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Experimental Studies on Dehydration Embrittlement of
Serpentinized Peridotite and Effect of
Pressure on Creep of Olivine

A Dissertation submitted in partial satisfaction
of the requirements for the degree of

Doctor of Philosophy

in

Geological Sciences

by

Gang Xia

March 2013

Dissertation Committee:

Dr. Harry W. Green, II, Chairperson

Dr. Larissa Dobrzhinetskaya

Dr. David D. Oglesby

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The Dissertation of Gang Xia is approved:

Committee Chairperson

University of California, Riverside

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ABSTRACT OF THE DISSERTATION

Experimental Studies on Dehydration Embrittlement of
Serpentinized Peridotite and the Effect of
Pressure on Creep of Olivine

by

Gang Xia

Doctor of Philosophy, Graduate Program in Earth Sciences
University of California, Riverside, March 2013
Dr. Harry W. Green, II, Chairperson

The origin of intermediate depth earthquakes has been debated for 90 years yet is still under active discussion. These earthquakes are localized in double seismic zones in descending lithosphere; both zones originate very close to oceanic trenches. A leading proposed initiation mechanism for these earthquakes since 1968 has been dehydration embrittlement of serpentine under stress. Despite the considerable evidence favoring this mechanism, a major argument against it has been that the lower seismic zone initiates at ~40 km depth almost immediately below trenches and there does not appear to be a vehicle to carry water sufficiently deep to hydrate otherwise dry lithosphere. To directly address this problem, an experimental study has been carried out to investigate the minimum amount of serpentine that is required to trigger the dehydration embrittlement instability in serpentinized peridotite at high pressure (1-3 GPa) and temperature (720-750°C). The results show that embrittlement occurs during

dehydration of antigorite (the phase of serpentine stable at elevated pressure) in a wide range of compositions but both nearly dry peridotite and extensively altered peridotite are ductile. Fresh, unaltered, synthetic harzburgite and harzburgite with 4 vol% distributed antigorite are ductile, as are specimens with greater than 65% antigorite. Only compositions between 8 vol% and 65 vol% antigorite develop the instability. We suggest that very small degrees of serpentinization do not release sufficient H₂O to trigger the instability and that extensive serpentinization avoids the instability because soft, ductile, antigorite becomes the interconnected matrix with olivine and pyroxene existing only as isolated crystals. In that case, dehydration simply facilitates flow.

These systematics suggest that small amounts of H₂O transported down deep normal (bending) faults at trenches are sufficient to enable the instability in the lower seismic zones, thus providing additional support for dehydration embrittlement as the mechanism of intermediate-depth earthquakes. At the other end of the spectrum of serpentinization, these results are consistent with previous suggestions that extensive dehydration of altered subducting crust and mantle release copious amounts of H₂O that rise to the surface of the descending slab and react with the cool mantle of the overlying plate to lead to extensive serpentinization, thereby explaining serpentine diapirs in the forearc and lack of seismicity along the plate interface deeper than about 35km.

Another long-lived controversy in mantle geophysics involves the pressure dependence of creep in olivine, the most abundant and softest phase in unaltered mantle rocks. The pressure dependence of any thermodynamically-controlled phenomenon is commonly expressed as the activation volume, ΔV^* . Previous experimental investigations on the effect of pressure on creep in olivine have produced bimodal results. ΔV^* obtained from solid-medium (Griggs) and gas-medium (Paterson) deformation apparatus at relatively lower pressures is $\sim 15 \text{ cm}^3/\text{mol}$ or higher. In contrast, higher-pressure studies using multianvil apparatus at sites of synchrotron X-radiation (D-DIA apparatus) report a ΔV^* near zero. To decipher this enigma and to provide a much-needed calibration of stress in the D-DIA apparatus, I have conducted systematic experiments on a synthetic, iron-free, forsterite at 1200°C and pressure between 1 and 2.5 GPa using the UCR 5 GPa modified Griggs apparatus, the only apparatus capable of performing these experiments. Our results show a robust ΔV^* value of $12 \pm 2 \text{ cm}^3/\text{mol}$, indicating a fairly significant pressure dependence of creep in olivine to pressures of $\sim 3\text{GPa}$ (approximately 100 km). In collaboration with other experimentalists, we plan to measure the ΔV^* for creep of this material over a pressure range of 2-8GPa in the D-DIA apparatus to both calibrate stress measurement in the D-DIA and resolve the question of change in ΔV^* at higher pressures.

Table of Contents

<u>LIST OF FIGURES</u>	XI
<u>LIST OF TABLES</u>	XIII
<u>CHAPTER 1</u>	
GENERAL INTRODUCTION	1
1.1 STRUCTURE OF THE DISSERTATION	2
1.2 GOAL OF THE DISSERTATION	4
1.3 GEOPHYSICAL SIGNIFICANCE OF SERPENTINE IN SUBDUCTION SYSTEMS	5
1.4 GEOLOGICAL IMPORTANCE OF OLIVINE IN UPPER MANTLE	9
1.5 METHOD OF INVESTIGATION	14
<u>CHAPTER 2</u>	
COMPOSITIONAL DEPENDENCE OF DEHYDRATION EMBRITTLEMENT IN SERPENTINIZED PERIDOTITE	26
2.1 ABSTRACT	27
2.2 INTRODUCTION	29
2.3 STARTING MATERIALS AND METHODS	39
2.4 RESULTS AND DISCUSSION	45
2.5 CONCLUSION	68
2.3 GEOPHYSICAL IMPLICATIONS	69
2.3 FUTURE PERSPECTIVES	71
<u>CHAPTER 3</u>	
EFFECT OF PRESSURE ON CREEP OF OLIVINE	72
3.1 ABSTRACT	73
3.2 INTRODUCTION	74
3.3 MOTIVATION OF THIS STUDY	78
3.4 STARTING MATERIALS AND EXPERIMENTAL DESCRIPTION	81
3.5 RESULTS AND DISCUSSION	84
3.6 CONCLUSION	98
3.7 FUTURE PERSPECTIVES	101

CHAPTER 4

CONCLUSIONS AND FUTURE WORK	103
4.1 SERPENTINE DEHYDRATION EMBRITTLEMENT	104
4.2 Δv^* IN CREEP OF OLIVINE	106
<u>REFERENCES</u>	109

List of Figures

	Page
Fig. 1.1 Photograph of Rig-2 Griggs apparatus	16
Fig. 1.2 Photograph of Rig-3 Griggs apparatus	17
Fig. 1.3 Schematic drawing of Rig-3 Griggs apparatus	18
Fig. 1.4 Schematic drawing of high pressure sample assembly for Griggs apparatus	22
Fig. 1.5 Photograph of Scanning electron microscope (SEM)	24
Fig. 1.6 Photograph of Transmission electron microscope (TEM)	25
Fig. 2.1 Globally averaged number of earthquakes per year as a function of depth (after Frohlich, 1989)	30
Fig. 2.2 Double seismic zone beneath northeast Japan (Peacock, 2001)	34
Fig. 2.3 Phase diagram for antigorite dehydration reactions (after Ulmer and Trommsdorff, 1995)	38
Fig. 2.4 SEM(BSE) Images of Starting materials	40
Fig. 2.5 Two types of specimen configurations	41
Fig. 2.6 Stress versus strain plot	47
Fig. 2.7 SEM (BSE) images of GL844	50
Fig. 2.8 Continued SEM (BSE) images of GL844	51
Fig. 2.9 SEM (BSE) images of GL840	52

Fig. 2.10 Continued SEM (BSE) images of GL840	53
Fig. 2.11 SEM (BSE) image of GL870	55
Fig. 2.12 TEM foil cut through focused ion beam (FIB)	56
Fig. 2.13 High resolution images of fault zone contents	58
Fig. 2.14 SEM (BSE) images of faulting in GB476	60
Fig. 2.15 SEM (BSE) images of GB513	61
Fig. 2.16 SEM (BSE) images of microcracks and fluid inclusion (bubble) trails in dehydrated material	63
Fig. 2.17 SEM (BSE) images of ductile harzburgite specimens	64
Fig. 2.18 SEM (BSE) image of GL495	65
Fig. 2.19 SEM (BSE) images of faulted experiment GB493	67
Fig. 3.1 Initial Stress/Ln stress versus pressure plots	86
Fig. 3.2 Stress drop before getting to steady state	87
Fig. 3.3 SEM images of high angle and cataclastic zones in specimens cracks	88
Fig. 3.4 Typical differential stress versus displacements curves	92
Fig. 3.5 Stress versus strain plot at a strain rate of $10^{-4}/s$	93
Fig. 3.6 Stress/Ln stress versus pressure plot at a strain rate of $10^{-4}/s$	94
Fig. 3.8 Stress versus strain plot of experiments at a strain rate of $10^{-5}/s$	96
Fig. 3.9 Stress/Ln Stress versus pressure plot at a strain rate of $10^{-5}/s$	97

List of Tables

	Page
Table 2.1 Composition of Starting materials used in experiments	42
Table 2.2 Experimental conditions and mechanical results	46
Table 3.1 Selected results from previous studies	79
Table 3.2 A summary of experimental condition and mechanical results	84

Chapter 1.
General Introduction

1.1 Structure of this dissertation

In this dissertation, I present the results from my experimental studies on two major projects:

- (i) Compositional dependence of dehydration embrittlement in serpentinized Peridotite;
- (ii) Pressure dependence of creep in olivine and its importance for measurement of upper mantle rheology.

In chapter 1, I present the geophysical settings and significances of olivine as well as serpentine, the two major minerals used in my studies. I also present the method of experimental investigation, including the description of apparatus, sample assembly and analytical techniques.

Chapter 2 provides a detailed description of the quantitative experimental study of dehydration embrittlement in serpentinized peridotite. I present an experimental investigation that covers a wide range of serpentinization, ranging from 4 vol% antigorite to ~75 vol% antigorite. Using two different starting material configurations, I show that at 1GPa, the serpentine dehydration-induced faulting instability is encountered between 8 vol% antigorite and 75 vol% antigorite. At 4 vol%, there is too little water being released to induce instability; at the other end, when serpentine, the weaker phase, is

the dominant mineral and becomes the interconnected framework in the whole specimen, the sample deforms plastically and faulting instability is absent.

Chapter 3 of this dissertation focuses on the measurement of activation volume of olivine, ΔV^* , which reflects the effect of pressure on the creep in olivine. I show that there is a great discrepancy between the ΔV^* results measured from low pressure (~ 2 GPa) to high pressure (> 5 GPa). Examination of experimental details suggests that the results from high pressure studies may very likely suffer from their small grain-size and/or inaccurate stress derivation. Small grain-size, less than $\sim 10 \mu\text{m}$, will favor grain boundary sliding rather than generation and movement of dislocations, and inaccurate stress derivation may lead to an erroneous ΔV^* value. Using the 5 GPa modified Griggs deformation apparatus with the molten salt assembly, we have obtained a robust and constant ΔV^* value of $12 \pm 2 \text{ cm}^3/\text{mol}$ between 0.4 GPa and 3 GPa for iron-free forsterite in the dislocation creep field. Although beyond the scope of this dissertation due to technical barriers, our overall intention is to use the same material to measure the ΔV^* for creep of olivine in additional experiments at higher pressures in the D-DIA apparatus using *in situ* experiments at synchrotron X-ray sources. Arrangements are in place to complete these experiments in collaboration with others. The result should enable resolution of the disagreements about ΔV^* of creep and simultaneously will provide an

important calibration standard for a wide variety of studies at elevated pressure using the D-DIA apparatus.

In chapter 4, I summarize the relevant works presented as well as the potential directions that experimentalists may take to move this subject forward in the future.

1.2 Goal of this dissertation

Two active debates in the scientific community relevant to the rheology of the upper mantle of Earth are:

- (1) What are the fundamental physical processes underlying dehydration embrittlement of serpentized peridotite and is this the mechanism of intermediate-focus earthquakes?
- (2) What is the pressure dependence of creep in olivine and does it change with pressure?

My experimental studies are designed to place quantitative constraints on these debates as follows:

- (i) Determine the minimum amount of serpentine required to produce faulting during its dehydration and the implications of that result for subduction zone Earthquakes.

- (ii) Measure the ΔV^* value for creep in iron-free forsterite below 3 GPa and provide a calibration standard for stress measurement in the D-DIA apparatus.
- (iii) To test how much serpentine, at maximum, can still trigger the instability.

The first two goals were designed at the inception of my PhD study; the last one was picked up adventitiously during my work on the first goal.

1.3 Geophysical Significance of Serpentine in Subduction Systems

Serpentinite, the official state rock of California, forms at the expense of hydration of peridotite which is the major constituent of Earth's upper mantle. Serpentinite is considered to be of salient importance for a variety of tectonic phenomena, including intermediate-depth earthquakes (e.g. Peacock, 2001; Dobson et al., 2002; Hacker et al., 2003; Jung et al., 2004), serpentinization of the forearc mantle wedge (e.g. Hyndman and Peacock, 2003; Rüpke et al., 2004), water recycling into the deep mantle (e.g. Thompson, 1992; Kawakatsu and Wadati, 2007) or not (Green et al., 2010; Kawakatsu and Yoshioka, 2011) and seismic anisotropy in the subduction systems (e.g. Long and Silver, 2008; Manuele et al., 2008; Katayama et al., 2009).

Serpentine has been under active study for over half a century since Gutenberg and Richter (1954) defined the intermediate-depth earthquakes as those occurred between ~ 70 to ~ 300 km depth range. Earthquakes in this depth range have been noted to define

two planes of seismicity in many subduction zones. Brudzinski et al. (2007) showed that all subduction zones show such double seismic zones and that the separation can vary between 15-40 km, depending on the age and rate of the subducting plate. Seismic studies have shown a strong correlation between seismicity and corresponding hydrous phases (e.g. Zhang H et al. 2004). Upper plane seismicity at shallow depths is associated with dehydration of altered oceanic crust whereas the lower plane seismicity, lying within the subducting mantle lithosphere, spatially corresponds to the location where dehydration of antigorite is to be expected (Peacock, 2001; Hacker et al., 2003; Yamasaki and Seno, 2003; Jung et al., 2004). Although another mechanism, shear heating instability, has been proposed to explain intermediate-depth earthquakes, dehydration embrittlement of serpentinite, regarding both geophysical and experimental evidence, remains the most viable mechanism for earthquakes at intermediate depth. The biggest concern currently is the level of hydration in the subducting mantle lithosphere. Seismic reflection study has shown that sea water can percolate along normal faults, which formed at the outer rise regions, to ~20 km depth (Ranero et al., 2003), but how and/or how much water can penetrate down over 40 km deep to hydrate the mantle where the lower plane of seismicity initiates is still enigmatic and awaits deciphering.

Serpentinization of the forearc mantle wedge, resulting from hydrothermal alteration of peridotite, was first proposed by Fyfe and McBirney (1975). As the backarc

is usually too hot ($> 800\text{ }^{\circ}\text{C}$) for hydrous phases to be stable, the forearc mantle, adjacent to the subducting oceanic crust, is a cool location ($400 \sim 600\text{ }^{\circ}\text{C}$) where hydrous phases, e.g. serpentine, can be stable (Bostock et al., 2002; Hyndman and Peacock, 2003). Seismic tomographic studies showed low velocity and high Poisson's ratio regions in the forearc mantle (Kamiya and Kobayashi, 2000; Bostock et al., 2002; DeShon, 2004; Seno, 2005), indicating extensive serpentinization of forearc mantle (serpentine has unusual low seismic wave velocity -- $V_p=5.5\text{ km/s}$, $V_s=2.6\text{ km/s}$ -- and remarkable high Poisson's ratio, 0.5 compared with 0.25 for ambient peridotite) (Christenson, 1996). Commonly observed serpentinite diapirs in the forearc provide strong evidence that the forearc mantle is partially serpentinized (Fryer, 1995). The aseismic forearc mantle may be explained by serpentinization since serpentinite is considerably weaker than dry peridotite (Hyndman and Peacock, 2003), and stable sliding of serpentinite may impede stress buildup and control the downdip limit of thrust earthquakes in subduction zones (Tichelaar et al., 1993; Hyndman et al., 1997; Oleskevich et al., 1999; Peacock and Hyndman, 1999; Seno, 2005; Hilairet et al., 2007). The serpentinized forearc mantle wedge can be dragged downward by the oceanic plate, leading to subsequent dehydration that may cause partial melting of mantle and eventually lead to arc magmatism (Davis and Stevenson, 1992; Hacker et al., 2003), Geochemical study has shown that arc lava components originate from the subducted slab (Elliot et al., 1997).

The deformation of serpentine has also been used to explain the trench-parallel seismic anisotropy in the hydrated mantle wedge (Katayama et al., 2009).

It is commonly believed that significant amounts of water are recycled into the Earth's deep interior through the subduction system. Water can be bound within hydrous phases and released through metamorphic dehydration reactions at elevated temperature and pressure. Serpentine, $\sim\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$, is the most abundant hydrous mineral in altered ultramafic rocks because the ratio of Mg/Si in serpentine is 1.5/1, sitting between that of olivine (2/1), and orthopyroxene (1/1) (Hydnman and Peacock, 2003). Serpentinite can store up to ~ 13 wt% water, which makes it the most important water carrier in hydrated mantle rock (Ulmer and Trommsdorff, 1995). Antigorite is the high pressure serpentine polymorph; its stability limit rises to 700°C at 2.2 GPa, then falls steadily to 600°C by 6 GPa. Seismic studies have shown a nearly one-to-one correlation between dehydration reactions and seismicity to a depth of around 250 km (Zhang et al., 2004; Green et al., 2010). Although clinohumite (Stalder and Ulmer, 2001) and dense hydrous magnesium silicates (DHMS) (Angel, 2001) are capable of carrying water further down, the correlation between dehydration reactions and seismicities is not observed at greater depth, suggesting dry slabs below ~ 400 km. Besides, metastable olivine, which is incompatible with the presence of H_2O (Diedrich et al., 2009), has now been reported from 5 subducting slabs (Iidaka and Suetsugu, 1994; Chen and Brudzinski,

2001; Kaneshima et al., 2007; Jiang et al., 2008; Kawakatsu & Yoshioka, 2011) requiring that only very small amounts of water could be transported below ~400 km in subducting slabs. If the mantle transition zone and lower mantle are not essentially dry (Green et al., 2010; Kawakatsu & Yoshioka, 2011), there must be another pathway.

1.4 Geological Importance of Olivine in Upper Mantle

Olivine is the magnesium/iron orthosilicate with the chemical formula $(\text{Mg, Fe})_2\text{SiO}_4$. Olivine has a complete solid solution between the two end members: forsterite Mg_2SiO_4 and fayalite Fe_2SiO_4 . Olivine is stable from the Earth's surface to the bottom of the upper mantle, corresponding to ~410 km where its structure becomes unstable and transforms to its higher pressure polymorph: wadsleyite.

Olivine dominates the composition of upper mantle, consequently, it controls the mantle rheology down to 410 km. One of the most intrinsic problems that is still under active investigation is the pressure dependence of the creep of olivine, represented by ΔV^* . The earlier low-pressure studies (less than 2.5 GPa) produced a ΔV^* around 15 cm^3/mol , suggesting a fairly large pressure effect of the creep of olivine (e.g., Bai and Green, 1998; Borch and Green, 1987; Green and Borch, 1987; Karato and Jung, 2003). With the development of technology, more results were reported from experimental studies using higher pressure limit apparatus, e.g., Deformation-DIA (e.g., Li et al., 2004;

Li et al., 2006; Raterron et al., 2007), these studies produced a ΔV^* near zero. However, it is not immediately obvious that the newer high pressure results are better than the older ones. Contrarily, it is quite possible that the newer ΔV^* derived from stress using synchrotron radiation is inaccurate in that the stress one derives from X-ray diffraction rings is different from each ring (reflecting the fact that different rings sample different populations of grains rather than the macro stress supported by the whole specimen). How one inverts such data to arrive at the macroscopic stress state necessary for comparison with conventional apparatus that measure stress with a load cell is controversial. There is currently no experimental "bridge" linking the measurement of ΔV^* from both low and high pressure studies. The results from chapter 2 in this dissertation provide a robust ΔV^* below 3 GPa and completion of our future study using the D-DIA on this same material should provide not only the resolution of the ΔV^* controversy but also the needed calibration standard for future studies using high pressure apparatus.

As the most abundant phase in the Earth's upper mantle, olivine also plays a key role in a wide range of dynamic processes, including upper mantle rheology, evolution of subduction zones (e.g., van Keken et al., 2002, Gerya and Stöckhert, 2002), Earth's deep seismicity (e.g., Green and Burnley, 1989; Green et al., 1990, Green and Houston, 1995; Green, 2007), upper mantle seismic anisotropy (e.g., Kneller et al., 2005 & 2007,

Mainprice et al, 2005; Jung et al., 2008), post-seismic mantle relaxation (e.g., Freed and Bürgmann) and attenuation of upper mantle seismic anisotropy (e.g., Hammond and Humphreys, 2000; Jackson et al., 2002). Among them, I would like to summarize some previous work on seismic anisotropy induced from olivine's lattice preferred orientation (LPO) because of its intrinsic relevance to this dissertation and its future perspectives, since recent studies have shown a potential correlation between transition of olivine's LPO and the discrepancy of ΔV^* values in olivine.

In addition to its well-known application in the scientific study of earthquakes, seismology is also capable of providing information about mantle deformation through the analysis of seismic anisotropy (Silver, 1996). Seismic anisotropy is a term used to describe that in anisotropic rocks, seismic waves exhibit birefringence, much like propagation of light through crystals; in such rocks seismic energy traveling in a particular direction is partitioned into two waves polarized perpendicular to each other and traveling at different speeds. Moreover, they travel at different velocities as a function of direction within the anisotropic materials. Seismic wave velocities depend on the elastic properties of materials; they are functions of temperature, pressure, composition and stress. Seismic anisotropy is mainly caused by the lattice preferred orientation (LPO) that arises during deformation of anisotropic minerals by dislocation creep (e.g. Karato, 2008). Therefore, one can determine the LPO of a mineral by

experimental deformation or, if one knows the dislocation mechanisms in a material one can calculate it. It follows that the relation between LPO and deformation conditions can be used to describe the mechanical behavior of upper mantle minerals and a variety of other geophysical and engineering problems. Experimentalists began to study the relationship between LPO of minerals and deformation conditions in the 1960s (e.g. Green et al., 1970); for olivine the major early papers were Carter and Ave Lallemant (1970), Nicolas et al. (1973) and Bouiller & Nicolas, (1973). Solid-medium Griggs apparatus were used in all of these studies except the last which addresses natural deformations in mantle xenoliths from kimberlite. Deformation-induced microstructures observed in the optical microscope level resolved that the (010) plane is perpendicular to the maximum shortening direction and that the [100] axis is parallel to the slip direction (Raleigh, 1968). Subsequent analysis using transmission electron microscopy (TEM) confirmed this mechanism as the dominant slip system of [100]/(010) (Green and Radcliffe, 1972), later referred to as the A-type LPO by Jung and Karato (2001). The latter authors extended the study of LPO in olivine and found that, depending on the water content, several other LPOs can develop.

More relevant to my study, the effect of pressure on olivine's LPO could also be significant. Although it was proposed that the pressure influence is only through its effects on water fugacity and homologous temperature, with no apparent effect of

pressure on olivine's LPO (Karato et al., 2008), evidence has accumulated showing that the reality may be more complex. Couvey et al. (2004) conducted simple shear experiments on olivine at 11 GPa and 1673 K, and reported a dominant slip system similar to the C-type LPO, $[001]/(100)$. They suggested that this transition is the result of the effect of pressure rather than H_2O as predicted by Jung and Karato (2001). However, post-experiment examination of their samples showed substantial amount of water (~ 2000 ppm H/Si), complicating the interpretation. Similar results were summarized by Mainprice et al. (2005), in which they also suggested that the attenuation of seismic anisotropy below ~ 250 km is induced from the transition of olivine's LPO from A-type to $[001]/(hk0)$, corresponding to B or C-type. Ratteron et al. (2007) carried out deformation experiments on "dry" forsterite single crystals under upper mantle conditions, their results showed that the dominant slip system changed from A-type to B-type LPO, $[001]/(010)$ around 5 GPa, although their results were at rather high stresses which also could be responsible for this transition. Results from our laboratory on dry harzburgite Jung et al (2009) were conducted at stresses equal to or lower than the wet experiments of Jung and Karato (2001), yet at 3 GPa showed the same LPO transition pattern as Ratteron et al. (2007). More recent experimental studies suggested that there is a potential link between the transition of olivine's LPO and the discrepancy from measurement of ΔV^* values that reflect the effect of pressure on creep

in olivine (e.g. Raterron et al., 2011). They reported A-type LPO below 5.7 GPa associated with a ΔV^* of $15 \pm 3 \text{ cm}^3/\text{mol}$, while above 5.7 GPa the B-type LPO appeared and resulted in a ΔV^* of $0 \pm 1.2 \text{ cm}^3/\text{mol}$.

