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Cathodoluminescence imaging of microindented {111} CdTe

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Résumé. — Des figures d'indentation développées à température ambiante sur les deux faces polaires {111} de CdTe ont été observées par cathodoluminescence dans un microscope électronique à balayage. La bonne résolution de cette technique apporte des renseignements nouveaux sur la configuration des dislocations développées autour des indentations.

Abstract. — Microhardness experiments have been carried out at room temperature on both polar {111} faces of n types CdTe specimens. The indentation figures is imaged by cathodoluminescence in a Scanning Electron Microscope (SEM / CL). The better spatial resolution of CL images, compared to that obtained by the etch-pit method, allows us to draw some new conclusions about the dislocation patterns around indentations on CdTe.

1. Introduction.

A few Vickers microhardness experiments have been undertaken on {001} and {111} oriented tetrahedrally coordinated polar semiconductors, like GaAs [1-4], GaAlAs [5] InSb [6, 7], CdTe [8, 9] and CdHgTe [9], to study the deformation mechanisms around microindentations, or to discriminate the fastest dislocation between the A(g) and B(g) type. The investigations were made either by etching [1, 3, 8, 9], or by transmission electron microscopy [2, 4, 5], or by X-ray transmission topography [6, 7].

From a geometrical analysis of the asymmetry of the rosette developed on (001) faces of n type GaAs crystals, a higher mobility has been attributed by Warren *et al.* [1] to the As(g) (B(g) type) dislocation, in agreement with previous results issued from macroscopic deformation experiments [10, 11].

Less numerous results are available in the literature for CdTe. Etch-pits on the {111} Te face of a p type specimen revealed that the rosette developed after room temperature microindentation exhibited three-fold symmetry [8]. Each of the six-double arms which form the rosette is along

a $\langle 110 \rangle$ direction, and consists of a long and short arm. It has also been shown that each double arm is formed by elongated loops gliding in the opposite $\{111\}$ planes and which are issued from the impact point; the loops are of screw and 60° character, with a Burgers vector parallel to the arm direction. The application of an external stress to the indented and etched sample, allowed Braun *et al.* [8] to conclude that $\text{Te}(g)$ and $\text{Cd}(g)$ dislocations were in the long and the short arm respectively. The higher velocity of the $\text{Te}(g)$ dislocation was in agreement with previous results obtained on CdTe samples [12, 13], but not with the conclusions drawn from the model of Hirsch *et al.* [3]. Nonetheless, this result has been confirmed by Barbot *et al.* [9] who studied the hardness as a function of face polarity.

In this paper, we show that a cathodoluminescence experiment in the Scanning Electron Microscope (SEM / CL) gives, for CdTe , more detailed information than does the etch-pit method. Therefore, it appears more appropriate to study the slip geometry of dislocations around microindentations. This is due to the higher spatial resolution of the SEM / CL technique. Conclusion about the nature of dislocations within the rosette arms and about slip mechanisms are drawn from observations of the in-depth extent of the microindentations made on Te and Cd faces.

2. Experimental.

Room temperature Vickers experiments were performed on monocrystalline bulk n-type $\{111\}$ CdTe specimens grown by the Travelling Heater Method [14] and annealed under saturated cadmium pressure for six hours at 700°C to achieve n type ($n \cong 10^{15} \text{ cm}^{-3}$). The specimens were mechanically polished with diamond paste. Then, the resulting damage was chemically removed by using a bromide-methanol solution. The polarity of the $\{111\}$ CdTe faces was determined by immersing the polished specimen in a lactic acid: nitric acid (70%): fluoridric acid (50%) (25:4:1) solution [16] for one minute: after rinsing in flowing water, the Te (111) B face remained dark, while the Cd (111) A face took a metallic appearance.

The diagonals of the indenter were along the $\langle 110 \rangle$ and $\langle 112 \rangle$ directions. The applied load varied in the range 5 g to 150 g, with a dwell time of 30 s.

Rosette patterns around the room temperature indentations could be observed straightaway, as CdTe is a plastic material at that temperature.

