

E. Thurel · P. Cordier · D. Frost · S.-I. Karato

Plastic deformation of wadsleyite: II. High-pressure deformation in shear

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Abstract We have studied the dislocation microstructures that develop in $(\text{Mg}_{0.9}\text{Fe}_{0.1})_2\text{SiO}_4$ wadsleyite deformed by simple shear at high pressure. The experiments were performed in a multianvil apparatus with the shear assembly designed by Karato and Rubie (1997). The samples were synthesized in a separate experiment from high-purity oxides. The deformation experiments were carried out at 14 GPa and 1300 °C with time durations ranging from 1 to 8 h leading to plastic shear strains of 60 and 73%, respectively. The microstructures investigated by transmission electron microscopy (TEM) show that dislocation glide is activated under these conditions over the whole experimental time. The easy slip systems at 1300 °C involve $1/2 \langle 111 \rangle$ dislocations gliding in $\{101\}$ as well as $[100]$ dislocations gliding in (010) and $\{011\}$.

Keywords Plastic deformation · Dislocations · Slip systems · High pressure · TEM

Introduction

We have described in Part I of this Study the microstructures of wadsleyite samples deformed in compression in a multianvil apparatus. This study led to a detailed characterization of the various dislocations and slip systems that can be activated in wadsleyite. How-

ever, this mineral bears significant plastic deformation during cold compression in the multianvil deformation experiments. The microstructures observed post mortem are thus a result of the annealing (plus further deformation) of the microstructures introduced at cold temperature. This makes it difficult to withdraw a temperature signature from these experiments. It is in principle possible to overcome this problem either by minimizing cold deformation or by increasing the strain at high temperature for the high-temperature microstructure to superimpose over the previous one. This can be achieved by using the shear deformation geometry developed by Karato and Rubie (1997) which allows large strains (over 100%) to be produced at high pressure and high temperature.

In this study, deformation of $(\text{Mg}_{0.9}\text{Fe}_{0.1})_2\text{SiO}_4$ wadsleyite was carried out using a multianvil apparatus. As in Part I, the samples were presynthesized in an initial high-pressure experiment and then compressed in a second experiment designed to induce shear deformation. The deformation microstructures are characterized by transmission electron microscopy and large-angle convergent beam electron diffraction.

Experimental

The high-pressure experiments were performed at the Bayerisches Geoinstitut. The experimental details have been described in Part I. For this study, we used San Carlos olivine (Fo90) as a starting material. The experimental conditions used for the synthesis of wadsleyite's are given in Table 1. After synthesis, the specimen was recovered and cut into slices. The slices were then sandwiched at 45° between two hard alumina pistons and placed in a 14-mm high-pressure deformation assembly (Fig. 1) as described by Karato and Rubie (1997). Stresses generated during compression induce simple shear of the specimen at high temperature. The total strain was measured after the experiment from the rotation of a Pt strain marker vapour deposited onto cut surfaces of the sample. The deformation conditions as well as the strains achieved are given in Table 2. After the deformation experiments, the samples were recovered and prepared for TEM examination at the University of Lille following the procedure described in part I.

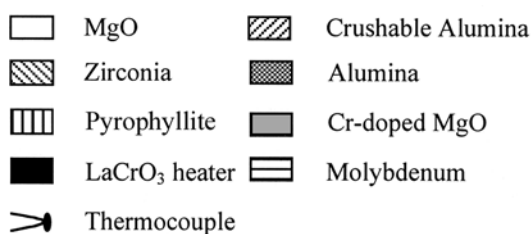
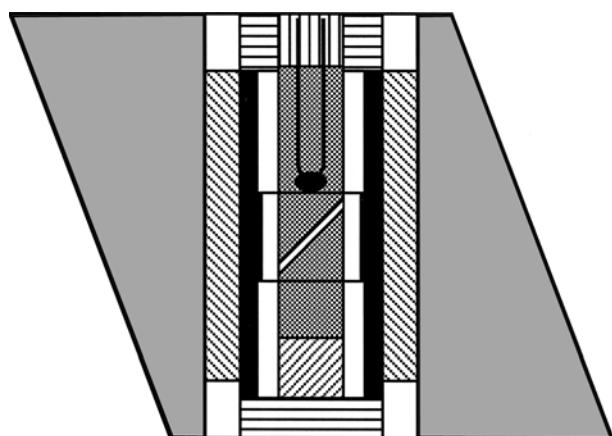
E. Thurel · P. Cordier (✉)
Laboratoire de Structure et Propriétés de l'Etat Solide,
UMR CNRS 8008
Université des Sciences et Technologies de Lille, France
e-mail: Patrick.Cordier@univ-lille1.fr
Tel.: +33 320 43 43 41; Fax: +33 320 43 65 91

