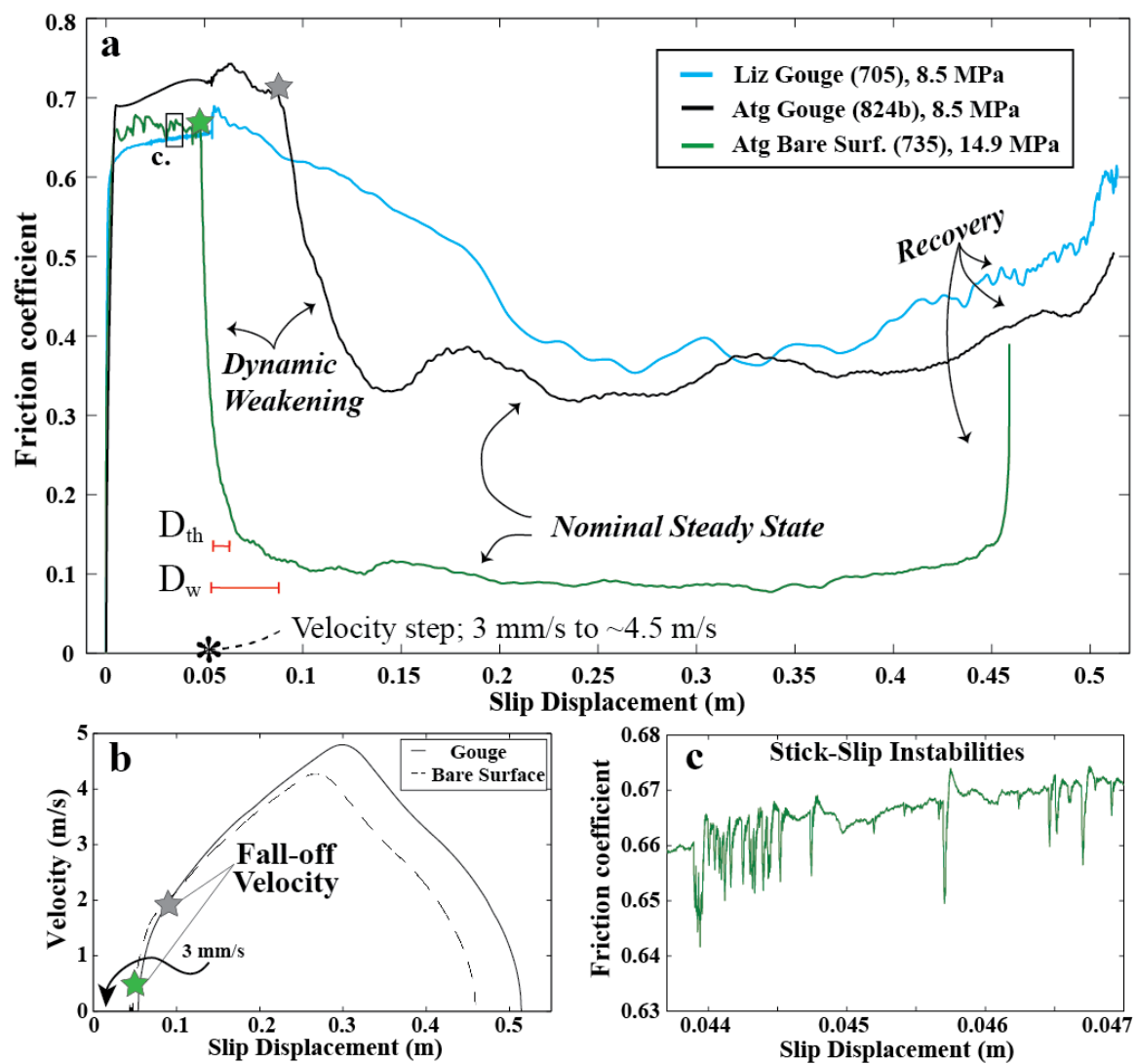


Figure 2

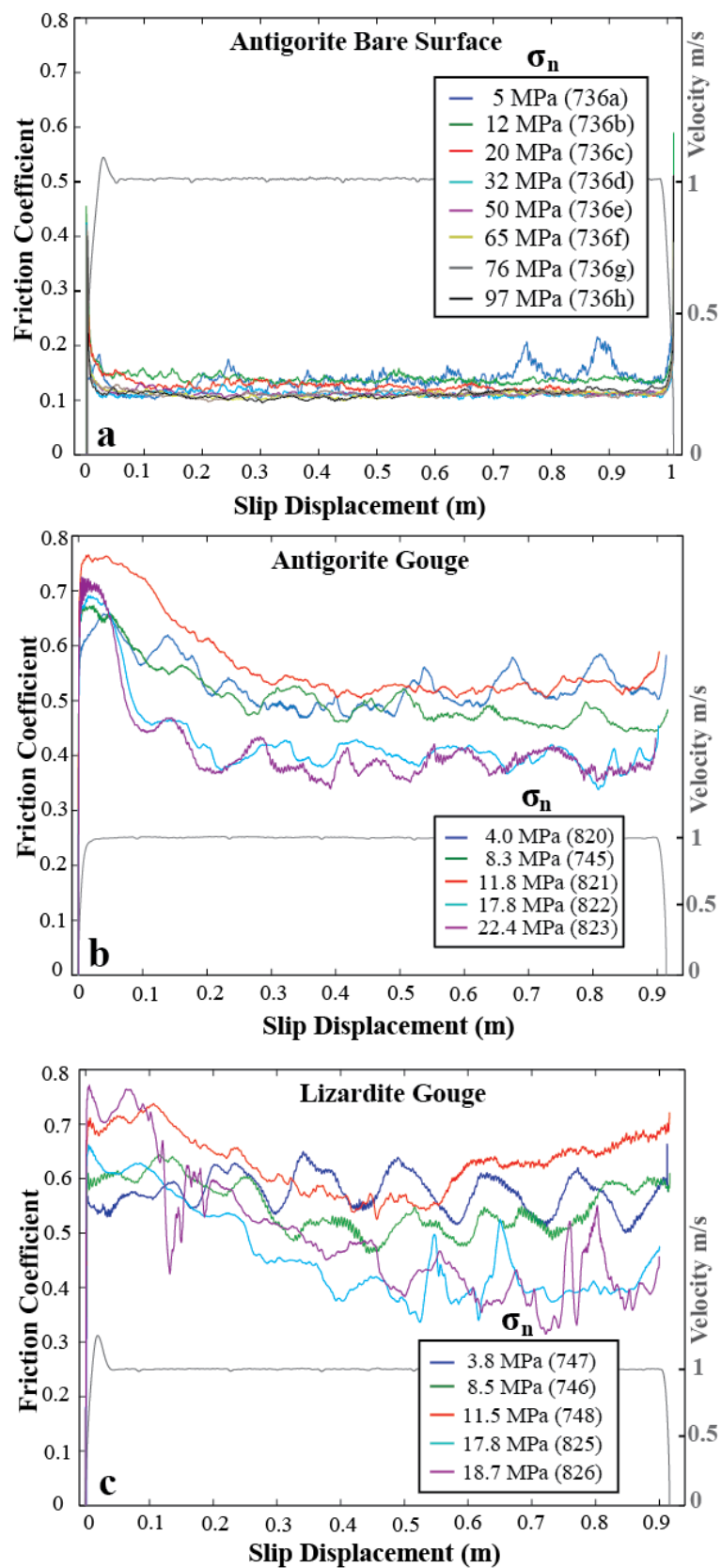


Figure 3

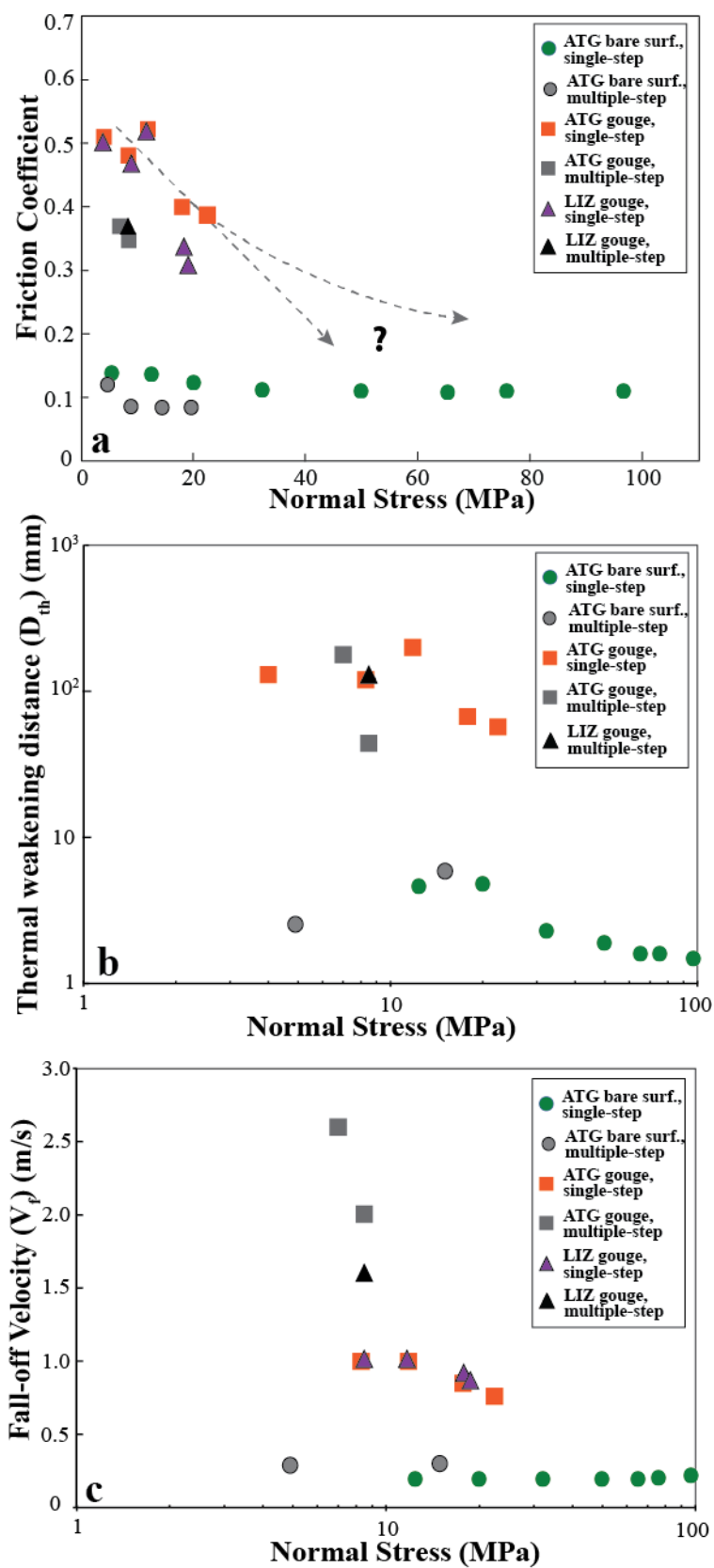


Figure 4

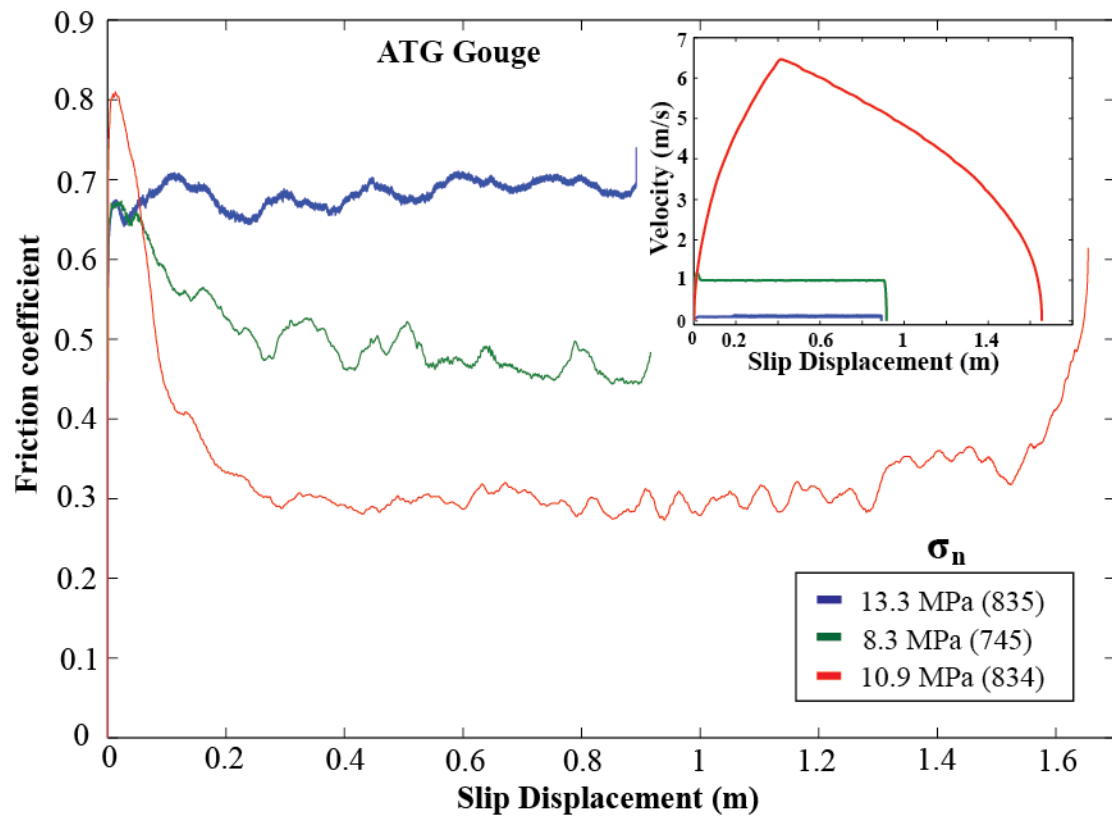


Figure 5

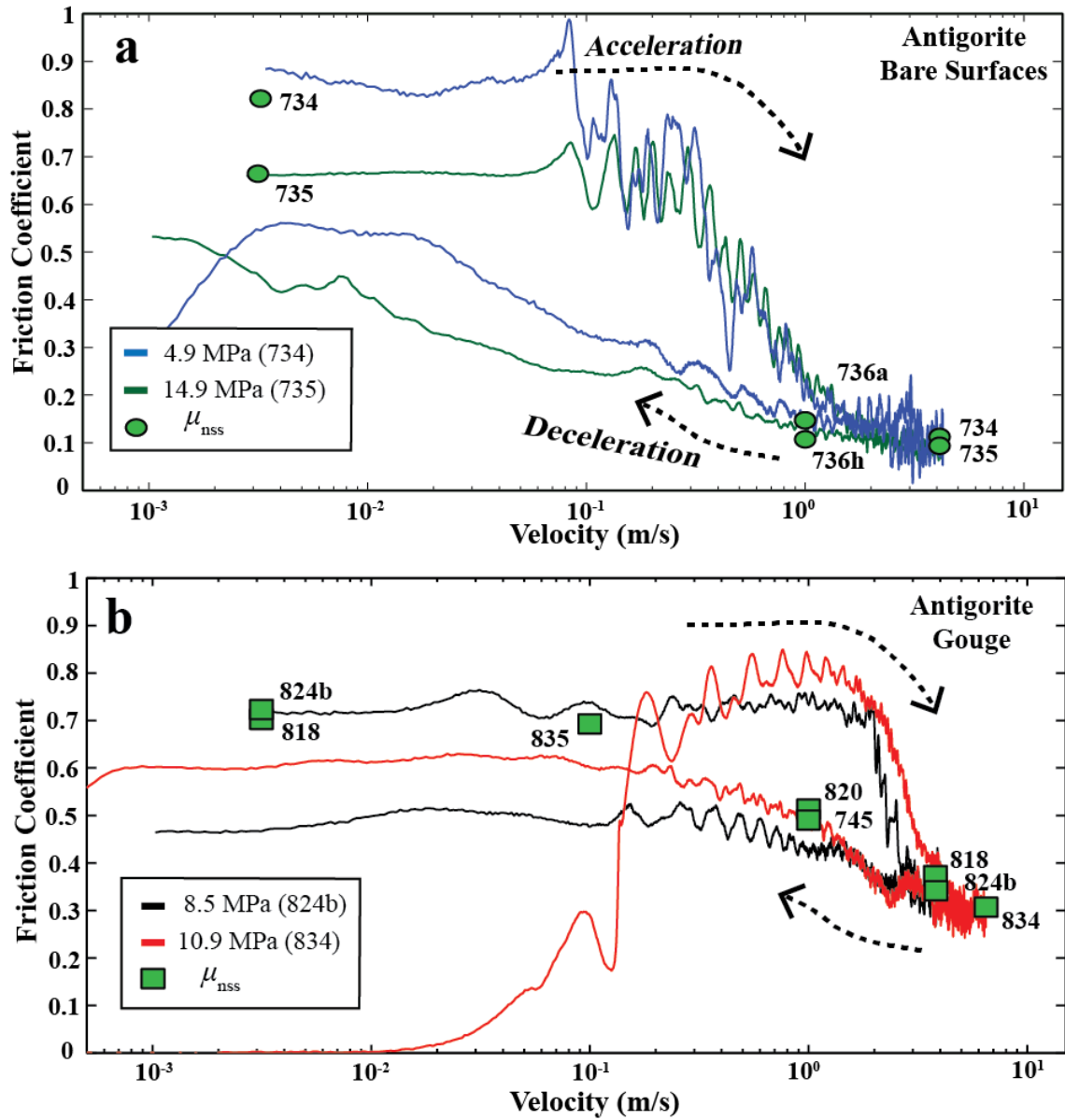


Figure 6

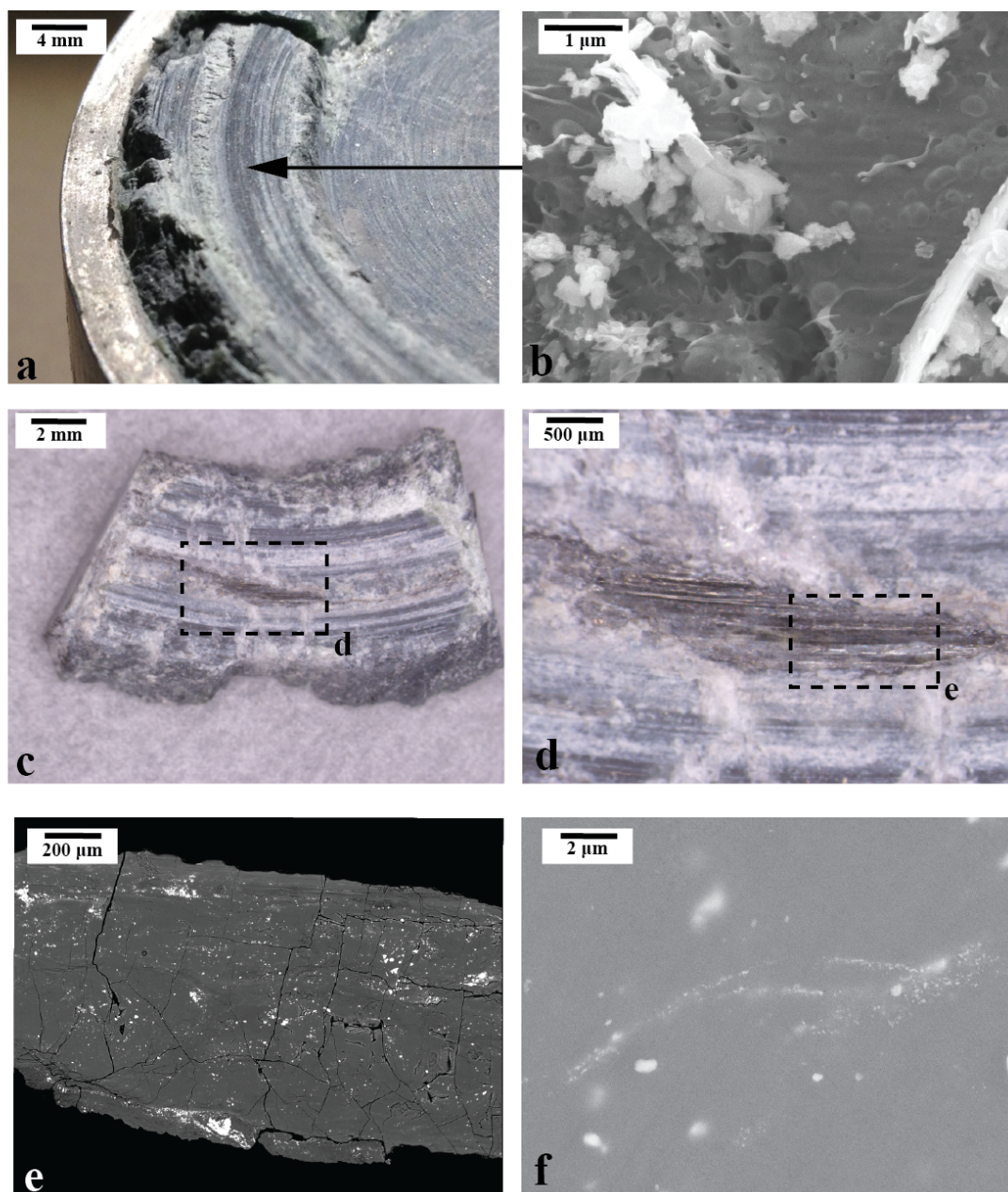


Figure 7

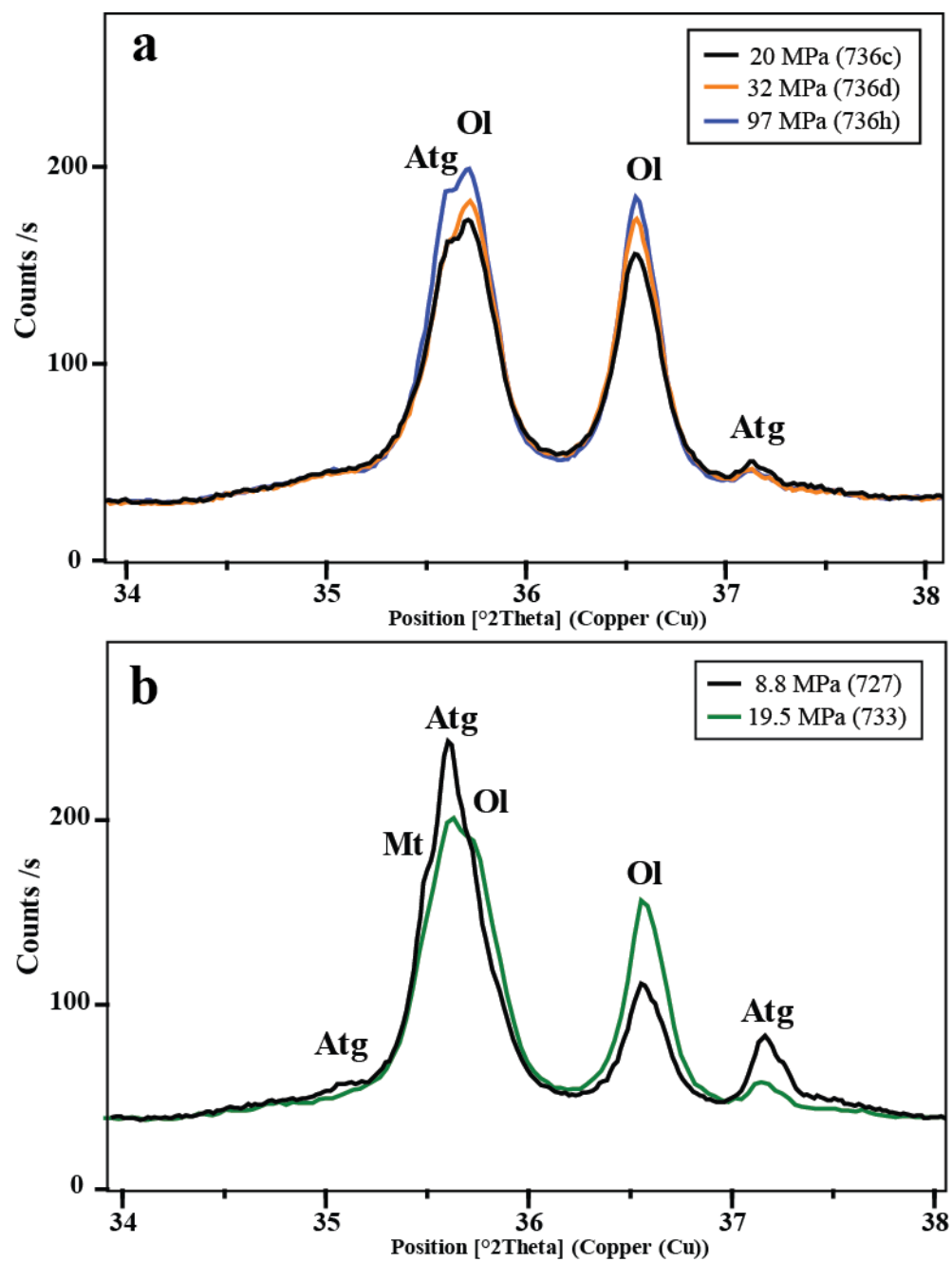


Figure 8

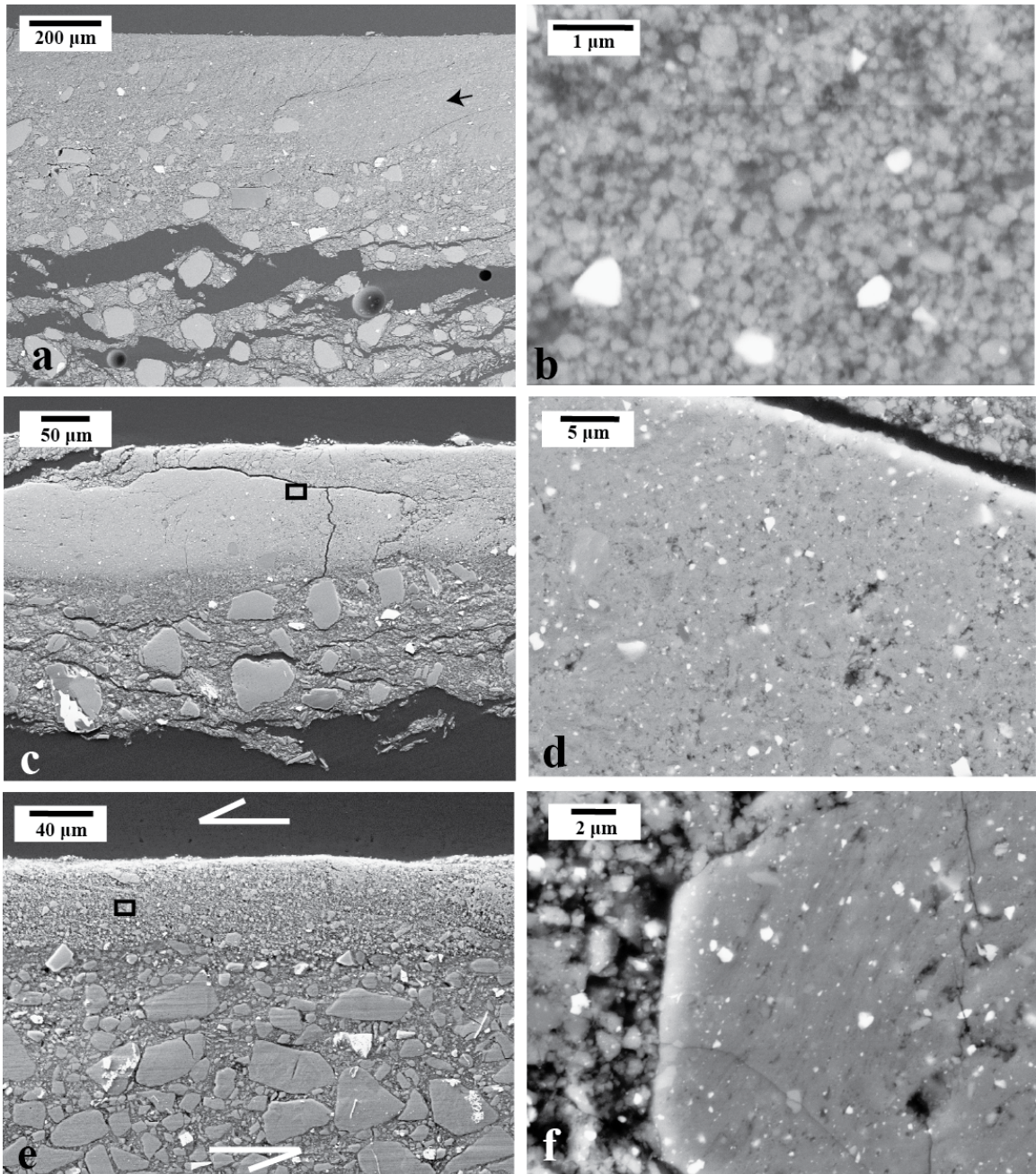


Figure 9

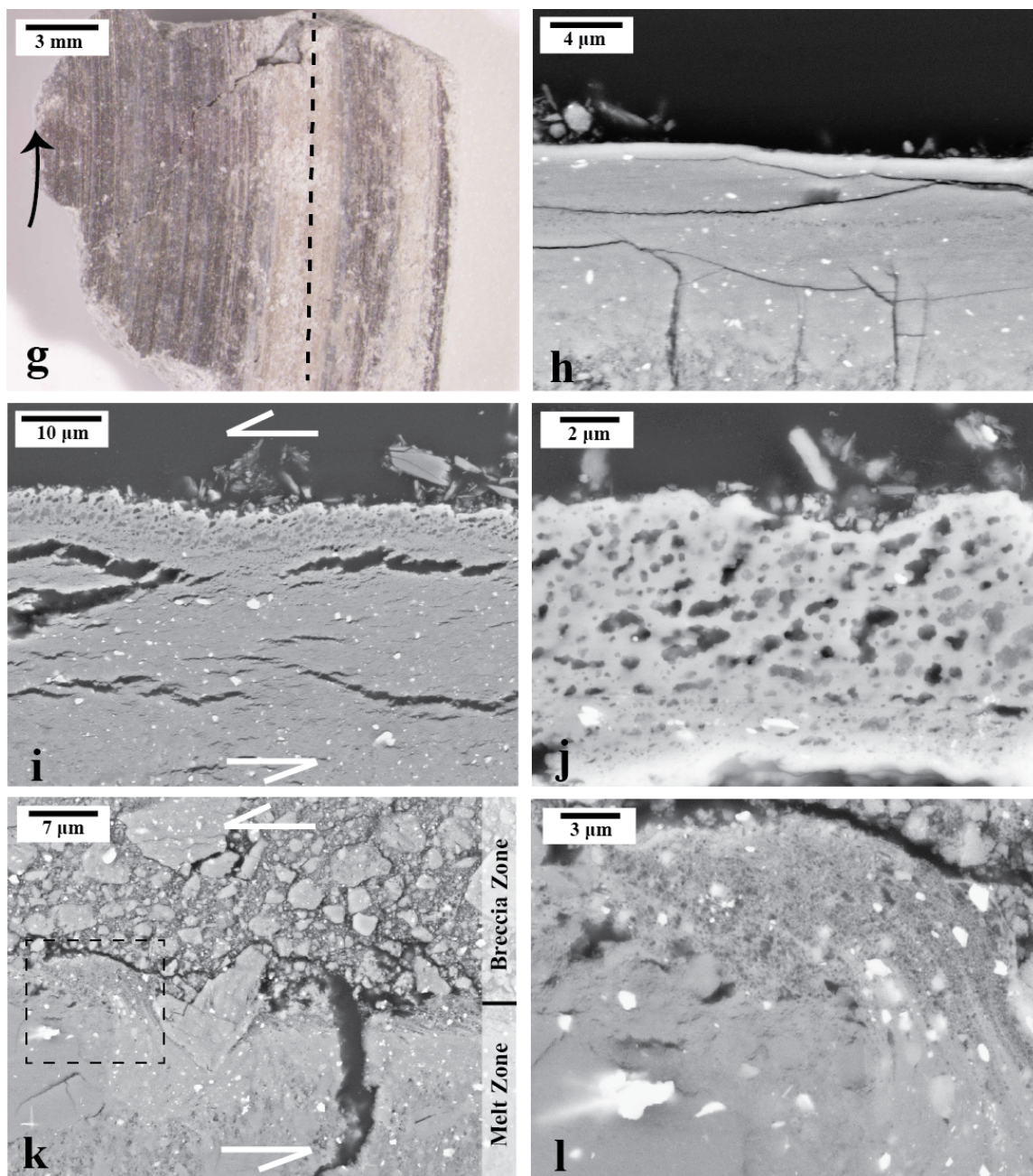


Figure 9 (continued)

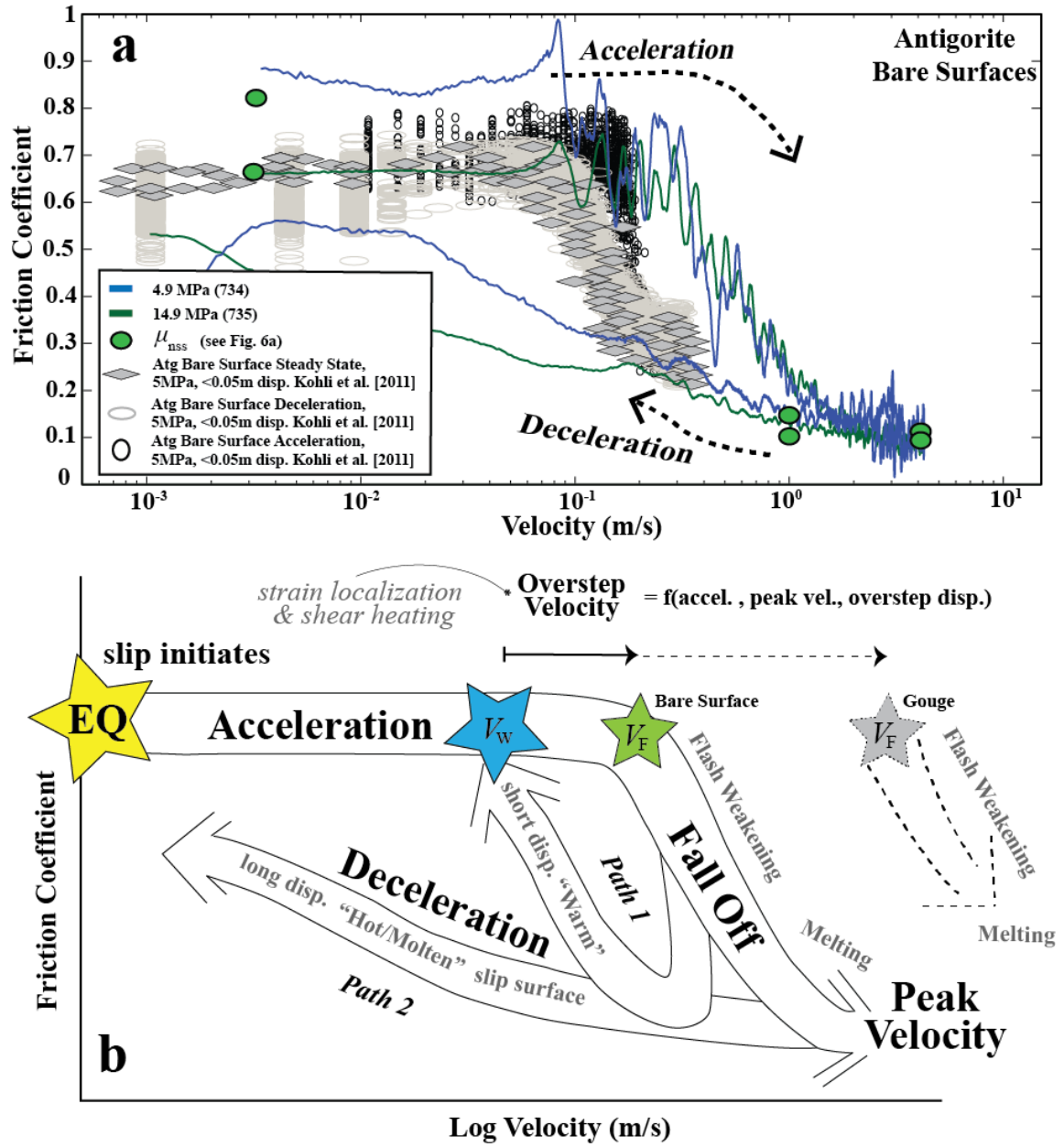


Figure 10.

