

Laboratory Studies of the Rheological Properties of Minerals under Deep-Mantle Conditions

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Most large-scale geological processes, such as mantle convection and plate tectonics, involve plastic deformation of rocks. However, quantitative experimental studies of plastic properties under deep-mantle conditions are challenging, and major progress in this area has often been associated with the development of new techniques. Until very recently, reliable studies have been conducted only at pressures less than ~0.5 GPa (~15 km depth in Earth). By combining novel techniques of synchrotron-based in situ stress-strain measurements with newly designed high-pressure apparatuses, a new generation of experimental studies of plastic deformation of minerals under deep-mantle conditions is emerging. These studies constrain the pressure dependence of deformation of minerals such as olivine and the slip systems in high-pressure minerals such as wadsleyite and perovskite. These results have important implications for the depth variation of mantle viscosity and the geodynamic interpretation of seismic anisotropy.

KEYWORDS: rheology, deep mantle, plastic deformation, seismic anisotropy, DDIA, RDA

INTRODUCTION

Plastic deformation of rocks occurs during translational motion of rock masses associated with large-scale geologic phenomena such as mantle convection, plate tectonics, and the formation of mountain belts. Consequently, knowledge of the rheological properties of minerals, particularly mantle minerals, is critical to understanding these geodynamic processes.

The rheological properties we need to know for geodynamical applications include the relationships between the creep strength (or strain rate) and various physical/chemical parameters, as well as the relationship between deformation conditions and the microstructures they produce. In both cases, experimental studies play a major role, but the experimental approach is not straightforward because of the complex nature of rheological properties and the large difference in timescale between geological deformation and deformation in laboratory experiments. Consequently, the development of new techniques and the careful evaluation of the validity of extrapolating experimental data to geological conditions have played a major role in the advancement of our knowledge of rheological properties.

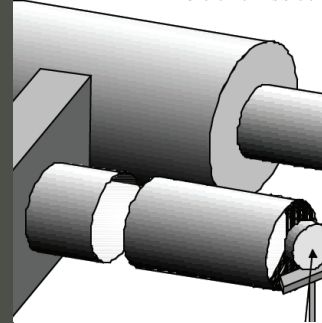
In this article, we present a brief summary of the nature of experimental studies on rheological properties and follow with a historical review of experimental studies of plastic deformation. Then we describe the development of new experimental techniques at pressures above 10 GPa (gigapascals), and last we summarize the important issues that need to be explored. Due to limited space, we focus on experimental studies of rheological properties under deep-mantle conditions. For a more comprehensive review, the reader is referred to textbooks by Karato (2008) and Poirier (1985).

WHAT DO WE NEED TO KNOW FROM LABORATORY STUDIES?

Experimental studies of plastic deformation present unique challenges compared to investigations of other properties, such as equations of state or elasticity. First, unlike elastic deformation, plastic deformation can occur by a variety of mechanisms (for details of deformation mechanisms see Frost and Ashby 1982). All of them involve motion of point defects, dislocations, and grain boundaries. These defects can be involved in plastic deformation either in isolation or in combination, and for each mechanism of deformation a particular type of flow law applies, with specific material constants. Important deformation mechanisms that may operate in Earth's mantle include (1) *diffusion creep* caused by stress-induced diffusion of atoms and (2) *dislocation creep* caused by thermally activated motion of dislocations. Dislocation motion occurs in a variety of ways, thus further complicating the plastic deformation processes. At relatively low stress levels, the rate of deformation by dislocation motion is proportional to some power of stress (*power-law creep*), whereas at high stress levels, the rate of deformation becomes an exponential function of stress (the Peierls mechanism, which applies to most silicates). Depending on the physical/chemical conditions and microstructures such as grain size, different mechanisms play an important role. Therefore in order to obtain experimental results that can be applied to Earth, one needs to make sure that the mechanisms studied in the lab are the same as those that may operate in Earth. Second, because plastic deformation occurs via a thermally activated motion of defects, the rate of plastic deformation is very sensitive to temperature, pressure, and chemical environment (such as water fugacity), and sometimes to grain size, and one needs to explore the influence of all these parameters.

