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Modelling in magnesium alloy with superplastic layer: implications for shear in fault zones induced in olivine by phase transformation

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Analysis of a recently discovered high-pressure phase-transformation-induced mechanism of shear failure in Mg_2GeO_4 olivine has produced evidence that sliding in the resulting fault zone is accomplished by superplastic flow of the extremely fine-grained high-density phase produced during the transformation. This failure mechanism is of interest because it may be the mechanism by which deep earthquakes are generated in the earth's mantle. To gain insight into this process, we have conducted model tensile experiments on coarse-grained, non-superplastic, specimens of Mg–15%Mn–0.3%Ce alloy, within which a fine-grained, superplastic, planar zone was fabricated at an orientation of 45° to the stress axis. Flow was largely restricted to shear offset within the superplastic zone. The experiments were interrupted periodically and microstructural observations were made. Repeated detailed observation of several regions at different strain levels showed that the main mechanism of shear operative in the superplastic region was grain-boundary sliding occurring in a layer-by-layer manner. The common features of microstructural change observed in the magnesium alloy and in the Mg_2GeO_4 olivine fault zones suggests that such cooperative grain-boundary sliding could be the mechanism of fault propagation in the deep earth and therefore important for understanding deep-focus earthquakes.

1. Introduction

The phenomenon of superplasticity (SP) has been observed in a vast range of metallic and ceramic systems having fine-grained structure [1–3]. The similarity in the features of superplastic behaviour in metals and ceramics, for example, low flow stress, equiaxed grain shape after significant deformation, etc., has been reported [4, 5]. Recently, SP flow has been suggested as an important process of deformation [6, 7] and failure [8–12] of geological materials deep in the mantle of the earth.

Green and Burnley [8], proposed a model of deep-focus earthquakes based on the hypothesis that fault development involves formation of and shear along thin spinel layers by superplastic processes proceeding in ultrafine-grained spinel. This model was derived from high-pressure experiments on Mg_2GeO_4 olivine that develops a faulting instability while undergoing incipient transformation to a denser phase with spinel structure. The model explains the microstructures produced [8–10] and it has successfully predicted acoustic emissions accompanying stress drop and fault formation [11], and the viscous manner of sliding on the fault after its formation [12]. The faulting has been confirmed in mantle olivine, $(Mg,Fe)_2SiO_4$ at a pressure of 14 GPa [13].

Experiments performed in tension in a magnesium alloy [14] and titanium alloys [15] with structure consisting of regions with SP structure and non-SP structure, confirm that localization of deformation occurs in SP region(s), but these previous works were restricted to coaxial deformation. In contrast, it is shown here that specimens of Mg–1.5%Mn–0.3%Ce alloy deformed in tension with a SP layer inclined to the specimen axis exhibit macroscopic shear of non-SP regions along the SP layer. This result is similar to that observed in the geological experiments. Repeated observation of the same region after successive increments of strain in the magnesium alloy showed that shear in the SP layer occurs through sliding of grains in layer-by-layer manner. This information may be important for better understanding of fault propagation at high pressure in geological materials and the mechanism for deep earthquakes.

2. Experimental procedure

The results from two different sets of experiments are

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reported here. The first set of experiments was performed on Mg_2GeO_4 olivine. The Mg_2GeO_4 olivine polycrystals had an initial grain size of approximately 30 μm, and contained 5% Mg_2GeO_3 pyroxene inclusions [10]. Right-circular cylinders of Mg_2GeO_4 were deformed in a modified Griggs piston-cylinder apparatus using solid NaCl as the pressure medium. Samples were deformed at strain rates of 10^{-3}–$10^{-5}\ s^{-1}$, temperatures of 1200–1500 K and pressures of 1–2 GPa. The details of mechanical testing procedures and preparation of samples of microstructural investigations are described elsewhere [10].

The magnesium alloy samples were fabricated to contain a layer 2 mm thick with a grain size of 10 μm sandwiched between two regions with a grain size of about 100 μm (Fig. 1). The orientation of the fine-grained layer was 45° to the tensile axis. This structure was produced in specimens with an initial grain size of 10 μm having shapes shown by the dashed line in Fig. 1 that were subjected to cold deformation up to a critical primary recrystallization strain of about 3% [14] followed by annealing at 670 K for 40 min. The region of each sample with a larger cross-sectional area remained undeformed and the size of grains was unaltered by annealing, whereas in the narrower regions of the gauge length the grain size after critical primary recrystallization increased to 100 μm. Finally, the cross-section was made uniform along the gauge length by machining. The Instron testing machine was used for deformation in the optimal superplastic regime for this material with a grain size of 10 μm [16]: $\dot{\varepsilon} = 4 \times 10^{-4}\ s^{-1}$; $T = 670$ K. The specimens were electrochemically polished prior to deformation and a network of scratches with cell size of 50 μm was inscribed by means of a diamond needle. Individual specimens were unloaded after deformation to 10%, 20%, 30% and studied by optical microscopy and scanning electron microscopy (SEM).