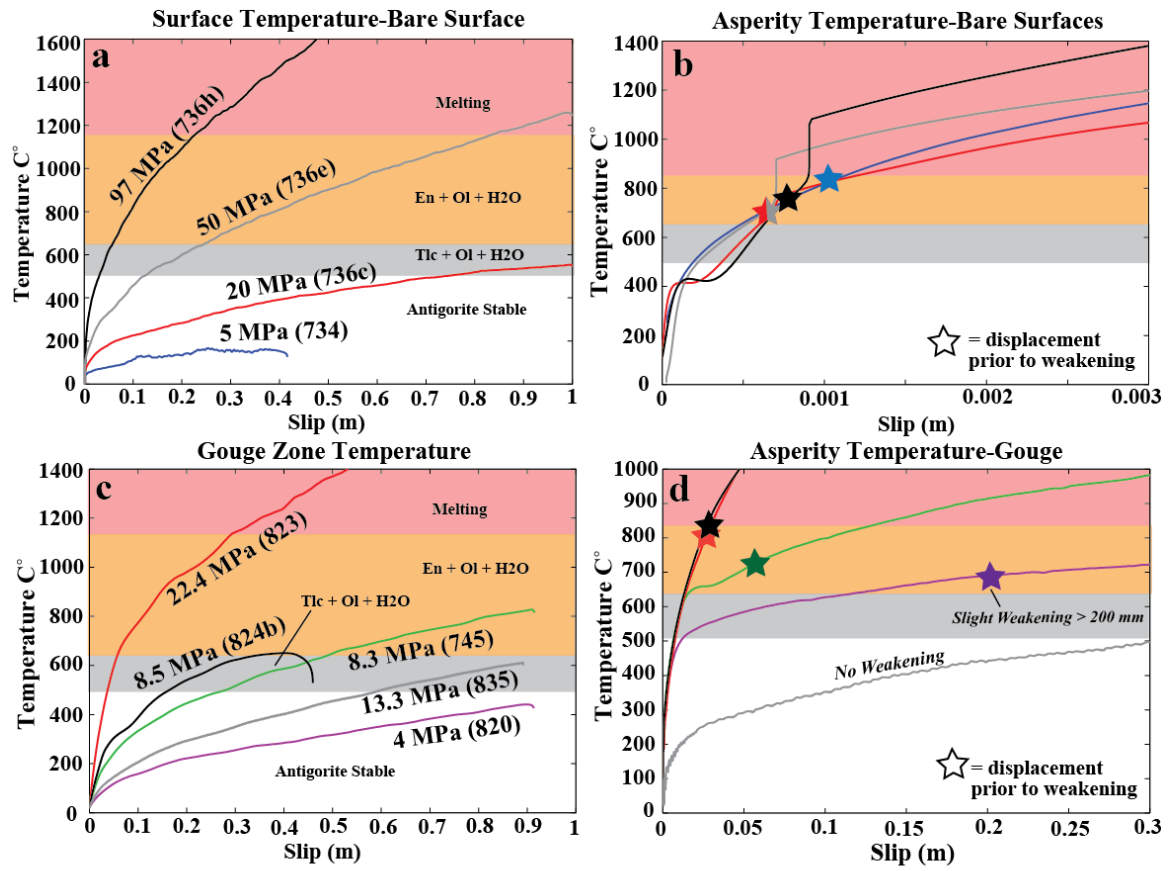


Figure 11

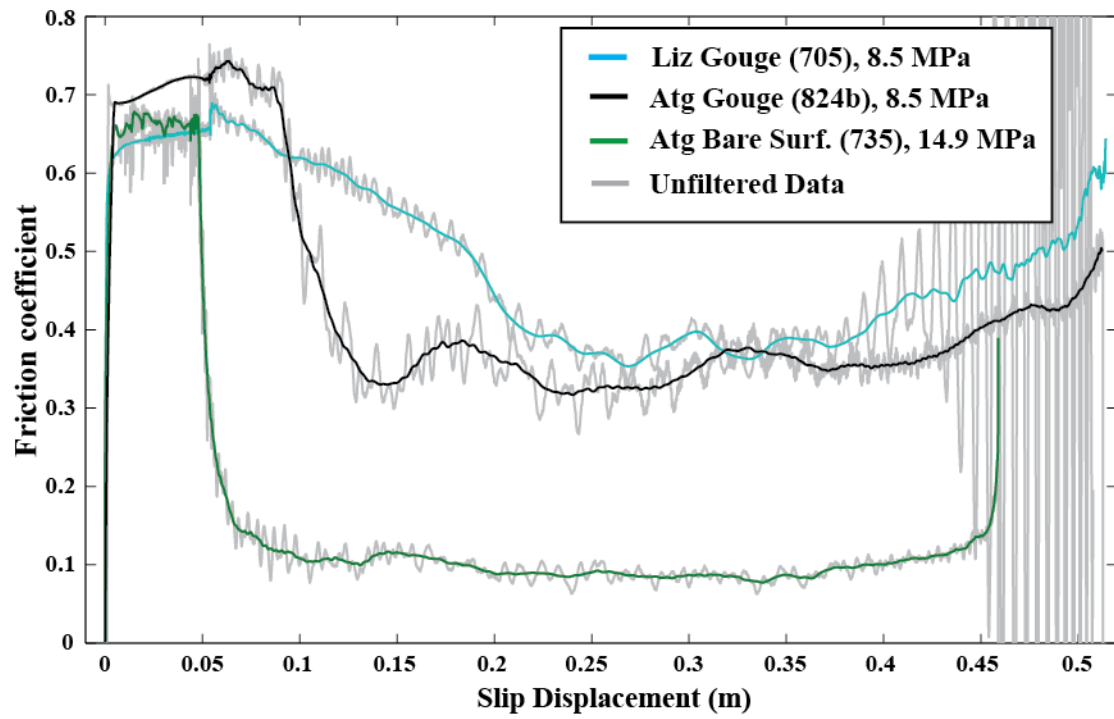


Figure A1

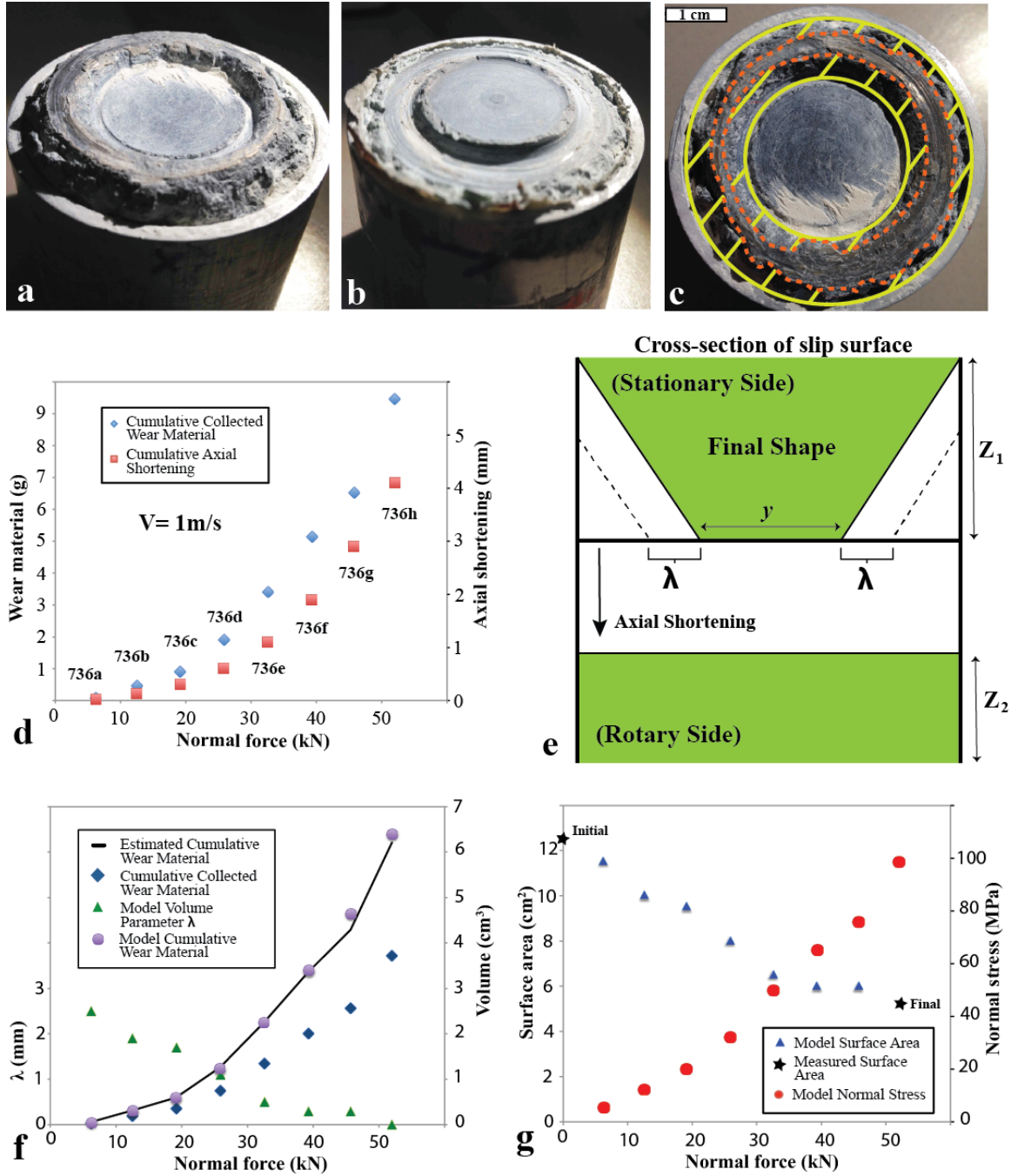


Figure C1.

Chapter 2

Role of pore fluid pressure on transient strength changes and fabric development during serpentine dehydration at mantle conditions: implications for subduction-zone seismicity

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Abstract

To further investigate the dehydration embrittlement hypothesis and its possible link to subduction-zone seismicity, we conducted deformation experiments on antigorite serpentinite in a Griggs-type apparatus at conditions below and above antigorite stability. Temperature ramps (crossing the antigorite thermal stability) were used in conjunction with a new experimental method that allows fluid produced during dehydration reactions to be drained, partially drained or undrained. During temperature ramps, weakening coupled with transient slip initiated at ~ 650 °C, coincident with the predicted phase transition of antigorite to olivine and talc at ~ 1 GPa. The weakening-rate and steady-state strength were dependent on drainage conditions; undrained samples weakened over a few minutes and supported the lowest shear stress (~ 50 MPa), while drained samples weakened over a few hours and supported the highest shear stress (~ 210 MPa). The coefficient of friction (shear stress over normal stress) in drained samples decreased from ~ 0.4 to ~ 0.16 after the temperature ramp. The strengths of samples that were first annealed at 700 °C for ~ 12 hours, then deformed, were similar to those observed in the temperature ramp experiments. Strain localization along fractures occurred in all samples during temperature ramping, regardless of the drainage conditions. However, microstructural observations indicate deformation by ductile mechanisms at higher strain under both undrained and drained conditions. The rheology and microstructures suggest dehydrating serpentinite deforms via semibrittle flow with grain-scale ductile deformation more active at high pore fluid pressures. Our results suggest that earthquakes in serpentinized mantle do not nucleate as a direct result of unstable frictional sliding along fractures generated at the onset of dehydration reactions.

1. Introduction

In subduction zones there is a strong correlation between the location of intermediate-depth earthquakes (50-200 km) and the predicted thermal stability of hydrous minerals, suggesting a link between dehydration reactions and seismicity [e.g., *Hacker et al., 2003; Abers et al., 2013*]. Earthquake hypocenters are typically observed along parallel trending planes within the subducting crust and mantle giving rise to the double Benioff zone [e.g., *Brudzinski et al., 2007*]. The overall distribution of these earthquakes is highly dependent on the thermal structure; in older (cold) lithosphere, earthquakes primarily occur within the subducting crust and deep within the mantle, whereas in younger (hot) lithosphere, earthquakes primarily occur within the mantle near the slab Moho [*Abers et al., 2013*].

Intermediate-depth earthquakes are commonly attributed to dehydration embrittlement, a phenomenon in which rocks containing hydrous minerals undergo brittle fracturing during prograde metamorphism [e.g., *Raleigh and Paterson, 1965; Murrell and Ismail, 1976; Green and Houston, 1995; Kirby et al., 1996; Davies, 1999; Hacker et al., 2003; Peacock, 2001; Jung et al., 2004; Milsch and Scholtz, 2005*]. Since its initial discovery, dehydration embrittlement has been almost exclusively explored (experimentally) with serpentine, a mineral that likely occurs within seismically active regions of the subducting mantle [e.g., *Hacker et al., 2003; Hyndman and Peacock, 2003; Reynard, 2013; Hirth and Guillot, 2013*]. Other hydrated rocks within the subducting crust that are associated with seismicity (e.g., lawsonite blueschist) are generally assumed to exhibit similar behavior [e.g., *Abers et al., 2013*].

Despite numerous studies that have documented dehydration embrittlement in serpentinite, it remains unclear if and how this phenomenon generates earthquakes [e.g., *Gasc et al*, 2011; *Chernak and Hirth*, 2011; *Reynard*, 2013]. The nucleation problem is two-fold; (1) we need to understand how strain becomes localized during metamorphism and (2) whether continued frictional sliding along the localized zone is unstable [e.g., *Kanamori and Brodsky*, 2004; *Scholz*, 1998]. As described below, existing experimental observations on serpentinite give conflicting observations on both strain localization and unstable fault slip during dehydration reactions.

There are many physical models to explain the link between serpentine dehydration and seismicity. The classic dehydration embrittlement model assumes simple Mohr-Coulomb failure caused by increasing pore fluid pressure [*Raleigh and Paterson*, 1965; *Murrell and Ismail*, 1976]; pore fluid is generated from the dehydration reaction and fluid pressure increases because the reaction has a positive volume change. The distribution of earthquake hypocenters in both hot and cold subduction zones appears to support this model [*Abers et al.*, 2013]. However, the experiments by *Jung et al.*, 2004, at confining pressures (P_c) as high as 6 GPa, demonstrated that partially serpentinized samples also fracture when the reaction volume is negative. Under similar conditions, *Dobson et al.*, [2002] and *Jung et al.* [2009] recorded acoustic emissions during dehydration, which they interpret to indicate unstable slip accompanied by fracture. Furthermore, unstable sliding was observed at dehydration conditions in experiments conducted on antigorite, the high-pressure serpentine polymorph, with a constant pore fluid pressure and at $P_c = 100$ MPa [*Takahashi et al.*, 2011]. Together,

these studies support the hypothesis that earthquakes can nucleate directly as a result of the promotion of unstable frictional sliding on fractures produced via dehydration.

Conversely, a second body of work illustrates stable frictional behavior of dehydrating serpentinite [Rutter and Brodie, 1988; Arkwright *et al.*, 2008; Rutter *et al.*, 2009, Chernak and Hirth, 2010; Gasc *et al.*, 2011; Chernak and Hirth, 2011]. Based partly on these observations, several alternative mechanisms have been proposed to generate intermediate (and deeper) depth subduction zone earthquakes [cf., Hacker *et al.*, 2003]; for example via dynamic weakening of fine-grained reaction products via superplastic flow [e.g., Schubnel *et al.*, 2013], thermal runaway within fine-grained olivine shear zones [Kelemen and Hirth, 2007; Prieto *et al.*, 2013], plastic faulting [Renshaw and Schulson, 2013], unstable slip promoted by stress concentrations at the edge of weak zones created during dehydration [Brantut *et al.*, 2012], or simply by fluid migration out of the region of dehydrating serpentinite into overlying mantle rocks that may exhibit unstable frictional behavior [e.g., Arkwright *et al.*, 2008].

The conflicting hypotheses surrounding the link between dehydration embrittlement and seismicity are in part due to the complicated nature of the mechanical behavior and reactions involved [e.g., Hacker *et al.*, 2003; Wong *et al.*, 1997, Brantut *et al.*, 2011], as well as the extreme pressures at which they occur in the Earth (>1 GPa). Our ability to explore processes that occur at high pressures is limited by the deformation apparatus and procedures used during experiments (e.g., constant stress vs. constant strain-rate; general shear vs. axial compression), each with their own limitations [e.g., Tullis and Tullis, 1986; Jaeger *et al.*, 2007]. One drawback of high confining pressure deformation machines (e.g., D-DIA, Griggs Rig, Multi-anvil) is a lack of pore fluid

pressure control. For this reason many studies have used lower confining pressure machines (< 500 MPa) [e.g., *Rutter and Brodie*, 1988; *Arkwright et al.*, 2008; *Rutter et al.*, 2009; *Takahashi et al.*, 2011; *Brantut et al.*, 2012]. The results are then extrapolated to higher pressure with the assumption that the effective pressure predominantly controls the rheology, and thus that the magnitude of the sample pressure (or depth) is irrelevant. To date this assumption remains untested.

In this study we provide a novel experimental method to vary the pore fluid pressure in a Griggs Rig deformation apparatus - a task not previously accomplished at confining pressures as high as ~ 1 GPa. We use this method to explore how pore fluid pressure affects transient weakening during dehydration at mantle pressures, following the approach employed by *Chernak and Hirth* [2011]. These tests allow us to further constrain the rheology of dehydrating serpentinite at mantle conditions and the possible link between dehydration embrittlement and seismicity.

2. Experimental Methods

2.1 Starting material and sample preparation

Experiments were conducted on an antigorite-rich serpentinite (ATG) collected near Rochester, Vermont that is composed of $\sim 90\%$ antigorite, 5-8% magnetite and minor amounts of magnesite, sulfides and oxides determined by X-ray diffraction and petrographic analysis [*Proctor et al.*, 2014]. The serpentinite was first crushed into a fine-grained powder, sieved to a grain-size of 37-53 μm , washed in distilled water to remove ultra fine-grained particles and dried on a hot plate at ~ 100 °C. The powder was next cold-pressed at room temperature into a ~ 1.1 mm thick gouge zone between two

alumina or yttria-stabilized zirconia (Y-zirconia) pistons cut at 45 degrees, then hot-pressed at 400 °C at a confining pressure of ~1 GPa for 12 hours prior to deformation.

2.2 Deformation apparatus and sample assembly

Experiments were conducted in solid confining media Griggs-type triaxial press [see *Tullis and Tullis*, 1986 for details]. The sample was encased in an assembly that rests within a cylindrical pressure vessel. The outer assembly components are shown in Figure 1A. We designed three versions of the inner assembly components to accommodate fluid that is generated within the sample at high temperatures (Figure 1B, 1C and 1D). In the undrained assembly (Figure 1B), the sample is confined between two solid Y-zirconia pistons cut at 45 degrees and encased in a 0.25 mm thick silver tube or jacket. The jacket is cut long enough such that the outer pistons partially fit into the ends of the tube (Figure 1B). This overlap keeps the pistons aligned during the initial sample loading. Platinum disks separate the inner shear pistons from the outer pistons and become mechanically welded to the jacket forming an impermeable chamber during the initial hot pressing of the sample at 400 °C and 1 GPa confining pressure. The partially drained assembly (Figure 1C) is similar to the undrained assembly except that the inner shear pistons are alumina and have a hollow core (1.6 mm diameter) creating an upper and lower cylindrical reservoir. The reservoirs are filled with either ~200 μm diameter Y-zirconia microspheres or 200-400 μm diameter glass carbon microspheres (Figure 1E and 1F). The drained assembly is further modified to vent fluid out of the pressure vessel (Figure 1D). In this assembly the lower Pt disk is removed and a ~ 2.5 mm thick split (i.e., with a vertical cut) alumina disk is added below the lower hollow shear piston, and the lower piston is replaced with a hollow alumina piston. The split alumina disk allows

only fluid to pass from the upper shear piston to the lower piston. The Ag jacket is extended to cover the disk and overlap the lower outer piston. Because there is no lower welded seal, to prevent salt from potentially leaking into the jacket and drainage system, a portion of the inner salt cylinder is replaced with a soft-fired pyrophyllite “can,” which prevents salt from migrating into the jacket. The can is machined such that the jacket is seated within a notch on the inner side. The solid tungsten carbide (WC) base plug is replaced with a WC plug that has a matching (1.6 mm diameter) drainage hole (Figure 1A). The plug rests on the base plate, which also has a drainage hole, thus providing a fluid pathway out of the apparatus.

To increase coupling between the shearing pistons and the sample, nine small grooves are cut into the surface of both shear pistons with a diamond wire saw. The grooves are $\sim 60\ \mu\text{m}$ wide, $\sim 30\ \mu\text{m}$ deep and spaced $\sim 800\ \mu\text{m}$ apart (Figure 1G).

Confining pressure, axial force, axial displacement and temperature were recorded at 1 Hz. Axial force is measured with an external load cell in line with the axial column and axial displacement is measured with a displacement transducer mounted on the load cell [e.g., *Tullis and Tullis*, 1986]. These measurements were converted to axial stress, shear stress and shear strain following methods outlined in the data reduction program RIG version 1.3 by Matej Pec (<https://sites.google.com/site/rigprogram/file-cabinet>). In all experiments axial stress or load values are corrected for friction between the axial loading piston and components of the assembly. The frictional stress is measured as the difference between the observed load value and the confining pressure when the loading piston first touches the upper piston; this value is assumed to be constant and is subtracted from measured load values. The resolution of stress is ± 25

MPa and the precision is ~ 1 MPa based on previous studies at similar conditions [Chernak and Hirth, 2010; Gleason and Tullis, 1993]. All unprocessed (raw) data are included in the supplemental materials.

2.3 Experimental procedures

The imposed experimental conditions are shown in Table 1. In all experiments general shear was imposed with a constant axial displacement rate of $1.8 \times 10^{-5} \text{ mm s}^{-1}$, corresponding to a shear strain rate of $\sim 2.3 \times 10^{-5} \text{ s}^{-1}$. All samples were initially deformed at 400°C until the differential stress approached a steady state value corresponding to a shear strain (γ) of ~ 0.75 and a differential stress of ~ 1.2 GPa. To further establish the rheology of serpentinite within its stability field, one sample (Run 1684) was deformed to $\gamma = 1.5$.

To explore the role of pore fluid pressure on transient weakening during dehydration, in six experiments (Runs 1707, 1768, 1773, 1848, 1865 and 1892) the temperature was increased at 0.5°C s^{-1} from 400°C to 700°C , after which samples were allowed to deform for an additional ~ 6 hours at 700°C (Figure 2). In Run 1892 the sample was quenched ~ 30 minutes after the ramp to characterize the reaction progress and microstructures after the initial stages of weakening. During the temperature ramps the pressure increased by ~ 100 MPa due to thermal expansion of the confining media (Table 1).

To further constrain the influence of pore fluid pressure on dehydrating serpentine, two samples were first heated to 700°C at ~ 1 GPa for ~ 12 hours, then deformed with a constant shear strain rate of $\sim 2.3 \times 10^{-5} \text{ s}^{-1}$ using the drained (Run 1775)

and undrained (Run 1703) assemblies. We term these experiments “cook and crank” (cf. *Trepmann, 2013*).

After deformation, all samples were first quenched under load to 300 °C in ~5 minutes and then returned to ambient conditions over 1-2 hours. Samples were impregnated with epoxy, cut and polished into thin-sections for microstructural analysis. Micro-imaging and micro-chemical analyses were conducted with an Olympus SZX16 optical microscope fit with a digital camera, a LEO 1500VP field-emission scanning electron microscope (FE-SEM) and a Cameca SX100 electron microprobe. Exploratory electron backscatter diffraction (EBSD) was conducted at the University of Otago, New Zealand on a Zeiss SIGMA FE-SEM equipped with Oxford Instruments NordlysF EBSD detector.

3. Results

3.1 Mechanical results

Stress-strain curves for 6 constant strain-rate experiments are shown in Figure 3A. At 400 °C, with no temperature ramp, (purple trace, Figure 3A) the shear stress (τ) first increased linearly, the sample then yielded and achieved a nominally steady-state shear strength of ~700 MPa. The temperature-ramp experiments (green, red, and black traces, Figure 3A) followed similar loading paths prior to the temperature increase; peak stresses in these experiments varied by ~100 MPa. *Chernak and Hirth [2011]* reported a similar range of yield stresses in axial compression experiments at similar conditions.

Weakening occurred in all samples during temperature ramping with the transient and steady-state behavior dependent on the sample assembly. Experiments using the undrained assembly (red trace, Figure 3A) showed the steepest unloading slope and the

lowest nominal steady-state shear stress (~ 50 MPa); experiments with the drained assembly (green trace, Figure 3A) showed a gradual unloading slope - never fully reaching a steady-state shear stress and the highest final shear stress (~ 210 MPa); experiments with the partially drained assembly (black trace, Figure 3A) showed an intermediate unloading slope. The unloading slope (dF/dx) never reached the apparatus stiffness and the unloading rate was gradual (Figure 3B), consistent with results from *Chernak and Hirth* [2011]. The results for undrained, partially drained and drained conditions were duplicated in Runs 1892, 1848 and 1865 (Figure 3C).

The steady-state shear stress values in cook and crank experiments were similar to those in the temperature-ramping experiments. The shear stress during experiments with the drained assembly (green dashed trace, Figure 3A) increased to a peak stress of ~ 360 MPa and then the sample strain weakened to a nominal steady-state value of ~ 275 MPa. The undrained sample (red dashed trace, Figure 3A) showed prolonged strain hardening and reached a nominal steady-state value of ~ 60 MPa.

After the temperature ramp, the amount of time (and strain) required to reach a nominal steady-state shear stress varies with the imposed drainage condition. The undrained and partially drained samples (red and black traces, Figure 3C) reached steady-state after ~ 40 minutes (shear strain, $\gamma \sim 0.6$). In contrast, the drained samples gradually approached a steady-state shear stress after ~ 350 minutes ($\gamma \sim 0.9$).

The evolution of differential stress and axial displacement during the temperature ramps are shown in Figure 3C and 3D. The weakening rate and associated displacement rate vary with the imposed pore-fluid drainage condition at temperatures above ~ 650 °C. During the initial temperature increase from 400°C to ~ 650 °C, the sample strength

followed similar quasi-linear unloading paths for all imposed drainage conditions (Figure 3D). Above ~ 650 °C the weakening rate varied for each assembly; the strength of the undrained sample (Run 1707) decreased by ~ 800 MPa over a 4-minute span, the strength of the drained sample (Runs 1773) decreased by ~ 150 MPa over the same 4-minute span and the partially drained sample showed an intermediate weakening rate. Similarly, the displacement rates (and strain rates) increased at temperatures > 650 °C, with the undrained samples showing the highest increase during the weakening (Figure 3D), and the partially drained and drained samples showing gradual increases in displacement rate during weakening. The displacement-rate increase reflects elastic unloading of the axial column during weakening; we term these events transient slip events.

The conditions where we observe an obvious deviation in the weakening rate (~ 650 °C, confining pressure of 1.0 GPa and the mean normal stress of ~ 1.3 GPa) correspond with the antigorite reaction boundary (Figure 2). Thus, we attribute the transient weakening and corresponding increase in displacement rate to the onset of dehydration. The mean normal stress (one third of the sum of the principle stresses) is by definition the isotropic component of the stress tensor or pressure of the sample [e.g., *Poirier*, 1985; *Jaeger et al.*, 2007], which, with caution, has been argued to be appropriate for thermodynamic treatments of deforming materials [e.g., *McLellan*, 1980].

3.2 Sample microstructures in temperature-ramp experiments

The sample deformed at 400 °C, with no temperature ramp, exhibits distributed deformation with no evidence of shear localization. Similar to observations of *Katayama et al.* [2009], the sample exhibits a modest lattice preferred orientation, as indicated by

continuous color zones in cross-polarized light with a full wavelength plate inserted (Figure 4A). At higher magnification, some grain-scale microcracks are observed.

In all temperature-ramp experiments we observe microstructural evidence for brittle deformation and strain localization, however the strain distribution within these samples varied with the sample assembly. The undrained sample (Run 1707) shows prominent shear fractures located near the piston/sample interfaces, and one fracture crossing the sample with little apparent offset (Figure 4B). However, the majority of the matrix appears nominally free of fractures. A similar fracture at the piston/sample interface was observed in Run 1892 (undrained), which was quenched ~30 minutes after the temperature ramp, indicating that this faulting occurs at the onset of weakening. In contrast, the drained and partially drained samples exhibited a network of fractures throughout the sample resembling R-type Riedel shears that accommodated varying amounts of strain (Figure 4C and 4D).

Higher magnification microstructures from temperature-ramp experiments are shown in Figure 5. In all temperature ramp experiments the dehydration reaction did not run to completion. The undrained sample (Run 1707) had significantly more reaction products than the drained sample (Run 1773) (~20 % vs. ~2 % by volume), even though both samples experienced about the same amount of reaction time (Table 1). Run 1892 (undrained), quenched after ~30 minutes at dehydration conditions, exhibited < 1 vol% reaction. For all experiments, Mg-rich olivine is the dominant reaction product with minor amounts of talc, as determined by electron microprobe analysis, energy-dispersive X-ray spectroscopy (EDS) and electron backscatter diffraction. In the undrained samples the olivine grains are equigranular to slightly elongated and range in size from 0.1 to 2

μm (Figure 5A). In the partially-drained and drained samples, the olivine grain-size is ~0.1 μm (at or just below the resolution of the SEM). Olivine forms preferentially within shear fractures and along the grain boundaries of antigorite, magnetite and magnesite, defining a “net texture” of interconnected reaction rims (e.g., Figure 5A). Talc occurs sparsely in undrained sample as 5 to 50 μm-long subhedral grains with the basal cleavage preferentially oriented 30° antithetic to the shearing direction (Figure 5D); in many cases, these grains appear to be intergrown with smaller olivine grains. Finer-grained talc (1-2 μm) is also observed within shear fractures, always with the basal cleavage oriented parallel to the fracture. Magnetite and magnesite grains have olivine reaction rims and are etched with serrated grain boundaries, suggesting that these minerals participated in the reaction (e.g., Figure 5A).

