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Regularities of MgO single-crystal failure in microindentation

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The peculiarities of the fracture of indentations on the (0 0 1) plane of MgO single crystals were investigated for two orientations of the Vickers indentor: (i) the indentation diagonals were parallel to the ⟨1 0 0⟩ direction, and (ii) the diagonals were parallel to ⟨1 1 0⟩. A brittleness anisotropy was revealed. A specific structure of "square" cracks was observed inside the indentations of the first orientation. Such a structure did not appear in the case of the second orientation. An explanation of the brittleness anisotropy was suggested using a model of material plastic flow in the indentation and considering the interaction of dislocations belonging to the active slip planes.

1. Introduction

In order to understand the mechanism of the process of material microindentation it is reasonable to investigate, together with the features of the plastic deformation, the peculiarities of the fracture as well. In the present paper such investigations are performed on the (0 0 1) plane of MgO single crystals. As is known from the literature [1–6] the specific cracks directed along ⟨1 1 0⟩ (i.e. not coinciding with the directions of cleavage plane emergence) arise near the indentation arranged on this plane. It is also true for alkali halide crystals such as NaCl [7–9]. These cracks are considered to have a dislocation origin [1–6, 8].

However, the fracture regularities inside the indentation as far as we know from the literature have practically not been investigated. The study of these peculiarities is the main aim of the present paper.

2. Experimental procedure

Crystal deformation was performed with the microhardness tester PMT-3. A Vickers diamond pyramid was used as an indentor. The indentor load P was varied in the range 10 to 200 g. The indentation was performed at room temperature. Indentations of two orientations were investigated: (i) the indentation diagonals (d) are parallel to the ⟨1 0 0⟩ direction, and (ii) d ∥ ⟨1 1 0⟩. Studies of the indentation appearance was made using a scanning electron microscope (Tesla BS-300).

3. Results and discussion

Inside the indentation with d ∥ ⟨1 0 0⟩ one can see the crack system on using even small indentor loads; the directions of the cracks coincide with ⟨1 1 0⟩ (Fig. 1a). Growth of these cracks occurred on increasing the load and their arrangement had the shape of a series of squares within each other (Fig. 2a). It should be noted that similar fracture patterns, known as a periodic fracture structure, develop in ionic crystals under the action of a powerful electron beam [10]. It is also interesting to note that an analogy between two other structures–the dislocation rosettes arising in the indentation of alkali halide crystals and in the action of laser radiation–was observed earlier [11]. Some other cracks (apart from those mentioned above) and some brittle damage appeared in the indentation process as well (Fig. 2a).

The failure patterns inside the indentations undergo an important change on turning the indentor through 45°. Cracks in the indentation centre do not appear in the case of low indentor loads (Fig. 1b). Near the indentation edges there are cracks parallel to the sides; some representative external cracks directed along ⟨1 1 0⟩ and mentioned above "penetrate" into the indentation. The degree of failure intensified on increasing the indentor load; cracks appear inside the indentation and brittle damage of the material occurs (Fig. 2b). However, a clear crack system similar to that arising for the first orientation (Fig. 2a) is not observed in this case.

As is known from the literature, the appearance of cracks in the indentation of different materials is accompanied by the initiation of acoustic emission signals [6, 12–16]. The acoustic emission method is sensitive enough for the detection of brittleness anisotropy [6, 14]. Therefore it was reasonable to use this method for study of the damage anisotropy revealed in the present paper for the (0 0 1) plane of MgO. The equipment for the registration of the acoustic emission signals arising from the indentation and the experimental procedure have been described earlier [6]. The acoustic emission impulse number N which exceeded the established discrimination level (cumulative acoustic emission calculation) and the rate of acoustic emission $\dot{N}$ were measured.

