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Cathodoluminescence at frictional damage in MgO single crystals

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The frictional damage in single-crystal MgO was studied using the cathodoluminescent mode of a scanning microscope as well as selected-area electron channelling pattern analysis. With a hemispherical diamond slider, the distorted structured layer beneath the surface was noted, the cathodoluminescence of which was quenched, presumably due to the high density of defects. This non-luminescent layer was encased in the plastically deformed luminescent zone, which extended downwards to where there was a (0 0 1) cleavage crack. It was also found that with a larger ball slider, luminescent slip lines on both $\{1\,1\,0\}_{90}$ and $\{1\,1\,0\}_{45}$ were pronounced inside the track after a single to-and-fro traversal, and successive traversals were able to generate a non-luminescent, distorted structured layer inside the track.

1. Introduction

There have been recent cathodoluminescent investigations of contact damage in single-crystal MgO [1–4]. Because the strained region in this material is highly luminescent, cathodoluminescence is a useful technique for observing damage due to static, sliding, and impacting contacts. Velednitskaya and co-workers [1, 2] found that cathodoluminescence occurs around an indentation but not at the centre of the indentation. They suggested that luminescence radiation was caused by interstitial point defects built up around the indentation, while the behaviour of the luminescence around the centre of the indentation was explained by the absence of such defects. The different mechanisms responsible for this phenomenon were suggested by Pennycook and Brown [3]. They argued that around the indentation the dilatation due to dislocation causes a substantial decrease in the band gap. This results in strong recombination of electrons and holes, while at the centre of the indentation the density of defects is very high, causing the luminescence to be quenched. Chaudhri *et al.* [4] recently found that the intensity of luminescence from screw dislocations around the indentation was markedly higher than that from edge dislocations. They pointed out that this behaviour is not clear in the band gap model.

In this paper, we describe cathodoluminescence studies on deformation and fracture due to sliding on MgO single crystals. The degree of crystallinity of the deformed zone is also studied by selected-area electron channelling pattern analysis.

2. Experimental details

An MgO single crystal of 99.9% purity was purchased from Fruuchi Chemicals Co and reduced to specimens of a suitable size by normal cleavage techniques. The frictional tester used was a simple reciprocating sliding-type apparatus in which an 80-μm radius diamond stylus or 2-mm diameter tungsten carbide ball was slid over the (0 0 1) cleaved surface. The tests were made in air at a sliding speed of 1 mm sec^{-1} and the load ranged from 0.3 to 2.9 N. After sliding, some specimens were cleaved through the frictional track to examine the subsurface deformation, and others were etched in a solution of $H_2O:H_2SO_4$:saturated NH_4Cl=1:1:5. Frictional damage was observed using a JEOL JL-35C electron microscope in cathodoluminescence (CL) mode as well as secondary electron (SE) mode at the accelerating voltage of 25 kV. Emitted light from the specimens was

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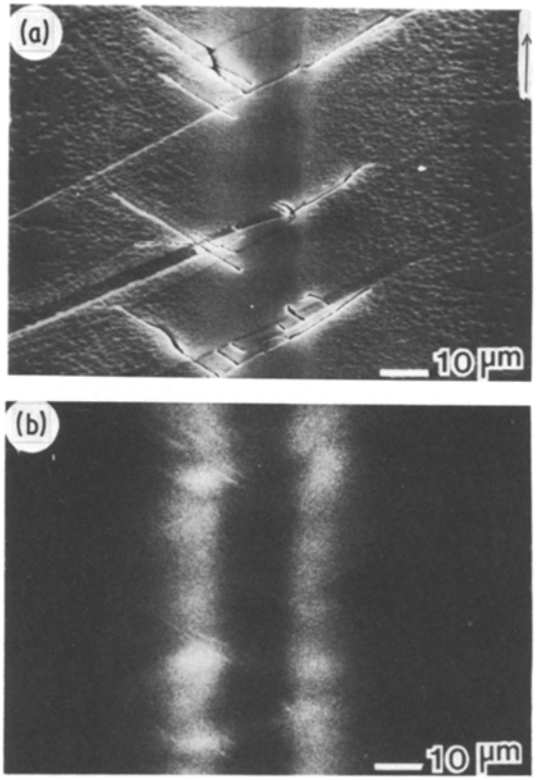