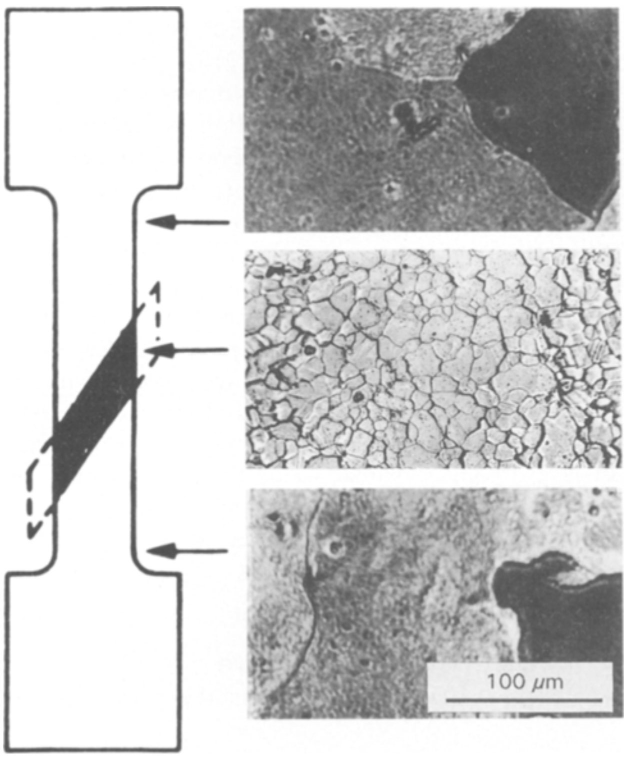
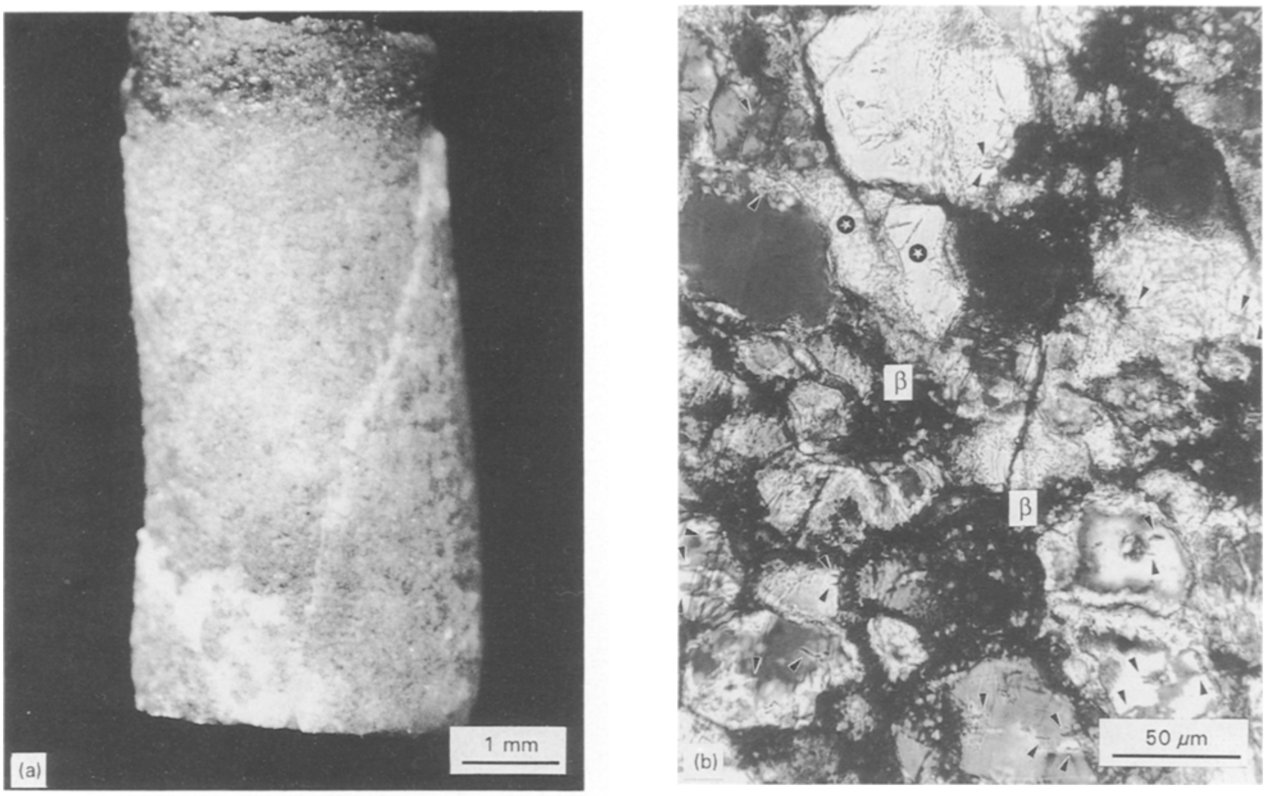