1.5 Method of Investigation

1.5.1 Griggs Deformation Apparatus

The Griggs apparatus is a piston cylinder high-pressure machine modified by inserting a movable piston down the center of the standard piston. The central piston is then driven by a motor through a gear train or a hydraulic drive into the high pressure volume to deform a cylindrical specimen that stands inside a graphite furnace. There are two Griggs apparatus in the Tectonophysics Laboratory at University of California, Riverside, “Rig-2” which is a standard Tullis-modified Griggs machine capable of ~ 1.8 GPa confining pressure (Figure 1.1) and “Rig-3”, a Green-modified 4-post machine capable of 5 GPa confining pressure (Figure 1.2). Rig-2 is utilized for lower-pressure experiments while all experiments above 1.8 GPa are accomplished in Rig-3. The different pressure limits are the result of the different designs; the 4-post machine can accommodate a larger pressure vessel. For stress control, Rig-2 (Figure 1.1) is equipped with a stepping motor and gear train and Rig 3 (Figure 1.2) with a servo-controlled hydraulic drive. Both are programmed and operated through a computer that also records confining pressure,

axial stress, axial piston displacement and two temperature measurements in each experiment. For both machines the confining pressure is calculated from the oil pressure behind the confining pressure ram measured by a pressure transducer (Precise Sensors Model 6550). Stress is calculated from the total load on the drive piston measured by a load cell (Houston Scientific Model 3500) that measures the axial force applied to the sample (Figure 1.3). Furnace power (1000 - 1600 W) is supplied by a 3 KVA AC power transformer (115:8) through a silicon-controlled rectifier (SCR), and the temperature is measured by two disposable Type-B thermocouples ($\text{Pt}_{94}\text{Rh}_6$ - $\text{Pt}_{70}\text{Rh}_{30}$) in each experiment, one of which is controlled by a Eurotherm temperature controller (Model 818P). Displacement, from which strain and strain rate are calculated, is measured by a linear voltage displacement transducer (Schaevitz Model DC-E500). From these data, confining pressure can be measured to within 10 MPa, temperature to within 1 K and displacement to 0.3 μm . Stress measurement depends on many things. The equipment is capable of recording differences of 1 MPa but in practice generally ± 10 MPa is attainable. With the molten salt cell in place, stresses can be measured to a precision of less than 10 ± 2 MPa.



Figure 1.1 Photograph of Rig-2 Griggs apparatus in the Tectonophysics Laboratory at University of California, Riverside.

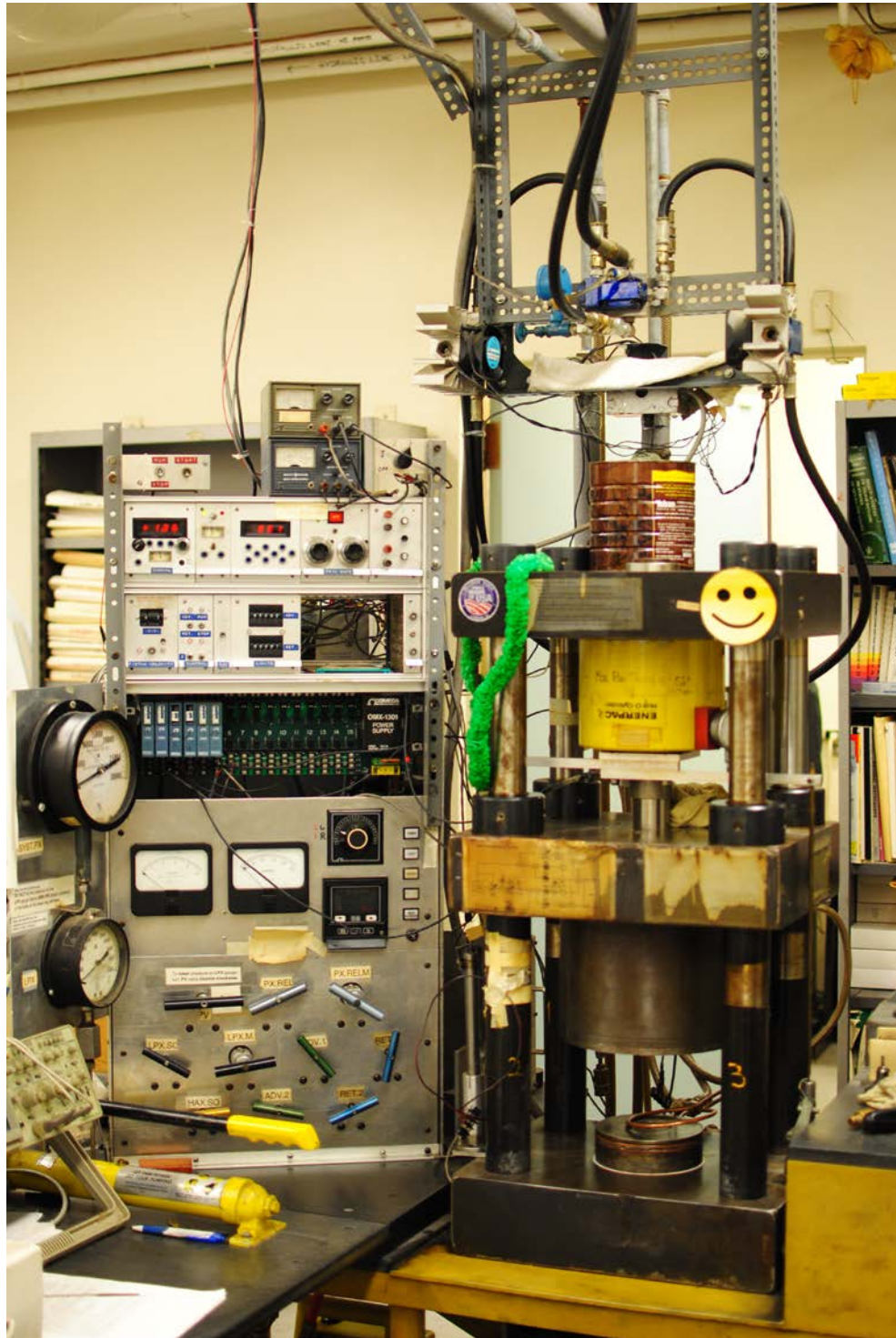


Figure 1.2 Photograph of Rig-3 Griggs apparatus in the Tectonophysics Laboratory at University of California, Riverside.

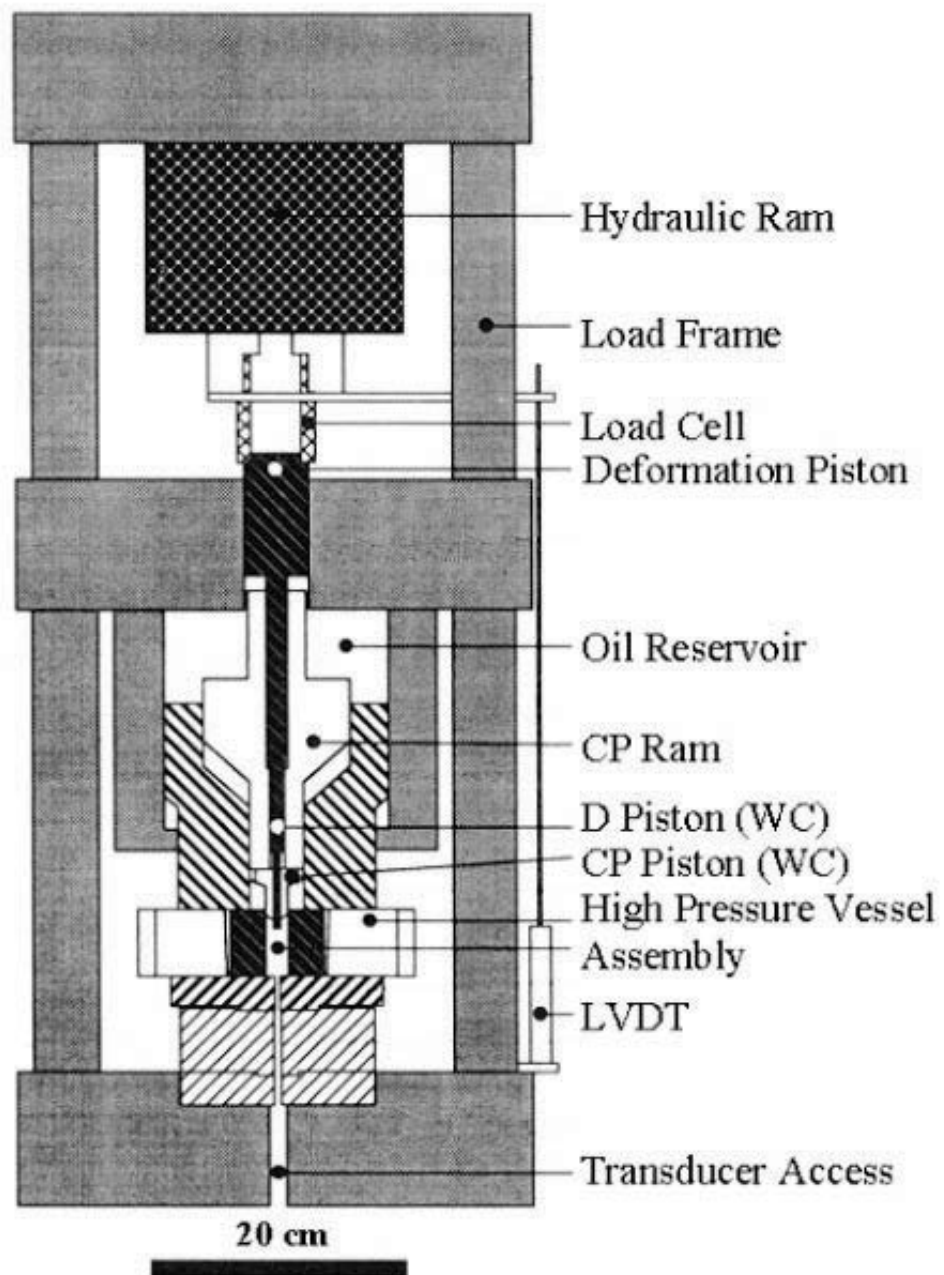


Figure 1.3 Schematic drawing of Rig-3 Griggs apparatus.

To prevent overheating, recirculated cooling water at 23 °C is used to cool the apparatus and room-temperature mineral oil is used to cool the pressure vessel. Data acquisition is programmed by FORTRAN language, the output data is then used to calculate the stress and strain using both Kaleidagraph and Origin software. For each machine, real-time output of all measured parameters is recorded on a strip-chart recorder to monitor experiment progress and to make decisions during the experiment if necessary.

1.5.2 Sample assembly

The sample assembly (Figure 1.4) is designed to (i) minimize stresses on the various parts during pressurization and, especially, during experiments; (ii) minimize temperature gradients within the sample and furnace lifetime; (iii) provide as weak a confining medium as possible; (iv) minimize friction on internal pistons and the sample itself. It is designed for experimental conditions up to 5 GPa and 1400 °C. The schematic cross section of Fig. 1.4 shows: From top to bottom, (i) the lower end of the tungsten carbide (WC) deformation piston that sticks up out of the pressure vessel, (ii) two packing rings on top of a lead cylinder that ensures that all parts of the assembly are stressed equally during pressurization. The outer packing ring forms a static seal during experiments to maintain constant confining pressure; the inner one forms a sliding seal around the deformation piston that progressively enters the high-pressure volume during an experiment. (iii) A graphite cap that grounds the graphite furnace to the wall

of the pressure vessel. The small gaps shown between the graphite cap and the tops of the furnace and the inner soft-fired pyrophyllite sleeve are there to protect those parts as pressurization begins; they close as the assembly compacts during pressurization. (iv) The graphite furnace is tapered at the bottom to generate extra heat to compensate for heat flow through the bottom of the assembly, thereby helping ensure an approximately isothermal specimen. The furnace sits on a graphite ring that cushions its thin fragile end and ensures good electrical contact. (v) the nickel sleeve provides a highly conducting material to assist in keeping the specimen approximately isothermal (vi) the inner and outer soft-fired pyrophyllite sleeves protect the furnace from reaction with the salt inside and outside the furnace, respectively; the inner sleeve also insulates the furnace from the nickel sleeve. It is particularly important to protect the furnace when the internal salt is molten because the molten salt, with time, destroys the furnace. (vii) The outer NaCl salt simply fills the unused volume of the assembly. (viii) The thermocouples are assembled and put into place prior to casting molten CsCl around the specimen. (ix) The two Al_2O_3 solid cylinders at the base provide a stand upon which the specimen sits within the hot spot of the furnace. (x) The WC base anvil serves as both the hot electrical lead and the strong base upon which everything fits and the hard-fired pyrophyllite sleeve + unfired pyrophyllite ring around the base anvil both insulate the base anvil electrically and serve as a very high friction seal to prevent blow outs at the bottom. (xi)

The upper CsCl part provides a very low friction sleeve around the Al_2O_3 upper piston that the WC piston impinges upon as the experiment begins. (xii) Prior to insertion of the assembly into the pressure vessel, the entire outer cylindrical surface is wrapped with teflon tape to minimize the friction between sample assembly and the tungsten carbide core as well as protect the core from salt and lead. A type-B ($\text{Pt}_{94}\text{Rh}_6\text{-Pt}_{70}\text{Rh}_{30}$) thermocouple is placed near each end of the specimen to monitor the homogeneity of temperature in the specimen. The optimal temperature difference between the two thermocouples is less than 15 °C. 99.8% dense alumina tubing is used for the short pieces of the thermocouples sheaths, to support the thermocouples where they penetrate the furnace and sleeves. Mullite, which can collapse around the thermocouple wires to protect them from breaking, is used for thermocouples sheaths outside the furnace.

1.5.3 Analytical Methods

In order to examine the post-experiment specimens, over 100 samples were prepared as doubly-polished petrographic thin sections, which were subsequently investigated utilizing polarized optical microscopy, scanning electron microscopy (SEM) (Figure 1.5), and transmission electron microscopy (TEM) (Figure 1.6), SEM and TEM are located in the Central Facility for Advance Microscopy and Microanalysis (CFAMM) on the UCR campus. All the SEM figures are back-scattered electron images taken with an

accelerating voltage of 15 kV in a Philips XL-30 field mission SEM equipped with energy dispersive x-ray microanalysis (EDAX). Samples with nanometric crystals were also examined in an FEI-Philips CM300 TEM equipped with Phoenix EDX microanalyzer at 300 kV accelerating voltage. The TEM foils ($10\text{ }\mu\text{m} \times 5\text{ }\mu\text{m} \times 100\text{ nm}$) were prepared by FEI Company using the focused ion beam (FIB) technique. The FIB technique is now widely used to prepare specimens for TEM observation that require very thin foils ($\sim 100\text{ nm}$). The FIB allows precise choice of the region to be thinned by use of SEM images for location. A platinum “strap” is evaporated along the line where the foil will be cut, thereby providing strength to hold the foil together if cracks develop during thinning and ensuring that the resulting foil will be electrically conductive and therefore much less vulnerable to charging than it otherwise would be. Then a high energy Ga^+ ion beam sputters away material and in that way “digs” two wedge-shaped troughs on both sides of the plane of the desired TEM foil. When the foil is thin enough to become transparent to electrons, it is separated from the bulk sample by a sophisticated “lift-out” device and placed on a TEM grid.

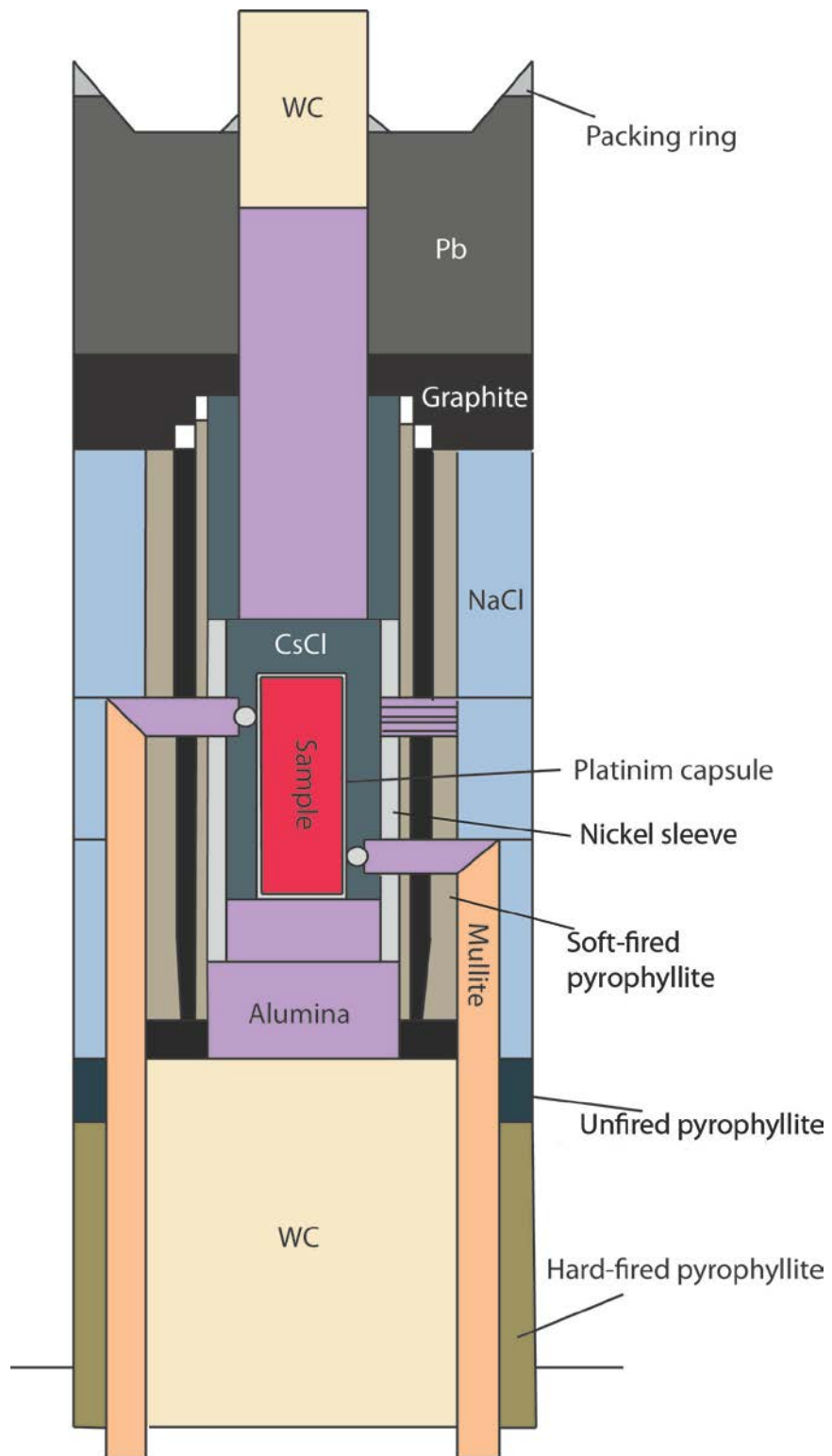


Figure 1.4 Schematic drawing of high pressure sample assembly for Griggs apparatus.

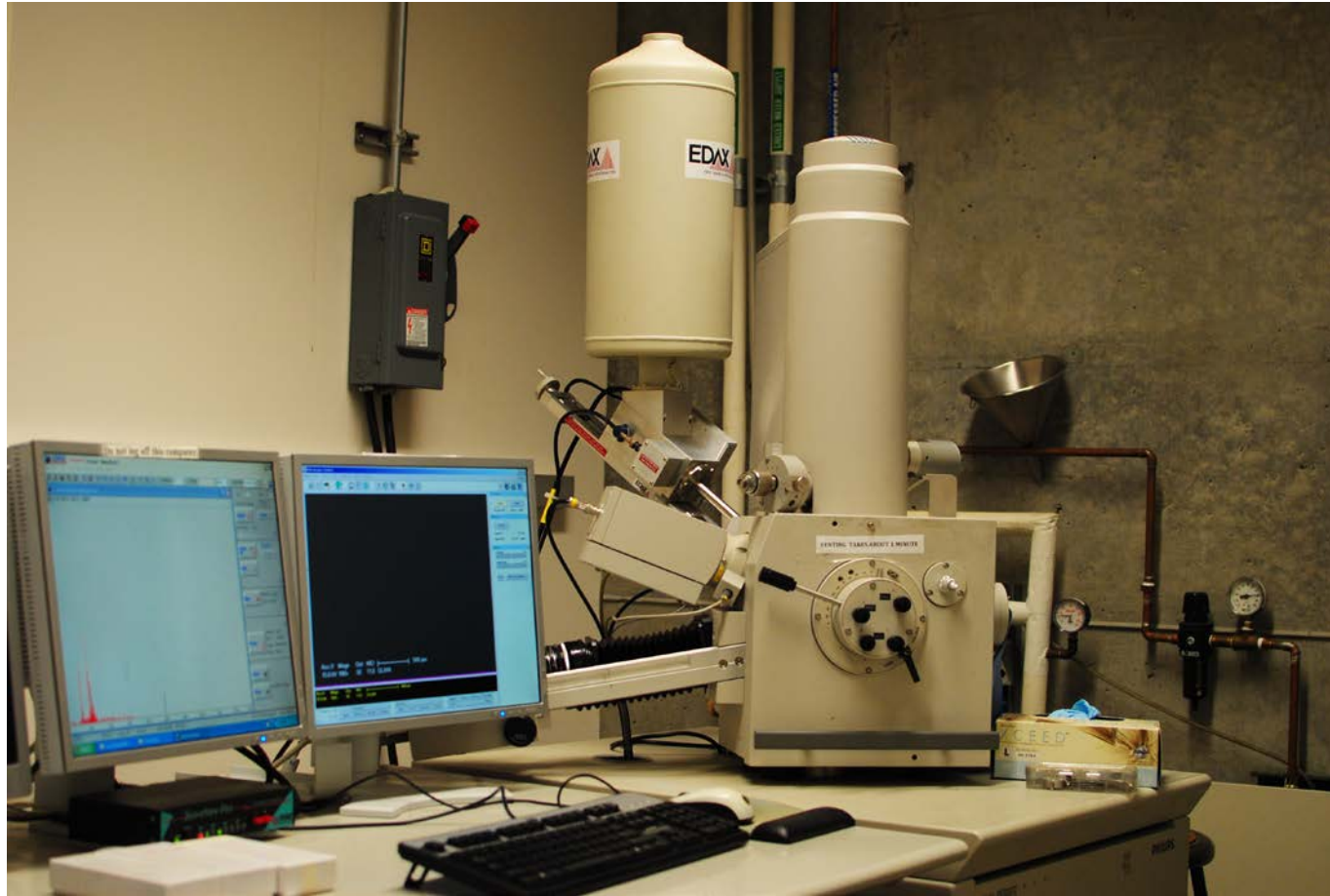


Figure 1.5 Photograph of Scanning electron microscope (SEM, XL30-FEG) in CFAMM at UC, Riverside.