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In most laboratory studies, deformation at “steady state” is emphasized. However, in some cases, deformation becomes unstable and localized in narrow shear zones. Processes of shear localization are also important, particularly in understanding the origin of plate tectonics (e.g. Bercovici 2003) and the deformation of deep subducting slabs (e.g. Karato et al. 2001).

EARLY EXPERIMENTAL STUDIES OF THE PLASTIC DEFORMATION OF MINERALS

First Generation of Rock-Deformation Studies

David Griggs at UCLA made a seminal contribution to the study of rock deformation in the 1960s (summarized by Heard et al. 1972). The effort at this stage focused on plastic deformation as opposed to brittle fracture, and therefore a confining pressure was used in order to suppress brittle fracture. For this purpose Griggs developed a high-pressure, high-temperature deformation apparatus by modifying the piston–cylinder-type high-pressure apparatus. Using this equipment (called the Griggs apparatus), the plastic properties and microstructural development of rocks were investigated to ~3 GPa and ~1600 K. Griggs and his coworkers deformed a large number of natural rocks and single crystals of minerals at high pressures and temperatures, and they compared their experimental observations with the microstructures of naturally deformed rocks and with seismological observations (e.g. seismic anisotropy). Most of the important concepts in the plastic deformation of rocks were established in these pioneering works, concepts such as the non-linear relationship between stress and strain rate (power-law creep), the weakening effect of water, the development of deformation fabrics (lattice-preferred orientation, LPO), and grain-size refinement (reduction) by deformation.

Second Generation of Rock-Deformation Studies

The applicability of the above-mentioned results is, however, limited because the stress measurements in these studies have very large uncertainties (stress is measured outside of a pressure vessel in this apparatus, and consequently the effect of friction is very large). As a result, rheological data at low stress levels, which are most critical to Earth-science applications, cannot be obtained using this apparatus. In addition, the use of natural rocks in these first-generation studies led to results that are very difficult to apply to Earth’s interior. In fact, experiments using “unnatural” synthetic rocks (unnaturally small grain size, unnaturally pure chemical composition) are better suited for applying results to Earth for the following reasons. First, natural rocks, almost without exception, contain various amounts of impurities, particularly hydrous minerals formed at grain boundaries as a result of weathering. The presence of hydrous minerals results in excess water at high temperatures and often leads to partial melting. Excess water and partial melting have important influences on rheological properties, but these factors are not well controlled if a natural rock is used as a sample. Second, the grain size of natural rocks is large in most cases. Because strain rates in laboratory experiments are much greater than those in Earth, stress levels in laboratory experiments are inevitably higher than those in Earth. Consequently, grain-size-sensitive creep processes that may play an important role in Earth cannot be observed in the laboratory if a natural sample is used. The use of synthetic rocks with smaller grain size and well-characterized composition (e.g. water content) provides us with data that can be applied to Earth with greater confidence.

Recognizing these issues, efforts over the next ~20 years (from the mid-1970s to the 1990s) focused on high-resolution experiments under lower stresses using well-characterized

synthetic polycrystalline aggregates or single crystals. In order to improve the resolution of stress measurements, lower-pressure apparatuses were used. For example, a room-pressure high-temperature creep apparatus was used for single-crystal studies (e.g. Bai et al. 1991). The applicability of results from room-pressure studies is very limited: the influence of water, for example, cannot be investigated because the water effect is too small to be detected at room pressure. In the gas-medium high-pressure deformation apparatus designed by Paterson (Paterson 1990), a sample is placed under a confining pressure of less than ~0.5 GPa and a temperature of up to ~1600 K, and a differential stress is applied and measured by a load cell (a device for the measurement of a force) inside the high-pressure chamber. Because the load cell is in the pressure vessel, there is no problem of friction, and high-resolution (better than ~1 MPa) measurements of stress can be made. Because of some confining pressure, the effect of water can be measured although it is not large at low pressures. Through these studies with synthetic samples, the influence of water and grain size was characterized (Karato et al. 1986). A number of studies were performed following this approach to obtain high-quality data on the rheological properties of minerals and rocks (e.g. Mei and Kohlstedt 2000).

