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T.E.M. investigations of room temperature microplasticity in MgAl_2O_4 spinel

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Résumé. — On a caractérisé par microscopie électronique les dislocations produites par indentation Vickers à température ambiante sur un monocristal de spinelle $\text{MgO} \cdot 1,1 \text{ Al}_2\text{O}_3$. Ces dislocations appartiennent aux systèmes $1/2 \langle 110 \rangle \{ 111 \}$. On ne détecte aucune trace de glissement dans $\{ 100 \}$ ou $\{ 110 \}$. En outre ces dislocations ne présentent ni orientation préférentielle, ni dissociation observable. Cependant après recuit de 1 jour à $1\,000^\circ\text{C}$, on observe des dissociations de l'ordre de $100 \text{ \AA}$.

Abstract. — Dislocations produced by room temperature Vickers indentation on a single crystal of $\text{MgO} \cdot 1,1 \text{ Al}_2\text{O}_3$ spinel have been characterized by T.E.M. These dislocations belong to $1/2 \langle 110 \rangle \{ 111 \}$ glide systems. No trace of $\{ 100 \}$ nor $\{ 110 \}$ glide is found. Moreover these dislocations show no preferred orientation nor visible dissociation. However after annealing for 1 day at $1\,000^\circ\text{C}$, dissociations of the order of $100 \text{ \AA}$ are observed.

Introduction. — Hardness tests have been extensively used to investigate properties of hard and brittle materials when uniaxial compression experiments are not possible [1]. The knoop indenter with an elongated diamond is particularly interesting for single crystals because hardness anisotropy for various crystallographic orientations gives further information on the activated glide systems [2].

Hardness tests have been successfully used on oxides with the spinel structure such as Fe_3O_4 [3], Ni ferrites [4, 5], Mn-Zn ferrites [6]. Glide line orientations around the indentations as well as knoop hardness anisotropy indicate that glide mostly occurs at room temperature on $\{ 111 \}$ glide planes. $\{ 100 \}$ and $\{ 110 \}$ are occasionally observed. Recently T.E.M. investigations around indentations have directly confirmed $\{ 111 \}$ slip planes in Ni ferrites [5].

For $\text{MgO} \cdot n\text{Al}_2\text{O}_3$ spinels, the influence of temperature and composition (n) on Vickers hardness have been reported by Westbrook [7]. Vickers [8] and knoop tests [9] have also been performed in studies of the precipitation sequence occurring during various time or temperature annealings.

We have performed knoop hardness tests on nearly stoichiometric spinel $n = 1.1$ with a view to determine the active glide systems of this material at room temperature. We find a mean knoop hard-

ness $(^1) H_K$ of the order of $1\,300 \text{ daN} \cdot \text{mm}^{-2}$ which is a rather high value (for comparison, at room temperature for Cu_2O $H_K \simeq 150$ [10], for MgO $H_K \simeq 600$ [2]) leading to small indentation lengths $\leq 60 \mu\text{m}$ and imprecise anisotropy curves (mainly because of numerous small microcracks occurring either on the sides or at the tip of the indentations, see fig. 1a). Around some indentations without cracks one could observe small glide lines, whose direction was consistent with both $\{ 111 \}$ and $\{ 110 \}$ glides (Fig. 1b) due to the choice of a (001) section which gives easily distinguishable profiles in the hardness *versus* orientation curves for these two known glide systems [11].

Direct T.E.M. investigations of the activated glide systems have thus been performed. We essentially find $1/2 \langle 110 \rangle \{ 111 \}$ glide. Moreover the dislocations produced by room temperature indentation do not appear dissociated in contrast with the case of high temperature deformation [11, 12, 13]. After

⁽¹⁾ The knoop hardness is deduced from the indentation length L produced by the applied force F owing to the semi empirical formula

$$H_K = 14.23 \frac{F}{L^2} \text{ daN} \cdot \text{mm}^{-2}.$$

It grossly corresponds to the resolved stress averaged on the various possible glide systems.

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Fig. 1. — *a*) Knoop indentation exhibiting microcracks which prevent precise evaluation of its length. *b*) Glide lines along the bottom edge on an indentation. They are consistent with $\{111\}$ or $\{110\}$ glide (Nomarski contrast).

annealing (1 day at 1 000 °C) the dislocations do not significantly move but they become clearly dissociated.

T.E.M. investigations. — The Vickers indenter, which is more symmetrical than the Knoop indenter, was chosen for this work. As large magnifications are available in T.E.M., very low forces were applied to prevent possible cracks (< 0.3 N against 3 N in previous Knoop tests). Thin samples were cut parallel to the (001) indented surface and ion thinned mainly on the opposite face. Figure 2 shows a typical indentation at low magnification. The cross corresponds to the traces in the foil of the four $\{111\}$ planes which are the principal activated glide systems. Dislocations have moved over a small distance of a few microns and the dislocation density is locally very large

Fig. 2. — Typical Vickers indentation ; (001) foil, the cross corresponds to the traces of the four $\{111\}$ activated glide planes.

