Pressure and strain dependence of the strength of sintered polycrystalline Mg$_2$SiO$_4$ ringwoodite

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1. Introduction

[1] Differential stresses in a cylindrical rock of polycrystalline Mg$_2$SiO$_4$ ringwoodite were measured at room temperature with pressures up to 10 GPa, axial strains in excess of 20%, and strain rates between $5 \times 10^{-5}$ and $4 \times 10^{-6}$ s$^{-1}$, using the deformation-DIA coupled with monochromatic X-rays. The sample exhibited ductile behavior in axial shortening-lengthening cycles with reproducible hysteresis loops, yielding multiple well-defined stress-strain curves. Significant strain hardening was observed beyond the yield point, which occurs at axial strains around 1.5%. Large discrepancies in stress data on ringwoodite reported in previous studies, where no strain information could be obtained, can be reconciled by the strain hardening behavior. Above 8% axial strain, sample stresses reach saturation and the deformation reaches steady state, during which process the ultimate strength increases with hydrostatic pressure but are insensitive to strain rate, suggesting that the sample deforms in low-temperature plasticity regime. Citation: Nishiyama, N., Y. Wang, T. Uchida, T. Irifune, M. L. Rivers, and S. R. Sutton (2005), Pressure and strain dependence of the strength of sintered polycrystalline Mg$_2$SiO$_4$ ringwoodite, Geophys. Res. Lett., 32, L04307, doi:10.1029/2004GL022141.

2. Experimental

[4] The deformation experiment was performed at the GSECARS 13-BM-D beamline (Advanced Photon Source) using D-DIA with monochromatic diffraction (wavelength 0.191 Å) and a radiographic imaging system (see details in work by Wang et al. [2003] and Uchida et al. [2004]).

[5] We used four sintered cubic boron nitride (cBN) anvils with truncated edge length (TEL) of 3 mm. These X-ray transparent anvils permitted observation of diffraction Debye rings over the entire 360° detector azimuth range, perpendicular to the incident beam direction. Two-dimensional (2-D) diffraction patterns were collected using an X-ray charge-coupled device (CCD) detector. Detector orientation relative to the incident beam was calibrated using a diffraction standard (CeO$_2$) and the detector-sample distance was determined by matching the observed ambient $d$-values of the sample inside the D-DIA to those reported by Sasaki et al. [1982]. The sample length was measured by radiography using a wide X-ray beam, by driving the WC slits out of the beam path. The cell assembly used in the present study was similar to that described by Uchida et al. [2004]. We used a pre-sintered, fully densified polycrystalline cylindrical specimen (0.8 mm in diameter and 1.2 mm in length), synthesized at 20 GPa and 1523 K using Orange-3000 (Kawai-type apparatus) at Geodynamics Research Center, Ehime University.

[6] Data reported here were collected from a single run, in which five shortening-lengthening deformation cycles exhibited microstructure associated with dislocation creep, whereas fine-grained samples showed evidence for diffusion creep or superplasticity. Wenk et al. [2004] made in-situ observation of texture development in ringwoodite in the diamond anvil cell (DAC) and determined active slip systems responsible for the observed fabric.
were carried out at a ram load of 30 T, and four more cycles at 50 T, by advancing and retracting the differential ram pistons at various speeds. In each cycle, an average strain at 50 T, by advancing and retracting the differential ram were carried out at a ram load of 30 T, and four more cycles were carried out at a ram load of 30 T, and eight at 50 T, with solid and open symbols denoted to indicate the cycles.

