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Title: An experimental investigation on fluid transfer mechanisms in ultramafic rocks

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Abstract

Static annealing and crack-cook experiments were performed on wet and dry fabricated San Carlos olivine aggregates and a core of Balsam Gap dunite to investigate fluid infiltration mechanisms for low-permeability mantle rocks. Deionized water (30 to 40 μL) was added to cold-pressed olivine powders before hot-pressing to make wet samples; samples hot-pressed without added water are dry ($< 30 \text{ H}/10^6 \text{ Si}$). Talc was added to all samples to provide fluid during the experiments. All samples were annealed at 1200°C and 300 MPa confining pressure for three hours, but crack-cook experiments were subjected to a 300-500 MPa axial differential stress at 600°C and 125 MPa confining pressure before annealing. All samples contain abundant fluid inclusions (FIs) after annealing. Olivine aggregate samples show FI-rich and FI-depleted regions. Grain size was reduced in all samples, particularly in FI-rich regions. Crosscutting FIs associated with fractures are only widespread in the dunite. Fluids were primarily transferred from talc along grain boundaries in olivine aggregate samples and along fractures in dunite. FIs inhibit grain growth and grain-boundary migration, leading to smaller than expected grain sizes at the experimental conditions. Results have important implications where excess fluid may affect grain-size sensitive deformation mechanisms in the upper oceanic lithosphere.

1. Introduction

Volatiles incorporated into the solid Earth have an effect on many geological processes. Physical and chemical properties (e.g. rheology, seismic velocity, melting temperature, diffusion rate, etc.) of mantle rocks may be fundamentally influenced by the presence of

fluids, and therefore affect mantle processes relating to geodynamics, mantle convection, partial melting, melt transport, fluid-rock reactions, and ultimately, the planet-scale cycling of volatiles (Chopra and Patterson, 1984; Karato, 1990; Inoue, 1994; Kohlstedt et al., 1995; Hirth and Kohlstedt, 1996; Asimow and Langmuir, 2003; Holtzman et al., 2003; Hirshmann, 2006; Kohlstedt, 2006; King et al., 2010; Malvoisin et al., 2012; Plummer et al., 2012; Cerpa et al., 2017). Some of the world's most diverse lifeforms are found adjacent to mid-ocean ridge and transform systems, and rely on deep circulation of seawater through prolonged (10^4 - 10^5) hydrothermal vents (Francheteau et al., 1979; Rona, 1988; Von Damm, 1995; Fouquet, 1997; Gebruk et al., 1997; Tivey et al., 2003; Bach et al., 2004; Kelley et al., 2005; Fouquet et al., 2010; McCaig et al., 2010). Because volatiles influence numerous geological processes, and volatiles may themselves be modified as they pass through the hot oceanic lithosphere, it is important to have a thorough understanding of their physical/mechanical interaction with mantle rocks.

Widespread field evidence for fluid-rock interactions exists in the oceanic lithosphere. Evidence for substantial water in abyssal peridotites is lacking, but mineral alteration of peridotite in the presence of seawater to serpentine, talc, brucite, and amphibole is evident from ocean dredging samples and drill cores adjacent to mid-ocean ridge, transform, and oceanic detachment systems (Cannat, et al., 1992; Bach et al., 2004; Gose et al., 2009; Ildefonse et al., 2007; McCaig et al., 2010; Grimes et al., 2011; Hesse et al., 2015; Hodgkinson et al., 2015; Peslier, 2010; Schmadicke et al., 2011; Warren and Hauri, 2014; Warren, 2016; Li et al., 2017). Furthermore, geochemical

signatures of fluids emanating from hydrothermal systems and recovered from oceanic shear zone rocks indicate hydrodynamic interaction between seawater and ultramafic rocks above ambient temperature conditions (Charlou et al., 2000; Boschi et al., 2006; Paulick et al., 2006; McCaig et al., 2007; Schmidt et al., 2007; McCaig et al., 2010; Seyfried et al., 2011; Seyfried et al., 2015).

Fluids also strongly influence the rheologic behavior of olivine, the constitutive mineral of the upper mantle and ocean lithosphere (e.g. Kohlstedt et al., 1995). Numerous experiments have been conducted over the last several decades on wet and dry olivine single crystals, olivine aggregates, and dunite to examine how fluids may affect the rheologic behavior of olivine, and to improve the understanding of mantle processes in the presence and absence of water and other volatiles (e.g. Chopra and Patterson, 1981; 1984; Cooper and Kohlstedt, 1984; Karato, 1989; Nichols and Mackwell, 1991; van der Wal et al., 1993; Farver et al., 1994; Hirth and Kohlstedt, 1995a; 1995b; Farver and Yund, 2000; Bystricky et al., 2000; Jung et al., 2006; Sundberg and Cooper, 2008; Demouchy, 2010; Wang et al., 2010; Hansen et al., 2011; 2012a, b, c; 2014; Carter et al., 2015; Tasaka et al., 2016; Marquardt and Faul, 2018). Anhydrous olivine creeps 6 times slower compared to hydrous olivine when the two are deformed at similar experimental conditions (Chopra and Paterson, 1984; Karato et al., 1986; Mei and Kohlstedt, 2000a, 2000b; Kohlstedt, 2006). Deformation of polycrystalline olivine is intrinsically grain size sensitive and accommodates deformation via diffusion creep or dislocation creep with and without a component of grain boundary sliding (Nabarro, 1948; Herring, 1950; Coble, 1963; Twiss, 1977; Jung and Karato, 2001; Hirth and

Kohlstedt, 2003; Wang et al., 2010; Rozel et al., 2011; Hansen et al., 2011, 2012a, 2012b, 2012c; Hirth and Kohlstedt, 2015).

There is strong petrographical and geochemical evidence of interaction between ultramafic rocks and seawater, and a firm understanding of the rheologic behavior of olivine. Chemical and textural evidence suggests that fluid infiltration along cracks and grain boundaries may facilitate fluid penetration into ultramafic rocks (e.g. Schwarzenbach et al., 2016), but a discernable mechanism to explain how fluids infiltrate into low-permeability ultramafic rocks is not as clear. To improve the understanding of fluid mobilization mechanisms related to fluid transfer into ultramafic rocks, a suite of high-temperature annealing and crack-cook experiments were conducted on fine-grained, fabricated, olivine aggregates and a sample of coarse-grained, Balsam Gap dunite. Results from these experiments suggest that fluids may be mobilized along grain boundaries and/or fractures and provides an explanation for how excess fluid may be transferred into and (re)distributed within the oceanic lithosphere.

2. Methods

2.1 Sample preparation

A suite of annealing and crack-cook experiments was conducted on hot-pressed cylindrical samples of powdered San Carlos olivine grains and on a rock core drilled from Balsam Gap dunite (North Carolina). Olivine powders were measured for grain size using a Horiba LA-920 particle size analyzer at the LacCore facility at the University of Minnesota prior to being fabricated for the experiments. Grain diameters of the

powdered San Carlos olivine range from $< 1 \mu\text{m}$ to $\sim 50 \mu\text{m}$ with an average of $\sim 15 \mu\text{m}$ (Fig. 1).

<Fig. 1 here>

Olivine powders were first cold-pressed into Ni-canisters of 25 mm in length, with a 9 mm inner diameter (ID) and a 10 mm outer diameter (OD) (Table 1). Cold-pressed samples were subsequently hot-pressed with and without added water. Wet samples were fabricated by adding 30 to 40 μL of de-ionized water to cold-pressed powders prior to sealing the Ni-canister and subsequently hot-pressing. While olivine is nominally anhydrous, hydrogen diffuses into olivine at $\sim 10^{-10} \text{ m}^2/\text{s}$ at 1200°C and 300 MPa, where it is incorporated into the olivine crystal structure as intrinsic point defects whose concentration is proportional to the water fugacity, $C^{\text{OH}} \sim f^r \text{H}_2\text{O}$, where $r \sim 1$ (Mackwell and Kohlstedt, 1990; Bai and Kohlstedt, 1992, 1993; Kohlstedt et al., 1996; Demouchy and Mackwell, 2003; 2006). Any excess oxygen in the sample combines with the Ni-canister to produce NiO, and the Ni-NiO buffer is within the stability field for San Carlos olivine for the experimental conditions (e.g. Mackwell and Kohlstedt, 1990; Demouchy and Mackwell, 2003; 2006). The mass of the Ni-canister before and after the addition of olivine powders $\pm$ de-ionized water was not measured for any sample during sample preparation; therefore, the proportion of H_2O was estimated from the sample dimensions and mass of added water. Given the sample dimensions and the amount of water added (0.03 to 0.04 g), it was estimated that the amount of water available for wet hot-pressed samples may be as high as 0.6-0.8 wt% H_2O (Table 1). Because San Carlos olivine has a nominal H/Si

content of 5-30 ppm and the crystal lattice is considered saturated at ~300 ppm H/Si, it is assumed that samples hot-pressed with added water have saturated crystal lattices; any additional water may fill pore space or be trapped as fluid inclusions (Bai and Kohlstedt, 1992; 1993; Kohlstedt et al., 1996; Demouchy and Mackwell, 2003; 2006). For the purpose of the work presented here, samples hot-pressed without added water are considered to be “dry” and those with added water are considered “wet”.

<Table 1 here>

After the cold-press and the addition (or not) of de-ionized water, each Ni-canister was inserted into a formed Fe-jacket along with flat and parallel alumina and zirconium pistons, and was annealed (hot-pressed) for 3 hours at 1200°C and 300 MPa confining pressure in a gas-medium deformation rig (Paterson and Olgaard, 2000). Samples were quenched to ambient P-T conditions in under 20 minutes at the end of each hot-press, sufficiently fast within the first 600°C of cooling to prevent diffusion of hydrogen out of the sample (e.g. Mackwell and Kohlstedt, 1990; Demouchy and Mackwell, 2003; 2006).

2.2 Experimental procedures

Experiments on dry and wet samples were performed with varying amounts of talc, which dehydrates at experimental conditions and contributes water to the system. Based on the dimensions of the talc and sample, a complete dehydration of talc to enstatite at the experimental conditions are estimated to be as much as 3.2-7.2 wt% H₂O to the samples (Table 1). Given that the experimental conditions are unfavorable

for hydrous mineral growth, the potential excess fluid is expected to be trapped as fluid inclusions (FIs) in olivine (e.g. Lamadrid et al., 2017).

Crack-cook experiments (e.g., Druiventak et al., 2012) were conducted by wrapping a cylindrical sample of wet olivine aggregate and a sample of cored dunite with a talc jacket (wet ol + tlc jacket; PI-1603, and dunite + tlc jacket; PI-1610, respectively) to investigate how differential stress may influence fluid mobility in samples with drastically different initial fluid content and grain size (Fig. 2A). Annealing experiments were performed on dry olivine wrapped with a talc jacket (dry ol + tlc jacket; PI-1647) and on a cored-out, dry olivine cylinder with an inner talc cylinder (dry ol + tlc cylinder; PI-1748) to investigate fluid mobilization mechanisms under static conditions and whether or not the placement and volume of the talc had an impact on fluid transfer into the sample during annealing (Fig. 2A). The dimensions of all samples were measured prior to the experiments (Table 1). Porous alumina spacers (2-4 mm thick) were placed at the top and bottom of each sample + talc to promote fluid flow from the talc into the sample during the experiment.

