Rheology of Synthetic Omphacite Aggregates at High Pressure and High Temperature

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Eclogite rheology, an essential control on the mechanics of subduction zones, is probably governed by the mechanical properties of the volumetrically dominant constituent, omphacite. Triaxial compression tests were performed in a solid-medium apparatus on synthetic aggregates with five compositions along the solid-solution series jadeite-diopside. The bulk water content of the synthetic material is of the order of $10^{-2}$ H$_2$O/Si. The deformation experiments were performed at a confining pressure of 2.5 GPa and temperatures between 900 and 1100°C (i.e. conditions in the stability field of jadeite). Strain rates ranged from $10^{-6}$ to $10^{-4}$ s$^{-1}$. For a given composition, strength decreases with increasing temperature and decreasing strain rate. The relation between strength and composition changes with increasing temperature. Whereas jadeite is the weaker endmember at all explored conditions, intermediate compositions are stronger than either endmember at 900°C and 1000°C. Samples with calcic compositions deformed at differential stresses above 500 MPa have abundant mechanical (100)[001] twins. In contrast, almost all deformation steps on jadeite aggregates yield strengths below 500 MPa and the resulting microstructures are dominated by well-developed subgrain boundaries and locally recrystallized grains. A weak crystallographic preferred orientation is developed in the deformed samples. Combining mechanical data and observed microstructures we hypothesize that the rate-controlling deformation mechanism differs for the two compositional endmembers. Deformation of diopside aggregates is controlled by dislocation glide, whereas the kinetics of dislocation climb control the deformation of jadeite aggregates at the explored conditions. Reasonable parameters are found when fitting corresponding, micromechanically motivated flow laws to our data. The variation in strength with composition obeys scaling based on the dependence of melting temperature and shear modulus on composition. Extrapolation of the constrained flow laws to geological strain rates indicates considerable strength for omphacite aggregates at temperatures up to 1000°C. Continuous deformation of eclogites by dislocation creep therefore appears unlikely in subduction zones. We suspect that exhumed eclogites exhibiting microstructures indicative of crystal plastic deformation experienced high-stress episodes related to seismic events.

KEY WORDS: omphacite; solid solution; deformation; dislocation; glide; climb; flow law; twinning

INTRODUCTION

Eclogites are found as xenoliths in kimberlites and alkali basalts, as layers and lenses in alpine-type peridotite bodies, and as blocks in blueschist-facies terrains, and also constitute large volumes of the exhumed crust in high-pressure metamorphic terrains (Carswell, 1990). Recently, the term ‘eclogite engine’ has been coined to emphasize that eclogitization is the expected universal fate of the crustal portion of aging lithosphere and thus constitutes a key player in plate tectonics (Anderson, 2007). In particular, eclogites are expected to replace the basalts and gabbros of the oceanic crust during subduction. The kinetics of the eclogite-forming reactions suggest a transition at depths exceeding about 50 km (e.g. Hacker, 1996; Kirby et al., 1996). At greater depths a continuous eclogitic layer with a thickness of several kilometres is predicted at the top of the downgoing slab. The deformation characteristics of this layer are of fundamental importance for stress transfer at the plate boundary and therefore the dynamics of subduction zones.
Eclogitized oceanic crust is primarily composed of omphacite and garnet. Natural omphacite is a solid solution of the sodic endmember jadeite (jd: NaAlSi$_2$O$_6$) and the calcic endmember diopside (di: CaMgSi$_2$O$_6$) with up to 10% of acmite (NaFeSi$_2$O$_6$), some hedenbergite (CaFeSi$_2$O$_6$) and Ca-ekspolite (Ca$_{0.5}$Al$_{0.5}$Si$_2$O$_6$), the last of these being restricted to high pressures and temperatures (Konzett et al., 2008). Microstructures of eclogites exposed at present at the Earth’s surface reveal that omphacite controls their bulk rheological properties at natural conditions. In many cases, undeformed garnet crystals are embedded in a continuous matrix of omphacite with shape- and crystallographic-preferred orientation. In places, omphacite microstructures are indicative of recovery and recrystallization; mechanical twins and kink bands are also observed (e.g. van Roermund & Boland, 1981; Philippot et al., 1992; Godard & Van Roermund, 1995; van der Klauw et al., 1997; Stockhert & Renner, 1998; Aoya, 2001; Pfeilbreier & Stockhert, 2001). With increasing temperature, omphacite undergoes a transformation from the ordered structure with space group $P2/n$ to the disordered $C2/c$ structure (Fig. 1a). The effect of the order–disorder transition on the activated glide systems and rheological properties of omphacite has been a matter of controversy (Brenker et al., 2002; Ulrich & Mainprice, 2005).

Given the challenges associated with performing deformation experiments at upper mantle conditions it is not surprising that the availability of mechanical data on clinopyroxenes correlates with the pressure necessary to stabilize a specific composition (Fig. 1b). The more sodic the composition, the higher are the required pressures. The calcic endmember, diopside, stable even at atmospheric pressure, has been extensively investigated as single crystals (Ave Lallemant, 1978; Ingrin et al., 1991, 1992; Raterron & Jaoul, 1991; Jaoul & Raterron, 1994; Raterron et al., 1994), natural rocks (Kirby & Kronenberg, 1984; Bystricky & Mackwell, 2001; Chen et al., 2006), and synthetic aggregates (Dimanov et al., 2003; Dimanov & Dresen, 2005; Hier-Majumder et al., 2005; Jin et al., 2001) and Zhang et al. (2006) performed deformation experiments at a pressure of 3 GPa on aggregates synthesized from natural omphacite crystals with compositions of di$_{73}$jd$_{27}$ (73 mol % di; 27 mol % jd) and di$_{58}$jd$_{42}$, respectively. Synthetic aggregates of jadeite were examined by Orzol et al. (2006).

Currently, it is difficult to deduce the effect of composition on flow properties, as different experimental techniques and samples of different origin were used in the studies performed so far. Early work suggested that hedenbergite (CaFeSi$_2$O$_6$) is stronger than chromian diopside (Kolle & Blacic, 1982). At face value, diopside aggregates...
appear to be stronger than jadeite aggregates at laboratory conditions yielding dislocation creep (Orzol et al., 2006). In contrast, it has been concluded that jadeite is stronger than diopside at low to intermediate temperatures where crystal plasticity controls deformation (Dorner & Stockhert, 2004). The lack of a profound understanding of the rheological characteristics of solid-solution series of silicates is not restricted to pyroxenes. Only limited work has been performed on important rock-forming minerals such as plagioclase (e.g. Rybacki & Dresen, 2000) and olivine (Zhao et al., 2007).

To determine the relevant deformation mechanisms and quantitatively constrain the rheology of omphacite, we conducted deformation experiments on synthetic polycrystalline clinopyroxene aggregates with five compositions along the omphacite solid-solution series, including end-members jadeite and diopside. The use of synthetic samples prepared following identical procedures and tested in a single deformation apparatus at identical conditions has the benefit of providing readily comparable data. Currently, experiments at the pressure mandated by the stability of sodic omphacite can be performed only in Griggs-type apparatus. These are limited in their stress resolution, even in their modified versions, because of uncertainty in friction associated with the external measurement of axial loads (e.g. Tingle et al., 1993; Rybacki et al., 1998). Clearly, mechanical data of higher quality can be gained for the calcic endmember in experiments at pressures <500 MPa using a gas apparatus. However, the benefits of a systematic study in a single apparatus outweigh the shortcomings of solid-medium apparatus in stress resolution when the prime interest is the effect of composition on the rheology of omphacite.

MATERIAL AND METHODS

Experimental procedures

Synthesis

Synthetic polycrystalline samples can be produced using gel, glass, or oxide powders of appropriate composition as starting materials. Building upon previous experience by Orzol et al. (2006), we used glass powders with five compositions (nominally \( \text{jdd}_{90} \), \( \text{d}_{25} \), \( \text{d}_{50} \), \( \text{d}_{25} \), and \( \text{d}_{50} \)) along the solid-solution series between diopside and jadeite. First, powders were pressed into gold capsules with a length of 18 mm, a wall thickness of 0.2 mm, and an outer diameter of 6 mm by uniaxial cold pressing at 100 MPa in several steps. After drying the cold-pressed samples at 150°C for at least 12 h, the capsules were mechanically sealed and stored in an oven, again at 150°C.

