Prolonged plastic deformation related to the microindentation of MgO single crystals

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The plastic deformation regions near the indentations on the (0 0 1) plane of MgO single crystals were investigated for an indentor under load and after unloading. It was established that the completion of slip-line structures arising during indentor penetration occurred during indentor removal. The presence of prolonged plastic deformation has been explained by considering the impulse character of the microindentation process of MgO at room temperature.

1. Introduction

It is known that change in the deformed region near the indentation occurs after indentor removal from the material [1–4]. This phenomenon was observed for both ionic [1–3] and semiconductor crystals [4] and was considered to be a reverse deformation or a recovery deformation [1, 2, 4], which was completely logical. However, an unexpected fact was revealed for MgO crystals [5] – the further development of the dislocation structure arising during indentor penetration took place after unloading. In this work, investigations were performed using the etch-pit technique. However, there is an opinion in the literature that this method is not very reliable, because change in the dislocation structure can occur under the action of etchant [6]. Therefore, the necessity arises to prove the presence of prolonged plastic deformation, but not the reverse, i.e. a deformation having the same sign during indentor penetration and whilst standing in the sample.

MgO crystals are a suitable object for such a study. The system of slip lines arises near the indentation in the case of deformation at room temperature, i.e. one can observe the deformed zone without the use of the etch-pit technique [6–12]. The crystals are transparent, which permits us, using the methods developed elsewhere [12, 13], to observe the deformed region related to the indentor under load and after unloading. Therefore, the purpose of the present work was to study the peculiarities of plastic deformation observed during the process of indentor removal using MgO crystals as an example.

2. Experimental procedure

The plates 1–1.5 mm thick were cleaved from the MgO single crystals by the $\{0\,0\,1\}$ cleavage planes. An installation comprising a metallography microscope and a loading device was used for the deformation [12, 13]. This installation permitted observation of the indentation and its surrounding deformed region, and the failure patterns made by the indentor under load and after unloading. The Vickers diamond pyramid was used as the indentor. The indentor loads, P, were varied within the limits 100–500 g. The experiments were performed for two indentor orientations: indentation diagonals, d, parallel to the directions $\langle 1\,0\,0\rangle$ and $\langle 1\,1\,0\rangle$. The surface relief near the indentations was studied using an interference microscope.

3. Results and discussion

A system of slip lines in the form of squares one inside the other (square sides parallel to the $\langle 1\,0\,0\rangle$ directions) arises around the indentations on the (0 0 1) face of MgO (Fig. 1). This distribution of slip lines was earlier observed in several papers [6–8, 10–12]. It was explained using a model of material plastic flow related to the indentation [6, 11, 12, 14]: the squares of the slip lines coincided with the emergence of slip planes, responsible for the upward movement of material, on the plane under investigation [6, 9, 11, 12]. The main slip system of MgO is $\{1\,1\,0\}$ $\langle 1\,1\,0\rangle$. The $\{1\,1\,0\}$ planes can be divided, taking into consideration their arrangement relative to the (0 0 1) plane into two types: $\{1\,1\,0\}_{90}$ which are perpendicular to the $\{0\,0\,1\}$ plane, and $\{1\,1\,0\}_{45}$ which form an angle of 45° with this plane.

Observation of the deformation patterns for the indentor under load (Fig. 1a) and after unloading (Fig. 1b) enabled us to establish that the slip-line squares arose during the loading process and their completion took place during load removal. Accordingly, the length of cracks directed along the square diagonals, i.e. along $\langle 1\,1\,0\rangle$, increases during the process of indentor unloading. These specific cracks have been observed by a number of authors for MgO single crystals and for the other ionic crystals with the NaCl lattice [6, 8, 10–12, 15–18]. They end up, as a rule, in the apexes of slip-line squares (Fig. 1) [6, 10]. Their formation seems to be related to the occurrence of pile-ups of sessile dislocations which arise by the