P. Cordier · D. Frost
Bayerisches Geoinstitut,
Universität Bayreuth, Germany

S.-I. Karato
Department of Geology and Geophysics, Yale University,
New Haven, USA

Table 1 Experimental conditions for wadsleyite synthesis in the multianvil apparatus

Run	Press	Oil pressure (bar)	Pressure (GPa)	Temperature (°C)	Duration (h)
S2326	Sumitomo	400–420	14	1300	3

**Fig. 1** Schematic of the shear deformation assembly. The non-hydrostatic compressive stress generated during compression causes plastic shear at high temperature of the thin sample disc sandwiched at 45° between the two central alumina pistons

TEM observations

Sample S2333

The specimen has a fairly homogeneous grain size of the order of 10 μm. Dislocations in glide configuration are found in all grains with an average density of $2 \cdot 10^{13} \text{ m}^{-2}$. Dislocations are mostly of the $1/2 \langle 111 \rangle$ and $[100]$ type as seen from the contrast and verified from large-angle convergent beam electron diffraction (LACBED). $1/2 \langle 111 \rangle$ dislocations are found to glide in $\{101\}$ (seven independent characterizations). $[100]$ dislocations glide in (010) and $\{011\}$ (Fig. 2) (one and two characterizations, respectively). Only one occurrence was found of $[010]$ dislocations (Burgers vector checked with LACBED) gliding in $\{101\}$. Most of the grains contain many stacking faults which belong mainly to the (010) plane, but exhibit numerous ledges (Fig. 3).

Table 2 Experimental conditions for plastic deformation experiments in the multianvil apparatus

Run	Press	Oil pressure (bar)	Pressure (GPa)	Temperature (°C)	Duration (h)	Shear strain (%)
S2333	Sumitomo	400	14	1300	1	60
S2336	Sumitomo	400	14	1300	8	73

Sample S2336

The microstructures in this sample are very similar to those observed in S2333. Gliding dislocations with a mean density of $2 \cdot 10^{13} \text{ m}^{-2}$ are ubiquitous. The slip system $1/2 \langle 111 \rangle \{101\}$ has been observed and fully characterized five times (Fig. 4). $[100]$ dislocations have been found to glide in (010) and $\{011\}$. Stacking faults with ledges are observed in every grain (Fig. 5). The faults are out of contrast when imaged with $g = 080$ (this can be seen on the part of the faults which are not parallel to (010)). This contrast is incompatible with a fault vector $1/4[010]$ and thus suggests a fault vector of $1/2 \langle 101 \rangle$ (Madon and Poirier 1983; Price 1983).

Discussion and conclusion

The two samples described in this study were deformed under identical conditions of pressure and temperature but for time durations of 1 and 8 h. All the indicators (rotation of the strain marker, microstructures) seem to imply that deformation was still taking place in experiments performed up to 8 h. The dislocation microstructures also appear very similar in both samples although the stresses and strain rates might differ. These microstructures are much more simple than those observed in compression deformation experiments (Part I). Only $[100]$ and $1/2 \langle 111 \rangle$ slip are significantly activated. $[010]$ dislocations, which were thought to result from cold compression and were very common in specimens deformed in compression, were detected only marginally. This suggests a thermomechanical history for shear deformation experiments which is very different from the one encountered in compression experiments. The cold deformation is probably minimized although our experiments give no hint about the magnitude of this effect. Whatever the importance of cold deformation, it is clear that the high-temperature deformation event is strong enough to impose its microstructure. It is also clear that a significant deformation regime can be maintained over long durations in these shear-type experiments.