A piston cylinder apparatus with a vessel of 25.4 mm diameter was used for the synthesis experiments. It comprises a servohydraulic control unit for confining pressure and a programmable temperature controller (Renner et al., 1997). Dry sodium chloride (NaCl) served as the pressure-transmitting medium. Heating was provided by a graphite resistance furnace. A NiCr–Ni thermocouple was used for measuring temperature in the upper part of samples. Three samples were synthesized in a single run. Several \( P-T-t \) paths in the stability field of jadeite were tested to optimize the microstructure of the samples. We found that a pressure \( P \) of 2.2 GPa, a temperature \( T \) of 1000°C, and a run time of 6 days are appropriate to reach a grain size of about 10 µm and a homogeneous microstructure. The actual chemical composition after synthesis was determined by electron microprobe analysis (\( \text{jdd}_{90} \): \( \text{Na}_{0.97}\text{Al}_{1.06}\text{Si}_{2}\text{O}_{6} \), \( \text{d}_{25} \): \( \text{Na}_{0.72}\text{Al}_{0.26}\text{Ca}_{0.26}\text{Mg}_{0.26}\text{Si}_{2}\text{O}_{6} \), \( \text{d}_{50} \): \( \text{Na}_{0.48}\text{Al}_{0.52}\text{Ca}_{0.50}\text{Mg}_{0.48}\text{Si}_{2}\text{O}_{6} \), \( \text{d}_{25} \): \( \text{Na}_{0.24}\text{Al}_{0.76}\text{Ca}_{0.76}\text{Mg}_{0.76}\text{Si}_{2}\text{O}_{6} \), and \( \text{d}_{50} \): \( \text{Al}_{0.48}\text{Ca}_{1.00}\text{Mg}_{0.92}\text{Si}_{0.92}\text{O}_{6} \)).

Deformation

Synthesized samples were machined to a cylindrical shape of ~7.3 mm length and ~3.7 mm diameter, and then dried at 150°C for at least 24 h. Platinum capsules with a thickness of 0.1 mm were used for jacketing. All deformation experiments were performed in a modified Griggs-type solid-medium apparatus (Rybacki et al., 1998). A eutectic mixture of CsCl–NaCl (65.5 mol % CsCl and 34.5 mol % NaCl) served as the confining pressure medium. At 2.5 GPa, the nominal confining pressure of all deformation tests, the melting temperature is about 1000°C (Kim et al., 1972). Two NiCr–Ni thermocouples were used for controlling and measuring the temperature at the lower and upper ends of the sample, respectively; a graphite furnace provided heating (Fig. 2).

Pressure and temperature were raised at 14 MPa/min and 5°C/min, respectively, to avoid damage to the graphite furnace and to minimize pressure gradients within the assembly. During this stage, the axial piston was periodically advanced until final conditions were reached. At run conditions, the hit point was first determined by advancing the deformation piston at a velocity of 1 mm/h. Subsequently, the deformation piston was retracted for ~1 mm before being advanced again for the actual deformation stage at specified strain rate. In some experiments, this procedure was repeated up to three times to explore the effect of strain rate (Fig. 3). At the end of the final deformation stage, the piston was retracted for at least 1 mm from the sample, before pressure and temperature were decreased at rates of 83 MPa/min and ~30°C/min, respectively.

Stress–strain curves were derived from the recorded force–displacement curves assuming constant sample volume and correcting for friction and stiffness of the axial load column. Strain was calculated from the displacement of the deformation piston beyond the hit point and the initial length of the sample at room temperature. In strain rate stepping tests, the shortening achieved in...
the previous stage(s) was taken into account. The nominal uncertainty in strain is about 0.001, accounting for the accuracy of the displacement transducer and uncertainty in stiffness correction. The uncertainty in reported strain rates of a factor of about 1 results mostly from the uncertainty in actual sample length during the various stages of an experiment. The nominal uncertainty in the derived stress is estimated to be 20 MPa, considering a load cell accuracy of 0.02% over its range of 160 kN and an uncertainty in the sample diameter of 0.1 mm. In addition, a significant methodological uncertainty for stress arises from the determination of the contribution of friction to the external load cell reading (Rybacki et al., 1998; Renner et al., 2001). Here, we applied a dynamic correction; that is, the approximately linear portion of the force-displacement curve prior to the hit point serves as the friction baseline (Fig. 4). A major source of uncertainty is the friction on the alumina piston on top of the sample. Uncertainty associated with the extrapolation of the friction baseline, the position of the baseline varying from test to test, and the issue of piston separation add up to an uncertainty of 50-100 MPa and therefore hinder reliable determination of the differential stress below about 100 MPa.

The confining pressure may be lower than the nominal pressure by about 10% as a result of friction at the sealing of the outer piston and the finite strength of the pressure transmitting assembly parts. The actual temperature is believed to be within about 20°C of the readings of the two thermocouples (see Rybacki et al., 1998). The thermocouple readings were not corrected for the effect of pressure (see Getting & Kennedy, 1970).

**Analytical techniques**

The cylindrical samples were cut into two parts along their axes. Doubly polished thin sections were prepared from...
Fig. 4. The processing of force–displacement curves involved a dynamic friction correction as exemplified for a di50jd50 sample deformed at a temperature of 900°C and a strain rate of 2×10^-4 s^-1.

one of these parts for optical and scanning electron microscope (SEM) inspection, as well as for electron backscatter diffraction (EBSD) and electron microprobe (EMP) analysis (Table 1). For EBSD analysis, the thin sections were chemically polished using a colloidal silica suspension (15–20 min). For a number of samples, the second half was used for the preparation of thin foils for transmission electron microscopy (TEM) and polished slabs with a thickness of 150–200 µm for Fourier-transform infrared microspectrometry (FTIR). The phases in the synthesized samples were determined by powder X-ray diffraction analysis (XRD). The crystallographic orientations and microfabrics were analyzed by SEM, using a LEO 1530 instrument with field emission gun and backscatter detector. For EBSD analysis, the SEM was operated at an accelerating voltage of 20 kV, the thin section being tilted at an angle of 70° with respect to the beam, and with a working distance of 25 mm. The HKL CHANNEL5 software was used for indexing and processing of the diffraction data. Automatic EBSD measurements were performed to obtain orientation maps and grain geometry. Reference files with reflectors optimized for jadeite and diopside were applied for jadeite samples and for all other samples, respectively. Reflectors were not critical for different compositions along the omphacite solid-solution series (Mauler et al., 2000). Six bands were used for indexing. Patterns indexed with a mean angular deviation (MAD) of >1·0 were discarded. Further processing included the automatic removal of isolated points that were incorrectly indexed and the replacement of non-indexed points by the most common orientation of a specified number (six or seven) of neighbouring indexed points. The original percentage of indexing is 45–70%. Processing of raw data resulted in an indexing percentage of 50–86%. The percentage of correct indexing increased with increasing grain size of the samples. A misorientation angle of 10° was chosen as the threshold between low- and high-angle grain boundaries. Grain size grows as the diameter of a circle of equivalent area. The shape preferred orientation (SPO) was gained from the orientation of the long axes of the grains in 2D section. Crystallographic preferred orientations (CPOs) are displayed as density plots in equal area projections of the lower hemisphere. The contours in the density plots are scaled by multiples of a uniform distribution (MUD). The maximum scaling contour was set to 2·0–2·4 MUD. Forward scattered orientation contrast (OC) imaging was performed to investigate the substructure within the grains, in particular the occurrence of twins. OC imaging provides qualitative information on the crystallographic orientation (e.g. Prior et al., 1999). Regions of different crystallographic orientation appear in different gray shades.

Arrangement and density of dislocations were analyzed by TEM with a Philips EM301 microscope operated at 100 kV. For preparation of foils a GATAN PIPS ion mill was used. All diffraction contrast images were produced using bright-field conditions. Type and concentration of water and water-related defects were studied by FTIR (e.g. Kronenberg & Wolf, 1990) at room temperature using a Bruker IFS 48 spectrometer with an attached IR-microscope (see the Electronic Appendix, available for downloading at http://www.petrology.oxfordjournals.org). The IR microscope permits spectrometric analysis with a spatial resolution of about 0·1 mm using polished sections of 0·1–0·3 mm thickness. The hydrogen concentration in samples was calculated from the absorption spectra in the wavenumber range 3000–4000 cm^-1 following the method proposed by Paterson (1982).