Figure 1 Mg–1.5%Mn–0.3%Ce alloy specimen with superplastic layer (fine-grained region in photomicrograph) before deformation.

3. Results

The high-pressure faulting phenomenon triggered by the densification phase transformations in Mg_2GeO_4 and $(Mg,Fe)_2SiO_4$ is illustrated in Fig. 2. Fig. 2a shows a specimen of Mg_2GeO_4 that developed two faults during failure. Both faults lie at about 25°–30° to the compression axis. A photomicrograph of a fault in $(Mg,Fe)_2SiO_4$ that has slipped only about 20–30 μm is shown in Fig. 2b. Regions of β-phase are found along the olivine grain boundaries and can also be seen lining the fault where it cuts through and displaces a single crystal. Fig. 2c shows the interior of a fault zone Mg_2GeO_4 that has slipped many tens of micrometres; it consists of a matrix of very fine-grained γ-phase ($\ll 1$ μm), with a larger fragment of olivine "floating" in it. The "anticracks" in which the very fine-grained denser phase is produced in both systems exhibit grain sizes of less than 50 nm [10].

The model experiments on magnesium alloy with the fine-grained superplastic (SP) layer were performed to test the hypothesis that uniaxial tensile testing of specimens with this microstructure would extend by macroscopic shear of the non-SP regions along the SP layer. Fig. 3a shows the surface of the prepolished sample after extension of 30%. Deformation is localized in the region having fine-grained superplastic structure. It is important to note that deformation in the fine-grained structure approximates simple shear. However, it did not occur by grain-boundary sliding (GBS) of individual grains distributed evenly throughout the SP region. Rather, it occurred by shear of fine-grained layers along grain-boundary surfaces, oriented approximately parallel to the boundaries of the SP region (at 45° with respect to the tensile axis). Such surfaces of cooperative GBS are seen more clearly under high magnification (arrowed in Fig. 3b). Scratch offsets at grain boundaries confirm that shear occurred primarily along grain boundaries. However, some grains were cut by propagating shear (Fig. 3c, arrowed). Fig. 4a–f shows a single interface region between fine and coarse grains after progressively increasing strain: $\varepsilon = 10\%$ (a), $\varepsilon = 20\%$ (b), $\varepsilon = 30\%$ (c). It is seen that coarse grains have deformed by dislocation slip, whereas the main deformation mechanism in the fine-grained layer is GBS (some non-uniformity of grain-boundary sliding in the fine-grained layer was observed). Little GBS occurred at the interface between the coarse-grained region and fine-grained region. The maximum of local deformation, determined by the change in spacing of nodes in the scribed network, occurred in the middle of the fine-grained layer (Fig. 5); at both sides of the SP region, there is a zone about one-fifth of the width of the layer where the GBS of fine grains is reduced. Irregularities at grain boundaries of coarse grains may be one of the reasons for the existence of the transition layers of restricted GBS.