<Fig. 2 here>

The assembly, consisting of the sample + tlc, porous alumina spacers, and flat and parallel alumina and zirconium pistons, was fitted into a formed Fe-jacket and inserted into a gas-medium deformation rig (Paterson and Olgaard, 2000). Pressure was first increased from 0.1 MPa to 100 MPa. Temperature was subsequently increased by ~ 1

°C/second. Once the temperature reached 900 °C, pressure and temperature were incrementally increased to 200 MPa and 1000 °C and then to 300 MPa and 1200 °C. Annealing experiments were held at static conditions of 1200 °C and 300 MPa for 3 hours. For crack-cook experiments, while pressure and temperature were held constant at 600 °C and 125 MPa, a 300 to 500 MPa axial stress (i.e. 2 to 3 times the confining pressure) was applied parallel to the long axis of the sample (Fig. 2A) for 5 to 10 minutes to induce cracking. After this crack phase, pressure and temperature were incrementally increased, similar to the annealing experiments, and the sample was annealed at static conditions of 1200 °C and 300 MPa for 3 hours for the “cook” phase.

2.3 Analytical methods

Doubly polished thin sections were made to characterize the starting wet and dry hot-pressed material as well as the experimental material. Radial thin sections were made from the starting material of wet and dry olivine aggregates and of cored dunite to preserve starting material for subsequent experiments (Fig. 2B). Both radial and axial thin sections were made from halved cylindrical samples of dunite and olivine aggregates, respectively, to characterize the experimental samples (Fig. 2B). All thin sections were ground to 20 to 30 µm thick, polished to a grade of 0.5 µm, and were examined with transmitted and reflected optical light microscopy. Owing to the small grain sizes and the size of FIs (see below), making the thin sections of this thickness helped to better observe the FIs while preserving them. Grain diameters were measured manually from scaled, cross-polarized light (XPL) thin section photographs using ImageJ.

208
209 Fourier-transform infrared (FTIR) spectra were acquired from a clean and polished
210 surface of the dry ol + tlc cylinder (PI-1748) sample using a Bruker Tensor 37
211 spectrometer with a Hyperion 2000 microscope. Spectra were obtained to determine the
212 volatile concentration and speciation within the sample. Only this sample was analyzed,
213 because it has the lowest potential water content (Table 1) and because material from
214 the other experiments was not available in sufficient quantity for FTIR analyses.
215 Hydroxyl content detected in this sample is, therefore, assumed to be present in
216 samples with higher estimated water content (Table 1).

217
218 The majority of inclusions in all the samples are small ($< 3 \mu\text{m}$ in diameter) and the
219 ability to observe phases within an individual inclusion is difficult. Recent unpublished
220 work has shown that samples hot-pressed under a vacuum contain ~2% fewer
221 “inclusions”/porosity compared to samples from a standard hot-press that was
222 performed in this study (Meyers et al., 2017). Moreover, it is possible that the smallest
223 of inclusions may not be “inclusions”, but rather partial-melt exsolutions (Raterron et al.,
224 1997; 2000). For simplicity of analysis, pores, partial-melt exsolutions, and/or FIs are all
225 referred to as FIs, whether fluid-filled or otherwise. Isolated FIs within olivine grains are
226 considered *intragranular* FIs, transgranular FIs that define planar trails are considered to
227 be *crosscutting*, and grain boundary FIs are considered *intergranular* FIs (e.g. Roedder,
228 1984). If present, the percentage of liquid and vapor phases within two-phase FIs at
229 ambient conditions are described.

230

<Fig. 3 here>

Fluid inclusion size, shape, and area percent were examined from thin section photographs with ImageJ. The wet and dry starting material was characterized prior to the experiments, and annealing and crack-cook samples were analyzed after each experiment. Samples that contain a lower abundance of FIs compared to the rest of the thin section are considered FI-poor, or FI-depleted, whereas adjacent areas that contain a higher abundance of FIs are considered FI-rich. Six photos in both FI-rich and FI-poor areas were taken of the upper thin section surface of each sample using a 40x objective lens in plane polarized light (PPL) with a confocal lens. However, only two photos could be taken in FI-poor domains of the wet ol + tlc jacket from the crack-cook experiment (PI-1603), because FI-poor domains are uncommon in this sample. Each photo was digitally edited to increase the contrast between FIs (dark) and olivine grains (white) and digital masks of FIs were identified with ImageJ to determine the size of FIs and the area percent of FIs relative to the sample (Fig. 3). Photos were taken only on the best-polished areas of the thin section where there were no substantial defects to allow for the most consistent comparison of FI content among the samples. The range of particle sizes of FIs was limited to 0.5 and 30 μm^2 , and circularity values were limited to 0.2 to 1.0 (1.0 being a perfect circle) to filter out noise from smaller particles, and to omit larger particles (e.g., incorporated phases from the San Carlos olivine powder) that were not considered to be FIs (Fig. 3). The range and average cross-sectional area and area percent of FIs in each sample is provided (Table 2). The dunite sample was not analyzed with ImageJ, because of its dissimilarity to the manufactured olivine samples.

Nonetheless, microstructural observations are described from this sample and compared to the other experiments.

<Table 2 here>

3. Results

3.1. Characterization of the starting material

3.1.1. Wet and dry hot-pressed olivine aggregate

Wet and dry hot-pressed samples were examined prior to anneal and crack-cook experiments. A freshly cut surface of the wet hot-pressed sample is light greyish green, whereas that of the dry hot-pressed sample is a darker pistachio green (Fig. 4A). In thin section, sub-equant to equant olivine grains for both wet and dry samples are 1-100 μm in diameter (average 15 μm), and are larger than the grains in the original powders (Figs. 1 and 5A; Table 2). Grains of 20-60 μm in diameter display different interference colors at the outer 2-4 μm of the grain rim, whereas smaller and the largest grain sizes do not display this variation (Figs. 5A-5D). Locally, 20-30 grains that are 15-85 μm in diameter form clusters in both wet and dry samples (Figs. 5C and 5D). Grain clusters in the dry hot-pressed sample have a microcrystalline matrix between larger grains (Fig. 5C), whereas those in the wet hot-pressed sample have 1-15 μm diameter olivine grains between larger grains instead of a microcrystalline matrix (Fig. 5D).

<Fig. 4 here>

<Fig. 5 here>

277
278 Intergranular and intragranular FIs are observed in both hot-pressed samples (Figs. 6A
279 and 6B). However, FIs are smaller and less abundant in the dry hot-pressed samples
280 compared to those in the wet samples (Table 2). Powders were not dried under a
281 vacuum, and likely contain pockets of air that manifest as pores, or FIs, in the hot-
282 pressed samples. The area percent FIs in the dry hot-pressed sample is 2-3 (average of
283 2), and is 3-9 (average 6) in the wet hot-pressed sample.

284
285 The morphology of intergranular and intragranular FIs is similar in both hot-pressed
286 samples. The cross-sectional area of FIs in the dry hot-pressed sample is 1.5-1.8 μm^2
287 (average 1.7 μm^2), whereas in the wet hot-pressed sample it is 1.8-2.6 μm^2 (average
288 2.2 μm^2) (Figs. 6A and 6B; Table 2). Intragranular FIs are circular to elongate in cross-
289 section, are $< 3 \mu\text{m}$ in diameter, and are either transparent with dark rims or appear
290 opaque in transmitted light (Figs. 6C and 6D). Intergranular FIs are irregularly shaped
291 and elongate, ranging from 2 to 10 μm^2 , rarely up to 30 μm^2 in cross-sectional area.
292 Intergranular FIs are dark and opaque in transmitted light and display irregular textures
293 (Figs. 6C and 6D). The majority of FIs are too small to observe phases present, as they
294 either appear empty or contain a single phase. Two-phase FIs (vapor and liquid) were
295 only observed in the wet hot-pressed sample and comprise $< 2\%$ of all observed FIs
296 (Fig. 6C). Where present, two-phase FIs contain $\sim 50\%$ vapor and $\sim 50\%$ liquid.

297
298 <Fig. 6 here>

299

3.1.2. *Cored dunite*

Balsam Gap dunite is dark green to black in hand sample with mm-scale grains of olivine, pyroxene, and an oxide phase (Fig. 4B). Thin sections reveal euhedral to sub-euhedral grains of 85-90% olivine, 8-10% pyroxene and 1-2% oxides (Figs. 5E and 5F). Olivine grains are 75 μm and up to 1 mm in diameter, pyroxene grains are 500 μm to 3 mm in diameter, and the oxide phase is 1-3 mm in diameter. FIs are rare in this starting material, likely making up < 1% of the thin section area (Figs. 6E and 6F). FIs are < 3 μm in diameter and observed along grains (intergranular) or define intragranular fluid inclusion planes that span < 30 μm in length (Figs. 6E and 6F). Where present, FIs display a single phase with dark rims.

3.2. *Characterization of material after crack-cook experiments*

3.2.1. *Wet ol + tlc jacket (PI-1603)*

A freshly cut surface of the sample is light greyish-green and does not appear substantially different in color compared to the wet hot-pressed starting material (Fig. 4A). Thin sections reveal that 85% of the thin section area is FI-rich whereas 15% is FI-poor (Fig. 7A). Irregular, FI-depleted areas are present as 100 to 200 μm wide, sub-horizontal zones that are at a minimum 200 μm long and range up to the length of the entire sample diameter (Fig. 7A). The area percent of FIs in FI-rich domains is 9-12 (average 10), whereas it is 2-4 (average 3) in FI-poor domains (Table 2). The sample is crosscut by sub-horizontal fractures that are 25-50 μm in width and sub-perpendicular to the stress load that was applied during the crack phase. Secondary FIs are not observed adjacent to these fractures.

<Fig. 7 here>

Intergranular FIs are more abundant than intragranular FIs, and their morphology also differs. Overall, the cross-sectional area of FIs in FI-rich domains is 6.7-12.4 μm^2 (average 9.0 μm^2), whereas it is 3.5-5.0 μm^2 (average 4.8 μm^2) in FI-poor domains (Figs. 8A, 8C and 8E; Table 2). Intragranular FIs are circular in cross-section, < 3 μm in diameter, and are translucent with dark rims (Figs. 8A, 8C, and 8E). Clusters of intragranular FIs (10s-100s of FIs) are observed in grains larger than 15 μm in diameter, but otherwise they are not common in this sample (Fig. 8B). Intergranular FIs are dark, and have highly irregular, elongate, and globular morphologies (Figs. 8A, 8C, and 8E). Similar to the wet hot-pressed sample, rare two-phase FIs (vapor and liquid) were observed, and where present they contain ~50% vapor and ~50% liquid (Fig. 8A).

Olivine grain diameters are smaller than the starting material and no clusters of larger grains were observed (Table 2). Olivine grains in FI-rich areas are 3-30 μm in diameter (average 11 μm), whereas grains are slightly larger in FI-depleted areas with 3-40 μm grain diameters (average of 14 μm) (Figs. 8B, 8D and 8F; Table 2). Grains are equant to sub-equant in FI-poor areas, whereas they are sub-equant to highly irregular and amoeboid in FI-rich domains.

