PRESSURE DEPENDENCE OF THE STRENGTH OF MAGNESITE DEFORMING BY LOW TEMPERATURE PLASTICITY, DIFFUSION CREEP, OR DISLOCATION CREEP

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Joseph W. Millard
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PRESSURE DEPENDENCE OF THE STRENGTH OF MAGNESITE DEFORMING
BY LOW TEMPERATURE PLASTICITY, DIFFUSION CREEP, OR DISLOCATION
CREEP

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Thesis

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ABSTRACT

Recent experiments by Holyoke et al. [2014] indicate that magnesite is weak compared to olivine possibly, leading to strain localization into magnesite-bearing horizons of downgoing subducting slabs, and causing intermediate depth (200 – 400 km) deep focus earthquakes (DFE). However, they did not determine the pressure dependence of the strength of magnesite. In order to determine the pressure dependence of magnesite deforming by low temperature plasticity (LTP) mechanisms (kinking and dislocation glide), diffusion creep, and dislocation creep, I deformed two natural magnesite aggregates (d ~ 3 and 80 μm) over a wide range of pressures ($P_{\text{eff}} = 0.76 – 7.5$ GPa) at strain-rates of $\sim 10^{-5}/s$ and at temperatures $T = 500, 750,$ and $900 \, ^\circ C$, respectively. Triaxial deformation experiments were conducted in the D-DIA at Beamline 6-BMB at the Advanced Photon source at Argonne National Lab and in the Griggs apparatus at the University of Akron. Differential stresses in all sets of experiments increase with increasing pressure. Microstructures in experiments performed on fine-grained magnesite at 500 \, ^\circ C include flattened, angular grains, which were slightly reduced in size; microstructures in experiments performed on fine-grained magnesite at 750 \, ^\circ C include rounded grains, increased porosity and four-grain junctions; and microstructures in experiments performed on coarse-grained magnesite at 900 \, ^\circ C include elongated grains, patchy undulatory extinction, and recrystallized grains at grain boundaries. Based on these results and results obtained by Holyoke et al. [2014], the pressure dependence or activation volume, $(V^*) = 33.8 \, (\pm 1), 2.2 \, (\pm 0.7),$ and $10.3 \, (\pm 2) \times 10^{-6}$ m$^3$/mol, for LTP, diffusion creep, and dislocation creep, respectively. With the addition of the influence
of pressure to the flow laws of Holyoke et al. [2014], the strength contrast between
magnesite and olivine decreases. However, magnesite remains orders of magnitude
weaker than olivine at all subduction zone depths which may lead to strain localization
and generation of DFE.
ACKNOWLEDGEMENTS

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I’d like to thank my parents and best friends Bill and Elaine Millard for the endless support they offered me throughout my entire schooling career.
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CHAPTER I
INTRODUCTION

Shallow earthquakes occurring at oceanic subduction zones at depths < 70 km are generally understood to occur due to brittle failure of cold crustal rock [Green and Houstin, 1995]. However, within subducting oceanic lithosphere, in the Wadati-Benioff zone at depths > 70 km, pressures are too high for brittle processes to occur [Wadati, 1928]. Instead, dehydration-embrittlement of serpentine may induce seismicity [Raleigh and Paterson, 1965; Green and Houstin, 1995; Peacock, 2001; Hacker et al., 2003].

Olivine, the dominant phase in lithospheric peridotite, is orders of magnitude stronger than serpentine at depths to which serpentine is stable [Ulmer and Trommsdorff, 1995; Hilairet et al., 2007]. As a consequence of this strength contrast, strain may be localized into serpentine under increasing temperatures and pressures as the subducting slab descends into the mantle. However, serpentine is only stable to depths of < 200 km which limits the maximum depth of earthquakes occurring by this mechanism [Hilairet et al., 2007]. Deep focus earthquakes may also occur due to mineralogical α-β-γ olivine transitions, which don’t occur until 400 km depth, where volume reduction of olivine forces surrounding rock to remobilize and compensate for the change in volume resulting in a release of energy [Kirby et al., 1996]. However, earthquakes still occur within some subducting slabs at depths between 200 – 400 km [Flinn and Engdahl 1965; Frohlich 1989] leaving a poorly understood seismically active gap.

Though most sedimentary carbonates are scraped from the oceanic crust onto the accretionary wedge, some have been found to be trapped within the subducting
lithosphere introducing CO$_2$ into the mantle wedge [Rea and Ruff, 1996; Plank and Langmuir, 1998; Kerrick and Connolly, 2001; Sleep and Zahnle, 2001; Snyder et al., 2001; Kilian and Behrmann, 2003; Zhang et al., 2003; Isshiki et al., 2004; Ducea et al., 2005; Goto et al., 2007]. The newly introduced CO$_2$ may react with Mg-rich peridotite in the mantle to form magnesian carbonates [Seto et al., 2008]. Carbonate inclusions such as dolomite and magnesite have been found in garnets exhumed from 150 – 280 km depth in Kazakhstan metapelites and marbles [Ishikawa et al., 2000; Zhu and Ogasawara, 2002; Dobrzinetskaya et al., 2003, 2006; Shatsky et al., 2005, 2006].

Unlike serpentine and carbonates like calcite and dolomite, magnesite is very stable at the high temperatures and pressures of the deep mantle [Newton and Sharp, 1975; Brey et al., 1983; Canil and Scarfe, 1990; Katsura and Ito, 1990; Biellmann et al., 1993]. Carbonaceous fluids react with peridotites, producing magnesite in networks of interconnected veins [Saldi et al., 2009; Kelemen et al., 2011; Quesnel et al., 2013]. The formation of magnesite itself may also cause earthquakes due to the large volume changed during the reaction between carbonated fluids and olivine [Kelemen and Hirth, 2012]. Considering magnesite is orders of magnitude weaker than olivine at all depths, strain localization into magnesite veins may lead to sudden shear instabilities at depths where serpentine and dolomite are unstable [Holyoke et al., 2014].

Holyoke et al. [2014] experimentally investigated the strain-rate and temperature dependence of magnesite deforming by low temperature plasticity (LTP), diffusion creep, and dislocation creep deformation mechanisms. They observed that both fine-grained ($d \sim 1 \mu$m) and coarse-grained ($d \sim 100 \mu$m) magnesite deformed by LTP processes at low temperatures ($T \leq 600$ °C). However, the fine-grained magnesite deformed by diffusion creep at high temperatures ($T > 600$ °C) and coarse-grained magnesite deformed by dislocation creep at high temperatures ($T > 600$ °C). They modeled and extrapolated these three deformation mechanisms to nature and found that magnesite was > 6 orders
of magnitude weaker than olivine along the path of a subducting slab. However, they did not determine the pressure sensitivity of the strength of magnesite deforming by each of these deformation mechanisms because their experiments were performed only at two effective pressures ($P_{\text{eff}} = 0.3$ and $0.9$ GPa for fine- and coarse-grained magnesite, respectively). Since the pressure gradient along the path of a subducting slab is very high ($0.6 – 14$ GPa) [Peacock, 2001], the pressure dependence of the deformation mechanisms of magnesite may affect the strength contrast between magnesite and olivine.

1.1 Pressure Dependence

The pressure dependence of the strength of a material (activation volume, $V^*$) is a measure of the change in strength as a function of increasing pressure. Pressure dependence is represented by:

$$\dot{\varepsilon} \propto \exp(E^* - PV^*/RT)$$

where $\dot{\varepsilon} = \text{strain-rate (1/s)}$, $E^* = \text{activation energy (J/mol)}$, $P = \text{pressure (Pa)}$, $V^* = \text{activation volume (m}^3/\text{mol)}$, $R = \text{universal gas constant (8.314 J/mol K)}$, and $T = \text{temperature (K)}$ [Durham et al., 2008].