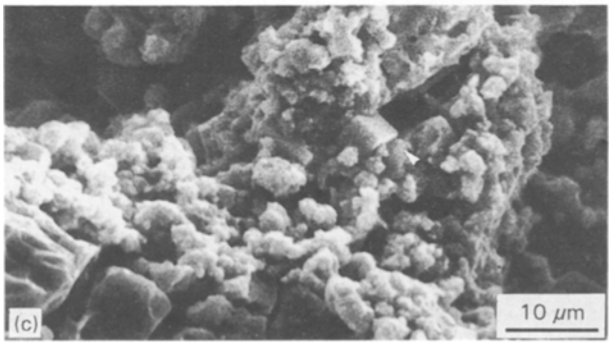


Figure 2 Phase-transformation-triggered faulting in olivine. (a) Specimen of Mg_2GeO_4 that failed at $T = 1200$ K, $P = 1$ GPa, $\dot{\varepsilon} = 10^{-4}\ s^{-1}$; (b) photomicrograph (crossed polarizers) of thin section of specimen of $(Mg_{0.9}, Fe_{0.1})_2SiO_4$ that failed at $T = 1550$ K, $P = 14$ GPa, $\dot{\varepsilon} = 10^{-4}\ s^{-1}$. The β-phase (lower birefringence and higher refractive index) can be seen on grain boundaries, in "anti-cracks" (arrowheads) and lining the fault zone where it cuts through a single grain (asterisks); (c) secondary electron image of fault zone in Mg_2GeO_4 etched with HCl which selectively attacks the olivine phase. Olivine at the border of the fault zone appears at lower left. Fault zone consists of spinel-structured polymorph with a grain size $\ll 1$ μm. White arrow points to an olivine fragment within the fault zone.