Microfabric of synthesized samples
Within the limit of detection, XRD analysis confirmed that the synthesized samples are composed of a single crystalline phase. All aggregates are in the disordered state (space group C2/c) in accord with the phase diagram (Fig. 1a). Only in the jd100 and di25jd75 samples did SEM images reveal the presence of a second phase (Fig. 5d). The volume percentage of this second phase is less than about 4% in jd samples, and even lower in di25jd75 samples (Table 1). The distribution of this phase at triple junctions and the gently curved interfaces suggest it to be derived from a melt present at run conditions. The second phase is characterized by a higher SiO2 and a lower Na2O content.
Table 1: Experimental conditions and results for the deformation experiments on the synthetic polycrystalline clinopyroxene aggregates at a nominal confining pressure of 2.5 GPa

<table>
<thead>
<tr>
<th>Composition</th>
<th>Sample</th>
<th>Temperature (°C)</th>
<th>Strain rate (1/s)</th>
<th>Stress (MPa)</th>
<th>Strain (%)</th>
<th>Micro-analysis</th>
<th>Grain size (µm)</th>
<th>Melt content (%)</th>
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The reported temperature is the average of the upper and lower thermocouple. In the test on sample omph11 the upper thermocouple failed. Melt content was estimated from SEM images. Axial strain is reported as derived from the recorded force–displacement curves (rec.) and from length measurements on samples recovered after quenching to ambient conditions (total).

*Grain sizes based on automated EBSD measurements (quoted as average ± standard deviation).

†Grain sizes based on line intercept determinations on SEM images.
compared with a jadeite composition, as indicated by energy-dispersive X-ray analysis.

The typical grain size of the starting material is in the range of 2–20 μm (Fig. 6a–c). In 2D sections perpendicular to the cylinder axis, grains do not show an SPO (Fig. 6a and b). In 2D sections parallel to the cylinder axis, grains with diameters >10 μm can exhibit a weak SPO (Fig. 6c). A significant crystallographic preferred orientation (CPO) was not found anywhere in the starting material (Fig. 6a–c). Grain-size distribution and grain shape vary systematically with composition. In diopside samples, grains are mostly equant and exhibit a narrow size range (Fig. 5b); in contrast, grain shape in jadeite samples is more anisometric and grain size varies (Fig. 5a). Samples with intermediate composition show microstructures that are transitional between those of the two compositional endmembers. Samples with the composition di25jd75 tend to show the smallest grain size. Generally, all grains appear internally strain-free under the optical microscope and the TEM (Fig. 5c and d).

In atom fractions, the estimated hydrogen contents range from slightly above to slightly below 10⁻² H/Si from jadeite to diopside aggregates, respectively. Water is most probably incorporated during glass powder preparation (Orzol et al., 2006). Intermediate compositions tend to contain the least amount of hydrogen. A large portion of the hydrogen is expected to be present in water inclusions along interfaces and in the quenched melt observed in the sodic samples. Spectra of undeformed samples indeed exhibit prominent broad band absorption between 3000 and 3700 cm⁻¹, but the jadeite and diopside samples also show distinct absorption peaks at 3614 and 3650 cm⁻¹, respectively, in agreement with previous studies (Bromiley & Keppler, 2004; Bromiley et al., 2004). Such peaks are not observed for intermediate compositions. Compared with experimentally determined water-solubility in synthetic Na-rich clinopyroxene and Cr-diopside (Bromiley & Keppler, 2004; Bromiley et al., 2004), all our samples contain excess H₂O. These observations qualify the experimental conditions as ‘wet’.

Fig. 5. (a) SEM image (secondary electron signal) shows irregular to prismatic grain shape of synthesized jadeite. Arrows point to an unspecified phase, interpreted to represent a quenched hydrous melt between the jadeite crystals. (b) SEM image (back-scatter electron signal) shows equant grains of synthesized diopside. (c, d) TEM bright-field micrographs of starting material (di75jd25 sample L1) showing grains devoid of dislocations with smoothly curved to straight boundaries.
Fig. 6. Selected crystallographic and shape orientation patterns obtained by automated EBSD measurements (2 μm step size) of starting materials (a–c) and deformed samples (d–g). Left: density plots (equal area projection of the lower hemisphere) are displayed for the poles to (100) and (010) planes and the [001] direction. Shortening direction is vertical for all diagrams. Right: 2-D orientation of long axes of grains with diameters <10 μm and >10 μm are displayed in a rose diagram. In (a) and (b), the section perpendicular to the cylinder axis of the samples is displayed, in (c–g) the section parallel to the cylinder axis.
RESULTS
Mechanical data
We performed deformation experiments on 32 samples at temperatures between 900 and 1100°C, a confining pressure of 2.5 GPa, and constant strain rates between $1 \times 10^{-6}$ and $8.5 \times 10^{-5}$ s$^{-1}$. A total of 24 tests were conducted at a single set of conditions, allowing an unequivocal correlation of the mechanical data and the microstructure of the deformed samples. In the eight step tests, a single sample was axially shortened at up to three strain rates. Typical examples of the derived stress vs strain curves are presented in Fig. 7. The majority of samples exhibit deformation at fairly constant stress. The occurrence of limited softening or hardening behavior does not systematically correlate with experimental conditions, indicating uncertainties in the friction correction as the potential cause, rather than actual material behavior. Indeed, the vast majority of samples appear homogeneously shortened and thickened after deformation. The reported flow stresses (Table 1), simply referred to as strength in the following discussion, correspond to the maximum stresses attained in a deformation step. The reproducibility of mechanical data obtained in tests performed at similar conditions on different samples is of the order of ±10%.

For all compositions, strength systematically decreases with increasing temperature and decreasing strain rate (Fig. 8a and b). The apparent effect of composition on relative strength evolves with temperature (Fig. 9a).

At 900°C, the lowest investigated temperature, clinopyroxenes of intermediate composition are stronger than either endmember, of which jadeite is systematically the weaker one. At 1100°C, we find an almost monotonic decrease in strength from diopside to jadeite aggregates. Notably,
the range in stress experienced by samples of the two compositional endmembers hardly overlaps. At the imposed strain rates, jadeite and diopside aggregates essentially deformed at stresses below and above 500 MPa, respectively.

We determined best-fit parameters of a power-law relation

$$\dot{\epsilon} = A\sigma^{n'} \exp\left(\frac{-Q}{RT}\right)$$

for the mechanical data, using a non-linear inversion method (Sotin & Poirier, 1984). This method accounts for the reported uncertainties in experimental data, strain rate $\dot{\epsilon}$, stress difference $\sigma$, and absolute temperature $T$ (Table 1). Here, $A$, $n'$, $Q$, and $R$ denote the pre-exponential factor, apparent stress exponent, apparent activation energy of creep, and universal gas constant, respectively. The values for $n'$ vary between $\sim 3$ and $\sim 10$, and generally seem to increase from sodic to calcic composition (Table 2, Fig. 9b). The values for $Q$ vary between 200 and 600 kJ/mol, without obvious relation to composition.

**Microfabric of deformed samples**

In some cases, the difference between axial finite strain, determined from the length of the cylindrical sample after removal from the apparatus, and permanent strain, calculated from the record of the displacement transducer, exceeds $\delta \epsilon = \pm 5\%$, the maximum uncertainty in the recorded strain resulting from the uncertainty in hit-point determination (Table 1). Deformed samples often show tensile cracks oriented normal to the sample axis. These cracks, which originate during quenching and release of the axial load, increase the apparent final sample length, causing the determined permanent strain to fall short of that recorded during the experiment. Samples that, in contrast, appeared shorter than the estimated recorded strain probably experienced non-hydrostatic quenching; these were therefore excluded from the microstructural analysis.

On the optical and the SEM scale, there is no marked difference in grain size between starting material and deformed samples (Table 1, Fig. 10). The deformed clinopyroxene samples show an SPO and a weak CPO (Fig. 6d–g).