Figure 1 Secondary electron (a) and cathodoluminescence (b) images of damage on (0 0 1) surface of MgO crystals produced by a single traversal in the [1 0 0] direction of the diamond stylus at a load of 0.3 N. The crystal was etched. The arrow indicates the sliding direction.

detected by an S-11 photomultiplier (detectable wavelengths range from 300 to 650 nm; maximum response at 440 nm). In order to examine the degree of crystallinity of the deformed zone, selected-area electron channelling pattern analysis was conducted using an electron microscope: JEM-100C with a scanning attachment, which was used to observe ECPs in beam-rocking mode. Because MgO is non-conducting, a thin conducting layer was needed to examine the sample; we deposited a gold layer a few hundred nm thick on the sample in the SEM observation and an 80 nm layer in the ECPs.

3. Results and discussion

3.1. Surface damage

Fig. 1 shows the SE and corresponding CL images of the damage produced by a single sliding in the [1 0 0] direction on the (0 0 1) surface of MgO with a diamond stylus at a load of 0.3 N. The surface was etched after the sliding. It can be seen that luminescence occurs around the track, but not inside the track. This observation agrees with previous work on indentation by static loading and by scratches [1–4]. The coefficient of friction was about 0.14. For a load greater than 0.8 N, chevron crack formations were observed around the track (not shown). Fig. 2a shows the same track as Fig. 1, but at a higher magnification of a different position, showing the distribution of dislocation etch pits around the track. The density of dislocations was estimated by counting the etch-pits. Fig. 2b shows the result. In the luminescent zone around the track, the density was approximately 3 to 10×10^9 lines cm^{-2}, whereas inside the track there were too many pits to count.

When a large spherical ball, a 2 mm diameter tungsten carbide ball, was slid over the (0 0 1) face at a load of 8 N, luminescent slip lines on both $\{1\,1\,0\}_{90}$ and $\{1\,1\,0\}_{45}$ can be seen inside the track after a single to-and-fro traversal (cf. Fig. 3a). These two slip planes were almost equally pronounced. If the sliding is repeated several times,

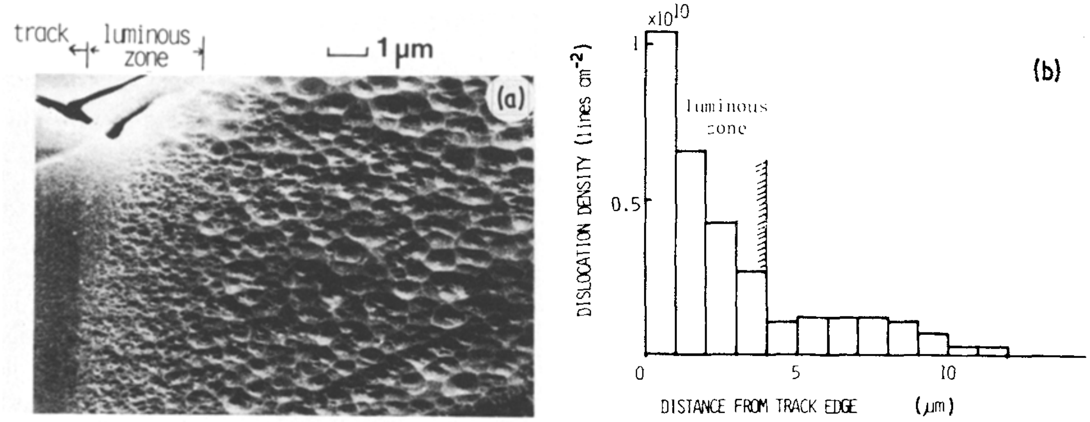

Figure 2 (a) Dislocation etch pits around the track, as in Fig. 1, but at a higher magnification of a diffferent position. (b) Distribution of dislocation densities around the track as estimated from etch-pit counting.