Table I lists the results of the measurements for two indentor orientations and two indentor loads. Each number in the table is obtained on the basis of averaging the results for 20 indentations. N_1, N_2 and N_3 denote the acoustic emission impulse numbers arising

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TABLE I Acoustic emission (AE) in the (0 0 1) indentation of MgO

AE impulse number	$P = 50$ g		$P = 100$ g	
	d ∥ ⟨1 0 0⟩	d ∥ ⟨1 1 0⟩	d ∥ ⟨1 0 0⟩	d ∥ ⟨1 1 0⟩
N_1	790	840	1140	1140
N_2	885	860	1200	1175
N_3	940	895	1390	1320

TABLE II Changes of cumulative acoustic emission in the indentation process of (0 0 1) planes

ΔN	$P = 10$ g		$P = 50$ g		$P = 100$ g		$P = 200$ g	
	d ∥ ⟨1 0 0⟩	d ∥ ⟨1 1 0⟩	d ∥ ⟨1 0 0⟩	d ∥ ⟨1 1 0⟩	d ∥ ⟨1 0 0⟩	d ∥ ⟨1 1 0⟩	d ∥ ⟨1 0 0⟩	d ∥ ⟨1 1 0⟩
ΔN_{21}	109	62	270	13	141	64	98	21
ΔN_{32}	8	84	24	63	91	141	135	184
ΔN_{31}	117	146	294	76	232	205	233	205

during the indentor penetration, after its standing in the sample (~ 15 sec) and after unloading, respectively.

As is seen from the Table I, brittleness anisotropy is practically absent in the first moments of the indentor penetration ($P = 100$ g), or the damage is somewhat more expressed for the d ∥ ⟨1 1 0⟩ orientation as compared with the d ∥ ⟨1 0 0⟩ one ($P = 50$ g). However, the situation changes when the indentor is kept in the sample: N_2 for the d ∥ ⟨1 0 0⟩ orientation turns out to be larger in comparison with that for the other orientation. Such a case is conserved after unloading. Therefore it is of interest to find not only the absolute values of N, but also the changes for different stages of the indentation process. Table II lists the corresponding data. Here $\Delta N_{21} = N_2 - N_1$, i.e. the acoustic emission impulse number accumulated while the indentor is held in the sample; ΔN_{32} is the impulse number arising during the unloading; $\Delta N_{31} = N_3 - N_1$ is the combined impulse number registered during these two processes.

As is seen from Table II, the crack accumulation process while the indentor is held in the sample is more intensive for the d ∥ ⟨1 0 0⟩ indentor orientation as compared with the d ∥ ⟨1 1 0⟩ one; this is true for all the loads under investigation. In contrast to this the relaxation resulting from the unloading is followed by more intensive material damage for the indentations of the second orientation.

Comparing the results obtained from acoustic emission and the electron microscope investigations, one can conclude that a well-developed system of crystallographically directed cracks observed inside the indentations of the d ∥ ⟨1 0 0⟩ orientation (Figs 1a and 2a) and missing for the other orientation (Figs 1b and 2b) evidently appears during the penetration process of the indentor and during its holding under load rather than in the moment of touching. The formation of these cracks can be connected with stress relaxation resulting from the annihilation of dislocation pile-up.

It is therefore reasonable to use the model of material plastic flow in the indentation on the (0 0 1) plane of ionic crystals with an NaCl lattice [6, 9, 17, 18] for explanation of the failure anisotropy observed experimentally. Let us consider the deformation peculiarities for two indentor orientations (Fig. 3). According to this model, two pairs of slip systems have to be active in the AOB region of the indentation: the first pair is $[1 0 \bar{1}]$ $(1 0 1)$ (providing downward material movement) and $[1 0 1]$ $(1 0 \bar{1})$ (upward material movement); the second pair is $[0 1 \bar{1}]$ $(0 1 1)$ (downward slip) and $[0 1 1]$ $(0 1 \bar{1})$ (upward slip). Dislocation reactions can take place when the dislocations of one pair belonging to the planes responsible for downward material movement meet the dislocation of the other pair belonging to the planes responsible for upward material movement. These reactions result in a sessile dislocation formation. For example, the dislocation reaction

$$\frac{a}{2}[1 0 \bar{1}] + \frac{a}{2}[0 1 1] \rightarrow \frac{a}{2}[1 1 0] \qquad (1)$$