Olivine, the dominant mineral in the mantle, has been found to increase in strength with increasing pressure at high temperatures with positive $V^*$ values ranging from $0 – 30*10^{-6}$ m$^3$/mol [Ross et al., 1979; Kohlstedt et al., 1980; Borch and Green, 1987, 1989; Karato et al., 1993; Wang, 2002; Hirth and Kohlstedt, 2003; Karato and Jung, 2003; Durham et al., 2008; Raterron et al., 2009]. The high variation in activation volumes for olivine may be attributed to the pressure dependence of water content [Hirth and Kohlstedt, 2003] and the pressure dependence of deformation in the dislocation creep regime [Hirth and Kohlstedt, 2003; Raterron et al., 2009; Durham et al., 2008].
The pressure dependence of the strength of calcite can potentially be used as a model for that of magnesite due to the two minerals’ similarities. The rhombohedral carbonates, calcite and magnesite, have the same crystallographic structure as one another with the only difference being whether Ca (calcite) or Mg (magnesite) cations are present in the structure. Both calcite and magnesite have the same \( \text{CO}_3 \) carbonate group arrangements and identical symmetries (\( \text{R} \overline{3} \text{c} \)) [Holyoke et al., 2014]. Calcite and magnesite have similar flow law parameters for each deformation mechanism (Table 1). Calcite deforming by dislocation creep has an activation volume of \( V^* = 4.4 (\pm 5.6) \times 10^6 \text{ m}^3/\text{mol} \) at experimental pressures ranging from 0.1 – 0.6 GPa [De Bresser, 2002]. The increase in strength of olivine and calcite with increasing pressure suggests that the strength of magnesite should also increase with increasing pressure.
Table 1. Deformation mechanism parameters and lattice parameters of calcite and magnesite.

<table>
<thead>
<tr>
<th></th>
<th>LTP</th>
<th>Diffusion Creep</th>
<th>Dislocation Creep</th>
<th>Lattice Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$n$</td>
<td>$H$ (KJ mol$^{-1}$)</td>
<td>$n$</td>
<td>$H$ (KJ mol$^{-1}$)</td>
</tr>
<tr>
<td>Calcite</td>
<td>15$^e$</td>
<td>250$^b$</td>
<td>1.1$^c$</td>
<td>200$^c$</td>
</tr>
<tr>
<td>Magnesite</td>
<td>19.7$^a$</td>
<td>233$^a$</td>
<td>1.1$^a$</td>
<td>209$^a$</td>
</tr>
</tbody>
</table>

$^a$Holyoke et al. [2014]
$^b$Rutter, [1974]
$^c$Herwegh et al. [2003]
$^d$Schmid et al. [1980]
$^e$Wang et al. [1996]
CHAPTER II
METHODS

My experimental study was performed using two natural magnesite aggregates from Nevada, USA, one fine-grained \((d \sim 3 \, \mu m)\) and the other coarse-grained \((d \sim 80 \, \mu m)\) over a wide range of effective pressures \(\left( P_{\text{eff}} = 0.3 - 7.5 \, \text{GPa} \right)\), strain-rates of \(\dot{\varepsilon} = 2.5 - 5.5 \times 10^{-5} / \text{s} \) and three temperatures \((T = 500, 750 \text{ and } 900 \, ^\circ \text{C})\). Experiments using fine-grained magnesite were performed using a multi-anvil Deformation DIA apparatus (D-DIA; Figure 1a,b) and a Griggs-type piston-cylinder solid-medium rod deformation apparatus (Griggs apparatus; Figure 1c,d), whereas all experiments performed on coarse-grained magnesite were only performed in the D-DIA.

2.1 Starting Materials

Both the fine-grained \((d \sim 3 \, \mu m)\) and the coarse-grained \((d \sim 80 \, \mu m)\) magnesite aggregates come from Nevada, USA (Figure 2). Grains of the fine-grained magnesite are angular, and the porosity is very low \((\sim 1\%)\) (Figure 2a,b). Grains in the coarse-grained magnesite are euhedral, relatively free of twins and deformation banding with straight extinction under cross-polarized light, and also has a low porosity \((\sim 1\%)\) (Figure 2c). Porosities of both starting materials were determined employing the Archimedes method in water. Inclusions \(<< 1\%)\) of opaque minerals, possibly ultramafic or altered ultramafic hydrous and oxide phases, are present. Microprobe analyses indicates
Figure 1. Deformation apparatuses used. (a) Durham-type assembly surrounded by three WC anvils and one sintered diamond anvil. Incident X-Rays pass through the assembly, diffract based on lattice strain and are recorded on the detectors positioned behind the D-DIA. (b) The Durham-type assembly used in experiments performed on 2 mm magnesite cylinders. Two samples can be stacked but separated with a Pt foil (not shown). (c) Griggs apparatus and (d) SSA assembly used in the Griggs apparatus modified from Holyoke et al. [2014].
Figure 2. Starting materials. (a) Cross-polarized ultra-thin (1 – 2 μm), doubly-polished thin section of fine-grained (3 μm) NV magnesite exhibits undeformed, angular grains with a porosity of ~1%. (b) SE SEM image of the same fine-grained magnesite etched with 10% dilute HCl acid for 10 min. (c) Ultra-thin, doubly-polished cross-polarized thin section of coarse-grained (80 μm) NV magnesite exhibits euhedral, straight extinction grains with little evidence of deformation.
coarse-grained Nevada magnesite contains trace amounts of Ca and Fe 
\((\text{Mg}_{0.994}\text{Ca}_{0.004}\text{Fe}_{0.002}\text{CO}_3)\) [Holyoke et al., 2014].

Cores for experiments performed in the D-DIA apparatus were collected from thin 
\((t \sim 5 \text{ mm})\) slabs of starting material with a 1 mm inner diameter core bit. The faces of 
the magnesite cylinders were ground perpendicular to the length of the cylinder using 
a fine diamond file and were dried in air for \(\sim 24\) hours prior to installation into the 
assembly.

Cores for experiments performed in the Griggs apparatus were cored from a block 
of the starting material using a 5 mm inner diameter core bit and cut perpendicular to 
the length of the core to generate a cylinder 10 mm in length. The ends of the cylinders 
were ground perpendicular to the length of the cylinder with silicon carbide 600 grit. The 
cylinders were cleaned in deionized water in an ultrasonic bath to remove grit and air 
dried for at least 24 hours prior to the experiment.

2.2 Experimental Techniques

Experiments were performed on fine- and coarse-grained magnesite at 
\(P = 0.85 - 7.9\ \text{GPa} \ (P_{\text{eff}} = 0.76 - 7.5\ \text{GPa})\) at \(\varepsilon = 5.5 - 2.5\times10^{-4}/\text{s}\) at \(T = 500, 750,\) and 
900 °C. Magnesite produces a partial pressures of CO\(_2\) with increasing pressure. This 
partial pressure of CO\(_2\) is 0 GPa at \(T = 500\ \text{°C}\), 0.09 GPa at \(T = 750\ \text{°C}\), and 0.4 GPa at 
\(T = 900\ \text{°C}\). The partial pressure of CO\(_2\) creates a pore pressure which reduces the 
pressure to an effective pressure:

\[
P_{\text{eff}} = P - P_{\text{CO}_2}
\]  

(2)

where \(P_{\text{eff}}\) is the effective pressure, \(P\) is the experimental confining pressure, and \(P_{\text{CO}_2}\) is 
the partial pressure of CO\(_2\). [Harker and Tuttle, 1955; Goldsmith and Heard, 1961; Irving 
and Wyllie, 1975].
2.2.1 D-DIA Apparatus

I performed experiments on fine- and coarse-grained magnesite using the D-DIA (Figure 1a,b) at beam-line 6-BMB at the Advanced Photon Source at Argonne National Laboratory, which is capable of reaching pressures of ~15 GPa and temperatures of ~ 2000 °C [Wang, et al., 2003; Raterron et al., 2012]. I used the Durham-type assembly [Durham et al., 2008] which consists of hollow cylinders of Boron Nitrate (BN) and graphite within a sphere of mullite in a soft-fired pyrophyllite cradle (Figure 1b). This assembly can accommodate a cylinder (or stacked cylinders) of 1 mm diameter by 2 mm long, with crushable $\text{Al}_2\text{O}_3$ pistons at each end of the cylinders.