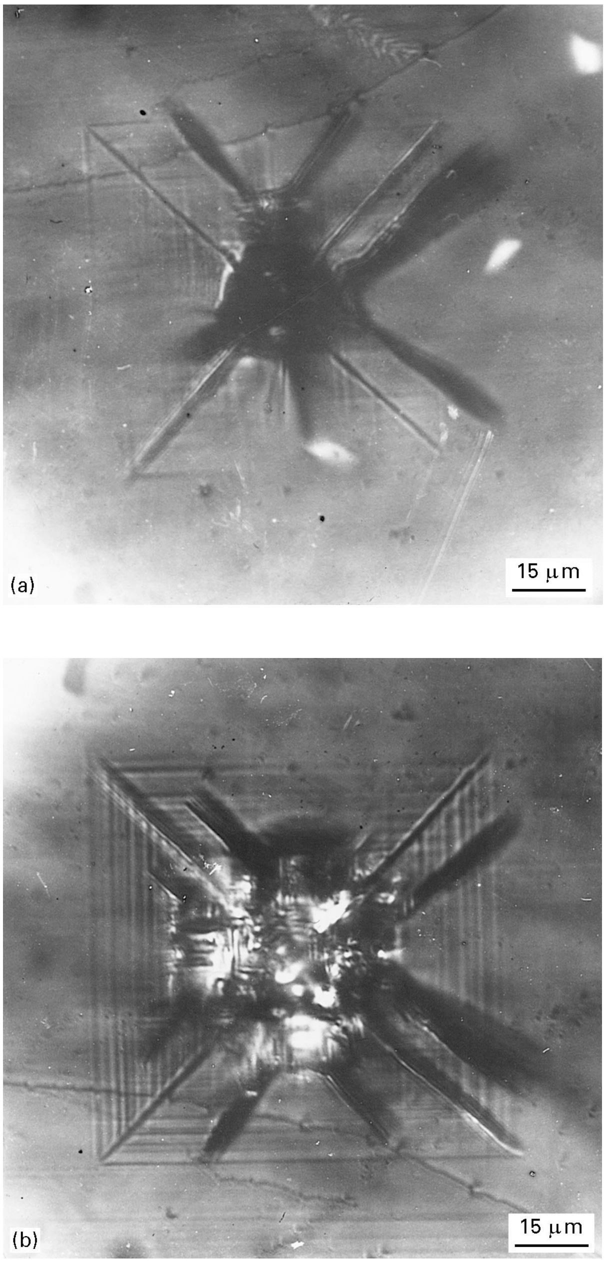

Figure 1 The slip lines and failure patterns near the indentations on the (0 0 1) plane of MgO crystals produced by the indentor (a) under load and (b) after unloading. $P = 500$ g.

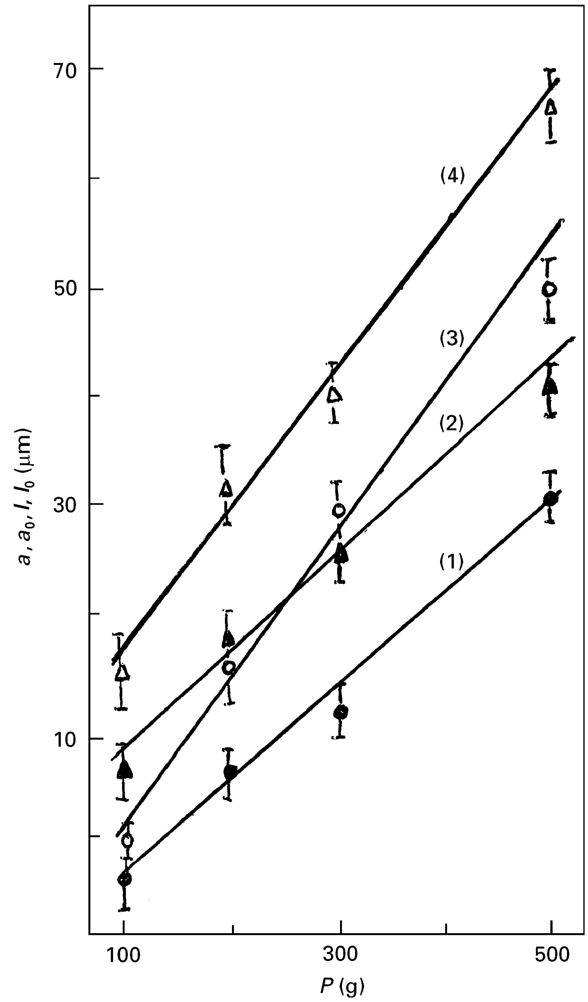

Figure 2 The dependence of the size of the slip-line squares (a_0, a) and length of cracks along $\langle 1\,1\,0 \rangle$ (l_0,l) on load. a_0,l_0 are the parameter values for the indentor under load, and a,l after unloading.

TABLE I The relative change in the sizes of slip-line squares and cracks along $\langle 1\,1\,0 \rangle$ during unloading

P(g)	$d \parallel \langle 1\,0\,0 \rangle$		$d \parallel \langle 1\,1\,0 \rangle$	
	$\lvert\Delta a\rvert / a_0$ (%)	$\lvert\Delta l\rvert / l_0$ (%)	$\lvert\Delta a\rvert / a_0$ (%)	$\lvert\Delta l\rvert / l_0$ (%)
100	190	195	143	97
200	75	70	86	66
300	70	80	98	74
500	34	32	46	42

interaction of dislocations moving in the $\{1\,1\,0\}_{45}$ slip planes [6, 8, 15].