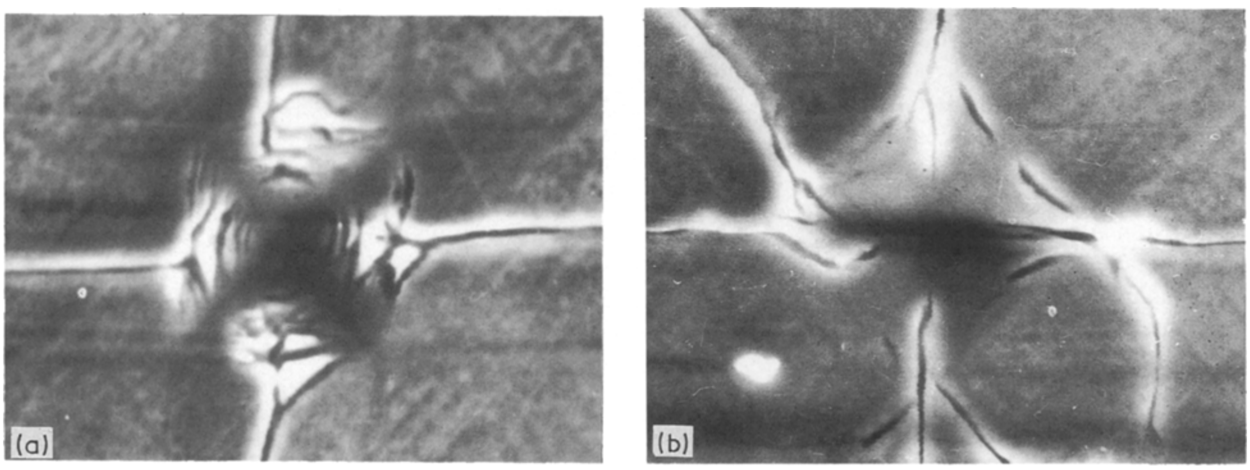

Figure 1 Fracture patterns inside the indentations on the (0 0 1) plane of MgO single crystals. $P = 10$ g. (a) d ∥ ⟨1 0 0⟩, (b) d ∥ ⟨1 1 0⟩. (× 4800).

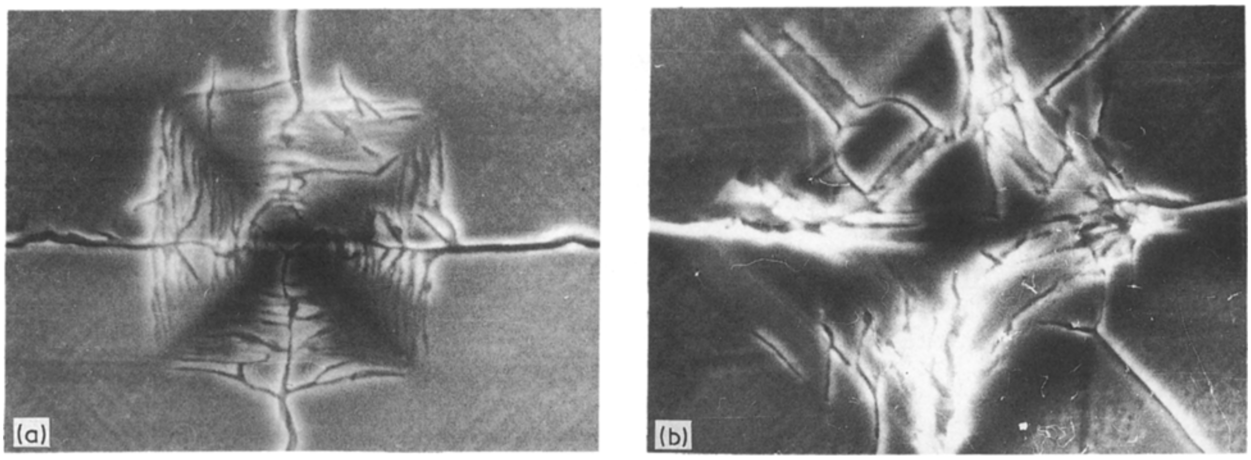

Figure 2 Fracture patterns inside the indentations on the (0 0 1) plane of MgO single crystals. $P = 100$ g. (a) d $\parallel \langle 1\,0\,0\rangle$, (b) d $\parallel \langle 1\,1\,0\rangle$. ($\times$ 2800).

is energetically advantageous. As a result of it an edge dislocation placed along the $[\bar{1}\,1\,1]$ direction in the $(1\,\bar{1}\,2)$ plane appears. This dislocation is a sessile one as planes of the $\{1\,\bar{1}\,2\}$ type are not slip planes of the crystals under investigation. The pile-up of the sessile dislocations results in a stress concentration. The relation of the latter is followed by the formation of cracks. These cracks have to be mainly arranged parallel to AB, as this direction coincides with the direction of $[\bar{1}\,1\,1]$ projection on the investigated plane.