In the partially drained and drained temperature ramp samples olivine is also observed filling fractures with little or no apparent offset oriented 30° antithetic to the shearing direction (Figure 5B). Individual grains are difficult to resolve in the SEM, indicating grain sizes of $\leq \sim 0.1 \mu\text{m}$. It is unclear if talc occurred in these samples due to finer grain-size of reaction products. In the partially drained and drained samples the reaction textures associated with magnetite and magnesite (particularly smaller grains) are less pronounced (e.g., Figure 5C). No reaction products are observed in the sample deformed at 400 °C.

3.3 Sample microstructures in cook and crank experiments

Microstructures from cook and crank experiments are shown in Figures 6 and 7. These samples show deformation styles and reaction textures similar to those observed in the temperature ramp experiments. The drained sample (Run 1775) developed a

pervasive network of shear bands oriented 30° antithetic to the shear direction with a spacing of $\sim 50\ \mu\text{m}$, resembling R-type Reidel shears (Figure 6A and 6B). This sample contains 30-40 vol% reaction products, mostly olivine as determined by EDS analysis (e.g., Figure 6C, 6D and 6E); it is unclear if talc also occurs due to the fine grain size of the reaction products. Most of the olivine has a grain size $\leq \sim 0.1\ \mu\text{m}$ (e.g., Figure 6E), however within higher-porosity zones along the Reidel shears, larger subhedral olivine grains (up to $\sim 4\ \mu\text{m}$) are observed (Figure 6F). The larger unreacted antigorite grains between the shear zones have a preferred orientation as indicated by continuous color bands in cross-polarized light with a full wavelength plate inserted (Figure 6B). Higher magnification reveals these grains are surrounded by a matrix composed of 20 to $< 1\ \mu\text{m}$ antigorite and $\leq 0.1\ \mu\text{m}$ olivine grains. The larger antigorite grains have cleavage parallel cracks and fractures (Figure 6C), bookshelf style rotations (Figure 6C), kinking (Figure 6G) and boudinage structures (Figure 6F). Brecciation of antigorite appears to be syn-metamorphic, as reaction products occur along fractures within antigorite grains. The matrix appears to be mechanically flowing around the larger grains, as indicated by the orientation of the long axis of smaller antigorite grains (Figure 6D). Within the matrix these smaller antigorite grains have “mica-fish” textures consistent with the applied sense of shear (Figure 6E). Aside from cracks, little to no porosity is observed within the reacting matrix.

The undrained cook and crank sample (Run 1703), in contrast with the drained sample, has more reaction products and microstructures that convey a more ductile mode of deformation. This sample contained ~ 90 vol% reaction products, mostly 1 to $4\ \mu\text{m}$ olivine as determined by EDS analysis; minor amounts of talc may occur between olivine

grains (see below) however this remains unconfirmed due to the fine-grained nature of this material. At the thin-section scale, Riedel shears oriented 30° antithetic to the shearing direction (Figure 7A, 7B and 7C) occur throughout the sample. Within the shear zones the olivine has an apparent lattice preferred orientation, indicated by continuous color bands in cross-polarized light with a full wavelength plate inserted (Figure 7B). At higher magnification (Figure 7D, 7E and 7F), the matrix olivine shows a strong shape-preferred orientation with the maximum elongation of the finite strain ellipse defining an S foliation, analogous with an S-C-C' mylonite [e.g., *Passchier and Trouw, 2005*] (e.g., Figure 7D). As such we term the Riedel shears C' shear planes. Furthermore, the long axis of olivine is apparently rotated into and deforming along C' shear planes oriented $\sim 30^\circ$ antithetic to the shear plane (Figure 7D) and along C shear planes oriented parallel to the shearing direction (e.g., Figure 7F), consistent with the color banding illustrated in Figure 7B. The olivine grains tend to be 'necked' or attenuated parallel to the stretching direction with an asymmetry consistent with the imposed sense of shear (Figure 7E and 7F). Around larger magnetite grains, we observe asymmetric strain shadows containing euhedral coarser-grained olivine. Additionally, within the olivine-rich matrix, there are subrounded cavities 1-3 μm in diameter filled with fibrous minerals (e.g., Figure 7D and 7E). The radial orientation of the fibrous minerals (likely talc) within some of the cavities (e.g., Figure 7G) suggests these cavities existed during deformation possibly as fluid filled pores (consistent with the imposed undrained condition). The porosity of the undrained sample was 10-15 vol %, notably smaller than predicted for 90 vol % reaction (~ 29 vol %). The "missing" fluid was likely trapped within dilatant zones that occur at the ends of the sample between the piston/jacket interface.

4. Discussion and implications

4.1 Reaction weakening during temperature ramping

Our results demonstrate that transient weakening and slip occur during serpentine dehydration. We find that the weakening rate depends on the drainage condition, indicating that the strength depends on pore-fluid pressure. The abrupt onset of weakening upon crossing the thermal stability of serpentine indicates a very small amount of reaction-derived water (< 0.25 vol%) has an enormous mechanical effect on strength. The production of porosity during dehydration can also cause weakening [Arkwright *et al.*, 2008]. However, this is probably not the cause of the initial weakening in our undrained experiments, because the increase in porosity is modest during the weakening. Similarly, fine-grained reaction products can cause weakening [e.g., Rutter and Brodie, 1988]. However, because fine-grained material was generated in all experiments yet we observed different weakening rates for different drainage conditions, we conclude that the onset of weakening is not produced by the presence of fine-grained reaction products.

To further explore the interaction between pore fluid pressure and weakening we assume a simple effective stress model [e.g., Scholz, 2002],

$$\tau = \mu(\sigma_n - \alpha P_f), \quad (1)$$

where τ is shear stress, σ_n is normal stress, P_f is the pore-fluid pressure and α is the effective stress coefficient. We assume τ and σ_n are defined as $\frac{1}{2} (\sigma_1 - \sigma_3)$ and $\frac{1}{2} (\sigma_1 + \sigma_3)$ because deformation was imposed on a sample inclined 45° to σ_1 (where σ_1 is axial load and σ_3 is the confining pressure), and that μ is simply τ / σ_n . Using this model we can first calculate antigorite friction. At 400°C (where there is no pore fluid) $\mu \approx 0.36$ (star,

Figure 3A). For temperatures $> 400\text{ }^{\circ}\text{C}$, μ for the drained sample decreases from >0.4 to ~ 0.16 (blue trace, Figure 3C), assuming $P_f \approx 0$, which is also consistent with the steady-state friction values determined in the drained cook and crank experiment (star, Figure 3A). These friction values are notably smaller than determined in lower confining pressure experiments at stable and dehydration conditions [e.g., *Hirose and Bystricky*, 2007; *Takahashi et al.*, 2011; *Okazaki et al.*, 2013 and reference therein]. With $\mu \approx 0.36$ the optimal frictional plane is 35° rather than the imposed 45° ; differential stresses could be up to 9% lower for deformation on the optimal plane.

Recall that the drained, undrained and partially drained samples have identical weakening trends prior to the onset of dehydration (Figure 3D), which indicates (1) friction evolves similarly with temperature (and time) in these samples and (2) there is a strong temperature dependence of stable and meta-stable antigorite friction (cf., *Hirth and Guillot*, 2013). Using this logic, we calculate a first-order estimate of the pore-fluid pressure in the undrained sample (Run 1707) during the temperature ramp. In this calculation, shown in Figure 8, we input into equation 1 the measured time-dependent τ and σ_n values from the undrained test (Run 1707) and the time-dependent μ values from the drained test (Run 1773), then solve for the effective fluid pressure (αP_f). The calculation predicts that αP_f increases and approaches the confining pressure only minutes after the onset of dehydration, indicating $\alpha \approx 1$. The relatively constant differential stresses observed in the undrained cook and crank sample indicates that the pore-fluid pressure does not increase further with 6.5 hours of continued reaction. This is reasonable if the pore-fluid pressure is maintained by expansion of the Ag jacket (and by dilatancy in the sample).

4.2 Semibrittle flow of dehydrating antigorite

At the conditions of our experiments, antigorite deforms by semibrittle flow, i.e., distributed deformation that involves both brittle and crystal-plastic mechanisms [e.g., *Kohlstedt et al.*, 1995], within its stability field. Because antigorite has only two dominant slip systems, the von Mises criterion is not satisfied and dislocation glide cannot fully accommodate strain [e.g., *Chernak and Hirth*, 2010; *Amiguet et al.*, 2014]. Thus, deformation must also involve cracking, frictional sliding and comminution [e.g., *Chernak and Hirth*, 2010; *Amiguet et al.*, 2014]. Brittle mechanisms may become less prominent at higher confining pressures. For example, *Hilaliret et al.*, [2007] show that the strength of samples deformed to low strain (axial strains less than ~ 0.15) at a confining pressure of 4 GPa can be fit with either a power law or an exponential (Peierls) law. However, we observed microcracks in all samples and strengths that are ~ 400 -500 MPa higher than predicted by the antigorite flow laws, indicating that strength was limited by brittle mechanisms, such as frictional sliding between grains, consistent with observations from previous studies [e.g., *Chernak and Hirth*, 2010; *Auzende et al.*, 2015].

During temperature ramps strain became localized along fractures. However, with continued deformation at dehydration conditions, our microstructural observations indicate that strain was distributed under both undrained and drained conditions. At these dehydration conditions, we observed microfractures and textures indicative of grain-scale ductile flow, indicating that both brittle and ductile mechanisms (not necessarily crystal-plastic) remained active. For these samples the effective stress law (equation 1) predicts that the shear stresses will approach zero as the pore fluid pressure approaches the confining pressure (as it appears to, based on the transient behavior observed in the

temperature-ramping experiments). However, in both the undrained temperature-ramp and cook and crank experiments we observed nominally steady-state shear stresses of ~60 MPa (e.g., Figure 3A). For comparison, a shear stress of 60 MPa indicates $\alpha P_f \approx 680$ MPa based on equation 1, significantly less than indicated by Figure 8. To reconcile these observations, we propose a generalized semibrittle flow law with the form:

$$\tau = \Delta + \mu(\sigma_n - \alpha P_f) \quad (2)$$

where Δ represents a ductile deformation mechanism (cf., *den Hartog and Spiers, 2014*). At high pore fluid pressures (as within undrained samples) the frictional term approaches zero such that $\tau \approx \Delta$, whereas at low pore fluid pressure (as within drained samples) both frictional sliding and ductile mechanisms are active.

The olivine deformation mechanism(s) controlling Δ remains unclear. However, we can provide a few first-order constraints from our results. First, for the conditions used in this study (700 °C, 1 GPa and 10^{-5} /s), the wet dislocation creep and wet diffusion creep olivine flow laws [*Hirth and Kohlstedt, 2003*] predict strengths on the order of 35-38 GPa, assuming a water fugacity of 1440 MPa (calculated from Tony Wither's calculator, <http://www.esci.umn.edu/people/researchers/withe012/fugacity.htm>) and a grain-size of 1 μm . These stress values are significantly higher than observed in undrained samples suggesting another deformation mechanism is active. Results of dehydration experiments at low confining pressure (< 200 MPa) led *Rutter and Brodie [1988]* to suggest that strength was controlled by diffusion-accommodated grain-boundary sliding (GBS) based on: a linear-viscous stress versus displacement rate relationship, a lack of sensitivity of stress to changes in effective confining pressure and small olivine grain size. However, based on: (1) the discrepancy between our results and

the olivine flow laws, (2) grain-scale microstructures showing elongation of olivine grains (e.g., Figure 6F) and (3) the high water contents in our undrained experiments, we hypothesize these textures are produced by pressure-solution [e.g., *Rutter and Elliott, 1976*]. This hypothesis is supported by the observations of more reaction products and larger olivine grains in undrained samples compared with drained samples as both reaction kinetics and grain-growth are promoted by enhanced grain boundary diffusion in the presence of water. Additionally, the strengthening observed in the undrained sample (Figure 3A), after the initial weakening, may be the result of olivine grain growth, which is consistent with our microstructural observations.

Pressure-solution has been shown to produce S-C fabrics in exhumed lower crustal rocks [e.g., *Wintsch and Yi, 2002*], but has not been previously demonstrated to occur in water-rich regions of the mantle wedge. The fact that similar textures are not observed in lower confining pressure experiments could be due to the lower temperature of the dehydration boundary and the positive pressure-dependence of the solubility of olivine in water [*Newton and Manning, 2002*].

In drained samples, the greater strength, finer olivine grain-size and lack of a shape preferred orientation (in olivine) indicate that the ductile mechanism observed in undrained samples is either inactive or less efficient in the absence of pore fluid. In this case, we conclude that the strength is limited by frictional sliding and fracturing of the reacting serpentinite, olivine and talc matrix, consistent with the abundant brittle microstructures in these samples (e.g., Figure 6). Alternatively, the (ultra) fine-grained olivine ($<1\ \mu\text{m}$) in these samples allows for the possibility that diffusion creep (GBS) is controlling the rheology [similar to the interpretation of *Rutter and Brodie, 1988*]. The

strength of these samples can be fit with a wet diffusion creep law [*Hirth and Kohlstedt, 2003*] by varying the grain-size and water fugacity. For example a strength of ~540 MPa is predicted if grain-size equals 0.1 μm and the water fugacity is 100 MPa, roughly similar to the observed differential stress of ~450 MPa.

4.3 Geophysical Implications

Our results suggest that subduction-zone earthquakes are not caused directly from dehydration embrittlement in serpentinite. In all of our experiments there was a lack of evidence for unstable frictional sliding, a key requirement for earthquake nucleation. In contrast with our findings, a few studies conducted at lower normal stresses, have reported stick-slip behavior in antigorite at dehydration conditions [*Hirose and Bystricky, 2007; Takahashi et al., 2011*]. Stick-slip behavior can be mitigated by higher apparatus stiffness, however, the machine stiffness of the Paterson gas apparatus used by *Hirose and Bystricky* (~150 kN/mm) is similar to that of our solid-medium apparatus (~120 kN/mm), and the lower effective stiffness of the Griggs apparatus (resulting from the smaller sample size) is offset by the much larger normal stress of our experiments. Other experimental parameters may also inhibit stick slip, such as the imposed constant displacement rate or the solid confining medium by its resistance to lateral sample displacement. However, we note that stick-slip behavior has been observed in other samples deformed in the same apparatus with similar experimental conditions [e.g., *Hadizadeh and Tullis, 1992*]. We suspect the lack of unstable frictional behavior in our experiments arises from a combination of the pressure/temperature dependence on antigorite friction, the inferred activation of pressure-solution creep, and the fact the temperature at dehydration is higher at the conditions of our experiments. Our results are

also consistent with previous studies that suggest subduction-zone earthquakes are not caused by the simple dehydration induced fracturing and unstable slip in serpentinite [Chernak and Hirth, 2011; Arkwright *et al.*, 2008; Rutter *et al.*, 2009]. These finding may apply more broadly to dehydrating rocks within the subducting crust. However, this assumption remains untested.

Our results therefore support the hypotheses that earthquakes are either not occurring in dehydrating serpentinite [e.g., Rutter *et al.*, 2009] or that another mechanism besides dehydration embrittlement promotes seismicity. In this case, owing to the strong correlation between the conditions where dehydration is predicted and the location of earthquakes, we review other ways in which the reaction can promote unstable fault slip. Other possible mechanisms include: creating a localized weak zone in dehydrating serpentinite that then promotes unstable slip in adjacent rocks owing to stress concentrations [Brantut *et al.*, 2012], dehydration induced unstable slip via dynamic feedbacks between shear heating, strain localization, pore fluid pressurization and compaction [Brantut *et al.*, 2011; Brantut and Sulem, 2012], superplasticity of nanograin reaction products [Schubnel *et al.*, 2013], viscous shear heating within fine-grained olivine shear zones (produced by dehydration reactions) leading to thermal run away [Kelemen and Hirth, 2007, Prieto *et al.*, 2013], plastic faulting [Renshaw and Schulson, 2013], or dynamic frictional weakening in antigorite shear zones at thermally stable conditions [e.g., Proctor *et al.*, 2014, Platt *et al.*, 2015]. Furthermore, the mechanisms involved may be scale-dependent and thus only resolved using numerical modeling. For example, Brantut and Sulem [2012] show that unstable slip can occur within dehydrating serpentinite owing to dynamic feedbacks between strain localization and pore-fluid

pressure, but only at scales significantly larger than laboratory samples. Similarly, the viscous shear-heating model proposed by *Kelemen and Hirth* [2007] requires displacements many orders of magnitude greater than the shear zone thickness, rendering this process difficult to simulate in the laboratory.

5. Conclusions

In this study a novel experimental technique is presented to explore the role of increasing fluid pressure on dynamic weakening during serpentine dehydration at mantle pressures. Temperature-ramping experiments on antigorite-rich serpentinite show that strain localization and weakening occurs at the onset of dehydration temperatures producing transient slip events. By varying the drainage condition we show that the weakening rate is strongly dependent on the pore fluid pressure. At the onset of dehydration μ reduces from ~ 0.4 to ~ 0.16 and the effective pore fluid pressures rapidly increase to values near the imposed confining pressure. With continued deformation during dehydration in undrained samples, strain becomes distributed and S-C and S-C' fabrics develop. We propose a generalized semibrittle flow law to model the rheology of these samples. By comparing drained and undrained experiments, we conclude that the pore fluid imparts both a mechanical and chemical effect on the overall rheology. Finally, our results suggest intermediate-depth earthquakes within serpentinite are not caused directly by dehydration embrittlement due to (1) lack of unstable sliding during transient slip events, (2) the tendency for strain to become distributed, (3) low friction coefficients and (4) activation of grain-scale ductile processes.

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Appendix A

Here we present all unprocessed data (see figure A1 below).

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Figure 1. Experimental apparatus and sample assembly. (A) Schematic cross-section of outer sample assembly; inner sample assembly components are noted with dashed line. Three versions of the inner assembly are used to create an (B) undrained, (C) partially drained, and (D) drained system. (E) and (F) Photomicrographs of yttrium-stabilized zirconia (Y-zirconia) and glass carbon micro-spheres used to fill reservoirs. (G) Photomicrograph of shear piston with reservoir opening and grooves cut into the surface.

Figure 2. MgO-SiO₂-H₂O phase diagram after *Ulmer and Trommsdorff* (1995) showing stability zones for mineral phase(s): antigorite, forsterite (Fo), talc (Tlc) and enstatite (En). Stars indicate experimental conditions. Arrows indicate P-T path in temperature ramping experiments.

Figure 3. (A) Mechanical results for temperature-ramp and ‘cook and crank’ experiments with undrained (UD), partially drained (PD) and drained (D) assemblies.

Stars indicate measured friction (μ) values. Experiment run number is noted in parentheses. (B) Evolution of axial force vs. axial displacement during the temperature ramp for Run 1707. Plot shows that the rate of unloading never exceeds the apparatus stiffness. (C) Evolution of differential stress with time after the onset of the temperature ramp from 400 °C to 700 °C. Blue trace notes the evolution of friction within Run 1773 (green trace). (D) Evolution of differential stress and axial displacement during the first twenty minutes after the initiation of the temperature ramp. Note that 650 °C corresponds with the predicted antigorite phase transition shown in Figure 1.

Figure 4. Temperature ramp microstructures. Photomicrographs in plane-polarized light; lower left diagram indicated the imposed drainage condition; arrows indicate the applied sense of shear. (A) Run 1684, deformed at 400 °C without temperature ramp shows no major fracturing. Inset photomicrograph, in cross-polarized light with full wavelength plate inserted, notes moderate preferred orientation of sample. (B) Run 1707 shows one shear fracture with little offset. Inset photomicrograph shows offset of top piston (white) with respect to the sample along a shear zone at contact. (C) Run 1768 shows through going shear fractures. (D) Run 1773 shows through going shear fractures.

Figure 5. Micrographs of reaction products in temperature-ramp experiments; lower-left diagram indicates the imposed drainage condition; arrows indicated sense of shear. (A) Electron backscatter image from Run 1707 with undrained assembly. Dark grey grains are antigorite (Atg), light grey grains are olivine (Ol) and white grains are magnetite (Mt). The olivine forms preferentially along antigorite grain boundaries and around

magnetite grains. (B) Electron backscatter image from Run 1768 showing micron-scale fractures filled with olivine oriented $\sim 30^\circ$ from the shearing direction. The wider fracture (black trace, sub-parallel to the shearing direction) has larger apparent offset than smaller fractures and also lined with fine-grained olivine; darker grains are magnesite (Mgs). (C) Electron backscatter image from Run 1773 showing trace amounts of ultra-fine-grained olivine within fractures and around antigorite grain-boundaries. (D) Photomicrograph in cross-polarized light with full wavelength plate inserted from Run 1707. Image shows subhedral talc grain (Tlc) with long axis oriented $\sim 30^\circ$ from the shearing direction.

Figure 6. Microstructures from drained ‘cook and crank’ experiments.

Photomicrographs in plane polarized light (A) and cross-polarized light with the full wavelength plate inserted (B) from Run 1775 exhibit closely spaced R-type Reidel shears truncating the sample. (C) Enlargement of 6A in electron backscatter showing fine-grained olivine (Ol) surrounding unreacted antigorite grains (Atg). (D) Enlargement of the structures within 6A. (E) Enlargement of fine-grained matrix showing deformation of antigorite. (F) Backscattered image showing boudinaged antigorite grain and Reidel shear (black); coarser-grain olivine occurs within the shear zone. (G) Backscattered image showing kinked antigorite grain.

Figure 7. Microstructures from undrained ‘cook and crank’ experiments.

Photomicrographs in plane polarized light (A) and cross-polarized light with the full wavelength plate inserted (B) from Run 1703 exhibits apparent S-C’ fabric defined by preferred orientation of olivine along shear bands oriented $\sim 30^\circ$ off of the shearing

direction. (C) Enlargement in electron backscatter showing fracture network sub-parallel to the C' shear and reacting magnetite grain (Mgt) with asymmetric strain shadow. (D) Electron backscatter image showing shape preferred orientation of olivine and antigorite defining an S foliation that is approximately perpendicular to the maximum principle stress direction and along C' oriented planes. (E) & (F) Electron backscatter images showing deformation textures in olivine. (G) Enlargement of cavities filled with euhedral fibrous minerals, in some cases oriented radially. The cavities are possibly relic pore spaces.

Figure 8. Calculated evolution of the effective pore fluid pressure within Run 1707 during a temperature ramp (see text for details).

Figure A1. Unprocessed axial load and confining pressure data for experiments reported in this study (refer to Table 1 for run conditions). Load values assume axial force is applied on a 6.35 mm diameter cylinder. Axial displacement values were corrected for the apparatus stiffness and sample shortening.

Table 1

Run Number	Confining Pressure * (MPa)	Temperature (°C)	Drainage Condition	Reservoir Material	Reaction Time (hrs)	Strain-Rate (s ⁻¹)	Total Strain (γ)	Deformation Type
1684	1010	400	Undrained	NA	NA	$\sim 2.3 \times 10^{-5}$	1.3	Distributed
1707	900, 1020	400 to 700	Undrained	NA	6.5	$\sim 2.3 \times 10^{-5}$	2.1	Localized
1892	970, 1090	400 to 700	Undrained	NA	0.47	$\sim 2.3 \times 10^{-5}$	0.8	Localized
1768	980, 1110	400 to 700	Partially Drained	Carbon Glass	5.6	$\sim 2.3 \times 10^{-5}$	1.7	Localized
1848	1020, 1140	400 to 700	Partially Drained	Y-Stab. Zirconia	4.8	$\sim 2.3 \times 10^{-5}$	NA	Localized
1865	998, 1100	400 to 700	Drained	Y-Stab. Zirconia	10.9	$\sim 2.3 \times 10^{-5}$	NA	Localized
1773	973, 1084	400 to 700	Drained	Carbon Glass	5.6	$\sim 2.3 \times 10^{-5}$	1.5	Localized
1703	1030	700	Undrained	NA	40	$\sim 2.3 \times 10^{-5}$	2	Distributed w/ spaced shear zones
1775	1130	700	Drained	Carbon Glass	39.5	$\sim 2.3 \times 10^{-5}$	1.8	Distributed w/ spaced shear zones

*-Pressure before and after temperature ramp indicated; NA-does not apply or value was not calculated