The completion effect of square-shaped slip traces in the unloading process was clearly observed for different indentor loads. Fig. 2 shows the result for the indentation orientation $d \parallel \langle 1\,0\,0 \rangle$. Analogous data were obtained for the other orientation – $d \parallel \langle 1\,1\,0 \rangle$. It is seen for the case of the indentor under load that the dependence of the side of the square region, a_0, on load is a linear one. After unloading, $a(P)$, a straight line is observed (a is the size of the side of the square region after unloading). It is practically parallel to $a_0(P)$ but is higher (Fig. 2, lines 1, 2), i.e. the increase in the size of the deformed region near indentations occurs during indentor removal for all the loads in question. However, the relative change $(a - a_0)/a_0 = \Delta a/a_0$ decreases with increasing load (Table I).

The same peculiarities are also observed in measuring the crack length (Fig. 2, straight line 3, 4; Table I): the distance between the ends of the cracks propagating along $\langle 1\,1\,0 \rangle$ in the opposite directions was determined (Fig. 1). Fig. 2 shows that the $l(P)$ and $l_0(P)$ straight lines (l_0 and l are the size of the crack length for the case of indentor under load and that after unloading, respectively) are deflected by a specific angle concerning $a(P)$ and $a_0(P)$. It is completely regular.

The ratios

$$k_1/k_3 = k_2/k_4 = 0.71 \tag{1}$$

must be fulfilled for perfect squares and cracks propagating along their diagonals and ending in their apexes, because $l = a \times 2^{1/2}$. Here k_i are the constants from the equations:

$$a = k_1 P + k_2 \qquad (2a)$$

$$l = k_3 P + k_4 \qquad (2b)$$

The plots presented in Fig. 2 (straight lines 2, 4) give us

$$k_1/k_3 = 0.6 \qquad (3a)$$

$$k_2/k_4 = 0.64 \qquad (3b)$$

In spite of the fact that the slip-line squares and cracks along $\langle 1\,1\,0 \rangle$ are observed for the different indentor orientations, the anisotropy of the parameters under investigation is revealed (Figs 3 and 4). The square size and the crack length are dependent on the directions of the indentation diagonals. This anisotropy is small and virtually within the error limits for indentor loads of 100–200 g. However, it increases when the load rises and values a_0, a, l_0, l parameters are greater for the $d \| \langle 1\,0\,0 \rangle$ orientation than for the $d \| \langle 1\,1\,0 \rangle$.

The presence of the investigated parameter anisotropy is in agreement with the fact that the microhardness anisotropy is clearly revealed on the (0 0 1) plane of MgO single crystals. So $H_{\langle 1\,0\,0 \rangle} > H_{\langle 1\,1\,0 \rangle}$ ($H_{\langle 1\,0\,0 \rangle}$ and $H_{\langle 1\,1\,0 \rangle}$ are the microhardness values for the orientations of $d \| \langle 1\,0\,0 \rangle$ and $d \| \langle 1\,1\,0 \rangle$, respectively) for the Vickers indentor [7, 11, 12, 19, 20].

All the results presented here permitted us to conclude that the unexpected phenomenon is observed on the (0 0 1) plane of MgO for the different indentor loads and different indentor orientations concerning the crystallographical directions of the deformed plane – plastic deformation having the same sign as in

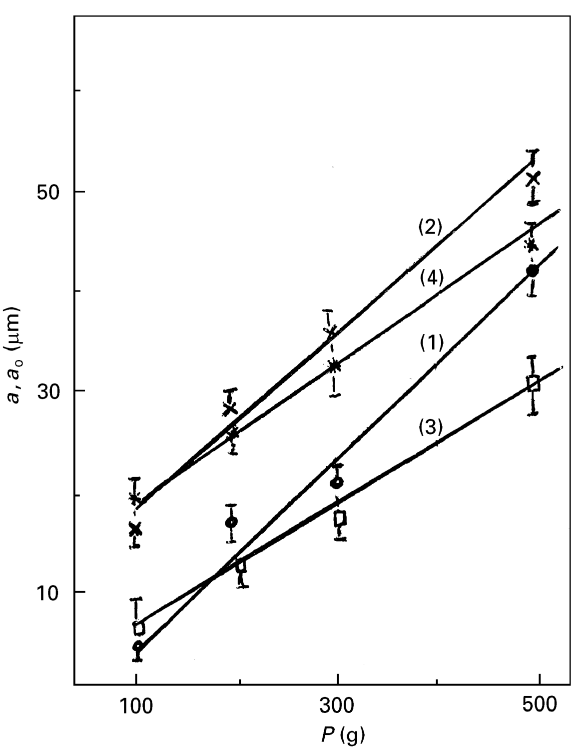

Figure 3 The comparison of $a_0(P)$ (1, 3) and $a(P)$ (2,4) dependences for two indentor orientations: $d \| \langle 1\,0\,0 \rangle$ (1, 2) and $d \| \langle 1\,1\,0 \rangle$ (3, 4).