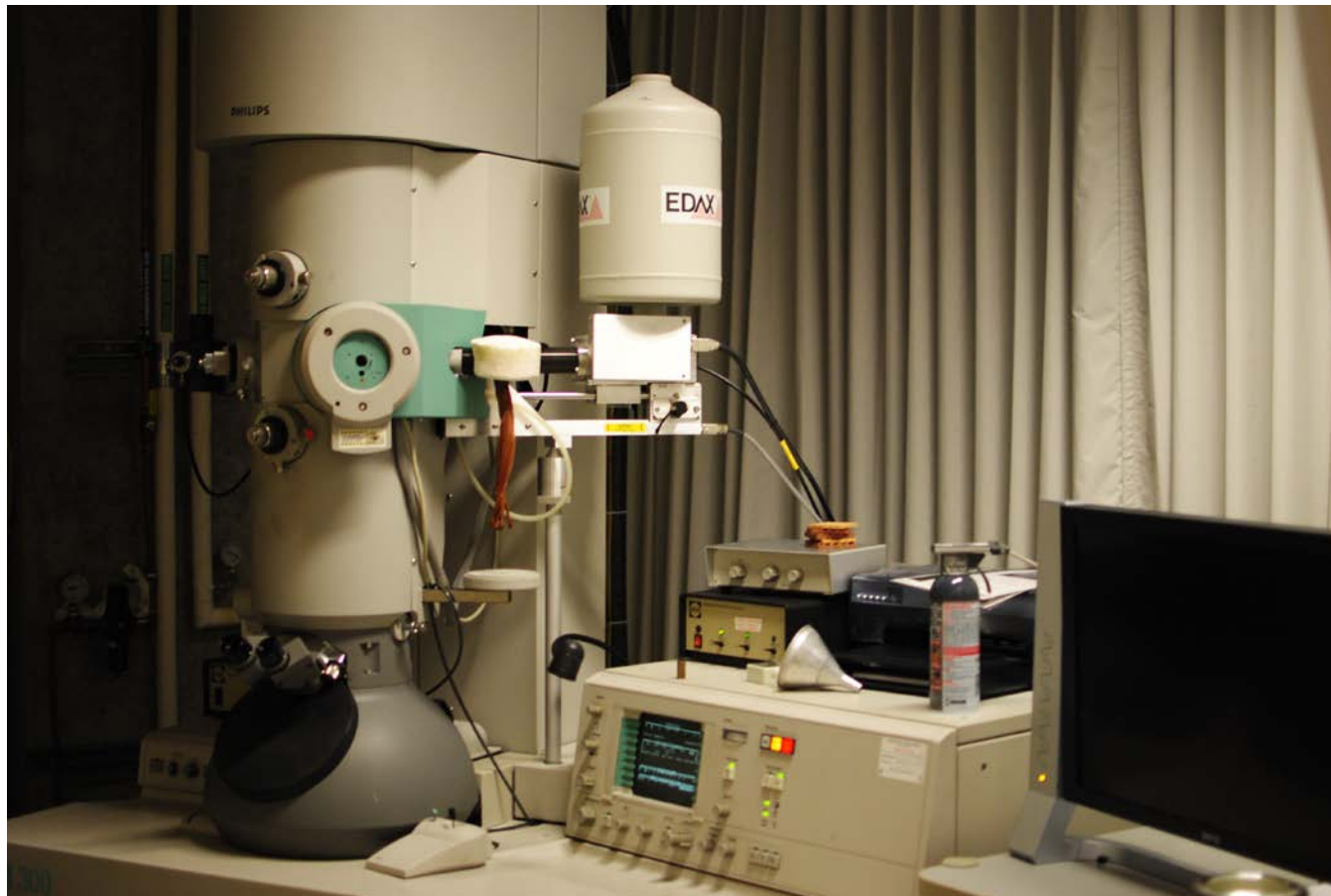


Figure 1.6 Photograph of Transmission electron microscope (TEM, FEI-Philips CM300) in CFAMM at UC, Riverside.

Chapter 2.

Compositional Dependence of Dehydration Embrittlement in Serpentinized Peridotite

2.1 Abstract

Double seismic zones (DSZ) which have two parallel planes of seismicity separated by 15-40 km are a global feature of subduction zones in the 70-250 km depth range (Brudzinski et al., 2007). While the physical mechanism of lower plane seismicity is still controversial, the leading hypotheses currently are associated with dehydration of antigorite serpentine within the subducting mantle plate (Peacock, 2001; Jung et al., 2004). In this study, we are conducting high-pressure (1-3GPa), high-temperature (720°C), deformation experiments on specimens of varying compositions of serpentine plus peridotite in our 4GPa Modified Griggs apparatus. Using samples composed of interlayered thin discs of antigorite and harzburgite, we find that dehydration embrittlement occurs down to less than ~30 vol % antigorite. Interlayered mineralogy was impractical at lower antigorite fractions so we prepared homogeneous mixtures of powders of the two rock types (35-75 μm grain-size) and “warm” pressed them to a coherent solid with little porosity. Subsequent deformation of these specimens extended the faulting regime to as little as ~8 vol % antigorite. In summary, we find that faulting occurs during dehydration in a wide range of serpentinized peridotite compositions but not during dehydration of nearly pure serpentinite nor nearly pure peridotite. We suggest that the lack of faulting in nearly pure peridotite is a consequence of too little H_2O production and the lack of faulting in nearly pure serpentine is due to extensive

crystal plasticity, as proposed previously by Chernak and Hirth (2011). We would expect that slower strain rates may change the compositions at which faulting stops but are unlikely to change the fact that there are cutoffs at both ends of the spectrum.

Likely consequences of this behavior in subduction zones are: (i) aseismic behavior of the plate interface that becomes serpentized as fluid escapes from the slab due to dehydration below; (ii) serpentine diapirs frequently found in forearc basins; (iii) earthquake generation from moderately serpentized lithosphere during dehydration.

2.2 Introduction

Earthquakes occurring at shallow depth ($< 70\text{km}$) result from brittle failure and frictional sliding along preexisting faults. However, rocks are expected to behave differently at greater depths. The fracture strength of rock increases rapidly with increasing pressure but is insensitive to temperature (Scholz, 2002), whereas the flow strength of rock decreases rapidly with increasing temperature and increases only moderately with increasing pressure (Poirier, 1985). The combination of these two facts indicates that unassisted brittle phenomena should be inhibited at depths greater than 50-70 km because both pressure and temperature increase with depth. Instead, rocks are expected to flow; any fault or potential fault would be replaced by homogeneous flow or, perhaps, a ductile shear zone. Nevertheless, Earthquakes are observed from the surface to the bottom of mantle transition zone ($\sim 700\text{km}$), where they cease abruptly (e.g. Frohlich, 1989). The overall number of subduction earthquakes decreases from ~ 50 km to a minimum at ~ 300 km. Then it increase between 400-600km before dropping exponentially to zero between 600 and 680 km (Figure 2.1). In addition, just like shallow earthquakes, deep earthquakes (> 70 km) display double-couple focal mechanisms, indicating offset across a plane, like slip on a fault (Kawakatsu, 1991; Green and Houston, 1995).

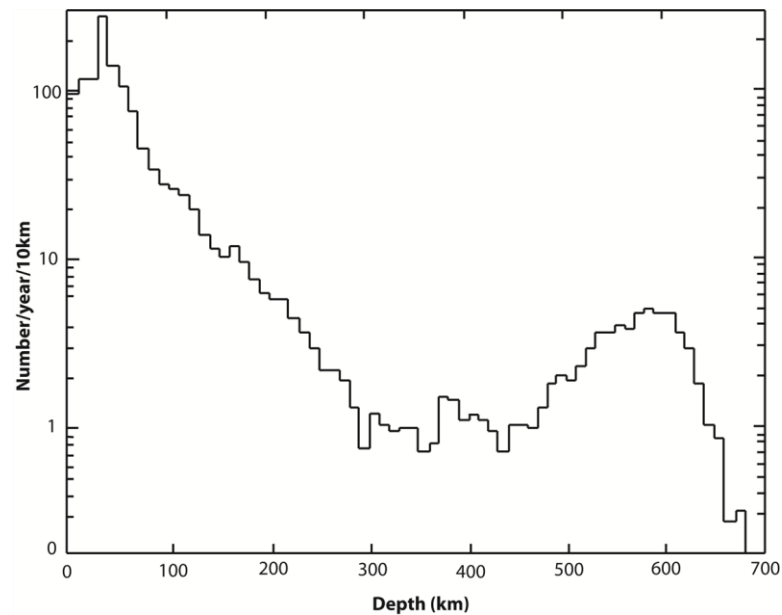


Figure 2.1 Globally averaged number of earthquakes (body wave magnitude, ≥ 5) per year as a function of depth (after Frohlich, 1989).

There are at least three physical mechanisms that have been proposed to answer this conundrum. Two of them have been tested in the laboratory, which appear to have counterparts in Earth, separately (Green and Houston 1995; Green, 2007):

i. Dehydration embrittlement of hydrous minerals, especially serpentine (eg: Raleigh and Paterson, 1965; Raleigh, 1967; Meade and Jeanloz, 1991; Escartin et al., 1997; Dobson, 2002; Jung et al., 2004), or even dehydration from nominally anhydrous minerals (Zhang J et al., 2004). In this process, the generation of a fluid phase during

dehydration reaction promotes the opening of microcracks and activate their interaction, which results in reactivation of a preexisting fault or nucleation of a new fault. Perrillat et al. (2005) showed that dehydration kinetics of antigorite, under undrained conditions, is faster than the relaxation time for antigorite (Hilariet et al., 2007), and therefore is enough to build up pore pressure and lead to earthquakes. Seismic and petrologic studies (Peacock, 2001; Hacker et al., 2003; Zhang H et al., 2004) strongly support this mechanism for intermediate-depth earthquakes (70-300 km). Both results from Meade and Jeanloz (1991) and Jung et al. (2004) suggested that this mechanism could support faulting to depths even greater than seismogenic zone if an appropriate dehydrating phase is present. However, the seismic identification of metastable olivine (incompatible with fluid released from hydrous phases) and the lack of evidence linking deep earthquakes with dehydration of high-pressure hydrous phases powerfully suggests that subduction zones are essentially dry below 400 km (Green et al., 2010; Kawakatsu and Yoshioka, 2011), limiting this mechanism to depths less than the mantle transition zone (MTZ) that begins ~400km.

ii. Phase-transformation -induced faulting (Green and Burnley, 1989; Green et al., 1990; Burnley et al., 1991; Kirby et al., 1991; 1996; Green and Houston, 1995; Green and Marone, 2002; Green, 2007). This mechanism only can operate under certain restricted conditions during exothermic polymorphic phase transformations involving a volume

change (Green and Zhou, 1996; Gleason and Green, 2008; Zhao et al., 2011). This reaction is known to occur when olivine transforms to its higher-pressure polymorphs under nucleation-inhibited condition and pressure comparable to that in the MTZ, the region between the two prominent seismic discontinuities at ~400 km and ~700 km. It means that this mechanism cannot operate at depths shallower than 400 km where olivine is stable. Ample seismic evidence for the presence of metastable olivine in the mantle transition zone of at least four cold subduction zones: Tonga (Chen & Brudzinski, 2001; 2003), southwest Japan (Iidaka & Suetsugu, 1992), Mariana (Kaneshima et al., 2007), and northeast Japan (Jiang et al., 2008), powerfully supports this mechanism to be responsible for the deepest earthquakes, making it a potential explanation for the second peak of bimodal distribution of earthquakes as a function of depth.

iii. Shear heating instabilities. Assuming hydrous phases are absent, it has also been proposed that localized shearing could generate heat faster than dissipation via conduction, which would speed up the slip and therefore generate more heat, etc. This shear-induced thermal runaway could lead to melting along the fault and trigger earthquakes (Ogawa, 1987; Karato et al., 2001). Kanamori et al. (1998) tried to use this mechanism to explain the 1994 Bolivian deep earthquakes which is the greatest rupture ever recorded below 300 km. Unfortunately, they explained why the earthquake could only grow so large because of shear heating mechanism based on an unspecified

mechanism that triggered the earthquake. More recently, Kelemen and Hirth (2007) used numerical simulation to model intermediate-depth earthquakes by such shear heating instability mechanism, but failure, in their model, can be achieved before reaching the melting point. Considering the lack of direct experimental studies supporting this mechanism, it is still not clear whether this mechanism can lead to the nucleation of deep earthquakes.

2.2.1 Double Seismic Zones

In a number of subduction zones, including Alaska (Abers, 1996), Aleutians (Abers and Sarker, 1992; Engdahl and Scholz, 1977), Tonga (Kwakatsu, 1986) and Northeast Japan (Hasegawa et al., 1978; Hacker et al., 2003), earthquakes occurring at intermediate-depths (70-350 km) define at least two planes of seismicities separated by 20-50 km, which appear to merge at greater depths (Figure 2.2).

Brudzinski et al. (2007) have now demonstrated that double seismic zones are a global feature observed in all subduction zones. They further demonstrated that the thickness of the "silent" zone between the two zones varies as a function of subducting plate age in such a way that they are consistent with the view that lower plane earthquakes are caused by dehydration embrittlement of antigorite serpentine, as originally proposed by Peacock (2001). Seismic study focused on the ratio of P- to S-wave

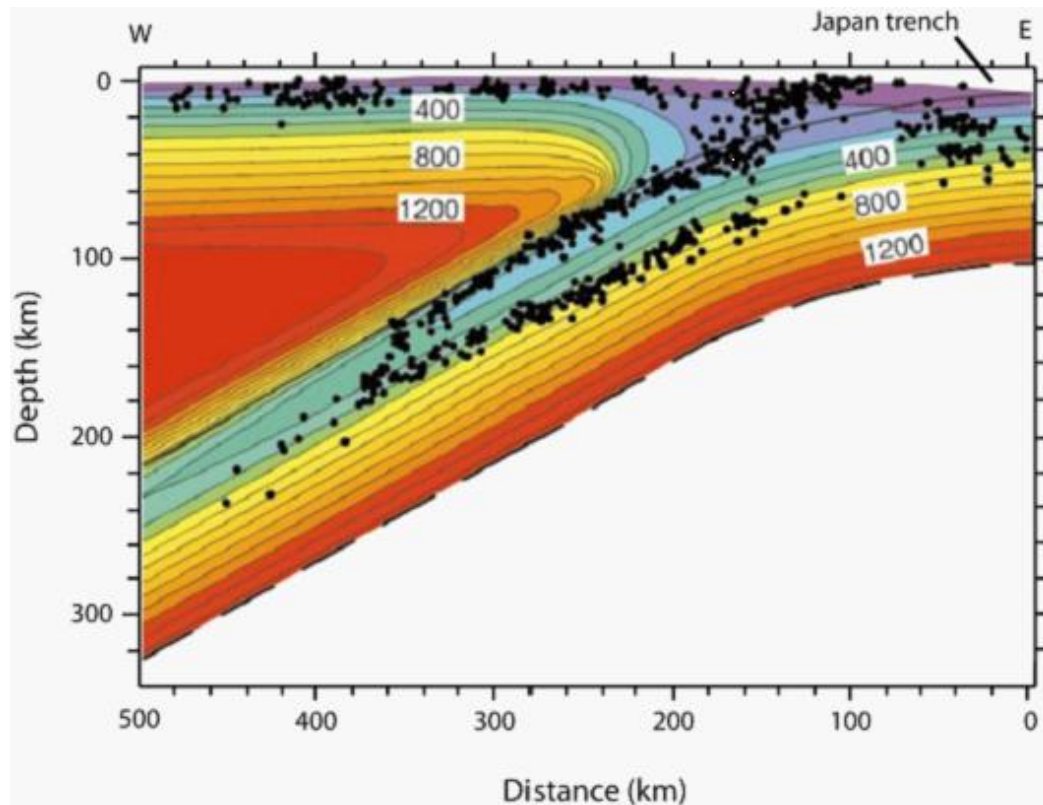


Figure 2.2 Double seismic zone beneath northeast Japan (Peacock, 2001). Epicenters, in black dots, highlight the double seismic zone. Temperatures reflected the calculated thermal structure, temperature decreasing pattern is represented by red to blue color sequence .

speed further supports that earthquakes occurring to depths as deep as 350 km (or at least the great majority of them) result from dehydration embrittlement, where the upper plane seismicities are associated with the transformation of altered oceanic crust

(Kirby et al., 1996; Zhang H et al., 2004) and the lower plane seismicities are triggered by dehydration embrittlement of antigorite in the oceanic mantle (Peacock, 2001; Hacker, 2003; Yamasaki and Seno, 2003; Jung et al., 2004).

2.2.2 Hydration of subducting lithosphere

A critical prerequisite for the dehydration embrittlement mechanism to function is for the lithosphere to be hydrated (e.g. serpentinized) continuously during subduction at least in the regions where seismicity is observed. Therefore, if the lower plane of seismicity in slabs is due to dehydration embrittlement, the slabs must be hydrated to a depth of 30-40 km almost immediately below trenches. Studies of ophiolites and ocean floor dredge hauls show that the oceanic crust is significantly altered by the hydrothermal circulation through the fractures created in the vicinity of mid ocean ridges. In contrast, mantle xenoliths from oceanic volcanoes show very little evidence of hydrous alteration and seismic velocities of the mantle immediately beneath the oceanic lithosphere are not consistent with significant serpentinization. As a consequence, if hydration is required almost immediately beneath trenches, that hydration must occur at the trench. However, this argument has been resisted for many years because it is not clear how hydration could occur so deeply along the faults that accommodate slab bending at the trench.

Seno and Yamanaka (1996) suggested that hydration of mantle lithosphere could occur when it passes over a plume or superplume, but this mechanism can only be used to explain a few subduction zones that have recently passed over such a feature. Moreover, we have every reason to expect that the earthquake distribution seen in slabs is a steady-state phenomenon that requires a continuing source of hydrous alteration, not something that might happen by chance now and then. The results from Brudzinski et al. (2007) required that the hydration corresponding to the lower plane seismicity in the mantle lithosphere must occur in all subducting slabs, which indicates that the hydration must take place at outer rise zones just before subduction. Ranero et al. (2003) demonstrated that significant numbers of normal faults are formed to depth in excess of 20 km as an ocean slab bends down for subduction, potentially providing conduits for deep hydration. A recent modeling study by Faccenda et al. (2009) produced more promising results. They envisioned that bending and unbending of oceanic lithosphere can provide a downward pressure gradient, pumping the water down the fault. Therefore, although the mechanism of water into the slab remains obscure, a plausible path is identified and the lower plane of seismicity is consistent with dehydration embrittlement of antigorite (Peacock, 2001; Brudzinski et al., 2007). In combination, this makes for a powerful argument.

2.2.3 Motivation and goal of this study

The antigorite phase diagram (Figure 2.3) shows that the total volume change, ΔV , of dehydration reaction changes from positive at pressure lower than 2.2GPa to negative at pressure above 2.2GPa. A negative ΔV is expected to inhibit dehydration embrittlement from the view of conventional rock mechanics (Ko et al., 1997; Wong et al., 1997). However, Jung et al. (2004) demonstrated that dehydration-induced faulting in peridotite that is partially altered to antigorite serpentine occurs even at pressures much higher than 2.2 GPa, under which the ΔV is negative. Nevertheless, a more recent study by Chernak and Hirth (2011) showed that experiments on pure serpentine did not lead to embrittlement during dehydration. Only localized shear zones and slow slip were found; macroscopic faulting was not observed during dehydration of antigorite.

Both seismic evidence showing limited deep mantle hydration and current experimental results indicate that there could be very small amount of serpentine formed at depth corresponding to the lower layer seismicity. Hence the first order issue we want to investigate is **how much serpentine at minimum** is required to produce such dehydration embrittlement. The result of this study will, in turn, put constraints on the level of hydration in subducting lithosphere and therefore the amount (lower bound) of H_2O that could penetrate into the deep mantle.

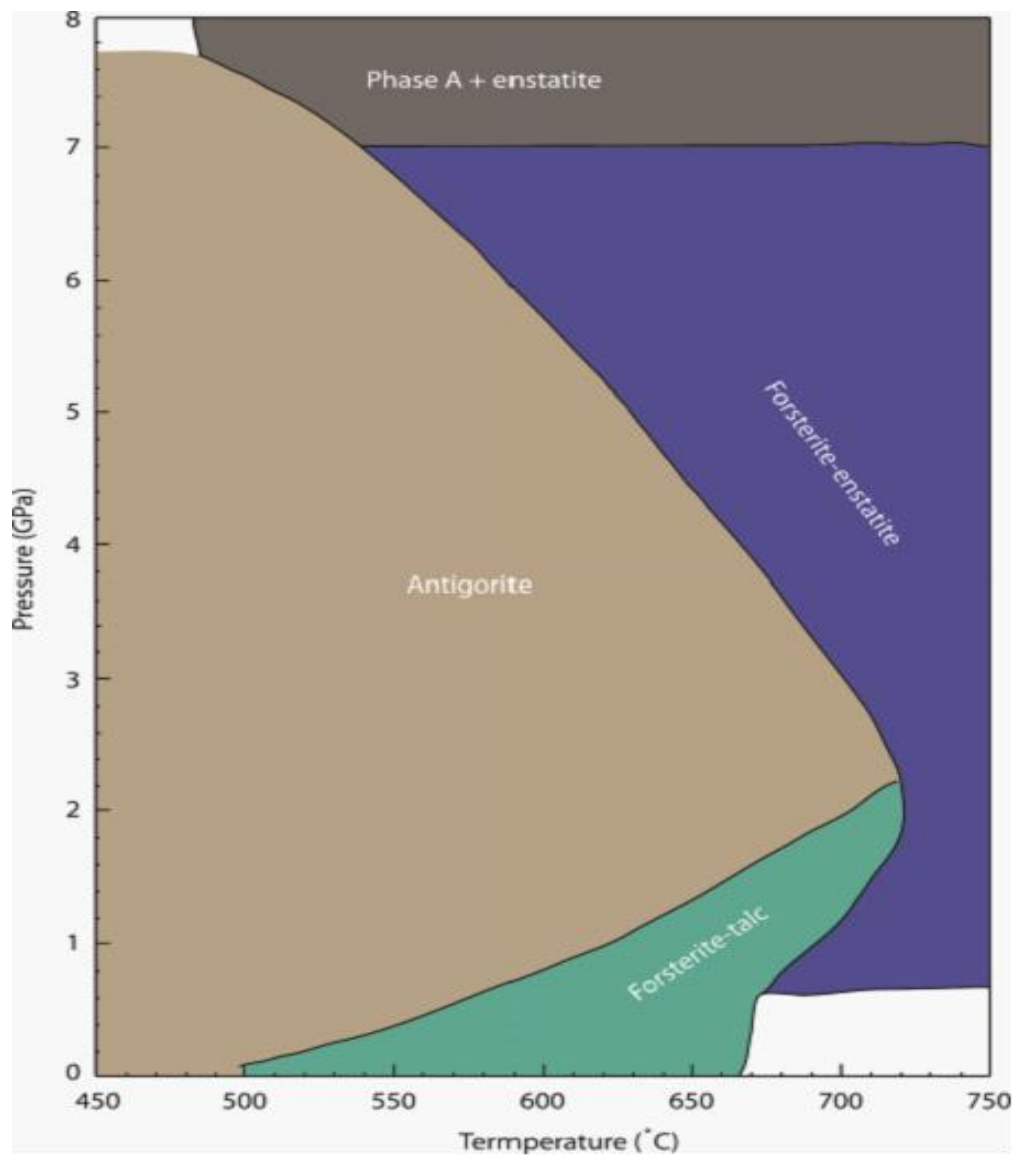


Figure 2.3 Phase diagram for antigorite dehydration reactions (after Ulmer and Trommsdorff, 1995).

Moreover, although in the earlier work of Jung et al. (2004) it was clear that the solid and fluid products of antigorite dehydration reaction separate almost immediately after their generation, the resolution and contrast were inadequate to examine how ultrafine-grained products are separated and distributed. In this study, I use high resolution SEM and TEM to investigate the fault configuration as well as the microstructure of dehydration products. This study will promote our understanding of the physics behind the dehydration embrittlement mechanism.