I performed experiments on fine-grained magnesite at 500, 750, and 900 °C in the D-DIA (Figure 1a,b; Tables 2 & 3). Fine-grained magnesite was stacked with (but separated by a Pt foil) fine-grained dolomite; each cylinder was 1 mm in diameter and 1 mm in length, and deformed simultaneously. The fine-grained dolomite in these experiments were analyzed by Blasko [2017], but not in this study. Because stacked cylinders are deformed in series, they deformed at identical stresses but different strains. The starting materials were wrapped in a thin Pt jacket and capped on both ends by thin Pt disks and thin Re foils adjacent to an alumina piston at each end.

Deformation temperatures were reached and maintained by applying a controlled wattage to the graphite furnace. The temperature gradient is estimated to be ~50 °C over a 1 mm length near the center of the magnesite cylinder [Raterron et al., 2013].

To apply pressure ($\sigma_j$), the D-DIA uses three tapered WC (X-ray opaque) anvils and one tapered sintered diamond (X-ray transparent) anvil oriented orthogonally around the assembly (Figure 1a). Two identical tapered WC, vertically-oriented anvils advance towards the assembly providing a vertical load ($\sigma_j$). When the horizontal and vertical forces are equal, the assembly is under static pressure ($\sigma_j = \sigma_j$). To apply differential
Table 2. List of experiments performed on fine-grained magnesite.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Temp</th>
<th>Pressure</th>
<th>Effective Pressure</th>
<th>Initial Strain Rate</th>
<th>Final Strain Rate</th>
<th>Strain</th>
<th>Peak Strength</th>
<th>Final Strength</th>
<th>Grain Size</th>
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<tr>
<td>MAG_004$^{ad}$</td>
<td>500</td>
<td>6.6 ± 0.2</td>
<td>6.6 ± 0.2</td>
<td>0.9</td>
<td>2.5</td>
<td>27</td>
<td>3.1 ± 0.3</td>
<td>3.1 ± 0.3</td>
<td>2.1</td>
</tr>
<tr>
<td>MAG_005$^{ad}$</td>
<td>500</td>
<td>3.4 ± 0.1</td>
<td>3.4 ± 0.1</td>
<td>0.7</td>
<td>2.7</td>
<td>27</td>
<td>1.6 ± 0.3</td>
<td>1.6 ± 0.3</td>
<td>2.5</td>
</tr>
<tr>
<td>MAG_006$^{ad}$</td>
<td>500</td>
<td>5.6 ± 0.3</td>
<td>5.6 ± 0.3</td>
<td>1.3</td>
<td>2.8</td>
<td>28</td>
<td>2.2 ± 0.3</td>
<td>2.2 ± 0.3</td>
<td>1.6</td>
</tr>
<tr>
<td>MAG_020$^{bcd}$</td>
<td>750</td>
<td>6.4 ± 0.1</td>
<td>6.3 ± 0.1</td>
<td>0.6</td>
<td>1.3</td>
<td>5</td>
<td>1.2 ± 0.2</td>
<td>1.2 ± 0.2</td>
<td>-</td>
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<tr>
<td></td>
<td></td>
<td>5.5 ± 0.1</td>
<td>5.4 ± 0.1</td>
<td>0.4</td>
<td>2.1</td>
<td>6</td>
<td>0.9 ± 0.2</td>
<td>0.9 ± 0.2</td>
<td>-</td>
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<tr>
<td></td>
<td></td>
<td>3.9 ± 0.1</td>
<td>3.8 ± 0.1</td>
<td>1.9</td>
<td>3.0</td>
<td>6</td>
<td>0.6 ± 0.2</td>
<td>0.6 ± 0.2</td>
<td>2.9</td>
</tr>
<tr>
<td>Z-100$^b$</td>
<td>750</td>
<td>0.85 ± 0.02</td>
<td>0.76 ± 0.02</td>
<td>1.5</td>
<td>2.1</td>
<td>15</td>
<td>0.50 ± 0.02</td>
<td>0.38 ± 0.02</td>
<td>2.3</td>
</tr>
</tbody>
</table>

$^a$Experiments performed in D-DIA apparatus (Durham-type assembly)
$^b$Experiments performed in Griggs apparatus (SSA)
$^c$Pressure stepping experiment
$^d$Stacked D-DIA experiments with fine-grained magnesite and dolomite.
Table 3. List of experiments performed on coarse-grained magnesite.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Temp (°C)</th>
<th>Pressure (GPa)</th>
<th>Effective Pressure (GPa)</th>
<th>Initial Strain Rate (*10^-5 s^-1)</th>
<th>Final Strain Rate (*10^-5 s^-1)</th>
<th>Strain (%)</th>
<th>Peak Strength (GPa)</th>
<th>Final Strength (GPa)</th>
<th>Grain Size (µm)</th>
<th>Recrystallized Grain Size (µm)</th>
<th>Grains w/ Kinks (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAG_008</td>
<td>900</td>
<td>5.6 ± 0.1</td>
<td>5.2 ± 0.1</td>
<td>1.5</td>
<td>2.9</td>
<td>30</td>
<td>1.1 ± 0.2</td>
<td>1.0 ± 0.2</td>
<td>63</td>
<td>2.0</td>
<td>10</td>
</tr>
<tr>
<td>MAG_010</td>
<td>900</td>
<td>7.9 ± 0.1</td>
<td>7.5 ± 0.1</td>
<td>1.2</td>
<td>2.9</td>
<td>36</td>
<td>1.7 ± 0.2</td>
<td>1.7 ± 0.2</td>
<td>59</td>
<td>1.8</td>
<td>92</td>
</tr>
<tr>
<td>MAG_012</td>
<td>900</td>
<td>6.2 ± 0.1</td>
<td>5.8 ± 0.1</td>
<td>1.8</td>
<td>3.3</td>
<td>30</td>
<td>1.6 ± 0.2</td>
<td>1.6 ± 0.2</td>
<td>75</td>
<td>1.9</td>
<td>14</td>
</tr>
<tr>
<td>MAG_014</td>
<td>900</td>
<td>3.2 ± 0.1</td>
<td>2.9 ± 0.1</td>
<td>1.5</td>
<td>3.5</td>
<td>36</td>
<td>0.9 ± 0.2</td>
<td>0.8 ± 0.2</td>
<td>65</td>
<td>2.2</td>
<td>0</td>
</tr>
<tr>
<td>MAG_016</td>
<td>900</td>
<td>6.7 ± 0.1</td>
<td>6.3 ± 0.1</td>
<td>1.1</td>
<td>2.9</td>
<td>27</td>
<td>1.3 ± 0.2</td>
<td>1.3 ± 0.2</td>
<td>74</td>
<td>1.8</td>
<td>82</td>
</tr>
</tbody>
</table>

All experiments were performed in D-Dia apparatus (Durham-type assembly).
loading \((\sigma_1 - \sigma_3 > 0)\), the vertical anvils advance towards each other, while the horizontal anvils retract to maintain constant confining pressure \((\sigma_3)\).

As the anvils apply stress to the assembly, the pyrophyllite cradle is squeezed between the anvils until a hardened pyrophyllite gasket forms. The pyrophyllite gasket does not deform uniformly between the anvils in each experiment so consistent forces applied by the anvils will not produce identical confining pressures in each experiment. Therefore, confining pressures are calculated after the experiment.