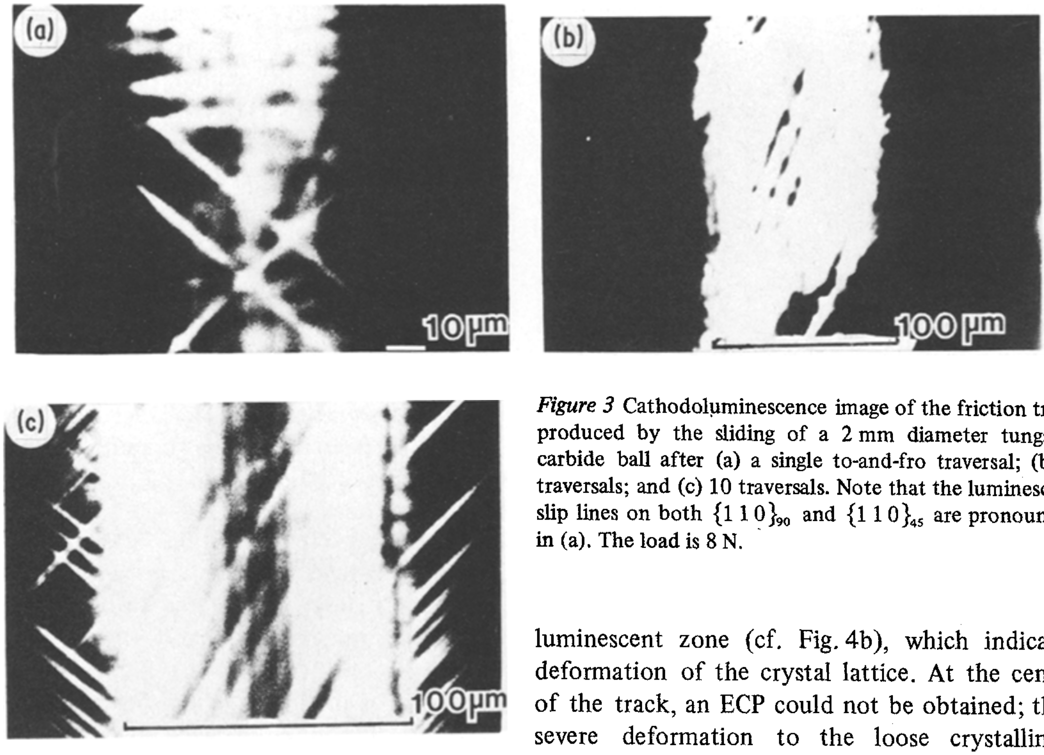

Figure 3 Cathodoluminescence image of the friction track produced by the sliding of a 2 mm diameter tungsten carbide ball after (a) a single to-and-fro traversal; (b) 6 traversals; and (c) 10 traversals. Note that the luminescent slip lines on both $\{1\,1\,0\}_{90}$ and $\{1\,1\,0\}_{45}$ are pronounced in (a). The load is 8 N.

the track becomes completely luminescent, which indicates that enough slip bands to cover the track (cf. Fig. 3b) were generated. After further traversals (cf. Fig. 3c), the luminescence around the centre of the track was quenched, and therefore this behaviour is similar to that in Fig. 1b. The slip lines on $\{1\,1\,0\}_{90}$ appeared to extend outward from the edge of the track.

The reason why there is no luminescence around the centre of the track can apparently be explained in two ways. The first explanation is that there are no dislocations [2], probably because of reorientation and/or recrystallization due to sliding, the behaviour of which in turn is similar to that of the non-luminescent non-deformed crystalline surface. The other one is that the very high density of defects causes the luminescence to be quenched [3]. To determine which of these explanations is more probable, we obtained selected-area electron channelling patterns from the (0 0 1) face at three different positions: (a) the non-deformed original surface, (b) the luminescent area around the track, and (c) the non-luminescent area around the centre of the track, as shown in Fig. 4a, b and c, respectively. Fig. 4d indicates the analysed positions in an SE image. The selected-area realizable was about 10 μm in diameter. The image quality deteriorated at the luminescent zone (cf. Fig. 4b), which indicates deformation of the crystal lattice. At the centre of the track, an ECP could not be obtained; thus severe deformation to the loose crystallinity occurred in that area, which may cause the quenching of the luminescence.

After the friction test a specimen was cleaved through the track into two halves, and one half was annealed at 800° C for 20 min. Fig. 5a shows the CL image of the frictional track at a load of 0.3 N with a diamond stylus before annealing. Fig. 5b shows that of the other half after annealing. It is clear that the area around the track became luminescent due to annealing as previous investigations have also observed [2, 3]. This luminescence is probably due to the reduction in the number of defects, caused by the annealing.