3. Results and Discussion

Representative X-ray diffraction patterns, converted from the original form in polar coordinates into Cartesian systems, are shown in Figure 1, where the horizontal and vertical axes are 2θ and detector azimuth χ (χ = 0 is parallel to σ1), respectively, with intensity represented by darkness. In Figure 1a (ambient; stress free), the positions of each diffraction line exhibit no χ dependence, indicating no residual stress in the starting sample. There is also no intensity variation with χ, indicating no preferred orientation. Figures 1b and 1c are patterns collected during shortening and lengthening of the first cycle at 50 T, respectively. Here the peak position of every diffraction line clearly varies with χ. The maxima in 20 angles at χ ~ 90 and 270° in Figure 1b correspond to the direction of the compressive principle stress σ1, with σ1 > σ3, where the sample underwent axial shortening. The minima in 20 at χ ~ 90 and 270° in Figure 1c correspond to σ1 < σ3, under which condition the sample underwent axial lengthening. Intensity variation with χ can also be clearly observed in Figures 1b and 1c. For example, intensities of the (440) peak show maxima at χ = 30, 90, 150, 210, 270, and 330°, with well developed six-fold symmetry. These observations have been used to determine texture development in ringwoodite during deformation. Based on this analysis, the deformation is dominated by the (111) (−110) slip system. Details of the analysis will be reported in a separate paper (H.-R. Wenk et al., Texture development and deformation mechanisms in ringwoodite, manuscript in preparation, 2005). Figure 1d is a diffraction pattern collected from the recovered sample. No χ-dependence in peak positions is observed, indicating that stress has been completely released.

Figures 2a and 2b show selected stress-strain curves at 30 and 50 T, respectively. Here, l0 is chosen to be the length at the beginning of the first deformation cycle under each ram load. For clarity, only the stresses determined from the (311) reflection are shown; other reflections show very similar behavior. A clear hysteresis behavior is observed from these curves. Ringwoodite is readily deformed in ductile regime even at room temperature. There is no evidence for fracture, such as a sudden drop of differential stress or sample length, during the deformation.

In order to examine more closely the stress-strain behavior, we decompose the curves into individual shortening and lengthening segments, and redefine l0 for each segment as the point where t is zero. It was necessary to perform interpolation in most cases to find the appropriate l0. Figures 3a and 3b show curves for (311) after l0 has been redefined. There are ten independent stress-strain curves at 30 T, and eight at 50 T, with solid and open symbols

Weidner et al., 2004]. Uchida et al. [2004] have demonstrated that this approach has yielded stress-strain relations similar to those obtained in a conventional deformation experiments for MgO. We used ambient single-crystal elastic moduli determined by Weidner et al. [1984] and the pressure derivatives by Sinogeikin and Bass [2001], assuming that the small amount of Fe (9%) present in the latter study does not affect the derivatives. The total sample axial strain is calculated using ε_total = (l0 - l)/l0, where l is the sample length measured during deformation, and the choices of the reference length l0 are discussed below.
representing data in shortening and lengthening, respectively. These curves are a good representative of the entire sample because the values of differential stresses determined using (311) are essentially identical to the average stresses determined by three major independent reflections of (311), (400), and (440) (see insets in Figures 3a and 3b).

In Figures 3a and 3b, all stress-strain curves exhibit a consistent trend. Stresses vary linearly with strain until \( \epsilon_{\text{total}} \) reaches about 1.5%, where curves start deviating from linearity. We define this point as the yield point \([Uchida et al., 2004]\). Below the yield point, ringwoodite deforms in the elastic regime, where all of the data points fall in a well-defined straight line. Beyond the yield point, the differential stress versus strain relation exhibits a strong non-linearity, indicating strain-hardening. Eventually, stresses are saturated when \( \epsilon_{\text{total}} > 8\% \). We refer to the stress after the attainment of steady-state flow as the ultimate strength.

The data points collected in the plastic regime show a range of stresses, which can be attributed to effects of pressure and total strain in different deformation cycles. Figure 4 shows stresses at selected sample strains (2, 5, and 10%) as a function of pressure. These stresses increase with pressure and axial strain, until the ultimate strength is reached. Changing strain rate from \( 5 \times 10^{-5} \) to \( 4 \times 10^{-6} \) s\(^{-1} \) has little effect on the ultimate strength under these conditions (see Figures 2 and 3), suggesting that deformation occurs in the low temperature plasticity regime at these pressure and temperature conditions \([Frost and Ashby, 1982]\). Both shortening and lengthening data show a consistent trend. Thus we conclude that stress-strain curves can be reproduced by repeated cycles at various pressures in a single experiment.