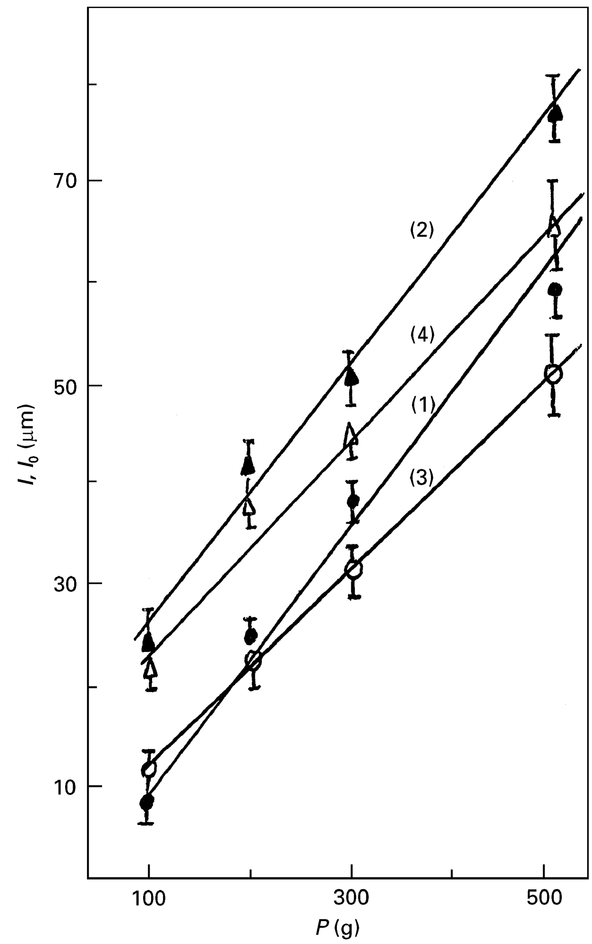

Figure 4 Comparison of $l_0(P)$ (1, 3) and $l(P)$ (2,4) dependences for two indentor orientations: $d \| \langle 1\,0\,0 \rangle$ (1, 2) and $d \| \langle 1\,1\,0 \rangle$ (3, 4).

the loading process takes place during indentor removal. The additional material is carried out to the surface by the $\{1\,1\,0\}_{45}$ convergent planes which are responsible for the formation of the hills of pressed-out materials near the indentations. According to the material plastic-flow scheme for the (0 0 1) plane indentation of ionic crystals such as NaCl, the $\{1\,1\,0\}$ active slip planes can be divided into two types: divergent ones which form tetrahedral pyramids with the apex near the surface and base at a depth, and convergent ones which form the inverted pyramid. The former planes are responsible for the material shift to a depth of the sample, and the latter ones for its transport to the surface [6, 12–14, 17]. The latter is evinced by the following facts. The bulging of the sample surface in the region of the slip-line square is observed for both MgO [6, 11] and other ionic crystals such as NaCl [12, 13, 17]. Our observation, performed by use of the interference microscope, confirmed in this fact (Fig. 5). The spreading distances of hills along $\langle 1\,1\,0 \rangle$ directions near the indentations on the (0 0 1) plane were measured for a series of ionic crystals, including MgO [21]. For MgO crystals deformed at room temperature, the parameter $L/d \sim 1.2$ (L is the spreading distance of hills from the side of the indentation with $d \| \langle 1\,0\,0 \rangle$ orientation). Measurements of L' from the indentation side to the apex of the slip-line square showed that $L/d = 1.25$, i.e. the hill along $\langle 1\,1\,0 \rangle$ ends at this apex.