<Fig. 8 here>

3.2.2. Dunite + tlc jacket (PI-1610)

Freshly cut surfaces of dunite reveal pale light green olivine grains and darker green pyroxene, which is overall much lighter in color compared to the starting material (Figs. 4B and 9A). In thin section, olivine grain diameters range 25-500 μm (average 200 μm) and are smaller than those in the starting material (Figs. 5E, 5F, and 9B; Table 2). Between larger grains of olivine and pyroxene, finer angular olivine grains of $< 40 \mu\text{m}$ in diameter are present (Fig. 9B). Pyroxene grains show no obvious change in size, but are highly fractured, particularly along their cleavage planes (Fig. 9B).

<Fig. 9 here>

Thin sections reveal abundant intragranular, intergranular, and secondary FIs (Figs. 9C, 9D, 9E, and 9F). Intragranular FIs are circular to elongate in cross-section, are 1-8 μm in diameter, and are transparent with dark rims (Figs. 9C and 9D). Intergranular and secondary FIs are irregular and elongate in cross-section and are commonly observed along olivine grain boundaries, conchoidal fractures in olivine, and cleavage planes in pyroxene (Figs. 9C, 9D, and 9E). Secondary and intergranular FIs display necking textures, are 2 to 4 μm in width, and are up to 15 μm in length (Figs. 9C, 9D, and 9E). Fluid phases are evident in both intragranular and intergranular FIs larger than 3 μm in diameter (Figs. 9D and 9F). Two-phase FIs are 70-90% vapor and 10-30% liquid at ambient temperature. Fluid phases are not easily observed in FIs smaller than 3 μm in diameter, and either display a single phase with dark rims or are too small to determine fluid phases (Fig. 9D).

3.3. Characterization of material after annealing experiments

3.3.1. Dry ol + tlc jacket (PI-1647)

The halved cylindrical hand sample is entirely light greyish green and is comparable in color to the wet ol starting material (Fig. 4A). Thin sections reveal the sample is 70-80% FI-rich and 20-30% FI-poor (Fig. **Error! Reference source not found.**A). FI-poor domains are present as heterogeneously distributed patches that are locally observed adjacent to fractures and along the margin of the talc jacket and porous alumina. The area percent of FIs in FI-rich domains is 3-5 (average 4), whereas in FI-poor domains it is 1-3 (average 2) (Table 2).

Sub-horizontal fractures are crosscut by sub-vertical fractures (Fig. 10A). Secondary FIs are present adjacent to sub-horizontal fractures as 50-100 μm wide zones and also along and between en échelon fractures; FIs are rare or non-existent near sub-vertical fractures (Figs. 10B-10E). Although secondary FIs are present adjacent to sub-horizontal fractures, the rest of the area surrounding the sub-horizontal fractures are relatively FI-poor (Figs. 10A, 10D, and 10E).

<Fig. 10 here>

Thin sections contain intragranular, intergranular, and secondary FIs (Fig. 11). In FI-rich domains, FIs have cross-sectional areas of 2.9-4.9 μm^2 (average 3.9 μm^2), whereas they are 2.5-3.7 μm^2 (average 2.9 μm^2) in FI-depleted regions (Table 2). Intragranular

FIs are circular to elongate in cross-section, are $< 3 \mu\text{m}$ in diameter, and are transparent with dark rims (Fig. 11E). Clusters of intragranular FIs (10s to 100s of FIs) are common in equant grains that are larger than $15 \mu\text{m}$ in diameter in both FI-rich and FI-poor areas (Figs. 11C, 11D, and 11F); these FI clusters are rare in grains smaller than $15 \mu\text{m}$ in diameter. Intergranular FIs and secondary FIs associated with sub-horizontal fractures are irregular and elongate in cross-section. No two-phase FIs were observed in this sample.

Olivine grains vary in size and shape depending on the area percent FIs, but overall the grain diameters are smaller than the starting material (Figs. 11A, 11B, and 11D; Table 2). Grains in FI-rich domains are $3\text{-}35 \mu\text{m}$ in diameter (average of $12 \mu\text{m}$) (Table 2). Grains in FI-poor domains have a comparable average grain diameter ($13 \mu\text{m}$) to FI-rich areas, but have a larger range in grain sizes of $3\text{-}70 \mu\text{m}$ in diameter (Figs. 11A and 11B; Table 2). Grains in FI-rich areas are sub-equant to irregular and amoeboid, whereas in FI-depleted domains they are equant to sub-equant.

<Fig. 11 here>

3.3.2. *Dry ol + tlc cylinder (PI-1748)*

Halved cylindrical hand samples are light green with a narrow ($< 1 \text{ mm}$) dark green zone adjacent to the inner talc cylinder (Fig. 12A). Thin sections reveal a FI-poor zone ($\sim 500 \mu\text{m}$ in width) adjacent to the talc cylinder, whereas the rest of the sample is relatively FI-rich (Fig. 12B). However, FI-poor bands of 100 to $400 \mu\text{m}$ in width radiate out from the FI-

poor zone adjacent to the inner talc cylinder toward the outer diameter of the sample (Fig. 12B). The area percent of FIs within FI-rich domains is 3-8 (average 6), whereas it is 1-3 (average 2) in FI-poor domains (Table 2).

The FIs are larger and more irregular in FI-rich domains. The cross-sectional area of FIs in FI-rich domains is 2.4-3.0 μm^2 (average 2.5 μm^2) and is 1.5-2.1 μm^2 (average 1.8 μm^2) in FI-poor regions (Table 2). Intragranular FIs are ubiquitous throughout the sample, are circular to elongate in cross-section with diameters of $< 3 \mu\text{m}$, and are either dark/opaque or are transparent with dark rims (Figs. 12C and 12E). Intergranular FIs are irregular and elongate along grain boundaries and triple junctions (Fig. 12E). No two-phase FIs were observed in this sample.

Grain size and shape are observed to vary with FI density (Figs. 12C-12F). In FI-rich areas, grains are 3-17 μm in diameter (average 8 μm) (Figs. 12C and 12D; Table 2). Grain diameters in FI-poor domains are 4-26 μm (average 9 μm) (Figs. 12E and 12F). Grains are equant to sub-equant throughout the sample, but tend to be more irregularly shaped in FI-rich domains (Figs. 12C-12F).

<Fig. 12 here>

Fourier-transform infrared spectroscopy (FTIR) from the dry ol + tlc cylinder (PI-1748) indicates hydroxyl content in both the olivine crystal lattice and inclusions (Fig. 13). A broad peak over 3600 to 3200 cm^{-1} represents OH in the FIs, whereas the peak at 3600

cm⁻¹ is typical of OH in the olivine crystal lattice. F-H and CO₂ were also detected in this sample. Owing to standard hot-pressing it is thought that most of the detectable CO₂ in the sample is trapped air (Meyers et al., 2017). Because the dry ol + tlc cylinder sample had the lowest available water content (Table 1), it is assumed that samples with higher potential water content also contain water as inclusions and in the olivine crystal lattice.

<Fig. 13 here>

4. Discussion

The results from the suite of anneal and crack-cook experiments suggest an important correlation between available fluid content and FI size, shape, and abundance. Fluid availability also appears to influence the size and shape of olivine grains. In olivine aggregate samples, fluids are thought to have been transferred from dehydrating talc into the sample along grain boundaries, whereas in dunite the dominant fluid infiltration mechanism was along fractures. FIs appear to inhibit grain growth and grain-boundary migration, leading to smaller grain sizes than would be expected at these experimental conditions.

4.1 Fluid infiltration mechanisms

Observations indicate that abundant water was available in all experimental samples. FI size and area percent FIs are higher compared to the starting material in all olivine aggregate samples, and FTIR results from the dry ol + talc cylinder experiment confirm that talc dehydration contributed water to the sample during the experiments (Figs. 13

and 14; Table 2). For example, the sample with the highest potential water content (wet ol + tlc jacket; PI-1603) has the highest area percentage of FIs as well as larger and more irregular FI shapes compared to all other olivine aggregate samples (Figs. 8, 10, and 11; Table 1). FIs are also abundantly evident in the dunite + tlc jacket sample (PI-1610), particularly along healed fractures (Fig. 9). From these observations, it is evident that excess fluid was successfully transferred from the dehydrating talc into all samples, no matter if they were initially wet or dry, and under both static (annealing) and stressed (crack-phase) conditions.

<Fig. 14 here>

Fluid inclusion rich and poor domains were not observed in any starting material sample or in the dunite specimen after the crack-cook experiment (Figs. 5A, 5B, 5E, and 9B). Yet, all olivine aggregate samples that were annealed with talc contain FI-rich and FI-depleted domains after annealing; samples with lower available water content contain fewer FI-rich regions (Figs. 7A, 10A, 11A, and 12B). It is interpreted that grain size heterogeneities may have been introduced to olivine aggregate samples during cold-pressing, or possibly hot-pressing, and are thought to have influenced the abundance and nature of FI domains. For example, sub-horizontal FI-depleted domains in the wet ol + tlc jacket sample (PI-1603) may represent layering (anisotropy) that developed during cold-pressing of olivine powder into the Ni-canister (Fig. 7A). In other cases, irregular patches of FI-depleted areas in the dry ol + tlc jacket (PI-1647) and the dry ol + tlc cylinder (PI-1748) may be related to larger grain clusters that were observed in the

starting material after a hot-press (Figs. 5D, 10A, and 12B). During the annealing phase of the experiments, these clusters of coarser grains may have had less fluid penetrate into them compared to finer grains in the rest of the sample.

In the dry ol + tlc cylinder sample (PI-1748), an anomalous FI-depleted zone formed adjacent to the inner talc cylinder during the annealing experiment (Fig. 10A); this type of zone was not observed in any other experiment with talc (Figs. 7A and 12B). This observation suggests that, despite being closest to the fluid source (i.e. talc), less fluid was trapped within this zone as intragranular or intergranular FIs compared to the rest of the sample. Dry olivine nearest the talc in this zone may have incorporated hydrogen into its lattice during an initial phase of talc dehydration and promoted an early phase of grain growth (e.g. Karato, 1989). After the zone experienced more rapid grain growth compared to the rest of the sample, inhomogeneous diffusion may have delivered hydrogen and excess fluid distally from the talc where FI-depleted bands and FI-rich areas formed (Fig. 12B). Fluid from the talc may have also simultaneously entered the porous alumina and subsequently diffused into areas of the sample away from the talc. In either case, the position of the talc (outer vs. inner) seems to have an effect on the development of FI-rich vs. depleted zones in this sample.

Evidence of stress-induced fracturing from the “crack phase” is only apparent in the dunite + tlc jacket sample (PI-1610). The dunite sample contains numerous fractures and associated crosscutting FIs exist along internal cracks in olivine grains and cleavage planes in pyroxene (Figs. 9B, 9C, 9E, and 15A-15D). Fractures in olivine

aggregate samples are not widespread and likely formed only during talc dehydration (crosscutting FIs present) and/or when the sample was quenched at the end of the experiment (no FIs observed). Fractures in the wet ol + tlc jacket crack-cook experiment (PI-1603) likely formed during quenching, because they do not contain associated crosscutting FIs along or adjacent to them. Further, these fractures are oriented sub-perpendicular to the applied stress load and centered within FI-poor zones where grain size is larger than the rest of the sample (Fig. 7D and 7E). Fluid-filled fractures in the annealing experiment on dry ol + tlc jacket (PI-1647) likely formed during talc dehydration, evidenced by FIs along sub-horizontal and sub-vertical fractures, and are crosscut by later-formed decompression fractures that developed during quenching (Fig. 10). Fractures in olivine aggregate samples are only evident in areas with larger relict grain sizes in FI-poor domains (i.e. wet ol + tlc jacket) and/or samples that had low initial water contents with a talc jacket (dry ol + tlc jacket). The only sample where fracturing seems to have played a substantial role in fluid infiltration is the coarse-grained dunite + tlc jacket sample (PI-1610).