It seems thus possible to gain information from these experiments about the deformation mechanisms which are active in wadsleyite at 1300 °C:

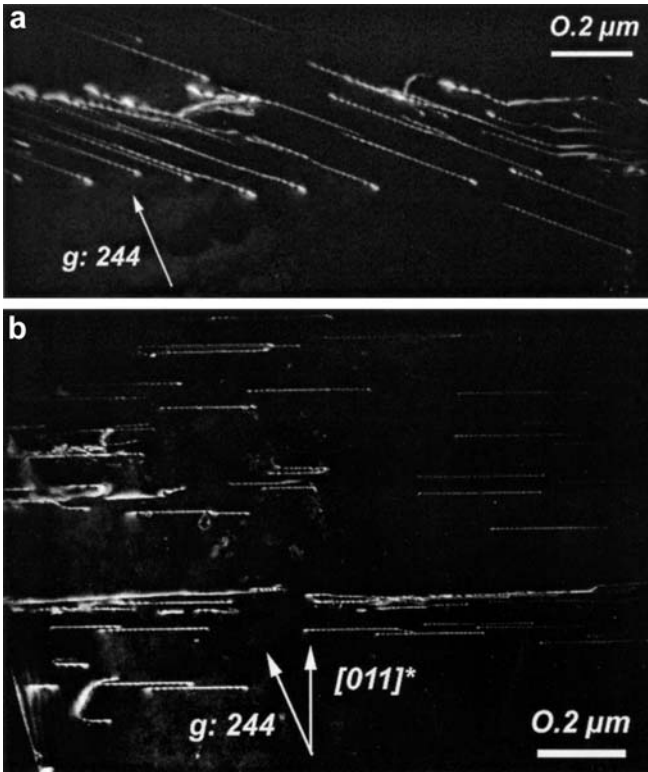


Fig. 2a,b Sample 2333. TEM micrographs showing $[100]$ dislocations in a glide band. Weak-beam dark field with $g: 244$. The specimen has been tilted so as to put the slip plane which is parallel to $\{011\}$ edge-on. Weak-beam dark-field with $g: 244$

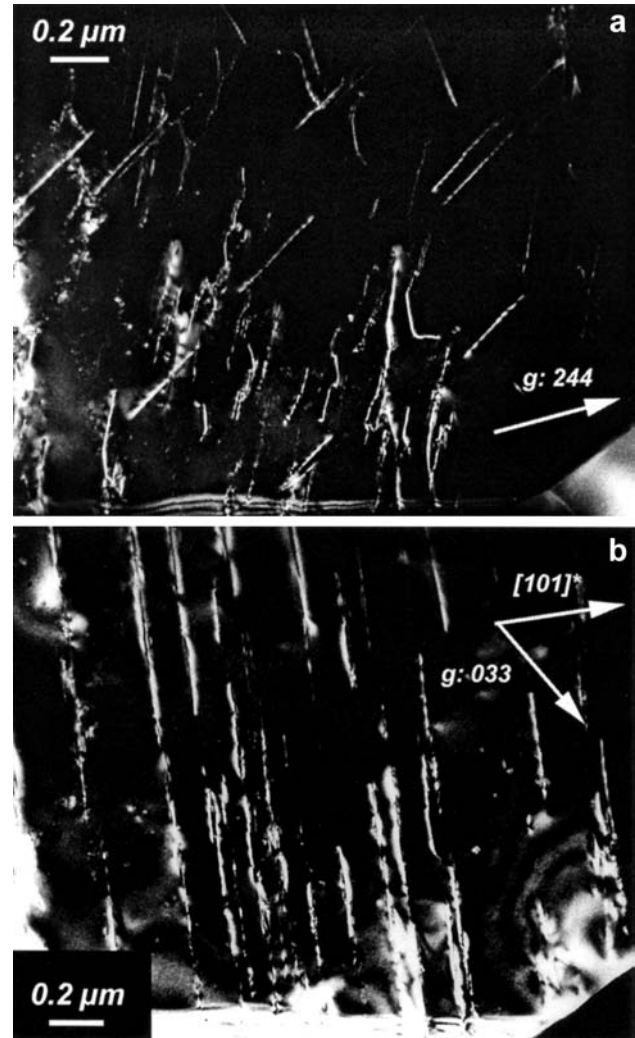


Fig. 4a,b Sample 2336. TEM micrographs showing $1/2\langle 111 \rangle$ dislocations. Weak-beam dark-field with $g: 244$. Same area (see dislocation 1) with the specimen tilted so as to put the slip, $\{101\}$, edge-on. Weak-beam dark field with $g: 033$