2.3 Starting Materials and Methods

Varying proportions of a natural antigorite serpentinite and a synthetic harzburgite are used as my starting materials. Antigorite, $(\text{Mg,Fe})_3\text{Si}_2\text{O}_5(\text{OH})_4$, is the high temperature and high pressure polymorph of serpentine group minerals, the other two polymorphs are chrysotile and lizardite. The antigorite serpentinite (Figure 2.4a) was collected from Val Malenco, Italy; there is a significant fraction of olivine (~40 vol%) within the serpentinite and the rock is highly foliated. This is the same antigorite serpentinite used by Ulmer and Trommsdorff (1995) to determine the phase diagram for antigorite. We used it for this study specifically for that reason because there has been considerable variation of phase diagrams produced for this mineral due to compositional differences. The synthetic harzburgite (Fig. 4b) is composed of 67 volume percent (vol%)

olivine, $(\text{Mg,Fe})_2\text{SiO}_4$ and 30 vol% ortho-pyroxene $(\text{Mg,Fe})\text{SiO}_3$, and ~3 vol% of diopside $\text{MgCaSi}_2\text{O}_6$. Such a combination (Table 1) provides a good approximation of the composition of upper mantle mineralogy of the subducting oceanic plate. Both interlayered and powder specimens were used to conduct experiments. I initiated experiments using cylindrical specimens that consist of a "stack of coins" -- interlayered serpentinite and harzburgite (Figure 2.5b). I started with 1-to-1 alternation of "coins", then the serpentine content was gradually reduced to ~30 vol%. To compare with more

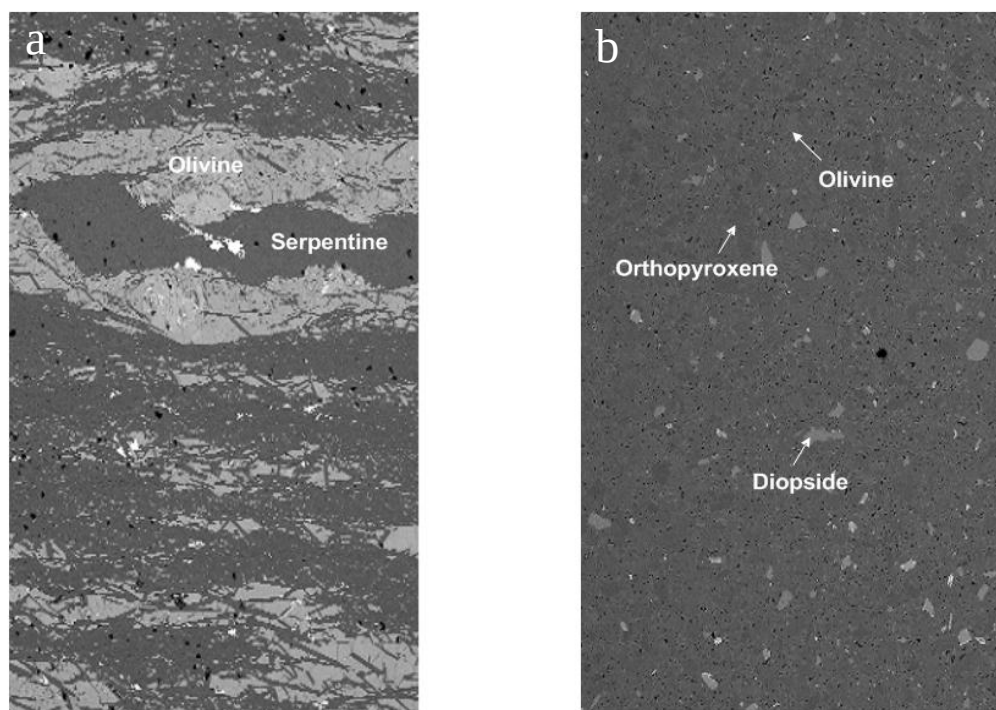


Figure 2.4 SEM(BSE) Images of Starting materials. a: Serpentinite; b: Harzburgite.

homogeneous specimens, I also performed a series of experiments with mixed powders of the starting materials (Figure 2.5a). Because of the way we consolidate the powders into rock for deformation (see below), the majority of antigorite grains in the mixed powder samples became oriented, forming a foliation perpendicular to the maximum

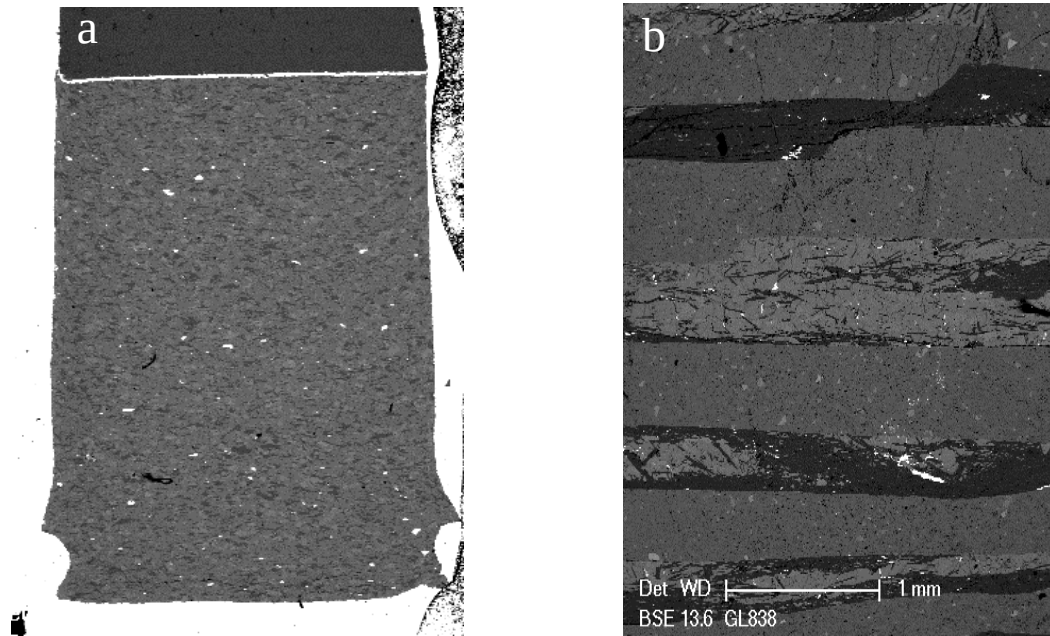


Figure 2.5 Two types of specimen configurations. a: mixed powder specimen; b: Interlayered specimen.

Table 2.1
Composition of Starting Materials Used in Experiments
(Energy Dispersive X-ray Spectroscopy)

Serpentinite (mol %)			Harzburgite (mol %)	
	Antigorite	Olivine	Olivine	Orthopyroxene
MgO	56.62	57.32	60.91	43.54
Al ₂ O ₃	1.18	-	-	2.17
SiO ₂	42.20	33.67	32.72	49.63
FeO	-	9.01	6.38	4.66
Antigorite: 60 vol. % Olivine: 40 vol. % magnetite: trace			Olivine: 67 vol. % Orthopyroxene: 30 vol. % Diopside (CPX): 3 vol. %	

compression direction, comparable with the orientation of serpentine grains in the interlayered samples. Both Serpentine and harzburgite separates were crushed and sieved to a grain size of 35~75 μ m for the mixed powder experiments.

Samples, both interlayered and mixed powders, were loaded into platinum capsules with dimensions 3 mm diameter and 9mm long. The capsules consist of two cylindrical parts, one of which slides smoothly into the other; both parts consist of tubes (0.01" thick) with one end welded shut with a platinum cap (0.01" thick). When assembled, the capsule has walls of thickness 0.02" thick. Pressurization produces a cold weld that becomes fully sealed. The oxygen fugacity was buffered by placing two thin Ni foils inside the capsule, one at each end of the specimen. The capsule was then placed into a standard high-pressure sample assembly for the Griggs apparatus (Figure 1.1 & 1.2).

Experiments with interlayered specimens were cold compacted with pressurization up to 280 MPa, then the temperature was increased to 300 °C at a heating rate of 7°/min. Further pressurization to 800 MPa was achieved at 2 MPa/min, followed by increasing temperature to desired temperature (720 °C). Samples were then annealed at target pressure 1 GPa and temperature 720 °C for ~60 min, and axially deformed at a strain rate of 10⁻⁴/s. Immediately following deformation, the sample was quenched to room temperature in seconds by turning off the power to the zero. In comparison,

experiments with mixed powder specimens were initially cooked at 3 GPa and 600 °C for 12 hours to produce fully compacted polycrystalline specimens. We established this protocol to ensure that all porosity was removed from the specimens prior to deformation. This “warm pressing” process results in a largely uniaxial shortening of the powder during compaction, thus producing the foliation of original antigorite grains mentioned in the previous paragraph. At the end of this protocol, the temperature was first reduced to 300 °C followed by slow pressure reduction to 1 GPa to insure integrity of the sample assembly during the process. Temperature was then ramped up again and deformation was carried out at $P = 1 \text{ GPa}$, $T = 720 \text{ °C}$ and $10^{-4} \sim 10^{-5}/\text{s}$, identical with that in interlayered-sample experiments.

2.4 Results and Discussion

2.4.1 Mechanical Results

In this study, a series of deformation experiments on serpentinized peridotite was carried out at $P = 1 \text{ GPa}$ and $T = 720 \text{ }^{\circ}\text{C}$, with different proportions of antigorite-harzburgite mixture. The experimental conditions and mechanical results are listed in Table 2.

Stress vs Strain curves are shown in Figure 6. To establish that the materials studied do not fail by normal brittle failure, we deformed a dry synthetic harzburgite at the same conditions as all other experiments. That specimen (GB478) was fully ductile with a flow strength 1600 MPa, stronger than all but one experiment containing antigorite (GB513, ~4 vol% serpentine content). Given the fact that the differential stress is almost double the confining pressure, the difference between these two experiments is not likely to be significant. The decrease of stress at the end of deformation of GB478 (dashed line) was caused by progressive tilting of the specimen. At constant temperature, the strengths of variably serpentinized specimens vary as a function of antigorite content in either mixed powder specimens or interlayered specimens. The more antigorite a specimen contains, the weaker the specimen is expected to be. That explanation is true for each type of experiment but, for a given amount of serpentine, the mixed powder specimens are weaker than the layered ones. We attribute that

systematic difference to the fact that, in the former, olivine crystals are disseminated in serpentine whereas in the latter, the olivine crystals in many cases constitute layers subnormal to compression (see below).

Table 2.2 Experimental Conditions and Mechanical Results

Expt No.	Composition	Strain rate(10^{-4}s^{-1})	Result
GL844	Serp ₆₅ Harz ₃₅	2.2	Faulted
GL840	Serp ₃₃ Harz ₆₇	2.3	Faulted
GL841	Serp ₂₇ Harz ₇₃	2.82	Faulted
GL842	Harzburgite	2.45	Flowed
GB495	Serp ₇₆ Harz ₂₄	4.10	Flowed
GL870	Serp ₄₅ Harz ₅₅	4.28	Faulted
GL865	Serp ₂₈ Harz ₇₂	3.87	Faulted
GB488	Serp ₈ Harz ₉₂	4.12	Faulted
GB513	Serp ₄ Harz ₉₆	4.05	Flowed
GB478	Harzburgite	3.97	Flowed
GB476	Serp ₈ Harz ₉₂	$3.94 \times 10^{-5}/\text{s}$	Faulted
GB493	Serp ₆₅ Harz ₃₅	4.13	Faulted

Note: All experiments, except GB493, were conducted at $P = 1 \text{ GPa}$, $T = 720 \text{ }^{\circ}\text{C}$. Experiment numbers in red represent interlayered specimens, while black ones represent mixed powder specimens. GB493 was conducted at $P = 2.7 \text{ GPa}$, $T = 750 \text{ }^{\circ}\text{C}$ and a strain rate of $10^{-5}/\text{s}$.

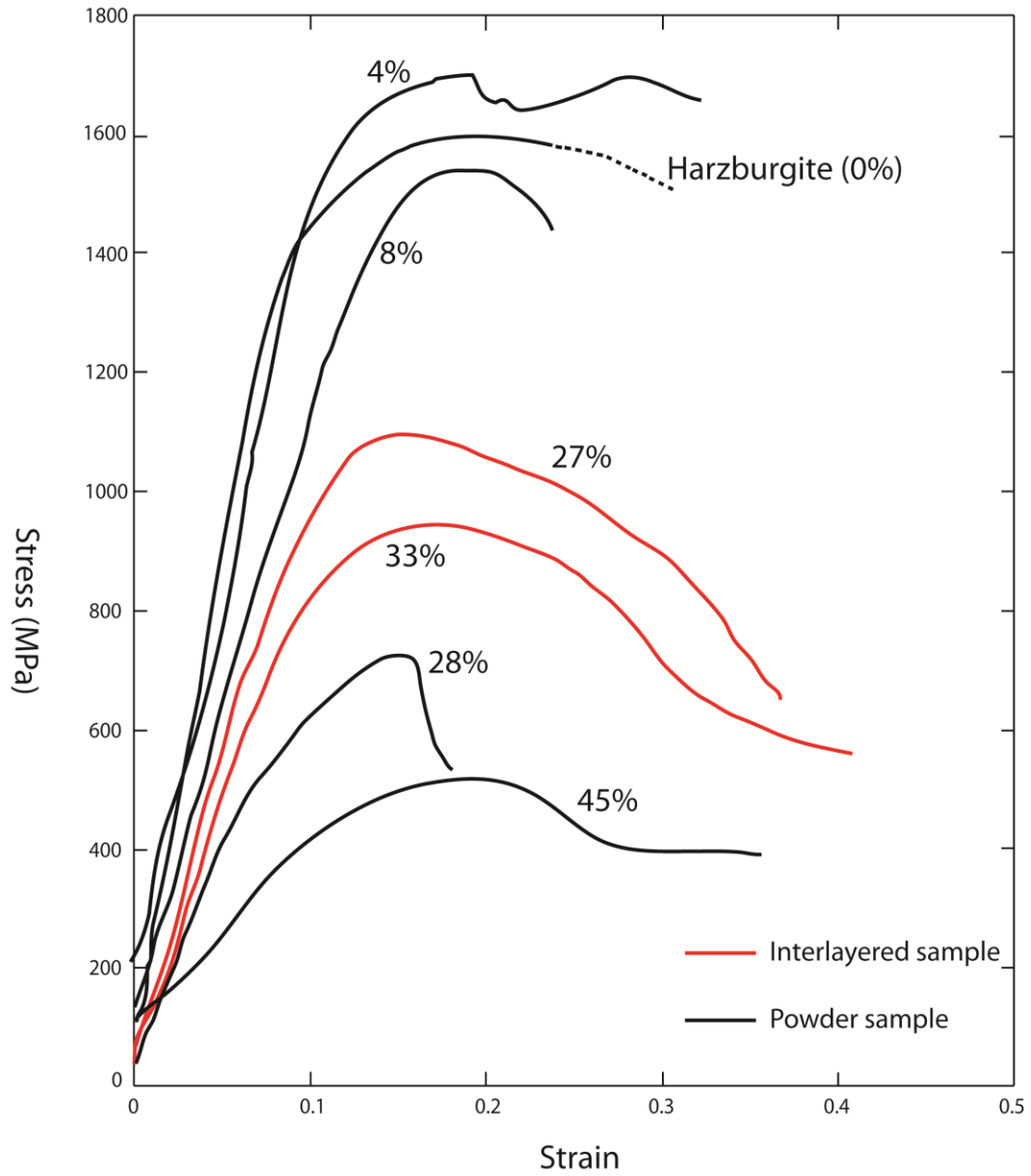


Figure 2.6 Stress versus strain plots. Note that at similar serpentine content, interlayered specimens are stronger than mixed powder specimens. In the former, olivine crystals are contiguous in layers whereas in the latter, olivine grains are isolated in a serpentine matrix, making the specimens weaker.

Experiments on interlayered specimens showed that dehydration-induced faulting occurred down to a serpentine content of 27 vol%. This specimen configuration allowed imaging of clear evidence of offset along faults (Figure 2.7a). Unfortunately, this technique is limited; specimens with lower amounts of serpentine could not be fabricated because making thinner layers of serpentine became essentially impossible due to their fragility. By contrast, one can fabricate a specimen with as little serpentine content as desired with mixed powder specimens. The latter also provides a much more homogeneous distribution of minerals throughout the specimen. With such specimens, dehydration embrittlement was observed in two specimens with only 8 vol% serpentine (GB476 & GB488) but GB513, with 4 vol% serpentine, was macroscopically ductile but displayed an irregular stress/strain curve after yielding (Figure 2.6) and a tiny incipient fault (see discussion of microstructures below).

2.4.2 SEM and TEM Microanalysis

In order to examine the fault configuration and evidence of dehydration reaction, Scanning Electron Microscopy (SEM) was applied in this study. All SEM images shown in this dissertation were taken using the Back Scattered Electron Detector (BSE) which provides sharp contrast among different phases. In our case, the darkest phase is antigorite; other phases (principally olivine and talc) are various shades of lighter gray.

Rare magnetite is very bright white. In addition, Transmission Electron Microscopy (TEM) was also adopted appropriately to study the nanostructure of dehydration products within fault zones.

Here, I describe several examples of our typical observations. Of the interlayered specimens, GL844 (Figure 2.7 & 2.8) contained the highest fraction of antigorite (65 vol%). The fault experienced a significant offset ($\sim 100\ \mu\text{m}$), as shown by displacement of olivine layers across the fault (Figure 2.7a). At larger magnification, it is clearly seen that the fault zone is sharply defined between coarse-grained antigorite and olivine (Figure 2.7b). Because the experiment was at an early stage of dehydration, away from the fault we can see that the reaction initiated at the grain boundaries of antigorite, leaving the grain interiors still un-reacted. The fault zone is less than $8\ \mu\text{m}$ thick (Figure 2.8a) and contains a high concentration of extremely fine-grained solid dehydration products. With further increased magnification, individual submicron-sized olivine grains are observed within the fault zone (Figure 2.8b).

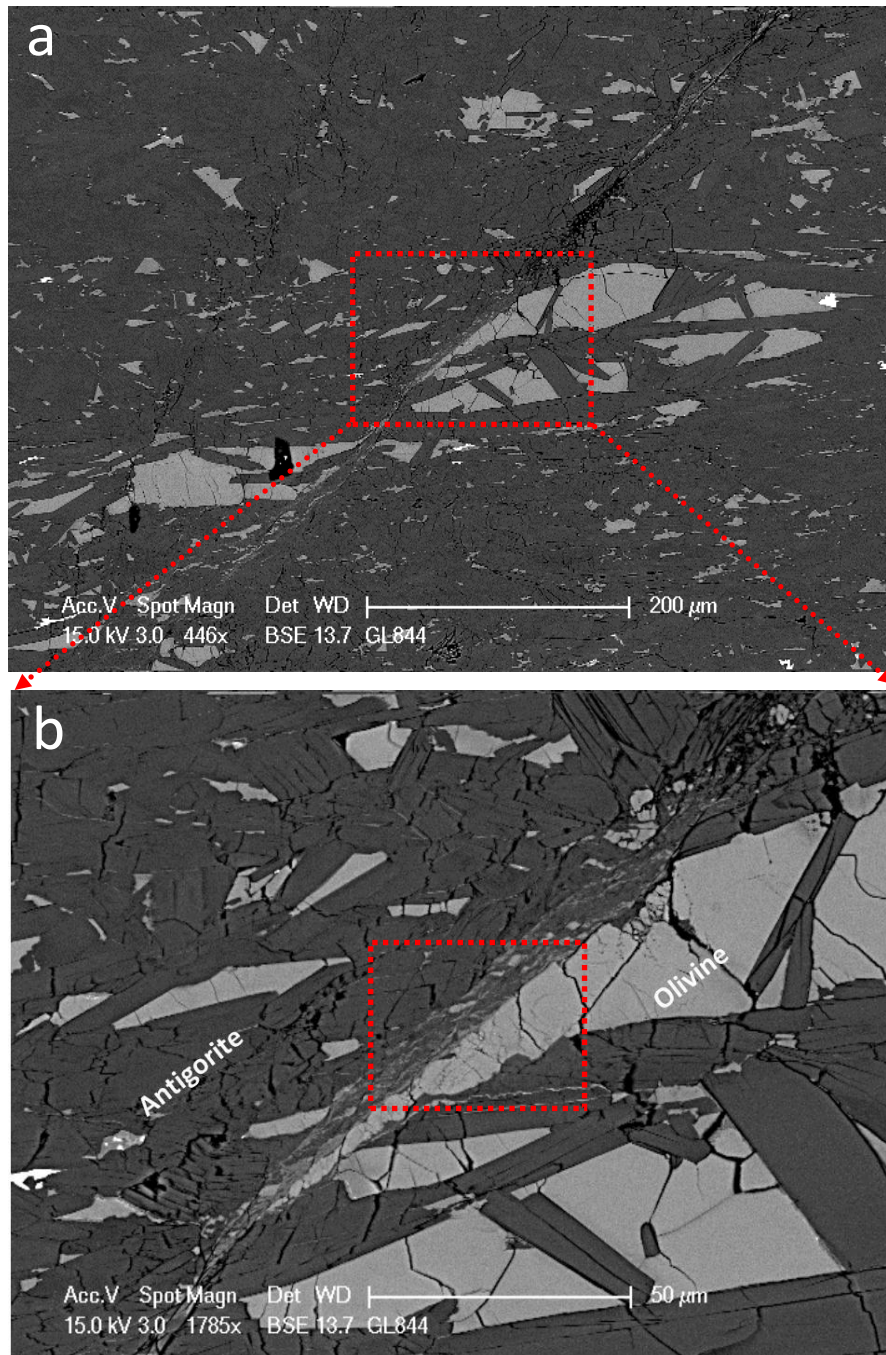


Figure 2.7 SEM (BSE) images of GL844. (a) Offset recorded by the large olivine grains (bright phase). (b) Fault zone is well constrained by coarse-grained antigorite and olivine. Red rectangle highlights the region for Figure 2.8a.

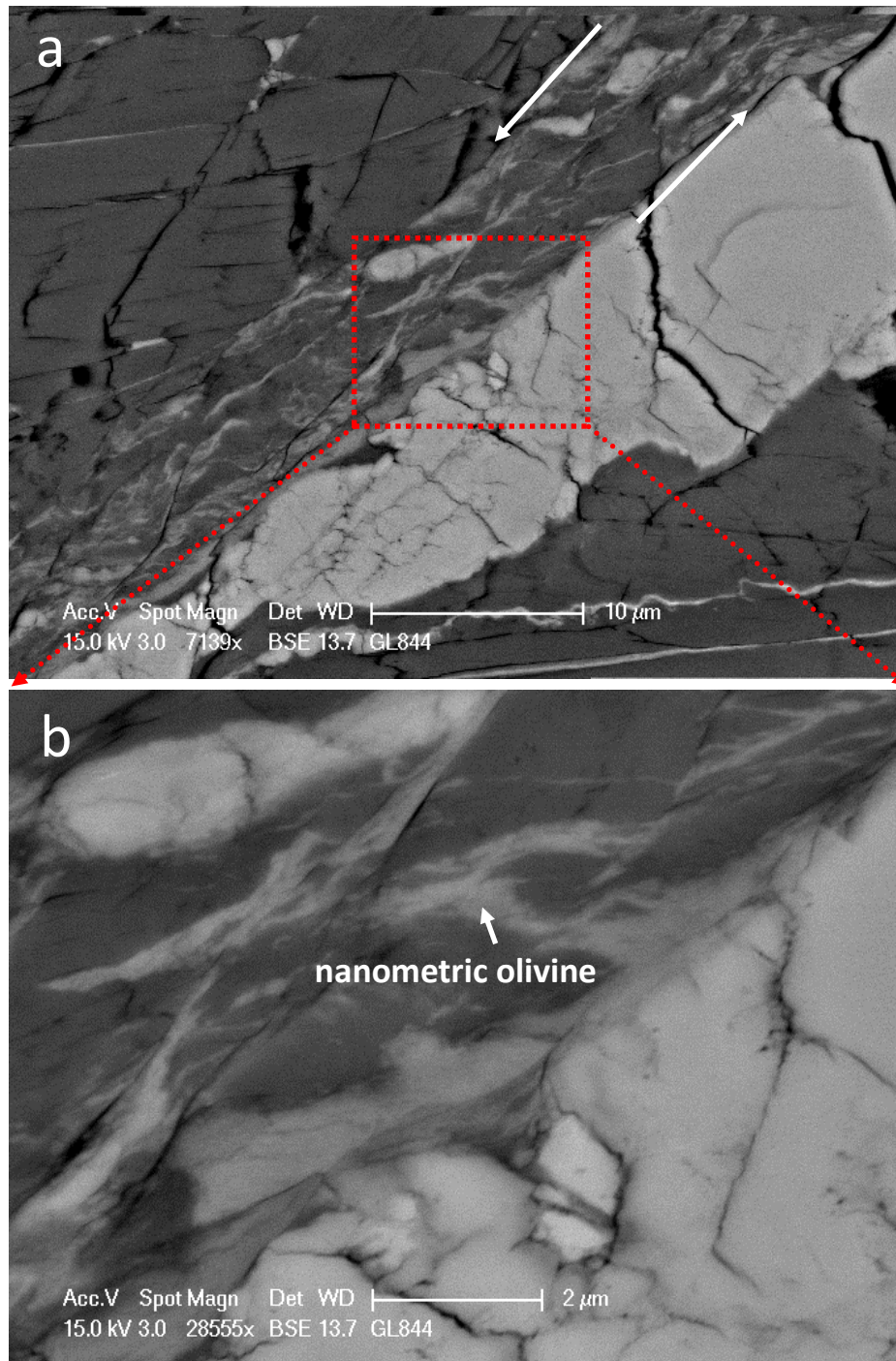


Figure 2.8 Continued SEM (BSE) images of GL844. (a) Fault zone is only ~8 μm thick. White arrows point out the sense of shear. (b) Nanometric olivine in the fault zone.