<Fig. 15 here>

The abundance of FIs along grain boundaries in all olivine aggregate samples suggests that fluids likely entered these samples along grain boundaries. Once the olivine crystal lattice is hydrogen-saturated (~300 ppm H/Si) excess incompatible hydrogen (i.e. fluid) may mobilize along the 0.4–1.2 nm grain boundary (Karato, 1989; Bai and Kohlstedt, 1992; 1993; Kohlstedt et al., 1996; Marquardt and Faul, 2018). Fluid infiltration along

fractures does not appear to be a dominant mechanism for fine-grained olivine aggregate samples, whether a stress load was applied or not.

4.2 The effect of FIs on olivine grain size

Observations from all olivine aggregate samples suggest that a larger area percent of FIs correlates with smaller grain diameters after annealing (Figs. 5, 7C, 7D, 9B, 11B, 11D, 12D and 12F; Table 2). Grain diameters within FI-rich domains, where present, in all olivine aggregate samples are smaller and more irregularly shaped compared to those in FI-depleted zones (Figs. 7C, 7D, 8D, 8F, 11B, 12D, and 12F; Table 2). FIs seem to have restricted grain growth and grain-boundary migration during annealing.

Water in a saturated olivine lattice (i.e. as intrinsic point defects) may promote grain growth (e.g. Karato, 1989), but grain boundary mobility may be prevented, depending on the grain boundary velocity and the drag force imposed by impurities (secondary phases) on the mobile grain boundary. Grain boundaries that pass over wetted grain boundaries may initially trap fluid as intragranular FIs, whereas subsequent grain-boundary migration may drag, drop, and/or break-up larger FIs (Roedder, 1984; Wanamaker and Evans, 1989; Karato, 1989; Schenk and Urai, 2005; Schmatz et al., 2011). An initial stage of abnormal grain growth during hot-pressing is interpreted from the presence of internal crystal defects (pores/FIs) in the dry ol starting material (Figs. 6B, 6D, and 15E), observed intragranular FIs in the wet ol starting material (Figs. 6A and 6C), larger grain sizes in the hot-pressed material compared to the powdered material (Figs. 1, 5A, and 5B), and larger grain clusters without groundmass between

them in the wet ol sample (Figs. 5C and 5D) (Cooper and Kohlstedt, 1984; Karato, 1989).

During annealing, subsequent grain growth and grain-boundary migration may have been inhibited (i.e. Zener pinning) by the presence of a secondary phase (FIs) (e.g. Olgaard and Evans, 1986; Manohar et al., 1998; Rollett, 2004; Roessiger et al., 2014). Relict larger grains that contain clusters of intragranular FIs appear to have been preserved from the hot-pressed material after the annealing phase in the experiments, whereas the majority of the smaller olivine grains contain fewer intragranular FIs and abundant FIs along their grain boundaries (Figs. 8B, 11C, 11F, and 15F). Reactivated strain energy introduced to the sample during hot-pressing (Fig. 15G, number 1), coupled with recrystallization under limited grain boundary mobility from Zener pinning, may have led to an overall reduction in grain size in the experimental samples (Fig. 15F). Because water is non-wetting in olivine (dihedral angle of $> 60^\circ$) excess water in the samples are confined to inclusions (e.g. Watson and Brenan, 1987). Furthermore, excess fluid may have promoted sub-micron-scale partial-melt exsolutions at grain triple junctions (Raterron et al.1997; 2000). Grain-boundary migration may have redistributed and swept earlier-trapped intragranular FIs to the grain boundary (Fig. 15G, numbers 2 and 3) during the earlier phases of the experiment, but was subsequently restricted by excess fluid, and/or partial-melt exsolutions, at the grain boundary as well as additional fluids infiltrating from talc along the grain boundary. In either case, it is unlikely that the sample was able to achieve complete recovery during the annealing period of only 3 hours (Farla et al., 2011).

576
577 The volume of fluid and gas phases observed in intragranular FIs at ambient
578 temperature as well as the textural evidence of FIs support FI re-equilibration and/or
579 modification during annealing. At the experimental conditions, the density of pure H₂O
580 at the experimental conditions is predicted to be 0.367 g/cm³ (~49 cm³/mole) (e.g. Pitzer
581 and Sterner, 1994). Given the density of liquid H₂O at ambient conditions is ~1.0 g/cm³,
582 a FI that traps pure H₂O with a density of 0.367 g/cm³ would contain ~ 63 volume
583 percent vapor at ambient temperature, provided that the FI was not re-equilibrated (e.g.
584 Bodnar, 1983). Phase ratios of a FI at ambient conditions do not constrain the P-T
585 formation conditions, rather a FI may have been trapped (or re-equilibrated) at any P-T
586 condition lying on or close to the 0.367 g/cm³ isochore. In our samples, only a few
587 intragranular FIs in the dunite + tlc jacket sample (PI-1610), the wet ol starting material,
588 and the wet ol + tlc jacket sample (PI-1603) display ~ 63% of vapor at ambient
589 temperature (Figs. 6C, 8A, 9D, and 9F), whereas the vast majority of intragranular FIs in
590 olivine contain > 90% vapor or were too small to observe individual phases (Figs. 6A,
591 6C, 6D, 11E, and 11F). FIs in the samples also do not likely contain pure H₂O, but
592 rather contain other volatiles that were present in the starting material (i.e. CO₂) and/or
593 minor components of dissolved olivine. Nonetheless, FIs in all experimental samples
594 display differing phase ratios at ambient temperature, indicating some re-equilibration
595 during the experiment. Furthermore, the morphology of intergranular FIs is similar to
596 those observed by Vityk and Bodnar (1995) for isothermal compression or isobaric
597 cooling, suggesting internal underpressure in the FIs from this study (Boullier et al.
598 1989; Sterner and Bodnar 1989; Herms and Schenk 1992; Bakker and Jansen 1994;

Vityk et al. 1994). We interpret that during an early phase of grain-boundary migration, larger intragranular FIs in olivine may have been broken up and/or swept to the grain boundary, coalescing into three-dimensional networks (Fig. 15E-G).

4.3 Implications for future experiments and natural systems

The results of the suite of experiments have important implications for experiments involving excess fluid. The presence of FIs may impact grain-size sensitive deformation mechanisms during deformation experiments (Karato et al., 1986; Mei and Kohlstedt, 2000a, 2000b; Kohlstedt, 2006; Hansen et al., 2011; 2012). Mantle rocks with large initial grain sizes deformed in the presence of fluids may accommodate deformation via fracturing, healing rapidly at ambient temperature, and trapping intergranular and crosscutting fluid inclusions (Figs. 15A-15D). Grain growth of olivine containing numerous FIs may be restricted during recrystallization, leading to a reduction in grain size (Figs. 145-145). Furthermore, the introduction of micron-scale FIs may lead to the formation of partial-melt exsolutions, promoting the onset of partial melting in ultramafic rocks (e.g. Raterron et al. 1997; 2000). Rocks containing FIs that are susceptible to long-term tectonic shearing may redistribute fluids (or melt) into localized bands (Fig. 15H). Indeed, many high-temperature shear experiments conducted with fluids, or low viscosity materials for that matter (i.e. melt), have shown that the low-viscosity phase will align into low-angle (C'-type) bands during deformation (Schmocker, 2003; Holtzman et al., 2003; Kohlstedt et al., 2010; Rybacki et al., 2008; King et al., 2009; Carter et al., 2015). Therefore, past and future experimental results involving fluids may

have to be viewed in light of the implications of this study on predicted deformation mechanism(s).

Although the conditions of the experiments are unlikely to be encountered in nature, the results suggest viable fluid infiltration mechanisms into low-permeability ultramafic rocks. Abundant fluids that are introduced into rocks of the oversaturated upper oceanic lithosphere may influence the grain size of the host rock, and may lead to altering the host rock's rheologic behavior and/or its melting temperature. For example, fluids introduced at depth in transform and oceanic detachment systems may promote strain partitioning, shear zone development, and exhumation of ultramafic rocks (Hirth and Kohlstedt, 1996; Mei and Kohlstedt, 2000a, 2000b; Bach et al., 2004; Ildefonse et al., 2007; McCaig et al., 2010; Grimes et al., 2011).

5. Conclusions

High-temperature annealing and crack-cook experiments of olivine aggregates and dunite indicate fluids may enter samples primarily along grain boundaries and in some cases along fractures. FI-rich and FI-poor domains developed in olivine aggregate experiments, suggesting inhomogeneous diffusion of water into the sample. FIs act as secondary phases (i.e. Zener pinning) that inhibit grain growth and may lead to a reduction in grain size.

Where water is introduced into the mantle in excess (i.e. in the mantle wedge and/or ridge-transform intersections) processes examined in this study have the potential to

occur. These results help to explain mechanisms to transfer fluids into low permeability rocks in the upper mantle and may help to understand processes relating to upper ocean lithosphere shear zone development, exhumation of mantle rocks, as well as mantle convection and planet-scale cycling of fluids.

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Tables

Table 1: Sample dimensions and potential H₂O content for starting material and all experiments.

Table 2: Grain size, mean FI cross sectional area, and area percent FIs for the starting material and for FI-rich and FI-depleted domains in crack-cook and annealing experiments.

Figure Captions

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Figure 2: (A) Configuration of the sample + tlc jacket (left) and sample + tlc cylinder (right) for the experimental setup. Note, the orientation of the axial stress load (σ_{axial}) is indicated on the sample + tlc jacket sample configuration. (B) Radial (left) and axial (right) thin section orientations that were made for microstructural analysis.

Figure 3: Example of image editing and processing with ImageJ from the wet hot press. The raw image (left image) was digitally enhanced (center image), black masks were created for FIs using applied thresholds (see text) and can be distinguished from white olivine grains (right image).

Figure 4: (A) Photo of dry and wet San Carlos olivine aggregate hand specimen of hot-pressed samples. Note, the lighter color of the wet sample. (B) Photo of the cored dunite sample. Note, the dark color of the sample.

Figure 5: Images of wet (A) and dry (B) hot-pressed samples in cross polarized light (XPL) to show size, shape and distribution of grain sizes. Images in XPL of wet (C) and dry (D) hot-pressed samples to show clusters of larger grains that formed during hot pressing. Note, the groundmass between grains in (D), which is absent in the wet hot press (C). Image of cored dunite in XPL (E) and PPL (F) to show grain size and distribution of olivine grains (E and F) and initial fracture distribution (F). All images are from radial thin sections.

Figure 6: Images of wet (A) and dry (B) hot-pressed samples in plane polarized light (PPL) to show size, shape and distribution of FIs (indicated). Note the higher abundance of FIs in the wet sample (A). Photomicrographs in PPL of wet (C) and dry (D) hot-pressed samples to show the general size and morphology of FIs. Note, rare two phase FIs exist only in the wet hot-pressed samples (L-liquid, V-vapor). Examples of irregularly shaped FIs (i) are indicated in (C) and (D). Figs. 6C and 6D are from Carter et al., 2015, and Fig. 6C is modified. (E) Image of cored dunite in PPL to show rare FIs (indicated). Note the small trail of FIs does not cross grain boundaries. (F) Image of cored dunite in PPL to show small size of FIs. All images are of radial thin sections.