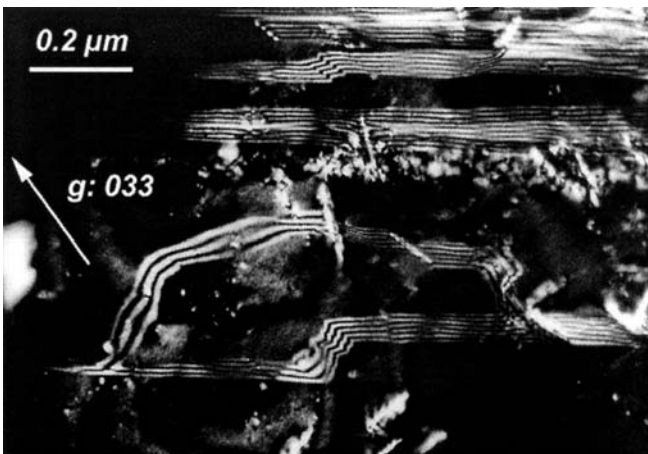


Fig. 3 Stacking faults in sample 2333. Note the numerous ledges when the fault changes its plane. Weak-beam dark field

– the most striking fact is that dislocation glide appears clearly to be the dominant mechanism. This is not in agreement with Dupas–Bruzek et al. (1998), who concluded that dislocation climb was activated in wadsleyite formed at $900\text{ }^{\circ}\text{C}$. This conclusion was further supported by Mosenfelder et al. (2000), who interpreted their microstructures (constituted mostly of subgrain boundaries) as resulting from plastic

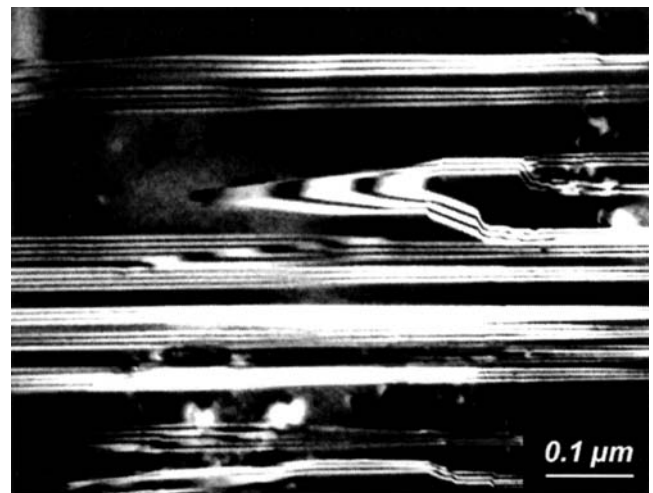


Fig. 5 Stacking faults in sample 2336. Note the numerous ledges and hairpin shapes. Weak-beam dark field

deformation. It must be stressed, however, that both studies are based on olivine to wadsleyite phase transformation experiments (under non-hydrostatic stresses). The microstructures observed are thus very likely to result from nucleation and growth of wadsleyite rather than from plastic deformation. This alternative interpretation would also explain why the rheological approach of Mosenfelder et al. gives rise to such surprisingly high values for the strength of wadsleyite.

- The easy slip systems activated in wadsleyite at 1300 °C involve $1/2 \langle 111 \rangle$ dislocations gliding in $\{101\}$ as well as $[100]$ dislocations gliding in (010) and $\{011\}$.
- The origin of the numerous stacking faults has been questioned. Their fault vector could be compatible with the glide of partial dislocations, as suggested by Sharp et al. (1994). However, detailed examination of the faults show many ledges as well as some folds which exclude any deformation origin. The higher density of faults in these samples compared to those observed in Part I could tentatively be interpreted as being a consequence of the higher content in iron. Indeed, stacking faults in (010) can be described in wadsleyite as a thin layer (one or two atomic layer) with the ringwoodite structure. Iron, which stabilizes the ringwoodite structure, could well stabilize the faults during growth as well.

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