These data provided a basic framework for understanding the rheological behavior of minerals in Earth’s interior: (1) For most mineral aggregates, the likely conditions in Earth’s mantle are close to the boundary between diffusion and dislocation creep. This means that significant grain-size reduction will lead to rheological weakening. Therefore it is important to understand the physical processes controlling the grain size of rocks. To evaluate the net effects of weakening, one needs to investigate grain size as determined by dynamic recrystallization (deformation-induced grain-size refinement), grain size after a phase transformation, and grain-growth kinetics. (2) The water (hydrogen) content has a strong influence on the creep strength of most rocks. Consequently, partial melting that will remove water from minerals (dehydration partial melting) will modify the creep strength of a rock considerably (Karato 1986; Hirth and Kohlstedt 1996). (3) Large-strain deformation of a rock leads to lattice-preferred orientation (LPO), which causes seismic anisotropy. This happens when deformation occurs by dislocation glide, but not by diffusion. However, the relation between LPO and deformation geometry is complicated in a material where deformation occurs along various crystallographic directions on various crystallographic planes (“slip systems”). In these cases, the relation between LPO and flow geometry can change with physical/chemical conditions.

LIMITATIONS OF EARLY STUDIES AND IMPROVEMENTS TO AN OLD APPARATUS

Although these high-resolution results form a basis for understanding the rheological behavior of rocks in the mantle, the low maximum pressure that can be achieved by this type of deformation apparatus poses severe limitations on the applicability of data obtained in this way. Note that the pressure range that can be reached by a gas apparatus is very limited compared to the pressures in Earth (Fig. 1). Because the maximum pressure is limited to such a low value, even the rheological properties of the lower continental crust (~30–70 km thick, i.e. ~1–2 GPa) cannot be investigated in any detail. In addition to this obvious limitation, there is a serious limitation to using low-pressure data in investigating the weakening effects due to water: results from pressures lower than ~0.5 GPa cannot be extrapolated to higher pressures due to the change in the thermodynamic properties of water at around ~0.5 GPa (Karato 2006).

Improvements to the Griggs apparatus and other high-pressure deformation apparatuses have also been made by Green and his colleagues, who developed a new sample assembly to reduce the uncertainties in stress measurements (e.g. Tingle et al. 1993). However, the maximum pressure range that one can explore with the Griggs apparatus is limited to ~3 GPa. Thus the rheological properties of more than 90% of Earth's mantle remain unexplored except in a very preliminary fashion.

WHOLE-MANTLE RHEOLOGY FROM LABORATORY STUDIES

Extending the pressure range of experimental studies of plastic deformation is therefore critical to understanding the rheological properties of Earth deeper than ~20 km. Two of the first attempts at extending the pressure range in rheological experiments were made by Kinsland and Bassett (1977) and Sung et al. (1977), who used a diamond anvil cell (DAC) as a deformation apparatus. The maximum pressure at which rheological studies have been performed using a DAC exceeds 200 GPa (Mao et al. 1998), and because of the transparency of diamond for various beams, DAC techniques can be used to measure a range of properties in situ (e.g. Wenk et al. 2004). However, most of the DAC deformation experiments were conducted at low temperatures and high stresses, where deformation mechanisms are probably different from those operating in Earth (Peierls mechanism in the experiments as opposed to power-law creep in Earth).