The room temperature CL was detected, by an ellipsoidal mirror mounted on a Jeol 840 SEM. The band gap wavelength, equal to $0.86 \mu\text{m}$ at 300 K ($E_{\text{gap}} = 1.44 \text{ eV}$) was detected by a S20 R photocathode. The depth expansion and the evolution of slip planes and dislocations arrays were analysed from both SEM / CL and compared with etch-pit pictures; the latter were obtained by immersing the specimen in a 10 ml HNO_3 (70%) – 20 ml H_2O – 4 g K_2CrO_7 solution [17]. We found, from the shift of a twin during etching, that the etch rate was of about $8 \mu\text{m}/\text{mn}$ in our experiments.

3. Rosettes and slip triangles around microindentations imaged by SEM / CL.

We have observed that, whatever is the applied load, a SEM/CL indentation figure on both $\{111\}$ CdTe faces is made of the four following features:

- a rosette which exhibits three-fold symmetry of the $\{111\}$ surface. Each double arm of the rosette, nearly parallel to a $\langle 110 \rangle$ direction, is made of a short and a long arm (SA and LA in Figs. 1a, 2a, 3a). $\langle 110 \rangle$ directions are traces of $\{111\}$ slip planes on the indented surface.

Fig. 1. — SEM / CL images of a room temperature microindentation on a Te (111) of a n type CdTe crystal. The applied load is 25 g. Figures b, c and d are taken at different depths d from the surface. a) Indented surface. The three-fold symmetry of the rosette is clearly visible. The arms of the rosette and the edges of the triangles are parallel to the three $\langle 110 \rangle$ directions. The extent of the long arms (LA) of the rosette is close to $400 \mu\text{m}$, and that of the short arms (SA) of about $220 \mu\text{m}$. Only the second and the third “slip” triangles are visible. The short arms do not all spread out the third triangle (see Fig. 3a). b) Depth $d = 60 \mu\text{m}$. No rosette is visible anymore; the third triangle has nearly disappeared. c) An inner triangle appears clearly at a depth of $90 \mu\text{m}$. It probably represents the apex of the internal tetrahedron (see text). d) Depth $d = 100 \mu\text{m}$. Same remarks as in figure 1c.

Dark spots are visible in each arm; they correspond to the emergences on the surface of dislocations which act as non-radiative recombination centers and therefore quench locally the luminescence. No dislocation segments could be detected within and between the arms.

– an inner “slip” triangle formed by the intersection of the short rosette arms (Fig. 3a). This triangle is, on the CL image of the indented surface, hidden by

– a second and larger “slip” triangle which corresponds to the long arms of the rosette (Figs. 1, 2 and 3). It roughly circumscribes the square trace of the indenter (Figs 3a, 3b),

Fig. 2. — SEM / CL images of a room temperature microindentation on a Cd (111) of a n type CdTe crystal. The applied load is 25 g. Figures b, c are taken at different depths d from the surface. a) Indented surface. The figure is identical to figure 1a, but the extent of the long arms (LA) of the rosette is close to $300\ \mu\text{m}$, and that of the short arms (SA) of about $200\ \mu\text{m}$. b) $d = 30\ \mu\text{m}$. No rosette is visible anymore; the third triangle has nearly disappeared. c) $d = 70\ \mu\text{m}$. An inner triangle appears clearly.

– a third “slip” triangle within which individual slip lines, parallel to three $\langle 110 \rangle$ directions, can be resolved. In fact, it is made of many triangles lying inside each other. When the applied load is larger than 25 g, the short arms of the rosette rarely spread outside this triangle (Fig. 4b). This could explain why they could not be always detected by the etch-pits method [9].

At the surface, the CL intensity within the inner and the second triangles is very low and uniform. This indicates that the dislocation density is higher within them than in the rosette arms and within the third “slip” triangle for which either dislocations or slip lines can be individually resolved.

Fig. 3. — a) Schematic drawing of an indentation figure, as imaged by SEM / CL, on both $\{111\}$ faces of n type CdTe specimen. Indenter trace is represented. Bended arrows suggest cross-slip of screw dislocations b) SEM / CL image of a room temperature 150 g indent figure on a Te face. The impact surface is clearly visible.

The dislocation density decreases rapidly outside the second triangle, as imaged in figure 3b which also shows that the impact surface is contained inside the second triangle.

