

## Final Project Report for DOE BES Grant DE-FG02-04ER15500

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In our first experiments with the deformation-DIA (D-DIA), the flow behavior of periclase (MgO), an important component in Earth's lower mantle, was investigated in order to measure the activation volume (*Mei et al.*, 2008). The activation volume quantifies the dependence of strength on pressure, which increases with increasing depth in the Earth. These experiments were some of the very first experiments carried out to high strains in compressional deformation with a D-DIA apparatus. Significant uncertainties in the values of stress and of temperature impaired our ability to measure flow parameters with sufficient accuracy unambiguously identify the dominant flow mechanism. Nonetheless, under the assumption that the mechanism of deformation the same as that in other high-temperature creep experiments carried out in conventional deformation apparatuses, our measurements indicate a positive dependence of the flow stress on pressure with a very modest value for activation volume of  $V^* \approx 2.4 \times 10^{-6} \text{ m}^3/\text{mol}$ . This work involved not only critical development the D-DIA, but also the formulation of an approach to handling a new type of input to deformation science, namely, the measurement of stress and strain rate at the grain scale using a synchrotron x-ray source. As has been observed previously in similar experiments on plastic flow of polycrystalline samples, a grain scale redistribution of differential stress occurs among different orientation populations of grains. Interpretation of the different flow laws obtained from analysis of different sets of diffraction peaks remains a topic of ongoing study. Also, the stress distribution is spatially significantly non-uniform within a sample. This observation opens the possibility of mapping *in situ* the stress distribution within a sample, as an important step in understanding from microscale measurements the macroscale deformation behavior of rocks.

During the course of our work, technological advances in hardware, software, and infrastructure associated with D-DIA experimentation, advances with which we were ourselves deeply involved, brought continuous improvements to efficiency, measurement precision, and ease of operation, and attracted more and more interest in the field (*Weidner et al.*, 2010). Synchrotrons on four continents are now equipped with D-DIA capability. We replaced key components of the x-ray optics to improve resolution and ease of alignment and calibration. We upgraded controlling and data reduction software that changed a labor-intensive, manual experimental procedure to one that can run in fully automated mode for hours at a time; and reduced the data reduction cycle (from gathering diffraction spectra to determining the state of stress) from several weeks to a few minutes.

We developed very specifically for our own interest in olivine an anhydrous cell assembly that for the first time prevented drift in the water content of samples. The flow strength of

olivine is notoriously affected by trace amounts of water. With the development of the new sample assembly, it became possible to pursue two fundamental studies of the strength of olivine in the D-DIA. In the first, the dependence of the viscosity of olivine rocks on pressure, that is, the activation volume was measured (*Durham et al.*, 2009). Most previous studies were limited to pressures of <0.5 GPa. Our experiments extended this pressure range by a factor of ten, thus greatly increasing the resolution. This study was the first to use the newly designed sample assembly that insured that experiments were carried out under anhydrous conditions. Previous studies were complicated by the lack of control of water content; even worse, the water content varied during most previous experiments, generally decreasing but, sometimes, increasing as the experiment progressed. Interpretation of results from such experiments is impossible and misleading, as indicated by the wide range of reported values for activation volume extending from  $\sim 0$  to  $30 \times 10^{-6} \text{ m}^3/\text{mol}$ . With the new sample assembly, this problem was avoided. The resultant value for  $V^*$  was  $(10 \pm 7) \times 10^{-6}$ . This study enabled us to benchmark our results from D-DIA experiment with those from higher experiments carried out in resolution, lower pressure instruments. This comparison demonstrated that the flow stress (that is, strength at a given strain rate) of olivine determined with the D-DIA is approximately the same as would be observed in the gas apparatus at identical conditions, thus validating the accuracy of stress measured at high pressure. Better resolution of the flow-law parameters with the D-DIA is now possible with enhancement made to the x-ray detector systems that enable greater precision in stress, and a follow-up study to these preliminary experiments was carried out as described below.

In the second part of our study of deformation of olivine rocks, emphasis was focused on experiments that would yield constraints on the strength of the lithosphere, the relatively strong outer layer of the Earth (*Mei et al.*, 2010a; *Hilaitet*, 2011; *Kohlstedt and Hansen*, 2015). This investigation was particularly well suited for D-DIA experiments as the strength of the lithosphere is relatively large, such that stress resolution is not a limitation. Prior to our study, the primary data used to constrain the deformation behavior of the lithosphere came from microindentation tests in which the rate of deformation is now well constrained. Our experimental results yielded a flow law with well-resolved values of activation energy (i.e., dependence of strength on temperature), theoretical yield strength (often call the Peierls stress), and flow parameters relating strain rate to applied stress. This flow law has since been implemented in a number of geodynamical models of flexure of the lithosphere in response to loading by tectonic stresses.