2.2.2 D-DIA Data Reduction

I used X-ray spectra of the \(\text{Al}_2\text{O}_3\) pistons in the load column to calculate pressure and differential stresses. The elastic deformation of the grains in the \(\text{Al}_2\text{O}_3\) pistons due to pressurization and axial loading causes changes in the lattice plane spacing. These changes in lattice plane spacing are determined by measuring the shift of the peaks of the X-ray spectra caused by diffraction. Spectra were not collected of the magnesite cylinders because \(\text{Al}_2\text{O}_3\) deforms elasticity relative to magnesite which deforms plastically during experiments. These spectra are converted to pressure and stress measurements using PLOT85, Python, and PolydefixED software (Figure 3a,b)

The X-ray beam is also used to collect radiographs which are used to measure strain (Figure 4). Calculations of strain and strain-rate are made during the experiments using Excel (Figure 3c). By correlating the time-stamps on the X-ray spectra files to the time strains were measured, a stress-strain curve can be calculated (Figure 3d). The increase and decrease of stresses between measurements are the result of spectra collected of the top and bottom \(\text{Al}_2\text{O}_3\) pistons producing slightly different stress measurements.

Differential stresses reported in this study are based on the average differential stress on five of the nine lattice planes in \(\text{Al}_2\text{O}_3\) during constant stress deformation (Figure 3a,b). Reaching constant stress deformation is to ensure defects such as mobile
Figure 3. D-DIA differential stress and strain measurements. (a) Five of nine lattice plane differential stresses are measured throughout each experiment. (b) The five differential stresses are averaged to obtain a single differential stress at a single point in time throughout each experiment. (c) Strain measurements are collected using the radiographs to calculate the change in strain relative to the undeformed original magnesite cylinder. Strain measurements collected by radiographs. (d) Differential stress and strain measurements are combined based on the time measurements were taken.
Figure 4. X-radiograph of sample assembly used to measure strain. Strain before (a: $\varepsilon = 0\%$), and strain after (b: $\varepsilon = 30\%$) deformation. MAG_012, $T = 900 \, ^{\circ}C$, $\dot{\varepsilon} = 3.3*10^{-3}/s$, $P_{eff} = 5.8 \, $GPa.
dislocation density and point-defect concentrations are generating and recovering at an identical rate homogeneously throughout the sample [Raterron et al., 2004]. If the sample did not reach constant stress deformation, the last two stresses collected were averaged and reported as the final stress. Peak stresses that occur before steady state deformation (i.e., strain weakening occurs) are reported in Tables 1 and 2, but are not used in calculations of constant stress behavior.

2.2.3 Griggs Apparatus

One experiment was performed on fine-grained magnesite using the Griggs apparatus, which is capable of reaching pressures of up to ~2 GPa and temperatures of ~1300 °C [Griggs, 1967; Tullis and Tullis, 1986; Holyoke et al., 2010] (Figure 1c,d). I used a solid salt assembly (SSA) for experiments [Holyoke and Kronenberg, 2010] because the stress measurement resolution is considerably greater than the D-DIA.

The SSA consists of concentric cylinders of NaCl, soft-fired pyrophyllite, and graphite which surround the Al₂O₃ pistons and magnesite cylinders (Figure 1d). The magnesite cylinder is jacketed with Ag. A 3x wrap of Ag is placed in the annular space between the magnesite cylinder and Ag jacket to limit void space and capped at each end with Ag discs. The Ag jacket is crimped over the Ag discs and Al₂O₃ pistons are adjacent to the jacketed magnesite cylinder (Figure 1d). The crimped ends of Ag jacket and discs create a mechanical weld at experimental pressure and temperature conditions.

The magnesite cylinder is heated by the graphite furnace and temperature is monitored by a pre-manufactured K-type thermocouple with its welded bead positioned just outside the edge of the Ag jacket centered vertically along the axis of the magnesite cylinder. The temperature gradient is ~5 – 10 °C along the length of the magnesite cylinder [Holyoke et al., 2014]. The assembly is pressurized over a period of ~4 hours.
To induce a load, the Griggs apparatus applies force to the sample with a load ram making contact with the WC load piston and alumina pistons (Figure 1c,d). The load ram is driven by a gear transmission system powered by a constant speed motor allowing constant strain-rates to be achieved (Figure 1c). The external nature of the load cell causes measurements of force applied to the sample to include friction between the assembly configuration, the load piston/ram, pressure piston/ram and stress transducer. Therefore, a correction must be made to so that they are equivalent to measurements made in the much more precise, low pressure \( P_{\text{max}} \sim 0.5 \text{ GPa} \), gas confining medium, Heard rock deformation apparatus:

\[
\sigma_{\text{corr}} = 0.73*\sigma_{\text{SSA}} - 0.048 \text{ GPa}
\]  

where \( \sigma_{\text{corr}} \) is the corrected stress, and \( \sigma_{\text{SSA}} \) is the stress measured by a SSA in a Griggs apparatus [Holyoke and Kronenberg, 2010].

2.3 Thin Section Sample Preparation

Samples for experiments performed using both D-DIA and Griggs apparatus were impregnated with epoxy and cut in half parallel to the direction of compression. The cut faces were ground using 3 \( \mu \)m silicon carbide grit and polished with 0.3 \( \mu \)m Al\(_2\)O\(_3\) powder. The samples were then cleaned in an ultrasonic cleaner and dried on a hot-plate overnight. The polished faces were mounted to a glass slide and polished with 0.3 \( \mu \)m Al\(_2\)O\(_3\) powder to \( (t \sim 1 – 2 \mu \text{m}) \) and imaged using a Carl ZEISS Axia Scope.A1 Microscope.
2.4 SEM Section Sample Preparation

Imaging samples of fine-grained magnesite in secondary electron (SE) or back-scatter electron (BSE) was difficult due to the low orientation contrast between individual grains. In order to enhance grain boundaries, I developed a method to etch grain boundaries. I polished a slab of fine-grained magnesite using the same techniques as the thin sections. I placed 10% diluted HCl on different spots of the fine-grained magnesite slab for 10, 100, 1000, and 10000 seconds and imaged these spots in the SEM (Appendix C). Etching for 1000 seconds enhanced grain boundaries without significantly affecting the grain size.

I used the University of Akron’s FEI Quanta 200 Environmental Scanning Electron microscope to analyze the etched samples using BSE and SE (30 kV) to determine grain-size and image other microstructures. All grain-sizes reported in this study were determined via tracing grain boundaries in SEM/BSE images or optical micrographs using Photoshop and converted to grain-sizes using Image SXM (Barbery, 2017).
In order to determine the pressure dependence of magnesite, I deformed fine-grained magnesite at $T = 500$ and 750 °C, $\dot{\varepsilon} = 1.3 - 3.0 \times 10^{-5}/s$, and a range of pressures $P_{\text{eff}} = 0.78 – 6.6$ GPa. I also deformed coarse-grained magnesite at $T = 900$ °C at $\dot{\varepsilon} = 2.9 – 3.5 \times 10^{-5}/s$ over a range of pressures $P_{\text{eff}} = 2.9 – 7.5$ GPa. I chose these temperatures and strain-rate conditions based on results of low pressure experiments performed on magnesite by Holyoke et al. [2014] who deformed fine-grained ($d \sim 1$ µm) magnesite by LTP at $T = 500$ °C, fine-grained ($d \sim 1$ µm) magnesite by diffusion creep at $T = 750$ °C and coarse-grained ($d \sim 100$ µm) magnesite by dislocation creep at $T = 900$ °C.

3.1 500 °C Fine-Grained Magnesite Deformation

I deformed fine-grained ($d \sim 3$ µm) magnesite cylinders using the D-DIA at $T = 500$ °C, $\dot{\varepsilon} = 2.5 – 2.8 \times 10^{-5}/s$, and three pressures ($P_{\text{eff}} = 6.6$, 3.4, and 5.6 GPa) to strains of 27, 27, and 28%, respectively (Table 1). In all experiments, differential stresses increased rapidly as the load rams were advanced, yielding at low strains ($\varepsilon \sim 6\%$) (Figure 5a). After yielding, differential stresses increased slowly until the end of each experiment.