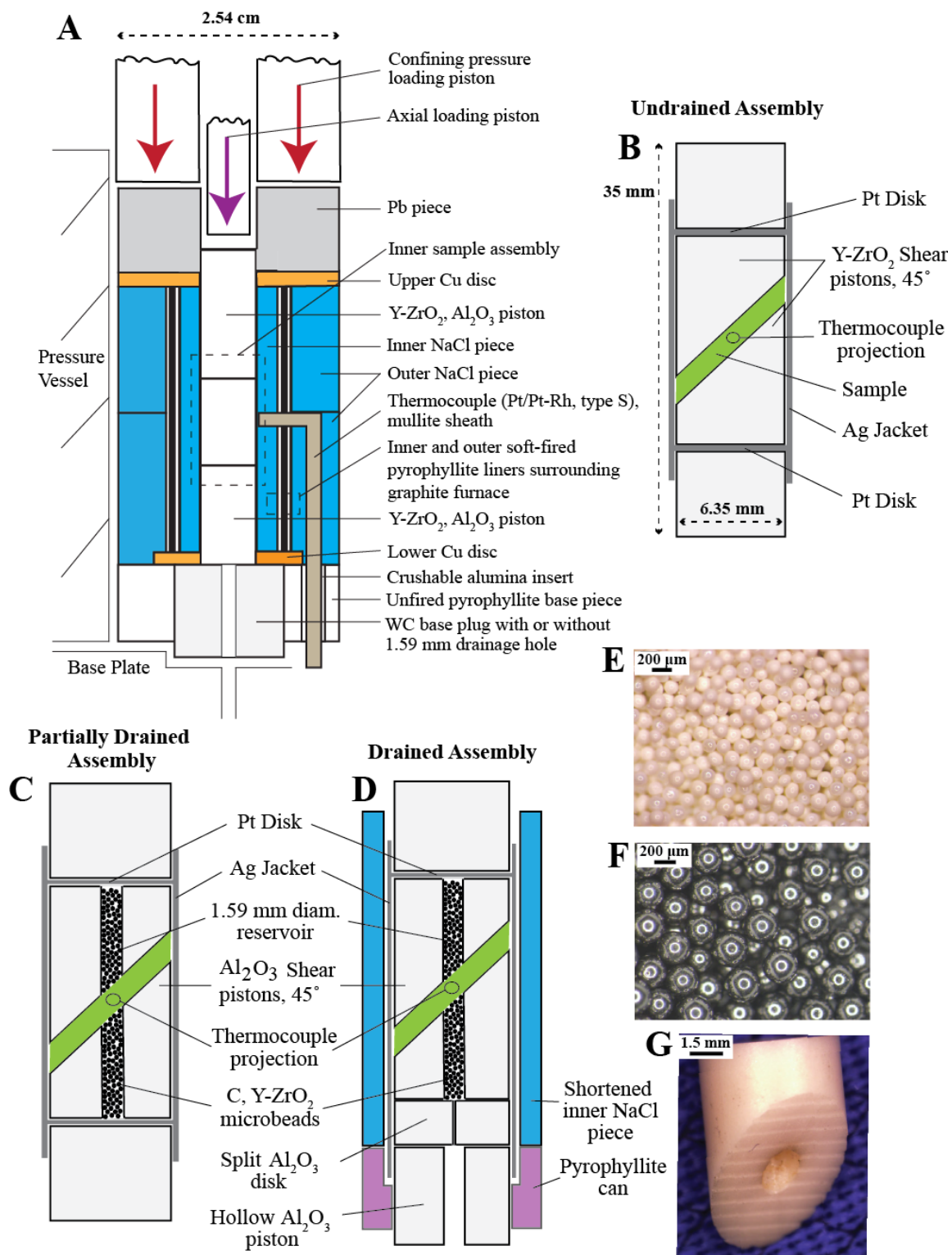


Figure 1

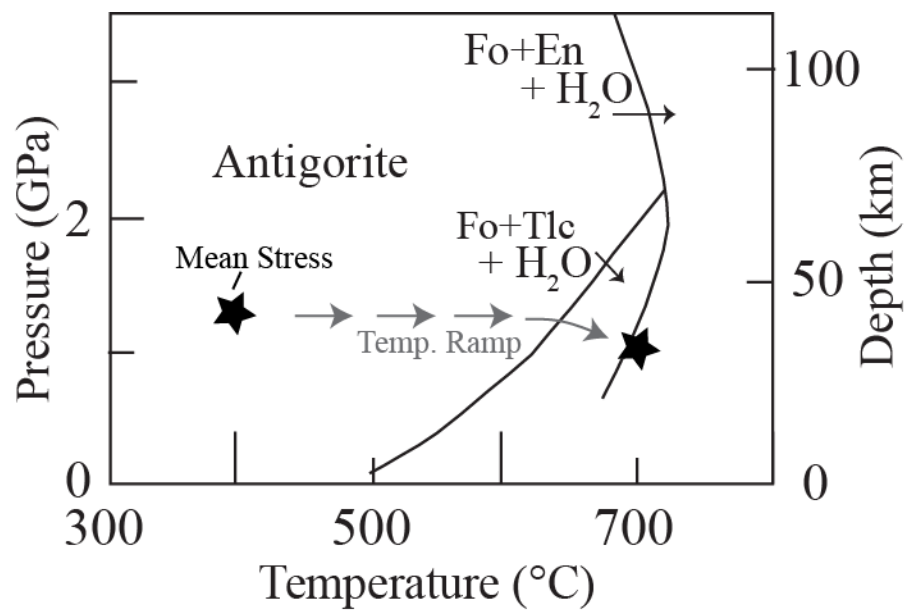


Figure 2

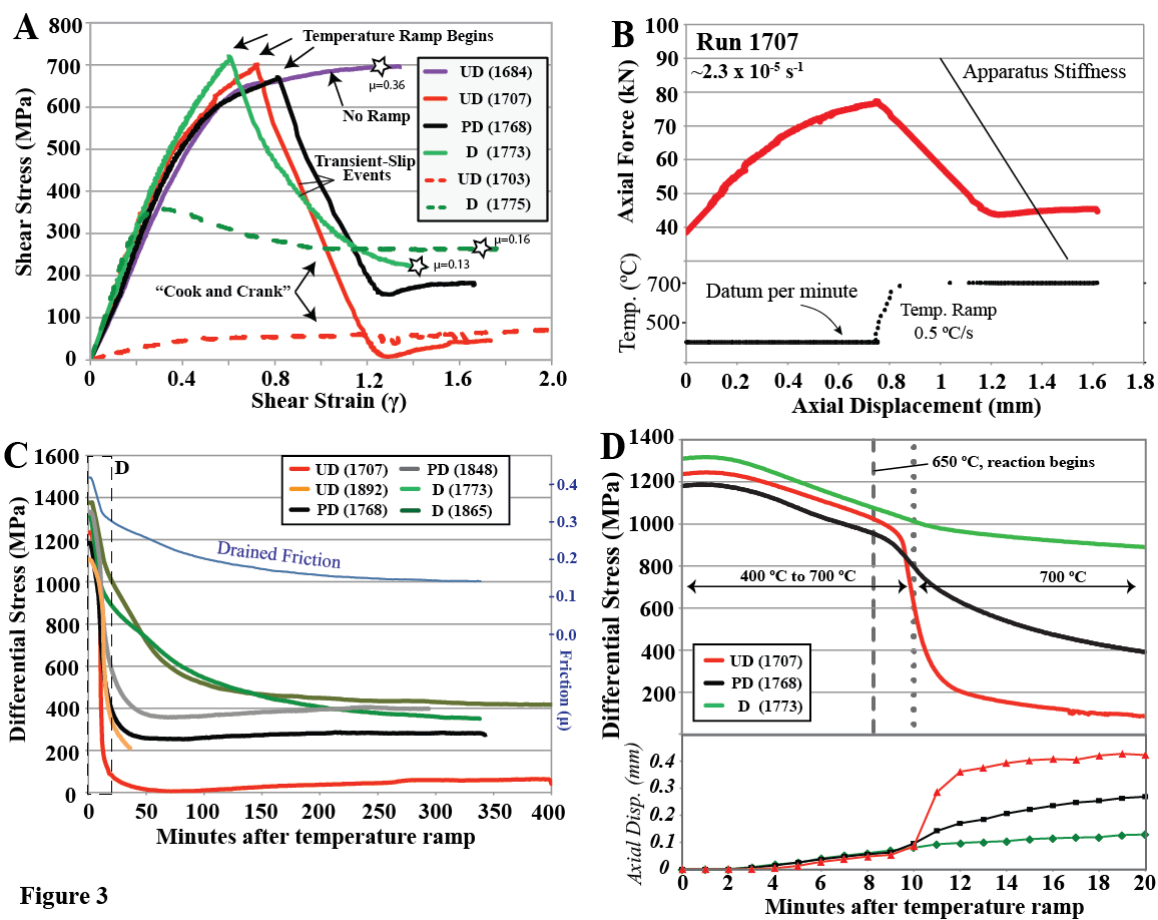


Figure 3

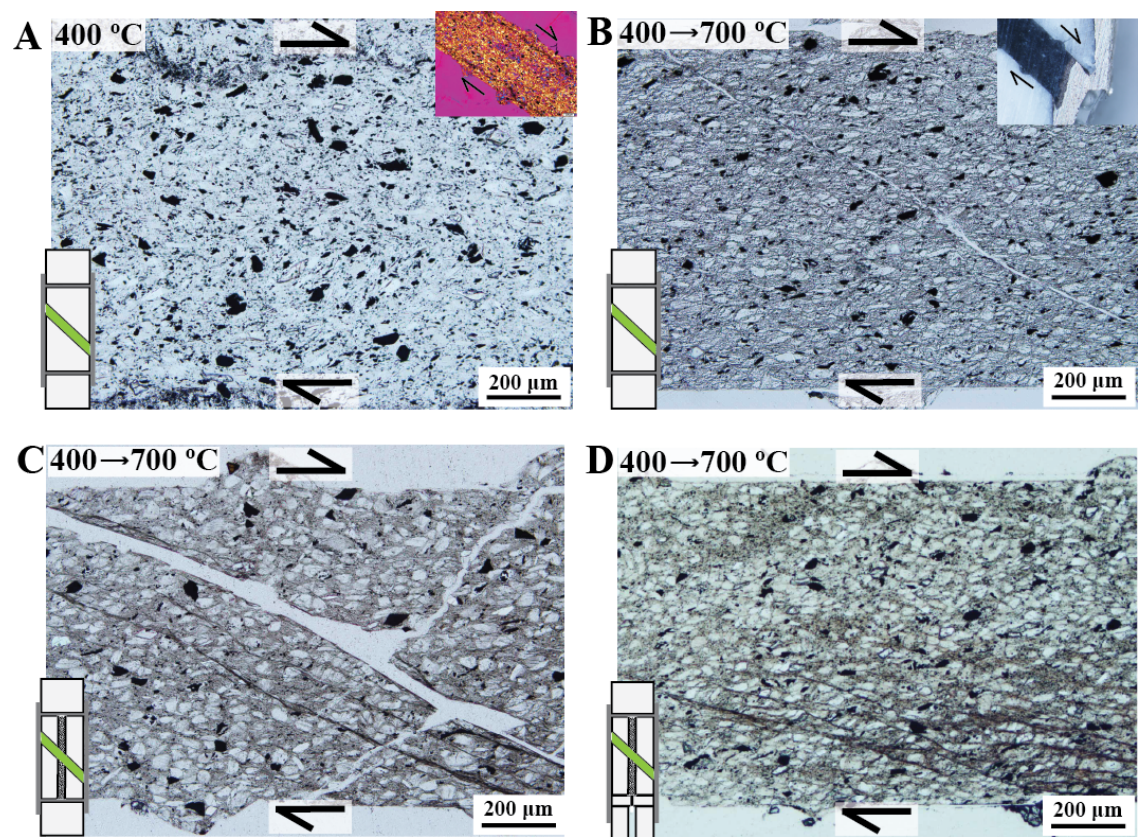


Figure 4

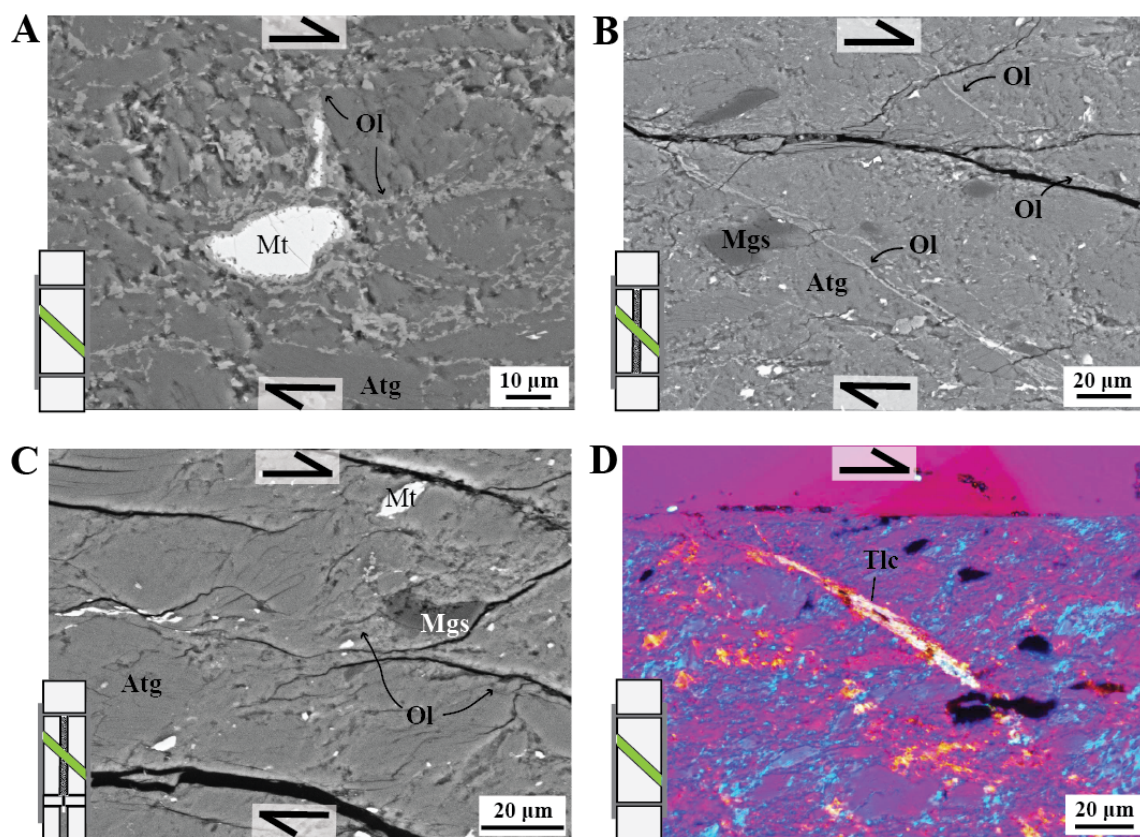


Figure 5

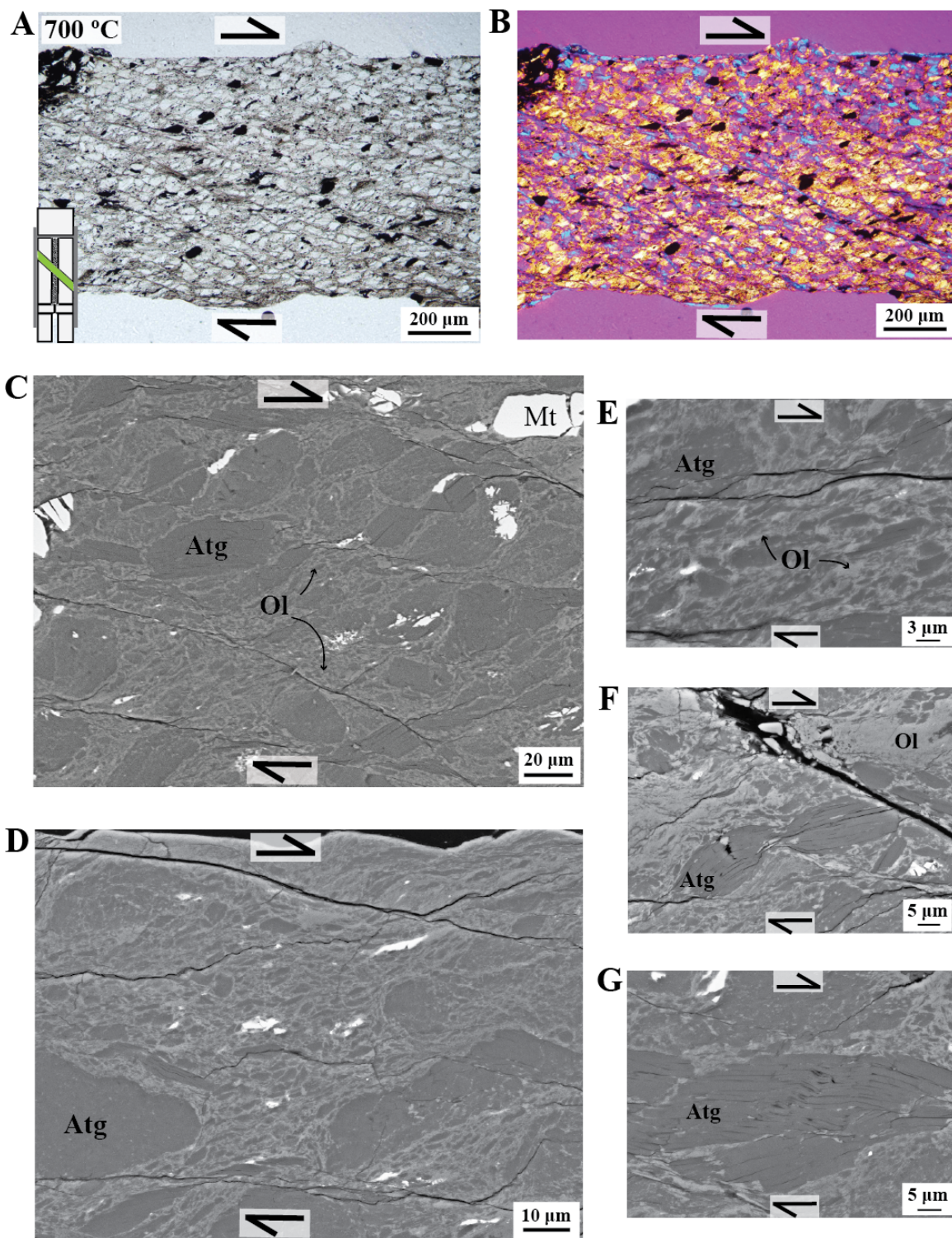


Figure 6

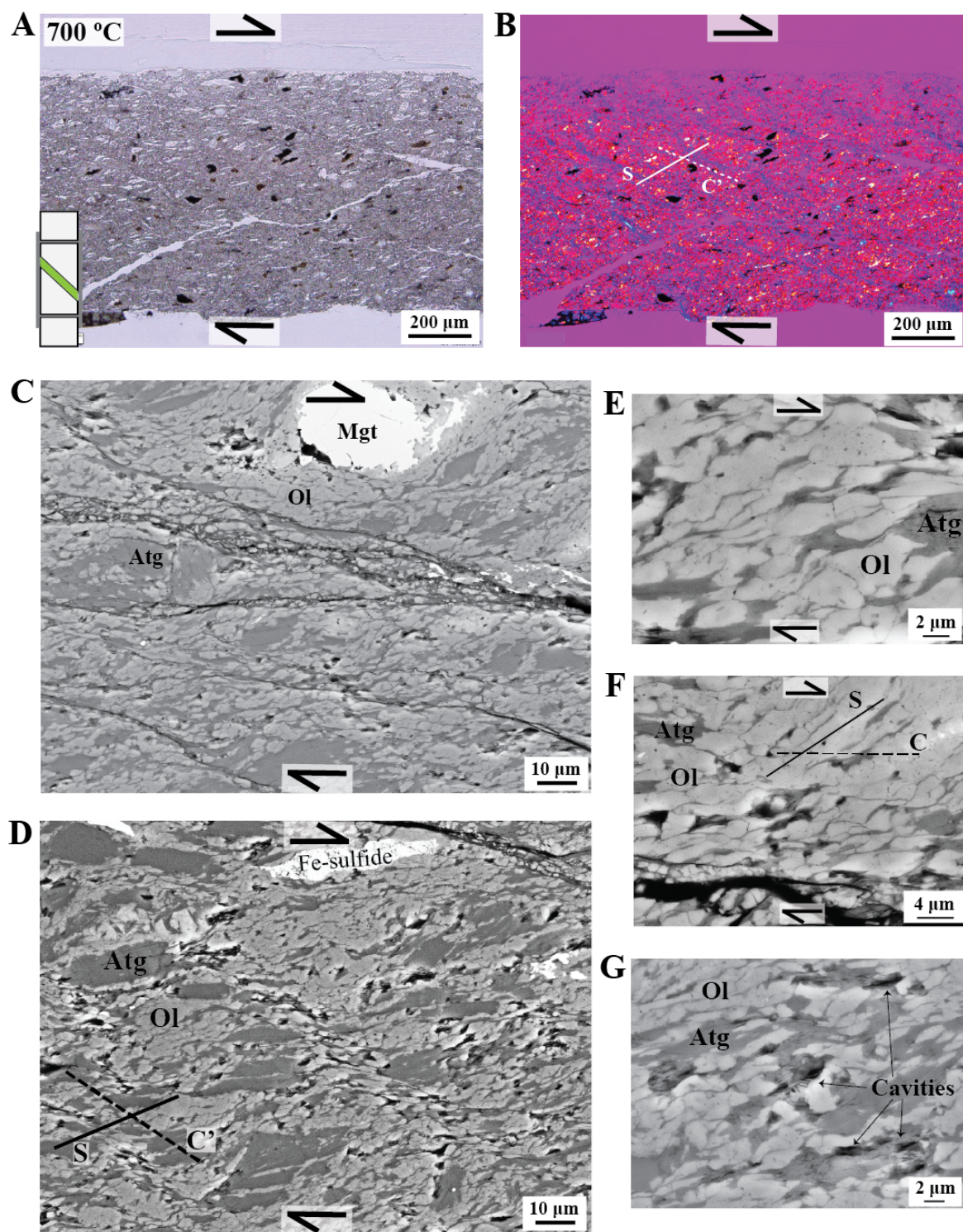


Figure 7

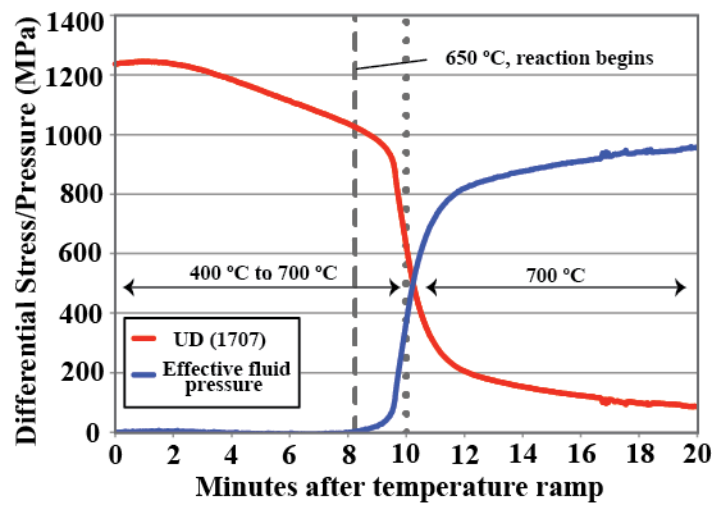


Figure 8

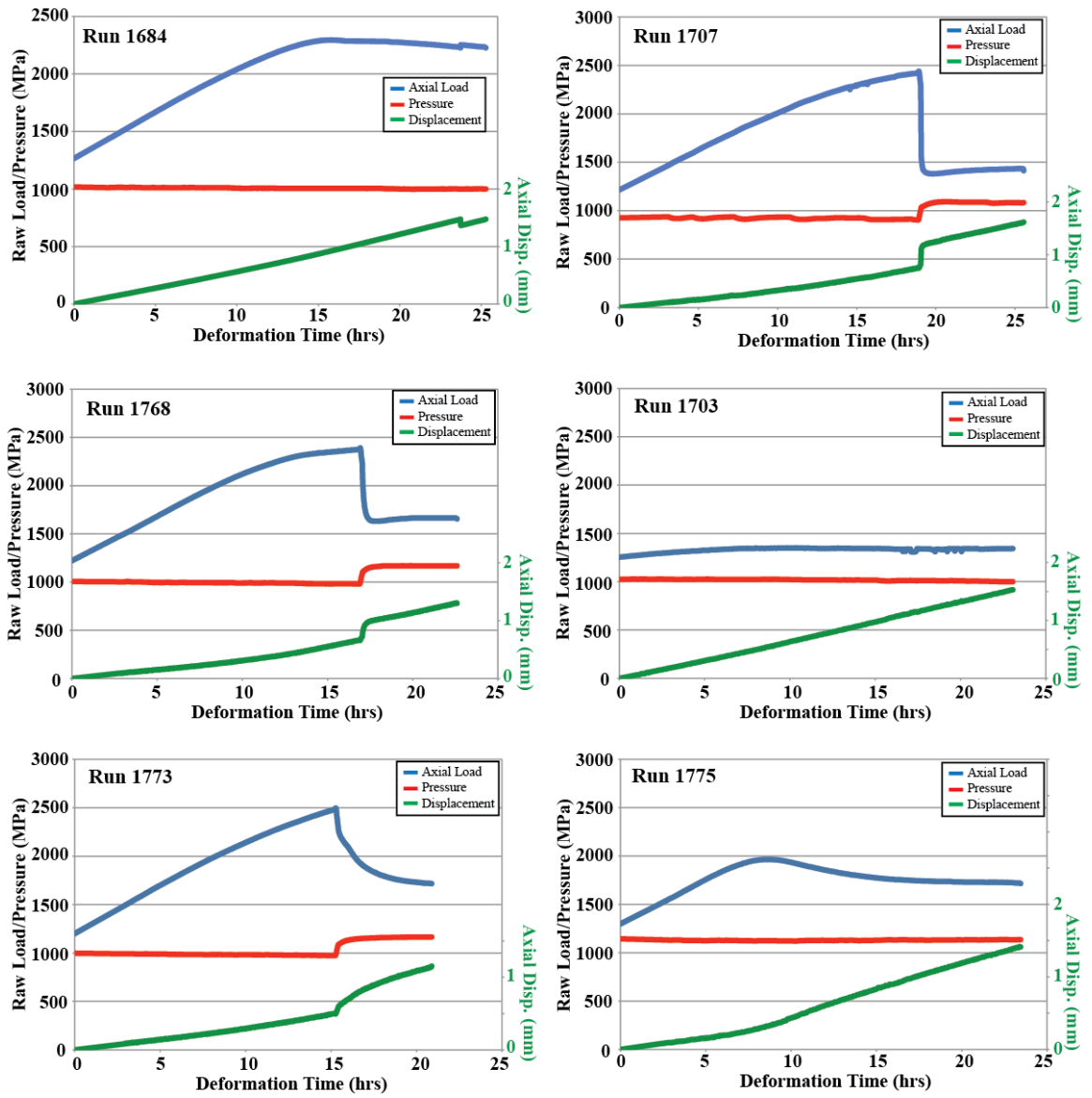


Figure A1

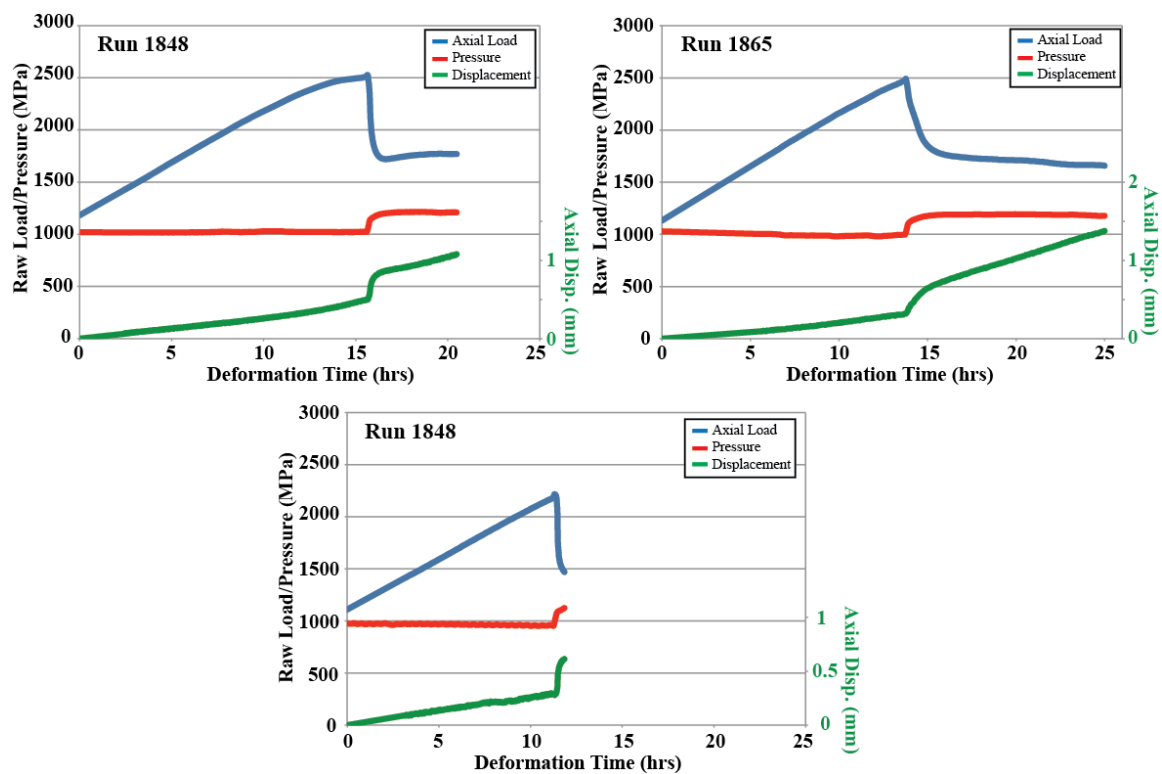


Figure A1 (continued)

Chapter 3

‘Ductile to brittle’ Transition in Thermally Stable Antigorite Gouge at Mantle Pressures

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Abstract

General shear experiments on antigorite-rich serpentinite show a transition from ductile (distributed) to brittle (localized) deformation with increasing temperature from 300 °C to 500 °C at confining pressures from 1 to 2 GPa. The coefficient of friction associated with slip along fractures decreased from 0.23 to 0.07 at 300 °C and 500 °C, respectively. Velocity stepping experiments exhibited a positive rate-dependence (a - b values) that decreased modestly with increasing temperature from ~ 0.015 to ~ 0.01 at 300 °C and 500 °C, respectively. Fractures contained fine-grained foliated antigorite. There was no evidence of dehydration. All samples had a moderate foliation and microstructural evidence for both plastic and brittle deformation mechanisms. The ductile to brittle transition observed for antigorite might explain the occurrence of some seismicity within subduction zones in serpentinitized regions that are apparently too cold to induce dehydration.

1. Introduction

Antigorite (serpentine) is generated in subduction zones as water-rich fluids interact with ultramafic rock within the mantle wedge and subducting mantle [e.g., *Reynard, 2013*]. As the subducting slab warms antigorite metamorphoses to either olivine + talc or olivine + enstatite releasing water in the process. Intermediate-depth earthquakes (50-300 km) within the subducting mantle are generally attributed to this dehydration reaction [e.g., *Peacock, 2001; Hacker et al., 2003; Jung et al., 2004*] based primarily on a correlation between the distribution of hypocenters and predicted phase boundaries and laboratory experiments. However, after numerous experimental studies the direct linkage between serpentine dehydration and seismicity remains unclear [e.g., *Proctor and Hirth, 2015*;

Chernak and Hirth, 2011; Gasc et al., 2011; Rutter et al., 1988]. One complicating observation is that many earthquakes are generated at temperatures too cold for serpentine breakdown [e.g., *Yamasaki and Seno, 2003; Abers et al., 2013*]. One possibility to explain the occurrence of these earthquakes is that serpentine, within its stability field, becomes brittle leading to earthquake nucleation. This hypothesis is motivated by axial compression experiments on talc and antigorite-rich serpentinite that demonstrated a peculiar tendency for strain to become localized with increasing temperature [*Escartin et al., 2008; Chernak and Hirth, 2010*]. In the case of talc, stick-slip events accompanied strain localization at temperatures up to 600 °C. These observations are intriguing because most materials become increasingly ductile and rate-strengthening with increasing temperature. The ‘brittle to ductile’ transition is a manifestation of this behavior and is generally coincident with the onset of aseismic creep [e.g., *Kohlstedt et al., 1995*]. However, antigorite and other strongly anisotropic minerals such as talc might be important exceptions.

Characterizing the rheology of antigorite remains a challenge due to its strongly anisotropic structure and relatively low dehydration temperature. The efficacy of trading off temperature for strain-rate is limited and to date it remains unclear if antigorite can deform by fully plastic flow and, if so, by what rate limiting deformation mechanism(s). At low confining pressures (< 300 MPa) antigorite is undoubtedly brittle at temperatures spanning its stability field (and above) [e.g., *Rutter and Brodie, 1988; Reinen et al., 1994; Escartin et al., 1997; Takahashi et al., 2010; Moore et al., 1997; Hirose and Bystricky, 2007; Okazaki et al., 2013; Okazaki et al., 2015*]. At higher pressures (as experienced in subduction zones), the brittle mechanisms should be increasingly limited owing to the

relative enhancement of ductile and possibly plastic flow. A few experimental studies have concluded that antigorite deforms by macroscopically ductile processes at confining pressures (> 500 GPa) based on observations of distributed (semi-brittle) flow, decreasing friction values with increasing pressure [Raleigh and Paterson, 1965; Escartin *et al.*, 1997] and evidence for grain-scale plasticity [Hilaliret *et al.*, 2007]. Hilaliret *et al.*, 2007 concluded that antigorite deforms by either power law creep or Peierls creep based on experiments conducted in a deformation-DIA apparatus (D-DIA) at pressure up to 4 GPa. More recently, Amiguet *et al.*, 2014 suggested pressure-solution creep in addition to Peierls creep allows for antigorite plasticity. The interpretations by Hilaliret *et al.*, 2007 (and Amiguet *et al.*, 2014) are complicated by the fact that only one sample was deformed for 14 consecutive cycles with different temperatures, strain-rates and pressures imposed for each cycle [see discussion in Chernak and Hirth, 2010]. Conversely, others have argued that antigorite may never achieve full plastic flow based on an insufficient number of dislocation glide planes (i.e., Von Mises criterion not met), a non-determinant Goetze's criterion and presence of semi-brittle microstructures in naturally deformed serpentinites [see discussions in Chernak and Hirth, 2010 and Auzuende *et al.*, 2015].

Nearly all of the previous high-pressure studies on antigorite employed axial-compression loading. One complication of axial compression on highly anisotropic antigorite is that grains progressively rotate into their hardest orientation for dislocation glide (basal cleavage perpendicular to maximum principal stress), which might bias the rheology and microstructure generated during these tests. General shear deformation may provide a better geometry for exploring plasticity because grains are progressively rotated into their weakest orientation for dislocation glide and higher strains can be

achieved. To date only one general shear experiment on antigorite has been reported (with mechanical data) at stable temperatures, mantle pressures and strains high enough to reach a steady-state strength [Proctor and Hirth, 2015]; two other studies have reported general shear experiments without mechanical data [Katayama *et al.*, 2009; Hirauchi and Katayama, 2013]. The microstructural observations alone from those studies are not sufficient to characterize the complex rheology of antigorite.

In this contribution we present results from general shear experiments on antigorite gouge. Our results demonstrate a high-temperature localization phenomenon similar to that observed in previous work, which challenges our understanding of the brittle to ductile transition in antigorite as well as the applicability of crystal plastic flow laws for antigorite in the Earth.

2. Methods

The starting material used in this study is a serpentinite collected from the Nagasaki metamorphic belt in Japan. This rock is composed of ~98 vol% antigorite, with minor amounts of oxides $\pm$ enstatite [Sawai *et al.*, 2013; Okazaki *et al.*, 2013]. The antigorite contains 1.7 wt% aluminum and 6.2 wt% iron [Sawai *et al.*, 2013]. The material was first crushed into a powder and sieved to a grain-size of 37-53 μm . The powder was next magnetically separated achieving >99% purity, washed in distilled water and dried on a hot plate at ~100 °C and stored at room humidity.

The starting material was deformed in general shear geometry with a solid confining media Griggs-type triaxial press [e.g., Tullis and Tullis, 1986]. The samples were sheared between grooved alumina pistons cut at 45° and encased in an undrained silver jacket (Figure 1a) [see Proctor and Hirth, 2015 for assembly details]. Sodium

chloride was used as the confining medium. Prior to deformation, 0.15 grams of powder was first cold-pressed within the assembly at room temperature by gradually increasing the pressure to ~150 MPa over ~30 minutes. The cold-pressed samples were next hot-pressed by gradually increasing temperature and pressure to run conditions (over 2-5 hours) and held hydrostatically for ~12 hours. The pressed gouge was ~1.2 mm thick, had no optically visible inter-granular porosity and had a moderately strong foliation sub-parallel to the shearing plane, as indicated by the continuous extinction color in cross-polarized light with a full wavelength plate inserted (Figure 1b).

In all experiments, general shear was first imposed with a constant axial shortening rate 18 nm/s, that resulted in a shear displacement rate (V) of 25.5 nm/s; this corresponds to a shear strain-rate of $\sim 2.3 \times 10^{-5}/s$ for fully distributed strain across the sample thickness. The confining pressures (σ_3) and temperature (T) were held constant throughout the experiments and were varied from 1 to 2 GPa and 300 °C to 500 °C, respectively. These conditions are all within the antigorite stability field (Figure 1c). All run conditions are shown in Table 1. To quantify the velocity (and strain-rate) dependence of strength, we conducted five velocity stepping experiments during which velocity was varied by up to two orders of magnitude. After the achievement of peak stress in these experiments, the velocity was stepped down from 25.5 nm/s to 2.6 nm/s for ~8 hrs and then down to 0.26 nm/s for ~80 hrs. The velocity was next stepped back up to 2.6 nm/s and 25.5 nm/s for ~8 hrs and ~0.8 hrs respectively.

Axial force, axial displacement and pressure data were converted to axial load (σ_I), shear stress (τ) and shear strain (γ) using methods described in *Proctor and Hirth, 2015*. Load values were corrected for the piston friction, which we assume is constant

for a given displacement rate. A suite of calibration experiments was conducted on sodium chloride and lead at run conditions to measure the rate-dependence of the piston friction (see supplement S1); these values were subtracted from the load values recorded in velocity stepping experiments. In two runs, small fluctuations in pressure (as high as 2 %) occurred due to variations in cooling water temperature. The resolution of stress is ± 25 MPa and the precision is ~ 1 MPa based on previous studies at similar conditions [Chernak and Hirth, 2010; Gleason and Tullis, 1993]. All unprocessed (raw) data are included in the supplemental material (Figure S3).

After deformation, samples were quenched by reducing the axial load and temperature in ~ 10 minutes to achieve a differential stress of ~ 200 MPa and temperature of 100°C . After quenching, the pressure and temperature were returned to ambient conditions over 1-2 hours. For thin-section preparation, the samples were impregnated with epoxy and cut parallel to shearing direction and perpendicular to the shearing plane. Microstructures were analyzed with an Olympus SZX16 optical microscope fit with a digital camera.

3. Results

3.1 Mechanical Data

Shear stress versus shear strain curves are shown for samples deformed at $\sigma_3=1$, 1.5 and 2 GPa and $V=25.5$ nm/s in Figure 2a, 2b and 2c. The corresponding Mohr circles are plotted in Figure 2d; stress values at $\gamma=1$ were used to plot the circles. For a given confining pressure, sample strength decreases with increasing temperature. At 300°C (Figure 2a and 2b), samples exhibit the highest yield stresses (Figure 3) and peak stresses, demonstrate continuous work hardening and show a modest pressure-dependence on τ

and yield stress (Figure 2d and Figure 3). At $\sigma_3=2$ GPa, we could not deform antigorite to its peak strength at 300 °C and 400 °C due to the strength exceeding the axial load limit of the carbide piston (~ 4.1 GPa). At 400 °C and 500 °C, the samples exhibit lower yield stresses (Figure 3), and in some cases reach nominally steady-state shear stresses at high strain (e.g., Figure 2). At 400 °C, gradual strain weakening is observed after the initial peak stress. At 500 °C, the stress drops associated with weakening were larger (in 3 of the 4 runs) and these samples achieve the lowest steady state stress values. For comparison with axial compression experiments we report the equivalent stress values (σ_{eq}) in Figure 4, where $\sigma_{eq} = \tau\sqrt{3}$ [e.g., *Paterson and Olgaard, 2000*].

The apparent coefficient of friction (μ_a) was calculated using the tangent approach by fitting a failure slope to Mohr circles (Figure 2d). μ_a values are ~ 0.23 , ~ 0.13 and ~ 0.07 with an apparent cohesion of 500-600 MPa for samples deformed at 300 °C, 400 °C and 500 °C respectively (Figure 2d). At lower confining pressures (< 200 MPa) antigorite gouge has higher friction values, ranging from ~ 0.45 to ~ 0.75 , and no cohesion (see summary in *Okazaki et al., 2013*). At 300 °C the shear strength is consistent with the failure envelope determined in room temperature experiments (grey line, Figure 2d), however at higher temperatures the friction slope decreases to almost zero.

The velocity dependence of friction was explored in five runs using velocity stepping tests. The velocity dependence was quantified as, $\delta\mu/\ln\delta V$, commonly parameterized as $a-b$ in rate and state friction [e.g., *Scholz, 1998*]. The $a-b$ values were positive in all steps, but decreased modestly with increasing temperature, coincident with decreasing strength values (Figure 4a). For velocity stepping tests, we calculate a friction coefficient (μ_β) as function of normal stress (σ_n) assuming $\mu_\beta = \tau/\sigma_n$ where $\tau = 1/2 (\sigma_1 -$

σ_3) and $\sigma_n = 1/2 (\sigma_1 + \sigma_3)$ because deformation was imposed on a sample inclined 45° to σ_1 [e.g., *Jaeger et al.*, 2007]. Cohesion was ignored for this part of the analysis, because we are interested in the relative change in friction, and consequently, the values of μ_β values are higher than μ_α . For each stepping interval, μ_β values evolved to a nominal steady-state friction (μ_{nss}); an average value was calculated over these intervals (see supplement A2). The average μ_{nss} values were corrected for the rate-dependence of the piston friction; the corrections were up to $\sim 7\%$ of the observed changes in friction (Figure A1).