Figure 7: Images from an axial thin section from wet ol + tlc jacket (PI-1603). (A) PPL image shows overall FI-rich domains (dark) with sub-horizontal FI-depleted domains (light). White box indicates the approximate image location for (B). Images in PPL (B) and XPL (C) to show the nature of the FI-depleted (light) and FI-rich domains (dark).

Note the sharp boundary between the two regions and the larger grain size in the FI-depleted area. (E) Image in PPL to show a FI-depleted zone along a crack in the sample. (F) Close up of image in (E) in XPL to show larger grain size in the zone adjacent to the crack. Note, the orientation of the axial stress direction is indicated (white arrows).

Figure 8: Images from an axial thin section from wet ol + tlc jacket (PI-1603). (A) PPL image to show the irregular morphology of FIs (indicated) observed throughout the sample. Note, arrows point to FIs that are sketched in insets to show rare two phase inclusions (L – liquid, V – vapor). (B) XPL image to show intragranular FI clusters that are present in larger olivine grains (diameters of $> 15 \mu\text{m}$). (C) Image in PPL of a FI-poor region surrounded by darker domains. Note, the morphology of FIs in the lighter region along grain boundaries. (D) Same image as (C) in XPL to show the variation in grain size. (E) PPL image of FI-rich domain. Note, the abundance of irregular FIs in this domain compared to (C) and (D). (F) Same image as (E) in XPL to show the variation in grain size and shape.

Figure 9: (A) Hand sample of dunite + tlc jacket (PI-1610) to show the lighter color compared with the starting material (Fig. 4B). Scale bar is in mm. (B) PPL image of dunite + tlc jacket (PI-1610) to show the reduction in grain size and increase in fracture abundance compare to the starting material (Figs. 5E and 5F). Box indicates photo area for (E). (C) PPL to show abundant intragranular and intergranular FIs in and between olivine grains, respectively. White box indicates the approximate location of (D). (D)

Magnified image of (C) to show morphology and phases of FIs. Note, elongate FIs along grain boundaries (GB). Inset indicates the phases present in the FIs (L- liquid, V- vapor). (E) PPL image of a pyroxene grain to show abundant two-phase FIs along cleavage planes. White box is approximate location of (F). (F) Magnified image of (E) to show morphology and phases of FIs. Inset indicates the phases present in the FIs (L- liquid, V-vapor). Images (B) through (F) are from radial thin sections.

Figure 10: Images from an axial thin section from dry ol + tlc jacket sample (PI-1647). (A) Full axial thin section in PPL shows sub-vertical fractures crosscut sub-horizontal fractures. Boxes for (B) through (E) are noted. (B) PPL image shows abundant FIs (indicated) along and adjacent to fractures. (C) Same image as (B) but in reflected light FIs appear white. (D) PPL image to show numerous crosscutting FIs adjacent to fractures. (E) PPL image to show FIs between en échelon fractures.

Figure 11: Images from an axial thin section from dry ol + tlc jacket sample (PI-1647). (A) PPL image to show FI-rich and FI-poor domains. (B) Same image as (A) in XPL to show grain size variation as a function of FI density. (C) PPL image to show dense FI clusters in larger grains (white arrows). Inset highlights one of the larger grains. (D) Same image as (C) in XPL to show grain sizes. (E) Image in PPL to show size and morphology of FIs (indicated). Note the lack of two phases in all FIs. (F) PPL image to show circular FIs along the grain boundaries of a circular grain.

Figure 12: Images from the dry ol + tlc cylinder (PI-1748) sample. (A) A halved hand sample to show a dark zone in ol adjacent to tlc (indicated). The black box outlines the approximate area of (B). Scale is in mm. (B) XPL image of half an axial thin section to show a FI-poor zone adjacent to the tlc and FI-poor bands (indicated) that run from the FI-poor zone to the edges of the sample. Note the larger grain size in FI-poor zones. (C) PPL image shows FI density and morphology in an FI-poor domain. Note dark grain boundaries (GB; black arrow). (D) Same photo as in (C) in XPL to show grain size. (E) PPL image of a FI-rich zone to show FI morphology and density. Note the contrast to (C). (F) Same photo as (E) in XPL. Note the smaller grain size compared to (D).

Figure 13: FTIR results from the dry ol + tlc cylinder experiment (PI-1748). Note the broad peak over 3600 to 3200 cm^{-1} is the OH in the FIs, whereas the peak at 3600 cm^{-1} is typical of OH in the olivine crystal lattice. The peak at 3712 cm^{-1} is likely related to F-H, and not shown are peaks relating to CO_2 .

Figure 14: Images of representative areas of FIs (dark) and olivine (light) for both starting material and experimental samples (not including dunite) in PPL to show an increase in FI area percent with available water content. Note the increase in abundance and size of intergranular FIs (dark) at higher water contents. Scale is the same for all photos.

Figure 15: Schematic representation of (A) the initial dunite sample containing olivine and pyroxene (pyx), (B) the dunite sample after the crack-phase, note the light grey

lines represent fractures (healed and unhealed) and areas for (C) and (D) are indicated with grey boxes, (C) olivine grain that contains abundant intergranular and crosscutting two-phase FIs (see also Fig. 9D), (D) the pyroxene grain contains two-phase FIs along cleavage planes, (E) grains and FIs of the initial wet ol hot press, (F) grains and FIs after the annealing phase, (G) the interpreted interaction of grain recrystallization and FI migration to grain boundaries and triple junctions (see Fig. 15F), note that intergranular FIs increase at the expense of intragranular FIs, but intragranular FIs are not eradicated, see text for the explanation to the numbers (1-3), (H) the result of deforming wet ol aggregates in shear. Note FIs are indicated in grey and are align into C'-bands. Shear direction and deformation conditions are indicated.

LIST OF TABLES

Sample ID	Starting Material or Experiment type	Sample description	Sample properties				Talc properties			total weight of sample (g)	wt H ₂ O added before hot press (g)	wt H ₂ O from complete talc dehydration (g)	total available wt % H ₂ O
			radius (mm)	height (mm)	weight (g)	outer diameter (mm)	inner diameter (mm)	weight (g)					
N/A	dry hot press	dry ol	4.5	25	5.3	-	-	-	5.3	-	-	-	
N/A	wet hot press	wet ol	4.5	25	5.3	-	-	-	5.3	0.03-0.04	-	0.6-0.8	
PI-1603	Crack-cook	wet ol + tlc jacket	4.5	4.0	2.3	11	9.0	1.6	3.9	0.03-0.04	0.3	7.2	
PI-1610		dunite + tlc jacket	5.0	25	6.5	13	10	3.7	10.3	-	0.7	6.5	
PI-1647	Annealing	dry ol + tlc jacket	4.5	5.4	1.2	10.5	9.0	0.3	1.5	-	0.06	4.2	
PI-1748		dry ol + tlc cylinder	1.8 ^a	4.0 ^a	0.6 ^a	9.0 ^b	3.7 ^b	0.1 ^b	0.8	-	0.02	3.1	
^a Dimensions/weight of the inner talc cylinder													
^b Dimensions/weight of the olivine jacket													
N/A - Not available													

^aDimensions/weight of the inner talc cylinder

^bDimensions/weight of the olivine jacket

N/A - Not available

Table 1. Sample dimensions and potential H₂O content for starting material and all experiments.

Sample ID	Starting Material or Experiment type	Sample description	FI domain type (rich or depleted)	Range of grain diameters (& average) [μm]	Total available wt % H_2O	Range of area % FIs (& average)	Range of FI cross sectional area (& average) [μm^2]
N/A	dry hot press	dry ol	N/A	1-100 (15)	-	1.6-2.8 (2.2)	1.5-1.8 (1.7)
N/A	wet hot press	wet ol	N/A	1-100 (15)	0.6-0.8	2.9-8.8 (5.9)	1.8-2.6 (2.2)
PI-1603	Crack-cook	wet ol + tlc jacket	FI-rich	3-30 (11)	7.2	8.8-12.1 (10.3)	6.7-12.1 (10.3)
PI-1610		dunite + tlc jacket	FI-depleted*	3-40 (14)*		2.0-3.5 (2.8)*	3.5-5.0 (4.3)*
PI-1647	Annealing	dry ol + tlc jacket	N/A	75-1000	6.5	N/A	N/A
PI-1748			FI-rich	3-35 (12)	4.2	3.2-5.2 (4.3)	2.9-4.9 (3.9)
			FI-depleted	3-70 (13)		1.2-2.5 (2.0)	2.5-3.7 (2.9)
		dry ol + tlc cylinder	FI-rich	3-17 (8)	3.1	3.4-8.1 (5.9)	2.4-3.0 (2.5)
			FI-depleted	4-26 (9)		1.3-2.6 (1.9)	1.5-2.1 (1.8)

*Note: Only two photos could be taken of the FI-depleted region in PI-1603 for the ImageJ analysis

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Table 2. Grain size, mean FI cross sectional area, and area percent FIs for the starting material and for FI-rich and FI-depleted domains in crack-cook and annealing experiments.

LIST OF FIGURES

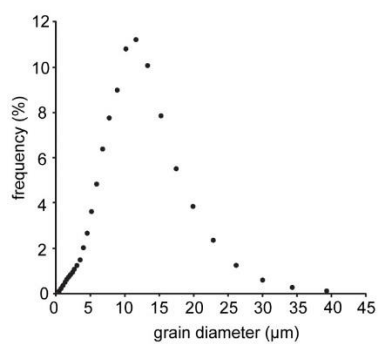


Figure 1. Plot of grain size (μm) vs. frequency (%) of powdered San Carlos olivine used for experiments.

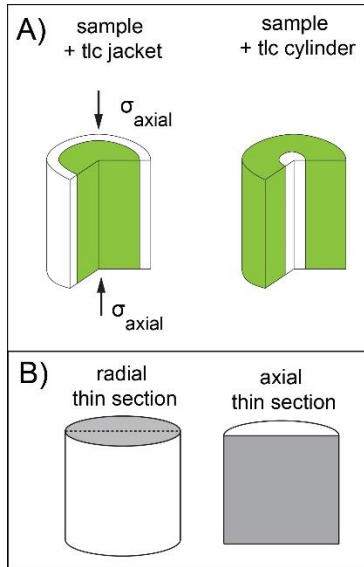


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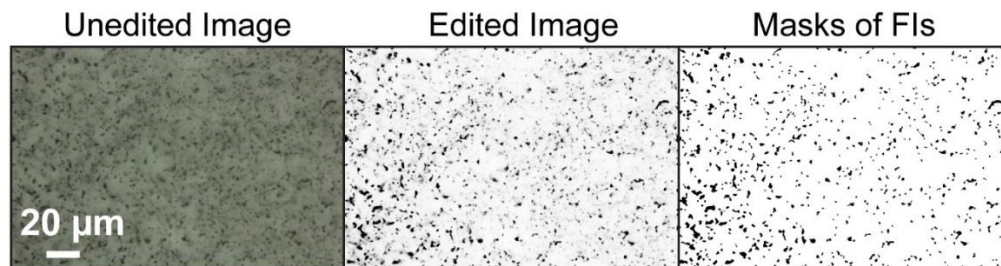


Figure 3. Example of image editing and processing with ImageJ from the wet hot press. The raw image (left image) was digitally enhanced (center image), black masks were created for FIs using applied thresholds (see text) and can be distinguished from white olivine grains (right image).