3.2. Subsurface damage

Fig. 6a shows a cross-sectional cleave through the track made with a diamond stylus at a load of 0.98 N. The CL image clearly shows that the non-luminescent zone inside the track has a clear boundary with a certain depth (cf. Fig. 6b). This zone is not, as the ECPs revealed, recrystallized, but very distorted due to frictional shear as well as hydrostatic pressure by normal loading. It was found that the depth (d) is about one-fourth the track width ($2a$); i.e. $d/a \approx 0.5$ for a load range between 0.29 and 2.9 N. Beneath this zone, the luminescent zone extends downwards to depth (c) where the (0 0 1) cleavage crack is parallel to the surface. In addition, the luminescent zone

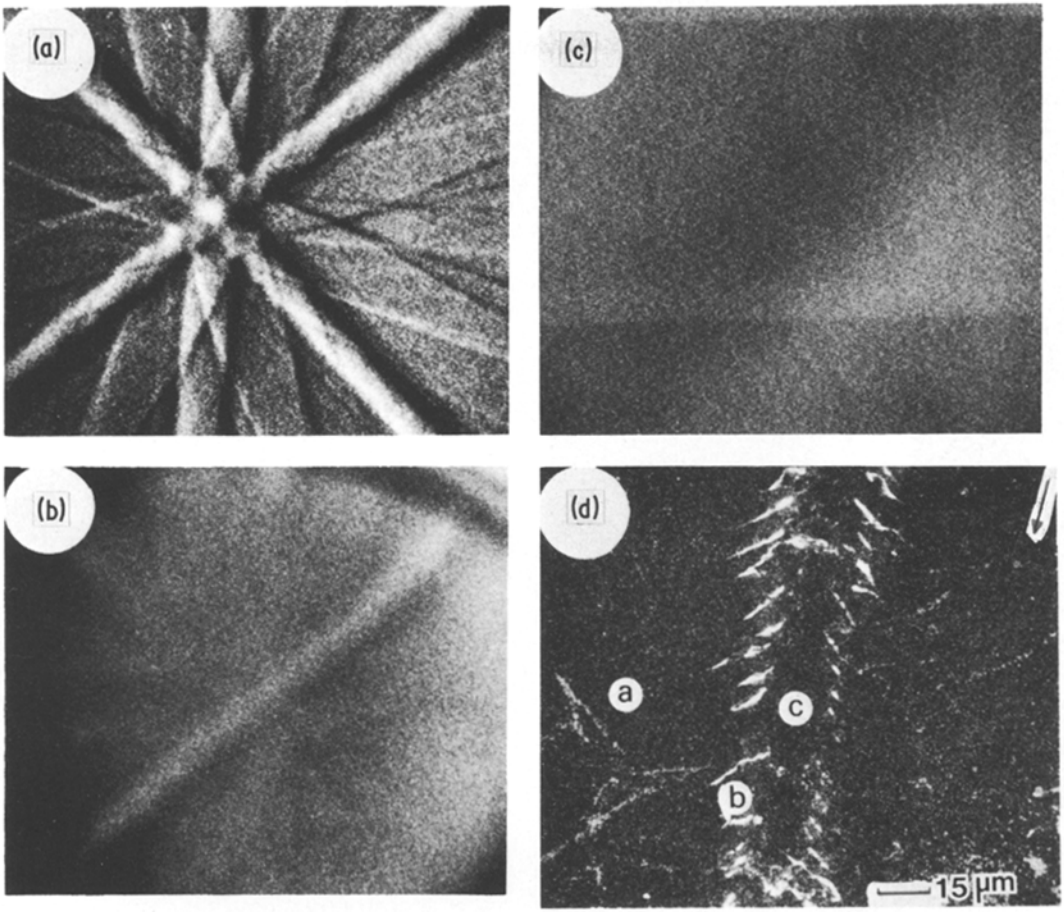

Figure 4 Selected-area electron channelling patterns obtained from (a) the non-deformed original surface; (b) the luminescent area around the track; and (c) the non-luminescent area around the centre of the track. (d) Secondary electron image of the track produced by sliding of a diamond stylus at 0.3 N load showing the position analysed in SA ECPs.