The SEM / CL images show that the in depth extent is smaller for the dislocations which have moved further from the impact point. This is illustrated in figures 1 and 2 where it is seen that the rosette disappears first when the depth increases (Figs. 1b and 2b), followed by the third “slip” triangle (Figs 1c and 2c); slip lines in the second “slip” triangle can be more and more easily detected whereas the CL intensity in an inner “slip” triangle underneath the impact point is still uniform (Figs 1c, 1d, 2c). At these depths and around this latter triangle many triangles appear inside each other.

Let us notice that these observations could not have been made with the etch-pit method, as shown in figure 5. They indicate that the deformation induced by the microindentation is mainly located underneath the impact point.

SEM / CL images of the indentation at different depths below the surface are qualitatively identical for both indented surfaces. The indent figure disappears at a higher depth for the Te face than for the Cd one (Figs. 1d and 2c). Furthermore, we could evidence cross-slip events (arrows in Figs. 1a and 4b). Finally, we found that the indentation surface area is nearly proportional to the applied load, indicating that, whatever is the applied load, dislocation equilibrium is reached at the end of the experiment.

Fig. 4. — SEM/CL images of room temperature microindentations on a $\{111\}$ face of an n type CdTe crystal. a) Cd face. The applied load is 10 g. The short arms of the rosette are visible. b) Te face. The applied load is 80 g. The short arms of the rosette do not spread out the third "slip" triangle. Cross-slip events are clearly visible.

Fig. 5. — Indents figures below a $\{111\}$ face. The depth under the surface is $150\ \mu\text{m}$. a) Te face, applied load of 120 g. SEM / CL image. b) Same indent as in a. Etch-pit figure which shows only emerging dislocations, whereas the CL image can evidence dislocations parallel to the surface. c) Cd face, applied load of 42 g. SEM / CL image. d) Etch-pit figure of the same indent as in c.

Fig. 5. — (*continued*)

4. Discussion.

The cathodoluminescence mode of the SEM has been used to an in depth visualisation of the disposition of dislocations underneath and around an micro-hardness. We have shown that the

resolution of this technique is higher than that obtained by the etch-pit method. This should allow us to derive more information on the way the deformation proceeds.

First of all it has to be noticed that the indent figures observed on Cd and Te faces are somewhat different from previous observations [3, 8, 9]. It has to be pointed out that, contrary to GaAs [3], we found that the indent figure was independent of the nature of the indented face.

The greater in depth extension of the indent figure below the Te surface related to the hardness anisotropy of polar faces (the Cd face seems a little harder than the Te face), could therefore be explained by a higher mobility of Te (g) dislocations, in agreement with previous results [9, 12, 13].

The first state of deformation, which occurs mainly below the impact surface, can be deduced from the indent figure at large depths (Figs. 1d-2c) where diamonds are observed at the edges of the triangle. These diamonds could result from the emission of prismatic loops, each of them gliding in two {111} planes with a $\langle 110 \rangle$ common direction and propagating along this direction (Fig. 6). The former loops emitted are pushed by the last ones in an efficient way as long as the distance between them is smaller than their diameter. Following figure 7a, two sides of the loops are of Cd(g) type and the other ones of Te(g) type. In the case of a Cd indented surface for instance, geometrical considerations show that slow Cd(g) dislocations are more efficiently emitted and glide more easily on the external apex tetrahedron than Te(g) dislocations. This easier glide is compensated by the higher mobility of Te(g) dislocations and all the parts of the loops move at about the same speed. This is not the case for a Te indented surface where the glide is favoured for fast Te(g) dislocations. This description is coherent with the indent figures at large depths where the diamonds are more easily observed for a Cd indented face (Figs. 2c, 5a, 1d).

The rosette arms are partly made of half loops such as those in figure 7b. Within our description, the "inner" triangle results from the intersection of short rosette arms (SA in Fig. 3a), but is not involved in the deformation process. The deformation starts on the external apex tetrahedron which corresponds to the second "slip" triangle as suggested by figures 3 b and 8. The short rosette arms correspond to dislocations which are not easily activated, ie slow Cd(g) dislocations for a Te indented surface and fast Te(g) dislocations for a Cd indented surface. It can therefore be suggested, contrary to [8], that the rosette arms lengths are not directly related to the anisotropy of dislocation mobilities. The difference in the rosette arms can be understood following a geometric description of the deformation process. It seems that, when the mobility ratio of A(g) and B(g) dislocations is high enough, as in GaAs for instance where the mobility of B(g) dislocations is 100 to 200 times greater than that of A(g) ones, this would lead to the appearance of single rosette arms [15], instead of double arms as in the case of CdTe. Thus, it can be concluded that the asymmetry of the dislocations mobilities is less important for CdTe than for GaAs, as suggested in [9].