The other example of cored-sample experiments is GL840 (Figure 2.9 & 2.10), which contains 33 vol% of antigorite. Again, the fault can be easily identified by the offset ($\sim 100\ \mu\text{m}$) of olivine layers (Figure 2.9). Note the sharp edges of the two olivine layers clearly showing fracture of those grains. Enlargement of the region between the two olivine layers (Figure 2.10a), shows extensive reaction; dehydration was highly promoted within the fault zone, probably caused by the strain induced by sliding on the fault, but away from the fault only small amounts of dehydration are seen along grain boundaries. When we look inside this fault (Figure 2.10b), recognizable broken olivine fragments are rare; extremely fine-grained olivine and talc are again observed, filling the fault zone. The very bright phase is magnetite fragments.

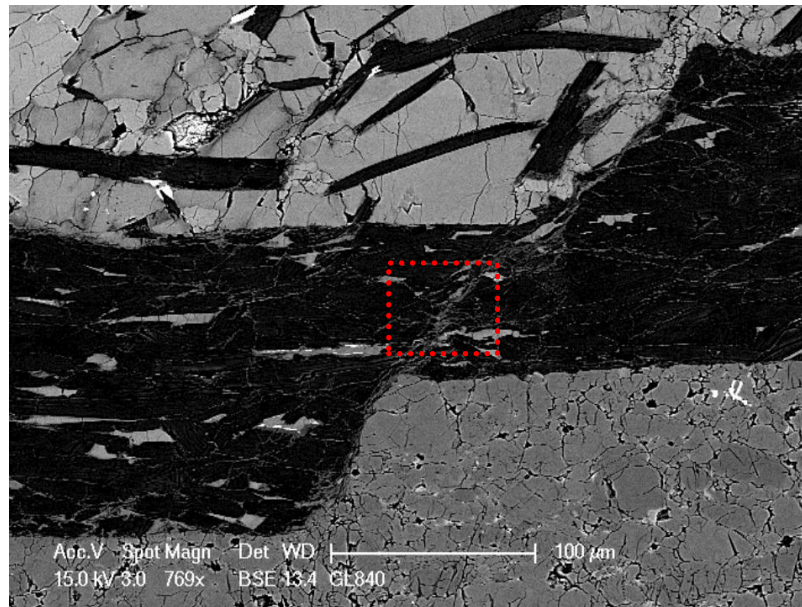


Figure 2.9 SEM (BSE) images of GL840. Fault cuts through the antigorite layer. Red rectangle highlights the region for Figure 10a.

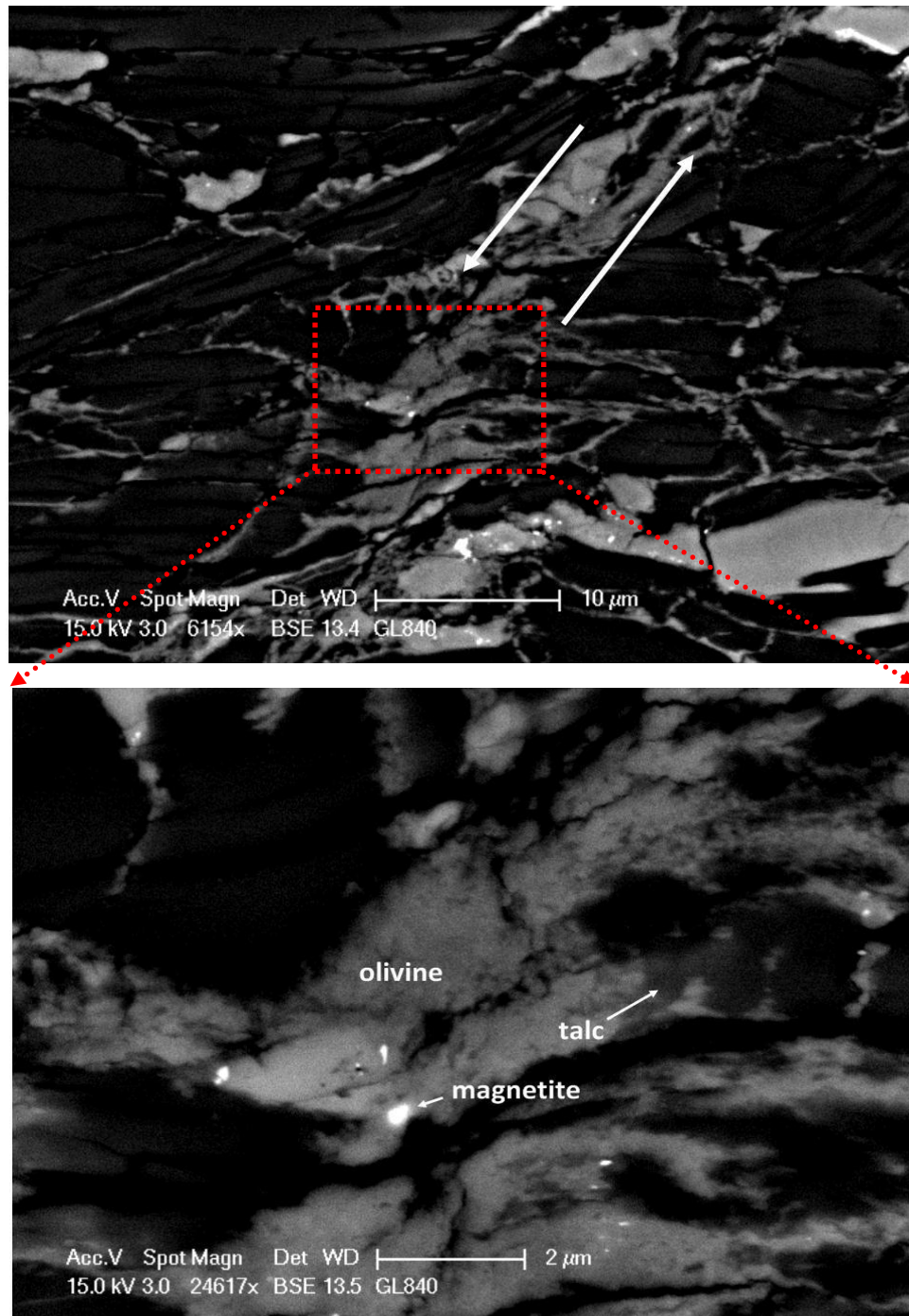


Figure 2.10 Continued SEM (BSE) images of GL840. (a) Dehydration reaction concentrated in the fault zone, highlighted by the bright dehydration product, olivine. White arrows point out the sense of shear. (b) Submicron olivine and talc grains in the fault zone. White phase is magnetite.

Regarding the powder-sample experiments, similar microstructures are observed. In specimen GL870 (Figure 2.11), which contains ~45 vol% of serpentine, the principal fault is ~30° running through the sample (Figure 2.11a); it splays out near the bottom of the specimen and a small conjugate fault is seen on the left, bottom. Bright white on the sides and bottom are Pt capsule; thin white line at top of specimen is a Ni foil separating sample from Al₂O₃ piston. Figure 2.11b shows broken fragments of olivine in the fault zone and, adjacent to the fault zone, a fluid-filled crack oriented parallel to the maximum compression direction (red arrow) and fluid inclusion trails denoting healed cracks. In the fault zone, we observe not only the nanometric dehydration products but also larger, broken, olivine fragments, which were probably dragged into the fault zone as the fault slipped. Figure 2.11c shows the fluidized new nanometric olivine grains flowing around the broken fragments.

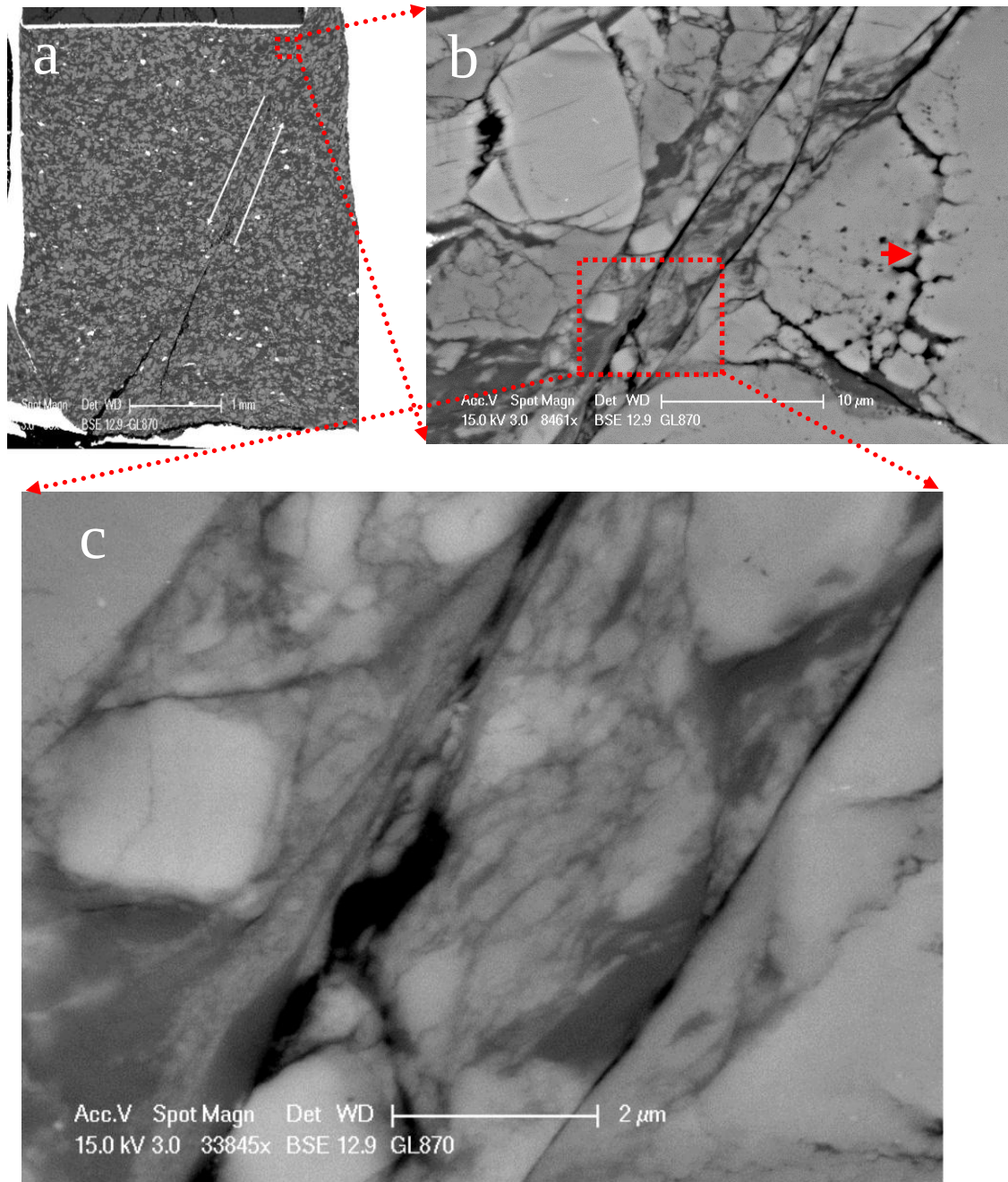


Figure 2.11 SEM (BSE) image of GL870. (a) Fault running through the sample at ~30° to the maximum compression direction. (b) Broken fragments of olivine and antigorite were observed in the fault zone (upper image). Fluid inclusion trails are also seen and highlighted by the mode I crack (red arrow). (c) Nanometric dehydration products are flowing around the large fragments (lower image).

To better understand the nanostructure of the dehydration products, GL870 was examined using TEM. The TEM foil (Figure 2.12) was cut using the Focused Ion Beam (FIB) technique (See chapter 1 for technical descriptions). A layer of platinum was deposited along the line where the foil is to be cut to prevent the foil from falling apart after it is prepared. As we focused in on the fault zone, we observed rare residual grains of bent antigorite showing 9Å fringes (Figure 2.13a) and new grains of talc (Figure 2.31b) with a dimension of ~10 nm, showing fringes of 9-10Å spacing. The apparently open triple junction and perhaps also grain boundaries in Figure 2.13c indicates either the former presence of fluid on the grain boundaries or the opening of boundaries during decompression or foil preparation; the suggestion of 3-5nm bubbles on several grain boundaries in this image strongly suggests the former presence of fluid. The diffraction pattern taken from within the fault zone displays multiple circles (Figure 2.13c), reflecting randomly oriented multiple phases in the fault zone, including relict antigorite as well as dehydration products, nanometric olivine and talc.

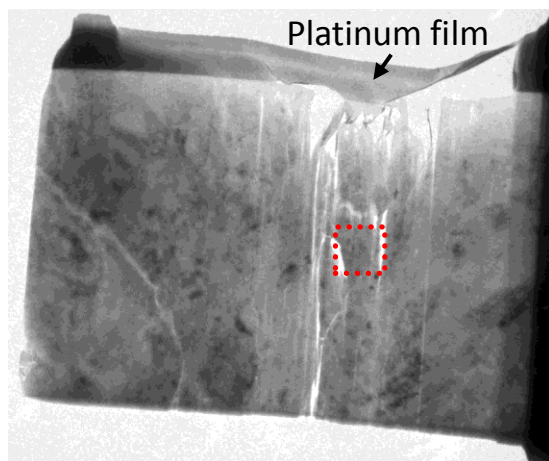
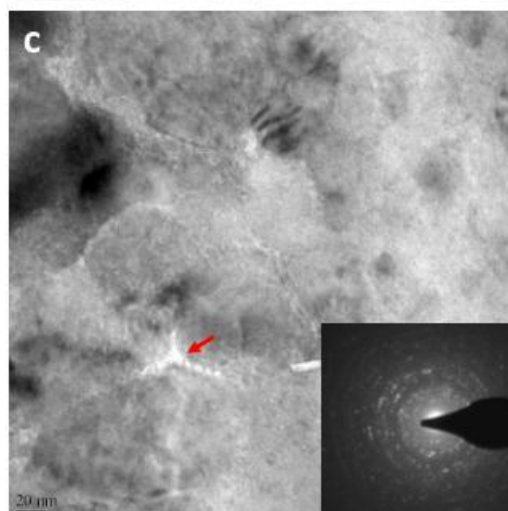
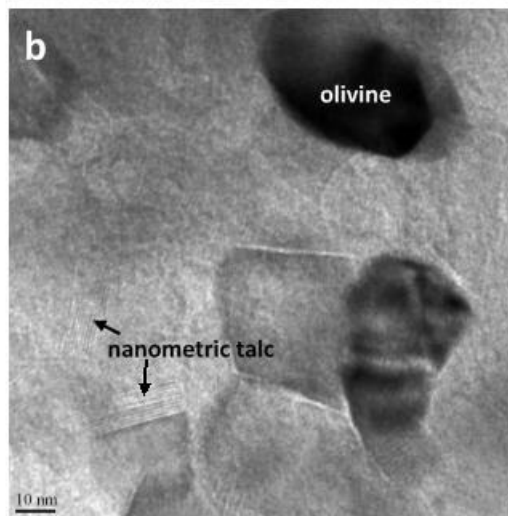
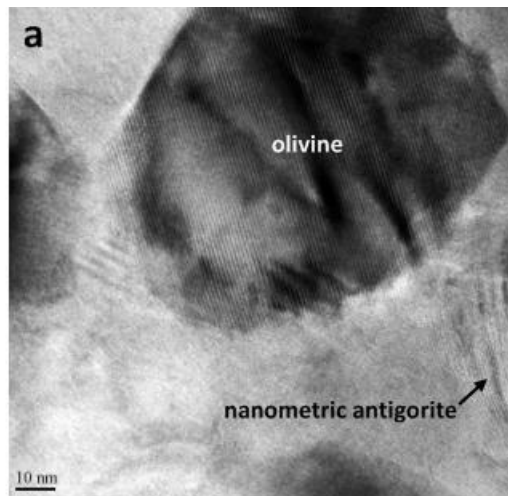


Figure 2.12 TEM foil cut through focused ion beam (FIB). A thin layer of platinum film was deposited on top of the foil. Red rectangle highlights the region for TEM analysis in Figure 11.

Figure 2.13 High resolution images of fault zone contents. (a) Residual olivine and antigorite in fault zone. Lattice fringes are visible in the relatively large olivine fragment (top) and also in the bent antigorite crystal. (b) Nanometric talc produced by dehydration. Note that the scale bar is 10 nm, and the dimension of talc is only 8 -10 unit cells thick. (c) Triple junction (black arrow) between nanometric grains of fault gouge. Open junction and some grain boundaries suggest former presence of fluid before foil preparation. Inset is the X-ray diffraction pattern from fault zone materials, indicating multiple phases with random crystallographic orientations.



Specimen GL476, with 8 vol% of antigorite, was conducted at a deformation rate of 3.94×10^{-5} /s to investigate the effect of strain rate on dehydration embrittlement. The sample shows faulting (Figure 2.14a) similar to that observed in the faster experiments. However, because of the much slower deformation rate, dehydration occurred over a longer time period (~5 hours) than other experiments (~1 hour). Fault zone does not appear to contain any gouge. At higher magnification (Figure 2.14b), we observed very limited amounts of antigorite remaining because it had reacted almost completely away and was replaced by the dehydration products with a spinifex-like texture, similar to that reported in nature by Trommsdorff et al., (1998).

To approach the lower bound of serpentine content that is required to produce faulting, we conducted another experiment containing only ~4 vol% of serpentine (GB513, Figure 2.15). The specimen was macroscopically ductile and showing a tiny incipient fault at the corner (Figure 2.15a) which might be responsible for the joggling of stress vs strain curve after yielding (Figure 2.6). Antigorite was under dehydration (Figure 2.15b), but there is too little H₂O released in this case, to produce a throughgoing fault in the specimen. We, thus, suggest that the dehydration embrittlement in serpentinized peridotite ceases at a sample composition of ~4 vol% serpentine.

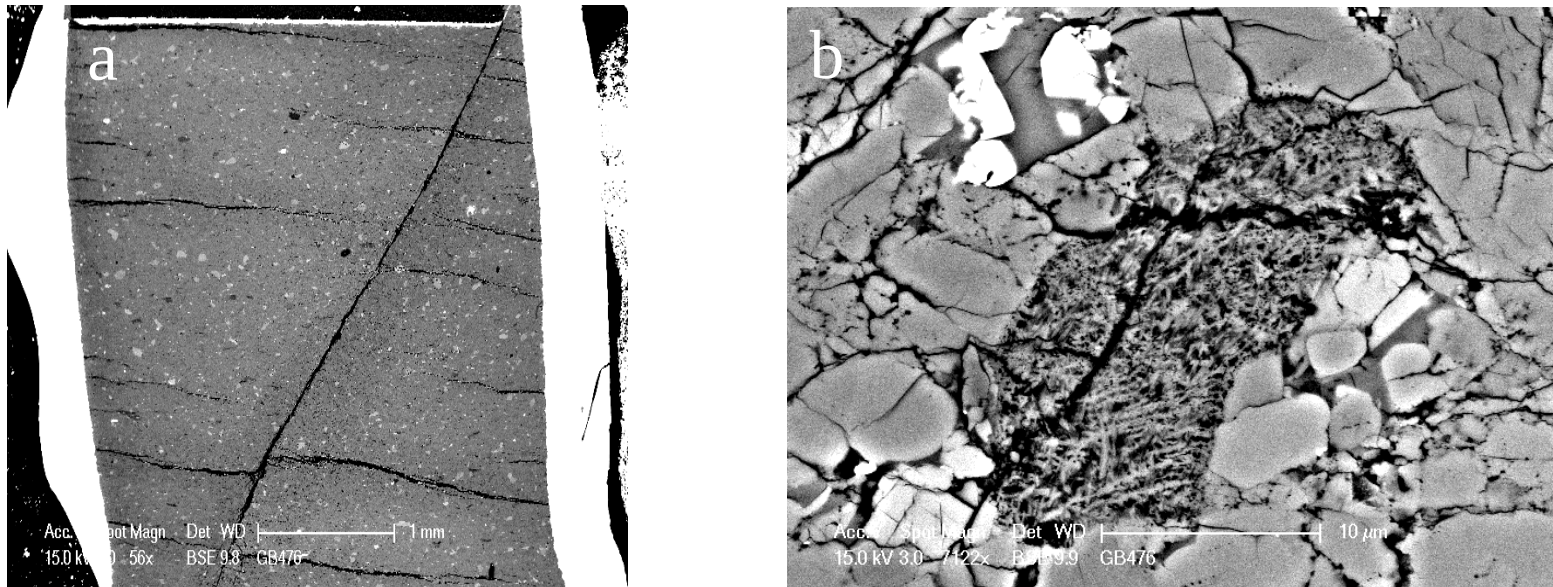


Figure 2.14 SEM (BSE) images of faulting in GB476. (a) The fault cut through the sample at $\sim 30^\circ$ to the maximum compression direction. (b) Spinifex-like structure in the fully dehydrated antigorite.

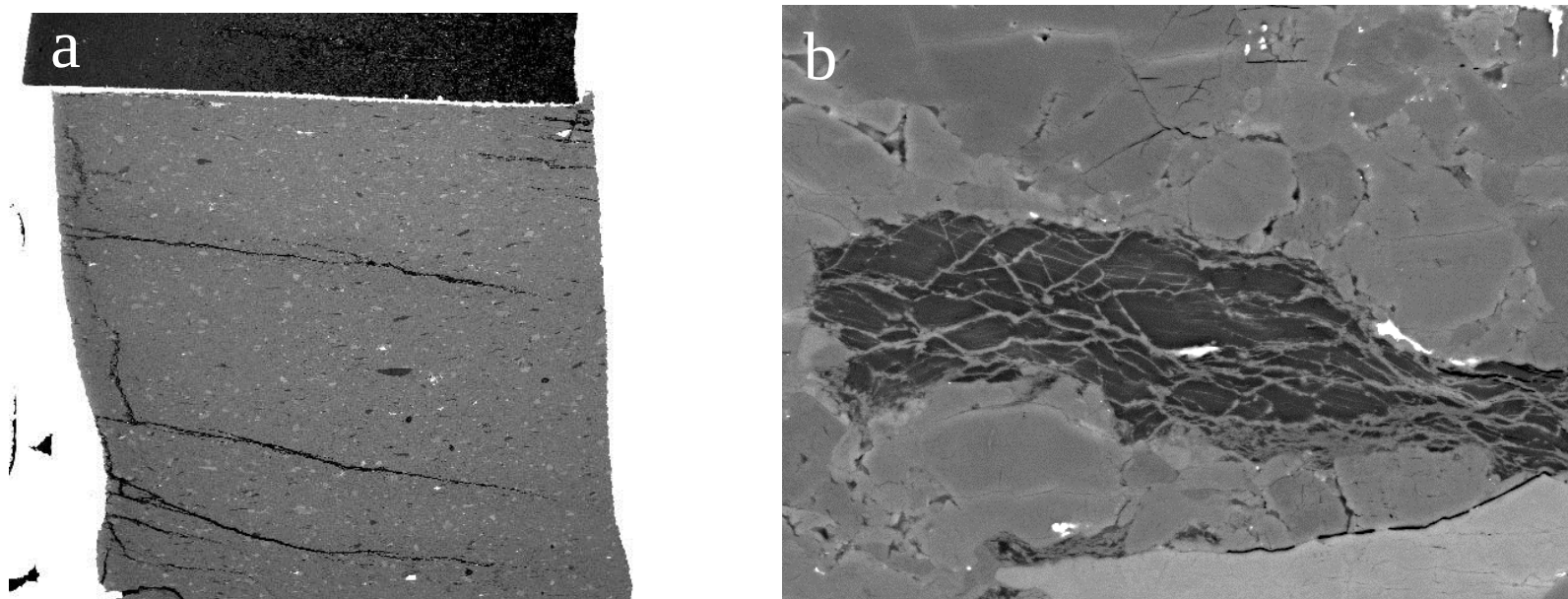


Figure 2.15 SEM (BSE) images of GB513. (a) Flow of this specimen was approximately homogeneous but a tiny, incipient, fault is visible in the upper right corner. (b) Dehydration is observed in individual antigorite domains, with porosity induced along grain boundaries of the adjacent harzburgite .