Another technique is to modify the sample assembly in a multianvil apparatus by which differential stress is generated in the sample space (Fujimura et al. 1981; Karato and Rubie 1997). For example, Thurel and Cordier (2003) and Cordier et al. (2004) used this technique to investigate the slip systems in wadsleyite and perovskite, respectively. These experiments can be made at high temperatures, and hence deformation mechanisms in these experiments could be similar to those operating in Earth. However, deformation in these experiments is not steady state, and in most cases stress levels are very high and not well known.

NEW APPROACHES TO THE EXPERIMENTAL STUDY OF DEEP-EARTH RHEOLOGY

Having realized the limitations of these previous efforts, a series of projects has been initiated by a team of scientists including ourselves. The aim is to develop new methods for quantitatively studying rheological properties and deformation microstructures under P–T conditions equivalent to Earth's deep interior. Technical developments have involved two steps. First, because there was no apparatus in which controlled stress could be generated at a pressure exceeding ~3 GPa, we needed to design new types of deformation apparatuses that work at pressures up to at least ~10 GPa (our goal is to go beyond ~24 GPa, i.e. lower-mantle pressures) and at high temperatures. Second, we needed to determine the rheological properties (that is, to measure stress and strain in a sample) under these conditions. We chose to apply and further develop synchrotron-based stress–strain measurement techniques that have been developed by the Stony Brook team (e.g. Chen et al. 2004). An X-ray probe is capable of measuring the stress and strain in the sample directly using diffraction and imaging techniques. This method avoids the previous issues related to friction corrections.

An appropriate high-pressure deformation apparatus must meet some conditions: (1) the piston (or anvil) with which a sample is squeezed must be well supported; otherwise the piston (or anvil) will fracture; (2) in some applications, a large strain is needed in order to characterize the

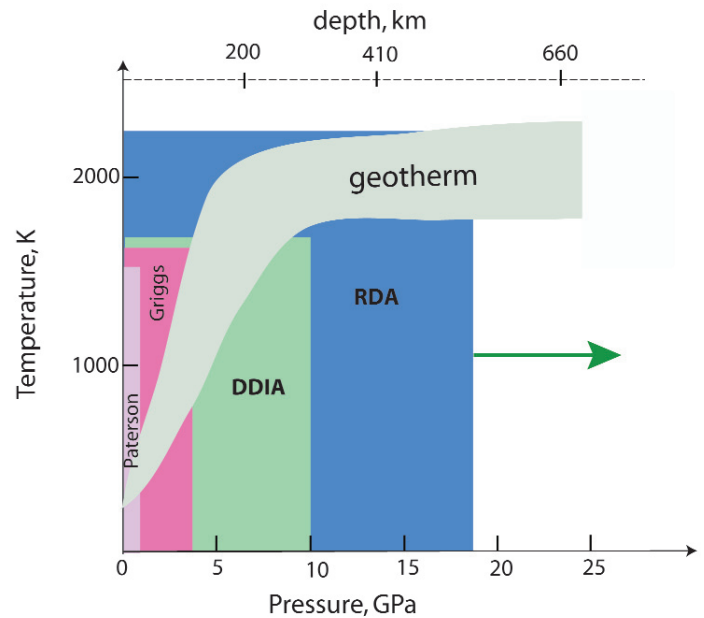


FIGURE 1 Pressure–temperature conditions in Earth (geotherm) and those achievable with high-pressure deformation apparatuses. The DDIA and RDA techniques are discussed later in the article. DDIA = deformation DIA, RDA = rotational Drickamer apparatus

microstructural developments; (3) in any apparatus to be used at a synchrotron facility, there must be a space through which X-rays can penetrate without much absorption; (4) stress and strain rates need to be applied in a controlled fashion so that steady-state rheological properties can be determined; (5) thermochemical conditions (temperature, water fugacity, etc.) must be homogeneous and well controlled; and (6) the sample volume must be large enough to explore the effects of grain size. Consideration of these points suggested to us that a large-volume apparatus is well suited to quantitative rheological studies, and two types of apparatuses have been designed and tested. These are modifications of preexisting apparatuses that were widely used with synchrotron X-rays.