The log stress versus log strain-rate values are shown in Figure 4b for run 1992. In this plot, the shear stress and shear strain-rate values were converted to σ_{eq} and equivalent strain-rate ($\dot{\epsilon}_{eq}$), where $\dot{\epsilon}_{eq}$ = shear strain rate divided by $\sqrt{3}$ [e.g., *Paterson and Olgaard*, 2000]. Stress values were corrected for the rate-dependent friction of the loading piston and strain-rate values are reported as an ‘applied strain-rate’ (i.e., we assume strain was fully distributed). In Figure 4b, we also plot the predicted strengths for both the power law creep and Peierls creep flow laws for the same deformation conditions imposed in run 1992 ($T=500^\circ\text{C}$ and $\sigma_3=1.5\text{ GPa}$). This comparison demonstrates that the rate dependence for samples deformed in this study is almost an order of magnitude lower (i.e., higher slope, n) than predicted by the flow laws: $n=3.8$ for power law creep versus $n=34$ for run 1992.

3.2 Microstructures

At all conditions tested antigorite deformed by a combination of slip along fractures and crystal plasticity. Interestingly, at higher temperatures strain was more localized than observed at lower temperatures (Figure 5). At 300°C the samples were

nominally ductile with strain accommodated via a network of anastomosing shear fractures. The fracture networks cut across the gouge zone from the upper to lower corner near the sample-piston contact, at an angle of 34°-37° to σ_1 (e.g., Figure 5). All fracture orientations are listed in Table 1. The fractures thickness ranged from 1-15 μm and contained finer-grained ($< 2 \mu\text{m}$) well-foliated antigorite. Likewise, the regions adjacent to the shear fractures had a noticeably stronger lattice preferred orientation (LPO) than the rest of the sample (Figure 6a). We also observed cleavage parallel micro-cracks and textures indicative of intra-granular plasticity- including undulose extinction and kinking - consistent with previous studies [e.g., *Chernak and Hirth*, 2010; *Amiguet et al.*, 2014; *Auzende et al.*, 2015]. We found no textural evidence for dynamic recrystallization or dehydration.

At 500 °C strain was fully localized along one prominent shear fracture that either cut across the gouge zone at up to 35° to σ_1 (e.g., Run 1937, Figure 5) or at 45° along piston-sample interface (e.g., Run 1979 and 1976, Figure 5). The higher fracture angle is consistent with the nearly zero friction coefficients reported for the “tangent method” above (e.g., Figure 2d). The localized shear fractures were observed in samples deformed at 1, 1.5 and 2 GPa with the lower angle (relative to loading direction) fractures occurring in samples deformed at lower confining pressures. The fractures ranged in thickness from 5-20 μm and contained finer-grained ($< 2 \mu\text{m}$) well-foliated antigorite, similar to distributed shear fractures that developed at 300 °C. In some places, the fabric within the fractures showed a sigmoidal “mica fish” texture (Figure 6b), which is common in sheared micaceous rocks. Adjacent to the localized shear fracture, grains with their cleavage planes oriented parallel to the fracture exhibit cleavage parallel micro-

cracks. Similar to the sample deformed at 300 °C, grains exhibit undulose extinction and kinking, and no evidence for dehydration or dynamic recrystallization. Samples deformed at 400 °C had intermediate textures and fracture orientations between those at 300 °C and 500 °C (see Table 1 and Figure A3).

4. Discussion

4.1 Non-typical “ductile to brittle” transition

Our results demonstrate that antigorite does not follow a typical brittle to ductile transition (BDT). As a rule of thumb with increasing pressure (for a given temperature) the BDT occurs when the fracture strength (or bulk strength) becomes less than the strength required for frictional sliding [e.g., *Byerlee*, 1967; *Kohlstedt et al.*, 1995]; at this transition, deformation is typically accommodated by distributed (semi-brittle) flow that is modestly pressure-insensitive [e.g., *Evans et al.*, 1990]. Increasing temperature also promotes ductility because crystal plastic deformation mechanisms are strongly temperature dependent. At temperatures ≤ 300 °C, dry antigorite exhibits a ‘typical’ BDT with increasing pressure. This behavior is demonstrated by our results at 300 °C and previous experiments conducted at room temperature (e.g., Figure 2d) [*Okazaki et al.*, 2013 and *Escartin et al.*, 1997]. However, at temperature > 300 °C, antigorite strength decreases and strain localizes, indicating a transition back to ‘brittle’ or localized deformation (Figure 2d). Thus, the BDT inverts with increasing temperature, counter to the behavior of many silicate minerals. *Chernak and Hirth*, 2010 recognized this phenomenon from axial compression experiments on antigorite at similar conditions. Following interpretations of similar observations for talc [*Escartin et al.*, 2008], *Chernak and Hirth*, 2010 proposed that the inverted BDT transition in antigorite arises because the

coefficient of friction has a greater temperature-dependence than the bulk strength, which is unusual in its own right [e.g., *Byerlee*, 1978]. Nonetheless, our results are consistent with this hypothesis (e.g., Figure 2d); when temperature is increased, friction drops and strain localizes on faults oriented $\sim 45^\circ$ to the maximum stress orientation.

It remains unclear why the coefficient of friction has an apparently strong temperature-dependence. One possibility is that this behavior is a manifestation of the extreme mechanical anisotropy of the crystal. In adhesion theory, the coefficient of friction is the ratio of the contact shear stress over the penetration hardness for a shearing asperity [e.g., *Scholz*, 2002]. For antigorite slip surfaces that are oriented parallel to the basal cleavage, asperity contacts will deform more easily in the shearing direction because the easy glide planes are oriented parallel to the basal cleavage. Increasing temperature may then preferentially reduce the contact shear stress (compared with the penetration hardness) by enhancing dislocation glide thus reducing the coefficient of friction and promoting slip [e.g., *Hirth and Guillot*, 2013]. The fact that grain-scale fractures are almost always observed parallel to the basal cleavage is consistent with this hypothesis [e.g., *Escartin et al.*, 1997; *Hilairet et al.*, 2007; *Chernak and Hirth*, 2010; *Proctor and Hirth*, 2015; *Auzende et al.*, 2015].

Kinking is a possible source for microcracks in deforming antigorite. Kink bands are commonly observed in deformed antigorite at pressures as high as 4 GPa [e.g., this study, *Chernak and Hirth*, 2010; *Boudier et al.*, 2010; *Amiguet et al.*, 2014; *Auzende et al.*, 2015] as well as within other micas [e.g., *Shea and Kronenberg*, 1992]. Kink bands are nominally caused by dislocation pile-ups along the basal cleavage [e.g., *Bell et al.*, 1986; *Christofferson and Kronenberg*, 1993; *Shea and Kronenberg*, 1992]. During kink

formation, cleavage parallel microcracks are nucleated to accommodate for lattice tilting associated with the kink [e.g., *Bell et al.*, 1986; *Christofferson and Kronenberg*, 1993; *Auzende et al.*, 2015]. Microcracks may also form without kinking due to interacting dislocations on adjacent glide planes [*Christofferson and Kronenberg*, 1993]. Because dislocation glide is promoted with increasing temperature, kinking and microcrack formation should also be enhanced with increasing temperature. Microcrack growth might be enhanced at higher temperatures due to the decreasing frictional resistance discussed above, which could account for the development of localized fractures.

We do not think that the onset of microcracks and strain localization at high temperature in our samples results from dehydration. Our experiments were conducted at temperature well below the lowest temperatures reported for dehydration of dry antigorite at pressures ≥ 1 GPa (~ 550 °C) [*Perrillat et al.*, 2005; *Chernak and Hirth*, 2010] and we did not observe significant weakening that has been observed with even a very small amount of reaction (< 1 vol%) [*Chernak and Hirth*, 2010; *Chernak and Hirth*, 2011; *Proctor and Hirth*, 2015; *Okazaki and Katayama*, 2015]. The range of temperatures where dehydration-induced weakening is observed are shown in Figure 3. One mechanism capable of generating dehydration at otherwise stable conditions is plastic faulting [e.g., *Renshaw and Schulson*, 2013]. The plastic faulting model could explain the occurrence of fracturing at $\sim 45^\circ$, however it seems unlikely this mechanism is occurring in our experiments. We did not observe velocity-weakening ($a-b < 0$) behavior or large stress drops during fracturing, which are implied for plastic faulting. Similarly, the opening of cracks might induce local dehydration due to the ‘low-pressure’ surface generated as the crack opens. However, *Auzende et al.*, 2015 found no evidence for

reaction products within microcracks in antigorite samples deformed at similar conditions used in this study.

4.2 Plastic flow in antigorite?

The experiments we ran were designed to maximize the possibility for demonstrating fully plastic flow of antigorite; tests were conducted at the highest confining pressure (to suppress fractures) and the lowest practical strain rates and temperatures (to promote crystal plasticity). Nonetheless, we found evidence for brittle deformation at all conditions tested. In addition, our results show higher strengths and a much lower strain rate dependence than predicted by previously published flow laws for antigorite (e.g., Figure 4b), consistent with previous work [*Chernak and Hirth*, 2010; *Auzende et al.*, 2015]. No study to date has replicated the strengths predicted by the flow laws. *Amiguet et al.*, 2014 proposed that pressure-solution creep might accompany Peierls creep at natural strain-rates. This hypothesis is difficult to test experimentally due to sluggish dissolution rates.

Despite the uncertainty regarding the applicability of the flow laws, there is no doubt that antigorite deforms at least partially by plastic deformation mechanisms. Many studies on natural and experimentally deformed antigorite-rich serpentinites report strong lattice preferred orientations (LPOs) [*Katayama et al.*, 2009; *van de Moortele et al.*, 2010; *Padron-Navarta et al.*, 2012; *Hirauchi and Katayama*, 2013], which are generally consistent with the operation of the easy slip systems [e.g., *Amiguet et al.*, 2014], as well as kinking and undulose extinction [e.g., this study; *Chernak and Hirth*, 2010; *Boudier et al.*, 2010; *Amiguet et al.*, 2014]. Overprinting mineral textures are also observed in natural samples, indicative of dissolution-precipitation style reactions consistent with

pressure-solution creep [e.g., *Auzende et al.*, 2015]. However, fabrics alone are not proof that the entire body is deforming plastically; for example, all of the samples deformed in this study had LPOs (as indicated by optical effects). Although it is difficult to determine the rheology of antigorite at natural strain-rates, the fact that earthquakes occur within the antigorite stability field in subduction settings [e.g., *Abers et al.*, 2013; *Yamasaki and Seno*, 2003] suggests that fully plastic flow of serpentinite is also limited at geologic conditions by brittle processes.

4.3 Geophysical implications

The inverted BDT in antigorite may provide new insight into the source of seismicity in serpentinitized rocks within subduction zones. Earthquakes are typically observed within the subducting lithosphere over a range of pressures and temperatures, some of which correspond to conditions conducive for serpentine dehydration [e.g., *Yamasaki and Seno*, 2003; *Hacker et al.*, 2003; *Abers et al.*, 2013]. Within the mantle wedge, slow earthquakes (slow slip events and tremor) are observed at locations in warmer subduction zones that are likely too cold for dehydration reactions [e.g., *Obara*, 2002; *Shelly et al.*, 2006]. Many studies have focused on understanding the link between dehydration and seismicity [see summary in *Proctor and Hirth*, 2015]. However, our results highlight another unique thermal region that may also generate earthquakes in colder serpentinite *not undergoing dehydration*. We find that at 400-500 °C antigorite becomes brittle and has a relatively low velocity-dependence that apparently decreases with increasing temperature (Figure 4a). For temperatures less than 400°C and greater than 700°C, antigorite is ductile and more velocity-strengthening and thus, less likely to generate earthquakes (Figure 4a). Experiments on partially dehydrated antigorite

demonstrate a linear viscous rheology [*Rutter and Brodie, 1988; Arkwright et al., 2008*] possibly caused by pressure-solution creep of olivine [*Proctor and Hirth, 2015*].

We speculate earthquakes could be generated in antigorite undergoing a brittle transition at 400-500°C. We acknowledge that the a - b values reported in this study are still positive, which will inhibit unstable frictional sliding. However, the brittle and localized nature of deformation in these rocks can promote dynamic weakening mechanisms, such as flash weakening [*Proctor et al., 2014; Platt et al., 2015*]; these processes may operate even when the quasi-static frictional behavior is velocity strengthening [*Brantut and Sulem, 2012*]. The presence of pore water might also induce unstable slip. In low confining pressure experiments, *Okazaki and Katayama, 2015* showed that antigorite gouge becomes velocity weakening ($a-b < 0$) at temperatures close to the dehydration temperature for samples deformed at hydrothermal conditions.

5. Conclusions

We question the idea that strain localization or embrittlement only occurs in antigorite-rich serpentinite during the onset of dehydration. Our results demonstrate that there is a distinct transition from ductile to brittle deformation with increasing temperature at temperatures well below the antigorite reaction boundary. Fractured serpentinite has a low velocity dependence that decreases with increasing temperature. This ‘high-temperature’ embrittlement is consistent with previous work and may explain the occurrence of seismicity in serpentinitized regions of subduction zones too cold for dehydration.

Appendix A

The piston friction was measured by conducting experiments on halite and lead. In Figure A1 we present results from velocity stepping experiments on halite (black symbols) and lead (grey symbols) at pressure and temperature conditions used in this study. The strengths of both materials at these conditions is on the order of a few MPa, thus the measured stress values indicate the frictional resistance of the loading piston. This plot shows that the piston friction is invariant with temperature and increases non-linearly with increasing loading velocity. The fit curve was used to correct for the change in piston friction during velocity stepping tests.

In Figure A2 the friction versus time is shown for a velocity stepping experiment. The imposed shearing velocity is indicated by the shaded regions. The change in nominal steady-state friction is noted with dashed lines.

In Figure A3 we present all unprocessed data.

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Table 1. Experimental details.

Figure 1. Sample assembly and run conditions. a) Schematic diagram of sample assembly: shearing velocity (V), axial load (σ_1) and confining pressure (σ_3) are noted. A

silver jacket and platinum disks (grey lines) surround alumina shear pistons and sample (see *Proctor and Hirth, 2015* for full description). b) Photomicrograph in cross-polarized light with full wavelength (gypsum) plate inserted of hydrostatic experiment at $\sigma_3 = 1.5$ GPa (Run 1973); continuous yellow coloring indicates a preferred orientation within the compacted gouge; dark regions are air bubbles. c) MgO-SiO₂-H₂O phase diagram after *Ulmer and Trommsdorf, 1995* (solid lines) with modified boundary (dashed lines) by *Perrillat et al., 2005* for dry antigorite (low water activity); stars indicate experiments conducted in this study.

Figure 2. Mechanical data for samples deformed at confining pressure of ~1 GPa (a), ~1.5 GPa (b) and ~2 GPa (c); shearing velocity was 25.5 nm/s for all runs. Run numbers are noted in black. d) Mohr circle plot for strength at $\gamma = 1$ from experiments shown in figures a, b and c. The slopes of tangential dashed lines (noted in bold black) indicated the apparent coefficient of friction for given temperature. Bold grey line indicates the room temperature failure envelope with coefficient of friction values (black) from *Okazaki et al., 2013* and *Escartin et al., 1997*.

Figure 3. Yield stress values versus confining pressure. Here the yield stress is the shear stress value at the onset of non-linear loading.

Figure 4. a) Evolution of strength and rate-dependence with temperature. The light gray band indicates the nominal steady-state strength. Equivalent stress values (see text) reported from this study (black symbols) at $\gamma = 1$ and $\sim 10^{-5}$ /s strain-rate, axial

compression experiments (green symbols) from *Chernak and Hirth*, 2010 at 20% axial strain and $\sim 10^{-5}$ /s, general shear experiments (blue symbols) from *Proctor and Hirth*, 2015 at $\gamma = 1$ and $\sim 10^{-5}$ /s with undrained (U) and drained (D) conditions. The dark gray band indicates weakening observed during temperature ramping experiments [*Chernak and Hirth*, 2010; *Proctor and Hirth*, 2015]. Antigorite flow law values for Peierls creep and power law creep were calculated with values reported in *Amiguet et al.*, 2012 for 10^{-5} /s strain-rate and 1.5 GPa pressure. Dashed lines separate results into either ductile or brittle regimes based on microstructures. Open symbols indicate rate-dependence (*a-b*) values for velocity stepping tests (see text for details). b) Log equivalent stress versus log equivalent strain rate plot; values from this study are from run 1992 ($T=500$ °C, $\sigma_3=1.5$ GPa); values for power law creep and Peierls creep [*Hilairet et al.*, 2007] were calculated for $T=500$ °C and $\sigma_3=1.5$ GPa and using equation parameters reported in *Amiguet et al.*, 2012. The samples deformed in this study exhibit a much larger slope than predicted by the flow laws.

Figure 5. Sample microstructures. Photomicrographs of samples cut perpendicular to the shearing direction. Solid and dashed red lines highlight prominent and poorly developed fractures respectively. Arrows note shearing direction. This figure shows that strain becomes more localized with increasing temperature (moving to the right) for all pressures tested.

Figure 6. Photomicrographs in cross-polarized light with the full wavelength plate inserted from Runs 1988 (panel a) and Run 1979 (panel b). a) Image shows distributed

network of fractures and well developed foliation indicated by continuous coloring across the sample in Run 1988; $T=300\text{ }^{\circ}\text{C}$, $\sigma_3=1.5\text{ GPa}$. Some fractures contained air bubbles trapped within the epoxy; these appear black. b) Enlargement of fracture containing 1-10 μm foliated antigorite in Run 1979; $T=500\text{ }^{\circ}\text{C}$, $\sigma_3=1.5\text{ GPa}$.

Figure A1. Rate-dependent machine friction (friction external to the sample). Results are reported from experiments on lead *Okasaki et al.*, (in prep) at similar conditions and one experiment on sodium chloride (this study). Dashed line traces the best fit through the data. At low axial velocities the positive rate-dependence is very modest, 3-5 MPa for an order of magnitude change in velocity.

Figure A2. Characteristic velocity stepping experiment; Run 1994 (see text for details).

Figure A3. Unprocessed axial load and confining pressure data (refer to Table 1 for run conditions). Load values assume axial force is applied on a 6.35 mm diameter cylinder. Axial displacement values were corrected for the apparatus stiffness and sample shortening.

Table 1.

Run Number	Average σ_3 (GPa)	Shear Velocity (nm/s)	Temp. (°C)	Total Shear Strain (γ)	Yield Stress (MPa)	Equivalent stress (GPa); $\gamma=1$	Strain Distribution; Fracture(s) Angle to σ_1
1982	1.009 $\pm$ 0.005	25.5	300	1.5	499	1.72	dist.; ~34°
1801	0.994 $\pm$ 0.014	25.5	400	2.69	504	1.33	trans.; 32-45°
1986	1.027 $\pm$ 0.003	25.5, 2.55, 0.255	400	1.69	481	1.38	loc.; 35-45°
1937	0.987 $\pm$ 0.008	25.5	500	2.54	401	1.2	loc.; 35-45°
1989	0.998 $\pm$ 0.004	25.5	500	1.63	371	1.21	loc.; 37-45°
1988	1.498 $\pm$ 0.006	25.5	300	1.79	681	1.96	dist.; ~35°
1996	1.494 $\pm$ 0.003	25.5, 2.55, 0.255	300	1.81	709	1.93	dist.; ~37°
1973	1.533 $\pm$ 0.006	NA, hydrostatic	400	0	NA	NA	NA
1802	1.470 $\pm$ 0.013	25.5	400	2.41	613	1.47	trans.; ~38°
1994	1.496 $\pm$ 0.003	25.5, 2.55, 0.255	400	1.46	619	NA	loc; 45°
1979	1.490 $\pm$ 0.008	25.5	500	1	489	1.22	loc; 45°
1992	1.498 $\pm$ 0.002	25.5, 2.55, 0.255	500	1.2	522	NA	loc; 45°
1959	1.905 $\pm$ 0.015	25.5, 2.55	400	1.34	800	NA	trans.; ~37°
1976	1.952 $\pm$ 0.01	25.5	500	1.45	703	1.46	loc.; 45°

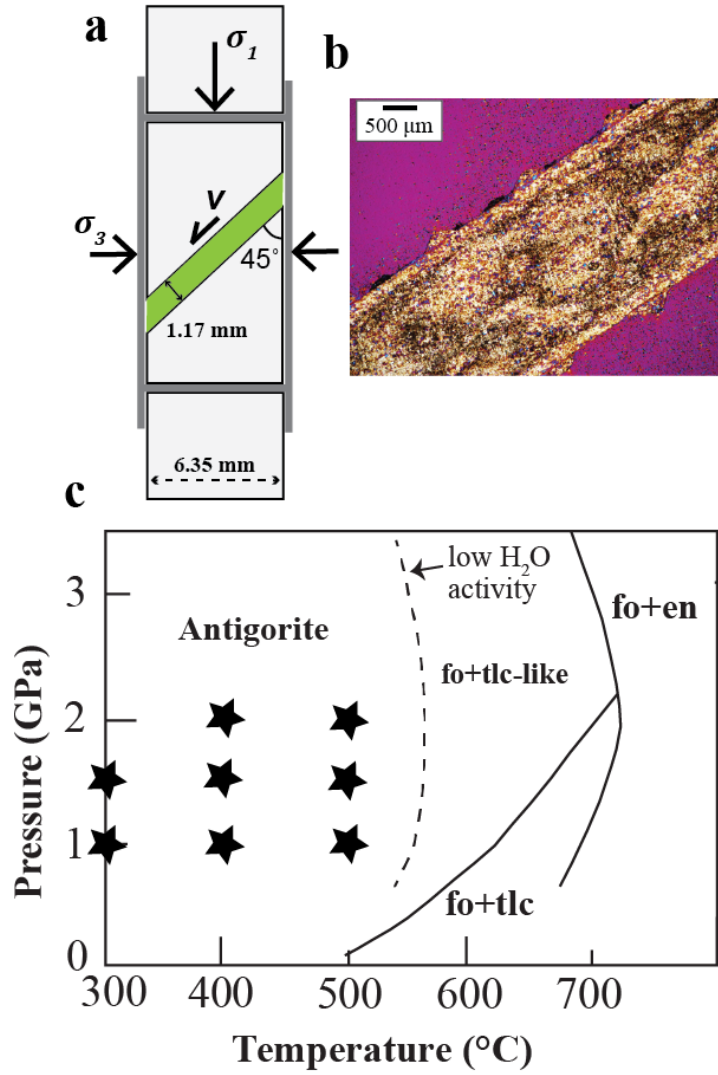


Figure 1.