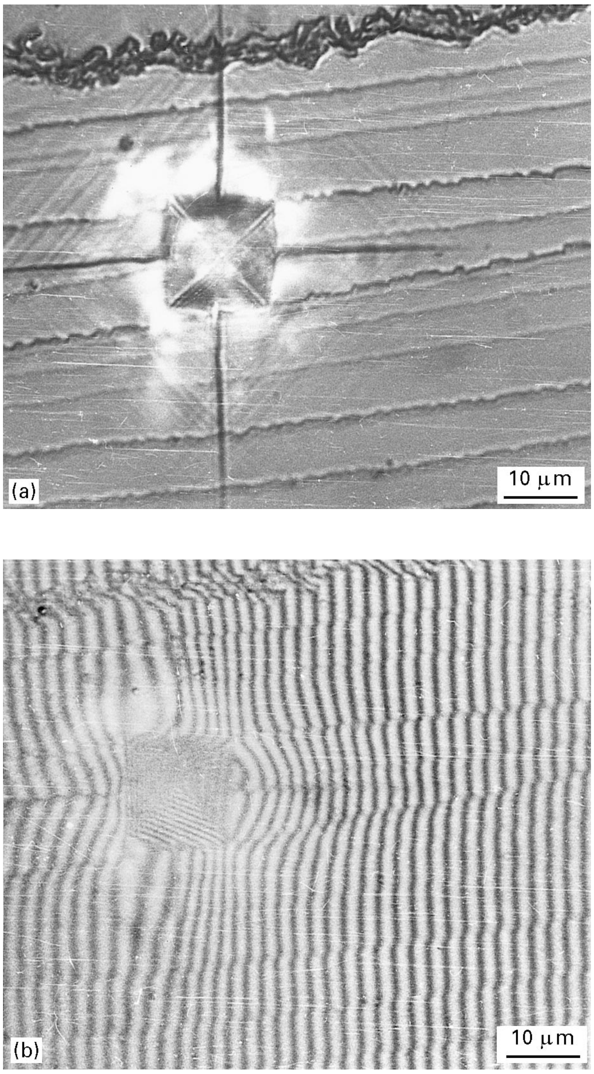

Figure 5 (a) The indentation on the (0 0 1) plane of MgO, $d \| \langle 1\,1\,0 \rangle$, $P = 200$ g, and (b) the interferogram of the surface nearby.

The occurrence of prolonged plastic deformation during the process of indentor removal can be explained as follows. An opinion was expressed concerning the presence of smooth and impulse mechanisms of the microindentation process of crystals [12, 22]. This opinion was confirmed by a series of experimental data [5, 23–25]. The synchronous development of the indentation and deformed zone around it takes place for the smooth mechanism. This synchronism is disturbed for the impulse mechanism. Rather strong dislocation pile-ups arise in the deformed region near the indentation, and the broadening of this region is stopped. At a certain time, the bursting of the dislocation pile-ups occurs and the intensive formation of a deformed region takes place. That is, in this case, the process of development of dislocation structures near the indentation has a relaxation character. The appearance of new slip bands, dislocation rosette arm formation, etc., is connected to the action of stress sources. Some of these sources can be non-active during indentor penetration, but they can act during the indentor resting in the sample and during unloading. The latter can operate as a trigger, resulting in the action of stress sources arranged in the deformed region. Therefore, the deformation during unloading can have the same sign as in loading and whilst the indentor rests in the crystal.

TABLE II The influence of indentor standing time in the sample, t, on the change of the size of the slip-line square and crack length during unloading. $P = 300$ g

Indentor orientation	Δa (μm)		Δl (μm)	
	$t = 1$ min	$t = 1$ h	$t = 1$ min	$t = 1$ h
$d \| \langle 1\,0\,0 \rangle$	14 ± 2.6	12 ± 3	22 ± 3	16 ± 3
$d \| \langle 1\,1\,0 \rangle$	17 ± 2.5	12 ± 2	21 ± 4	17 ± 2.5

Such considerations are quite applicable to MgO crystals. The opinion has been expressed earlier that an impulse mechanism of microindentation must occur in these crystals, in contrast to the alkali halide ones, at room temperature [12, 22]. Therefore, one can expect the following. If the time an indentor stands in the sample is sufficient, the stress sources can be partly brought into action under load. Then Δa and Δl changes during the unloading must be less in comparison with those for the usual loading time (~ 15 s). The results listed in Table II show the validity of this suggestion.

4. Conclusion

An unusual phenomenon was revealed by the use of MgO single crystals as an example: further development of the plastic deformation region near an indentation occurs after indentor removal from the sample. The enhancement of slip-line squares formed near the indentations on the (0 0 1) plane of MgO takes place during unloading. These slip lines are the traces of the $\{1\,10\}_{45}$ planes which are responsible for the material transport from under the indentor to the surface, and for the formation of hills of pressed-out material. Thus the additional material transport to the surface occurs during the unloading process. The phenomenon of prolonged plastic deformation can be explained by considering the fact that the impulse mechanism of the microindentation process of MgO takes place at room temperature.

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