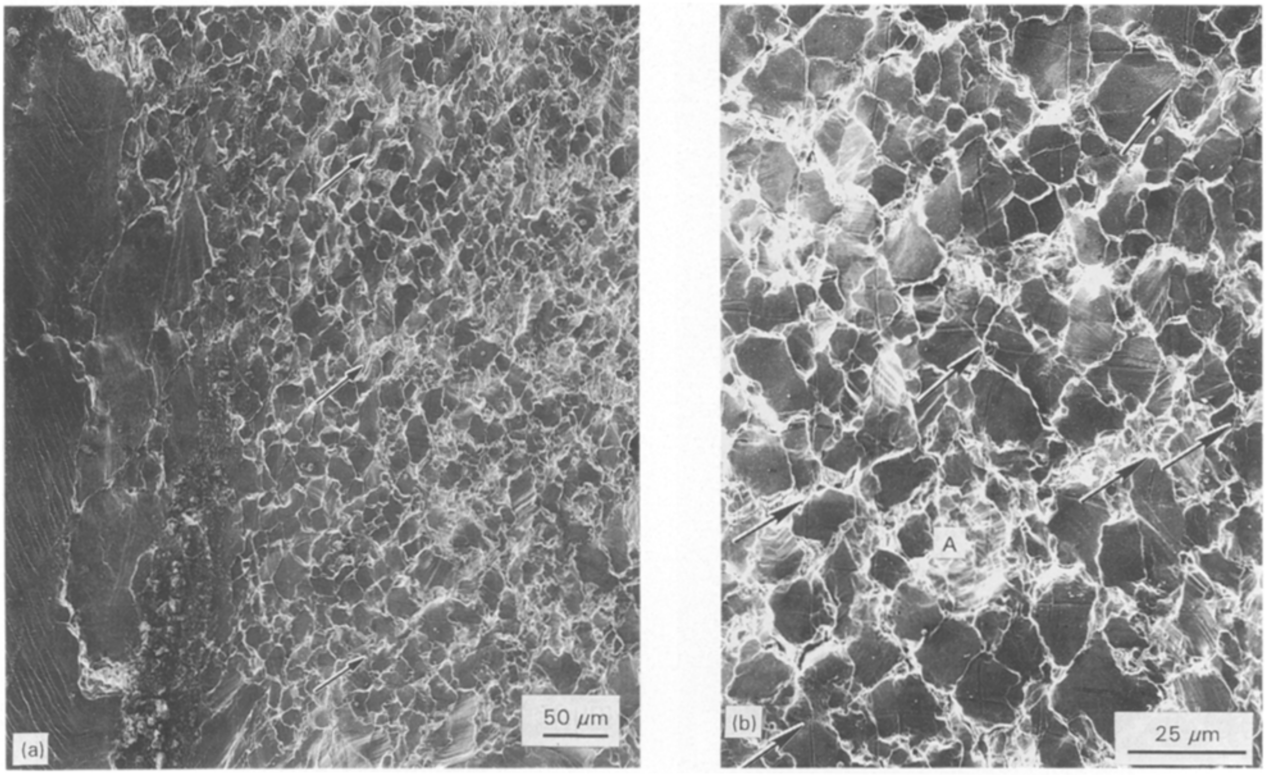
4. Discussion

Fig. 6 schematically shows the possible ways deformation can occur for cases in tension (Fig. 6a–c) and in compression (Fig. 6d–f). There are two possibilities for concentration of deformation in the SP layer: (1) shear of the coarser grained regions along the SP layer (Fig. 6b and e) or (2) axial strain (Fig. 6c and f). The first alternative has been observed in the present experiments in magnesium alloy. It supports the basic suggestions of Green and coworkers [8–13] of their model of localized shear propagation in olivines. Importantly, however, the detailed observations of the deformation of the SP region in this study enable us to clarify the mechanism by which shear takes place in such thin SP layers, There are four possible mechanisms by which such shear can occur:

1. GBS of individual grains;
2. localized GBS causing shear of grains in layer-by-layer manner (Fig. 7a);
3. sliding on the interface between SP and non-SP regions due to rotation of fine grains (Fig. 7b) as has been discussed by Vladimirov [17];
4. intragranular shear deformation of fine grains (Fig 7c).

In this study, repeated observation of the same region at successive stages of deformation showed slip lines in only a few grains and retention of an equiaxed grain shape even after large strain. Hence, the fourth mechanism, which calls for significant intragranular deformation, cannot play an important role in shear. Deformation was concentrated in the middle of the SP layer, suggesting that the third mechanism, which predicts more active deformation near the SP and non-SP interface is not important either. The offsets of scratch marks characterizing the shear of grain groups and the traces of the intensive shear of grain layers (Fig. 3) suggest that mechanism two, layer-by-layer shear of grains is the dominant deformation mechanism. This conclusion is supported by recent observation of similar cooperative GBS in SP flow in other materials [14, 18, 19].

There are two main difficulties in extending the observed features of the deformation in magnesium alloy to that for transformation-induced faulting in



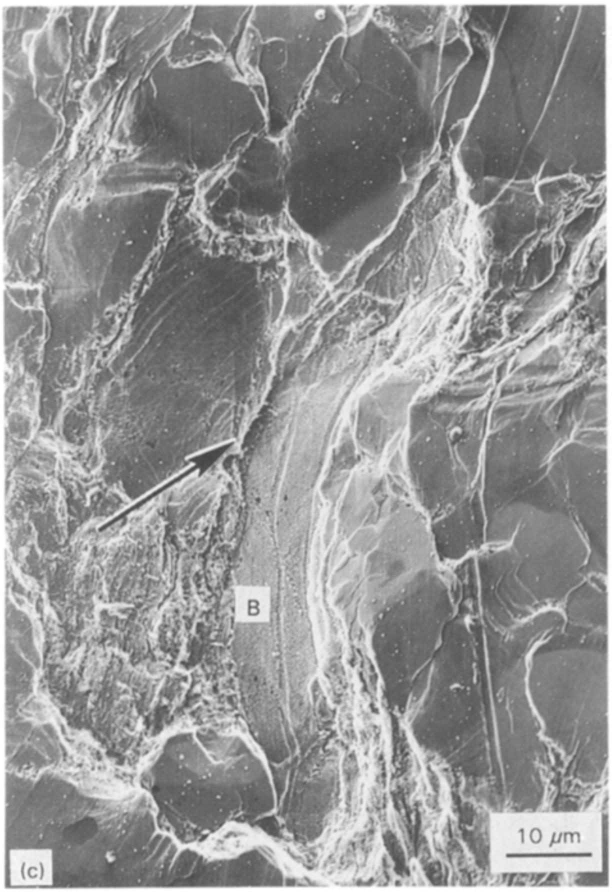


Figure 3 Deformation relief, arising at the prepolished surface of Mg–1.5%Mn–0.3%Ce alloy after superplastic deformation up to 30%, $\dot{\varepsilon} = 4 \times 10^{-4}\ s^{-1}$, $T = 673$ K. (a) Traces of active deformation in SP layer, (b) shear along grain boundary surfaces (arrowed) and (c) grains cut during shear process (labelled A in (b) and B in (c)) are seen under different magnification. Tensile axis is vertical.