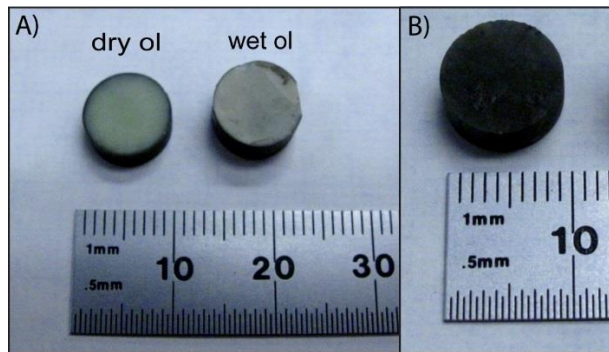


Figure 4. (A) Photo of dry and wet San Carlos olivine aggregate hand specimen of hot-pressed samples. Note, the lighter color of the wet sample. (B) Photo of the cored dunite sample. Note, the dark color of the sample.

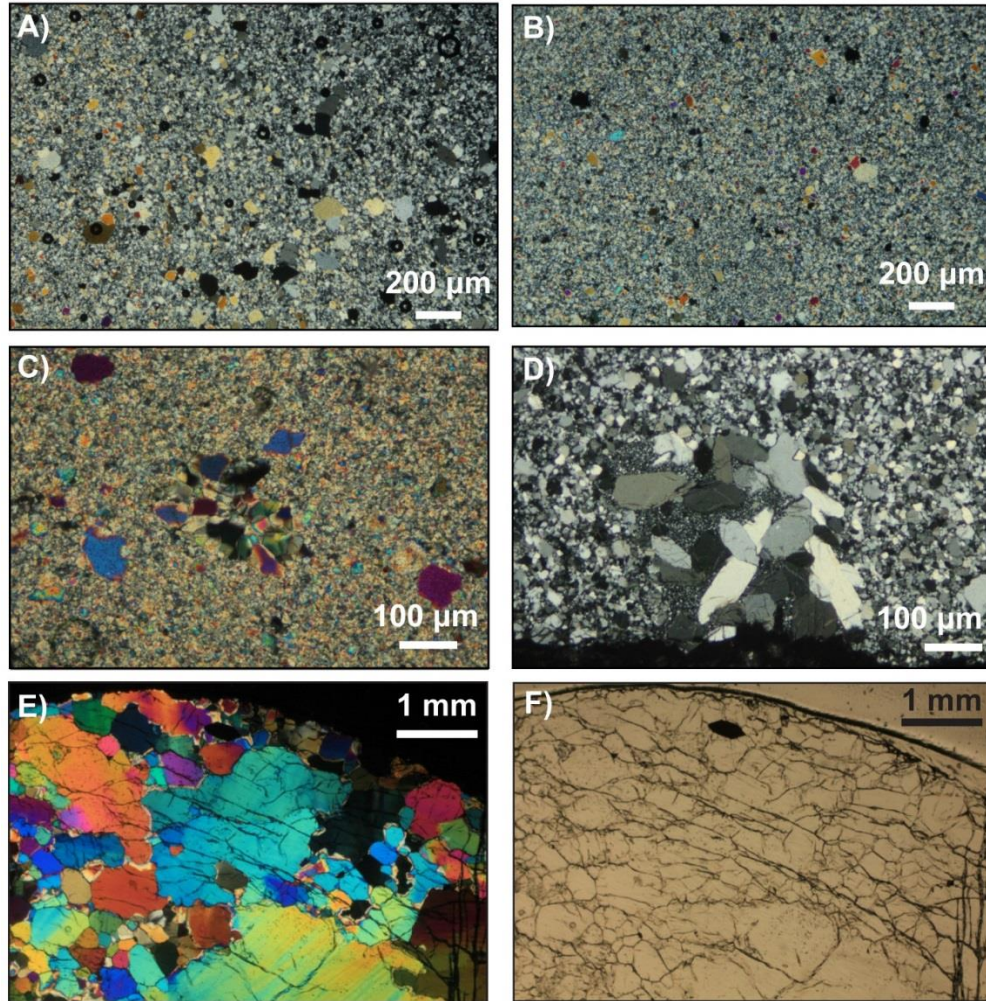


Figure 5. Images of wet (A) and dry (B) hot-pressed samples in cross polarized light (XPL) to show size, shape and distribution of grain sizes. Images in XPL of wet (C) and dry (D) hot-pressed samples to show clusters of larger grains that formed during hot pressing. Note, the groundmass between grains in (D), which is absent in the wet hot press (C). Image of of cored dunite in XPL (E) and PPL (F) to show grain size and distribution of olivine grains (E and F) and initial fracture distribution (F). All images are from radial thin sections.

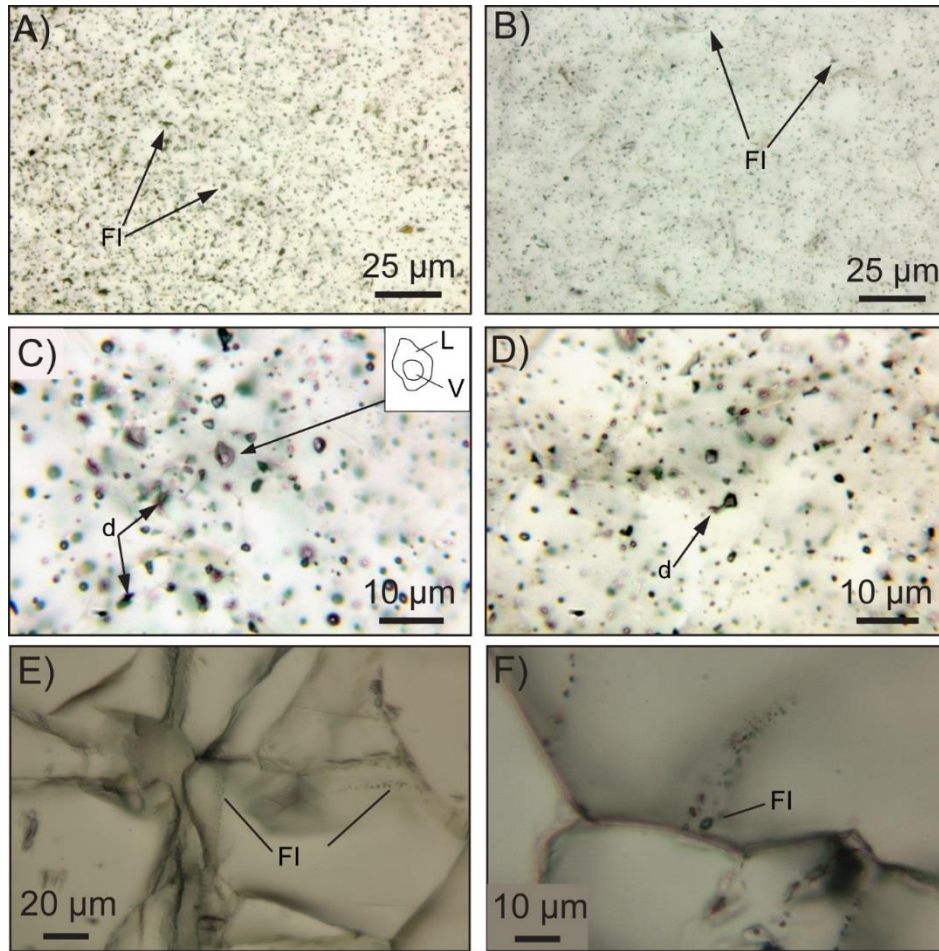


Figure 6. Images of wet (A) and dry (B) hot-pressed samples in plane polarized light (PPL) to show size, shape and distribution of FIs (indicated). Note the higher abundance of FIs in the wet sample (A). Photomicrographs in PPL of wet (C) and dry (D) hot-pressed samples to show the general size and morphology of FIs. Note, rare two phase FIs exist only in the wet hot-pressed samples (L-liquid, V-vapor). Examples of decrepitated FIs (d) are indicated in (C) and (D). Figs. 6C and 6D are from Carter et al., 2015, and Fig. 6C is modified. (E) Image of cored dunite in PPL to show rare FIs (indicated). Note the small trail of FIs does not cross grain boundaries. (F) Image of cored dunite in PPL to show small size of FIs. All images are of radial thin sections.

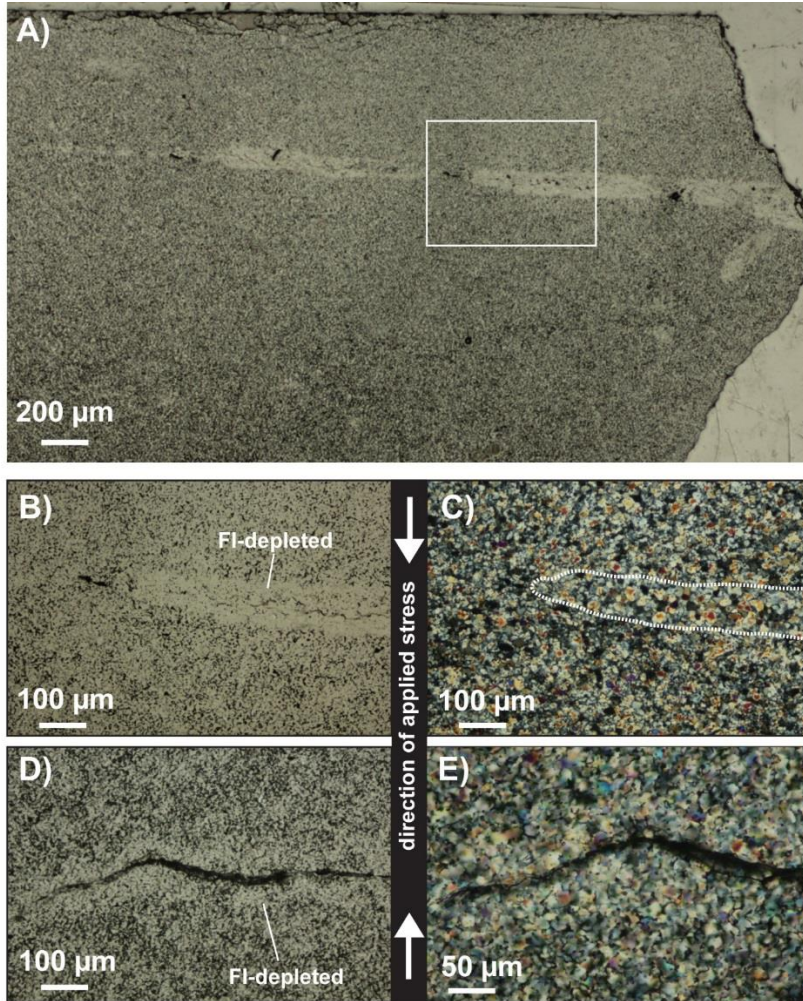


Figure 7. Images from an axial thin section from wet ol + tlc jacket (PI-1603). (A) PPL image shows overall FI-rich domains (dark) with sub-horizontal FI-depleted domains (light). White box indicates the approximate image location for (B). Images in PPL (B) and XPL (C) to show the nature of the FI-depleted (light) and FI-rich domains (dark). Note the sharp boundary between the two regions and the larger grain size in the FI-depleted area. (E) Image in PPL to show a FI-depleted zone along a crack in the sample. (F) Close up of image in (E) in XPL to show larger grain size in the zone adjacent to the crack. Note, the orientation of the axial stress direction is indicated (white arrows).

