Laboratory Studies of Rheological Properties of Minerals Under Deep Mantle Conditions

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Most large-scale geological process 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 the major progress in this area has often been associated with developments 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 new techniques of synchrotron-based *in-situ* stress-strain measurements with newly designed high-pressure apparati, a new generation of experimental studies of plastic deformation of minerals under deep mantle conditions is emerging that constrain the pressure dependence of deformation of some 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.

Introduction

Plastic deformation of rocks occurs during the large relative translation of rock masses that has important control over large-scale geologic phenomena such as mantle convection, plate tectonics and formation of a mountain belt. Consequently, the knowledge of rheological properties of (mantle) minerals is critical to understand these geodynamic processes.

Rheological properties that we need to know for geodynamical applications include the relationships between the creep strength (or strain-rate) and various physical/chemical parameters and the relationship between deformation microstructures and deformation conditions. 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 developments of new techniques and careful evaluation of the validity of extrapolation of experimental data to geological applications have played a major role in the advancement of our knowledge on rheological properties.

In this article, we first present a brief summary of nature of experimental studies on rheological properties followed by a historical review of experimental studies of plastic deformation. Then we describe the developments of new experimental techniques at pressures beyond 10 GPa, and finally summarize the important issues that are needed to be explored. Due to the limited space, we will focus on experimental studies on rheological properties under deep mantle conditions. For a more comprehensive review, a reader is referred to other textbooks such as (Karato 2008; Poirier 1985).

What do we need to know from laboratory studies?

Experimental studies of plastic deformation have unique challenges compared to than those on other properties such as equation of state or elasticity for several reasons. 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 defects such as point defects, dislocations and grain-boundaries. Each of 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 materials 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, in most of the 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. One needs to explore the influence of all important parameters including pressure, temperature, water fugacity and grain-size.

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

Earlier experimental studies on plastic deformation of minerals

The 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 was to focus 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 apparatus (called the Griggs apparatus) plastic properties and microstructural development of rocks were investigated to ~3 GPa and ~1600 K. Griggs and his co-workers deformed a large number of natural rocks and single crystals of minerals at high pressures and temperatures, and compared their experimental observations with microstructures of naturally deformed rocks as well as with seismological observations (e.g., seismic anisotropy). Most of the important concepts in plastic deformation of rocks were established in these pioneering works including non-linear relationship between stress and strain-rate ("power-law creep"), weakening effects by water, development of deformation fabrics (LPO; lattice-preferred orientation), and grain-size refinement by deformation.

The second generation of rock deformation studies

However, the applicability of these results is 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 raised an issue of applicability to Earth's interior. Interestingly, the use of natural rocks leads to results that are very difficult to apply to nature (Earth). The use of "unnatural" synthetic rocks (unnaturally small grain-size, unnaturally clean chemical composition) are better suited for applying results to Earth. Firstly, natural rocks almost without exception contain various amounts of impurities particularly hydrous minerals at grain-boundaries as a result of weathering. They produce excess water at high temperatures, leading to partial melting. Both of these factors have an important influence on rheological properties, but are not well controlled, if a natural rock is used as a sample. Secondly, the grain-size of natural rocks is large in most cases. Because strain-rates in laboratory experiments are much faster 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 higher confidence.

Recognizing these issues, efforts over the next ~20 years (from mid-1970s to 1990s) were 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 apparati 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 of 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 some pressure (less than ~0.5 GPa) and high-temperature (to ~1600 K) conditions, and a deviatoric stress is applied and measured by a load cell (a device for the measurement of a force) inside the high-pressure chamber. Because a 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 rheological properties of minerals and rocks (e.g., (Mei and Kohlstedt 2000)).

These data provided a basic framework to understand the rheological behavior of minerals in Earth's interior: (1) For most mineral aggregates, likely conditions in Earth's mantle are close to the boundary between diffusion and dislocation creep. This condition means that significant grain-size reduction will lead to rheological weakening. Therefore it is important to understand the physical processes by which grain-size of rocks is controlled. One needs to investigate the grain-size determined by dynamic recrystallization (deformation-induced grain-size refinement), grain-size after a phase transformation as well as the grain-growth kinetics to evaluate the net effects of weakening. (2) The water (hydrogen) content has a strong influence on the creep strength of most of rocks. Consequently, partial melting that will remove water from minerals will modify the creep strength of a rock considerably (Hirth and Kohlstedt 1996; Karato 1986). (3) Large-strain deformation of a rock leads to lattice-preferred orientation (LPO) that causes seismic anisotropy. This condition occurs when deformation proceeds 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.

Limitation of earlier studies and some improvements to the use of 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 from such an apparatus. Note that the pressure range that can be reached by a gas-apparatus is very limited in comparison 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 of these low-pressure data in investigating the mechanisms of water weakening effects: 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 apparatus 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 by 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.