extends from the surface around the track edge downwards along the (1 1 0) slip planes which intersected the (0 0 1) face at 45°. The depth (c) is found to be $c/a \approx 2$. It is interesting to note that the value c/a agrees with the elastic–plastic boundary given by Hill's theory [5], although such agreement is somewhat fortuitous; i.e. $c/a = [E/3(1-\nu)Y]^{1/3} \approx 2.2$, assuming a Young's

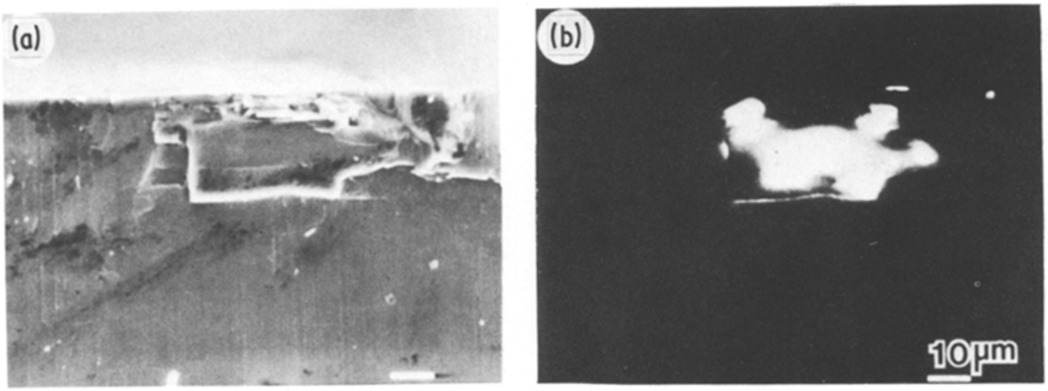

Figure 5 Cathodoluminescence image of the track produced by a single sliding of a diamond stylus at a load of 0.6 N: (a) before annealing, and (b) after annealing for 20 min at 800° C.

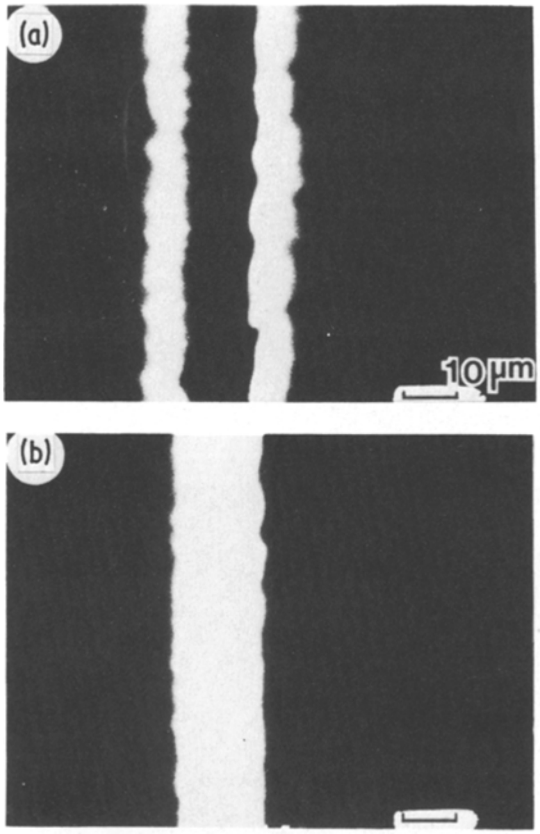

Figure 6 Secondary electron (a) and cathodoluminescence (b) images of the cross-sectional cleave through the track produced by the sliding of diamond stylus at a 0.98 N load.

modulus, E, of 210 GPa, a yield stress, Y, of 8.2 GPa, and a Poisson ratio of 0.25.

4. Conclusion

The present study has again demonstrated that observation of cathodoluminescence provides us with a versatile method for studying the particular characteristics of frictional damage, as proposed by Chaudhri *et al.* [4]. By observation with an SEM, the cathodoluminescence of frictional damage in MgO produced by a single traversal of a sliding stylus, we established that the layer inside the track is distorted with a high density of defects. The thickness of the layer is one-fourth the track width. This layer is encased in a luminescent zone which involves the dislocations of 10^9 to 10^{10} lines cm^{-2}. The depth of this zone is equal to the track width, which seems to agree with an elastic–plastic boundary.

These particular characteristics of subsurface damage can presumably be correlated to the frictional behaviour of the material. Work on this behaviour will be reported elsewhere.

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