**Table 2: Parameters of a power-law [equation (1)] fit to the data reported in Table 1**

<table>
<thead>
<tr>
<th>Composition</th>
<th>In($A$) (MPa$^{-n'}$ s$^{-1}$)</th>
<th>$n'$</th>
<th>$Q$ (kJ/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td>di</td>
<td>$-29.6 \pm 4.8$</td>
<td>$8.1 \pm 1.5$</td>
<td>$391 \pm 79$</td>
</tr>
<tr>
<td>di$_{25}$</td>
<td>$-10.3 \pm 3.2$</td>
<td>$5.5 \pm 1.9$</td>
<td>$414 \pm 136$</td>
</tr>
<tr>
<td>di$_{75}$</td>
<td>$-4.7 \pm 1.1$</td>
<td>$3.5 \pm 0.2$</td>
<td>$323 \pm 18$</td>
</tr>
<tr>
<td>di$_{50}$</td>
<td>$4.3 \pm 3.6$</td>
<td>$3.0 \pm 0.5$</td>
<td>$281 \pm 63$</td>
</tr>
<tr>
<td>jd</td>
<td>$35.6 \pm 6.3$</td>
<td>$4.2 \pm 0.8$</td>
<td>$413 \pm 79$</td>
</tr>
</tbody>
</table>
Fig. 10. OC images of (a) jadeite (sample omph57) deformed at 1108°C; (b) jadeite (sample omph6) deformed at 1100°C; (c) di50jd50 (sample omph52) deformed at 1100°C; (d) di50jd50 (sample omph22) deformed at 912°C; (e) di25jd75 (sample omph16) deformed at 1110°C; (f) di25jd75 (sample omph19) deformed at 1114°C; (g) diopside (omph50) deformed at 993°C; (h) diopside (omph53) deformed at 882°C. For all samples, the confining pressure during deformation was 2.5 GPa (for details see Table 3). Subgrains are widespread in jadeite (a,b), whereas mechanical twins tend to be more frequent in diopside-rich compositions (e–h). High-angle grain boundaries between grains of low twin density and grains with a high twin density are strongly sutured (g).
The long axes of the grains, as seen in axial 2D sections, are oriented normal to the direction of shortening. The CPO is very weak with the maximum MUD typically <2. The strongest CPO with a maximum MUD of 2-4 is recorded in the sample of intermediate composition di$_{50}$jd$_{50}$ (Fig. 6c). Samples of the diopside endmember composition show the least pronounced CPO (Fig. 6g). For all compositions, the normal to the (010) planes tends to be oriented parallel to the shortening direction, defining a point maximum. The [001] direction is oriented perpendicular to the shortening direction, forming a girdle in the foliation plane.

Mechanical twins are found in almost all deformed samples (Figs 10d–h and 11). The overall twin density decreases with decreasing maximum differential stress experienced by the sample. As such, twins are widespread in deformed samples of calcic composition and rare or absent in those of more sodic composition. EBSD measurements and TEM observations reveal that the twin plane is (100) and the glide direction is [001]. Most twin lamellae are less than 0.5 µm wide (Figs 10d–h and 11). Some are also visible on the optical scale. In inhomogeneously deformed grains, the twins can be wedge-shaped and taper out in the grain interior (Fig. 10f and g). At serrated high-angle grain boundaries, the diameter of bulges corresponds to the thickness of the twin lamellae (Fig. 10g).

Dislocation density reaches about $10^{15}$ m$^{-2}$. The highest dislocation densities are observed for intermediate compositions (Fig. 11e and f) in accord with the observation of their highest strength. Subgrains are widespread in the more sodic compositions and rare in the more calcic compositions (Fig. 10a and b). The low-angle grain boundaries are frequently oriented normal to [001] (Fig. 11a and g). Recrystallized new grains with a diameter of the order of 1 µm are observed on the TEM scale within deformed samples of jadeite (Fig. 11c and d). The small recrystallized grains show very few dislocations. The FTIR spectra of deformed samples are similar to those obtained for the starting material, indicating that water speciation is not significantly affected by deformation (see the Electronic Appendix).

**DISCUSSION**

The mechanical data indicate thermally activated creep. The strength of the synthetic clinopyroxene aggregates decreases with decreasing strain rate and increasing temperature. Although the sodic endmember jadeite is consistently weaker than the calcic endmember diopside, the details of the variation in strength with composition change with temperature. At 900 and 1000°C, some intermediate compositions are significantly stronger than either compositional endmember. Only at 1000°C does strength increase almost monotonically from jadeite to diopside aggregates. In the following discussion, we first compare our observations with those of previous studies. Then, the likely active deformation mechanisms are identified by combining key mechanical and microstructural observations to fit micromechanically motivated flow laws to the data. The dependence of strength on composition is discussed in the light of first-order scaling relations. Finally, the geological implications of these data are considered.

**Comparison with previous work**

In direct comparison, stress exponent and activation energy values derived for our jadeite aggregates agree well with those reported by Orzol et al. (2006). However, the absolute strength values determined in this study are systematically lower than those previously determined (Fig. 9a). At a given stress, the difference corresponds to less than an order of magnitude in strain rate. Compared with Orzol et al. (2006), we used a different glass precursor and we did not employ seeding for synthesis. Nevertheless, grain sizes achieved by the two approaches are comparable. Bulk water contents of our samples estimated by FTIR spectroscopy exceed the contents quoted by Orzol et al. (2006) by a factor of 3–5. Microscopic inspection reveals that samples rich in the jd-component (jd$_{30}$ and jd$_{50}$) contain up to 4% of an unspecified phase (Table 1), probably a quenched hydrous melt. The presence of this melt phase is thought to be responsible for the observed difference in strength between this study and that of Orzol et al. (2006). Such a moderate effect of melt on strength is comparable with findings by Dimanov et al. (2003) for diopside, and by Cooper & Kohlstedt (1986) and Hirsh & Kohlstedt (1995) for olivine aggregates. The correspondence in the stress exponent and activation energy between melt-free and melt-containing samples shows that the rate-controlling mechanism remains unaffected but that the average load-bearing area of the grains changes.

The mechanical data for omphacite aggregates of intermediate composition compare fairly well with the results of Zhang et al. (2006), who investigated synthetic aggregates prepared from crushed natural omphacite crystals (di$_{30}$jd$_{45}$ FeO = 174 wt %; about 10$^{-2}$ H/Si). Combined, the two datasets yield an apparent stress exponent of $\sim$6.5 at a temperature of $\sim$1100°C (Fig. 12a); that is, a value higher than that deduced by Zhang et al. (2006) on the basis of their data alone. Jin et al. (2001) deformed syngneous Fe-bearing omphacite aggregates (di$_{20}$jd$_{25}$; FeO/MgO = 0.17; about 10$^{-2}$ H/Si) at a temperature of 1500 K and a confining pressure of 3 GPa. At a strain rate of $4.6 \times 10^{-4}$ s$^{-1}$, a strength of $\sim$300 MPa was derived from two stress–strain curves. The conditions of these experiments are too different from ours for a direct comparison.

Absolute strength determined for our synthetic fine-grained, wet diopside aggregates compares well with the results of Kirby & Kronenberg (1984) obtained for...
Fig. 11. TEM bright-field micrographs of deformed clinopyroxenes. (a) Deformed jadeite shows mechanical (100) twins and a high dislocation density. A low-angle grain boundary is marked by an arrow (jd100 sample omph57). (b) Bulges in high-angle grain boundaries correlated with twins indicate that grain boundary mobility is affected by orientation relations (black arrow, jd100 sample omph57). (c, d) Recrystallized micron-sized grains are free of dislocations and show various crystallographic orientations, as indicated by the inserted diffraction pattern in (c) (jd100 sample omph57). (e, f) Slightly curved twin lamellae in an inhomogeneously deformed omphacite grain with high dislocation density (di3jd25 sample omph22). (g) Wide twin lamella and a low-angle grain boundary marked by an arrow (di2jd25 sample omph28). (h) High density of narrow twin lamellae in diopside (di100 sample omph47).
samples of coarse-grained Sleaford Bay clinopyroxenite at 1·5 GPa confining pressure (Fig. 12b). At face value, this agreement suggests a modest effect of compositional details, confining pressure, and grain size on the flow behavior of diopside aggregates at the explored conditions. The strength observed by Chen et al. (2006) at a confining pressure of 0·3 GPa for this rock is, however, slightly lower than that observed in the two studies at higher pressure and as such is close to the strength of diopside single crystals (Fig. 12b). A direct one-to-one comparison of the stress—strain rate relations obtained by deformation experiments on single crystals with those obtained on polycrystalline samples is not possible. However, our observations on \( \text{di}_{50}\text{jd}50 \) and \( \text{di}_{75}\text{jd}25 \) samples agree well with the strain rate dependence of strength obtained for single crystals at temperatures above \( \sim1000^\circ\text{C} \), where the glide system \{110\} \( \frac{1}{2} <a \pm b> \) was activated (Raterron & Jaoul, 1991; Fig. 12b). Between 800 and 1000°C, the glide system \{100\}[001] dominates deformation of single crystals in the tested orientation (Raterron & Jaoul, 1991).