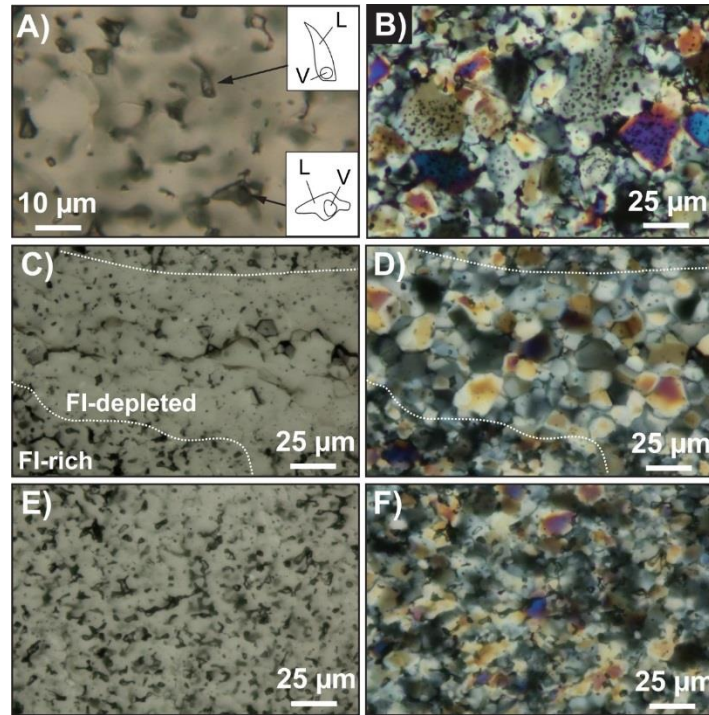


Figure 8. Images from an axial thin section from wet ol + tlc jacket (PI-1603). (A) PPL image to show the irregular morphology of FIs (indicated) observed throughout the sample. Note, arrows point to FIs that are sketched in insets to show rare two phase inclusions (L – liquid, V – vapor). (B) XPL image to show intragranular FI clusters that are present in larger olivine grains (diameters of $> 15 \mu\text{m}$). (C) Image in PPL of a FI-poor region surrounded by darker domains. Note, the morphology of FIs in the lighter region along grain boundaries. (D) Same image as (C) in XPL to show the variation in grain size. (E) PPL image of FI-rich domain. Note, the abundance of irregular FIs in this domain compared to (C) and (D). (F) Same image as (E) in XPL to show the variation in grain size and shape.

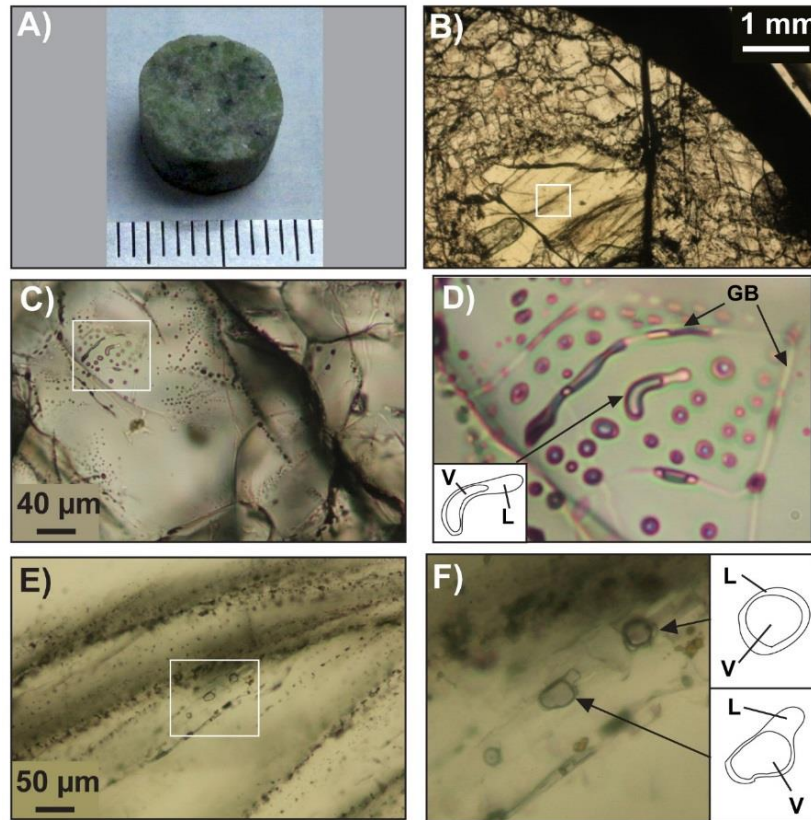


Figure 9. (A) Hand sample of dunite + tlc jacket (PI-1610) to show the lighter color compared with the starting material (Fig. 4B). Scale bar is in mm. (B) PPL image of dunite + tlc jacket (PI-1610) to show the reduction in grain size and increase in fracture abundance compare to the starting material (Figs. 5E and 5F). Box indicates photo area for (E). (C) PPL to show abundant intragranular and intergranular FIs in and between olivine grains, respectively. White box indicates the approximate location of (D). (D) Magnified image of (C) to show morphology and phases of FIs. Note, elongate FIs along grain boundaries (GB). Inset indicates the phases present in the FIs (L- liquid, V-vapor). (E) PPL image of a pyroxene grain to show abundant two-phase FIs along cleavage planes. White box is approximate location of (F). (F) Magnified image of (E) to show morphology and phases of FIs. Inset indicates the phases present in the FIs (L- liquid, V-vapor). Images (B) through (F) are from radial thin sections.

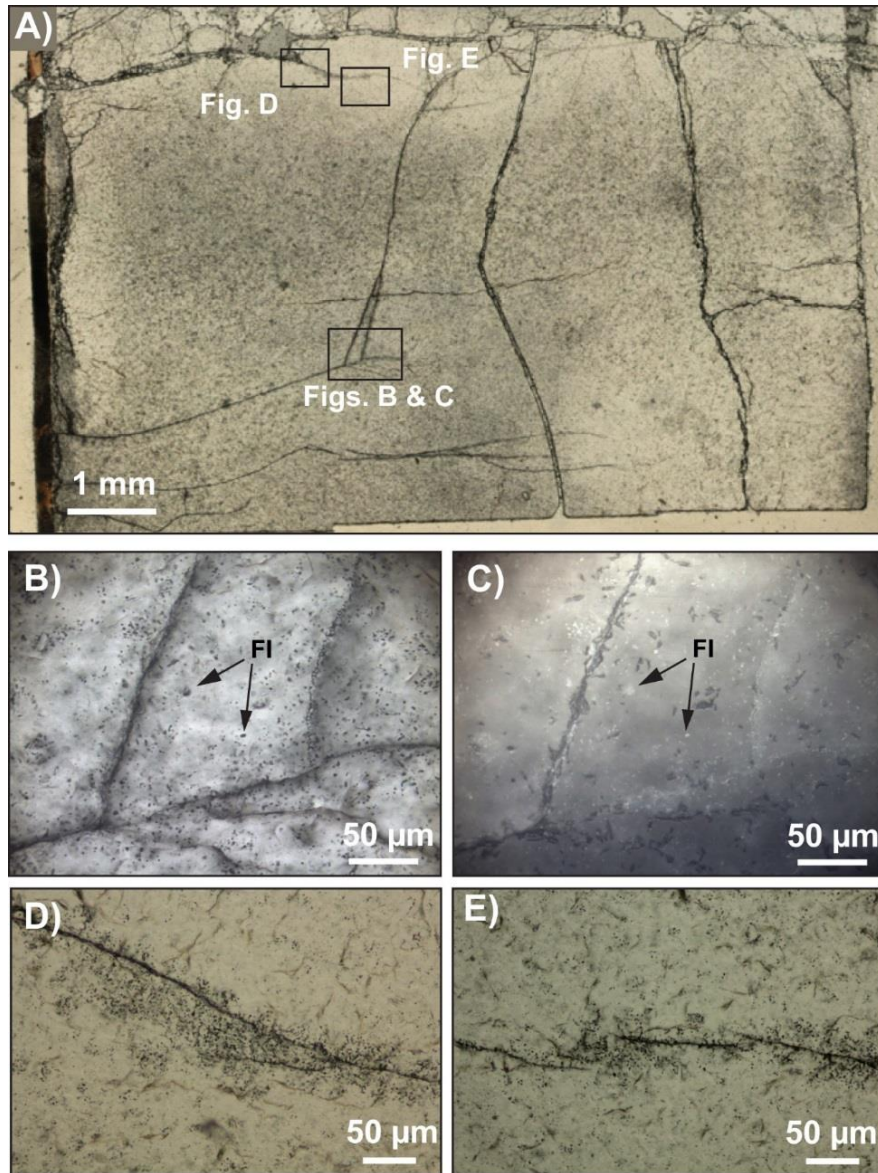


Figure 10. Images from an axial thin section from dry ol + tlc jacket sample (PI-1647). (A) Full axial thin section in PPL shows sub-vertical fractures crosscut sub-horizontal fractures. Boxes for (B) through (E) are noted. (B) PPL image shows abundant secondary FIs (indicated) along and adjacent to fractures. (C) Same image as (B) but in reflected light FIs appear white. (D) PPL image to show numerous secondary FIs adjacent to fractures. (E) PPL image to show FIs between en échelon fractures.

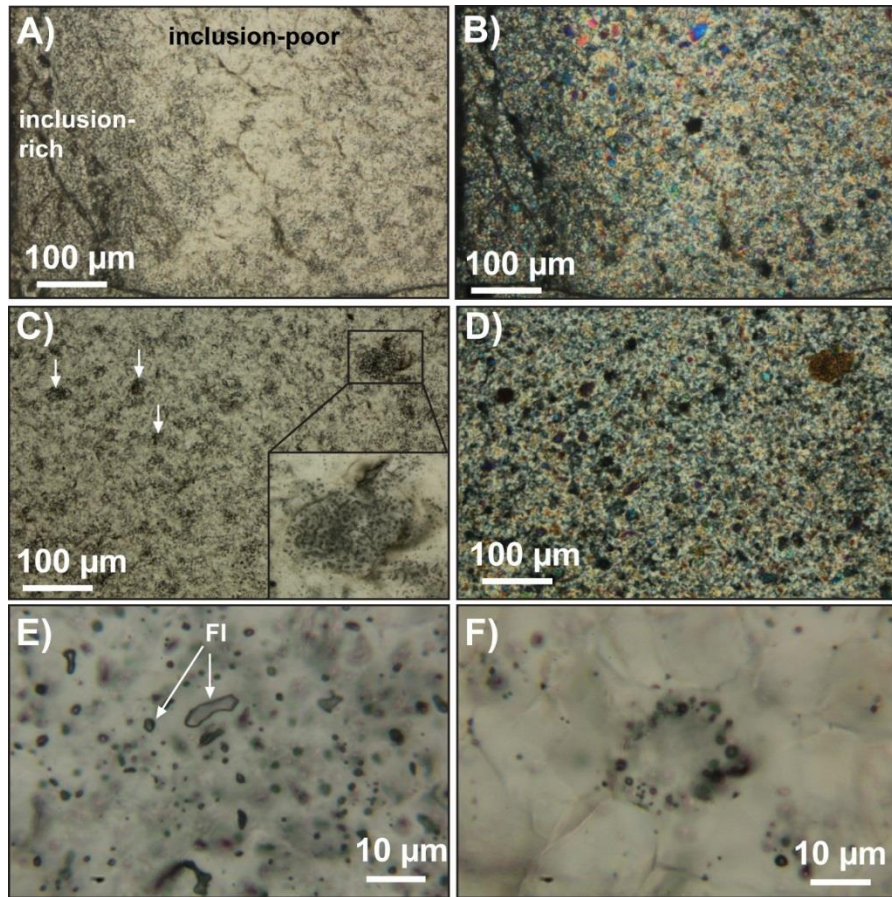


Figure 11. Images from an axial thin section from dry ol + tlc jacket sample (PI-1647). (A) PPL image to show FI-rich and FI-poor domains. (B) Same image as (A) in XPL to show grain size variation as a function of FI density. (C) PPL image to show dense FI clusters in larger grains (white arrows). Inset highlights one of the larger grains. (D) Same image as (C) in XPL to show grain sizes. (E) Image in PPL to show size and morphology of FIs (indicated). Note the lack of two phases in all FIs. (F) PPL image to show circular FIs along the grain boundaries of a circular grain.