Microstructures observed in all samples include flattening of angular grains perpendicular to compression, and slight grain-size reduction (Figure 6). However, kink
Figure 5. Differential stress-strain curves for each experiment. (a) Differential stresses in experiments performed on fine-grained magnesite at $T = 500 \, ^\circ C$ increase with increasing pressure. Error bars correspond to the experiment with the highest degree of error for each set of temperatures. (b) Differential stresses in the experiment performed on fine-grained magnesite at $T = 750 \, ^\circ C$ increase with increasing pressure. In experiment MAG_020, depressurization between deformation steps 1-2 and 2-3 occur over 110 and 82 min, respectively. (c) Differential stress in experiments performed on coarse-grained magnesite at $T = 900 \, ^\circ C$ increase with increasing pressure.
Figure 6. Fine-grained magnesite deformed at $T = 500 \, ^\circ\text{C}$ and etched with 10% HCl acid. (a) Microstructures include flattened, angular grains and slight grain-size reduction. At $P_{\text{eff}} = 6.6 \, \text{GPa}$ (MAG_004) significant kinking is observed relative to lower pressure experiments at the same temperature. (b) Close up of MAG_004. (c) no kinking is observed at $P_{\text{eff}} \leq 5.6 \, \text{GPa}$, $T = 500 \, ^\circ\text{C}$ (MAG_005; $P_{\text{eff}} = 3.4 \, \text{GPa}$).
bands are only observed at the experiment performed at the highest pressure (MAG_004; \( P_{\text{eff}} = 6.6 \) GPa) (Figure 6b).

3.2 750 °C Fine-Grained Magnesite Deformation

I deformed a fine-grained (\( d \sim 3 \) µm) magnesite cylinder using the D-DIA at 
\( T = 750 \) °C, \( \dot{\varepsilon} = 1.3 - 3.0 \times 10^{-5} \)/s, and three pressures (\( P_{\text{eff}} = 6.3, 5.4, \) and \( 3.8 \) GPa) to strains of 6, 6, and 5%, respectively (Table 2). I also deformed a fine-grained (\( d \sim 3 \) µm) magnesite cylinder using the Griggs apparatus at \( T = 750 \) °C, \( \dot{\varepsilon} = 2.1 \times 10^{-5} \)/s, at \( P_{\text{eff}} = 0.76 \) GPa) to a strain of 15% (Table 2). In all experiments, differential stresses increased rapidly as the load rams were advanced yielding at low strains (\( \varepsilon \sim 3\% \)) (Figure 5b). After yielding, differential stresses decrease slightly until the end of each experiment.

Microstructures observed in all samples include rounded grains, increased porosity, and four-grain junctions (Figure 7). However, grain roundness is more significant at the lowest pressure, single-step, highest strain deformation experiment performed in the Griggs apparatus (Z-100: \( P_{\text{eff}} = 0.76 \) GPa; \( \varepsilon = 15\% \)) (Figure 7a).

3.3 900 °C Coarse-Grained Magnesite Deformation

I deformed coarse-grained (\( d \sim 80 \) µm) magnesite cylinders using the D-DIA at 
\( T = 900 \) °C, \( \dot{\varepsilon} = 2.9 - 3.5 \times 10^{-5} \)/s, and four pressures (\( P_{\text{eff}} = 2.9, 5.2, 5.8, 6.3, \) and \( 7.5 \) GPa) to strains of 36, 30, 30, 27, and 36%, respectively (Table 3). In all experiments, differential stresses increased rapidly as the load rams were advanced yielding at low
Figure 7. Fine-grained magnesite deformed at $T = 750 \, ^\circ C$ and etched with 10% HCl acid. (a) Microstructures include rounded grains, increased porosity from the starting material, and four-grain junctions ($Z$-100; $P_{eff} = 0.76 \, \text{GPa}$). (b) Microstructures of pressure-stepping experiment MAG 020 ($P_{eff} = 3.8 - 6.3 \, \text{GPa}$) have more angular grains than the single-step experiment ($Z$-100).
strains (ε ~ 8%) (Figure 5c). After yielding, differential stresses increase slowly and reach constant stress deformation.

Microstructures observed in all samples include flattening of grains perpendicular to compression, patchy undulatory extinction, and recrystallization at grain boundaries (Figure 8). Kinking is observed in all samples except at the lowest pressure (MAG_014; $P_{\text{eff}} = 2.8$ GPa). However, significant kinking is observed at high pressures ($P_{\text{eff}} \geq 6.7$ GPa) (Figures 8b and 9).
Figure 8. Coarse-grained magnesite deformed at $T = 900 \, ^\circ\text{C}$. (a) Microstructures include elongated grains, patchy undulatory extinction, and recrystallization at grain boundaries (MAG_014; $P_{\text{eff}} = 2.8 \, \text{GPa}$). (b) Kink density increases with increasing pressure ($P_{\text{eff}} \geq 5.2 \, \text{GPa}$) with a significant increase in kink density occurring at $P_{\text{eff}} \geq 6.7 \, \text{GPa}$ (MAG_010; $P_{\text{eff}} = 7.5 \, \text{GPa}$).
Figure 9. Kink density of coarse-grained magnesite. Increased pressure of coarse-grained magnesite deformed at $T = 900^\circ C$ increases kink density but a significant increase in kink density occurs at $P_{\text{eff}} \geq 6.7$ GPa. An increase in kink density does not correlate with an increase in differential stress.
CHAPTER IV
DISCUSSION

The mechanical and microstructural results from deformation experiments performed on magnesite were used to determine its pressure dependence deforming by LTP, diffusion creep, and dislocation creep. At each set of temperature conditions, a different response to pressure sensitivities was observed.

4.1 Deformation Of Fine-Grained Magnesite At 500 °C

Strengths of fine-grained magnesite deformed at $T = 500$ °C increase with increasing pressure and are greater than those of magnesite deformed at $T = 750$ and 900 °C (Figure 5a). Microstructures include flattened angular grains, a slight reduction in grain size, with kinking occurring only at the highest pressure (MAG_004; $P_{\text{eff}} = 6.6$ GPa) (Figure 6). These microstructures are similar to those observed in fine-grained magnesite [Holyoke et al., 2014], calcite marble [Liu et al., 2002], olivine [Raleigh, 1968], and calcite synthetically hot pressed with dolomite [Kushnir et al., 2015] deformed by LTP.

Holyoke et al. [2014] performed one experiment on fine-grained magnesite ($d \sim 1 \mu$m) at 500 °C, $\dot{\varepsilon} = 1.6 \times 10^{-5}$/s, and $P_{\text{eff}} = 0.3$ GPa, which was similar to mine, yielding at low strain ($\varepsilon \sim 2\%$) and work hardening until the end of the experiment ($\sigma_{\text{diff}} = 0.335$ GPa, $\varepsilon = 6\%$). They observed similar microstructures, including angular grains and a similar porosity to the starting material. Liu et al. [2002] observed kinking in naturally deformed Damara orogen, Namibia calcite attributed to deformation by LTP mechanisms.
Raleigh [1968] experimentally deformed olivine at 0.5 GPa which deformed by plastic flow on several active slip systems which were studied by analyzing the slip bands deformation lamella, and kink bands. Kushnir et al. [2015] performed deformation experiments on hot isostatic composites of variable amounts of calcite and dolomite to determine the role of dolomite plays in the strength of limestone. They found that the strong crystallographic preferred orientation and lack of sub grains in the calcite were consistent with grain boundary sliding assisted by limited dislocation glide [Schmid et al., 1987; Rutter et al., 1994].