In addition to fluid inclusion trails mentioned briefly above (Figure 2.11b), evidence of fluid inclusion trails was wide-spread in all specimens containing enough coarse-grained olivine to allow them to be seen (Figure 2.16). Everywhere, healed cracks and fluid inclusion trails are approximately parallel to the plane of least compression (parallel to σ_1). The starting material shows no fluid inclusion trails, hence their presence here records the formation of Mode I microcracks and their healing before the end of the experiment that occurred only a few minutes later. These healed cracks and also those that remained open at the end of the experiment also display braches (smaller cracks), radiating from them. Compared to the solid reaction products that remain where they formed, fluid inclusion trails and cracks initiated from antigorite and propagated into relict olivine grains, demonstrating that separation of solid and fluid reaction products is very fast after dehydration (Jung et al., 2004).

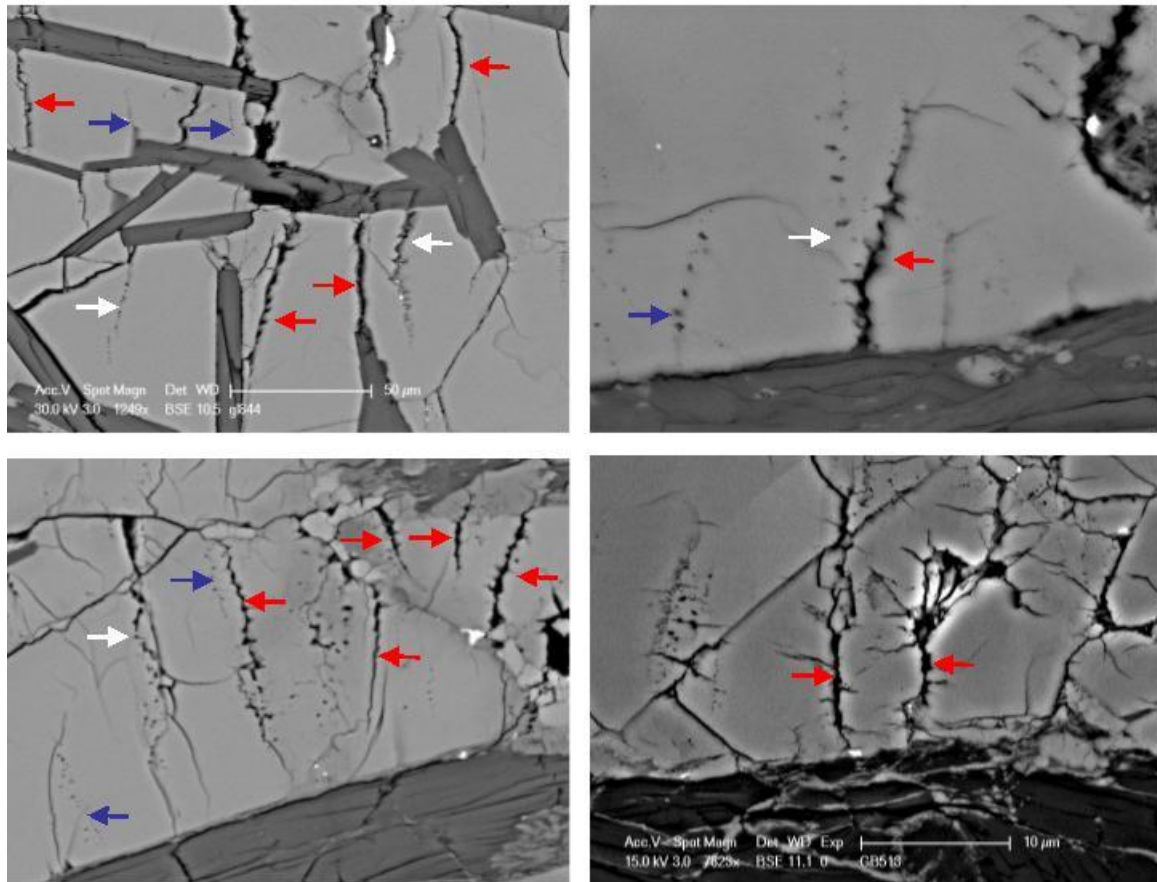


Figure 2.16 SEM (BSE) images of microcracks and fluid inclusion (bubble) trails in dehydrated material. Note that cracks and trails begin at antigorite crystals and run approximately parallel to maximum compression direction (N-S in the images). Red arrows delineate open cracks and blue arrows mark healed ones (bubble trails), white arrows mark some others showing partial healing at their tips.

These experiments were conducted at 1 GPa confining pressure, so normal brittle fracture phenomena are not fully precluded, as evidenced by the broken olivine fragments found in fault zones. Therefore, to examine the effect of fluid and to exclude the possibility that the faulting I observe was a result of fully brittle behavior instead of dehydration embrittlement, I conducted a couple of experiments using pure harzburgite under the same conditions as the antigorite experiments were performed. The samples were stronger than all of the serpentine-bearing samples except for GB513, yet they ended up flowing rather than faulting (Figure 2.17).

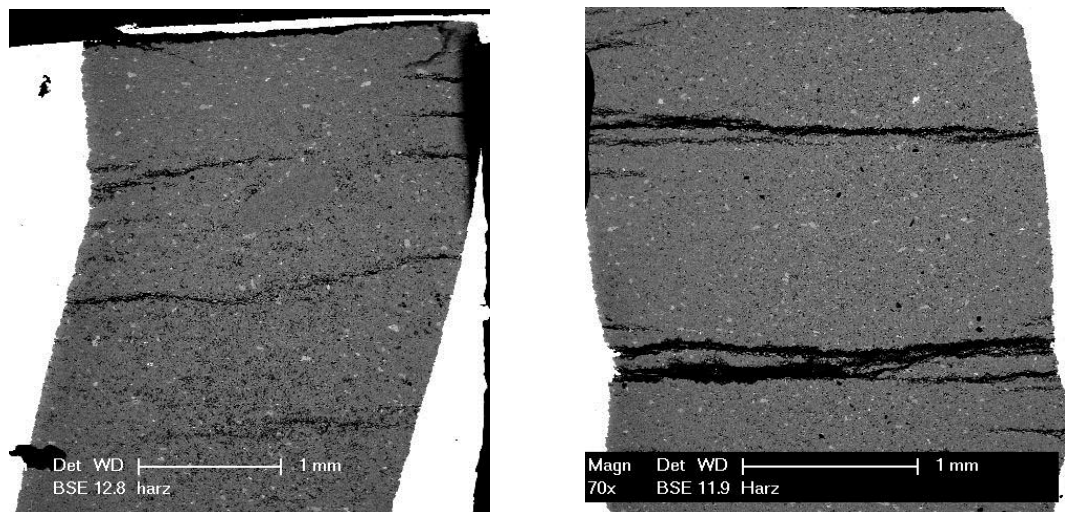


Figure 2.17 SEM (BSE) images of ductile harzburgite specimens. GB478, mixed powder specimen, on left, and GL842, cored specimen, on right.

GL495, a specimen fabricated from powder and composed of 76 vol% antigorite, is an experiment designed to extend the investigation at the antigorite-rich end of the composition range. It can be seen (Figure 2.18) that antigorite grains are surrounded by thin white lines, which consist of dehydration products. This specimen was fully ductile, dehydration enhanced its plastic deformation rather than inducing failure.

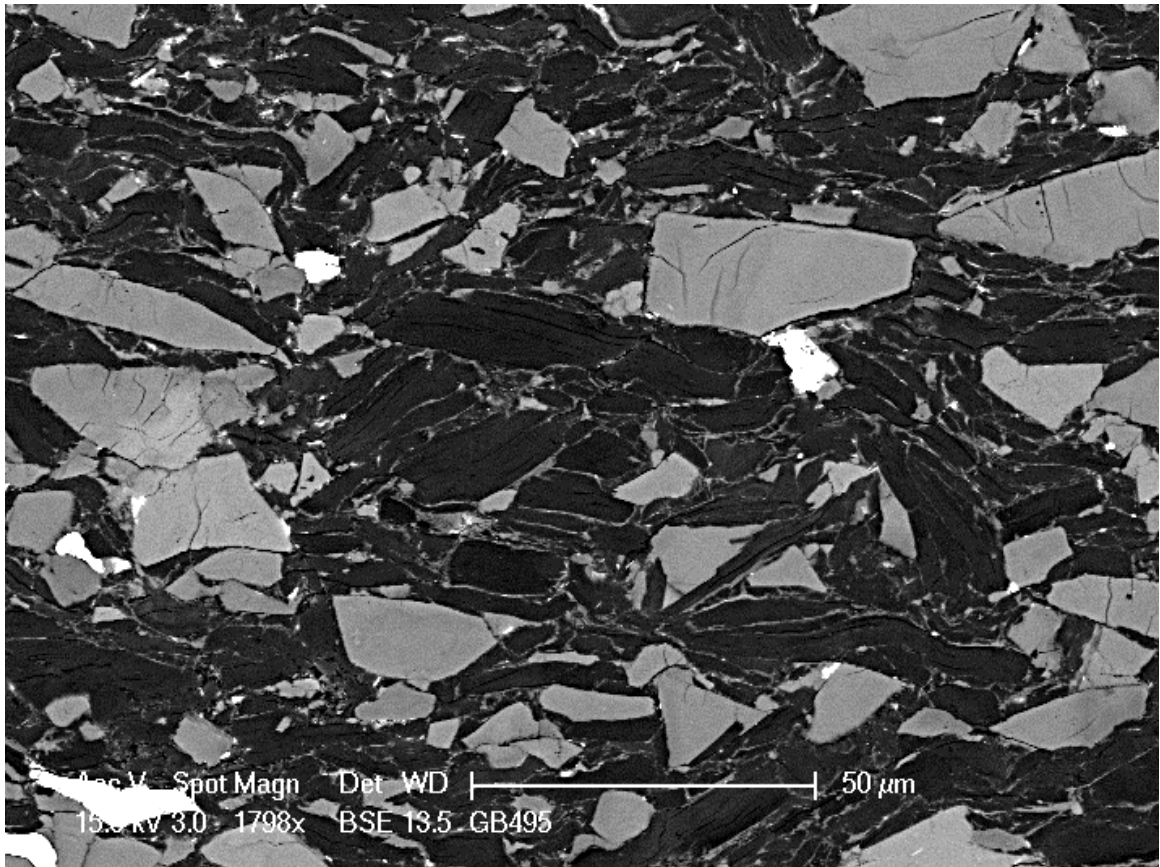


Figure 2.18 SEM (BSE) image of ductile sample experiment (GL495, 76 vol% serpentinite). Dehydration reaction products (bright) can be observed along antigorite grain boundaries (dark). Light gray phase is olivine; bright white phase is minor magnetite.

To further investigate the dehydration embrittlement phenomenon, I conducted an additional experiment at 2.7 GPa and 720 °C (GB493, ~60 vol % serpentine content), where unassisted brittle failure would be extremely unlikely and where the slope of the dehydration reaction is negative, implying that the volume change of the reaction is also negative. Given that these specimens are very strong, performing this experiment was quite challenging. Because the strength would be very high at 2.8 GPa and 720 °C, there was a significant probability that the stress on the tungsten carbide (WC) load piston could reach the fracture strength, resulting in catastrophic failure. To control the stress on the deformation piston safely below its failure limit, I used a sample of smaller diameter (~2.55 mm) and placed a sleeve of palladium around it to fill out the specimen capsule. Therefore, the same level of stress on the WC piston would generate a larger stress on the sample because of its reduced diameter. The specimen was prepared as for lower-pressure powder experiments; the original antigorite serpentinite was crushed, mixed and encapsulated. The experiment was successful; at the end of deformation, the specimen failed just below the stress limit of the piston. Faulting and dehydration products were observed in the specimen by post-experiment SEM imaging, revealing similar microstructures as those produced at lower pressures (Figure 2.19).

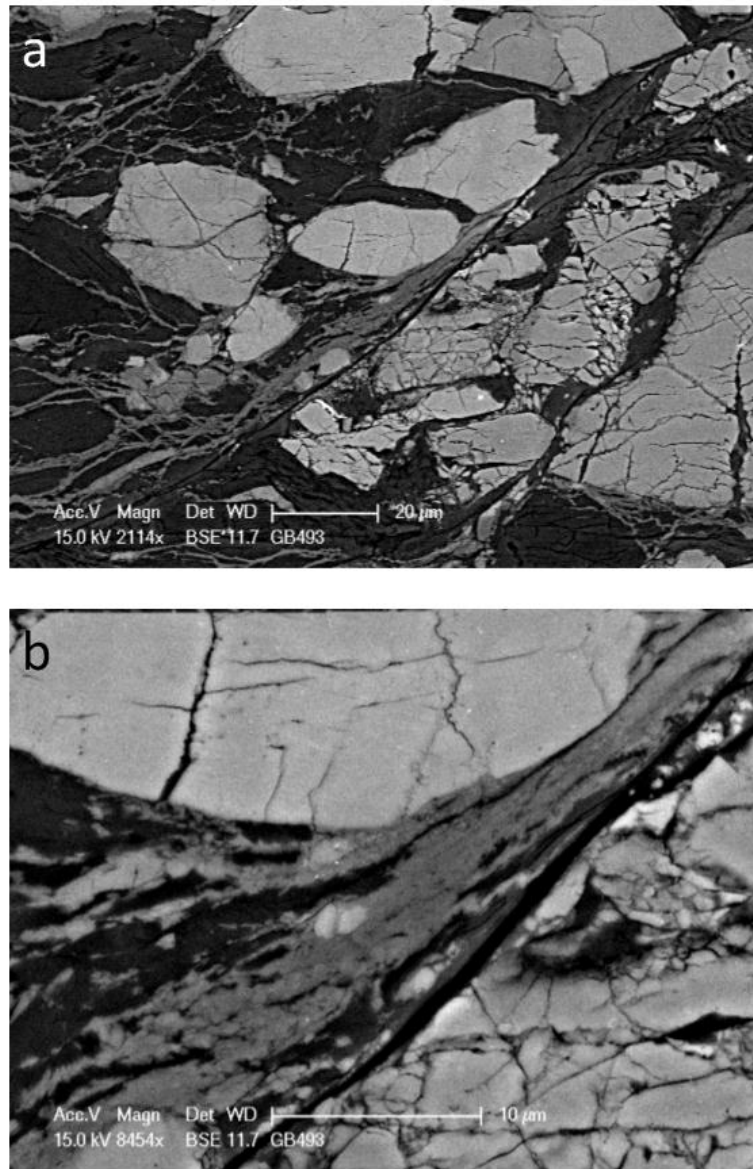


Figure 2.19 SEM (BSE) images of faulted experiment GB493. (a) Fault cutting through the sample is highlighted by the concentrated dehydration products and broken fragments in the fault zone. (b) Nanometric dehydration products in the fault zone are similar to those observed at lower pressure.

This finding is consistent with previous work from our laboratory (Jung et al., 2004; Jung and Green, 2004) that was also confirmed by acoustic emissions during dehydration (Jung et al., 2009). As previously, our faulting microstructures are closely similar at all pressures, even though here we have observed the faulting in a much wider composition range. We, thus, confirm that the dehydration embrittlement mechanism is working even under conditions where the total volume change of the dehydration reaction is negative.

2.5 Conclusion

Over the last several years, experimental as well as numerical studies from different groups have reached contrary conclusions about whether or not serpentinite fails by ductile or brittle processes (e.g. Jung et al., 2004; Jung et al., 2009; Hirth and Kelemen, 2007; Chernak and Hirth, 2011). Results from my experiments provide an answer to this dilemma, showing that dehydration-induced faulting occurs in a wide range of compositions but not in nearly pure serpentinite nor nearly pure peridotite (Xia et al., 2012). Close to the peridotite end (GB513), there is too little H₂O being released during dehydration to produce bulk faulting. Similarly, as one looks at specimens of increasing proportions of serpentine, specimens also become ductile. However, this is not because more fluid is released but because the composition reaches sufficient antigorite so that

relict peridotite mineralogy is dispersed in the serpentinite matrix (GB495). Under such conditions, antigorite rheology becomes the dominant factor in bulk rheology, the rock is further weakened by dehydration but the material flows rather than fractures. The amount of serpentine required to trigger the instability, in my study, is very limited, as low as ~8 vol% at 1GPa, 993K and 10^{-4}s^{-1} .

Microanalysis from SEM and TEM strongly supported that both fluid release and the solid dehydration products (nanometric olivine and talc) were involved in the weakening process and fault formation. However, it is still not clear whether the fault is weakened by the super-fine-grained materials or the fault itself promoted the super-fine-grained materials within the fault zone.

2.6 Geophysical Implications

We observed, from SEM images, that in most specimens antigorite was at very early stage of dehydration; only less than 5 vol% of antigorite was decomposed to olivine, talc and water. This observation, combined with the fact that as little as 8 vol% antigorite within the specimen was sufficient to induce faulting, indicates that less than 1 vol% of water, released during dehydration, was enough to produce instability. This result is consistent with that demonstrated by Zhang et al. (2004) who showed that less than 0.1 vol% fluid was sufficient to induce faulting and that whether or not specimens failed

could be controlled by manipulating the flow stress to be above or below the fracture stress. Based on this result, we also suggest that faulting by this mechanism would still occur even if hydration of oceanic mantle lithosphere is very limited, which is consistent with the current consensus that there is not an apparent conduit for ocean water to percolate down deep and extensively hydrate mantle peridotite. Perhaps partial hydration of the deep portions of surficial faults produced during slab bending could be the answer, although most such faults would be quasi-vertical in subducting lithosphere and it has been shown by Warren et al. (2007) and Kiser et al. (2011) that intermediate-depth earthquakes occur on quasi-horizontal faults. Indeed, our results also suggest a way out of this latter conundrum. If the abundant trenchward-dipping faults in subducting slabs are sufficiently serpentized to enable them to slip silently and at the same time small amounts of serpentine form on grain boundaries of bulk mantle harzburgite, then the partially-altered harzburgite could fail during dehydration yielding earthquakes on new faults in the conjugate orientation (subhorizontal).

Moreover, we suggest that the results of Chernak and Hirth (2011) may very well explain the aseismic creep along the plate interface if that interface is cold enough to stabilize antigorite as fluid evolves from below as the altered crust and/or mantle of the down-going slab dehydrates. Should that serpentine reach its dehydration point, the

fluid released would enhance ductility as proposed by Jung and Green (2005) and demonstrated by Chernak and Hirth (2011).

2.7 Future Perspective

Most of experiments in this study are accomplished at 1 GPa. As it has been proposed that the dehydration embrittlement of serpentine can still function at elevated pressure conditions even when the total volume change of dehydration reaction becomes negative, therefore It would be important to further investigate the pattern of dehydration embrittlement of serpentinized peridotite at elevated pressure conditions, corresponding to the deeper range of intermediate-depth earthquakes. Another series of deformation experiments, similar to that accomplished at 1GPa, could be carried out at 3GPa and 6GPa in the Deformation-DIA apparatus installed at a synchrotron X-ray radiation source, using acoustic emission monitoring, which would allow capture of faulting "in situ". Subsequent tomographic imaging of the 3-D specimen geometry and electron microscopy would confirm fault configurations.

Chapter 3.

Effect of Pressure on Creep of Olivine

3.1 Abstract

Rheological properties of mantle minerals are critical for understanding the dynamics of Earth's deep interior. Polycrystalline olivine, which makes up more than 60 vol% of mantle portion of oceanic lithosphere, predominantly represents the rheology of this region. Nevertheless, previous experimental studies carried out at varied pressures, measuring the rheological properties of olivine at high temperature, produce bimodal results. The activation volume (ΔV^*) for creep of olivine reported from Griggs and Paterson apparatus at relatively lower pressures is $\sim 15 \text{ cm}^3/\text{mol}$ or even higher, while higher pressure studies from D-DIA apparatus report a ΔV^* near zero. In order to address this issue and provide a standard for strength comparison between different apparatus, we have conducted a series of experiments on a synthetic polycrystalline forsterite, the Mg end member of olivine, in a modified Griggs apparatus with molten or near-molten CsCl pressure media that is capable of high-quality measurement of stress during deformation experiments under confining pressures up to $\geq 3 \text{ GPa}$. Our results show a tightly constrained ΔV^* of $12 \pm 2 \text{ cm}^3/\text{mol}$, which is similar to that for Mn_2GeO_4 olivine measured over the range of 0.4 - 4.0 GPa (Bai and Green, 1998), demonstrating a significant pressure dependence of creep in nominally dry olivine at pressure lower than 3 GPa in the dislocation creep field.

3.2 Introduction

Olivine is stable over a wide pressure range, from that at Earth's surface to approximately 15 GPa, corresponding to a depth of ~410 km where it transforms to its higher pressure polymorph, wadsleyite. As the most abundant phase and the weakest phase in the upper mantle, the rheological properties of olivine control the dynamic processes of the upper mantle, including evolution of subduction zones (e.g. van Keken et al., 2002, Gerya and Stöckhert, 2002), Earth's deep seismicity (e.g. Green and Burnley, 1989; Green et al., 1990, Green and Houston, 1995), upper mantle seismic anisotropy (e.g. Kneller et al., 2005 & 2007, Mainprice et al., 2005; Jung et al., 2008), post-seismic mantle relaxation (e.g. Freed and Bürgmann, 2004) and seismic attenuation (e.g. Jackson et al., 2002). It has been known that the dominant deformation mechanism in olivine is different from low temperature to high temperature ($> \sim 900$ °C) (Carter and Ave'Lallemant, 1970; Nishihara et al., 2010; Raleigh, 1968; Tsenn and Carter, 1987;).

In this study, high temperature dislocation creep behavior of olivine in the upper mantle is described by the power-law equation:

$$\dot{\epsilon} = A \cdot \sigma^n \cdot (1 + f_{H_2O})^h \cdot \exp\left(-\frac{\Delta E^* + P\Delta V^*}{RT}\right) \quad (1)$$

where $\dot{\epsilon}$ is strain rate, A is material constant, σ is flow stress, n is stress exponent, h is the water fugacity exponent for creep, ΔE^* is activation energy, P is confining pressure,

ΔV^* is activation volume, R is gas constant, T is temperature. Among these parameters, the effect of pressure, represented by ΔV^* , is probably one of the most intrinsic issues that is still controversial. It is often argued that the strength of plastic flow material is relatively insensitive to pressure compared to that in brittle regime where brittle strength increases rapidly with pressure. However, the pressure range in Earth's mantle is so great that the ΔV^* of mantle rocks is very important for Earth's deep interior. Plastic deformation always involves overcoming the barrier for defect motion, which is expressed by the effect of pressure on free energy for defect motion, namely the activation volume ΔV^* (Karato, 2008). The larger the pressure, the higher energy for defect motion will be required. More importantly, the effect of pressure on fugacity of some chemical species, e.g. H_2O , will change the defect mobility with pressure. Borch and Green (1989) also suggested that water enhances the pressure dependence of creep in olivine, at least to pressure around 2 GPa. Therefore pressure dependence on plastic deformation can be quite complicated under conditions of Earth's deep interior.

The earliest attempt to investigate the pressure dependence of creep in olivine can be traced back to 50 years ago when Griggs et al. (1960) tried to test olivine-bearing rocks under pressure. Currently the highest quality data on plastic deformation are obtained by using the Paterson apparatus with gas pressure medium and internal load-cell (Paterson, 1990). Experimental studies using Paterson apparatus with

well-controlled chemical environment have produced very important and accurate results on plastic deformation of olivine (e.g., Mei and Kohlstedt, 2000). Despite the high accuracy of stress measurement from Paterson apparatus, this apparatus can be operated only at pressures up to 0.5 GPa, hence rheological properties under most upper mantle condition have been inferred from results under low pressure condition, but this kind of extrapolation could be wrong. Taking ΔV^* as an example, it has been argued that the effect of pressure on creep of minerals may itself be a function of pressure (Borch and Green, 1989) rather than a constant. It becomes a more complicated case when water is taken into account because it is known that water has a significant effect on rheological properties of silicates, and the amount of water dissolved in a mineral depends on the fugacity of water which increases as a function of pressure (Karato, 2009). Bai and Green (1998) conducted experiments on $\alpha\text{-Mn}_2\text{GeO}_4$ (olivine structure) and their results showed that the strengthening effect of pressure was counteracted by the water-weakening effect, and the water weakening effect increases with pressure because of higher H_2O fugacity. This observation may explain the small ΔV^* value measured from high pressure studies (e.g. Li et al., 2006) as they used water-bearing materials for their pressure media. All of these factors will make the extrapolation questionable. Therefore, despite its accuracy in stress measurement, the Paterson apparatus is limited by its limitation to low pressures.