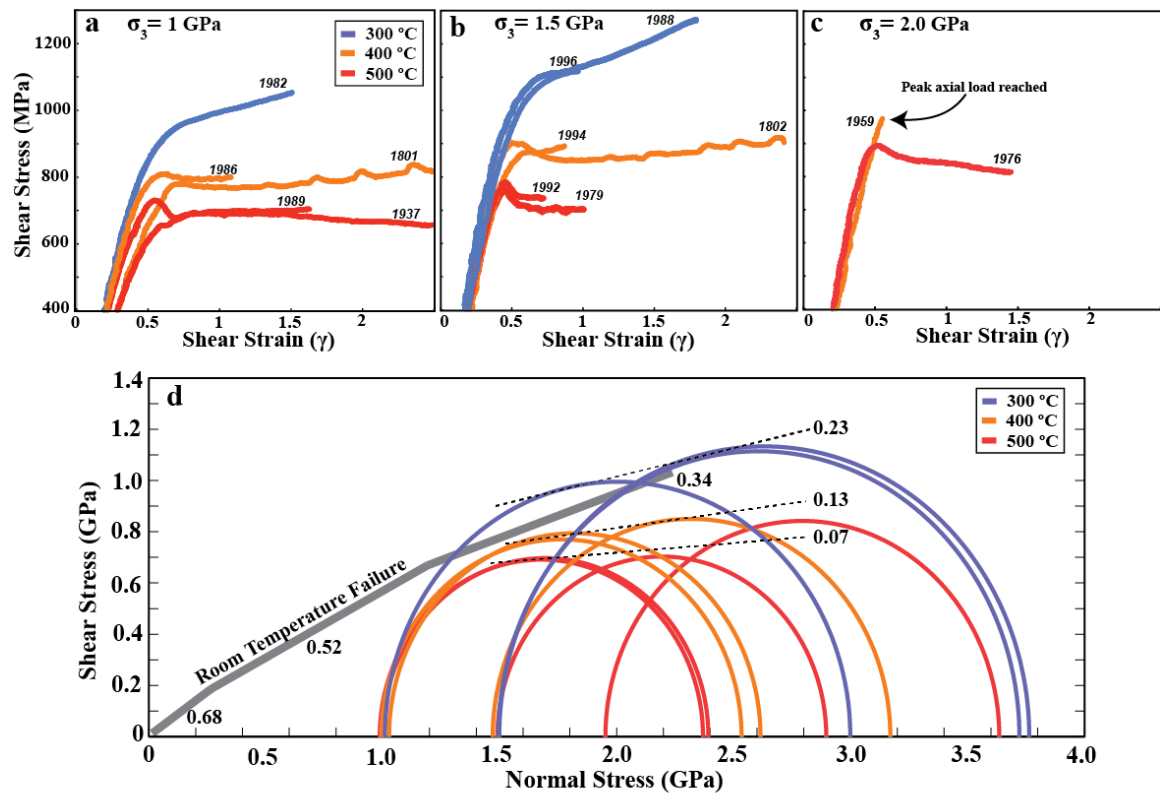


Figure 2

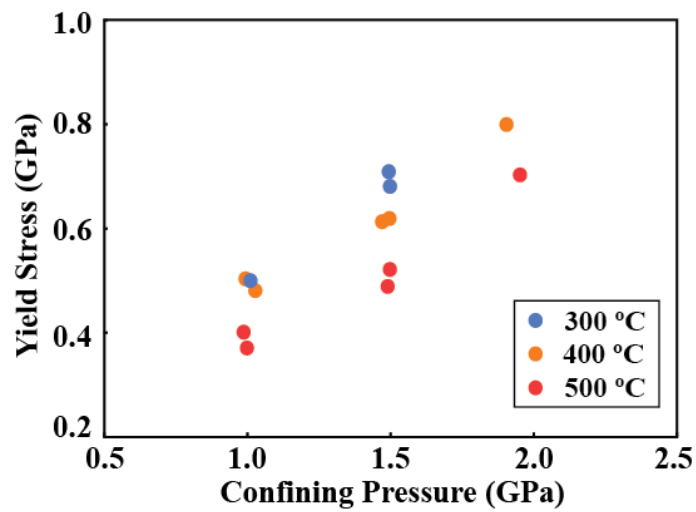


Figure 3

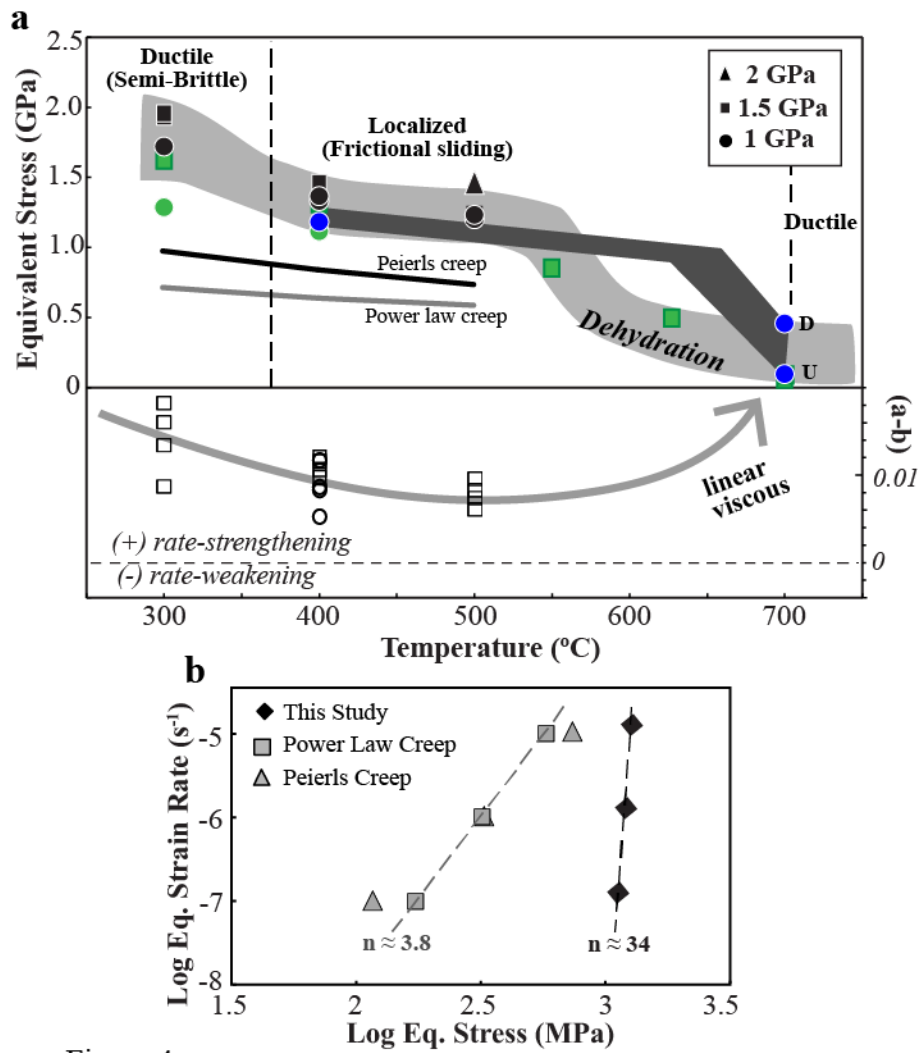


Figure 4

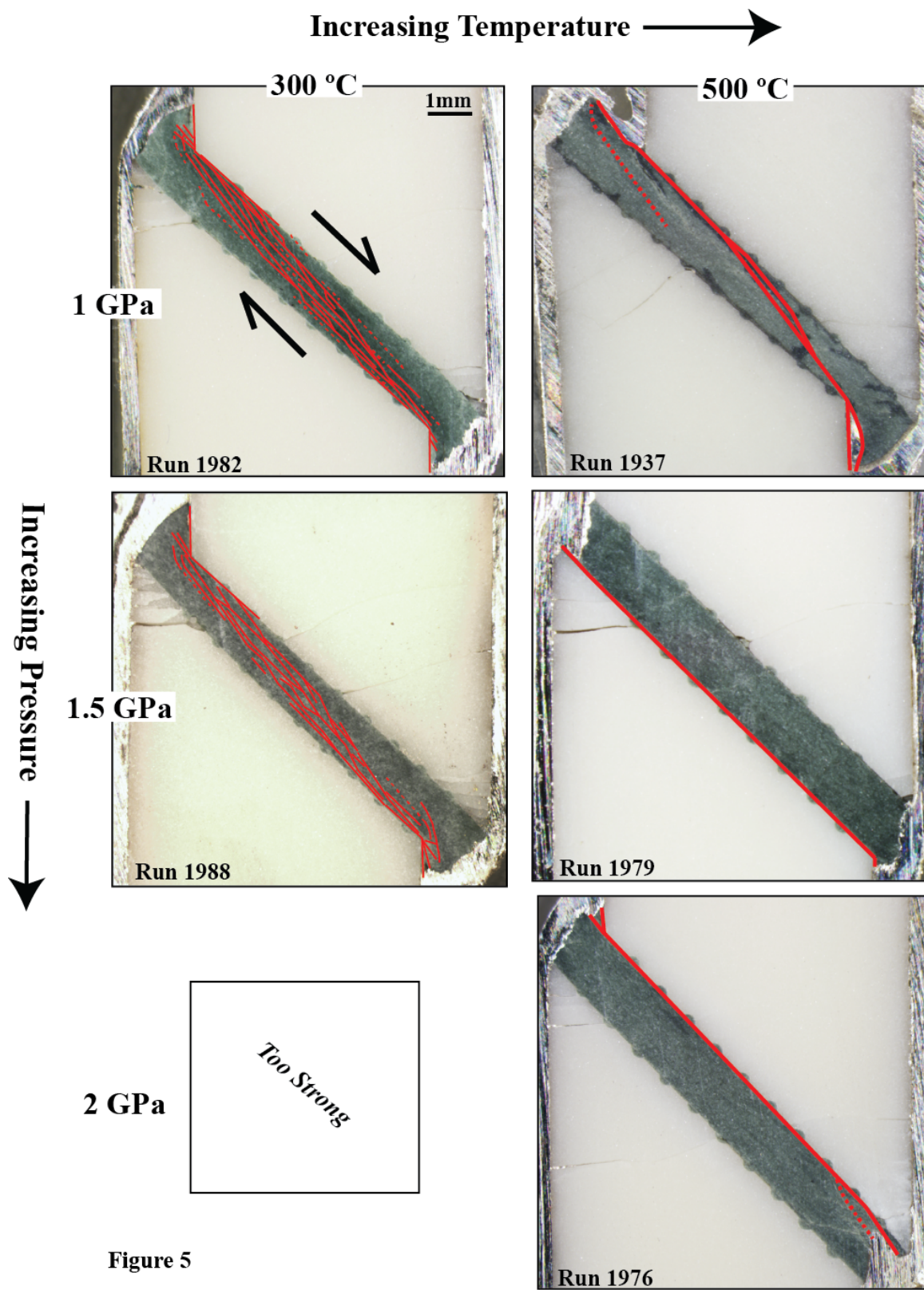


Figure 5

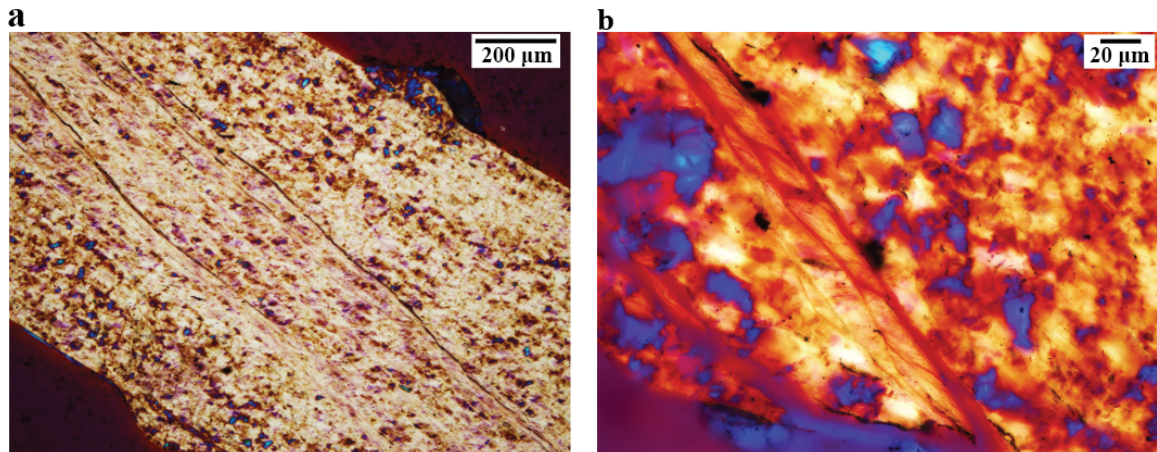


Figure 6

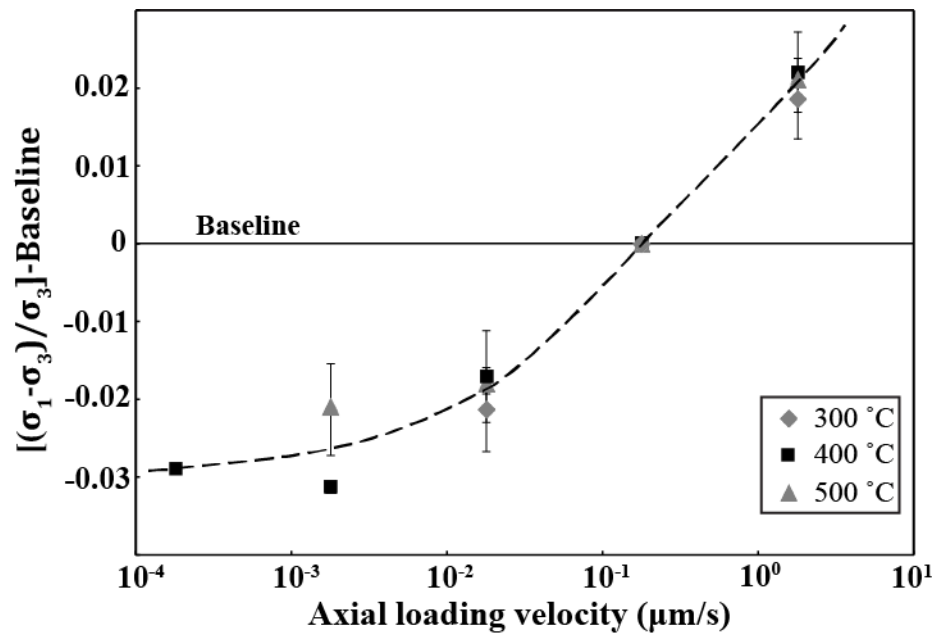


Figure A1

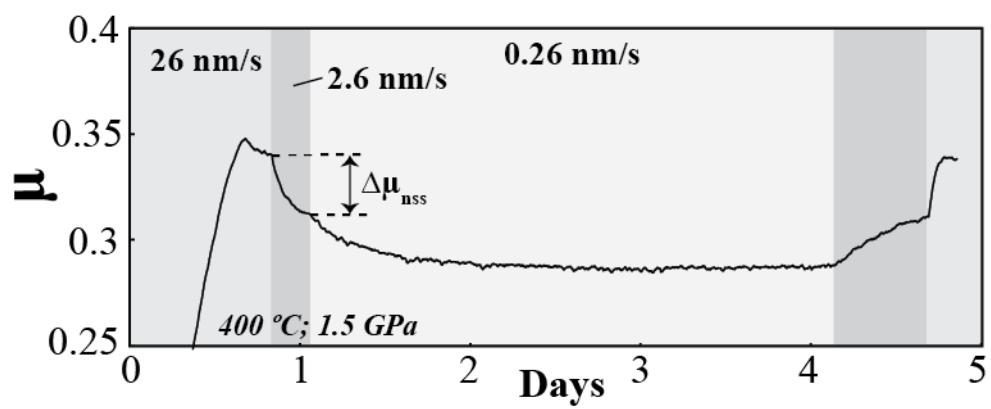
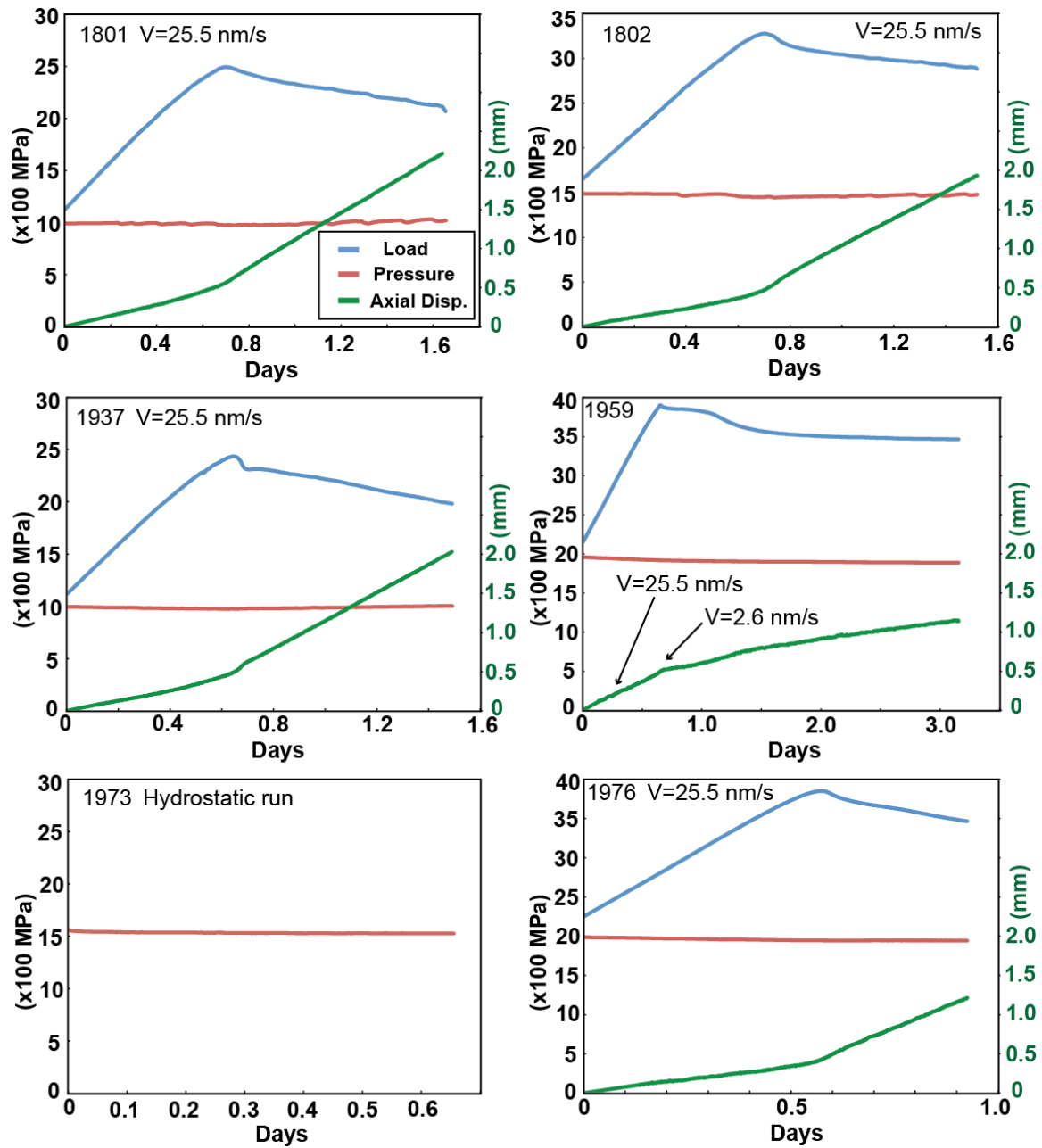
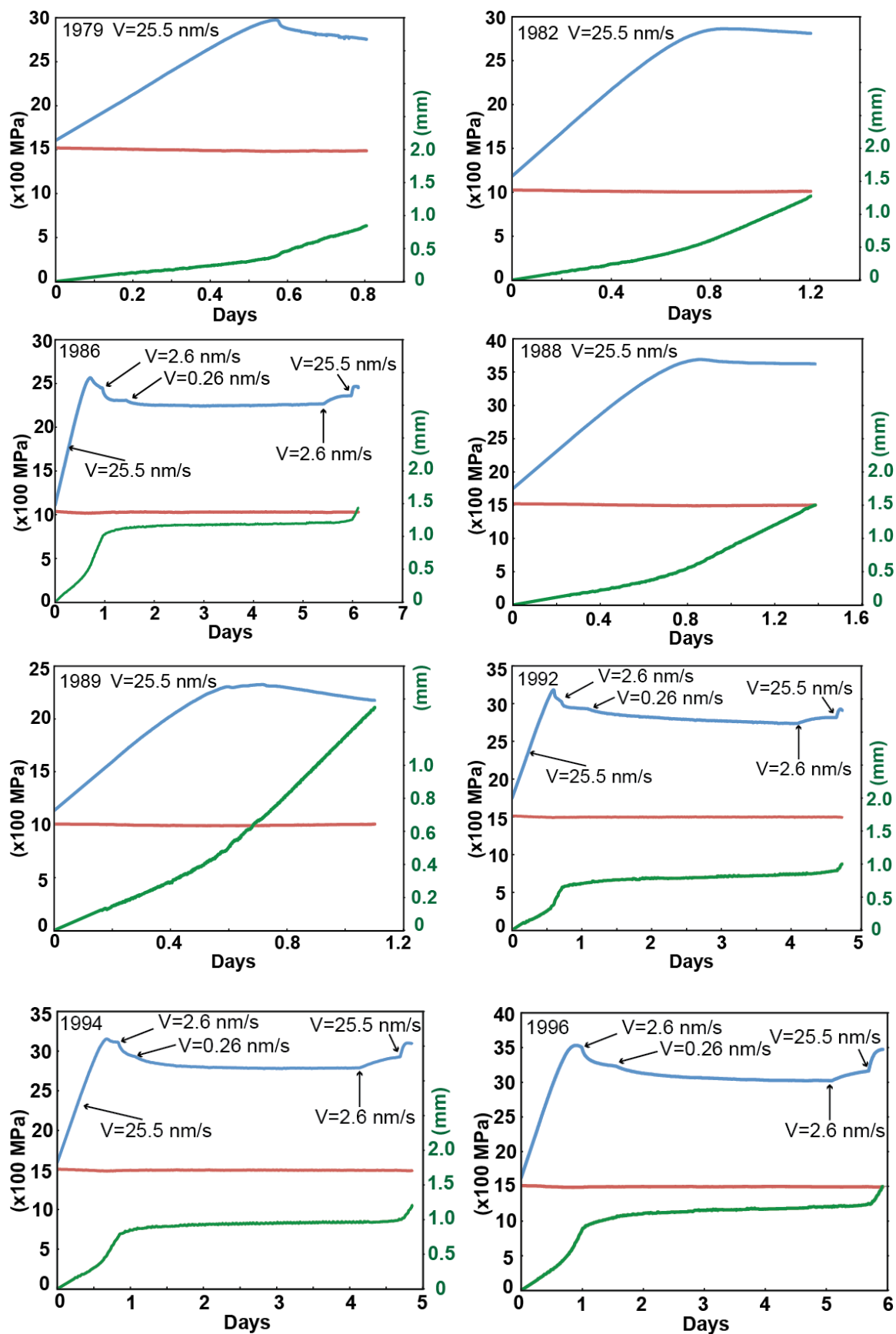


Figure A2

Figure A3





Chapter 4

Talc-Induced Pressure-Solution in San Carlos Olivine

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Abstract

Prograde metamorphism of antigorite-rich serpentinite within subduction zones is hypothesized to cause earthquakes, nucleate slow-slip and tremor events, and affect slab/wedge coupling and corner flow. Despite its importance, relatively little is known about the rheology of the olivine and talc-rich rocks produced during serpentine dehydration. Recent experiments by *Proctor and Hirth, 2015* at 700 °C and 1 GPa suggest actively reacting samples deform partly by pressure-solution, a deformation mechanism not thought to exist within these rocks, allowing olivine to deform ductily at lower stresses than previously thought. Motivated by these findings, we conducted general shear experiments on San Carlos olivine and fully dehydrated serpentine at 700 °C and 1 GPa. Our results show that the presence of talc is required to promote flow of olivine at these conditions.

1. Introduction

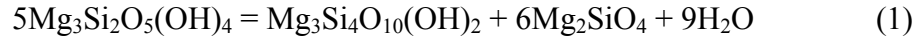
The antigorite breakdown reaction plays an important role in many subduction zones processes. For example, the reaction is hypothesized to directly or indirectly cause large earthquakes [e.g., *Davies, 1999; Hacker et al., 2003b; Kirby et al., 1996; Peacock, 2001*], low-frequency earthquakes, tremors and slow-slip events [e.g., *Brown et al., 2009; Schwartz and Rokosky, 2007*] and initiate slab/wedge coupling, thus affecting corner flow [e.g., *Furukawa, 1993; Peacock, 2003; Wada et al., 2008*]. In chapter 2 we explored the transient rheologic changes associated with the onset of antigorite dehydration. In that study two experiments were conducted to explore the rheology of mostly dehydrated serpentinite (i.e., most of the sample was composed of reaction products). These experiments demonstrated that (1) pore fluid pressure had a significant effect on the

overall strength and (2) olivine apparently deformed plastically at relatively cold (700 °C) and wet conditions. The olivine textures reported in chapter 2 [now published in *Proctor and Hirth, 2015*] were perplexing because plasticity should not be possible at these temperatures and stresses based on the current understanding of olivine rheology [see discussion in *Proctor and Hirth, 2015*]. We suggested pressure-solution creep (P-S) maybe contributing to the overall deformation, based on the morphology of the deformed grains and the fact that sample was saturated with water. However, it remains unclear if water alone can promote low stress deformation (at these conditions), as well as how talc effects the overall rheology of a rock composed of talc and olivine. Talc is an extremely weak phase that may mechanically weaken the rock just by being present [e.g., *Escartin et al., 2008; Moore and Rymer, 2007*]. Talc may also enhance P-S creep, and hinder grain-growth by pinning olivine grain boundaries. Previous work by *Hickman and Evans, 1995* demonstrated that mica grains placed between sodium chloride and silica dramatically enhances dissolution (pressure-solution) of halite along stressed contacts. We hypothesize that a similar process may occur between talc and olivine in the presence of water.

We conducted general shear experiments on pure olivine aggregates and fully dehydrated serpentinite samples using experimental techniques developed in *Proctor and Hirth, 2015*. These experiments indicate that talc is the primary factor promoting low-temperature and low-stress olivine deformation. We also show that talc and olivine, in the presence of a high pore pressure, are stable to temperatures much higher than previously observed.

1.1 Antigorite dehydration

The antigorite breakdown reaction varies as a function of pressure for a given temperature (Figure 1). At lower pressures (< 1.5 GPa) antigorite reacts to talc, forsterite (Mg-endmember olivine) and water:



[e.g., *Deer et al.*, 1996; *Spear*, 1995]. This reaction releases ~12 wt% water and produces a rock composed of ~60/40 vol% forsterite and talc [e.g., *Hacker et al.*, 2003a]. Up to 0.1 wt% OH can dissolve into olivine at 700 °C and 1 GPa [e.g., *Mosenfelder et al.*, 2006]. At higher temperatures talc and forsterite react to form enstatite (Mg-endmember orthopyroxene):



[e.g., *Spear*, 1994]. This reaction releases ~2 wt% water and produces a rock composed of ~50/50 vol% forsterite and enstatite [e.g., *Hacker et al.*, 2003a]. The pressures and temperatures (P-T) at which reaction 2 occurs are well established [e.g., *Spear*, 1995; *Ulmer and Trommsdorff*, 1995; *Perrillat et al.*, 2005]. At higher pressures (> 1.5 GPa), talc becomes energetically unfavored and antigorite reacts directly to forsterite and enstatite [e.g., *Spear*, 1995; *Ulmer and Trommsdorff*, 1995]. However, there is some debate on whether talc or a talc-like phase is stable to pressure >1.5 GPa [*Perrillat et al.*, 2005].

1.2 Olivine rheology

For temperatures greater than ~1100 °C, there is clear experimental (and geophysical) evidence that olivine deforms plastically via either dislocation creep or diffusion creep [e.g., *Hirth and Kohlstedt*, 2003]. The dominant mechanism is primarily dependent on grain size, water content, strain-rate, stress and the presence of melt [e.g.,

Hirth and Kohlstedt, 2003; Cooper and Kohlstedt, 1984]. Both mechanisms produce unique microstructural textures and crystallographic fabrics (or lack thereof) (e.g., Figure 2a & 2b) [*Jung and Karato, 2001; Katayama et al., 2004; Miyazaki et al., 2013*] and have distinct stress/strain-rate relationships: $\dot{\epsilon} \propto \sigma^{3.5}$ for dislocation creep and $\dot{\epsilon} \propto \sigma d^{-3}$ for diffusion creep, where $\dot{\epsilon}$ is strain-rate, σ is differential stress and d is grain size [*Hirth and Kohlstedt, 2003*]. At temperatures less than ~ 1100 °C there is more debate on how olivine deforms. In low confining pressure experiments (≤ 300 MPa), *Boettcher et al., 2007* observed semi-brittle deformation up to ~ 1000 °C with rate-weakening (frictional) sliding at up to 800 °C. Scaling of *Boettcher et al* results suggests rate-weakening is limited to temperatures $< \sim 600$ °C under natural strain rates. Conversely, olivine single crystal experiments show “Peierls creep” (dislocation glide) at these conditions [*Demouchy et al., 2013*]. Similarly, experiments on olivine aggregates conducted at much higher pressures (≥ 4 GPa), where cracking is suppressed, show Peierls creep deformation [e.g., *Mei et al., 2010; Raterron et al., 2004*].

2. Methods

2.1 Starting materials and sample preparation

Three starting materials were used in this study: an antigorite-rich serpentinite collected near Rochester, Vermont (serpentine #1) (same material used in Chapter 1 and Chapter 2), an antigorite-rich serpentinite collected near Nagasaki Japan (serpentine #2) (same material used in Chapter 3) and olivine collected near San Carlos, Arizona (SC olivine). These materials were crushed into a powder, sieved and stored at room temperature and humidity. Serpentine #1 and serpentinite #2 have a grain-size between 37 and 53 μm and contain ~ 90 and 99 vol % antigorite, respectively (see Chapter 2 and 3).

The SC olivine powder has a grain-size $< 25 \mu\text{m}$ with most grains $< 10 \mu\text{m}$ (Figure 3), and contains $> 99 \text{ vol\%}$ olivine with a forsterite content of $\sim\text{Fo}90$, as determined by electron microprobe analysis (Table 1). The powdered samples were cold-pressed at room-temperature into $\sim 1 \text{ mm}$ thick gouge layers between two alumina or yttria-stabilized zirconia (zirconia) pistons cut at 45 degrees, then hot-pressed at run conditions for at least 12 hours prior to deformation (experimental conditions are compiled in Table 2).

2.2 Deformation apparatus and sample assembly

General shear experiments were conducted in a solid confining medium Griggs-type triaxial press (same as used in Chapters 2 and 3). In all experiments, the samples were deformed in an undrained assembly (refer Fig. 1B in Chapter 2). For hydrostatic and ‘cook and crank’ experiments (discussed below) we used zirconia pistons inside silver jackets (identical to experiments in Chapter 2). In one experiment on dry SC olivine (run 2024), alumina pistons were used due to the anticipation of higher stresses in this experiment; the zirconia plastically deforms at high stress. In one experiment on wet SC olivine (run 2026), zirconia pistons and a double platinum-nickel jacket were used. The double jacket system has been shown to effectively seal water added to the sample prior to cold pressing [see *Getsinger and Hirth*, 2014 description of the double jacket]. Mechanical data were collected and reduced using methods described in Chapters 2 and 3.

2.3 Experimental procedures

In three runs (1935, 2023, 2025) ‘cook and crank’ style experiments were employed to induce reaction prior to deformation. In these experiments, serpentine #2 was held at $\sim 1 \text{ GPa}$ confining pressure and 800°C for up to 83 hours prior to axial

loading (see Table 2 for run conditions). After the cook period, the temperature was reduced to 700 °C and the loading piston advanced. In all experiments, general shear was imposed with a constant axial shortening rate of 18 nm/s, which corresponds to shear displacement rate (V) of ~ 25.5 nm/s or a shear strain-rate of $\sim 2.3 \times 10^{-5}/s$ for strain distributed across the entire thickness of the sample. In runs 1775, 2023 and 2025, the axial shortening rate was stepped by one or two orders of magnitude. To explore the extent of reaction progress during the cook period three hydrostatic experiments were conducted at ~1 GPa for 15 and 44 hours at 700 °C (runs 1737 and 1929) and 83 hours at 800 °C (run 1932).

Two experiments were conducted on pure SC olivine at ~1 GPa, 700 °C and an axial shortening-rate of 18 nm/s (Run 2024 and 2026) to investigate how talc affects the rheology of dehydrated serpentinite. There was no cook period in these experiments; the loading piston was advanced directly after the sample was brought to run conditions. In run 2026, ~4.4 μ l of deionized water (~4.0 wt %) was added to the SC olivine powder before the sample was cold pressed. In this run, the loading path (and quench) was set such that the density of water was maintained at ~1.0 g/ml (Figure A1); at peak P and T, water has a density of ~0.93 g/ml.

All samples were quenched under load and pressure to 300 °C in ~5 minutes then returned to ambient conditions over 1-2 hours. After quenching the jacketed samples were covered with epoxy, cut and polished for microstructural analysis. Micro-imaging and micro-chemical analyses were conducted with an Olympus SZX16 optical microscope fit with a digital camera, a LEO 1500VP field-emission scanning electron microscope and a Cameca SX100 electron microprobe. Electron backscatter diffraction

(EBSD) was conducted at the University of Otago, New Zealand on a Zeiss SIGMA FE-SEM equipped with Oxford Instruments NordlysF EBSD detector. In two runs a portion of the sample was removed for analysis using powder x-ray diffraction (XRD). XRD was conducted at Brown University using a Bruker D2 Phaser equipped with a Cu x-ray source and a LYNXEY detector. We used a 0.6 mm divergence slit, a 1 mm anti-scatter shield, a 2.5 degree soller slit, and a Ni filter. The analyses were run from 5 to 50 degrees 2theta with a 0.004 degree step size and 0.3 sec/step. Diffrac.EVA V3.1 was used for qualitative search and peak matching.

3. Results

3.1 Mechanical Data

Shear stress (τ) versus shear strain (γ) curves are shown in Figure 4. Cook and crank experiments on serpentine #1 (orange trace) and serpentine #2 (green trace) had strengths < 150 MPa for all shear velocities (strain-rates) tested. These runs generally had lower loading slopes, with the exception of run 1749, which had a shorter cook period at 700 °C (Table 2). In runs with longer cook periods at 800 °C, the samples exhibit relatively small stress drops (on the order of 10 MPa) followed by gradual reloading back up to nominal steady state flow values (e.g., Figure 4b and 4c). All small stress drops occurred over a period of a few minutes.

Two cook and crank experiments reported in *Proctor and Hirth, 2015* are included in Figure 2a (runs 1703 and 1775). The strength of the undrained sample (run 1703) is consistent with the results from this study while the strength of the drained sample (run 1775) is ~150 MPa higher.

In four runs the slip velocity was stepped by either 1 or two orders of magnitude to investigate the velocity and (strain-rate) dependence of flow stress (see Table 2). During the velocity steps the sample exhibit modest increases in stress with increasing strain-rate (e.g., Figure 4c). The log-stress versus log-strain-rate relationships are shown in Figure 5. In this plot, the shear stress and shear strain-rate values were converted to equivalent stress (σ_{eq}) and equivalent strain-rate ($\dot{\epsilon}_{eq}$) (for comparison with axial compression experiments, e.g., *Paterson and Olgaard*, 2000), stress values were also corrected for the rate-dependent friction of the loading piston (following methods discussed in Chapter 3) and strain-rate values are reported as an ‘applied strain-rate’ (i.e., we assume strain was fully distributed). All samples had slope of ~ 4 in log-log space (Figure 5).

The strengths of dry and wet SC olivine deformed at 700 °C and ~ 1 GPa confining pressure are shown in Figure 4a (black traces). Wet SC olivine (run 2026; 4 wt % water added), has a higher loading slope and peak strength than observed in the cook and crank runs. This sample exhibited a small stress drop at the peak stress that occurred over a period of ~ 6 minutes. The stress drop was followed by strain strengthening and a second, more gradual stress drop (Figure 4a).

Dry SC olivine (run 2024; no water added) was much stronger than all other samples tested. In this experiment, a large stress drop occurred after the sample achieved a shear stress of ~ 1100 MPa, which was followed by two cycles of restrengthening and smaller stress drops. Details of the stress drops are shown in Figure 6. During the first drop, ~ 5.5 mm of slip occurred in less than 1 second (Figure 6a and 6b). The apparent unloading slope (axial force/external axial displacement) during the first and third drops were ~ 218 kN/mm and ~ 199 kN/mm respectively, which are larger than the rig stiffness

(~120 kN/mm) (Figure 6c). The second drop had an unloading slope of ~93 kN/mm. The higher unloading slopes are consistent with values observed in blueschist and antigorite during large stress drops in a Griggs rig at similar conditions [Okazaki and Hirth, submitted]. The coefficient of friction (μ) for quasi-steady state sliding after the third stress drop was ~0.32 (assuming no cohesion) (see Chapter 2 for μ calculation). Boettcher *et al.*, 2007 observed a friction value of ~0.5 at similar temperatures and shear velocities, but at lower confining pressures (300 MPa).

3.2 Microstructures

3.2.1 Hydrostatic experiments

Microstructures from three hydrostatic experiments are shown in Figure 7. After ~15 hours at 700 °C serpentine #1 shows 2-3 vol % reaction products (run 1737, Figure 7a), which were estimated from the relative proportion of regions inferred to be products in BSE images. The reaction products have a grain size < 1 μ m and occur mostly along grain-boundaries, with thicker bands rimming magnesite grains (darker gray grains in Figure 7a). The product phases are likely olivine and talc based on XRD analyses from longer cook runs with coarser-grained products (see below), however XRD analysis was not conducted on this sample. Serpentine #2, reacted for ~44 hours at 700 °C shows ~1 vol % reaction products (run 1929, Figure 7b); the reaction products in this sample have a grain size < 1 μ m and occurred preferentially along grain boundaries. After 83 hours of reaction time at 800 °C, serpentine #2 had nearly complete metamorphism (run 1932, Figure 7c). This sample contained subhedral olivine grains, 1-10 μ m in size that comprise ~60 vol % of the sample (Figure 7d). The olivine was surrounded by randomly oriented, euhedral talc grains making up 20-30 vol % of the sample. The mineralogy was

determined with EDX, XRD and EBSD analysis. The XRD spectrum from run 1932 is shown in Figure 7e. This analysis shows strong olivine and talc peaks and weak clinocllore (chlorite group; $\text{Mg}_5\text{Al}_2\text{Si}_3\text{O}_{10}(\text{OH})_8$) peaks. Because the antigorite in serpentine #2 contains ~1.7 wt% aluminum [Sawai *et al.*, 2013] a small amount of clinocllore is expected as a reaction product. There are no antigorite peaks in Figure 7e, however <1 vol % antigorite could remain within the sample (XRD resolution is ~1 vol%). For comparison, an XRD analysis of un-dehydrated serpentine #2 is shown in Figure 7f; this material has no talc or olivine peaks, strong antigorite peaks and very weak clinocllore peaks, which might also account for the occurrence of clinocllore in the dehydrated sample.

We qualitatively estimate that the porosity in run 1932 is ~15 vol % based on the relative proportion of black area surrounding olivine and talc grains in BSE images. This estimate is notably less than predicted by the reaction (~30 vol %). However, the thickness of the sample reduced by ~16% during the cook period, indicating some compaction and fluid redistribution occurred.

3.2.2 Dry SC olivine

Deformation within the dry SC olivine was localized along one fracture located parallel to the piston/sample contact (Figure 8a and 8c). The majority of the sample underwent very little shear strain. Within the low strain region, larger olivine grains are angular (similar to the starting material), contain microcracks and, in places, appear brecciated. Grains with a high aspect ratio appear to be rotated, such that the long axis is perpendicular to the orientation of the maximum principal stress (Figure 8c); some grains were fractured to accommodate the rotation. The sample was not fully dense; dark

regions within BSE images appear to be voids (porosity) that compose 2-4 vol % of the sample (e.g., Figure 8b). Conversely, the olivine grains adjacent to the shear fracture contain more microcracks (than away from the fracture) and, apparently, have less void space around grains (Figure 8d). These microstructures resemble highly damaged rocks commonly observed near active fault zones [e.g., *Mitchell et al.*, 2011]. No ware material was recovered along the slip surface; if this material existed it may have been washed out during the sample preparation process.

3.2.3 Wet SC olivine

Strain was also highly localized within wet SC olivine along one shear fracture at the piston/sample contact (Figure 9a). The fracture is $\sim 2\ \mu\text{m}$ wide and contains finer-grained ($< 1\ \mu\text{m}$) material (Figure 9b). The olivine grains that were truncated by the fracture contain very little evidence of deformation aside from the truncation. Very few microcracks or other textural evidence of deformation are observed throughout the sample. The sample had $\sim 5\%$ porosity (estimated using method described above).

3.2.4 Cook and crank

In cook and crank experiments, the distribution of strain at the sample-scale and grain-scale vary between samples. Run 1749 (this study) and runs 1703 and 1775 [Proctor and Hirth, 2015] show nominally distributed strain, whereas runs 1935, 2023 and 2025 show either fully localized strain or a combination of localized and distributed deformation throughout the samples (Table 2). In run 2025, talc defines a S-C' type shear fabric (Figure 10a). The olivine grains between the talc grains are elongate and apparently rotated into the S and C' fabrics. Nearly all olivine grains show highly attenuated or 'necked' grain-boundaries in the orientation of maximum elongation

(Figure 10a and 10b). The necked grain boundaries are often curved into C' shear bands forming sigmoidal shapes and, in some cases, become highly elongated into ribbon-like textures within the shear bands themselves (Figure 10b). In nearly all cases, the deformed olivine grains are contacting talc basal cleavage. Conversely, olivine grain-boundaries that abut talc edges, perpendicular to the basal cleavage, are typically pinned and appear relatively undeformed (Figure 10b). There is no evidence of dynamic recrystallization or grain-size reduction of the olivine (as observed from BSE images) and very few microcracks are observed throughout the sample. Thick talc bands were also observed filling fractures and vein-like structures that transected the sample (Figure 10c). The talc bands are up to 20 μm thick and contain 1 to 3 μm long grains; the long axes of the talc grains are typically oriented parallel to the shearing direction (Figure 10c). These microstructures are consistent with those previously described in *Proctor and Hirth, 2015* at similar conditions.

In one cook and crank experiment (run 2025), strain was completely localized along the piston/sample contact; little to no strain occurred within the sample (Figure 10d). XRD analysis of this sample shows strong olivine and talc peaks and weak chlorophyll peaks (Figure 10e). The olivine grains are subhedral, with a grain-size from 1-10 μm and talc grains are euhedral, occurring in randomly oriented patches, and have a grain size from 3 to 30 μm (Figure 10d). These grains are slightly larger than observed in the hydrostatic sample (run 1932, Figure 7d), indicating some grain growth occurred during the ~ 29 additional hours this sample was held at dehydration conditions compared with run 1932. This sample also appears to have less porosity than in run 1932.

In runs that were deformed to high strain, the shearing pistons ruptured through the confining jacket into the salt medium (see Table 2). In these experiments, there are no apparent changes in the mechanical data to indicate the onset of the rupture and no textural differences are observed in comparison with runs without jacket ruptures.