In the first half of our investigation of the strength of garnet rocks at high pressures and high temperature, experiments were performed to quantify the dependence of viscosity on pressure as well as temperature using the D-DIA (*Mei et al.*, 2010b). Differential stress and pressure were measured using X-ray diffraction techniques based on the elastic strain of

various lattice planes as a function of orientation with respect to the applied stress field. The plastic strain of a deforming sample was monitored in-situ through a series of radiographs. Our results provide a measure of the dependence of creep rate of garnet on the temperature with an activation energy of  $\sim 280$  kJ/mol and on pressure with an activation volume of  $\sim 10 \times 10^{-6}$  m<sup>3</sup>/mol. The volume fraction of garnet is significant in regions along subducting slabs and in the transition zone such that the deformation behavior of garnet plays an important role in mantle dynamics. As examples, the volume fraction of garnet in the major rock compositions in these regions (i.e., eclogite, pyrolite and piclogite) ranges from  $\sim 50\%$  to  $80\%$ . Thus, garnet has an important influence on viscosity structure. Furthermore, in the garnetite layer that subducts in descending oceanic lithosphere and possibly deflects laterally into the transition zone, garnet constitutes more than  $90\%$  of the rock; hence, garnet controls its strength. Therefore, the flow law generated from this study provides a solid constraint on the rheological response of those garnet-rich regions in the Earth. In particular, quantification in this study of the influence of pressure on the viscosity of garnet is critical for extrapolating laboratory results to geological conditions. The flow behavior of garnet quantified by this study provides the basis for modeling geodynamic processes occurring within subducted lithosphere.

The second portion of our research on garnet concentrated on the role of water on the viscosity of this important mineral (*Xu et al.*, 2013). Our experimental results demonstrate that water has a significant influence on the creep strength of garnet aggregates in the dislocation creep regime. Most previous investigations of the microstructures of naturally and experimentally deformed samples emphasized the high creep strength of garnet, consistent with quantitative experimental studies carried out under anhydrous conditions. However, based on the results from our study, the lower creep strength of garnet in the naturally deformed garnet-rich rocks from the shallow upper mantle reported in previous studies indicates the important role of water weakening. Thus, the flow law for garnet samples deformed in the dislocation creep regime under hydrous condition in our study provides an important constraint on the viscosity of the garnet-rich layer of the subducted oceanic lithosphere in a subduction zone and in the transition zone, where water can be enriched. Our experimental results demonstrate that the viscosity of a garnet-rich layer in a subducting slab is lowered by the presence of water in the shallow upper mantle. However, although garnet becomes weaker with increasing depth down to  $\sim 100$  km, it then becomes strong with further increase in depth. This transition occurs at about the pressure at which water solubility begins to decrease with increasing pressure. Furthermore, at depths greater than  $\sim 170$  km, anhydrous garnet becomes weaker than hydrous garnet, reflecting the relatively large value for activation volume determined for hydrous conditions. Thus, in the deeper part of the upper mantle, the viscosity of subducting oceanic crust is governed by the creep of dry garnet since the water weakening effect fades away.

The deformation behavior of enstatite rocks was investigated with creep experiments at pressures of 3.8 to 6.3 GPa and temperatures of 1323 to 1573 K using a deformation-DIA apparatus (*Zhang and Mei, 2016*). Experiments were carried out at the National Synchrotron Light Source at Brookhaven National Laboratory and Argonne National Laboratory. Differential stress and sample displacement were monitored in-situ using synchrotron x-ray diffraction and radiography, respectively. The flow law developed from our experimental data describes the dependence of deformation rate on stress, water fugacity, temperature, and pressure. Specifically, creep rate depends on stress with  $n \approx 1$ , indicating that samples deformed in the diffusion creep regime. Dependences of creep rate on temperature and pressure with an activation energy of  $\sim 200$  kJ/mol and activation volume of  $\sim 14 \times 10^{-6}$  m<sup>3</sup>/mol, respectively. The water weakening effect for enstatite is smaller than for olivine and clinopyroxene.