The first one is a modified DIA apparatus (called DDIA; see FIG. 2A), in which deformation experiments are conducted at high pressures by moving two sets of anvils independently. This apparatus has been operated to P ~10 GPa and T ~1600 K (or to 19 GPa and ~900 K), and has provided a large amount of new data on plastic deformation under these conditions (Wang et al. 2003). This apparatus has the advantages of having a simple diffraction geometry and a relatively large and homogenous sample space, a design that allows detailed studies of rheological properties. However, since the anvils are not well supported in this design, the maximum pressure of operation with tungsten-carbide anvils is limited to ~10 GPa (at 1600 K).

The second type of equipment is a modification of the Drickamer apparatus. A rotational actuator is added to a Drickamer apparatus to conduct torsion experiments (called RDA; see FIG. 2B) (Yamazaki and Karato 2001). The torsion design was chosen for two reasons: (1) Anvils are well supported in the Drickamer apparatus, and static experiments have been conducted to ~20 GPa and ~50 GPa using tungsten-carbide anvils and sintered diamond anvils, respectively. By rotating one of the anvils, the support for the anvils is identical to that for the static high-pressure experiments, and therefore deformation experiments can be performed at high pressures, i.e. exceeding ~18 GPa (at

temperatures above ~2200 K). (2) Because of geometry, one can deform a sample to high strain in torsion experiments. This is critical to the study of microstructural developments such as LPO. However, RDA has some disadvantages compared to DDIA. Because of the torsion design, both stress and strain change as a function of distance from the center of rotation, and consequently there is some uncertainty in the measured stress and strain. Also, the sample space with RDA is rather small, and many different components are present next to the sample. Consequently, the analysis of X-ray diffraction data from an RDA apparatus is more complicated than the analysis of data obtained with DDIA equipment.

A synchrotron radiation facility produces an intense X-ray that plays an important role in high-pressure mineral physics. An intense X-ray can penetrate through a gasket material to probe a sample under high pressure and tem-

perature. The X-ray provides two types of data from a sample. One is the "image" of a sample resulting from X-ray absorption. The image provides a direct measurement of deformation geometry (total strain). The other is a diffracted signal that yields the distance between lattice planes.

The underlying principle in the stress measurement is that the distance between atoms represents the elastic strain of individual grains caused by the stress. X-ray diffraction measures the spacing between planes that are oriented so that their normal bisects the angle formed by the incoming X-rays and the diffracted X-rays. Since the diffraction angle is small (typically 6 degrees), the normal to the planes are oriented nearly perpendicular to the X-ray beam. Weidner and Li (2006) employ a slit system that allows a cone of X-rays to pass through the slit (Fig. 3). Diffracted X-rays are examined at different positions around this cone to define the lattice spacings in the sample at different orientations relative to the applied stress system. Stress is inferred from the elastic distortion of the material. That is, planes perpendicular to the applied compressional stress will be closer together than planes perpendicular to the tensional stress. Stress can be calculated from the variations of lattice spacings if the elastic properties of the material are known. This is the case when only elastic deformation is involved as assumed by Singh (1993). Recent results have shown that this is not the case and that plastic deformation also plays an important role in controlling the distribution of stress, as will be discussed below.

Some New Results

A large number of results have already been obtained using the DDIA to ~10 GPa and ~1600 K and the RDA to ~18 GPa and ~2200 K. One of the first results from the application of DDIA surprised us: stress values estimated from various lattice planes showed markedly different stress magnitudes beyond the values predicted by the model of elastic deformation (Li et al. 2004). It is likely that the large variation in stress values is caused by plastic anisotropy, and hence such data sets will give us new information on plastic anisotropy under deep-Earth conditions. In the same way, the X-ray technique for stress measurement provides us with data on stress in individual materials in a multiphase mixture (Li et al. 2006). Consequently, the stress distribution in a multiphase mixture can be investigated using this technique, and new insights into the processes of deformation of a multiphase material can be obtained.