olivine; (i) the difference in nature of materials, and (ii) the difference in type of deformation mode, i.e. compression of olivine compared with tensile tests of magnesium alloy. Repeated observation of the same region in olivine at successive stages of deformation cannot be accomplished because of the high pressure required for deformation and the very short time scale of fault generation and slip. It also is difficult to produce the required starting microstructure in magnesium alloy in compression. The common structural characteristics observed in Mg_2GeO_4, $(Mg,Fe)_2SiO_4$ and magnesium alloy, i.e. localized shear in a fine-grained layer; equiaxed shape of fine grains implying grain-boundary sliding as the main mechanism; shear of grains cut by the shearing surface; intragranular slip deformation of coarse grains, suggest that the mechanism of shear of non-SP regions along the SP layer in magnesium alloy during superplasticity and in olivine during fault propagation is similar. This is not a surprising result because metals and ceramics exhibit similar characteristics for superplasticity [4, 5].

One conceptual difficulty in visualizing GBS as the flow mechanism involved in the generation of deep earthquakes has been the intuitive expectation that GBS requires accommodation by diffusion and that diffusive processes may be too slow to allow the sudden accelerations that radiate seismic energy. The experimental results on Mg_2GeO_4 and $(Mg,Fe)_2SiO_4$ make clear that the anticrack faulting instability can operate at high pressure and does radiate energy, but understanding of the details of the process are still lacking. The magnesium alloy experiments show that GBS can occur in a cooperative fashion that probably does not involve complicated neighbour-switching events and therefore probably requires diffusion only