3.3 Olivine fabric analysis

Olivine fabrics were analyzed using EBSD on two experimental samples and one natural deformed sample. In Figure 11 the olivine fabric is shown from a cook and crank experiment on serpentine #1 from *Proctor and Hirth, 2015* (run 1703, Table 2). An analysis conducted over a larger region of the sample shows a weak olivine LPO, in which the c-axes align sub-parallel to the pole of the shear plane and the b-axes align sub-parallel to the shearing direction (Figure 11c). A second analysis, on a smaller region of the sample (Figures 11e and 11d) that contained a C' shear band (refer to Chapter 2 for fabric description), also shows a weak olivine LPO (Figure 11f), consistent with the broader analysis (Figure 11c).

In Figure 12, the olivine fabric is shown for an axial compression experiment on serpentine #1 from *Chernak and Hirth, 2010* (Run W1464). This sample underwent dehydration during deformation at 700 °C and ~1.5 GPa confining pressure to an axial strain of ~ 37%. In this sample, the olivine a and c-axes are oriented perpendicular to the axial loading direction and b-axes are oriented sub-parallel to the loading direction.

The olivine fabric for a natural sample from the Erro-Tobbio ultramafic terrane (NW Italy) is shown in Figure 13. The olivine from this sample occurred within a fracture that truncated antigorite-rich serpentinite. The olivine is interpreted to have formed during slip along the fracture, in which high temperatures and subsequent

dehydration of the serpentine occurred [unpublished, Luc Mel and G. Hirth]. The sample, shown in Figure 13a, is oriented such that the apparent shear direction is parallel to the long axis of the image (X0). Here olivine b-axes tend to be oriented perpendicular to the shearing direction and c-axes appear to be rotating into the shearing plane.

4. Discussion

4.1 Talc-induced pressure-solution creep in olivine

Similar to *Proctor and Hirth, 2015* we propose that the presence of talc promotes pressure-solution (P-S) creep of olivine. Our results demonstrate that adding water as a pore fluid to olivine gouge reduces the strength of the sample, but does not induce grain-scale ductility. In both dry and wet experiments on SC olivine, the deformed grains closely resemble the starting material and, in both cases, exhibit only brittle microstructures (cracks and fractures). In contrast, in the presence of the talc that formed during dehydration, the olivine became highly deformed (in high strain regions of the samples) and the morphology of the olivine was indicative of ductile (or plastic) deformation. Because the experiments without talc were deformed at conditions nearly identical to those with talc we rule out the possibility that dislocation creep or glide was the dominant deformation mechanism in olivine. Other factors that are inconsistent with dislocation creep or glide include: (1) the strengths of the olivine + talc samples were one to two orders of magnitude lower than predicted by the respective flow laws [e.g., *Hirth and Kohlstedt, 2013; Demouchy et al., 2013*], (2) grain boundary migration or dynamic recrystallization textures were not observed, and (3) the weak olivine LPO's observed in these samples are inconsistent with established dislocation creep fabrics [e.g., *Jung and Karato, 2001*]. Furthermore, the deformed olivine samples exhibit many structures

consistent with fluid-accommodated dissolution and precipitation (i.e., P-S) including: S-C-C' foliations [e.g., *Bell and Cuff*, 1989; *Wintsch and Yi*, 2002], strain-shadows (reported in *Proctor and Hirth*, 2015) [e.g., *Passchier and Trouw*, 2005], monomineralic veins [e.g., *Passchier and Trouw*, 2005], and enhanced grain-growth [see *Proctor and Hirth*, 2015 for discussion].

Talc cleavage surfaces appear to be key for promoting P-S creep in olivine. Our microstructural analysis revealed that deformed olivine grains were almost always contacting talc basal cleavages. Conversely, olivine grain-boundaries that abutted talc edges, perpendicular to the basal cleavage, were typically pinned and appeared relatively undeformed. In general, the olivine grain morphology appears to be controlled by the development of fabrics in talc, i.e. olivine was passively responding to talc reorienting during deformation.

It is well known that quartz or carbonate grains in contact with mica grains dissolve faster than without mica present [e.g., *Heald*, 1955; *Weyl*, 1959; *Engelder and Marshak*, 1985; *Tada and Seiver*, 1989; *Bjorkum*, 1996; *Renard et al.*, 1997]. Mica is commonly observed at dissolution surfaces in stylolites within limestone and quartz-rich sandstone [e.g., *Tada and Seiver*, 1989] and in tectonic cleavage formations [e.g., *Wright and Henderson*, 1992, *Engelder and Marshak*, 1885]. These geologic observations are consistent with experiments by *Hickman and Evans*, 1995 that demonstrate a five-times greater convergence rate between halite and silica glass when thin-film of montmorillonite is added between the phases. More recent work by *Kristiansen et al.*, 2011 showed that increased dissolution rates along clay-quartz contacts are linked to differences in electrochemical surface potentials.

4.2 Rheology of dehydrated serpentinite

Despite the overwhelming textural evidence for P-S, the overall rheology of the dehydrated serpentinite was not limited by this deformation mechanism. For P-S limited deformation strain-rate is linearly proportional to stress, i.e., $\dot{\epsilon} \propto \sigma$ [e.g., *Rutter*, 1983]. Recall that in our experiments strain-rate was proportional to stress raised to the power of ~ 4 [Figure 5]. However, the inconsistency in rate-dependence is not surprising considering the semi-brittle nature of the deformation in these samples. Nearly all samples showed highly localized structures. Only one experiment deformed in this study and one from *Proctor and Hirth*, 2015 exhibited nominally ductile deformation, but even within these samples strain was partitioned into a network of shear fractures. Interestingly, run 1749 (this study) had a slightly higher strain-rate dependence (i.e., lower stress exponent) than the other samples with highly localized fractures, suggesting at lower strain rates the strength may approach a linear stress-strain rate dependence, consistent with P-S creep.

The development of brittle shear zones in cook and crank samples might be related to migration of pore fluid and porosity during deformation. Many brittle shear zones that occur within these samples are oriented 15-30° antithetic to the shearing direction, resembling R-type Reidel shear [e.g., Figure 10a, *Proctor and Hirth*, 2015]. These shear zones appear to have higher porosity and, in some cases, brecciated material [e.g., *Proctor and Hirth*, 2015]. Previous work, on partially molten samples, has demonstrated that melt tends to migrate into similarly oriented bands antithetic to the shearing direction [e.g., *Holtzman et al.*, 2003; *Rybacki et al.*, 2008]. *Rybacki et al.*, 2008 showed that strain localization along melt bands can lead to brittle failure and stress

drops. We hypothesize that a similar mechanism is occurring in cook and crank experiments as water and porosity reorganizes during deformation. The occurrence of small stress drops at the beginning of our experiments is consistent with this hypothesis.

The strength of dehydrated serpentine is apparently limited by a combination of frictional sliding of talc and grain-scale ductile flow of olivine by P-S creep. Recall that our microstructural analysis showed talc occurred within fractures and shear zones, as well as deformed olivine. *Proctor and Hirth, 2015* demonstrated that the strength is also sensitive to pore fluid pressure. One possibility is that the strength is simply limited by the frictional strength of talc, following an effective stress relationship. However, at the conditions of our experiments, talc has a lower strength than observed in this study (~50 MPa differential stress with no pore fluid pressure) and almost no rate dependence [*Escartin et al., 2008*]. We therefore suggest the semi-brittle flow law proposed by *Proctor and Hirth, 2015*:

$$\tau = \Delta + \mu(\sigma_n - \alpha P_f) \quad , \quad (3)$$

where τ is shear stress, σ_n is normal stress, P_f is pore fluid pressure, α is the effective stress coefficient [e.g., *Scholz, 2002*], Δ represents P-S creep and μ represents the friction coefficient of talc. Δ may be fit with a P-S creep flow law [e.g., *Rutter, 1983*], however quantifying the parameters associated with this flow law are outside the scope of this study. To a first order, the ductile and brittle components of equation 3 are dependent on one another, for example, if $P_f = 0$ then $\Delta = 0$ (i.e., P-S requires water). There are also second order feedbacks between the pressure of the water and rates of dissolution and precipitation. For example, *Newton and Manning, 2002* demonstrated that the solubility of olivine in water increases by a factor of ~6 for an increase in water pressure from 100

MPa to 1000 MPa at 700 °C. The stability of talc also depends on the pore fluid pressure (see below), which adds further complication.

Previous experiments conducted on partially dehydrated and fully dehydrated serpentinite at lower confining pressures (≤ 300 MPa) and temperatures concluded that the strength of rocks composed of olivine + talc is limited by diffusion creep of olivine [Rutter and Brodie, 1988; Arkwright *et al.*, 2009]. These studies did not observe textural evidence for P-S creep. However, the lower pressures and temperatures used in these studies may have sufficiently reduced dissolution/precipitation rates, such that P-S did not cause observable deformation.

4.3 Ultra-high temperature talc?

Our experiments suggest that the assemblage of olivine and talc may be stable at higher temperatures than previously thought. Recall that our experiments on dehydrated antigorite at temperatures up to 800 °C at 1 GPa contained only olivine, talc and a small amount of chlorite (Figure 7e). These results conflict with theoretical calculations for the phase boundary between forsterite + talc and orthopyroxene (~ 675 °C, Figure 1) [e.g., Spear, 1995], as well as experimental observations [Ulmer and Tromsdorff, 1995; Perrillat *et al.*, 2005]. One possible explanation for the occurrence of talc at such high temperatures is that high water pressures are stabilizing talc via a Le Chatelier-type reaction effect, i.e., if the product concentration is sufficiently high then higher temperatures are required to drive the reaction. We know that high fluid pressures (at least as high as the confining pressure) are generated within our undrained assemblies based on the results from Chapter 2.

Without a more rigorous thermodynamic model (and more data) it remains unclear if the phase boundary is actually shifted and if high fluid pressures are causing the shift. Nonetheless, some aspects of the previous experimental studies add further legitimacy to our hypothesis. The experiments by *Ulmer and Trommsdorff, 1995* are complicated by the fact that their starting material contained ~ 10 wt % enstatite and olivine and only a small amount of reaction products were apparently generated during their tests. To infer reaction occurrence, they measured the relative change in XRD peak intensity for a given phase. For example, if enstatite peaks increased then they assumed that it was stable and increased in volume during the reaction. The problem with their results is that, in most cases, they could not resolve the relative decrease in antigorite peak intensity, which should correspond with the increase in product phase. This calls into question their ability to resolve (or infer) small increases in enstatite and forsterite. In the *Perrillat et al., 2005* study, enstatite was observed in only two jacketed experiments conducted at ~700 °C and $P > 3.5$ GPa. For these tests it is possible that talc is unstable regardless of the water pressure.

4.4 Geophysical implications

P-S primarily occurs within the upper to middle crust [e.g. *Rutter, 1983*]. With increasing depth and temperature, free water within grain boundaries becomes less abundant and dislocation creep becomes increasingly dominant [e.g., *Kohlstedt et al., 1995*]. Dehydration reactions in subduction zones may produce transient environments conducive for P-S creep. Previous studies have suggested that P-S may play an important role in the rheology along the slab wedge interface based on geologic observations from exhumed high-pressure terranes [e.g., *Fagereng, 2011*; *Wassman and Stockhert, 2013*,

and references therein]. Our work confirms these observations and further suggests P-S extends to the limit of talc stability - deeper and hotter than previously thought possible.

5. Conclusions

In this study we demonstrate that talc promotes flow of olivine at 700 °C and 1 GPa at hydrothermal conditions. Based on our microstructural observations, we conclude that pressure-solution along olivine-talc surfaces is causing olivine deformation at differential stresses significantly lower than predicted by existing flow laws. Despite the overwhelming textural evidence for pressure-solution, we show that the strength of olivine + talc is not limited by this mechanism in our experiments. We speculate that the strength is limited by a combination of frictional flow in talc and pressure-solution creep, both of which are dependent on pore fluid pressure. However, it remains unclear if the semi-brittle nature of these samples extrapolates to natural strain rates. The occurrence of pressure-solution in our experiments suggests this mechanism is occurring in dehydrating serpentine within the mantle wedge and possible within subducting lithospheric mantle.

Appendix A

In Figure A1 the density of water is shown for a range of P-T conditions used in this study. The loading and unloading path used for most experiments is shown (white trace). This figure shows that the density of water is ~1 g/ml for the duration of the loading and unloading cycle.

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Table 1. Results from electron microprobe analysis of SC olivine.

Table 2. Experimental conditions: * samples from *Proctor and Hirth*, 2015; # Amount of time after loading piston is advanced; NA – does not apply.

Figure 1. MgO-SiO₂-H₂O phase diagram after *Ulmer and Trommsdorff* (1995) showing stability zones for mineral phases: antigorite, forsterite (Fo), talc (Tlc) and enstatite (En). Dashed line indicates reaction boundary for low water activity [*Perrillat et al.*, 2005].

Figure 2. Dislocation creep fabrics in experimentally deformed olivine. (a) Pole figures of crystallographic axes after *Jung and Karato* (2001); warmer colors indicate higher density. (b) Fabric diagram as a function of stress and water content after *Katayama et*

al. (2004). The dominant slip direction and plane are indicated with brackets and parentheses respectively.

Figure 3. Electron backscatter image of uncompacted serpentine #2.

Figure 4. (a) Mechanical results (see text for description). Data shown in (b) and (c) are enlargements from figure a. Arrows indicate onset of a step in the imposed shear velocity (noted in black).

Figure 5. Results from strain-rate stepping experiments (see text for description). A slope of 4 in log-log space is noted in red.

Figure 6. Differential stress versus shear displacement (a) and time (b) from run 2024; dry SC olivine. (c) Axial force versus external axial displacement. Dashed lines highlight the unloading slope for each large stress drop; the magnitude of the unloading slope is noted in black. The rig stiffness is plotted in red.

Figure 7. Results from hydrostatic experiments. Electron backscatter images are shown from (a) run 1737, serpentine #1, 14.8 hours at 700 °C; (b) run 1929, serpentine #2, 43.5 hours at 700 °C; (c) run 1932; serpentine #2, 83 hours at 800 °C. (d) Enlarged region from run 1932. (e) XRD analysis from run 1932 (see text for description). Colored lines indicated the expected peak positions for the phases: forsterite (red), talc (blue) and

clinochlore (green). (f) XRD analysis from undehydrated serpentine #2. This analysis shows no peaks for talc or forsterite.

Figure 8. Microstructures from dry SC olivine. (a) Photomicrograph of deformed sample; sample is cut parallel to the shearing direction; large shear fracture is highlighted with red line. (b) Electron backscatter image of sample. Black arrows indicate sense of shear. Image shows abundant microcracks and slight preferred orientation of elongated grains. (c) Electron backscatter image of region adjacent to the shear fracture. This images shows highly fractured grains. The opposing sides of the slip surface were separated during unloading and sample preparation. The brighter regions, adjacent to the fracture surface, are an artifact of surface charging.

Figure 9. Microstructures from wet SC olivine. (a) Photomicrograph of deformed sample, cut parallel to the shearing direction; large shear fracture is highlighted with red line. (b) Electron backscatter image of sample showing fracture truncating undeformed olivine grains. Fracture trace is highlighted with red dashed line.

Figure 10. Microstructures from cook and crank experiments. Electron backscatter images from run 2023 are shown in (a), (b) and (c) and from run 2025 in (d). (a) Images shows lighter gray (olivine) and darker gray (talc). An S-C' fabric is developed (see text); red dashed lines note the trace of C' shear planes and solid red line notes the trace of S planes. Unloading cracks (black) preferentially localize along shear zones parallel to C'. (b) Enlarged image from run 2023. Images show highly deformed olivine grains that are

being dragged into two shear zones that transect the image (now partially dilated by an unloading crack, black). (c) Image shows fracture with slight shear offset filled with talc (darker gray). (d) Image shows relatively undeformed olivine and talc grains. (e) XRD analysis from run 2025; colored lines indicated the expected peak positions for the phases: forsterite (red), talc (blue) and clinocllore (green).

Figure 11. Olivine fabric analysis from cook and crank experiment, run 1703. (a) Electron backscatter image of sample; arrows note imposed sense of shear. (b) Enlargement of (a) showing indexed grains; olivine (red) and antigorite (blue). (c) Olivine pole figure projections with shearing direction oriented E-W. Note c-axis is oriented perpendicular to the shearing direction. (d) Enlargement of region surrounding a C' shear band; shear band is traced in red, and deformed antigorite grains with left-lateral sense of shear are traced in green. (e) EBSD analysis region (white box in 11d); colors indicate olivine orientation. (f) Olivine pole figure projections with shearing direction oriented E-W.

Figure 12. Olivine fabric analysis from an axial compression experiment that underwent syn-dehydration deformation, run 1464 (see text for details). (a) Electron backscatter image of sample. (b) Enlargement of (a) showing indexed grains; olivine (red), antigorite (blue) and magnetite (yellow). (c) Olivine pole figure projections with the compression direction oriented N-S.

Figure 13. Olivine fabric analysis from natural sample from Italy (see text for details); shearing direction is inferred to the E-W. (a) Image showing indexed grains; olivine (red), antigorite (blue), magnetite (yellow) and talc (pink). (b) Olivine pole figure projections with shearing direction oriented E-W.

Figure A1. Density of water as a function of P-T conditions. Data is from *Lemmon et al.*, 2011.

Table 1

Material	Na ₂ O	SiO ₂	MgO	Al ₂ O ₃	K ₂ O	CaO	TiO ₂	Cr ₂ O ₃	MnO	FeO	Total	Fo mol%
San Carlos Ol	0.01	40.94	48.86	0.01	0	0.07	0	0	0.14	10.47	100.51	91
San Carlos Ol	0.04	40.29	48.25	0	0.01	0.07	0	0.04	0.15	10.45	99.31	91
San Carlos Ol	0	40.8	48.82	0.01	0.01	0.06	0.01	0.03	0.14	10.33	100.21	92

Table 2

Experiment	Starting Material	Cook Temperature (°C)	Cook Time (hrs)	Cook Pressure (GPa)	Deformation Temperature (°C)	Deformation Pressure (GPa)	Deformation Time ^a (hrs)	Shear Velocity (nm/s)	Strain Distribution	Jacket Tare
1737	Serp. #1	700	14.8	1.1	NA	NA	NA	NA	NA	no
1929	Serp. #2	700	43.5	1.05	NA	NA	NA	NA	NA	no
1932	Serp. #2	800	83	1	NA	NA	NA	NA	NA	no
1749	Serp. #1	700	19	1	700	1.02	65	25.5, 2.55	Distributed	no
1935	Serp. #2	800	80.4	1.02	700	0.98	54.5	25.5, 25.5	Localized	yes
2023	Serp. #2	800	68.2	1.05	700	1.03	67	25.5, 2.55	Localized	yes
2024	SC Olivine	NA	NA	NA	700	0.89	37.6	25.5	Localized	no
2025	Serp. #2	800	68.5	1.1	700	1.03	41.6	25.5, 25.5, 2.55	Localized	yes
2026	SC Olivine	NA	NA	NA	700	0.94	37	25.5	Localized	no
1703*	Serp. #1	700	14.5	1.02	700	1.01	23.2	25.5	Distributed	no
1775*	Serp. #1	700	15.7	1.1	700	1.12	23.8	25.5	Distributed	no