These techniques have been applied to the plastic deformation of various materials including MgO, olivine, serpentine, iron, wadsleyite, and ringwoodite. The results for olivine (power-law creep under dry conditions) are summarized in FIGURE 4. While it is still unclear why the results of Li et al (2006) (activation volume of $0-5 \times 10^{-6} \text{ m}^3/\text{mol}$) differ from those of Kawazoe et al. (2007) ($15-20 \times 10^{-6} \text{ m}^3/\text{mol}$), experiments are in progress to test the models. Some results under deep-mantle conditions suggest possible changes in the dominant slip systems in olivine and provide some constraints on the slip systems in deep-mantle minerals (e.g. Thurel and Cordier 2003; Cordier et al. 2004; Couvy et al. 2004). However, the dominant slip systems in a given material are controlled by a number of parameters, and consequently the applicability of these results to deformation in deep Earth is unclear because only a limited range of parameter space was explored in these studies.

Future Prospects

The development of high-pressure deformation apparatuses used in combination with synchrotron X-rays is an important step forward in the experimental study of whole-Earth rheology. However, we need to be cautious before we make

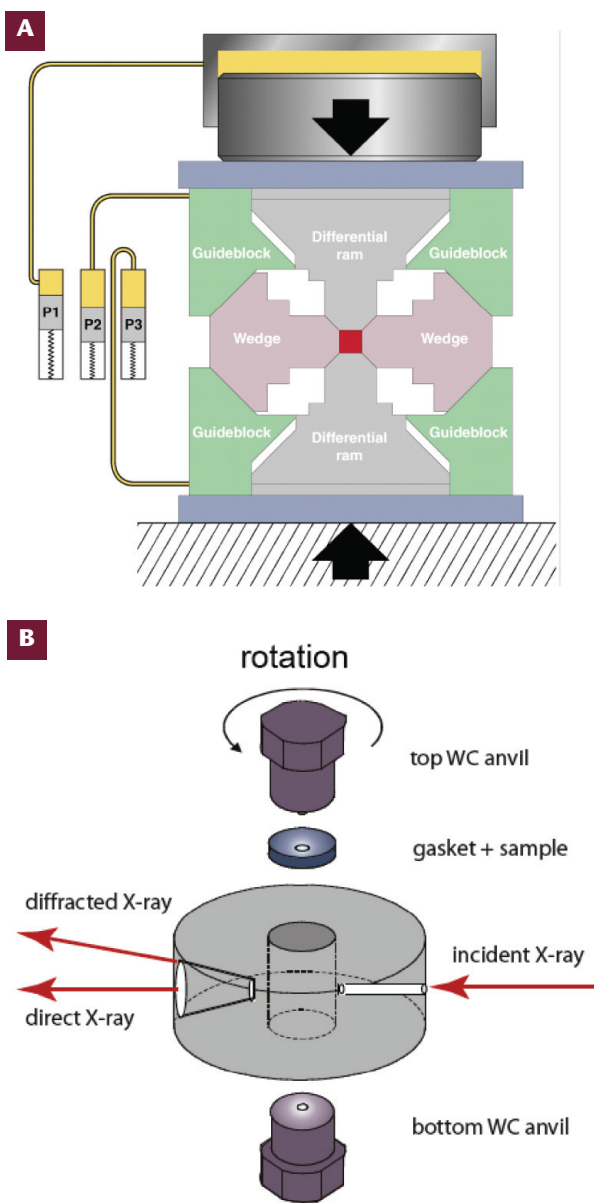


FIGURE 2 Schematic diagrams showing DDIA (A) and RDA (B) apparatuses. WC = tungsten carbide