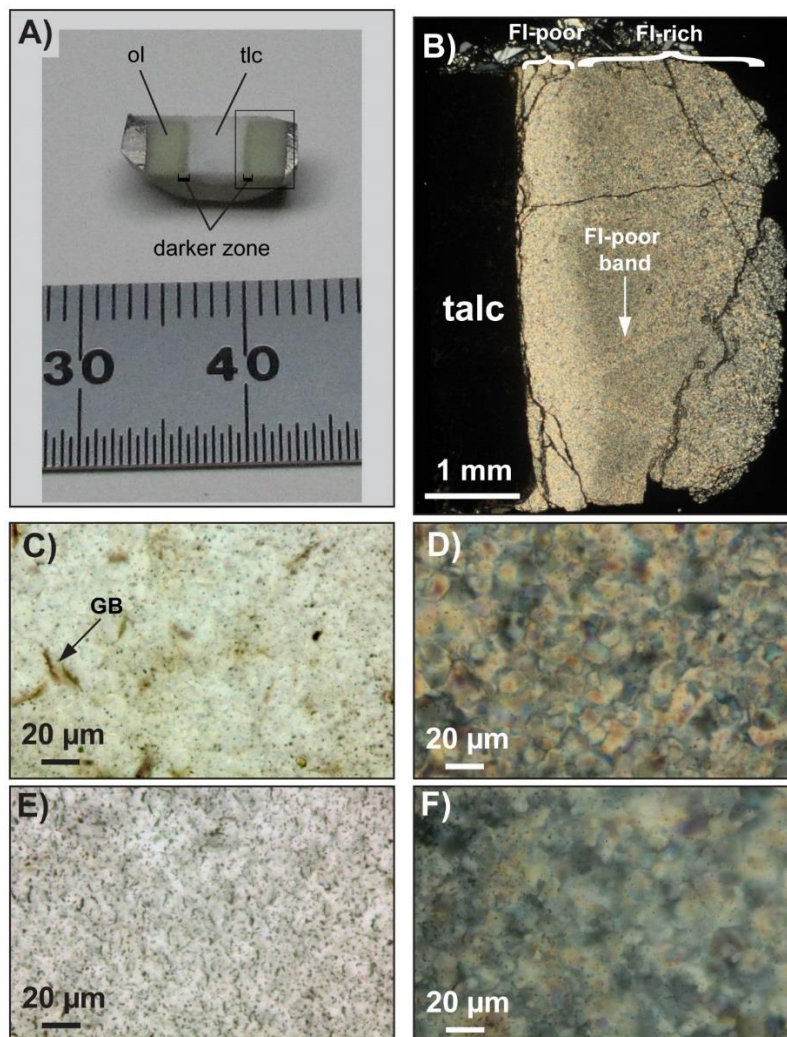


Figure 12. Images from the dry ol + tlc cylinder (PI-1748) sample. (A) A halved hand sample to show a dark zone in ol adjacent to tlc (indicated). The black box outlines the approximate area of (B). Scale is in mm. (B) XPL image of half an axial thin section to show a FI-poor zone adjacent to the tlc and FI-poor bands (indicated) that run from the FI-poor zone to the edges of the sample. Note the larger grain size in FI-poor zones. (C) PPL image shows FI density and morphology in an FI-poor domain. Note dark grain boundaries (GB; black arrow). (D) Same photo as in (C) in XPL to show grain size. (E) PPL image of a FI-rich zone to show FI morphology and density. Note the contrast to (C). (F) Same photo as (E) in XPL. Note the smaller grain size compared to (D).

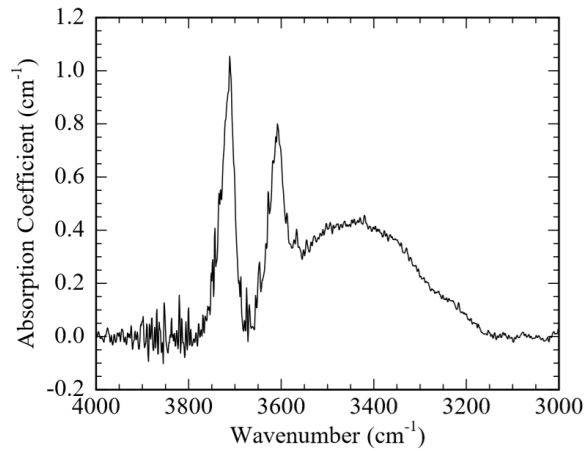


Figure 13: FTIR results from the dry ol + tlc cylinder experiment (PI-1748). Note the broad peak over 3600 to 3200 cm^{-1} is the OH in the FIs, whereas the peak at 3600 cm^{-1} is typical of OH in the olivine crystal lattice. The peak at 3712 cm^{-1} is likely related to F-H, and not shown are peaks relating to CO_2 .

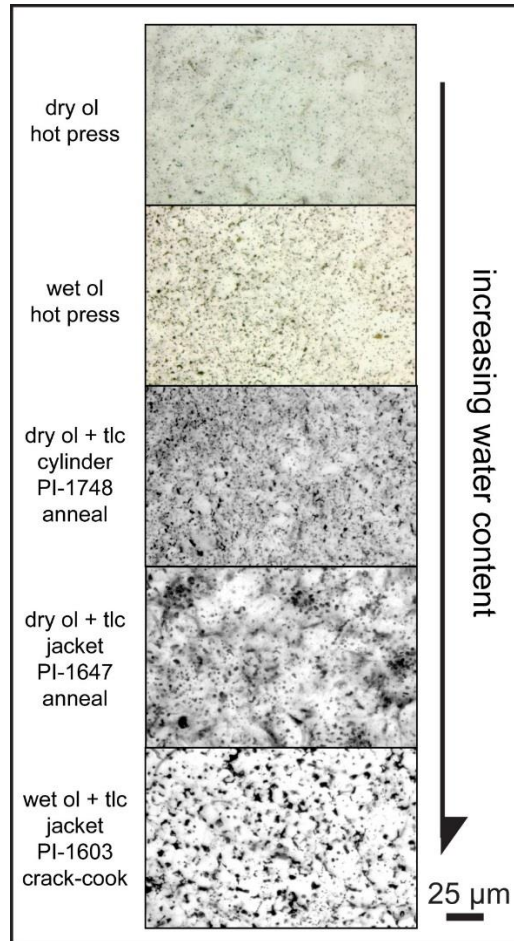


Figure 14. Images of representative areas of FIs (dark) and olivine (light) for both starting material and experimental samples (not including dunite) in PPL to show an increase in FI area percent with available water content. Note the increase in abundance and size of intergranular FIs (dark) at higher water contents. Scale is the same for all photos.

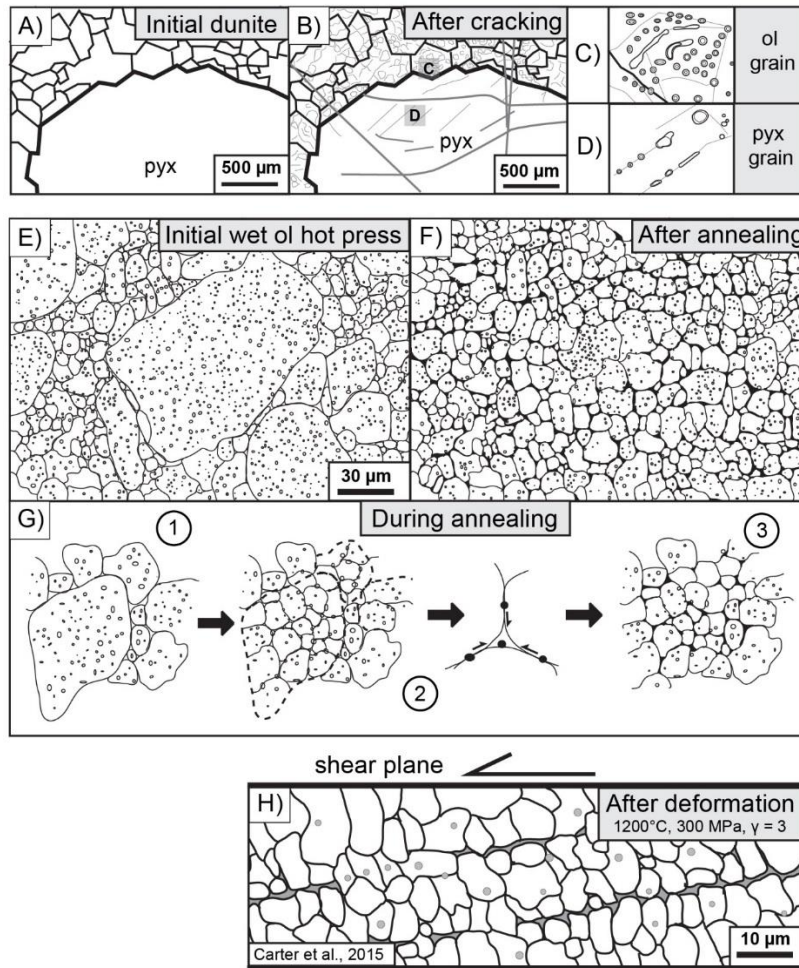


Figure 15. Schematic representation of (A) the initial dunite sample containing olivine and pyroxene (pyx), (B) the dunite sample after the crack-phase, note the light grey lines represent fractures (healed and unhealed) and areas for (C) and (D) are indicated with grey boxes, (C) olivine grain that contains abundant primary and secondary two-phase FIs (see also Fig. 9D), (D) the pyroxene grain contains two-phase FIs along cleavage planes, (E) grains and FIs of the initial wet ol hot press, (F) grains and FIs after the annealing phase, (G) the interpreted interaction of grain recrystallization and FI migration to grain boundaries and triple junctions (see Fig. 14F), note that intergranular FIs increase at the expense of intragranular FIs, but intragranular FIs are not eradicated, see text for the explanation to the numbers (1-3), (H) the result of deforming wet ol aggregates in shear. Note FIs are indicated in grey and are align into C'-bands. Shear direction and deformation conditions are indicated.