As the indenter enters the specimen, more and more glide planes are activated; the size of the external apex tetrahedron, made of three {111} glide planes, increases in all three $\langle 110 \rangle$ directions as deformation goes along. This could explain the low dependence of the inner triangle with depth as suggested by the dislocations position in figure 7a. The better resolution of the SEM/CL indent figure at large depths (Figs 1b, 1c) comes from the decrease of the dislocation density as depth increases (Fig. 7).

The steep decrease of the dislocation density outside the second triangle can be explained by annihilation of common Burgers vectors in neighbouring glide systems (Fig. 8a) along the edge of the tetrahedron, and by the existence of dislocation locks in the volume underneath the impact surface (Fig. 8b). The locks can be created, in the case of $(11\bar{1})$ and $(\bar{1}\bar{1}1)$ slip planes for instance, by the interaction of dislocations with Burgers vectors $b = 1/2 [\bar{1}\bar{1}0]$ and $b = 1/2 [101]$. They can induce dislocation cross slip which gives rise to the external triangle, as observed in figures 4 b and 9. Dislocation dissociation should not prevent such events since the dissociation width is quite small in CdTe at room temperature (5 nm for screw dislocations [18]). Such dislocations move in

Fig. 6. — First prismatic loops emission at the beginning of the deformation.

Fig. 7. — a) Schematic first dislocations emission below the indented surface. The dislocations are emitted in the $(11\bar{1})$ plane at the immersion point of the indenter on both sides of the tip. The dislocations emission is favoured in side labelled (1) whatever is the dislocations mobility. The flow of matter from side (2) in symmetrical when dislocations are emitted in other $(11\bar{1})$ and $(\bar{1}11)$ planes. b) Plan-view of dislocations in $(11\bar{1})$ plane. Screw parts of dislocations can cross-slip.

an internal apex tetrahedron, the base of which corresponds to the external triangle observed on the surface and roughly circumscribed to the second triangle. This shows that there is indeed cross-slip of dislocations in the second triangle which is limited by the locks detected at large depths (Figs. 1d and 2c). Note that such a cross-slip induces a change in the dislocation character, ie a Te (g) dislocation becomes a Cd (g) one.

Another dislocation interaction is possible, between dislocations gliding on the $(11\bar{1})$ and $(\bar{1}11)$ slip planes, following the sequence for Burgers vectors:

$$\frac{1}{2} [0\bar{1}\bar{1}] + \frac{1}{2} [101] \rightarrow \frac{1}{2} [\bar{1}\bar{1}0]$$

The resulting Burgers vector is parallel to the (111) surface, and the dislocation, which glides in the $(11\bar{1})$ plane, can expand in the rosette arms parallel to the $[\bar{1}\bar{1}0]$ direction contained in the (111) surface plane. Dislocations in the other arms can be created in a similar way.

Fig. 8. — Schematic representation of few interactions of dislocations gliding in the $(\bar{1}\bar{1}1)$ and $(11\bar{1})$ slip planes of the external apex tetrahedron leading to locks. a) Result of annihilation of Burgers vectors common to two slip systems along the edge of the tetrahedron. b) Locks.

Fig. 9. — Schematic representation of dislocations cross slips from one tetrahedron to the other one. These deviations induce a change in the dislocation character.

5. Conclusions.

We have used the SEM / CL technique to visualise the in-depth extent of indentations made on both Cd and Te $\{111\}$ faces of n types CdTe crystals. We have shown that this technique is more powerful than the etch-pit method, due to its higher spatial resolution. The indent figures have been found to be independent of the nature of the indented face. The dislocations which move inside the crystal seem to glide, whatever is the indented polar face, within the external apex tetrahedron. The observed different hardness of the Cd and Te faces could be explained in terms of dislocations mobilities, in agreement with [3]. The detection, by cathodoluminescence, of cross-slip events from one slip triangle to another allowed us to conclude that the length of the rosette arms is not directly correlated with the dislocation type. The rosette arms contain dislocations with parallel (to the indented surface) and inclined Burgers vectors.

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