The CPO patterns observed in our deformed samples are weak (Fig. 6d–g), probably because of the limited total strain. Qualitatively, the weak CPO is similar for all our deformation experiments, independent of temperature and clinopyroxene composition. The [001] orientation describes a girdle perpendicular to the shortening direction, whereas the poles to the [010] plane define a point maximum parallel to the shortening direction. Bascou et al. (2001, 2002) obtained a similar CPO of omphacite in numerical simulations, and interpreted the pattern to result from the combined activation of the slip systems \{110\} \( \frac{1}{2} <a \pm b> \), \{110\}[001], and {100}[001]. These slip systems are consistent with TEM observations on naturally deformed omphacite (e.g. Buatier et al., 1991; Godard & Van Roermund, 1995; Ulrich & Mainprice, 2005). According to Bascou et al. (2001, 2002), slip on the \{110\} planes is responsible for the orientation of \{010\} planes parallel to the foliation, whereas the predominant glide direction [001] defines a girdle in the foliation. These patterns are comparable with the S-type of Godard & van Roermund (1995) observed in naturally deformed eclogites that is also consistent with axial symmetric compression (Ulrich & Mainprice, 2005). Similar patterns were observed in experimentally deformed clinopyroxene (Mauler et al., 2000) and jadeite (Orzel et al., 2006) aggregates. For natural eclogites, a second type of CPO pattern was reported, the L-type, characterized by a point maximum of [001] parallel to the lineation and a girdle of [010] perpendicular to the lineation (e.g. Helmstaedt et al., 1972). Based on TEM observations, Brenker et al. (2002) postulated that cation ordering in omphacite controlled the development of either L-type (ordered structure) or S-type patterns (disordered structure) in natural eclogites. A subsequent study on three mantle eclogite xenoliths did not support this hypothesis (Ulrich & Mainprice, 2005). Omphacite is disordered in our experiments and the CPO patterns are consistent with the predictions (Bascou et al., 2001, 2002; Ulrich & Mainprice,
2005) for axial symmetric compression. With respect to the activated glide system, the orientation of (010) normal to the shortening direction suggest {110}[001] to be predominant, as proposed by Bascou et al. (2001, 2002). Activation of the glide system {100}[001], observed by Raterron & Jaoul (1991) and Raterron et al. (1994) to be the most active between 800 and 1000°C in experiments on diopside single crystals, is not reflected by the CPO. The Burgers vector [001] common to both glide systems leaves the possibility of cross slip.

**Mechanisms controlling the plastic deformation of omphacite**

*Inferences from the microfabric*

Observed microfabrics and relations between strength and strain rate or temperature indicate that samples of all compositions are plastically deformed by a combination of mechanical twinning, dislocation glide and climb. Twin lamellae parallel to (100) have previously been observed in clinopyroxenes from eclogite-facies rocks (Carter & Raleigh, 1969; Wenk, 1970; Ave Lallemant, 1978; Muller, 1993; Godard & Van Roermund, 1995; Trepmann & Stockhert, 2001; Muller & Franz, 2008) and in samples deformed in the laboratory (Kirby & Christie, 1977; Kolle & Blacic, 1982; Orzol et al., 2003; Muller et al., 2007). The mechanisms of twinning in pyroxenes have been discussed in some detail (Kirby & Christie, 1977; Kolle & Blacic, 1982). Kolle & Blacic (1982) proposed a composition-insensitive critical resolved shear stress (CRSS) of about 140±10 MPa for mechanical [100][001] twinning of clinopyroxene. Experiments on natural polycrystalline jadeite (Orzol et al., 2003) indicated a CRSS of 150±25 MPa, in accord with the suspected negligible influence of composition. Based on these results, mechanical twinning should occur with increasing frequency in clinopyroxene aggregates subject to a differential stress exceeding about 300 MPa and the observed higher frequency of mechanical twins in deformed samples of more calcic composition is probably a result of the generally higher differential stresses attained in these experiments compared with those on samples of more sodic composition (Table 3). Low-angle grain boundaries form by rearrangement of geometrically necessary dislocations and recrystallized grains by migration and/or formation of high-angle grain boundaries driven by the reduction of stored strain energy during high-temperature creep or static annealing (e.g. Nicolas & Poirier, 1976; Poirier, 1985). Geometrically necessary dislocations are introduced during inhomogeneous crystal plastic deformation, dominated by dislocation glide. Although the importance of dislocation glide is also reflected by the CPO, the low-angle grain boundaries indicate dislocation climb. Strain-induced grain boundary migration is documented by serrated high-angle grain boundaries (Fig. 10g). Here, the diameter of bulges corresponds to the thickness of twin lamellae, indicating that grain boundary mobility is affected by the orientation relations between adjacent grains and strain gradients as a result of accommodation processes at the interface.

The observation that low-angle grain boundaries and recrystallized grains are more widespread in the sodic pyroxenes suggests that dislocation climb is more effective in those compositions compared with the calcic pyroxenes at the conditions of the deformation experiments. This assertion is consistent with the observation that the CPO is more pronounced in the more sodic and less pronounced in the more calcic compositions. CPOs observed in this study are generally weak, probably as a result of the limited finite strain. A weak CPO in fine-grained

### Table 3: Results of micromechanical modeling

<table>
<thead>
<tr>
<th>Composition</th>
<th>Climb-controlled creep according to equation (2)</th>
<th>Glide-controlled creep according to equation (3)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$n$</td>
<td>$\ln(A_{\text{climb}})$</td>
</tr>
<tr>
<td>jd</td>
<td>4.5</td>
<td>4.8 ± 5.6</td>
</tr>
<tr>
<td>di</td>
<td>3.5</td>
<td>0.7 ± 4.3</td>
</tr>
</tbody>
</table>

Italic font indicates parameters that were prescribed in the inversion [stress exponents in (a) and the range of dislocation source density in (b)]. Bold values are actual inversion results whereas normal font indicates parameters calculated from the inverted ones using $\mu_{\text{di}} \approx 56$ GPa and $v_{p,\text{di}} \approx 4140$ m/s.
experimentally deformed clinopyroxene aggregates has been taken as an indication of diffusion creep (Mauler et al., 2000). The observed dislocation densities, subgrains, recrystallized grains, and serrated grain boundaries (Figs 10 and 11) as well as the mechanical behavior (large apparent stress exponents, Fig. 9b), however, clearly argue for dislocation-accommodated deformation in our samples.

**Inferences from the mechanical data**
Two state variables, temperature and stress, are central for the mechanics and kinetics of deformation of crystals by motion of dislocations. The relative magnitude of these two variables with respect to melting temperature \( T_m \) and shear modulus \( \mu \) are associated with regimes dominated by different dislocation processes (Frost & Ashby, 1982). Dislocation climb accommodated by the formation and migration of jogs is a thermally activated process realized by thermally activated vacancy diffusion with an activation enthalpy that roughly scales with the melting temperature of a substance (e.g. Poirier, 1985; Karato et al., 1995). For a dislocation to glide, it has to be lifted out of its potential trough in the lattice. Overcoming this potential barrier that scales with the shear modulus can be achieved by mechanical force.

Whereas melting temperatures increase from jadeite to diopside (Bell & Davis, 1969), the relation between shear modulus and composition is opposite (Duffy & Anderson, 1989). Thus, the explored conditions yield separate ranges of scaled temperature and strength for the compositional endmembers (Fig. 13). The tests on jadeite aggregates correspond to the highest homologous temperatures and the lowest normalized strengths whereas those on diopside aggregates represent the lowest homologous temperatures and the highest normalized strengths. The boundary between the conditions for diopside and jadeite aggregates is close to a temperature of \( \sim 2/3 T_m \), considered to separate ‘low-temperature glide’ and ‘high-temperature climb’ for metals (Weertman, 1999).

The notion that the deformation of aggregates with a calcic composition is controlled by glide kinetics is supported by further details of the mechanical data. Apparent stress exponents as large as those derived for calcic clinopyroxene (Table 2) are considered unlikely for cases where dislocation climb is the rate-controlling step. Furthermore, the increase in strength from diopside to intermediate compositions found here for 900 and 1000°C (Fig. 9a) correlates with the composition dependence of the shear modulus, suggesting glide as the relevant dislocation mechanism. Dorner & Stockhert (2004) found such a correlation between shear modulus and strength of pyroxene single crystals by indentation hardness measurements at temperatures between 300 and 750°C. In these comparatively low-temperature and high-stress experiments, climb cannot be significantly activated at laboratory strain rates, leaving glide as the predominant mechanism of plastic deformation. The correlation between the trends of shear modulus and observed strength with composition near the calcic endmember suggests that the deformation of the calcic pyroxenes is still controlled by dislocation glide, despite the higher temperatures of our experiments.

The relative weakness of the aggregates with the most sodic composition indicates that for these the transition to climb-controlled deformation occurs at temperatures between those attained in the present study and those explored by Dorner & Stockhert (2004). Considering the deformation of the more sodic aggregates to be controlled by dislocation climb in perfect agreement with the observed apparent stress exponents (Table 2), the ubiquity of subgrain boundaries (Figs 10 and 11), and the relation between melting temperature and composition.