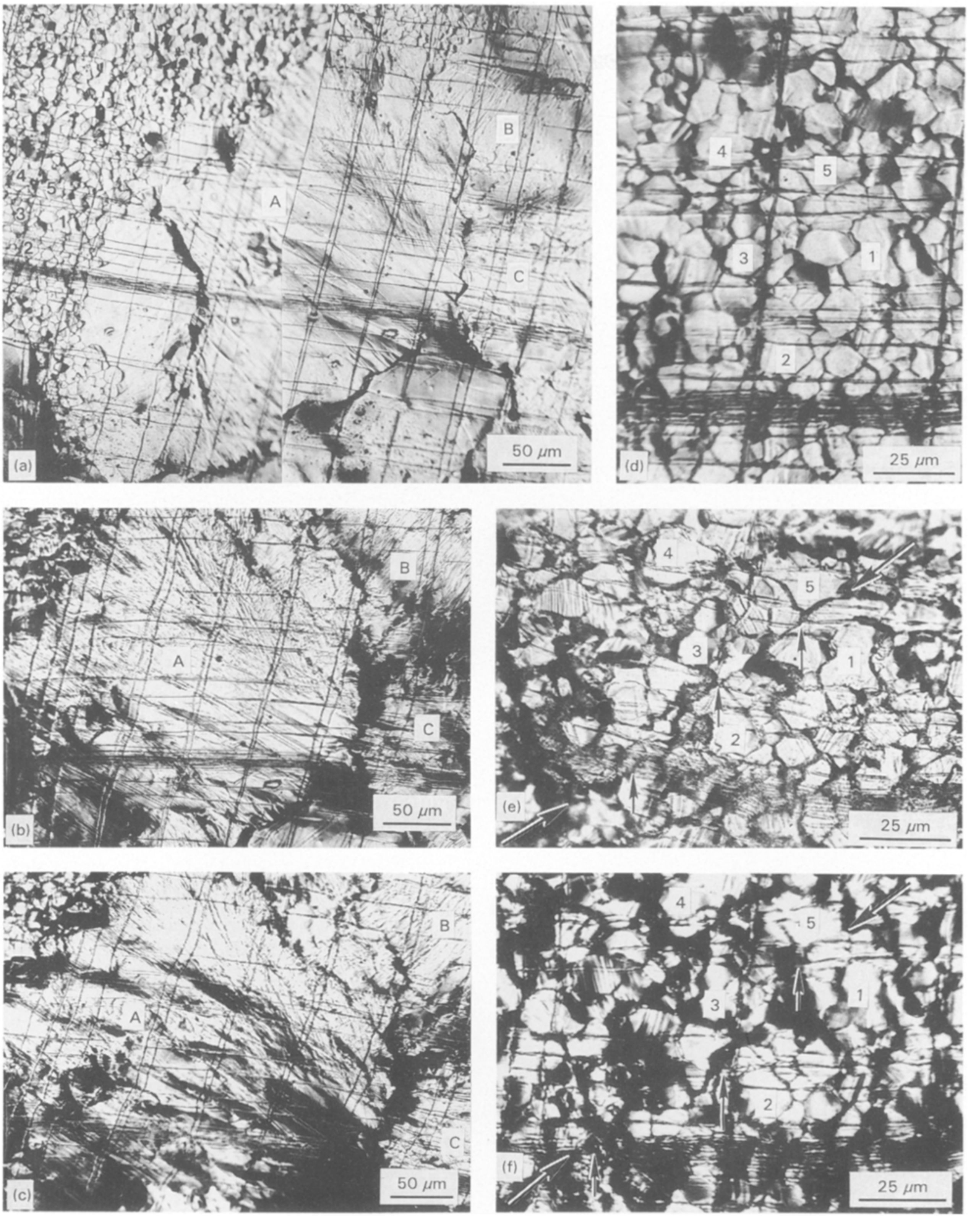


Figure 4 Deformation relief in Mg–1.5%Mn–0.3%Ce alloy on the prepolished surface of (a–c) the coarse-grain region and (d–f) the fine grain region. The same regions of coarse and fine grains are shown after bulk deformation (a, d) 10%, (b, e) 20% and (c, f) 30%.

on a smaller length scale than in GBS of individual grains. It is appealing to envision such cooperative GBS as active during high-pressure faulting.

5. Conclusions

1. The similarities between the flow in fault zones of Mg_2GeO_4 olivine and in Mg–1.5% Mn–0.3% Ce alloy with a superplastic zone in shear orientation suggest that the flow mechanisms in the two situations may be the same.

2. The magnesium alloy deformed by shear along the superplastic zone, with flow occurring by cooperative grain-boundary sliding in a layer-by-layer manner.

3. Flow in olivine fault zones in the earth by such cooperative grain-boundary sliding would be an efficient way for the system to minimize the diffusive

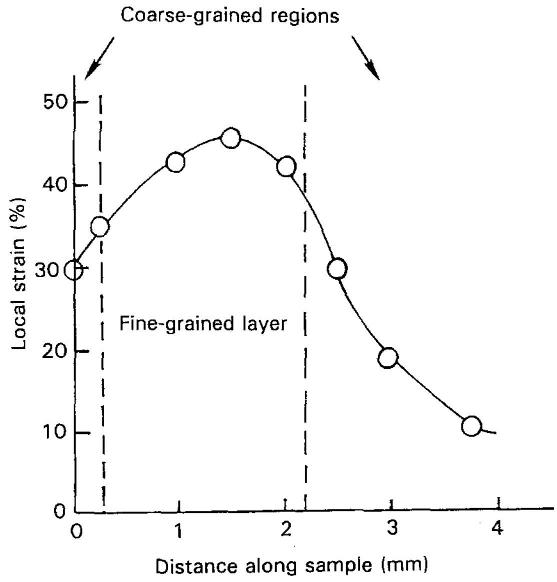


Figure 5 Local strain versus distance along tensile axis of Mg–1.5% Mn–0.3% Ce alloy sample with superplastic layer.

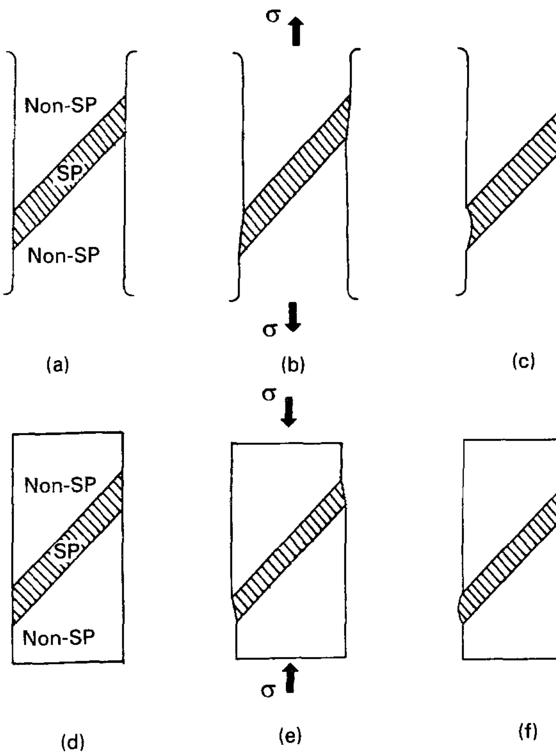


Figure 6 Schematic illustration of possible ways of deformation in material with superplastic layer in (a–c) tension and (d–f) compression through shear of regions with non-SP structure along the superplastic layer (b, e) or (c) elongation and (f) compression of the superplastic layer.

accommodation step and therefore may be important in deep-focus earthquakes.