4.2 Deformation Of Fine-Grained Magnesite At 750 °C

Strengths of fine-grained magnesite deformed at \( T = 750 \, ^\circ\text{C} \) increase with increasing pressure and are the lowest differential stresses relative to experiments performed at \( T = 500 \) and 900 °C (Figure 5b). Microstructures include rounded grains, increased porosity, and four-grain junctions (Figure 7). These microstructures are similar to those of fine-grained magnesite [Holyoke et al., 2014] and calcite [Schmid et al., 1977] deformed by diffusion creep.

Holyoke et al. [2014] performed one single-step and one strain rate-stepping experiment on fine-grained magnesite \((d \sim 1 \, \mu\text{m})\) at 750 °C, \( \dot{\varepsilon} = 1.6*10^{-5}/\text{s} \) and \(1.4*10^{-6} - 1.6*10^{-4}/\text{s} \), respectively at \( P_{\text{eff}} = 0.3 \, \text{GPa} \). Their mechanical response of the single-step experiment was similar to mine yielding at low strain \((\varepsilon \sim 2\%)\) and strain weakened until the end of the experiment \((\sigma_{\text{diff}} = 0.004 \, \text{GPa}, \varepsilon = 4\%)\). The microstructures observed in their experiments were similar to those observed in Z-100 including rounding of grains and an increase in porosity. Schmid et al. [1977] deformed Solnhofen limestone at \( T = 900 \, ^\circ\text{C} \) at \( \dot{\varepsilon} = 6.7*10^{-4}/\text{s} \) in regime 3 or superplastic flow regime (diffusion creep) and also observed grain rounding.
4.3 Deformation Of Coarse-Grained Magnesite At 900 °C

Strengths of coarse-grained magnesite deformed at $T = 900$ °C increase with increasing pressure and are intermediate between those observed in experiments performed at $T = 500$ and 750 °C. Prior to yielding, coarse-grained magnesite deforms at a constant stress at $P_{\text{eff}} \geq 5.8$ GPa and strain weakens at $P_{\text{eff}} \leq 5.2$ GPa (Figure 5c). Microstructures include flattening of grains perpendicular to compression, significant recrystallization at grain boundaries, patchy undulatory extinction, with significant kinking occurring only at high pressures ($P_{\text{eff}} \geq 1.3$ GPa) (Figure 8). These microstructures are similar to those observed in coarse-grained magnesite [Holyoke et al, 2014], dolomite [Davis et al., 2008; Holyoke et al., 2013], and Solnhofen limestone [Schmid et al., 1977] deformed by dislocation creep.

Holyoke et al. [2014] performed two single-step experiments on coarse-grained magnesite ($d \sim 100$ μm) by dislocation creep at 900 °C, $\dot{\varepsilon} = 1.6 \times 10^{-5}$/s and $1.6 \times 10^{-4}$, respectively at $P_{\text{eff}} = 0.9$ GPa. Their mechanical response was similar to mine yielding at low strain ($\varepsilon \sim 8\%$) and strain weakened until the end of the experiment ($\sigma_{\text{diff}} = 0.080$ GPa, $\varepsilon = 47\%$, and $\sigma_{\text{diff}} = 0.055$ GPa, $\varepsilon = 16\%$, respectively). The microstructures observed in their experiments were very similar to those observed in MAG_010, MAG_012, MAG_014, and MAG_016 include patchy undulatory extinction, recrystallization at grain boundaries, and limited kinking. Holyoke et al. [2013] deformed coarse-grained dolomite by dislocation creep at $T = 700 – 1000$ °C at $P_{\text{eff}} = 0.9$ GPa at, $\dot{\varepsilon} = 1.7 \times 10^{-7}$/s – $1.4 \times 10^{-4}$/s and observed undulatory extinction, recrystallization along grain boundaries, and twinning. Davis et al. [2008] deformed natural coarse-grained Madoc dolomite at $T = 850$ °C, $P_{\text{eff}} = 200$ MPa, at $\dot{\varepsilon} \sim 10^{-5}$/s and found smooth undulatory extinction, with some finely recrystallized grains at grain boundaries consistent with deformation by dislocation creep. Schmid et al. [1977] deformed Solnhofen limestone in
“regime 2” (dislocation creep) at $T = 700\,^\circ$C, $\dot{\varepsilon} = 2\times10^{-3}/s$ and observed flattening of grains, undulatory extinction and subgrain boundaries.

4.4 The Effect Of Pressure On Magnesite Deformation Mechanisms

In order to determine the pressure dependence of the strength of magnesite ($V^*$) deforming by LTP, diffusion creep, and dislocation creep, I have combined my data with select data from experiments from Holyoke et al. [2014]. For experiments performed at $500\,^\circ$C, the strengths were normalized to the same strain-rate ($\dot{\varepsilon} \sim 10^{-5}/s$) using the flow law of Holyoke et al. [2014] and compared to pressure (Figure 10a)

$$\text{Ln}(\sigma) = (V^*/nRT)*P$$

where $\sigma$ is the differential stress, and $n$ is the stress exponent. I used this relationship to calculate $V^*$ for experiments performed at $500\,^\circ$C (LTP: $V^* = 33.8\,(\pm1)*10^{-6}\,m^3/mol$) (Table 4). I used the same procedure to calculate $V^*$ for experiments performed at $750\,^\circ$C (diffusion creep: $V^* = 2.2\,(\pm0.7)*10^{-6}\,m^3/mol$) and $900\,^\circ$C (dislocation creep: $V^* = 10.3\,(\pm2)*10^{-6}\,m^3/mol$) (Figure 10b,c; Table 4).

The low value of $V^*$ for magnesite deforming by diffusion creep is similar to the low value of wet olivine deforming by diffusion creep ($V^* = 4*10^{-6}\,m^3/mol$) [Hirth and Kohlstedt, 2003]. The higher value of $V^*$ for magnesite deforming by dislocation creep is similar to the higher values of wet and dry San Carlos olivine deforming by dislocation creep ($V^* = 12.8*10^{-6}\,m^3/mol$) [Bollinger et al., 2014] as well as the higher values of calcite single crystals and Carrara marble deforming by dislocation creep ($V^* = 15$ and $16.4*10^{-6}\,m^3/mol$, respectively) [de Bresser, 2002].

I used the $V^*$ values I calculated and the values ($n$ and $H$) Holyoke et al. [2014] calculated for LTP and dislocation creep and combined data from all experiments from...
Figure 10. The activation volumes calculated for LTP, diffusion, and dislocation creep. Pressures range $P_{eff} = 0.3 – 6.6, 0.76 – 6.3, \text{ and } 0.9 – 7.5 \text{ GPa}$ for LTP, diffusion creep and dislocation creep, respectively. Data is incorporated from Holyoke et al. [2014].
Table 4. List of parameters to describe the pressure dependence of magnesite.