The original Griggs apparatus, a modified piston cylinder device, extended the experimental investigation of rock deformation up to ~1.8 GPa. Ross et al. (1979) carried out the first creep experiments on dunite (polycrystalline olivine) and reported $\Delta V^* \approx 13.4$ cm³/mol. Unfortunately, their results are suspect because of the low accuracy of data resulting from the very strong solid pressure medium that was available at that time; the friction between the solid pressure medium and both the sample and the deformation piston significantly compromised their stress measurements. Borch and Green (1989) solved this problem by using the molten salt assembly, although their samples may have contained sufficient water to interfere with their measurements (Hirth and Kohlstedt, 1996). They found their data to be inconsistent with a constant ΔV^* ; the implication of their data was that ΔV^* falls with increasing pressure. Karato and Jung (2003), using solid NaCl or KCl as pressure medium reported ΔV^* values of 14 ± 1 cm³/mol under dry conditions and 24 ± 2 cm³/mol under wet conditions. Their stress values were deduced from dislocation density; whether this method is appropriate for stress calculation without adjustment for pressure effects needs to be further examined.

With significant technical progress in the last few decades, deformation apparatus covering larger pressure ranges have been developed, including the Deformation-DIA (D-DIA) apparatus (Wang et al., 2003) and Rotational Drickamer apparatus (RDA) (Yamazaki and Karato, 2001). Li et al. (2004) reported a smaller ΔV^* (less than 10

cm³/mol) at 8 GPa and 1200°C. Multi-anvil apparatus were used in their studies, in which control and knowledge of the level and state of differential stress is difficult. Li et al., (2006) extended their study using *in situ* D-DIA experiments while measuring stress and strain with synchrotron X-ray radiation. They concluded that there is little or no effect of pressure on dislocation creep of olivine, yielding a ΔV^* of 0 ± 5 cm³/mol. Because they used natural rocks and water-bearing pressure media, the effect of water was not well characterized. Raterron et al., (2007) also reported a small ΔV^* value of less than 3 cm³/mol, but a small activation energy for "dry" olivine single crystal (100 kJ/mol compared to the well-known 400-500 kJ/mol), suggesting that Peierls mechanism, rather than dislocation creep may have dominated deformation in their experiments. Kawazoe et al. (2009) conducted shear deformation experiments on dry polycrystalline olivine using RDA. They obtained a ΔV^* of 15-20 cm³/mol. But their results have been questioned for the macroscopic stress derivation (Burnley et al., 2008).

3.3 Motivation of this study

Table 3.1 lists some results from previous experimental studies. In spite of substantial technical development during the last few decades, the high-pressure studies, using the D-DIA apparatus, have amplified the discrepancy instead of placing constraints on the earlier results. Reported ΔV^* values, ranging from 0 to 28 cm³/mol, can cause

Table 3.1 Selected results from previous studies

Apparatus	P (GPa)	T (K)	ΔV^* (cm ³ /mol)	Water content (ppm H/Si)	Reference
Griggs	0.6-2.1	1250-1700	not a constant	N/A	Borch and Green (1989)
Griggs and Paterson	0.4-3.8	1000-1200	10.6	dry	Bai and Green (1998)
Griggs	2.0	1573	14 ± 2	< 100	Karato & Jung (2003)
Multi-anvil	8	618-1780	<10	N/A	Li et al. (2004)
D-DIA	3.5-9.6	1073-1473	0 ± 5	570-3800	Li et al. (2006)
D-DIA	2.1-7.5	1373-1677	3 ± 0.5	<90	Raterron et al. (2007)
RDA	4.9-9.6	1300-1870	15 - 20	<50	Kawazoe et al. (2009)
D-DIA	2.8-7.8	1153-1670	N/A	>100	Hilairt et al. (2012)
D-DIA	3.5-8.5	1373-1673	12±4 for [100]/(010) 3±4 for [001]/(010)	32-700	Raterron et al. (2009)
D-DIA	> 5.7	~1675	15±3 for [100]/(010) 0±1.2 for [001]/(010)	36-138	Raterron et al. (2011)

Note: Green and Borch (1989) demonstrated that the ΔV^* is decreasing with increasing pressure.

a difference in effective viscosity of olivine of 10 orders of magnitude at the bottom of the upper mantle (~410 km) (Durham et al., 2009). Therefore, the effect of pressure on creep in olivine is, at best, poorly understood. Part of this problem involves different studies using different starting materials and part of it may well be due to apparatus calibration for the very high-pressure apparatus.

In addition, evidence has emerged that there is a potential correlation between the discrepancy of ΔV^* value and the transition of lattice preferred orientation (LPO) in olivine (Raterron et al., 2007 2009 & 2011, Hilairet et al., 2012). LPO (sometimes called CPO, crystallographic preferred orientation) records the crystallographic rotations induced by the active slip systems in materials flowing by dislocation creep. The pattern of LPO in a material is termed its “fabric” (sometimes “microfabric”) in the geological literature and is referred to as “texture” in the materials literature. It is very important microstructural characteristic by which one can determine the underlying physical process(es) of deformation or how a rock was formed in a certain environment. There are quite a few LPOs that have been documented for olivine, among which A-type LPO [100]/(010), a-slip, and B-type LPO [001]/(010), c-slip, could be responsible for the difference of ΔV^* value between low and high pressure studies. It has been proposed that a-slip is inhibited with increasing pressure, resulting in a large ΔV^* value, while c-slip, which is virtually insensitive to pressure, results in a small ΔV^* value (e.g., Raterron et al.,

2009). As single crystals were used in these studies, whether the result can be directly applied to Earth's mantle, where polycrystalline aggregates are present, is not yet established.

In order to examine the potential correlation between LPO and ΔV^* in olivine using a polycrystalline sample, as well as to obtain a robust ΔV^* value and simultaneously provide a standard for strength examination and comparison for various types of apparatus, we have conducted a series of experiments on synthetic polycrystalline forsterite with a confining pressure from 1 to 2.5 GPa to measure ΔV^* of high temperature dislocation creep in olivine.

3.4 Starting Materials and Experimental Description

3.4.1 Starting Materials

Olivine is both the weakest phase in the upper mantle and also the most abundant one, making up more than 60 vol% of the upper mantle at depths less than 410 km. Therefore, to a first order approximation, upper mantle rheology is that of olivine. In our study, we use iron-free forsterite (Mg_2SiO_4) plus a small excess of SiO_2 that results in ~ 3 vol% coexisting enstatite, MgSiO_3 , to buffer the point defect populations appropriately for comparison to the mantle. It has been shown that olivine has different point defect distributions depending on whether it is in equilibrium with enstatite (MgSiO_3) or with

periclase (MgO) and such differences can significantly affect the flow properties of olivine (Kohlstedt et al., 1995), so a little extra SiO₂ in the bulk materials can ensure that the defect populations in forsterite are in equilibrium with enstatite (as in the mantle), not periclase. Using forsterite, the Mg end member of the olivine series, undoubtedly somewhat overestimates the strength of mantle olivine (Mg_{0.9},Fe_{0.1})₂SiO₄. However, it also avoids the myriad problems that Fe can add to the experiments because of the sensitivity of Fe to the oxygen fugacity, meaning that it can change from Fe²⁺ to Fe³⁺, with a variety of corresponding difficulties. Our samples are essentially dry in that molten alkali halides are very strong “getters” for H₂O, hence any remaining grain-boundary H₂O is efficiently scavenged by the confining medium prior to deformation.

3.4.2 Sample Assembly and Experimental Procedure

Cylindrical Specimens, approximately 9mm long and 3 mm diameter were cored from a ~7cm diameter specimen that was prepared by commercial hot pressing at 1400 °C and 100 MPa. Both ends of each specimen were separately polished flat and square. Each specimen was preheated in the vacuum oven at 110 °C for ~12 hours to remove any interstitial or adsorbed molecular water before loading it into a cylindrical platinum capsule. Capsules consist of two “half-capsules” of different diameter, each with a blind end; one of which fits snugly over the other to make a closed capsule that cold welds

upon pressurization. A 25 μm thick Nickel foil was included within capsules at both top and bottom to buffer the oxygen fugacity at Ni/NiO. The specimens, within their Pt capsules, were then cast into molten salt pressure media as appropriate (CsCl or NaCl). A standard sample assembly would be put together and seated into our modified 4 GPa Griggs apparatus.

In our early experiments, specimens were first pressurized at room temperature to $\sim 280\text{MPa}$, the temperature was then ramped to 300°C , followed by resumed pressurization to target values. The temperature was then increased to 1200°C , followed by 1 hour annealing at target pressure and temperature. Deformation experiments were conducted at strain rates of 10^{-5} - $10^{-4}/\text{s}$ and a temperature of 1200°C . Molten alkali halides are very strong “getters” for H_2O and hydrogen can diffuse through Pt at elevated temperatures, hence any remaining grain-boundary H_2O is efficiently scavenged by the confining medium prior to deformation. The small amount of oxygen generated by this loss of H as well as any residual oxygen trapped within the capsules during assembly will react with the Ni foils at the ends of the specimens, thereby buffering the experiments at the Ni-NiO oxygen buffer.

3.5 Results and Discussion

3.5.1 Initial Results and Discussion

In an effort to maximize the efficiency of each experiment, we carried out two cycles of deformation on each specimen at two strain rates, $10^{-5}/s$ and $10^{-4}/s$, respectively and 1200 °C. Mechanical results of selected experiments are summarized in Table 2.

Table 3.2 A summary of experimental conditions and mechanical results

Run #	P (MPa)	$\dot{\epsilon}(10^{-5}/s)$	σ (MPa)	$\dot{\epsilon}(10^{-4}/s)$	σ (MPa)
GL787	1000	2.3	230	2.7	520
GL797	1500	2.5	290	2.9	670
GB353	2000	2.1	550	2.2	1000
GB355	2500	2.2	920	2.3	1470
GB352	3000	2.2	1390	N/A	N/A
$\dot{\epsilon}$ (s^{-1})		2.3×10^{-5}		2.5×10^{-4}	
ΔV^* (cm^3/mol)		36.7		25.7	

$\dot{\epsilon}$: strain rate; σ : strength. GL- experiments were conducted in Rig-2 below 1.8 GPa; GB- experiments were conducted in Rig-3 above 2 GPa.

As I discussed above, olivine's high-temperature creep behavior can be best described by the power law empirical equation (1). Because the effect of pressure on dislocation creep is represented by ΔV^* , we made the following transformation to equation (1):

$$\dot{\epsilon} = A \cdot \sigma^n \cdot (1 + f_{H_2O})^h \cdot \exp\left(-\frac{\Delta E^* + P\Delta V^*}{RT}\right) \quad (1)$$

$$\longrightarrow \quad \ln \dot{\epsilon} = \ln A + n \ln \sigma - \frac{\Delta E^* + P\Delta V^*}{RT}$$

$$\longrightarrow \quad \ln \sigma = \left(\frac{\Delta V^*}{nRT}\right)P + \frac{\Delta E^* + RT(\ln \dot{\epsilon} - \ln A)}{nRT} \quad (2)$$

$$\longrightarrow \quad \frac{\Delta V^*}{nRT} = \frac{\partial \ln \sigma}{\partial P} \quad (3)$$

We use dry samples in this study. Therefore, equation (1) transforms to equation (2) by taking the natural logarithm of both sides and rearranging terms. Equation (3) is obtained by taking the partial derivative with respect to pressure (P) of both sides of equation (2). In this study, T = 1473 K, R = 8.31 J/K, and we take n = 3 (Mei and Kohlstedt, 2000) for dislocation creep. ΔV^* can be derived from the slope of the plot of natural log of stress versus pressure for our data (Figure 3.2). From this relationship, we derive $\Delta V^* = 25.7 \text{ cm}^3/\text{mol}$ at a strain rate of $2.5 \times 10^{-4}/\text{s}$ and $\Delta V^* = 36.7 \text{ cm}^3/\text{mol}$ at a strain rate of $2.3 \times 10^{-5}/\text{s}$, respectively.

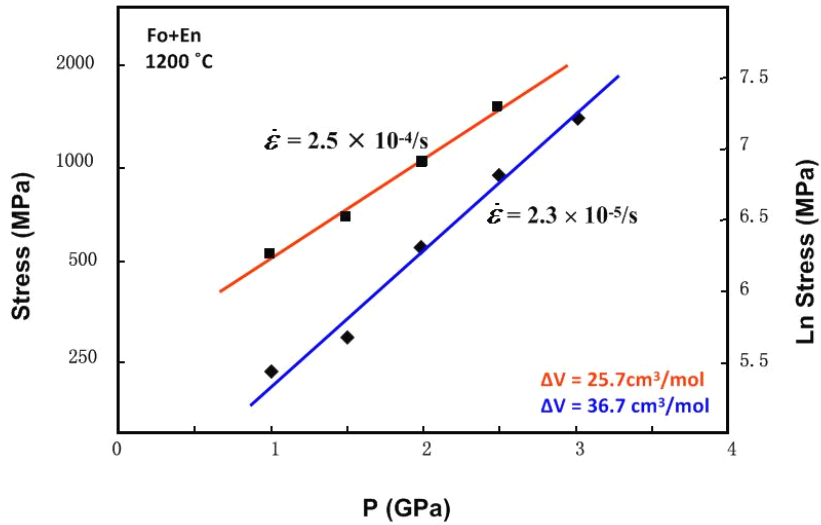


Figure 3.2 Initial Stress/Ln stress versus pressure plots. Red line shows the result from a strain rate of $2.5 \times 10^{-4}/s$, blue line is the result from a strain rate of $2.3 \times 10^{-5}/s$.

From the extended discussion in the Introduction, these results are clearly suspect. The former value is as large as anything previously determined (Borch and Green, 1987; Green and Borch, 1987) and the latter value is almost 50% larger than that. Moreover, despite the internal consistency of the data, the flow stresses of our specimens are much lower than were obtained during preliminary results performed by others (HWG, personal communication).

Because of these anomalies, we turned back to check the procedures and specimens of our experiments to seek the answers for why the pressure dependence of creep is so great (yielding such large ΔV^* values) and why the data from the two different strain rates yield different values. Firstly, it was noticed that some of our specimens experienced stress drops before getting to the steady state (Figure 3.3). Microscopic re-examination of samples revealed that these specimens had sustained brittle damage,

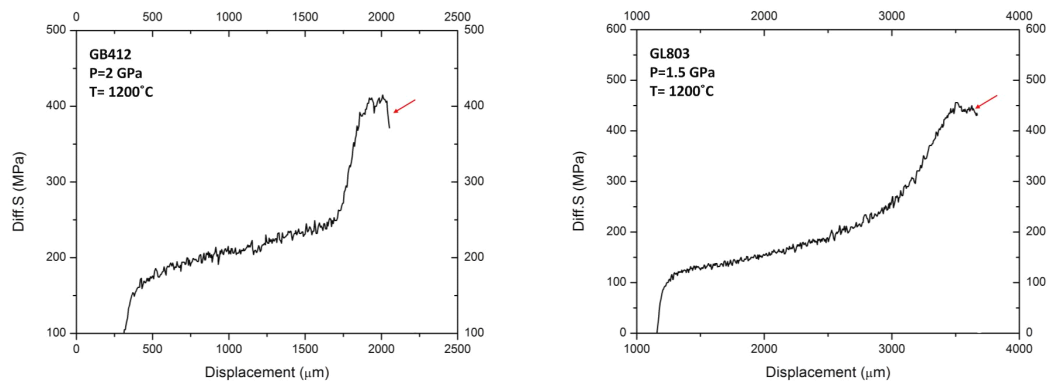


Figure 3.3 Stress drop before getting to steady state, pointed out by red arrows.

commonly represented by cracks running through some specimens at very high σ_1 and cataclastic zones in some other specimens (Figure 3.4). Riggs and Green (2005) pointed out that cored specimens can be damaged at during the early stage of pressurization, prior to initial heating. If this should be the case, even if the sample is not cracked, the

cataclastic accommodated flow can significantly reduce the flow strength of the sample. Early damage is likely to be partially repaired by void closer during further pressurization, hence, although all specimens will be weakened, the effect should be worst at lower pressure. As a consequence, not only will strengths be reduced but they will be reduced most at low pressure, resulting in abnormally high ΔV^* values.

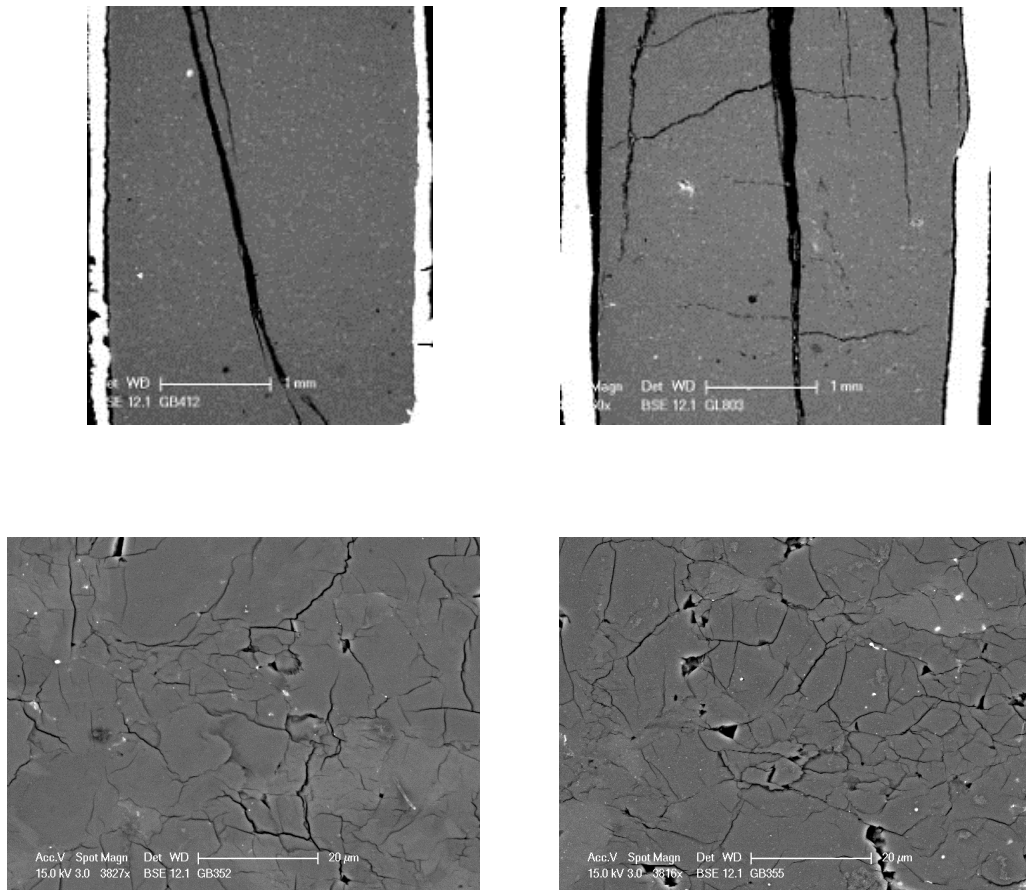


Figure 3.4 SEM (BSE) images of high angle cracks in specimens (Upper images) and cataclastic zones in specimens (Lower images).

3.5.2 Modified Procedure and Results

In order to rectify these problems, I started over this series of experiments with revised experimental procedures developed by Riggs and Green (2005). Brittle damage was avoided by much more gentle pressurization. In our sample assembly, void space is purposefully left between the upper graphite disk and the carbon furnace to protect the furnace from damage while going to pressure (see discussion in Chapter 1). However, a side effect of that procedure is that until the graphite disk and the furnace make contact, the temperature cannot be raised. Therefore, to enable sample heating at a lower pressure to avoid brittle damage, we shortened the top salt ring of the assembly (see Figure 1.4) by 0.03" (0.76mm) to allow contact between graphite cap and furnace at a lower pressure and thereby enable initiation of heating earlier. Previously, cold compression to ~ 200 MPa was required to establish furnace contact and initiate heating. With this small change, early stage heating could begin at a pressure of 40 MPa. In addition, because the sample material of this study is very refractory, we accomplish the remainder of pressurization very slowly (~ 1 MPa/min) at an elevated temperature of 527 °C. As a consequence, the modified procedure is very beneficial overall; it allows early softening of assembly parts which efficaciously avoids brittle damage to the specimen and to the furnace.

In addition, in the new series of experiments, we carried out only one cycle of deformation for each specimen to minimize the possibility of specimen damage during back and forth of the deformation piston at high temperature. CsCl and NaCl salt pressure media were used for experiments below 1.5 GPa and above 2 GPa, respectively. Using these two salts in this way, we were able to have the confining medium molten or very close to the melting temperature (and therefore very weak) in all experiments, maximizing the accuracy of stress measurement at all pressures to obtain a better-constrained ΔV^* value. We changed salts at 2 GPa because the molten salt cell works best when the salt around the specimen is molten but that otherwise the salt is solid, stabilizing the assembly.

With these changes in place, we repeated our previous experiments, focusing on a strain rate of 10^{-4}s^{-1} . Typical force-time curves (expressed as stress on the piston vs. displacement) are shown in Figure 3.5. Red arrows point out the abrupt steepening of the force curve when the Al_2O_3 piston encounters the specimen. Note that the force record prior to the “hit point” is essentially horizontal, indicating that the friction on the piston is constant as it advances through the low-viscosity molten salt and abruptly “hits” the specimen (Borch and Green, 1989). As a result of the friction not being time dependent, the force difference between the hit point and the flow stress is unambiguously an accurate measurement of the stress applied to the specimen.

Using the modified procedure, we conducted experiments covering a pressure range from 1 GPa to 2.5 GPa. To ensure the highest-possible quality data, experiments were considered a success only if both thermocouples survived the experiment and if the recorded temperature difference between them remained within 20°C, indicating that the temperature was very close to homogeneous. The latter is one of the most critical parameters for rheological deformation experiments. Stress- strain curves for these experiments is shown in Figure 3.6. The total strain in these experiments was up to 30%, indicating that all specimens went well into steady-state plastic deformation. Plotting these data as a function of pressure yielded a linear relationship between the natural log of the flow stress and pressure (Figure 3.7), and a well-constrained ΔV^* of 11cm³/mol. This result falls into the range of previously reported results and is essentially identical to the results on dry Mn₂GeO₄ olivine by Bai and Green (1998). Most importantly, the early heating procedure protected the specimen and the furnace from brittle damage during pressurization, with the result that the measured strengths of specimens are much stronger and are comparable with results from earlier preliminary results.

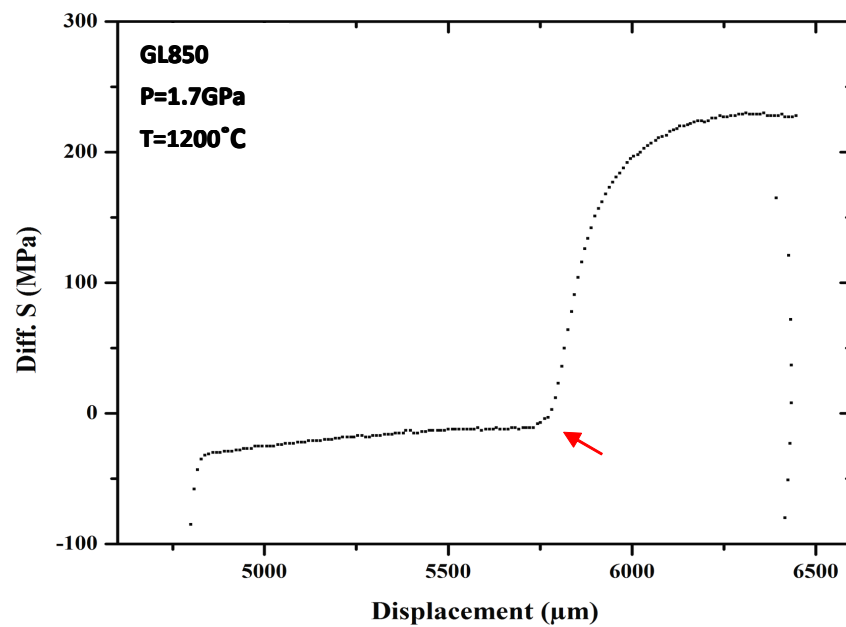
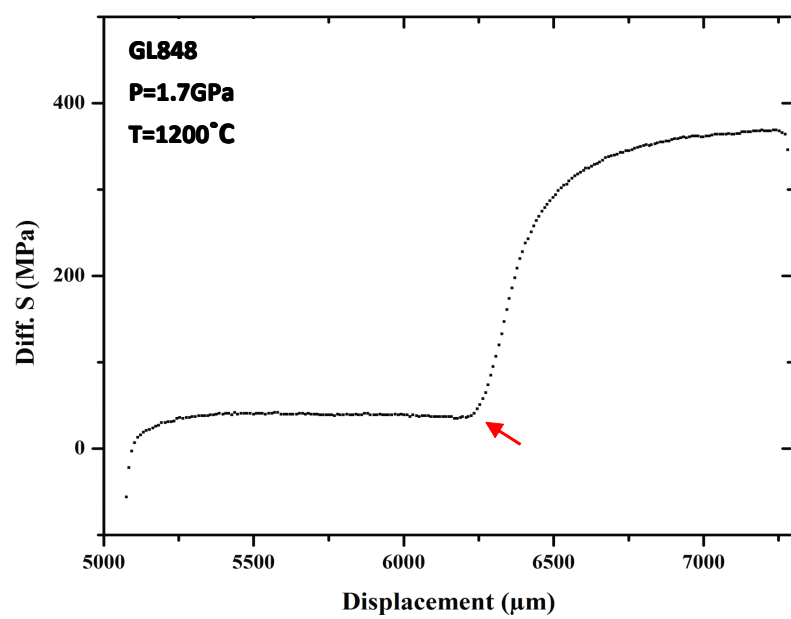


Figure 3.5 Typical differential stress versus displacements curves. Sharp hit-points are pointed out by red arrows.