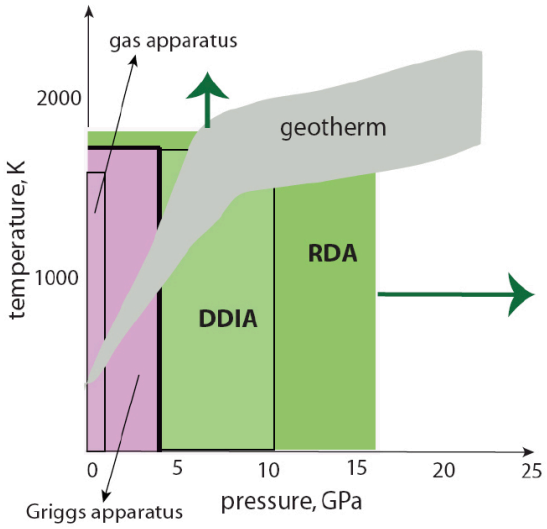

Fig. 1 Pressure-temperature conditions in Earth and those achievable by some high-pressure deformation apparati

Various approaches to whole mantle rheology from laboratory studies

Extending the pressure range of experimental studies of plastic deformation is therefore critical to the understanding of rheological properties of regions of Earth deeper than ~20 km. One of the first attempts at extending the pressure range for rheological experiments was made by (Kinsland and Bassett 1977; Sung, et al. 1977) who used a diamond anvil cell (DAC) as a deformation apparatus. The maximum pressure at which rheological studies were performed using a DAC exceeds 200 GPa (Mao, et al. 1998). However, most of the DAC deformation experiments were conducted at low temperatures and high stresses where deformation mechanisms are likely different from those operating in Earth (the Peierls mechanism as opposed to the power-law creep). 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 type of 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 is not steady-state and in most cases, stress levels are very high and not well known.

New approaches to the experimental studies of deep Earth rheology

Having realized the limitations of these previous efforts, a series of project have been initiated by a team of scientists including ourselves. The aim is to develop new methods of quantitative studies on rheological properties and deformation microstructures that are applicable to the conditions equivalent to Earth's deep interior. Technical developments involve two steps. First, because there was no apparatus in which controlled generation of stress can be made at pressures exceeding ~3 GPa, we needed to design new types of deformation apparati for this purpose that work at pressures of at least to ~10 GPa (our goal is to go beyond ~24 GPa, i.e., lower mantle pressures) at high temperatures. Second, we need to be able to determine the rheological properties of a sample under these conditions. This condition means that we need to be able to measure stress and strain of 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 defining the stress and strain in the sample directly using diffraction and imaging techniques. This technique avoids the previous issues related to friction corrections.

Appropriate high-pressure deformation apparatus must meet some conditions. First, the piston (or an anvil) by which a sample is squeezed must be well supported: otherwise a piston (or an anvil) will be fractured. Second, in some applications, a large strain is needed in order to characterize the microstructural developments. Third, in any apparatus to be used at a synchrotron facility, there must be a space through which X-rays can penetrate without much absorption. Fourth, stress or strain-rate needs to be applied with a controlled fashion so that we can determine steady-state rheological properties. Fifth, thermochemical conditions such as temperature, water fugacity etc. must be homogeneous and well controlled. Sixth, 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, as opposed to a DAC, is better suited for quantitative rheological studies, and two types of apparatus have been designed and tested. These are the modifications of pre-existing apparati that were widely used with synchrotron X-ray. One is a modification to the DIA apparatus in which deformation experiments are conducted at high pressures by moving two sets of anvils independently (called DDIA; see **Fig. 2a**). This apparatus has been operated to P~10 GPa and T~1600 K (or to 19 GPa and T~900 K), and provided a large number of new data on plastic deformation under these conditions (Wang, et al. 2003). This apparatus has an advantage of a simple diffraction geometry and relatively large and homogenous sample space that allows us to conduct 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). Another type of apparatus 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 the static experiments were conducted to ~20 GPa or to ~50 GPa using WC anvils and a sintered diamond anvils, respectively. By rotating one of the anvils, the support for the anvils will be identical to that for the static high-pressure experiments, and therefore deformation experiments can be performed at high-pressures exceeding ~15 GPa (at temperatures beyond ~1800 K). (2) Because of geometry, one can deform a sample to large strain in torsion experiments. This condition 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 changes as a function of distance from the center of rotation, and consequently, there is some ambiguity in the measured stress (and strain). Also, the sample space is rather small and many different components are present next to a sample. Consequently, the analysis of X-ray diffraction data is more complicated than that for DDIA.