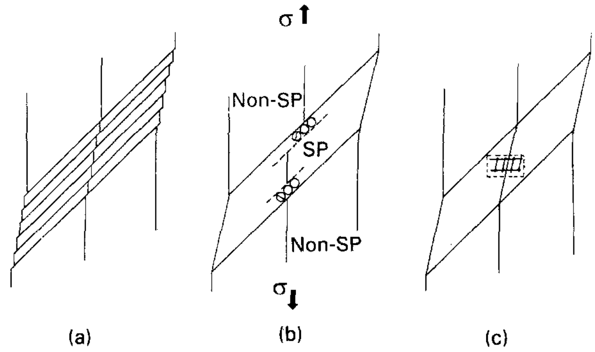


Figure 7 Schematic illustration of possible micromechanisms of shear of regions with non-SP structure along the SP layer: (a) cooperative grain-boundary sliding in layer-by-layer manner; (b) rotation of fine grains along grain-boundary surface of coarse grains; (c) shear strain of fine grains.

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References
1. C. H. HAMILTON and N. E. PATON, "Superplasticity and Superplastic Forming" (TMS-AIME, Warrendale, PA, 1988).
2. M. KOBAYASHI and F. WAKAI, "Superplasticity", Vol. 7 (Materials Research Society Symposium Proceeding, MRS, Pittsburgh, PA, 1988).
3. S. HORI, M. TOKIZANE and N. FURUSHIRO (eds), "Superplasticity in Advanced Materials", (Japan Society for Researches on Superplasticity, Osaka, 1991).
4. A. K. MUKHERJEE, in "Plastic Deformation and Fracture of Materials", edited by H. Mughrabi, (VCH Verlagsgesellschaft, Germany, 1993), Ch. 9, p. 407.
5. M. S. PATERSON, in "Superplasticity in Metals, Ceramics, and Intermetallics", Vol. 196, edited by M. J. Mayo, M. Kobayashi and J. Wasdsworth (Materials Research Society Symposium Proceeding, MRS, Pittsburg, 1990) p. 303.
6. E. ITO and SATO, *Nature* **351** (1991) 733.
7. S. KARATO and P. LI, *Science* **255** (1992) 1238.
8. H. W. GREEN and P. C. BURNLEY, *Nature* **341** (1989) 733.
9. H. W. GREEN and P. C. BURNLEY, in "Deformation Mechanisms, Rheology and Tectonics", edited by R. J. Knipe and E. H. Rutter, Geological Society of London Special Publication **54** (Geological Society, London, 1990) p. 133.
10. P. C. BURNLEY, H. W. GREEN and D. PRIOR, *J. Geophys. Res.* **96** (1991) 425.
11. H. W. GREEN, C. H. SCHOLZ, T. N. TINGLE, T. E. YOUNG and T. A. KOCZYNSKI, *Geophys. Res. Lett.* **19** (1992) 789.
12. T. N. TINGLE, H. W. GREEN, C. H. SCHOLZ, and T. A. KOCZYNSKI, *J. Structural Geol.* (1992) in press.
13. H. W. GREEN, T. E. YOUNG, D. WALKER and C. H. SCHOLZ, *Nature* **348** (1990) 720.
14. M. G. ZELIN and M. V. ALEXSANDROVA, in "Superplasticity in Advanced Materials," edited by S. Hori, M. Tokizane and N. Furushiro (Japan Society for Researches on Superplasticity, Osaka, 1991) p. 63.
15. P. H. PARTRIDGE, D. S. McDARMAID and A. W. BOWEN, *Acta Metall.* **33** (1985) 571.
16. R. Z. VALIEV, O. A. KAIBYSHEV, V. I. SERGEEV and M. G. ZELIN, *Res. Mech.* **12** (1984) 219.
17. V. I. VLADIMIROV, "Physical Nature of Metals Failure", (Metallurgy, Moscow, 1984).
18. V. V. ASTANIN, O. A. KAIBYSHEV and S. N. FAIZOVA, *Scripta Metall, Mater.* **25** (1991) 2663.
19. H. S. YAUNG, M. G. ZELIN, R. Z. VALIEV and A. K. MUKHERJEE *ibid.* **26** (1992) 1707.

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