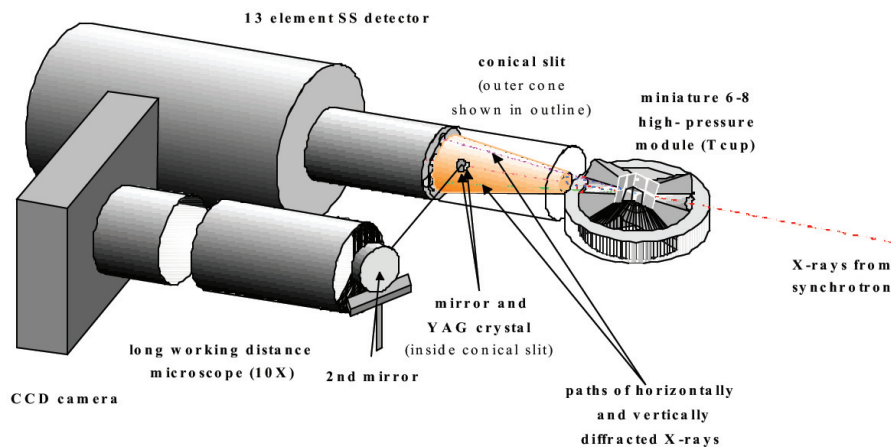


FIGURE 3 System for measuring stress by X-ray diffraction and strain by imaging. A sample is placed in the high-pressure module and struck by synchrotron X-rays. The conical slit collimates the diffracted X-ray beam at a fixed scattering angle, and the 13-element solid-state detector is placed so that some of the active detectors are exposed to the collimated beam. This beam is analyzed to define the lattice spacings. A YAG crystal is used to create an image of the sample from the through-passing X-ray beam which is imaged on a CCD camera. This produces a direct image of the sample.

any specific conclusions derived from these new results. The development of new technology for investigating high-pressure rheology is still in its infancy, and some important issues addressed in the “second-generation studies” have not been fully incorporated into these new high-pressure technologies. These issues include characterizing plastic deformation at lower stress levels and the need for a good control or at least a good characterization of water fugacity (or water content). Such issues are particularly critical for the use of the DAC in deformation studies where the control of temperature, stress, and chemical conditions is difficult. But these issues are also important (although to a lesser extent) in experiments with large-volume deformation apparatuses.

One of the fundamental problems that has been recognized through the application of these new techniques is that the widely used theory by Singh (1993) is not adequate when plastic deformation redistributes stress in individual grains. There is a need to improve the theory to quantify the influence of plastic anisotropy in the analysis of X-ray diffraction as a function of orientation (radial X-ray diffraction).

Many results using these new techniques are controversial. For example, there is a wide variation in the reported pressure dependence of olivine that must be explained. A strategy should be established to obtain geologically relevant results on plastic deformation under deep-mantle conditions. Very few quantitative data are available for plastic deformation of minerals under transition-zone and lower-mantle conditions. There is an obvious need to extend quantitative deformation experiments to higher pressures (and temperatures). Also, work on the dominant slip systems in minerals under deep-mantle conditions will have an important bearing on the interpretation of seismic anisotropy. For example, no consensus exists as to the influence of pressure on the dominant slip systems in olivine. In addition, there are few constraints on the dominant slip systems in deep-mantle minerals such as wadsleyite, perovskite, and post-perovskite (for slip systems in lower-mantle minerals, see Yamazaki and Karato 2007). A major challenge in inferring the dominant slip systems will be determining the influence of a range of important parameters.