Another situation is observed in the d $\parallel \langle 1\,1\,0\rangle$ indentor orientation (Fig. 3b). In this case one pair of slip planes which are responsible for the downward and upward material movement acts roughly in the AOB region. Such a reaction is possible among the dislocations belonging to these planes ((1 0 1) and $(1\,0\,\bar{1})$):

$$\frac{a}{2}[1\,0\,\bar{1}] + \frac{a}{2}[1\,0\,1] \rightarrow a[1\,0\,0] \qquad (2)$$

There is no guarantee that the Reaction 2 will take place, because no decrease of elastic energy occurs in this process; however, in KCl crystals this reaction was observed under certain conditions [19]. If such a reaction takes place in MgO crystals, it will cause the formation of an edge dislocation parallel to the [0 1 0] direction with the a[1 0 0] Burgers vector. This dislocation is placed in the (0 0 1) plane, i.e. it is a sessile dislocation. The pile-up of such dislocations may result in the formation of cracks along the AB direction, i.e. parallel to the indentation sites. Separate cracks of such a type are observed for indentations with the d $\parallel \langle 1\,1\,0\rangle$ orientation (Figs 1b and 2b); however, inside these indentations the system of "square" cracks clearly revealed for the other orientation d $\parallel \langle 1\,0\,0\rangle$ (Figs 1a and 2a) is absent. This may easily be understood because the dislocation Reaction 1 contributing to the formation of this system is energetically advantageous and consequently more probable in comparison with Reaction 2, which is not followed by a decrease of elastic energy.

4. Conclusions

1. Brittleness anisotropy was revealed in the indentation of the (0 0 1) plane of MgO single crystals using electron microscopy and acoustic emission methods.

2. A specific structure of "square" cracks was observed inside the indentations with a d $\parallel \langle 1\,0\,0\rangle$ orientation (where d is the indentation diagonal). Such a structure did not appear for the indentations having d $\parallel \langle 1\,1\,0\rangle$ orientation.

3. An explanation of the brittleness anisotropy was suggested using a model of material plastic flow in the indentation and considering the interaction of dislocations belonging to the active slip planes.

4. The results obtained in the present paper confirm indirectly the correctness of an interpretation of the main plastic deformation peculiarities in the indentation of crystals such as NaCl, as suggested earlier [6, 9, 17].

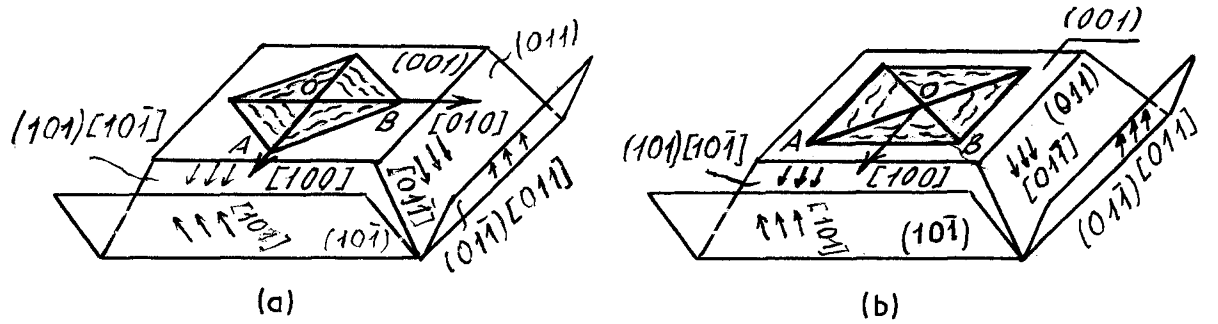

Figure 3 A scheme of material plastic flow in (0 0 1) indentation of crystals such as NaCl Indentation orientation: (a) d $\parallel \langle 1\,0\,0\rangle$, (b) d $\parallel \langle 1\,1\,0\rangle$.

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