Finally, the effect of pressure on the creep strength of olivine under anhydrous conditions was systematically investigated with a series of newly designed experiments (*Dixon, 2014; Dixon and Durham, 2016*). An obstacle to understanding the influence of olivine on mantle viscosity has been the difficulty of determining the effect of pressure on its high-temperature creep strength. The bulk of previous studies examining  $V^*$  were conducted over small ranges of pressure at low pressure ( $< 300$  MPa), limiting precision and raising questions about application to deep geological conditions. New generation high-pressure apparatus such as the D-DIA now operate to 15 GPa or more, allowing access to the full range of upper mantle pressures. As a practical matter, however, high-pressure creep experiments in the D-DIA on samples with very small volumes (order of 1 mm<sup>3</sup>) are plagued by resolution issues related to temperature measurement, water content (i.e., water weakening), and mechanical disruption within the deformation column to the simple axisymmetry of the applied load. Therefore, dry polycrystalline San Carlos olivine samples were deformed with the focused intent of making the best achievable measurement of  $V^*$ , varying only pressure (1.5 to 9 GPa) while holding temperature (1373 K) and strain rate ( $1 \times 10^{-5}$  s<sup>-1</sup>) approximately fixed, using our anhydrous cell assembly (*Durham et al., 2009*) to prevent water uptake by the sample, and having a thermocouple-free deformation column for minimum mechanical disruption. Stress and strain were measured in-situ with synchrotron x-rays. Refinement of diffraction techniques has allowed stress resolution of  $\pm 0.02$  GPa (*Weidner et al., 2010*). For the pressure range in this study, an activation volume of  $(16 \pm 5) \times 10^{-6}$  m<sup>3</sup>/mol was determined for dry polycrystalline San Carlos olivine. This value indicates a substantial pressure effect, which in the absence of a temperature gradient would represent a large viscosity increase across the range of pressures from the top to bottom of the upper mantle. The diffraction technique used for stress measurement in these experiments also illuminates the relative strength of differently oriented grains in our polycrystalline sample, providing new experimental evidence for change in dominant dislocation slip systems in olivine with pressure.

### **Publications of Research Supported by DOE BES Grant DE-FG02-04ER15500**

- “Pressure sensitivity of creep of dry olivine at upper mantle conditions”, N. A. Dixon and W. B. Durham, *in preparation*, (2016)
- “Creep behavior of enstatite at high pressures”, G. Zhang and S. Mei, *in preparation*, (2016)
- “Constitutive equations, rheological behavior, and viscosity of rocks”, D.L. Kohlstedt and L.N. Hansen, in *Treatise on Geophysics*, 2<sup>nd</sup> edition, ed. G. Schubert, Oxford: Elsevier, vol. 2, pp. 441-472 (2015) <http://dx.doi.org/10.1016/B978-0-444-53802-4.00042-7>
- “Experimental Constraints on the Rheological Behavior of Olivine at Upper Mantle Conditions”, N. A. Dixon, Ph. D. Thesis, Massachusetts Institute of Technology, 125 pp (2014).
- “Effect of water on rheological properties of garnet at high temperatures and pressures”, L. Xu, S. Mei, N.A. Dixon, Z. Jin, A. Suzuki, and D.L. Kohlstedt, *Earth Planet. Sci. Lett.*, **379**, 158-165 (2013) <http://dx.doi.org/10.1016/j.epsl.2013.08.002>
- “Deformation mechanisms of olivine under upper mantle conditions from in-situ, high-pressure deformation using monochromatic synchrotron radiation”, N. Hilairet, Y. Wang, T. Sanehira, S. Merkel, C. Tomé, and S. Mei, *J. Geophys. Res.*, **117**, B01203 (2011) [doi:10.1029/2011JB008498](http://dx.doi.org/10.1029/2011JB008498)
- “Precise stress measurements with white synchrotron x rays”, D.J. Weidner, M.T. Vaughan, L.P. Wang, H B. Long, L. Li, N.A. Dixon, and W.B. Durham *Rev. Sci. Instrum.*, **81**(1), 013903, (2010) [doi:10.1063/1.3263760](http://dx.doi.org/10.1063/1.3263760)
- “Experimental constraints on the strength of the lithospheric mantle,” S. Mei, A.M. Suzuki, D.L. Kohlstedt, N.A. Dixon, and W.B. Durham, *J. Geophys. Res.* **115**, B08204, (2010a) [doi:10.1029/2009JB006873](http://dx.doi.org/10.1029/2009JB006873)
- “Experimental investigation of the creep behavior of garnet at high temperatures and pressures”, S. Mei, A.M. Suzuki, D.L. Kohlstedt, and L. Xu, *J. Earth Sci.* **21**, 532-540 (2010b) [doi:10.1007/s12583-010-0127-8](http://dx.doi.org/10.1007/s12583-010-0127-8)
- “New measurements of activation volume in olivine under anhydrous conditions”, W.B. Durham, S. Mei, D.L. Kohlstedt, L. Wang, and N.A. Dixon, *Phys. Earth Planet. Inter.* **172**, 67-73, (2009) [doi:10.1016/j.pepi.2008.07.045](http://dx.doi.org/10.1016/j.pepi.2008.07.045)
- “Experimental investigation of the creep behavior of MgO at high pressures”, S. Mei, D.L. Kohlstedt, W.B. Durham, and L. Wang, *Phys. Earth Planet. Inter.* **170**, 170-175, (2008) [doi:10.1016/j.pepi.2008.06.030](http://dx.doi.org/10.1016/j.pepi.2008.06.030)