CONCLUSIONS

In this article, we have focused our attention on technical developments in the experimental study of the rheological properties of deep-Earth minerals. Understanding the rheological properties of the whole mantle is an important step toward a better comprehension of whole-mantle dynamics,

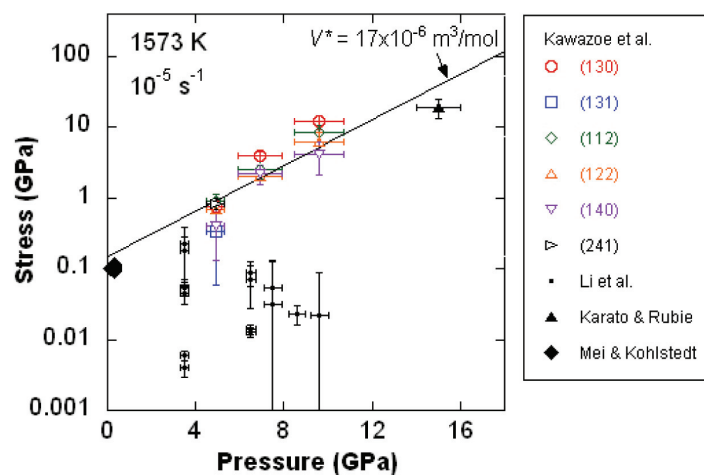


FIGURE 4 Some representative results from high-pressure deformation experiments on olivine under dry conditions (after Kawazoe et al. 2007) showing a wide range of pressure effects. Results shown by colored symbols are for steady-state deformation in power-law creep and yield an activation volume of $\sim 15\text{--}20 \times 10^{-6} \text{ m}^3/\text{mol}$. (hkl) indicates diffraction planes used to estimate the stress. The thin line shows the pressure dependence of stress for $V^* = 17 \times 10^{-6} \text{ m}^3/\text{mol}$.

and experimental investigations play a central role in the study of rheological properties. Technical development is key because no routine techniques are available for the quantitative study of rheological properties at pressures greater than a few gigapascals. Due to the complexities of rheological properties, great care needs to be taken in conducting rheological experiments and applying these results to geological problems. Only with the development and careful application of novel techniques will important new experimental results on rheological properties and deformation microstructures be obtained to help us better understand the dynamics and evolution of Earth and other terrestrial planets.

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GLOSSARY

DAC – Diamond anvil cell

DIA – A type of high-pressure apparatus in which six orthogonally oriented anvils are used to generate pressure

DDIA – Deformation DIA

Diffusion creep – Plastic deformation caused by the diffusion of individual atoms. The rate of deformation is sensitive to grain size and linearly proportional to stress.

Dislocation – Plastic deformation of a crystal often occurs by slip. Slip occurs along particular directions (slip directions) and on particular planes (slip planes), which together define a slip system. In an actual crystal, slip occurs inhomogeneously by the propagation of a slip front. The slip front that defines the boundary between slipped and unslipped regions is a dislocation where deformation is localized.

Dislocation creep – Plastic deformation caused by the motion of crystal dislocations

Drickamer apparatus (DA) – A type of high-pressure apparatus in which a sample is squeezed between a set of opposed anvils that are supported by a gasket.

Elastic deformation – Instantaneous and recoverable deformation caused by a small displacement of atoms from their equilibrium positions

LPO – Lattice-preferred orientation: a non-random distribution of crystallographic axes in a polycrystalline aggregate. The geometry of LPO reflects the geometry of flow, and hence when LPO is measured in deformed rocks or inferred from seismic anisotropy, the results can be used to infer the flow geometry in Earth's interior.

Peierls mechanism – When high stress is applied, a dislocation will move over a potential barrier (the Peierls potential). When stress is high, the activation enthalpy for dislocation motion is reduced by the applied stress. In such a case, the rate of deformation is an exponential function of stress. When deformation is due to this type of dislocation motion, it is referred to as the Peierls mechanism.

Power-law creep – One of the creep mechanisms in which strain rate is proportional to some power of stress (usually the exponent is 3–5). This mechanism involves thermally activated motion of dislocations at high temperatures and low stresses.

RDA – Rotational Drickamer apparatus