Fig. 3. — Sharp $\{111\}$ glide bands. Dark field.

(10^{13} to 10^{14} m^{-2} depending on the chosen region). At higher magnification (Fig. 3) standard stereographic determinations of the dislocation line orientations and standard determinations of their Burgers vector clearly show that the dislocations which form the cross are confined in some very sharp and discrete glide bands parallel to $\{111\}$ planes. There is no evidence for any preferred orientation and configurations like the one shown on figure 4 is the result of the intercept of circular loops produced by a Frank-Read source in a $\{111\}$ glide plane by the thin foil parallel to (001).

Between the arms of the cross, one finds other dislocations which are better seen on figure 5. They seem to lie in the foil plane (001) but their Burgers vectors are at 45° to this plane. We believe that we are concerned here with prismatic loops. More precisely a detailed stereographic analysis performed on numerous parts of these loops shows that these segments belong to the $\{111\}$ planes in zone with their Burgers vector. Therefore, these loops might be created at the indent and they would glide on the prism surfaces formed by the $\{111\}$ faces in zone with their Burgers vector. They would have slipped a distance comparable to that travelled by the dislocations on the $\{111\}$ arms of the cross shown in figure 2. This observation is consistent with the fact that the easiest glide system at room temperature occurs in $\{111\}$ planes.

All these dislocations do not appear to be dissociated, even in usual weak beam conditions [14]. If dissociated, their width is smaller than 50 Å while

Fig. 4. — Intersection of a Frank-Read source in a $\{111\}$ glide plane with the thin foil parallel to (001).

Fig. 5. — Half prismatic loops systematically present between the arms of the cross (cf. Fig. 2).

dislocations produced by high temperature deformation exhibit dissociation widths as large as 100 to 200 Å depending on the stoichiometric ratio n [9, 11, 13].

We have annealed an indented crystal for one day at 1 000 °C. Thin foil examinations show that dislocations around indentations are now clearly dissociated ⁽²⁾ (see Fig. 6a and b showing how both cases compare at the same magnification). This observation raises the question of equilibrium configurations in the room temperature pictures. Recently Veys-

Fig. 6. — Weak beam images of dislocations produced by indentation. *a*) After annealing (1 day at 1 000 °C). They are clearly dissociated, their dissociation width as projected on the micrograph plane is about 70 Å. *b*) Before annealing, there is no visible dissociation ; both micrographs are taken at the same magnification with the same diffraction conditions (same g , same s for same Burgers vector).

sière [5] has reported T.E.M. investigations on indented Ni ferrites at room temperature (they have an inverse spinel structure). From several well characterized features (glide in $\{111\}$ planes but with well marked preferred orientations along the $\langle 110 \rangle$ directions leading to 60° characters, numerous screw dipoles with a few hundreds Angstroms wide), he concludes that these dislocations have to be slightly dissociated (less than 50 Å) off their $\{111\}$ glide plane, and by comparison with calculated stacking fault energies he suggests that a glissile dissociation in $\{100\}$ into two collinear partials could occur. In our case no preferred orientation nor screw

⁽²⁾ The two peaks profile of dislocations in figure 6b cannot come from some special contrast effect. Indeed : i) figure 6a and 6b show the same dislocation type (same Burgers vector, same glide plane) ; they are taken under the same diffraction conditions (same g , same s) ; ii) it has been experimentally shown that, in the case of spinels, with these diffraction conditions, the two contrast peaks effectively correspond to two partials with equal and collinear Burgers vectors $1/4 \langle 110 \rangle = 1/2$ perfect Burgers vector ; indeed the apparent separation of both partials on the micrograph plane varies with the tilt conditions (for given g and s) and from these experiments the dissociation plane could be determined [11, 13].

dipoles are observed but the room temperature elastic limit of stoichiometric Mg-Al spinel is larger than that of Ni ferrite (approximately three times from comparison of indentations for the same load) and the resulting isotropic lattice friction could prevent any appreciable glissile dissociation in any plane, thus strongly decreasing the Peierls frictions observed in the case of Ni ferrite. On the other hand, during the annealing process at 1 000 °C, this isotropic lattice friction is much lower and dislocations can reach an equilibrium configuration characterized by a larger dissociation (either a glissile configuration by dissociation in their { 111 } glide plane or sessile dissociations as those observed in samples deformed at high temperature).

The dislocations studied in weak beam do not significantly move during the annealing process but it is to be noted that for the sake of commodity we have chosen regions with a low dislocation density (i.e. far from the indentation) for our weak beam observations. In these regions the internal stress is low and has no appreciable effect, dislocations do not significantly move and thus no preferred orientation is observed after annealing.

In conclusion, a limited amount of plastic deformation can be produced at room temperature. It mostly occurs by glide on { 111 } planes when the load is weak. There is no preferred orientation nor visible dissociation for these glide dislocations.

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