(a) (b)

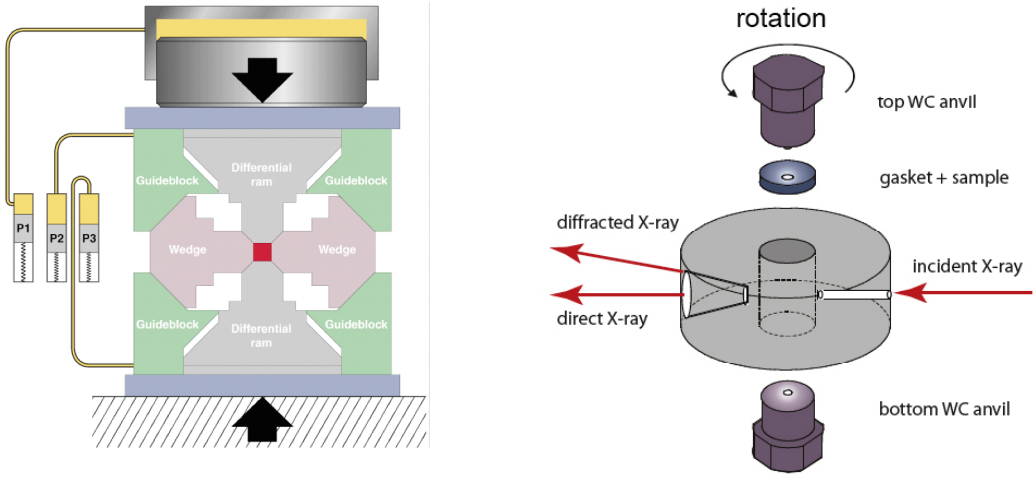

Fig. 2 Schematic diagrams showing (a) DDIA, (b) RDA

A synchrotron radiation facility produces an intense X-ray beams 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 temperature. X-ray provides two types of data from a sample. One is the "image" of a sample by 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 is insensitive to plastic deformation, but rather to elastic strain. 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 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 system (**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 showed that this is not the case as will be discussed later).

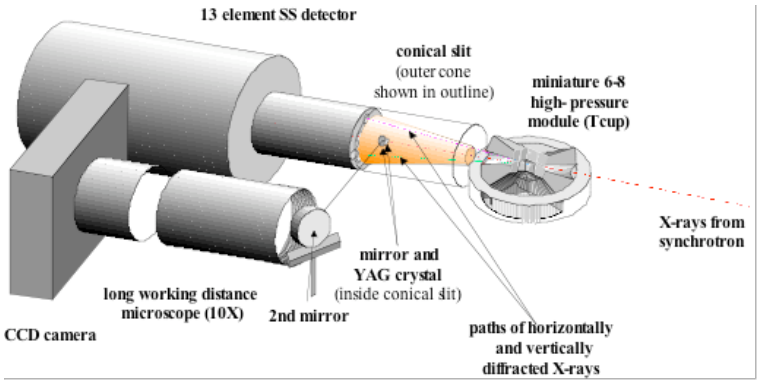

Fig. 3. Measurement system for both diffraction and imaging to define stress and strain. The conical slit collimates the diffracted X-ray beam at a fixed scattering angle, the 13 element solid state detector is placed so that some of the active detectors is exposed by the collimated beam. This beam is 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.

Some new results

A large number of results have already been obtained using the DDIA to ~10 GPa, ~1600 K and the RDA to ~18 GPa, ~2000 K. One of the first results from the application of DDIA brought us a surprise: stress values estimated from various lattice planes show markedly different stress magnitude beyond the model of elastic deformation predicts (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 provide us with new data on plastic anisotropy under deep Earth conditions. By the same token, the X-ray technique of stress measurements provide us with the data on stress in the individual materials in a multi-phase mixture (Li, et al. 2007). Consequently, the stress distribution in a multi-phase mixture can be investigated through this technique by which new insights into the processes of deformation of a multi-phase material can be obtained.

These techniques have been applied to plastic deformation of various materials including MgO, olivine, serpentine, iron, wadsleyite and ringwoodite. The results on

olivine (power-law creep under "dry" conditions) are summarized in **Fig. 4**. Results on dense aggregates at "steady-state" show large pressure effects (activation volume of (15-20) $\times$ 10^{-6} m^3/mol) although results showing much smaller pressure dependence were also published (Li, et al. 2006). The cause for such a large difference in the results is not well known although the possibilities include the influence of transient creep in low strain measurements and the influence of water and/or grain-size. 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., (Cordier, Ungar, Zsoldos and Tichy 2004; Couvy, et al. 2004; Thurel and Cordier 2003)). 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.