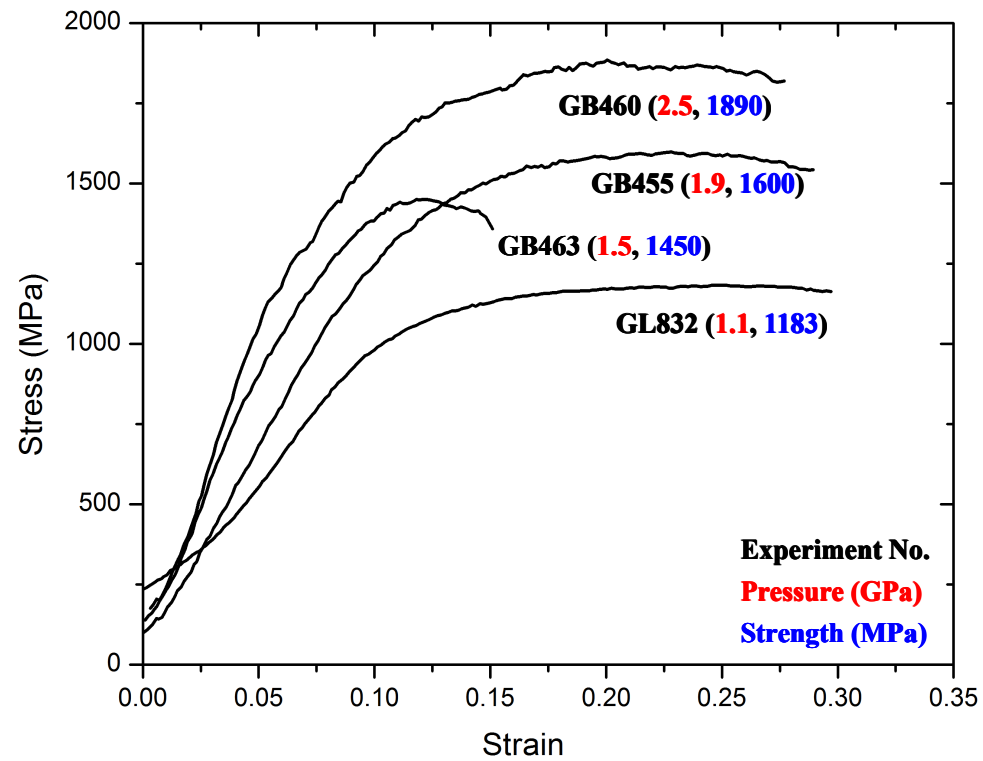


Figure 3.6 Stress versus strain curves. The stress drop GB463 is caused by falling over of the specimen at the end of deformation.

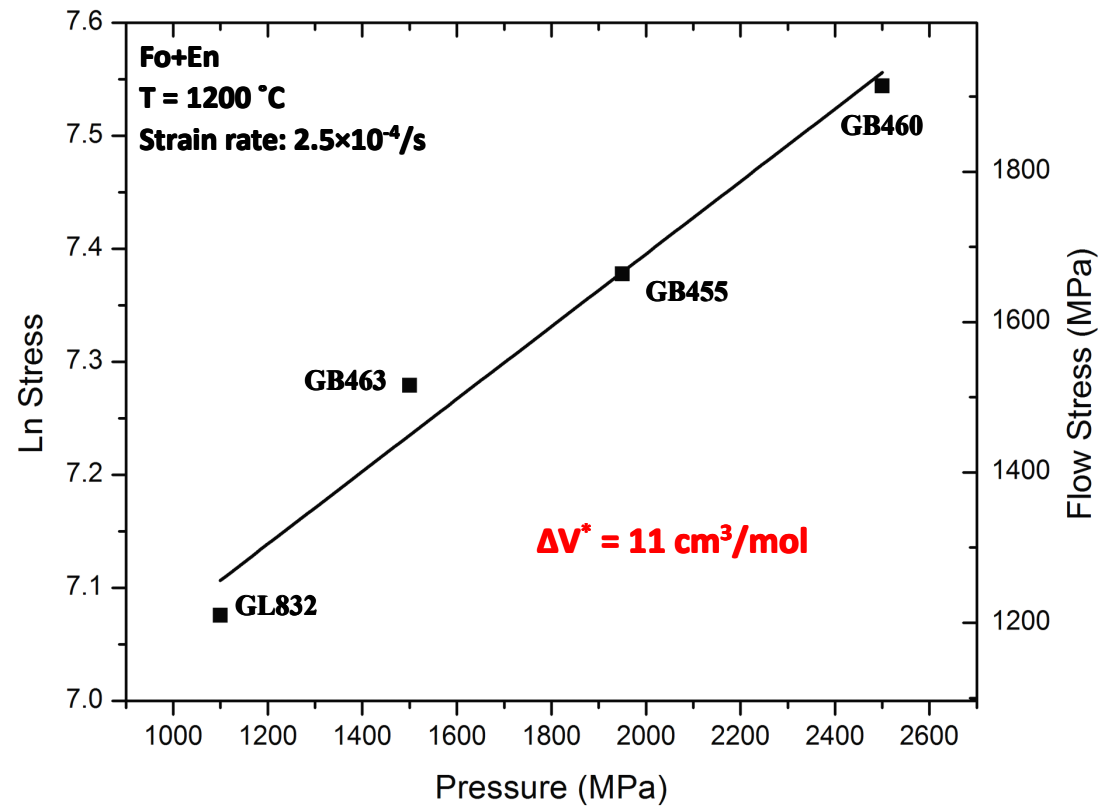


Figure 3.7 Stress/Ln stress versus pressure plot at a strain rate of $10^{-4}/s$. ΔV^* was derived from the slope of the curve.

In order to confirm the ΔV^* value as well as to examine the strain rate effect on ΔV^* , we carried out a few experiments at strain rates of $10^{-5}/s$ and a temperature of $1200\text{ }^{\circ}\text{C}$. The stress versus strain curves are shown in Figure 3.8. These curves were smoothed by polynomial fitting to minimize the disturbance from mechanical noise in the apparatus due to a noisy control valve on the hydraulic drive. As we found for $10^{-4}s^{-1}$ experiments, measured strengths are higher than those in the initial series of experiments. Again, there is a linear relationship between the natural log of stress and pressure (Figure 3.9), but in this case resulting in a ΔV^* value of $14.7\text{ cm}^3/\text{mol}$. Given the noisy data at a strain rate of $10^{-5}/s$ and the less-abrupt hit points in the measurements, we do not consider this slightly larger ΔV^* value to be significantly different from the value obtained at $10^{-4}/s$. We believe that our results are best represented by a value of $12 \pm 2\text{ cm}^3/\text{mol}$.

Based on the presentation above and the rigorous control of these experiments, we conclude that $\Delta V^* = 12 \pm 2\text{ cm}^3/\text{mol}$ is a robust value for creep of dry olivine at $P < 3\text{ GPa}$.

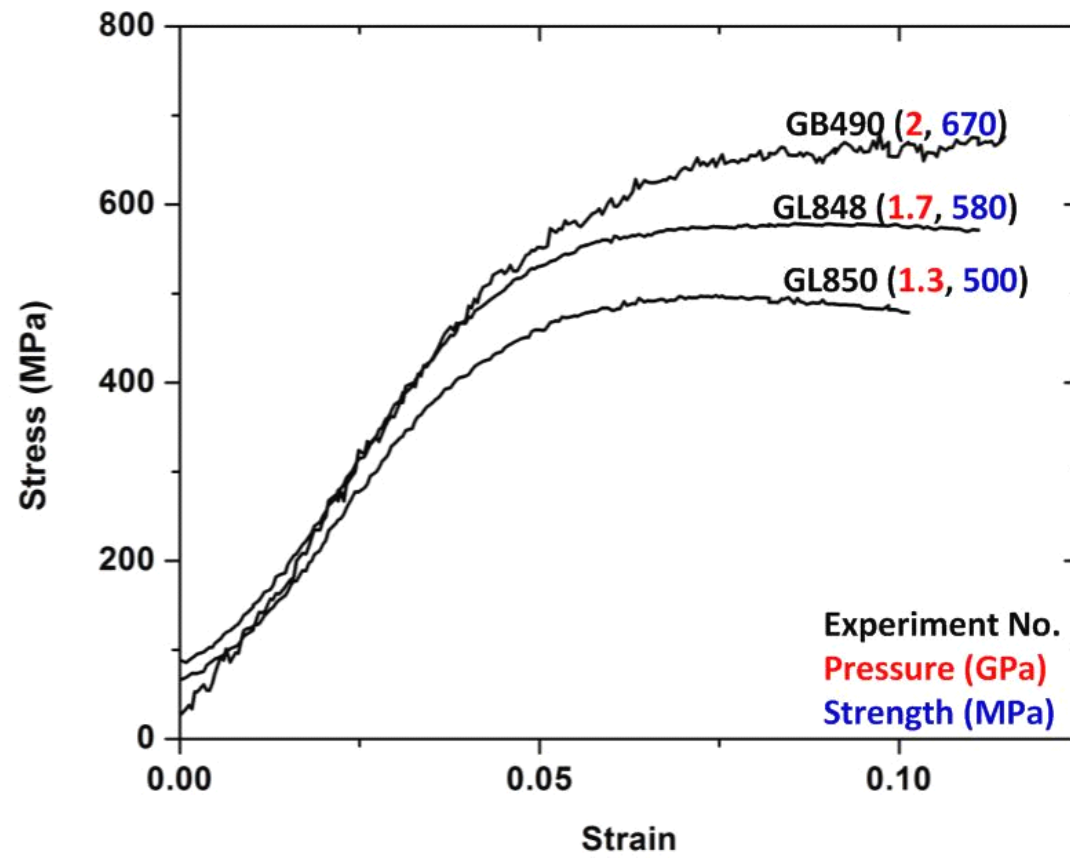


Figure 3.8 Stress versus strain plot of experiments at a strain rate of $10^{-5}/s$.

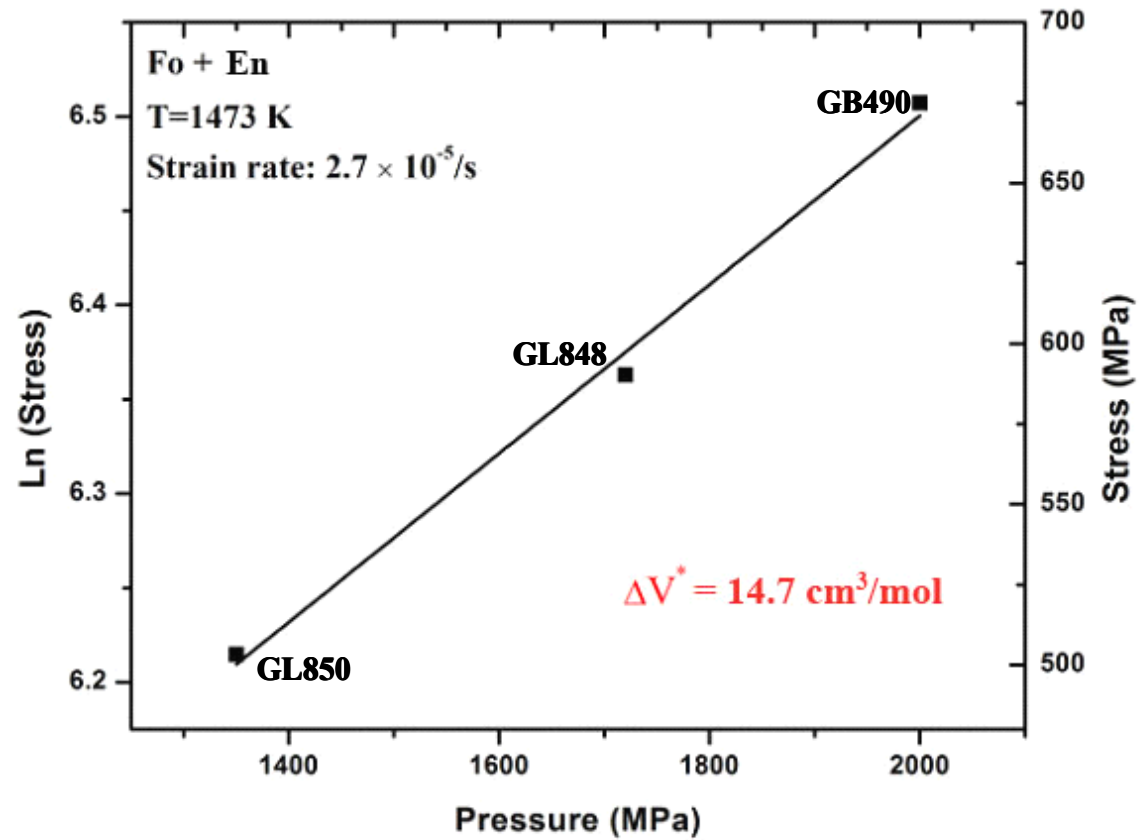


Figure 3.9 Stress/Ln Stress versus pressure plot for experiments conducted at a strain rate of $10^{-5}/s$.

3.6 Conclusion

The results of this study provide a very robust result for the ΔV^* of olivine at relatively low pressures under dry conditions. By comparison, ΔV^* values from very high pressure studies ($> \sim 6$ GPa) are very small, near zero (Li et al., 2004 & 2006). The most obvious difference between the very high pressure studies and ours is the starting material. We have used synthetic forsterite and others have used natural rocks that contain significant amounts of water, plus they used water-bearing pressure media. Much of the difference between their results and ours, therefore, may be that their ΔV^* measurement are a result for wet olivine. Bai and Green (1998), using the same apparatus as ours (and including low-pressure data obtained in the Kohlstedt gas apparatus) demonstrated that the presence of water can significantly weaken the flow strength of manganese germanate olivine, and this water weakening effect is greater at higher pressure than at lower pressure. It is likely that this is the result of both the solubility of water in olivine and the fugacity of H_2O increase substantially with increasing pressure.

Another major difference between our 5 GPa Griggs apparatus and multi-anvil apparatus is that in the latter case, stress measurement is by X-ray diffraction (using synchrotron radiation) and the correct procedure for reducing such microstress measurements to macroscopic stress remains controversial (Burnley and Zhang, 2008). The problem is that X-ray diffraction measurements of specimens under stress

in synchrotron experiments yields individual diffraction ellipses that differ in their ellipticity and therefore, by implication, different elastic strains from which different stresses are calculated. The explanation of this apparent paradox is that each diffraction ellipse is sampling a different population of grains and those different populations actually are supporting different stresses. A nontrivial question then arises concerning how to extract the macroscopic stress like that measured in conventional apparatus (like ours) from these families of crystal orientations. Fortunately, important progress has been made to solve this problem during the last few years. Using the Elastic-Plastic Self Consistent (EPSC) modeling, coded by Tuner and Tome (1994), Burnley and Zhang (2008) successfully calculated the macroscopic stress and strain for their quartz specimens deformed in the D-DIA. At minimum, this process yields an objective result relieving the experimentalist from the danger of perhaps choosing a favored diffraction ellipse that yields the wrong answer. However, in the particular case of Burnley and Zhang (2008), their calculations yielded the counter-intuitive result that the macrostress supported by the sample was greater than the stress inferred from any of their diffraction ellipses. Such a result is not impossible; the experimentalist is restricted to the strongest diffraction lines and those may not sample the most highly stressed family of grains. Stress measurement in the synchrotron remains an area of active research in itself.

Another complexity was alluded to earlier in this dissertation. The discrepancy between our ΔV^* value and the smaller values found at higher pressure could be related to a pressure-induced transition of easiest slip systems in olivine. Evidence has been accumulated that there is correlation between the drop of ΔV^* value and transition of slip system in olivine (Durinck et al., 2005; Raterron et al., 2007, 2009 & 2011; Hilairet et al., 2012). Raterron et al. (2007) documented a slip system transition in dry olivine with increasing pressure from a-slip to c-slip, with a-slip dominating at pressure lower than 3 GPa. This could explain the attenuation of seismic anisotropy in the upper mantle, while c-slip, dominant at pressure higher than 7 GPa, could explain the seismic-anisotropy anomalies observed along subduction zones. Jung and Karato (2001) demonstrated that water fugacity can change the dominant slip system and thereby produce LPO transitions in olivine. However, Jung et al. (2008) later showed that pressure alone can induce the LPO transition in olivine from a-slip to c-slip at ~ 3 GPa. More recently, Raterron et al. (2009) carried out D-DIA experiments on San Carlos olivine single crystals, oriented to promote either a-slip or c-slip, respectively. Their result pointed out, the first time, that the discrepancy of ΔV^* measurement between low and high pressure studies could be explained by the transition of slip system in olivine. They found that a-slip is very sensitive to pressure, resulting in a large ΔV^* (12 ± 4 cm³/mol) that is similar to our results whereas c-slip is essentially insensitive to pressure, resulting in a small $\Delta V^* = 3 \pm 4$ cm³/mol that is similar to other high pressure results. Hilairet et al. (2012) resolved the dominant slip

system using EPSC modeling, showing a gradual transition of slip system in olivine between 3-4 GPa. They also suggested that the comparison of ΔV^* value should be based on more detailed work on the deformation mechanism, as most high pressure studies on synchrotron facilities use samples with very small grain sizes (less than 10 μm) that will significantly affect the dominant deformation mechanism.

3.7 Future perspectives

Based on the discussion above, resolution of understanding of the pressure dependence of creep in olivine would appear to be on the horizon. Questions remaining involve the accuracy and procedure of stress measurement in the D-DIA as well as more detailed understanding of the effects of water and slip systems. Based upon our robust determination of the pressure dependence of creep in olivine at pressures below 3 GPa, we are planning to perform collaborative experiments in the D-DIA apparatus on the same forsterite polycrystalline sample we used in this study. Using an overlapping pressure range should allow us to both calibrate stress measurement in the D-DIA and simultaneously investigate the ΔV^* of forsterite over a wide range of pressure, perhaps to as high as 12 GPa.

Moreover, with the progress of technical development, electron backscatter diffraction (EBSD) measurements in the SEM have been widely used to study the LPO of various phases. We are planning to carry out the post-experiment analysis,

combining the use of EBSD to examine the LPO in olivine deformed at both low pressure and high pressure conditions as well as direct slip-system determination using TEM and oxidation techniques in the SEM. These studies should enable us to determine whether the difference of ΔV^* at low and high pressures is induced by slip system transition or is somehow the result of inappropriate calibration of high pressure apparatus (e.g. D-DIA).

Chapter 4.
Conclusions and Future Work

In this dissertation, I report my experimental studies on compositional dependence of dehydration embrittlement in serpentinized peridotite and the effect of pressure on creep in olivine, including detailed description of systematic experimental investigation on serpentinized peridotite and forsterite, extensive SEM analysis and a TEM analysis on the microstructure of fault zone materials induced by serpentine dehydration. My results provide new pieces of important information for intermediate-depth earthquakes, hydration of oceanic mantle lithosphere, and upper mantle rheology.

4.1 Serpentine Dehydration Embrittlement

4.1.1 Summary

I conducted high pressure (1-2.7 GPa) and high temperature (720-750 °C) deformation experiments on variably serpentinized peridotite. Microscopic analysis shows coherent submicron-sized solid dehydration products, olivine and talc, in the fault zones, and Mode I cracks as well as fluid inclusion trails in surrounding olivine grains close to the fault. At 1 GPa and a strain rate of 10^{-4} /s, dehydration induced faulting is observed from ~8 vol% antigorite specimen to ~65 vol% antigorite specimen. The 2.7 GPa experiment, containing ~60 vol% serpentine, demonstrates similar dehydration-induced faulting configuration as well as the nanometric dehydration products in the fault zone.

4.1.2 Conclusion

I conclude that (i) dehydration embrittlement occurs in a wide window of the composition of serpentinized peridotite, ranging from very small amounts of antigorite to more than half antigorite; (ii) near the composition of pure harzburgite, there is too little H₂O production during dehydration to produce a faulting instability; (iii) In compositions in which antigorite, greatly dominates, dehydration simply facilitates flow of serpentinite because soft and ductile antigorite becomes the interconnected matrix and relict peridotite mineralogy is dispersed in the matrix. This observation can be used to explain the serpentine diapirs in the forearc and the aseismic forearc mantle wedge that is believed to be highly serpentinized. (iv) Observation of nanometric solid dehydration products in the fault zone, and extensive Mode I cracks as well as fluid inclusion trails in the surrounding olivine grains indicate their involvement in the fault initiation process. (v) Dehydration embrittlement can still function at elevated pressure conditions where the total volume change becomes negative (Jung et al., 2004; Jung and Green, 2004). (vi) serpentinization of oceanic mantle lithosphere could be very limited and yet still trigger faulting during dehydration, providing a potential answer to the conundrum of how ocean water can percolate as deep as 40 km beneath trenches and trigger earthquakes.

4.1.3 Future Work

Most of my experiments were carried out at 1 GPa. Whether such a compositional dependence pattern for serpentine dehydration embrittlement is repeatable at higher pressures is still unknown. **In the future**, it would be of salient interest to investigate the pattern of dehydration embrittlement of serpentized peridotite at higher pressure conditions, corresponding to the deeper range of intermediate-depth earthquakes. Another series of deformation experiments, similar to that accomplished at 1GPa, could be carried out at 3GPa and even 6GPa in a Deformation-DIA apparatus installed at a synchrotron X-radiation source, using acoustic emission monitoring which would allow capture of faulting "in situ". Subsequent tomographic imaging of the 3-D specimen geometry and electron microscopy would confirm fault configurations.

4.2 ΔV^* in Creep of Olivine

4.2.1 Summary

Because the ΔV^* value is still poorly constrained, I conducted high pressure (1-2.5 GPa) and high temperature (1200 °C) deformation experiments on forsterite using the 5 GPa Green-modified Griggs apparatus at strain rates of 10^{-4} /s and 10^{-5} /s respectively, and showed a robust ΔV^* of $12 \pm 2 \text{ cm}^3/\text{mol}$ below 3 GPa for iron-free forsterite in the dislocation creep field.

4.2.2 Conclusion

My results indicate a significant pressure dependence of creep in olivine down to ~100km. After careful examination, we suggest that the ΔV^* (near zero) obtained from higher-pressure experimental studies, using D-DIA apparatus at synchrotron x-ray radiation source, are questionable because of the small sample grain size (less than 10 μ m) used and, perhaps, inadequate stress derivation from X-ray diffraction patterns. Moreover, results from single-crystal experiments by Ratteron et al. (2009 & 2011) may not be directly extrapolated to Earth which are made up of polycrystalline minerals. Last but not least, when natural rocks and/or water-bearing pressure media are used, it could be H₂O, rather than pressure, that is responsible for a ΔV^* near zero, e.g. Li et al., 2006. That argument was initially made 15 years ago with a very-high-quality data set on the flow of Mn₂GeO₄ olivine (Bai and Green, 1998).

4.2.3 Future Work

In collaboration with other experimentalists, we will carry out experiments on the same forsterite starting material as this study using D-DIA apparatus and derive stress via EPSC modeling that can generate more representatively macroscopic stress. The result will eventually provide an important calibration standard for a wide variety of studies at elevated pressure using the D-DIA apparatus. As it has been proposed that there is a correlation between the transition of LPO in olivine and the discrepancy in ΔV^* values, it would be of great importance to measure the LPO in

olivine from both low and high pressure experiments. The results should enable resolution of the disagreements about ΔV^* of creep and improve our understanding on the correlation between LPO and ΔV^* of olivine.

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