**Constraints on flow law parameters**
Experimentally constrained flow laws can be extrapolated to geological conditions with some confidence only when based on micromechanical models (e.g. Paterson, 1987). In particular, the power-law parameters quoted in Table 2 constitute purely empirical fits unsuitable for reliable extrapolation. Therefore, we made an effort to choose flow laws whose underlying models are in agreement with our...
mechanical and microstructure data and to derive physically reasonable parameters for them. As shown in the Appendix, jadeite samples are found to closely obey the flow law for dislocation climb

\[ \dot{\varepsilon}_{W,\text{climb}} = \kappa \frac{\mu \Omega D_{\text{eff}}}{b^5} \sigma^{4.5} \left( \frac{\sigma}{\mu} \right) \]

proposed by Weertman (1968) for physically reasonable magnitudes of the parameters involved (Table 3). Although the diopside data can, in principle, also be modeled by (2), the required magnitude of the parameters involved appears unreasonable (see the Appendix), and the microstructures and the observed relation of strength to composition suggest dislocation glide-controlled deformation instead. We employed a flow law

\[ \dot{\varepsilon}_{W,\text{glide}} = A_{\text{glide}} \sigma^{3.5} \exp \left[ -\frac{Q_{\text{glide}}}{RT} \left( 1 - \frac{\sigma}{\sigma_p} \right) \right] \]

as proposed by Weertman (1957) and found interestingly reasonable parameters (Table 3). In contrast, the power-law parameters quoted in Table 2 are purely empirical. In particular, the estimates of apparent activation energies elude a substantial discussion of their relation to composition or independent kinetic data as they correspond to different apparent stress exponents.

Starting from the endmember flow laws, simple scaling relations can be used to derive a zero-order description of the behavior of omphacite aggregates of intermediate composition within the two separate deformation regimes of glide and climb. The lack of understanding of how the sequential processes of glide and climb can be combined in a unifying flow law hinders a more comprehensive quantitative modeling of the compositional dependence of the rheology of omphacite aggregates across the regime boundaries.

In the case of climb-controlled creep [equation (2)], the thermal activation of the effective diffusion coefficient, presumably scaling with homologous temperature, dominates the effect of composition (see the Appendix). A flow law involving a parameter for bulk composition (\(X = 0\) corresponds to jadeite, \(X = 1\) corresponds to diopside) is thus potentially

\[ \dot{\varepsilon}_{W,\text{climb}}(X) \approx A_{\text{jd}} \sigma^{4.5} \exp \left( -\frac{Q_{\text{climb,jd}} + \lambda_1 X}{RT} \right) \]

with \(\lambda_1 \approx (72 \pm 10) \text{ kJ/mol}\). For our experimental conditions the relation between strength and bulk composition leads to

\[ \frac{\partial \ln \sigma^{\text{climb}}}{\partial X} \bigg|_{\dot{\varepsilon}, T} \approx 1 \cdot 1 \pm 0 \cdot 2. \]

For glide-controlled creep [equation (3)], the dependence of shear modulus on composition dominates the scaling relation. A composition-dependent flow law is

\[ \dot{\varepsilon}_{\text{glide}}(X) \approx A_{\text{glide,di}} \sigma^{2.5} \exp \left\{ -\frac{Q_{\text{glide,di}}}{RT} \left[ 1 + \lambda_2 (1 - X) \right] \left( 1 - \frac{\sigma}{\sigma_p,\text{di}} \right) \right\} \]

with \(\lambda_2 \approx \Delta \mu / \mu_0 \approx 0.2\). For our experimental conditions the expected effect on strength amounts to

\[ \frac{\partial \ln \sigma^{\text{glide}}}{\partial X} \bigg|_{\dot{\varepsilon}, T} \approx -\left( 1 \cdot 3 \pm 0 \cdot 6 \right). \]

The strength scaling according to equations (5) and (7) is in reasonable qualitative agreement with our observations (Figs 8 and 9a). In the glide regime, predictions and observations are fairly close, whereas the trend with composition in the climb regime appears underestimated. This discrepancy may be an artifact of transitional behavior (Fig. 14) or may be related to the finite melt content of those samples with the most sodic compositions.

**Geological implications**

Extrapolation of flow laws derived from laboratory experiments to natural conditions requires addressing two central issues. Are the same deformation mechanisms operative in nature and the laboratory? Which geological environments are characterized by thermodynamic conditions comparable with those in the experiments? At face value, our tests apply only to temperatures at which disordered omphacite is stable (\(T > 700^\circ C\), Fig. 1) and to ‘wet’ high-pressure environments as probably found in subduction zones. The previously suggested difference in CPO-relevant glide systems for ordered and disordered omphacite (Brenker et al., 2002) was questioned subsequently (Ulrich & Mainprice, 2005) and our results may well apply to ordered omphacite, too.

Our diopside samples qualify as ‘wet’; however, the kinetics of dislocation glide are intrinsically much less sensitive to point defect chemistry than the kinetics of dislocation climb and are probably less sensitive to the presence of water-related defects in the nominally anhydrous pyroxenes. Thus, the parameters (Table 3) of the Peierls-type flow law [equation (3)] used to model the glide-controlled deformation of diopside aggregates probably apply also to less hydrogen-rich environments. Numerical studies revealed that the Peierls Law may play a crucial role for plastic deformation to localize in shear zones (Karato et al., 2001; Kaus & Podladchikov, 2006).

It is probably impossible to judge whether glide was rate limiting during ancient deformation of omphacite in rocks exposed at present. Glide-controlled deformation may be inferred from the microstructural record, where inhomogeneous crystal plastic deformation is evident, whereas
features indicating climb and dynamic recrystallization are absent. Microstructures observed in omphacite in eclogitic rocks are variable (e.g. van Roermund & Boland, 1981; Boland & Tullis, 1986; Buatier et al., 1991; Godard & van Roermund, 1995; Muller & Franz, 2008). In most studies, deformation by dislocation creep is inferred from the presence of a CPO, without being supported by a characteristic microstructural record. Only recently, Mauler et al. (2001) suggested instead that the CPO in natural eclogites may be related to anisotropic growth rather than deformation.

Piepenbreier & Stockhert (2001) appear to have given the only report of unequivocal evidence for dynamic recrystallization of omphacite in eclogite rocks. The inferred temperature at deformation is slightly below 500°C. In accord with the constraints from this field study, the extrapolation of the flow laws derived in this study to geological strain rates indicates a switch from glide to climb between 400 and 500°C (Fig. 15). It should be noted that the predicted transition temperature is the lowest possible because our laws for the deformation of melt-bearing jadeite aggregates kinetically controlled by climb (Table 3, upper part) and diopside aggregates controlled by glide (Table 3, lower part) constitute lower bounds for the two deformation mechanisms in the system diopside-jadeite according to the discussed scaling relations. When allowing for the presence of minor Fe as typical for natural omphacites, however, some weakening compared with the current predictions is possible because of (1) the reduction in shear modulus (Duffy & Anderson, 1989) and (2) the acceleration of diffusion kinetics resulting

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**Fig. 14.** Schematic illustration of compositional dependence of (a) scaled and (b) absolute strength at indicated homologous and absolute temperatures, respectively. The combination of the opposite dependence of melting temperature and shear modulus on composition and of a switch in deformation regime leads to the increase in strength from either compositional endmember towards intermediate compositions at intermediate temperatures (900°C ≤ T2 ≤ 1000°C; this study). The increase from the sodic side where deformation is controlled by dislocation climb results from the increase in melting temperature with increasing calcic component, whereas the increase from the calcic side results from the increase in shear modulus with decreasing calcic component (the illustrated iso-temperature lines represent observations at T1 ≤ 750°C by Dorner & Stockhert (2004) and at T3 ≥ 1100°C).
from the relation between the multivalence of iron and the incorporation of hydrogen (e.g. Chen et al., 2006; Costa & Chakraborty, 2008). The weakening associated with the presence of Fe is probably limited considering the good quantitative correspondence of our results to the study of Zhang et al. (2006) on aggregates prepared from crushed natural, Fe-bearing omphacite crystals.