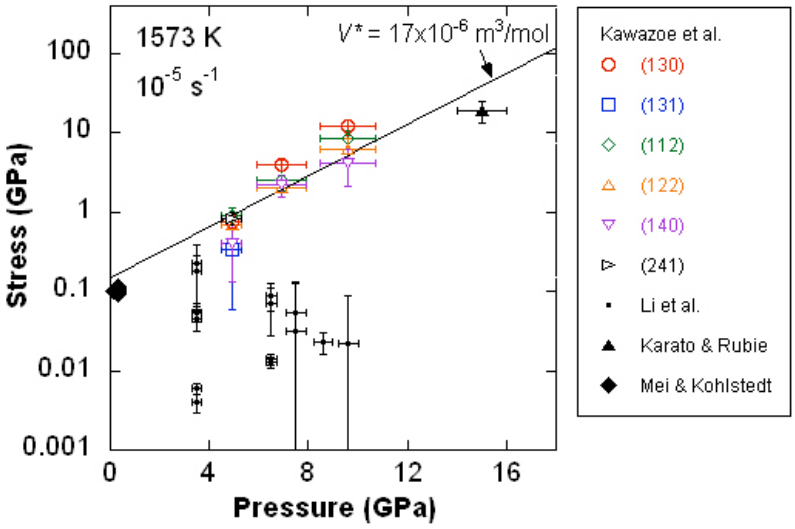

Fig. 4 Some representative results from high-pressure deformation experiments on olivine under dry conditions (after (Kawazoe, et al. 2007)) showing a wide range of pressure effects. Results shown by colored symbols are for steady-state deformation in the power-law creep showing an activation volume of ~(15-20) $\times$ 10^{-6} m^3/mol.

Future prospects

The developments of high-pressure deformation apparatus in combination with synchrotron-produced X-rays are an important step toward the experimental studies on whole Earth rheology. However, we need to be cautious before we derive any specific conclusions by applying the new results obtained so far. We recognize that the developments of new technology of high-pressure rheology are still in its infancy, and some important issues that were addressed in the "second-generation studies" have not been fully incorporated in these new high-pressure technologies. These are the issues of characterizing plastic deformation at low stress levels, and the need for a good control or

at least a good characterization of water fugacity (or water content). Such issues are very critical for the application of DAC for deformation studies where the control of temperature, stress and chemical conditions is difficult. But these issues are also important (although to a lesser extent) for experiments with large volume deformation apparati.

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

As discussed briefly in the previous section, technical development is still in its infancy and many results using new techniques are controversial. There is a wide variation in the reported pressure dependence of olivine that must be explained. Strategy should be established to obtain geologically relevant results on plastic deformation under deep mantle conditions. Very little quantitative data is available for plastic deformation of minerals in the transition zone and the lower mantle under deep mantle conditions. There is an obvious need to extend quantitative deformation experiments to higher pressures (and temperatures). Also there are debates on the dominant slip systems in minerals under deep mantle conditions that 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. Also there are few constraints on the dominant slip systems in deep mantle minerals such as wadsleyite, perovskite or post-perovskite (for slip systems in lower mantle minerals see (Yamazaki and Karato 2007)). The major challenge in inferring the dominant slip systems is to explore the influence of a range of important parameters.

Summary

In this article, we have focused our attention to the technical developments in experimental studies of rheological properties of minerals of the deep interior of Earth. This is based on our belief that understanding the rheological properties of whole mantle is an important step toward the better understanding of whole mantle dynamics, and that experimental studies play a central role in the study of rheological properties. Technical development is a key in the experimental study because no routine techniques are available for the quantitative study of rheological properties beyond a few GPa. We have highlighted important new technical developments with the emphasis on those on large volume apparati at high pressures and high temperatures. We have also discussed that due to the complexities of rheological properties, great care needs to be paid in conducting rheological experiments and applying those results to geological problems. Only with the careful applications of these techniques, and with the further development of techniques, important new experimental results on rheological properties and deformation microstructures will be obtained from laboratory studies to help us better understand the dynamics and evolution of Earth and other terrestrial planets.

Acknowledgments

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GLOSSARY

DAC – Diamond anvil cell

DDIA – Deformation DIA (DIA: a type of high-pressure apparatus in whichsix orthogonally oriented anvils are used to generate pressure)

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) on particular planes (slip plane) that define a *slip system*. In an actual crystal, slip occurs inhomogeneously by the propagation of "slip front". The slip front that defines the boundary between slipped and unslipped regions is a dislocation where deformation is localized.

Dislocation creep – A mechanism of plastic deformation caused by the motion of crystal dislocations

LPO – Lattice-preferred orientation (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 on deformed rocks or inferred from seismic anisotropy, these observations can be used to infer the flow geometry in Earth's interior.

Peierls mechanism – When high stress is applied, then dislocations will move over the 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 relatively high temperatures and low stresses.

RDA – Rotational Drickamer apparatus (Drickamer apparatus: a type of high-pressure apparatus in which a sample is squeezed by a set of opposed anvils that are supported by a gasket)

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