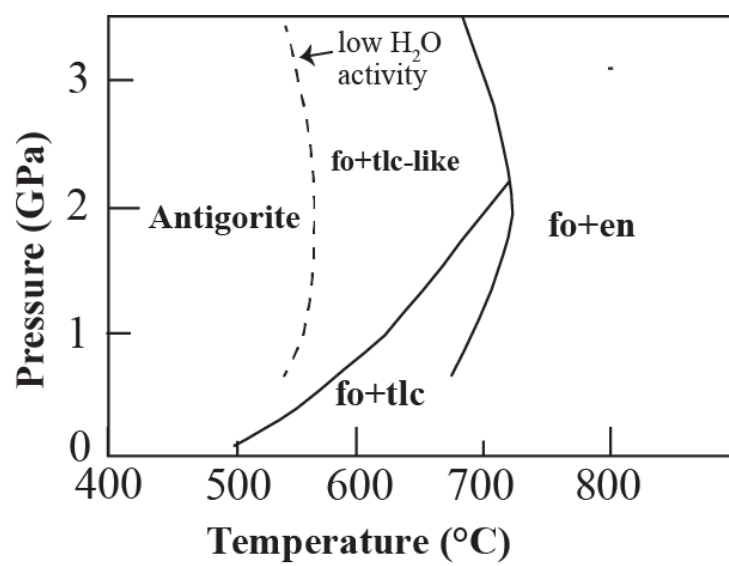


Figure 1

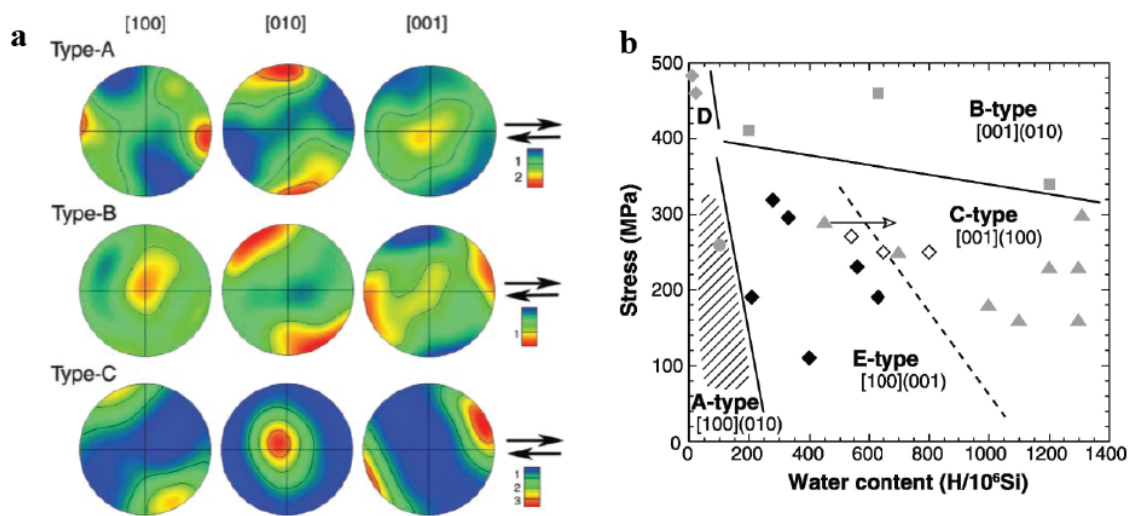


Figure 2

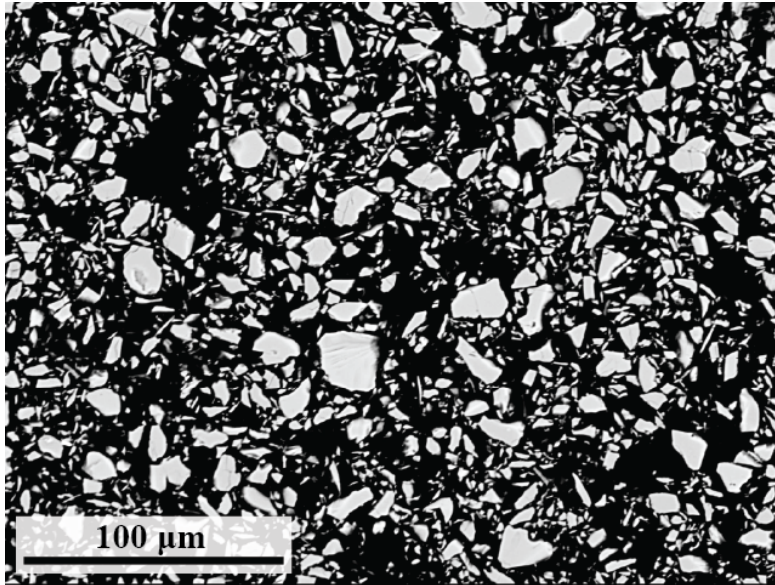


Figure 3

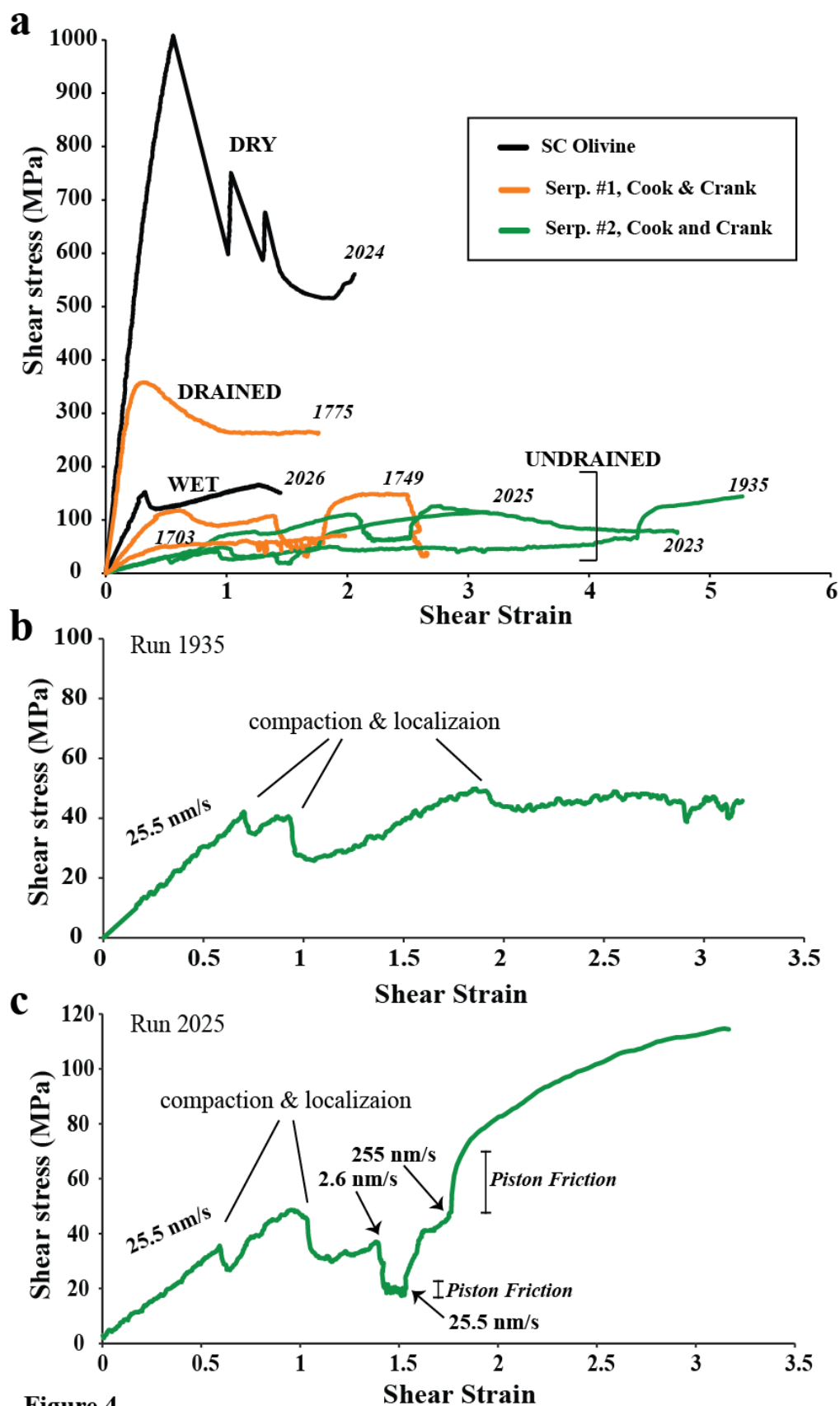


Figure 4

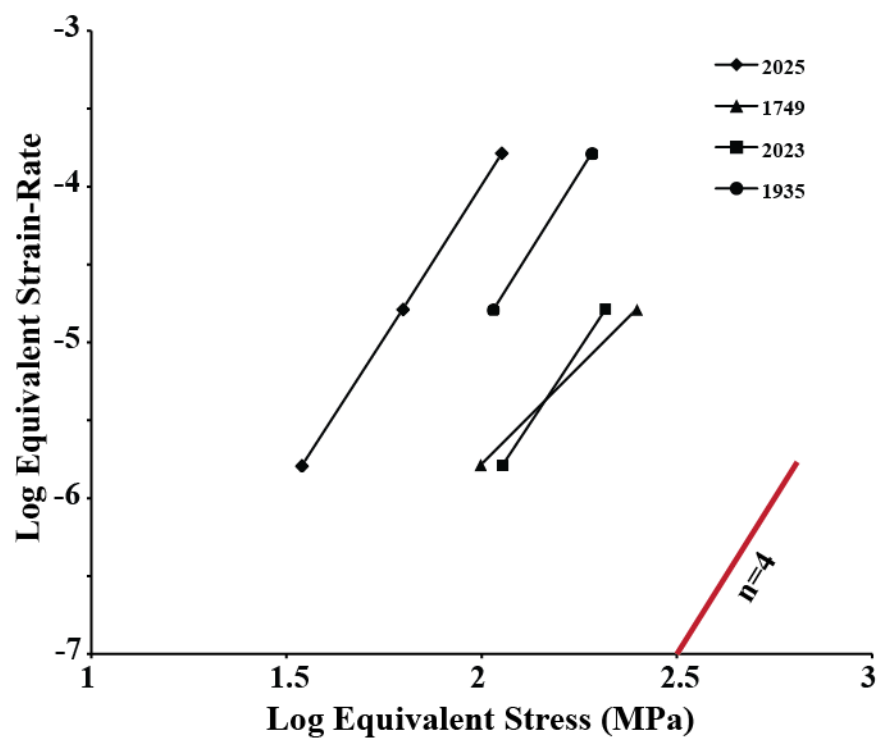


Figure 5

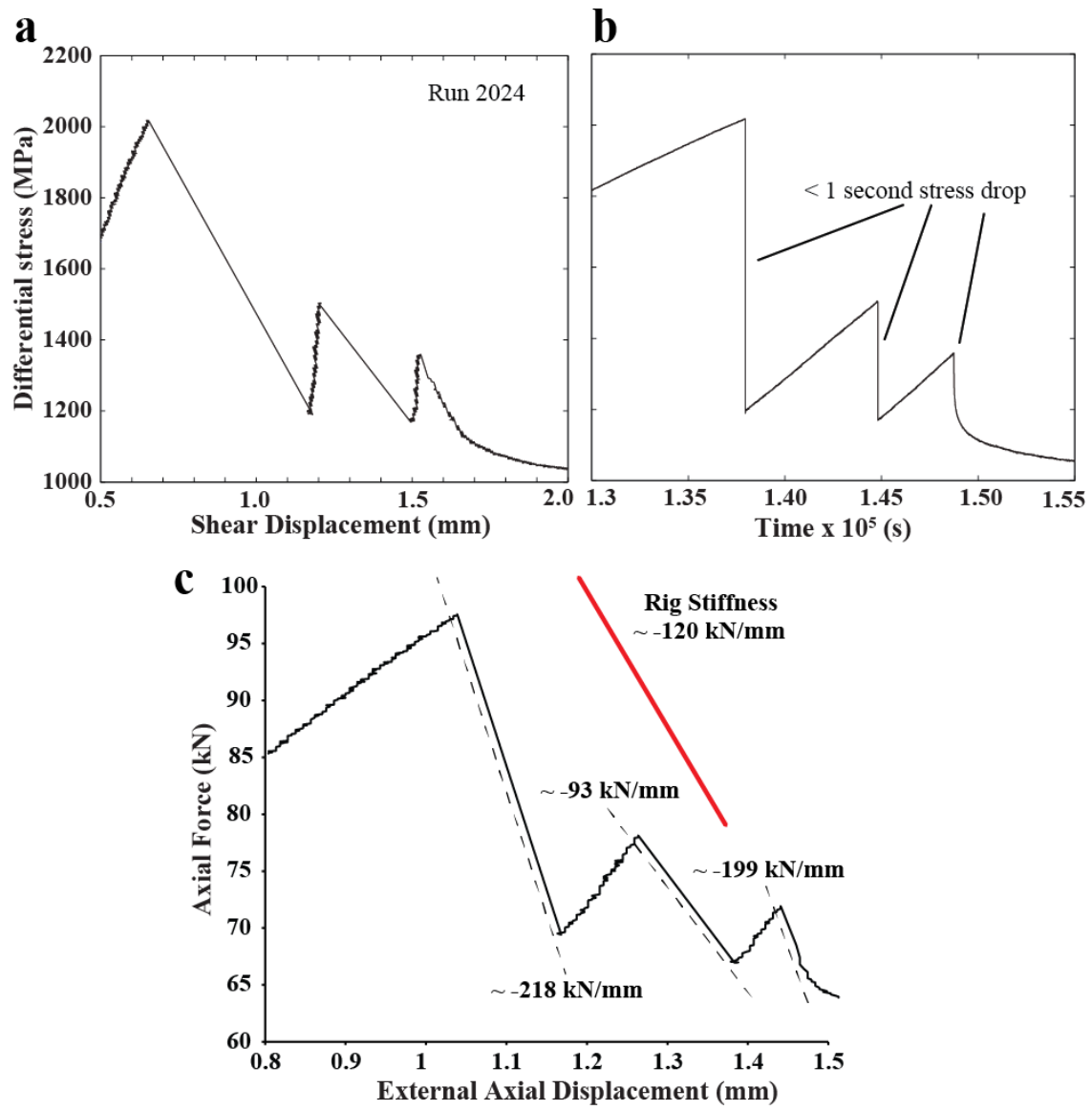


Figure 6

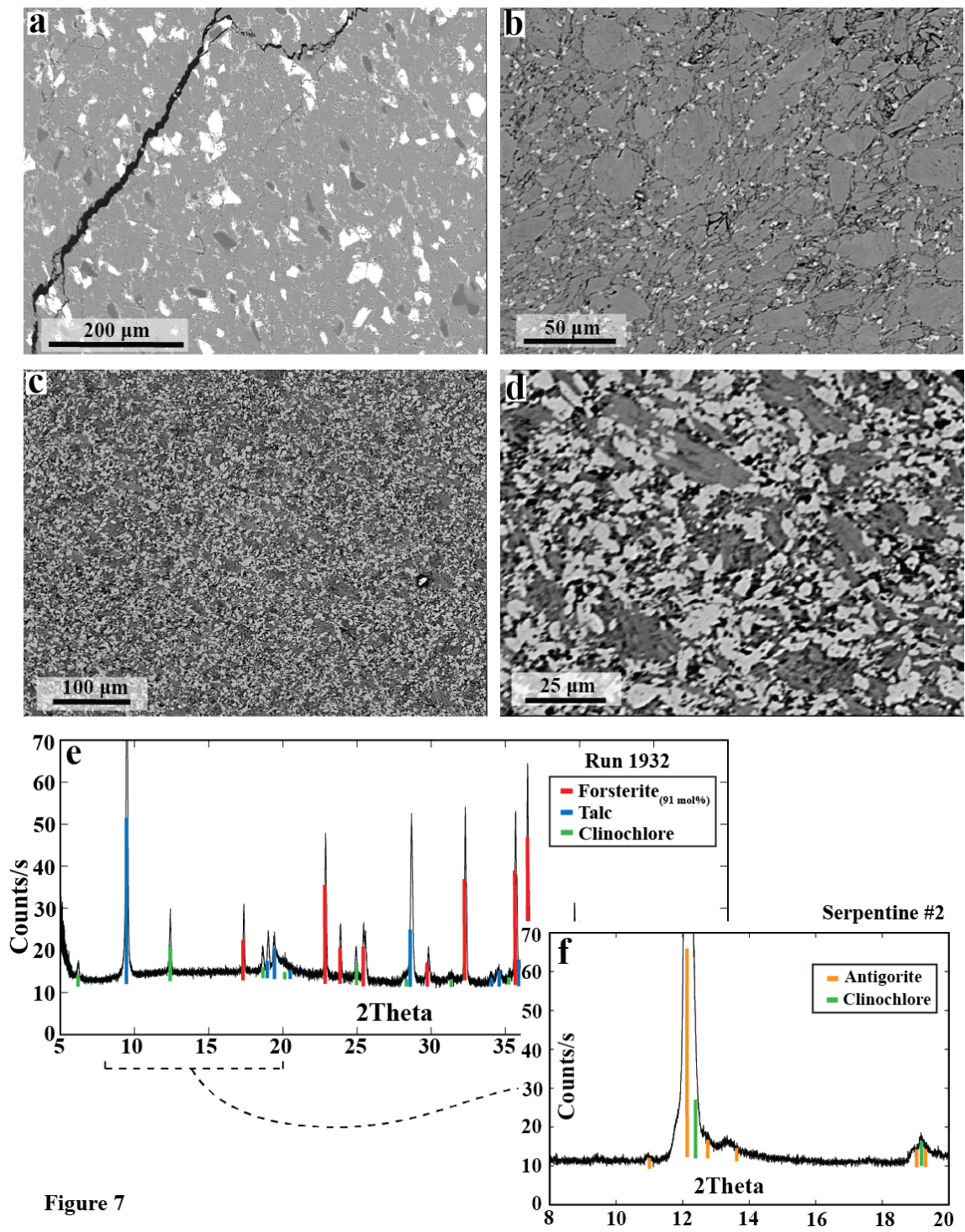


Figure 7

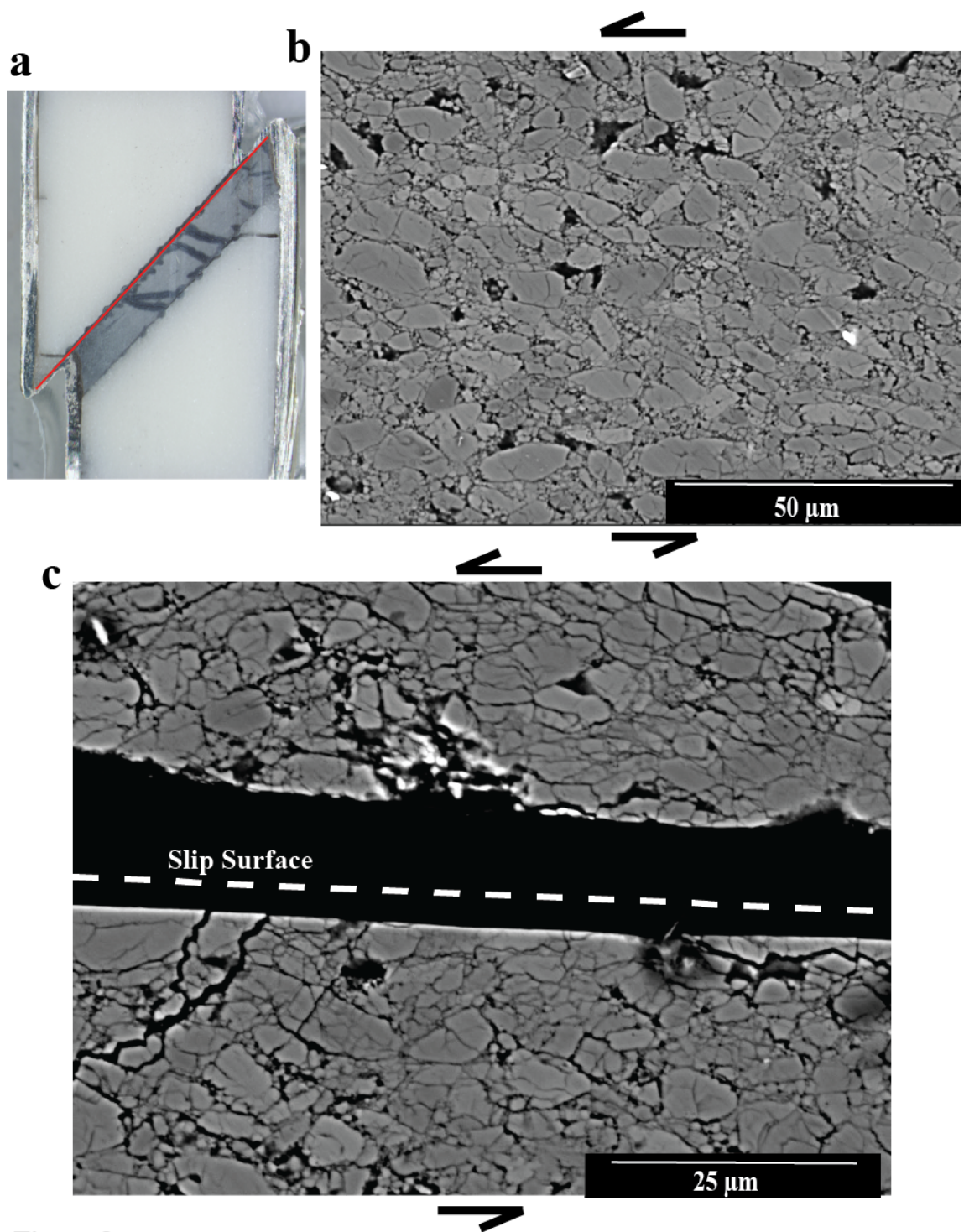


Figure 8

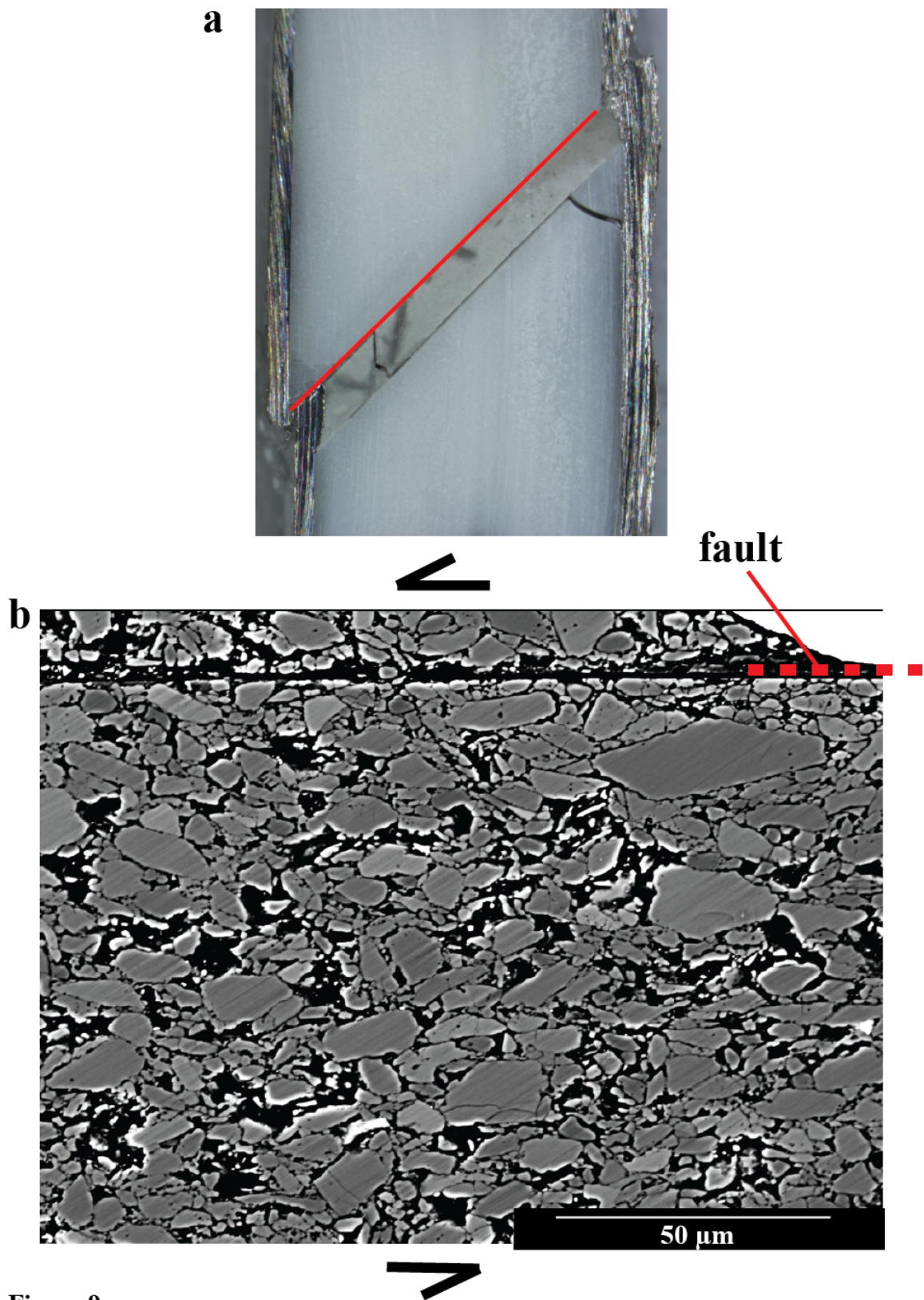


Figure 9

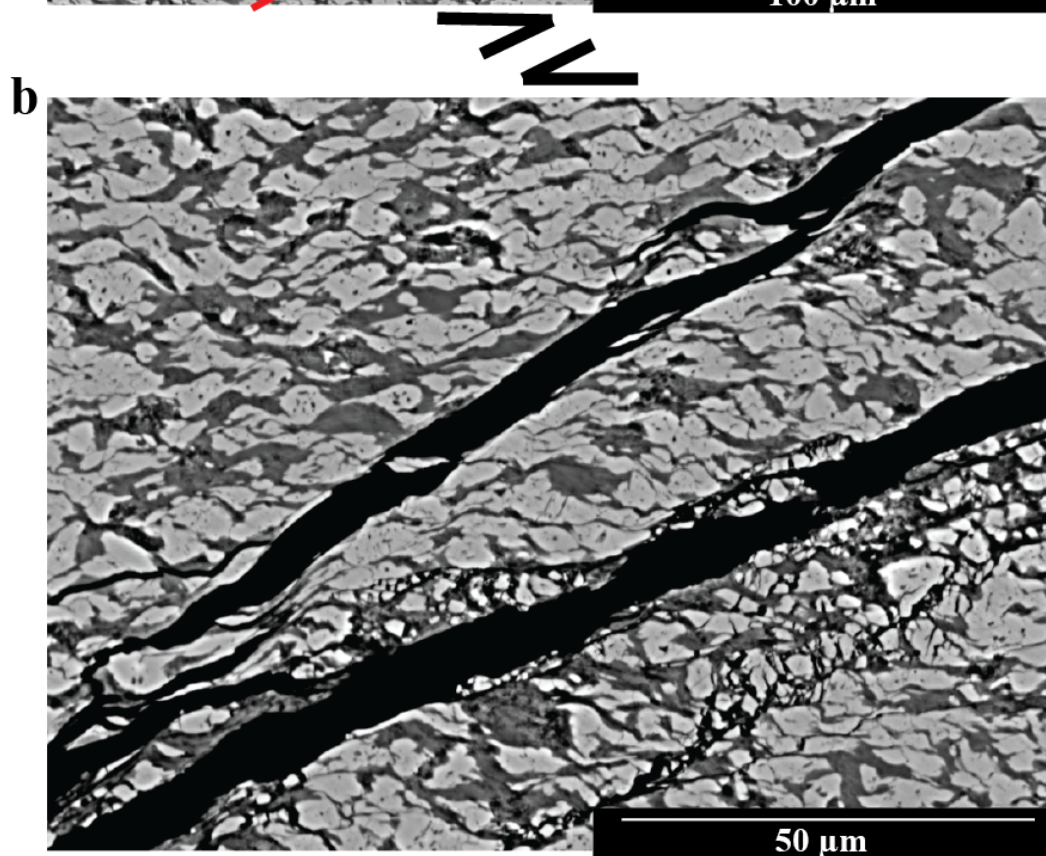
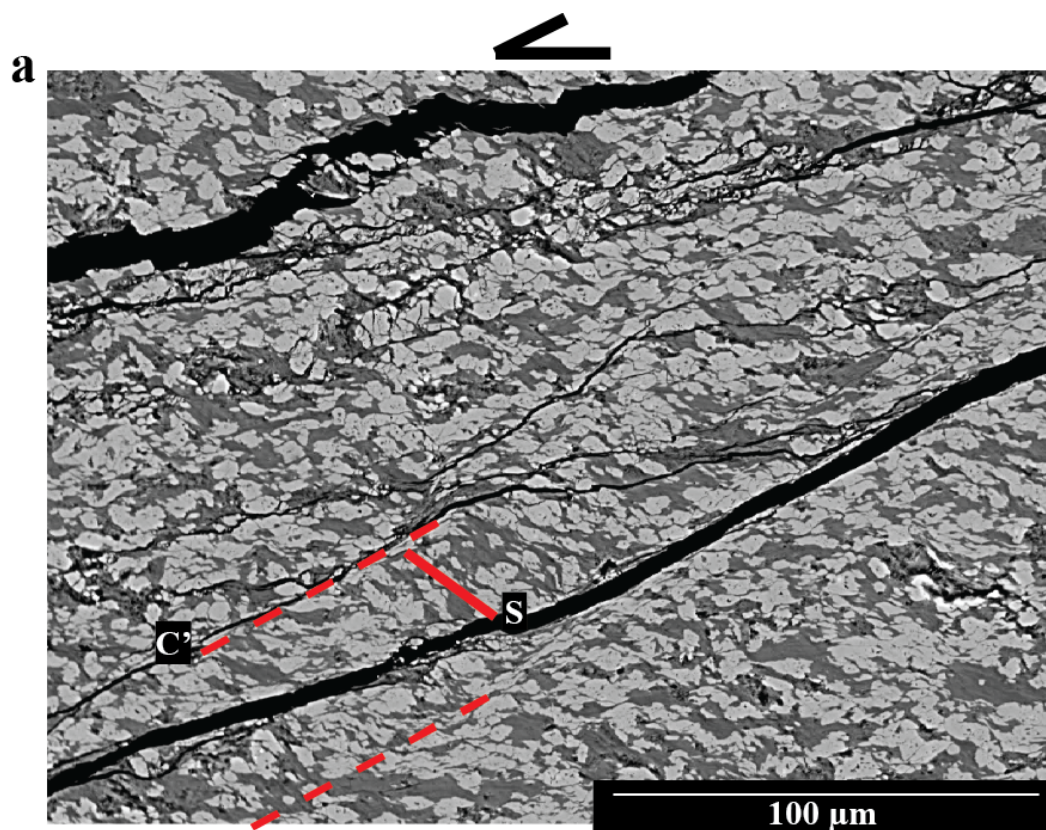


Figure 10

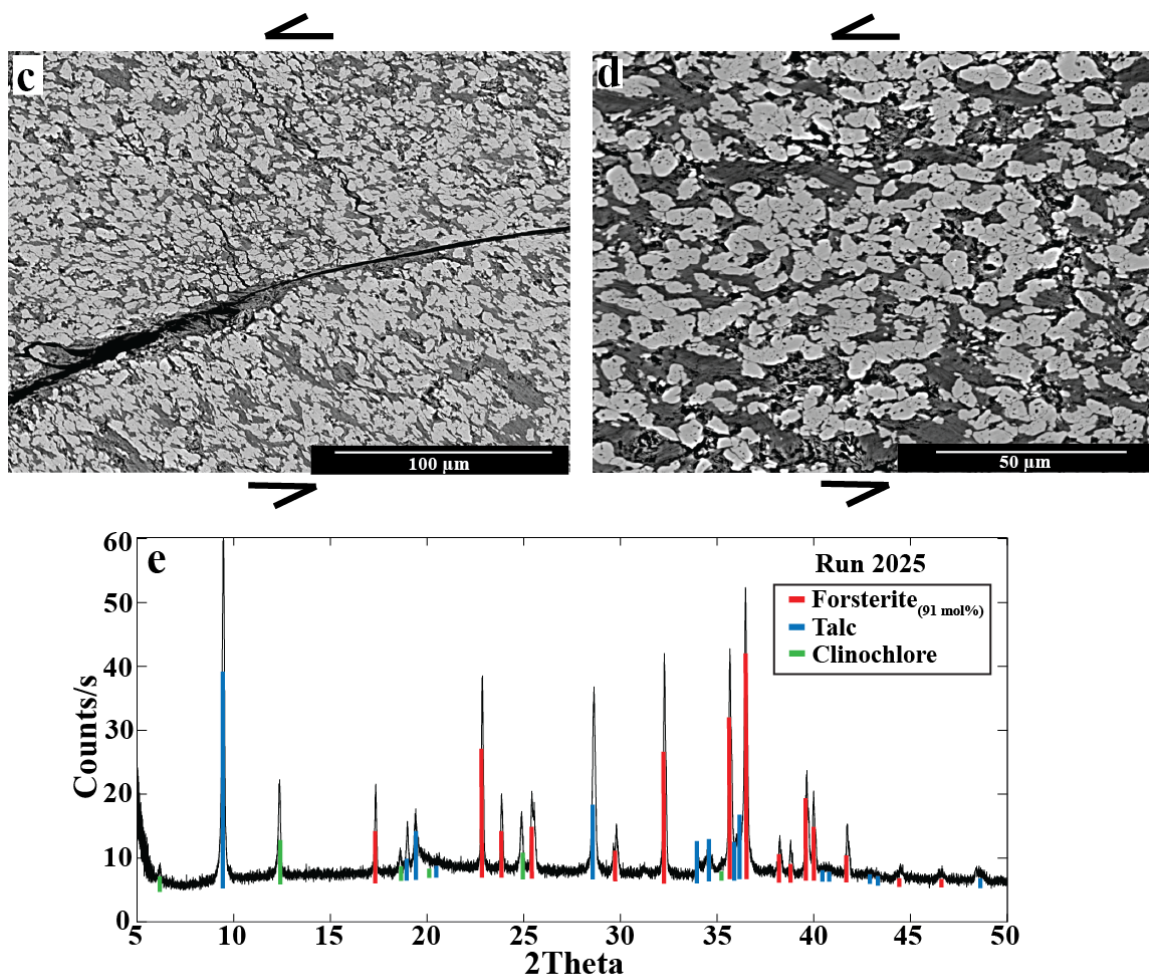


Figure 10 (continued)

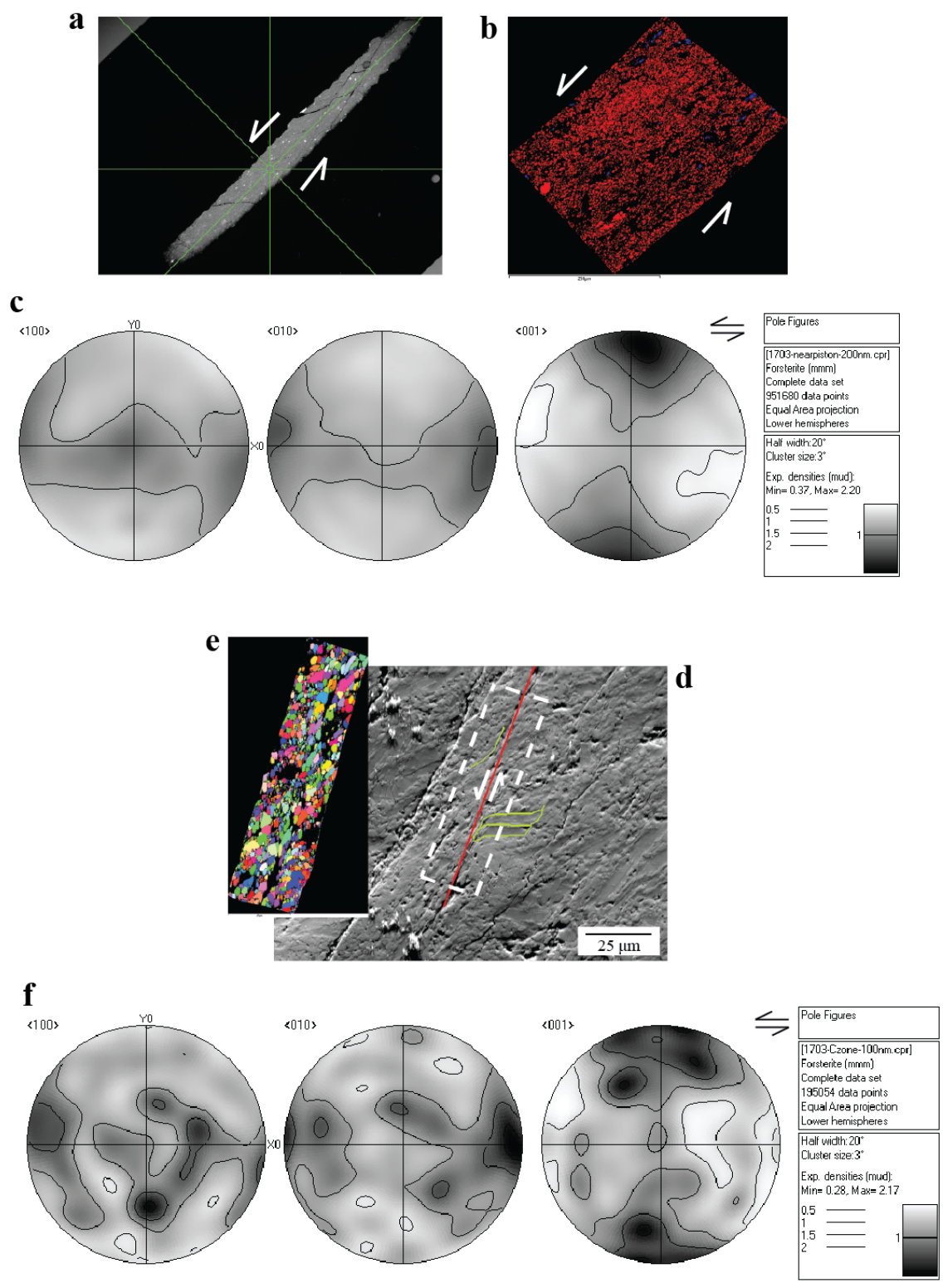


Figure 11

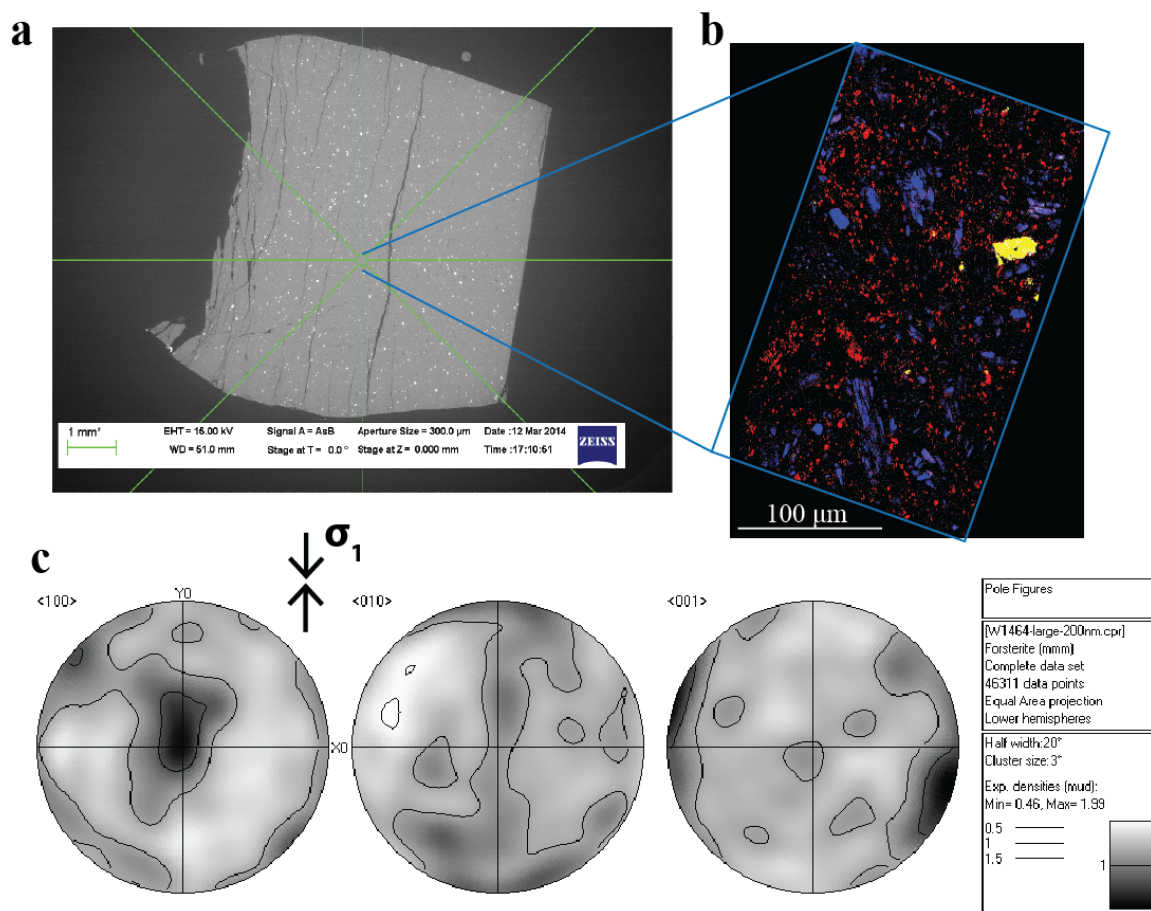


Figure 12

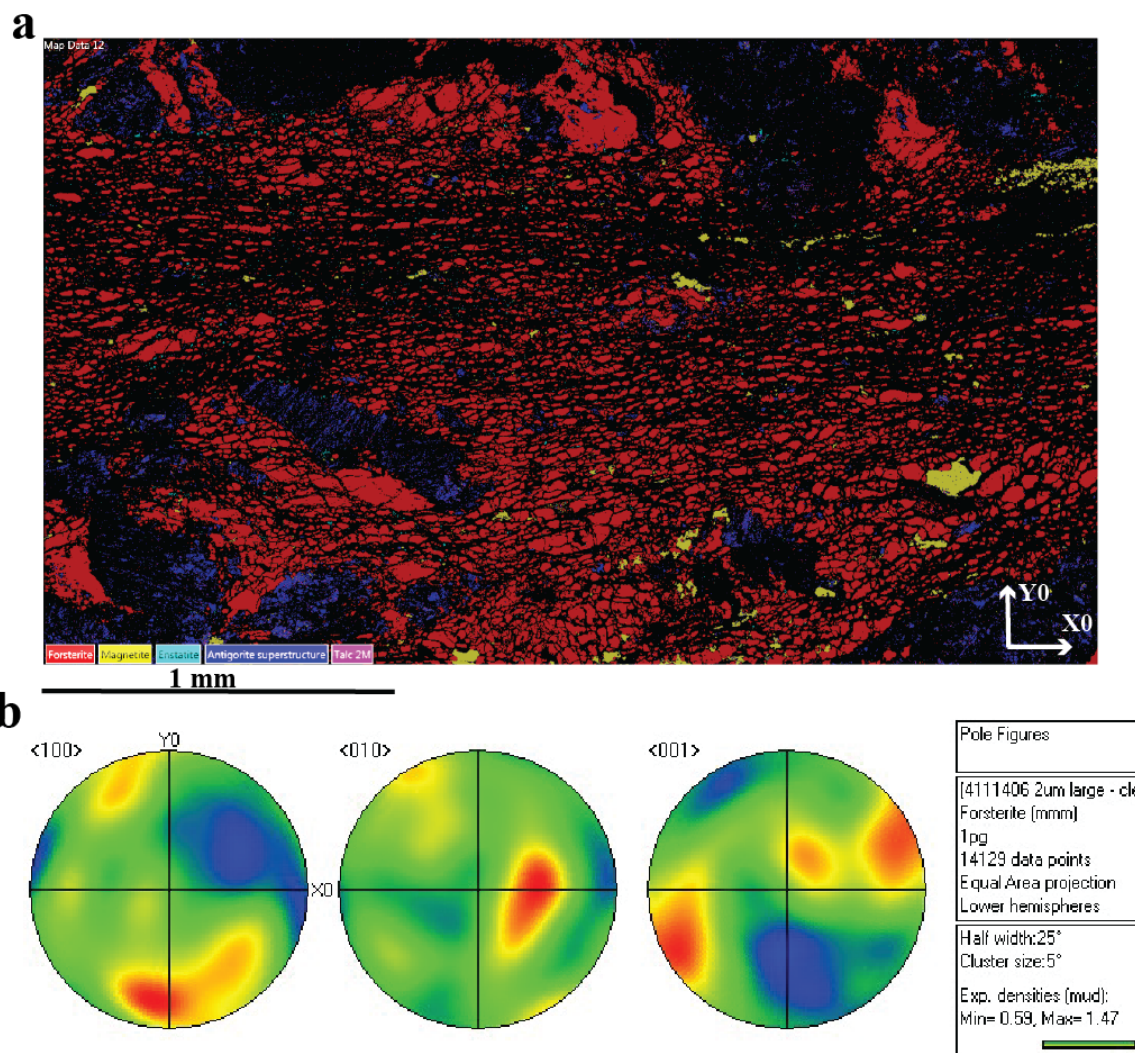


Figure 13

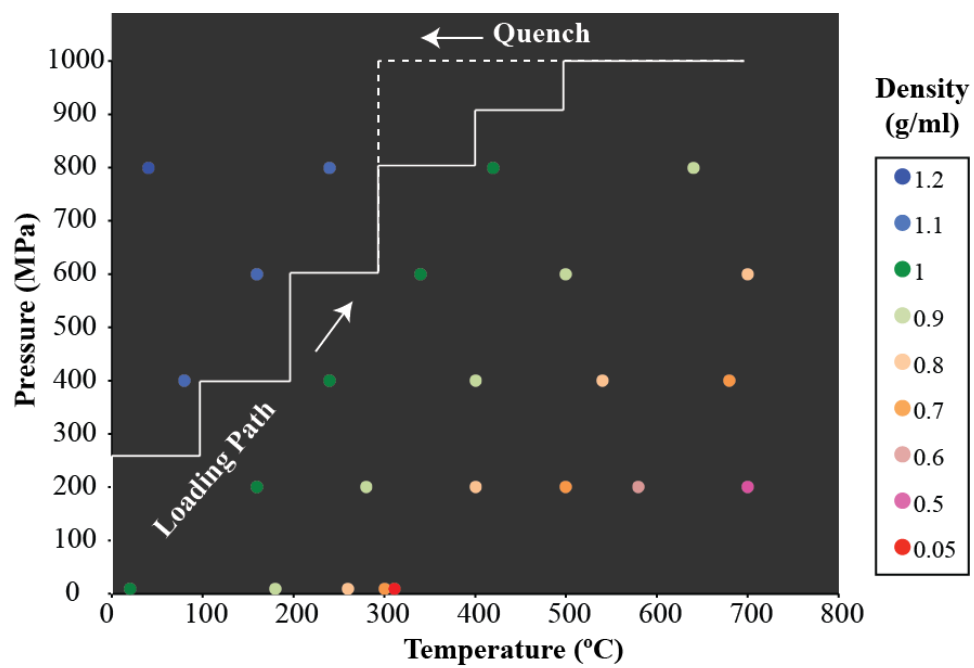


Figure A1