Although constituting lower bounds for Fe-free systems, the predicted stresses are high, of the order of 1 GPa, for a temperature of about 500°C. Geological observations suggest that stresses of this magnitude are not characteristic for long-term deformation in high-pressure subduction zone environments (for a review see Stockhert, 2002). We therefore propose that crystal plastic deformation, mechanical twinning, and dynamic recrystallization of natural omphacite at high stresses, as reported by Piepenbreier & Stockhert (2001), indicates localized and episodic high-stress cycles related to seismic activity in the subduction zone. This hypothesis is in accord with the geological record, experimental results, and methodological considerations. The structural record of such cyclic deformation with high peak stresses was previously identified in several crustal environments (Kuster & Stockhert, 1999; Trepmann & Stockhert, 2003; Birtel & Stockhert, 2008; Nüchter & Stockhert, 2006), and also was studied in laboratory experiments by Trepmann et al. (2007). The observation of recrystallized grains being restricted to the immediate vicinity of garnet crystals (Müller & Franz, 2008) is in accord with plastic deformation driven by local stress concentration.

Finally, considering that our samples qualify as ‘wet’ and that the incorporation of hydrogen into silicates tends to cause ‘hydrolytic weakening’, it is unlikely that drier natural omphacite deforms extensively by dislocation creep anywhere inside the Earth unless temperatures exceed about 700°C.

**CONCLUSIONS**

Triaxial compression experiments were performed on synthetic pyroxene aggregates with five compositions along the diopside–jadeite solid-solution series, in a solid-medium apparatus at a confining pressure of 2.5 GPa, temperatures between 900 and 1100°C, and strain rates between $10^{-15}$ and $3.5 \times 10^{-9}$ s$^{-1}$. The mechanical data indicate thermally activated creep and the microfabrics of the deformed samples show evidence of various deformation mechanisms involving dislocations. The observed magnitude of the stress exponent and the microstructural features, notably the abundance of subgrains, suggest that the deformation of the most sodic aggregates is controlled by dislocation climb. In contrast, the large apparent stress exponent and the positive correlation between strength and shear modulus near the diopside endmember strongly suggest that dislocation glide is the rate-controlling mechanism for the most calcic aggregates at the explored conditions. The apparent insensitivity of the deformation behavior of diopside aggregates to Fe and H content as deduced from a comparison with previous studies is in accord with these conclusions; in contrast to
climb-controlled deformation, glide-controlled deformation should be insensitive to impurity concentrations that affect diffusion kinetics. Our micromechanical modeling yields physically reasonable flow-law parameters. The extrapolation of the derived flow laws predicts stresses probably exceeding those actually realized on long time and space scales in subduction zones. As suggested previously for intermediate to lower crustal environments, localized and episodic high-stress stages related to Benioff zone seismicity may be responsible for the microstructural record of crystal plastic deformation of omphacite observed in natural eclogites.

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SUPPLEMENTARY DATA
Supplementary data for this paper are available at Journal of Petrology online.

REFERENCES


APPENDIX: SELECTION OF FLOW LAWS AND PARAMETER DETERMINATION

Various mechanisms have been considered rate-controlling in the deformation of polycrystalline silicates. Here, the small amount of melt in the sodic aggregates is thought to cause moderate weakening of our jadeite samples in comparison with the results of Orzol et al. (2006), as a result of its effect on the load-bearing area (e.g. Cooper & Kohlstedt, 1986) rather than by altering the rate-limiting deformation mechanism, as indicated by the agreement of the observed apparent stress exponents and activation energies between the two studies. The large stress exponents also exclude substantial contribution of diffusion creep. A contribution of grain boundary sliding — although unlikely considering the serrated grain boundaries — cannot be excluded per se, but the grain-shape accommodation has to be realized by dislocation processes rather than by diffusional transport, again because of the high stress exponents.

Twinning cannot be considered as rate controlling in steady-state flow, nucleation being quasi-instantaneous at a certain threshold of resolved shear stress, and growth by motion of partial dislocations generally being controlled by strain accommodation at the interfaces with adjacent grains of a polycrystal. Moreover, twinning is restricted to grains with a suitable crystallographic orientation, and these sites will be rapidly exhausted at a given stress level. Mechanical data and observed microstructures strongly suggest deformation related to dislocation processes. The models adopted below rest on the notion that the task of quantifying the overall kinetics of a large number of conceivable processes affecting the motion of a huge number of dislocations in a large number of grains constituting a polycrystalline aggregate can be reduced to identifying the slowest of sequential and the fastest of parallel processes. The motion of a dislocation comprises stages of unhindered glide and stages during which obstacles are overcome by climb of edge segments or cross-slip of screw segments (e.g. Hirth & Lothe, 1982; Reed-Hill & Abbaschian, 1994). After discussing potential rate-controlling mechanisms, we make the simplifying assumption that the available data for the two compositional end-members, jadeite and diopside, represent two distinct deformation regimes kinetically controlled by two different dislocation mechanisms, dislocation climb and glide, respectively. We neglect transitional behavior although deformation of diopside samples may involve dislocation climb at the highest temperature of ~1100°C as indicated by the near absence of a peak in strength for the intermediate compositions (Fig. 9a). Flow-law parameters are derived from sets of strain rate \( \dot{\varepsilon} \), strength or differential stress \( \sigma \), and absolute temperature \( T \) using a non-linear inversion method that accounts for the reported uncertainties in experimental data (Sotin & Poirier, 1984).

Is cross-slip relevant?

Cross-slip is described by rate equations (Poirier, 1985; de Bresser, 2002) that are formally similar to the glide equation employed below and, thus, cross-slip can be considered to be implicitly included in our approach. Cross-slip was explicitly discussed for olivine (Poirier & Vergobbi, 1978; Raterron et al., 2007) and calcite (de Bresser, 2002), yet its role as a rate-controlling step in silicates remains uncertain, climb being probably more effective at high temperatures because of fewer crystallographic constraints. Cross-slip requires the association of dissociated dislocations for the switch onto an alternative glide plane. Whereas dissociation of dislocations in clinopyroxene has been observed by TEM (e.g. Skrotzki, 1994), the role of cross slip in crystal plastic deformation of clinopyroxenes has not been explored in detail. In principle, for a glissile screw dislocation to overcome a discrete obstacle by changing the glide plane depends on the availability of a suitable plane in zone with the Burgers vector and in appropriate orientation, and the stress state to overcome the Peierls potential in the new system. The availability of a suitable plane is easily conceivable for metal phases with high symmetry and an according number of potential glide planes (e.g. Puschl, 2002; Lu et al., 2004; Wang et al., 2007). For clinopyroxenes, various glide systems have been invoked from CPO patterns and the post-mortem analysis of dislocations by TEM, in materials that have been deformed both naturally (Buatier et al., 1991; Godard & Van Roermund, 1995) and experimentally (Raterron & Jaoul, 1991; Raterron et al., 1994). In experiments at temperatures below 1000°C the glide system \{100\}[001] appears to be dominant, and at higher temperatures this is accompanied by \{110\}[001] and \{110\}1/2<110>, among others. The increased number of active glide systems may render cross slip a feasible mechanism at higher temperatures, at which climb also becomes more effective, as documented by the formation of low-angle grain boundaries in our experiments.

Climb-controlled deformation (jadeite aggregates)

The velocity with which dislocations climb is limited by the formation and mobility of jogs realized by vacancy...
diffusion. Weertman (1968) derived a general rate equation for climb-controlled creep:

\[
\dot{\varepsilon}_{W,\text{climb}} = \frac{\alpha}{b^3 \mu^2 D_{\text{eff}}} \left( \frac{\sigma}{\mu} \right)^{4.5}
\]

(8)

Here given according to Poirier (1985) (\(\mu\) is shear modulus, \(\Omega\) is atomic volume, and \(b\) is the Burgers vector). The dimensionless factor \(\chi\) is of order unity and may differ for the two diffusion processes considered to contribute to the effective self-diffusion coefficient:

\[
D_{\text{eff}} = D_l + \pi R_p^2 \rho_d D_{dc}
\]

(9)