<table>
<thead>
<tr>
<th>Deformation Mechanism</th>
<th>$A$ (±)</th>
<th>Units</th>
<th>Pressure Range</th>
<th>$n^a$</th>
<th>$m$</th>
<th>$E^*^a$ (KJ mol$^{-1}$)</th>
<th>$V^*$ ($10^{-6}$ m$^3$/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LTP</td>
<td>$7.44 \times 10^{-41}$</td>
<td>$3.57 \times 10^{40}$</td>
<td>MPa$^{-n}$ s</td>
<td>0.3 - 6.6</td>
<td>19.7</td>
<td>233 ± 16</td>
<td>33.8 ± 3</td>
</tr>
<tr>
<td>Diffusion Creep</td>
<td>$9.75 \times 10^4$</td>
<td>$5.87 \times 10^4$</td>
<td>MPa$^{-n}$ s</td>
<td>0.76 - 6.3</td>
<td>1.1</td>
<td>209 ± 10</td>
<td>2.2 ± 0.5</td>
</tr>
<tr>
<td>Dislocation Creep</td>
<td>$3.81 \times 10^8$</td>
<td>$7.66 \times 10^8$</td>
<td>MPa$^{-n}$ μm$^{-m}$ s</td>
<td>1.3 - 7.5</td>
<td>3</td>
<td>410 ± 20</td>
<td>10.3 ± 2</td>
</tr>
</tbody>
</table>

$^a$Based off of Holyoke et al. [2014]
both studies to calculate a modified pre-exponential ($A$) value for each experiment using same flow law as Holyoke et al., [2014].

$$\dot{\varepsilon} = A\sigma^n \exp\left(\frac{E^* + PV}{RT}\right)$$  \hspace{1cm} (5)

The pre-exponential $A$ values for each experiment from both studies were grouped by their respective deformation mechanism deforming magnesite and averaged to calculate the modified $A$ value for LTP and dislocation creep (Table 4). The same method for calculating a modified $A$ value for diffusion creep was used, however, the addition of a grain size ($d = 1$ or $3 \, \mu m$ from Holyoke et al. [2014] or this study, respectively) and grain size exponent ($m = 3$) was incorporated into the flow law [Holyoke et al., 2014].

$$\dot{\varepsilon} = A\sigma^n d^{-m} \exp\left(\frac{E^* + PV}{RT}\right)$$  \hspace{1cm} (6)

4.5 Application To Nature

Magnesite can form interconnected networks of veins [Quensel et al., 2013] when carbonaceous fluids react with peridotite [Saldi et al., 2009] in a subducting oceanic slab where it remains stable into the deep mantle [Newton and Sharp, 1975; Brey et al., 1983; Canil and Scarfe, 1990; Katsura and Ito, 1990; Biellmann et al., 1993; Isshiki et al., 2004; Panero and Kabbes, 2008].

I calculated the viscosity of each deformation mechanism operating in magnesite aggregates along the pressure-temperature path of a subducting slab [Peacock et al., 2005] using the $V^*$ and $A$ values I determined and the $n$ and $H$ values determined by Holyoke et al. [2014] (Figure 11a). Assumptions made to predict the absolute viscosity of each deformation mechanism of magnesite included: (1) Changing temperature-pressure conditions experienced by subducting oceanic lithosphere [Peacock et al., 2005], (2) a differential stress of $0.01 \, \text{GPa}$ along the entire length of the subducting slab, (3) a grain size grain size exponent of $m = 3$ [Herwegh et al., 2003; Delle Piane et al., 2008]
Figure 11. Magnesite viscosity in a subduction plate. (a) Magnesite will deform primarily by diffusion creep if $d \leq 180 \ \mu\text{m}$, but if $d \geq 1000 \ \mu\text{m}$, deformation by dislocation creep will be the predominant mode of deformation at depths $\geq 180 \ \text{km}$. Magnesite deforming by LTP will be insignificant at all depths. (b) Magnesite continues to be orders of magnitude weaker than olivine and stronger than serpentine and dolomite at all depths of a subducting slab with the addition of pressure dependence unless grain sizes are close to $d = 18 \ \mu\text{m}$ in which only dolomite will be stronger along its stability field. The slope of the viscosity contrast between magnesite with grain size $d = 1000 \ \mu\text{m}$ differs from $d = 18$ and $180 \ \mu\text{m}$ at $\sim 180 \ \text{km}$ depth due to dislocation creep becoming weaker than diffusion creep.
for magnesite deforming by diffusion creep. Magnesite deforming by LTP contributes
to the deformation the least being > 15 orders of magnitude more viscous than diffusion
creep and dislocation creep, but has a \(V^*\) high enough for pressure to influence the
viscosity more than temperature at depths > 180 km. Magnesite deforming by diffusion
creep is the weakest mechanism with the strength contrast is 3 orders of magnitude
weaker than dislocation creep if grain sizes are \(\sim 18 \, \mu m\) at a depth of 400 km. However,
if grain sizes are \(\sim 180 \, \mu m\), the strength contrast of magnesite deforming by diffusion
creep and dislocation creep ranges from 10 – 0 orders of magnitude from
17 – 400 km depth, respectively. The strength of magnesite deforming by dislocation
creep is intermediate between LTP and diffusion creep if grain sizes are \(\sim 180 \, \mu m\).
However, if grain sizes are \(\sim 1000 \, \mu m\), deformation by dislocation creep will become
weaker than diffusion creep at > 180 km depth and will become \(\sim 2\) orders of magnitude
weaker at \(\sim 400 \, km\) depth (Figure 11a).

The model created by Holyoke et al. [2014] found magnesite of grain size \(\sim 180 \, \mu m\)
will be 7 – 9 orders of magnitude weaker than olivine from 200 – 400 km depth,
respectively, in a subducting slab without including activation volume in their magnesite
flow laws. With the addition of activation volumes of each deformation mechanism to
the magnesite flow laws, the strength contrast between magnesite and olivine decreases
from 7 – 9 orders of magnitude weaker to 6 – 5 orders of magnitude weaker from
200 – 400 km depth, respectively. If grain sizes are < 1000 \(\mu m\), the viscosity contrast
between olivine and magnesite decreases at a constant rate between 180 – 400 km depth.
However, once magnesite grain size \(\geq 1000 \, \mu m\), the viscosity contrast between magnesite
and olivine (\(\sim 5\) orders of magnitude) remains constant over the same range of depths
because dislocation creep becomes the dominant deformation mechanism in magnesite
aggregates. This significant strength contrast, although less than predicted by
Holyoke et al. [2014], still supports the hypothesis that strain may localize within magnesite resulting in DFE occurring by shear instabilities.

Intermediate depth DFE are not observed in all subducting plates [Karato et al., 2001]. Earthquakes are observed from the surface to the lower mantle transition in subducting plates in the Tonga and Mariana subduction zones. However, there are seismically inactive zones from ~300 – 500 km depth in the subducting plates in the Mariana, Chile, Izu-Bonin, and Banda subduction zones. Plates which are seismically active from the surface to the lower mantle transition may contain magnesite, but plates which have a seismically inactive zone likely do not contain magnesite. This variation in seismicity may be due to the variation in composition of subducting sediments or carbonated fluids from underlying mantle altering the composition of the subducting plate.
Triaxial deformation experiments were performed on fine- and coarse-grained Nevada magnesite in D-DIA and Griggs apparatuses at $P_{\text{eff}} = 0.76 - 7.5$ GPa at $T = 500, 750$, or $900 \, ^\circ\text{C}$ at $\dot{\varepsilon} \sim 1.6 \times 10^{-5}$/s. Fine-grained magnesite deformed at $500 \, ^\circ\text{C}$ characterized by high stresses (1.6, 2.2, and 3.1 GPa), angular grains, kink bands and lack of recrystallized grains is consistent with deformation by LTP. Fine-grained magnesite deformed at $750 \, ^\circ\text{C}$ characterized by low stresses (0.38, 0.6, 0.9, and 1.2 GPa), rounded grains, increased porosity, and four-point junctions is consistent with deformation by diffusion creep. Coarse-grained magnesite deformed at $900 \, ^\circ\text{C}$ characterized by moderate stresses (0.8, 1.0, 1.3, 1.6, and 1.7 GPa), fine recrystallized grains along grain boundaries, patchy undulatory extinction, and kinking at high pressures is consistent with deformation by dislocation creep. The following conclusions were made:

- The activation volumes calculated for LTP, diffusion creep, and dislocation creep of magnesite aggregates are $V^* = 33.8 \, (\pm 1)$, 2.2 $(\pm 0.7)$, and $10.3 \, (\pm 2) \times 10^{-6}$ m$^3$/mol, respectively.
- Olivine is $\geq 5$ orders of magnitude stronger than magnesite with the inclusion of pressure dependence.
- Strain may localize within magnesite resulting in DFE.
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APPENDIX A