Several previous studies reported high-pressure strengths of polycrystalline ringwoodite at room temperature \([Meade and Jeanloz, 1990; Chen et al., 1998; Kavner and Duffy, 2001]\). There is a large discrepancy among the reported values, which may be attributed to grain size, strain rate, and total strain \([Kavner and Duffy, 2001]\). In those studies, both strain and strain rate were not controlled parameters and could not be measured. Our results, obtained in the D-DIA where differential stress, total strain, and strain rates are controllable and measurable, show that ringwoodite deforms with significant strain hardening at high pressures, before reaching the ultimate strength.

The inset of Figure 4 compares differential stresses measured in this study with those reported in previous studies. The data of Meade and Jeanloz [1990] were based on characterizing pressure gradients in the DAC. Their stress levels are much higher than those determined by all other studies, including this study. Possible reasons for this discrepancy have already been discussed by Kavner and Duffy [2001]. The trend of our data at 10% strain, where steady state flow was observed, is in general agreement with the DAC results of Kavner and Duffy [2001].

![Figure 2](image-url) **Figure 2.** Representative stress-strain curves of ringwoodite at (a) 30 and (b) 50 T. Stresses were determined using the (311) reflection and the reference sample length \( l_0 \) is chosen to be the length at the beginning of the first deformation cycle under each ram load. Strain rates \((\dot{\epsilon})\) of some deformation segments are shown: numbers represented by bold italic are average strain rates when the sample exhibits steady-state flow; the others are average strain rates throughout whole deformation segment (from elastic to plastic).

![Figure 3](image-url) **Figure 3.** Representative decomposed stress-strain curves of ringwoodite at (a) 30 and (b) 50 T. Stress was determined using the (311) reflection. Each curve is labeled by a numeric followed by a letter. For example, 4C and 4E represents the 4th shortening and lengthening segments, respectively. Solid and open symbols represent data points collected in shortening and lengthening, respectively. Numbers in the parentheses represent average hydrostatic pressures above 5% total strain. Insets show stress-strain curves of the fourth compression at each ram load; stresses were determined using all of usable reflections. Error bars are smaller than the size of the symbols.
Figure 4. Pressure and strain dependence of differential stress of ringwoodite. The present data are represented at three selected strains: 2% (diamonds), 5% (triangles) and 10% (circles). Closed and open symbols indicate data from compression and extension cycles, respectively. Solid lines represent linear least-square fits of data at 2 and 10%; the dotted line is a guide to the eye for the 5% data. Error bars for pressure and differential stress represent linear least-square fits of data at 2 and 10% strain. A T-cup apparatus was used in the present study, where stresses were generated by grain-to-grain contact in compressing a powdered sample and determined by the broadening of diffraction line widths. It appears that the total strain generated in their samples was limited and hence ultimate strength was not achieved.

Acknowledgments. We thank two anonymous reviewers for their constructive comments. GeoSoilEnviroCARS is supported by the National Science Foundation - Earth Sciences (EAR-0217473), Department of Energy - Geosciences (DE-FG02-94ER14466) and the State of Illinois. Use of the APS was supported by the U.S. Department of Energy, Basic Energy Sciences, Office of Energy Research, under Contract No. W-31-109-Eng-38. N.N is partly supported by Postdoctoral Fellowships for Research Abroad of Japan Society for the Promotion of Science.

References


Sinoeikin, S. V., and J. D. Bass (2001), Single-crystal elasticity of γ(Mg0.91Fe0.09)2SiO4 to high pressures and to high temperatures, Geochem. Phys. Earth. Sci. Lett., 8, 4325–4338.


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