For volume diffusion through the lattice \((D_l)\) and short-circuit diffusion along dislocation cores \((D_{dc})\) of width \(R_p\). Equation (8) covers popular special cases of ‘power-law creep’ with stress exponents in the range of 3–6.5, when the stress dependence of the density of dislocation sources, \(M\), and dislocation density, \(\rho_d\), are incorporated appropriately (see Poirier, 1985). The formulation of the effective diffusion coefficient assumes that the fastest of the alternative paths for the slowest constituent controls the overall kinetics. The exact formulation of the contribution from core diffusion is a matter of debate (e.g. Frost & Ashby, 1982; Balluffi et al., 2005). In this model, the temperature dependence of creep is determined by the activation enthalpies of the involved diffusion coefficients; for example, \(D_l = D_0 \exp(-\Delta H_l/RT)\). The apparent stress exponent of close to four observed for jadeite aggregates (Table 2, Fig. 8a) is most obviously explained by a combination of a moderate dependence of dislocation density on stress and lattice diffusion as the rate-controlling parameter in equation (8). Specifically, for \(M = \text{const.}\) and \(D_{\text{eff}} = D_l\) the stress exponent assumes a value of 4.5. We consider \(M \sim 10^{6.5 \pm 1.5}\) m\(^{-3}\) to represent a reasonable range for the dislocation source density; that is, 1–1000 sources in a grain of a diameter of \(\sim 10\) \(\mu\)m. The lower bound of this range was quoted by Weertman (1957) when modeling the deformation of zinc. With this estimate and \(p_d \sim 75\) GPa, \(b \sim 5 \times 10^{-10}\) m, and \(\Omega_p \sim 10^{-29}\) m\(^3\), we gain an order of magnitude for \(D_l\) at 1000°C where we used the pre-exponential factor \(A_{\text{climb}}\) and an activation enthalpy for creep \(\Delta H_{\text{climb}}\) inverted for a prescribed stress exponent of 4.5 (Table 3). Alternatively, for \(M = M_0\sigma/\sigma_0^2\) and \(D_{\text{eff}} \approx D_l\) the stress exponent amounts to 3.5. With \(M_0 \sim 10^{6.5}\) m\(^{-3}\) at \(\sigma_0 \approx 100\) MPa, we gain almost the same estimate for \(D_l\). These two estimates compare well with directly measured lattice diffusion coefficients for Si in various silicates (Bejina & Jaoul, 1997), in particular considering (1) that it may be overestimated by up to an order of magnitude because of the weakening effect of melt and (2) the relatively low melting temperature of jadeite. Direct determinations of diffusion kinetics are lacking for jadeite crystals. A dominance of dislocation core diffusion (i.e. \(D_{\text{eff}} \approx \pi R_p^2 \rho_d D_{dc}\)) is in contrast unlikely for jadeite aggregates, because reaching a stress exponent of about four then requires an unrealistically strong dependence of the dislocation source density on stress of \(M \propto \sigma^4\) respecting the well-established scaling relation \(b^5 \rho_d = (\sigma/\mu)^2\) with \(\sigma\) of the order of 0–1 (Taylor, 1934; Takeuchi & Argon, 1976; Kohlstedt et al., 1995).

In principle, the diopside data can also be modeled by equation (8) which allows for stress exponents as high as 6.5 that reach the lower limit of the determined range (Table 2). An exponent of 6.5 results for a dominance of core diffusion (i.e. \(D_{\text{eff}} \approx \pi R_p^2 \rho_d D_{dc}\)) and a stress-independent density of dislocation sources. For this scenario, we gain an order of magnitude estimate of the coefficient for dislocation core diffusion of \(D_{\text{climb}}^{ij} = \sqrt{\gamma^i \gamma^j} \mu^2 b^3 M^5 k T\exp(-\Delta H_{\text{climb}}^{ij}/RT)/(\pi \Omega_p \Delta H_{\text{climb}}^{ij})\approx 10^{-16.5} \text{m}^2/\text{s}\) that appears too low compared with the order of magnitude of interface diffusion coefficients in silicates (Farver & Yund, 2000; Milke & Heinrich, 2002) that, in the absence of appropriate data, may serve as a lower bound for dislocation core diffusion coefficients (Balluffi et al., 2005). An estimate too low in comparison with independently determined diffusion coefficients is in accord with our reasoning that climb is not rate-controlling in diopside aggregates at the explored conditions. Climb could yield a faster than the observed rate, but glide as the slower sequential step is rate-limiting. Because cross-slip is operating parallel to climb, it can also be discarded as rate-limiting.

**Glide-controlled deformation (diopside aggregates)**

The lattice resistance represented by the Peierls stress \(\sigma_p\) constitutes the central mechanical parameter for dislocation glide. An explicit flow law for glide-controlled creep

\[
\dot{\varepsilon}_{\text{glide}} = 12\nu \frac{\sigma}{b^2 \mu^2 M^5} \left( \frac{\sigma}{\mu} \right)^{2.5} \exp \left( -\frac{Q_{\text{glide}}}{RT} \left( 1 - \frac{\sigma}{\sigma_p} \right) \right)
\]

(10)

was proposed by Weertmann (1957), which formally coincides with that given by Frost & Ashby (1982) when the dislocation source density \(M\) is assumed to be linearly proportional to stress \((a\) is the distance between glide planes). The frequency of lattice vibrations

\[
\nu = \sqrt{\frac{5 \sigma_p}{4ab \mu}}
\]

(11)

(shear-wave velocity \(c_s^2 = \mu/\rho\)) and the apparent activation energy

\[
Q_{\text{glide}} = N \lambda \frac{32a^3 b^5 \mu \sigma_p}{10 \pi^2} \approx 0.57 a^3 \lambda b^5 \mu \sigma_p
\]

(12)
both scale with the square root of the Peierls stress. Relying on the estimate of the Peierls stress for an edge dislocation from elastic theory \( \text{e.g. Hirth & Lothe, 1982} \), the prefactor \( 12\pi a/b^2M^0X^1 \) in equation (10) can be written as

\[
12\pi a/b^2M^0X^1 = \frac{5\alpha}{\pi(1-\rho)ME^0} \exp\left(-\frac{\pi a}{(1-\rho)b}\right)
\]

\( \approx \frac{v_s \sigma_{P}^{11/12}N_{A}^{3/4}}{M^{1/12}Q_{Gl}^{3/2}}. \)  

Because Peierls stress, apparent activation energy, and pre-exponential factor are not independent of each other, we can either continue with the traditional parameterization of the flow law using \( \sigma_{P} \) and \( Q_{Gl} \), or alternatively take the perspective that the three central, purely geometrical characteristics of the gliding dislocations, \( a, b \) and \( M \), control strain rate. It is important to emphasize that in equations (11)–(13) derived from elastic models the parameters \( a \) and \( b \) have phenomenological rather than a simple crystallographic character for multi-constituent minerals with low symmetry and complex bonding (Hirth & Lothe, 1982). Furthermore, the representation of the elastic behavior of a symmetrical and complex bonding (Hirth & Lothe, 1982) of intermediate composition. The activation enthalpy observed for jadeite aggregates (Table 3) yields an estimate of \( \kappa \approx 32.3 \pm 4.1 \), a value well within the expected range (see Poirier, 1991).

For our analysis, we express bulk composition such that \( X = 0 \) and \( X = 1 \) correspond to jadeite and diopside, respectively. We assume \( \partial \sigma^0/\partial X \approx \partial \sigma/\partial X \approx \partial \Omega/\partial X \), that is, the geometrical characteristics of the glide system vary with composition as the unit cell does, and estimate (1) \( \partial \ln \sigma/\partial X \approx -0.02 \) using density values \( \rho_{jd} = 3260 \text{ kg/m}^3 \) and \( \rho_{di} = 3400 \text{ kg/m}^3 \), (2) \( \partial \ln \mu/\partial X \approx -0.25 \) using \( \mu_{jd} = 87.8 \text{ GPa} \) and \( \mu_{di} = 70 \text{ GPa} \), and (3) \( \partial \ln \Omega/\partial X \approx -0.46 \) with melting temperatures of \( T_{m,jd} \approx 1653 \text{ K} \) and \( T_{m,di} \approx 1923 \text{ K} \) at the pressure of our experiments (Bell & Davis, 1969). tracking explicitly the dependence of all parameters involved in equation (8) on composition, we find

\[
\frac{\partial \ln \sigma_{P}^{\text{climb}}}{\partial X} \approx 3 \cdot \frac{\partial \ln \mu}{\partial X} + \frac{\partial \ln \Omega}{6 \partial X} + \frac{\partial \ln D}{\partial X} \approx -\frac{\kappa}{\Theta^2} \frac{\partial \sigma}{\partial X}
\]

\( \text{(14)} \)

demonstrating that the homologous temperature scaling of the diffusion coefficient contributes dominantly to the dependence of climb-controlled strength on composition. The dependence of shear modulus on composition is subordinate and the relation of lattice parameters on composition is negligible.

In contrast, the dependence of shear modulus on composition dominates the scaling relation for glide-controlled creep, as \( \partial \ln \mu/\partial X \gg \partial \ln \Omega/\partial X \). From the full analysis, we find that the variation of strain rate with composition is actually controlled by the relation between the apparent activation energy and shear modulus (12)

\[
\frac{\partial \ln \sigma_{P}^{\text{glide}}}{\partial X} \approx \frac{Q_{Gl}}{RT} \left(1 - \frac{\sigma}{\sigma_{P}}\right) \frac{\partial \ln \mu}{\partial X}
\]

\( \text{(15)} \).