COMPLETE LIST OF EXPERIMENTS

On the following pages, a complete list of experiments performed in my study are presented.
Apendix A. Complete list of experiments performed in this study.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Coarse/Fine</th>
<th>Temp (°C)</th>
<th>Pres (GPa)</th>
<th>Eff Pres (GPa)</th>
<th>Strain Rate ($10^{-5}$ s$^{-1}$)</th>
<th>Strain (%)</th>
<th>Peak Strength (GPa)</th>
<th>Final Strength (GPa)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAG_004</td>
<td>F</td>
<td>500</td>
<td>6.6 ± 0.2</td>
<td>6.6 ± 0.2</td>
<td>1.4</td>
<td>27</td>
<td>3.1 ± 0.25</td>
<td>3.1 ± 0.25</td>
<td>Successful experiment, Blasko et al., 2017</td>
</tr>
<tr>
<td>MAG_005</td>
<td>F</td>
<td>500</td>
<td>3.4 ± 0.1</td>
<td>3.4 ± 0.1</td>
<td>1.6</td>
<td>27</td>
<td>1.6 ± 0.25</td>
<td>1.6 ± 0.25</td>
<td>Successful experiment, Blasko et al., 2017</td>
</tr>
<tr>
<td>MAG_006</td>
<td>F</td>
<td>500</td>
<td>5.6 ± 0.3</td>
<td>5.6 ± 0.3</td>
<td>2.2</td>
<td>28</td>
<td>2.2 ± 0.25</td>
<td>2.2 ± 0.25</td>
<td>Successful experiment, Blasko et al., 2017</td>
</tr>
<tr>
<td>MAG_008</td>
<td>C</td>
<td>900</td>
<td>5.6 ± 0.1</td>
<td>5.2 ± 0.1</td>
<td>1.6</td>
<td>30</td>
<td>1.1 ± 0.2</td>
<td>1.0 ± 0.2</td>
<td>Successful experiment</td>
</tr>
<tr>
<td>MAG_010</td>
<td>C</td>
<td>900</td>
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<td>MAG_012</td>
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<td>5.8 ± 0.1</td>
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<td>1.6 ± 0.2</td>
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<td>MAG_014</td>
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<td>36</td>
<td>0.9 ± 0.2</td>
<td>0.8 ± 0.2</td>
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</tr>
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<td>6.3 ± 0.1</td>
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<td>1.3 ± 0.2</td>
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<tr>
<td>MAG_020</td>
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<td>750</td>
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<td>6.3 ± 0.1</td>
<td>0.7</td>
<td>5</td>
<td>1.2 ± 0.2</td>
<td>1.2 ± 0.2</td>
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<td>5.4 ± 0.1</td>
<td>0.5</td>
<td>6</td>
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<td>3.8 ± 0.1</td>
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<td>1.0 ± 0.2</td>
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<td>5.3 ± 0.1</td>
<td>1</td>
<td>4</td>
<td>1.1 ± 0.2</td>
<td>1.1 ± 0.2</td>
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<tr>
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<td>3.5 ± 0.1</td>
<td>1.7</td>
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<td>TC Failed</td>
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<tr>
<td>Z-94</td>
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<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>TC Failed/Salt breached into Sample/Sample too strong</td>
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<tr>
<td>Z-96</td>
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<td>Z-98</td>
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<td>-</td>
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</table>
Apendix A. Complete list of experiments performed in this study (continued).

<table>
<thead>
<tr>
<th>Experiment</th>
<th>F</th>
<th>750</th>
<th>0.85 ± 0.02</th>
<th>0.76 ± 0.02</th>
<th>2.1</th>
<th>15</th>
<th>0.50 ± 0.02</th>
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</table>
APPENDIX B

GRAPHICAL DATA

On the following pages, complete graphical data for stress-strain, stress/pressure-time, and strain-time recorded for each experiment in my study are presented.
MAG_004 Fine-grained magnesite

$T = 500^\circ$C  $P_{\text{eff}} = 6.6$ GPa

$\dot{\epsilon} = 2.5 \times 10^{-5}$ s$^{-1}$

d = 3 $\mu$m
MAG_005  Fine-grained magnesite

\[ T = 500^\circ C \quad P_{\text{eff}} = 3.4 \text{ GPa} \]
\[ \dot{\varepsilon} = 2.7 \times 10^{-5} \text{ s}^{-1} \]
\[ d = 3 \mu m \]
MAG_006  Fine-grained magnesite

$T = 500\,^\circ C$  $P_{eff} = 5.6$ GPa

$\dot{\varepsilon} = 2.8 \times 10^{-5}$ s$^{-1}$

d = 3 \, \mu m

Differential Stress vs. Strain (%)

Confining Pressure vs. Differential Stress

Strain vs. Time (min)
MAG_008  Coarse-grained magnesite

$T = 900^\circ C$  $P_{eff} = 5.2$ GPa

$\dot{\varepsilon} = 2.9 \times 10^{-5}$ s$^{-1}$

$d = 80$ $\mu$m

---

![Graphs showing stress-strain and pressure-time relationships for a magnesite sample.](image-url)
MAG_010  Coarse-grained magnesite

$T = 900^\circ$C  $P_{eff} = 7.5$ GPa

$\dot{\varepsilon} = 2.9 \times 10^{-5} \text{s}^{-1}$

d = 80 \mu m
MAG_012 Coarse-grained magnesite

\[ T = 900^\circ C \quad P_{eff} = 5.8 \text{ GPa} \]
\[ \dot{\varepsilon} = 3.3 \times 10^{-5} \text{ s}^{-1} \]
\[ d = 80 \mu m \]
MAG_014 Coarse-grained magnesite

\[ T = 900^\circ\text{C} \quad P_{\text{eff}} = 2.9 \text{ GPa} \]
\[ \dot{\varepsilon} = 3.5 \times 10^{-5} \text{ s}^{-1} \]
\[ d = 80 \text{ \textmu m} \]
MAG_016  Coarse-grained magnesite

$T = 900^\circ C \quad P_{\text{eff}} = 6.3 \text{ GPa}$

$\dot{\epsilon} = 2.9 \times 10^{-5} \text{ s}^{-1}$

$d = 80 \mu m$
MAG_020  Fine-grained magnesite

$T = 750^\circ C$

$\dot{\varepsilon} \sim 10^5 \text{ s}^{-1}$

$d = 3 \mu m$

Step 1

$P_{eff} = 6.3 \text{ GPa}$

Step 2

$P_{eff} = 5.4 \text{ GPa}$

Step 3

$P_{eff} = 3.8 \text{ GPa}$

Differential Stress (GPa)

0 0.5 1 1.5 2 2.5 3 3.5

Strain (%)

0 5 10 15 20

Differential Stress or Pressure (GPa)

0 1 2 3 4 5 6 7 8 9

Confining Pressure

Differential Stress

0 1 2 3 4 5

110 min b/w steps 1 & 2

82 min b/w steps 2 & 3

Differential Stress

0 1 2 3 4 5 6

110 min b/w steps 1 & 2

82 min b/w steps 2 & 3

Strain

0 2 4 6 8 10 12 14 16 18 20

Time (min)

0 50 100 150 200 250 300
Z-100 Fine-grained magnesite

$T = 750^\circ C$  $P_{eff} = 0.76$ GPa

$\dot{\varepsilon} = 2.1 \times 10^{-5} \text{s}^{-1}$

$d = 3 \mu m$
APPENDIX C
GRAIN ETCHING

On the following page, the technique for etching fine-grained magnesite to image in the SEM is presented. Grain boundaries of fine-grained magnesite were difficult to image in the SEM. The starting material ($d \sim 3 \, \mu m$) was etched with standard field geology 10% HCl acid for 10, 100, 1000, and 10000 seconds. It was found that etching for 1000 seconds allowed for easy viewing of grain boundaries without causing significant grain rounding or porosity increase. All magnesite samples imaged using the SEM were etched with standard field geology 10% HCl acid for 1000 seconds.