Anisotropy of microhardness on the (001) plane of ionic crystals such as NaCl

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The anisotropy of microhardness, H, was investigated for the series of KCl–NaCl–NaF–LiF–MgO crystals using a Vickers indentor. The anisotropy had a similar character for deformation temperatures of 77, 293 and 800 K ($H_{[100]} > H_{[1\overline{1}0]}$); however, the main factors affecting it were different for low and high temperatures. The dependence of the anisotropy coefficient $K = (H_{[100]} - H_{[1\overline{1}0]})/H_{[100]}$ on the crystal hardness was found and it was shown that this dependence was significantly influenced by the deformation temperature. The results obtained support the dislocation mechanism of indentation formation on the crystals investigated.

1. Introduction

The microindentation method permits the anisotropy of the mechanical properties of crystals to be revealed. The shape and size of the indentations, pictures of the slip bands and the destruction are dependent on the indentor orientation relative to the crystallographic directions of the sample [1–4]. It is of interest to study this phenomenon as it allows to gain understanding of the nature of the microindentation process which has been under discussion until now.

Several facts which may be responsible for the microhardness anisotropy observed experimentally, have been considered in literature. They are: the distortion of the surface relief near the indentation [1, 3, 4]; the dependence of the effective shear stresses, arising in the slip planes along the slip directions, on the indentor orientation [5–8]; and the elastic recovery of the indentations which takes place after unloading [3, 4, 9–11]. The last factor is significant only for the hard and markedly anisotropic crystals [3, 4]. A study of the anisotropy of the mechanical properties of crystals such as NaCl revealed that the indentation shape on the cube plane was dependent on the Vickers indentor orientation: the indentations had convex sides if the indentation diagonals, d, were orientated parallel to the $\langle 100 \rangle$ directions; the indentations had concave sides when the indentor was turned through 45°, i.e. for the orientation $d \parallel \langle 110 \rangle$ [3, 12–15]. It has been shown [3, 13, 15] that this specific shape of the indentations was formed during the indentation process, i.e. it was not connected with elastic recovery nor with dislocation motion after unloading. Its appearance was connected with the hills of the pressed-out material arising along the $\langle 110 \rangle$ directions, independent of indentor orientation [3]. Different opinions about the nature of these hills are found in the literature [3, 16, 17].

The orientation dependence of the indentation shape, the occurrence of material hills and the dependence of the effective resolved shear stresses, acting in the slip planes, on the indentor orientation, resulted in the microhardness anisotropy observed on the (001) plane of NaCl [3, 12], LiF [18], MgO [19, 20] single crystals. Both factors (the features of the hills and the difference in the effective shear stresses for different orientations) operate in a similar manner and result in a maximum microhardness, H, for the $d \parallel \langle 100 \rangle$ orientation and a minimum H for the $d \parallel \langle 110 \rangle$.

Using NaCl single crystals as an example, the influence of temperature and doping on mechanical properties anisotropy, observed in the indentation, has been investigated [3, 21, 22]. It has been reported that the microhardness anisotropy was connected with the peculiarity of the crystal slip, but its correspondence with the anisotropy of the elasticity has also been noted [22]. It has been shown that the anisotropy of the indentation shape on the (001) plane of NaCl crystals was markedly decreased at elevated deformation temperatures [3]. Therefore, it was natural to expect a decrease in the H anisotropy at these temperatures. However, a tendency towards an increase of this characteristic but not a decrease was observed for NaCl crystals [3]. From this it may be concluded that further investigation of the microhardness anisotropy of crystals is needed.

Therefore, a study of this characteristic for the series KCl–NaCl–NaF–LiF–MgO was attempted in the temperature region 77 to 800 K; these results are reported here. The crystals have the same lattice structure, the same slip systems $\{110\}\langle \overline{1}10 \rangle$ and a gradually rising hardness (from $10 \, \text{kg mm}^{-2}$ for KCl to $660 \, \text{kg mm}^{-2}$ for MgO at room temperature).

2. Experimental procedure

The (001) plane of the crystals under investigation was obtained by normal cleavage techniques. Measurements of microhardness were performed with the microhardness tester PMT-3. The Vickers diamond was used as an indentor. The indentor loads were selected in such a way as to obtain indentation diagonals approximately equal for all the crystals under investigation, and to eliminate the significant brittle

TABLE I Microhardness anisotropy of ionic crystals

Crystal	T = 77 K				T = 293 K				T = 800 K			
	H (kg mm^{-2})		ΔH (kg mm^{-2})	K (%)	H (kg mm^{-2})		ΔH (kg mm^{-2})	K (%)	H (kg mm^{-2})		ΔH (kg mm^{-2})	K (%)
	$d\|\langle 100\rangle$	$d\|\langle 110\rangle$			$d\|\langle 100\rangle$	$d\|\langle 110\rangle$			$d\|\langle 100\rangle$	$d\|\langle 110\rangle$		
KCl	51.2	20.7	30.5	60.0	10.0	9.8	0.2	2.3	1.9	1.2	0.7	35.0
NaCl	78.0	54.1	23.9	30.6	20.0	19.3	0.7	3.3	3.5	2.0	1.5	42.0
NaF	184.7	151.9	32.8	18.0	74.0	69.6	4.4	6.0	12.6	11.0	1.6	12.6
LiF	245.8	213.6	32.2	13.1	108.0	101.1	6.9	6.4	16.0	17.2	−1.2	7.5
MgO	670.0	454.3	215.7	32.2	662.0	492.0	170.0	25.0	471.0	422.0	49.0	10.4

fracture arising during indentation. The error in the diagonal measurement was $\sim 3\%$. The microhardness was calculated using the usual formula [1]. A furnace for sample heating, which allowed the deformation in the temperature interval 293 to 800 K to be performed, or a special liquid nitrogen bath for the indentation at 77 K, were mounted on the testing table.

3. Results and discussion

The results obtained from microhardness measurements of the crystals under investigation at different temperatures are given in Table I. The size of the indentation diagonals for all the materials and all the temperatures is approximately 22 μm. As seen, the temperature significantly influences the value of the microhardness, especially for soft crystals. For example, the microhardness of KCl and NaCl decreases by a factor of 28 and 20, respectively, in the temperature range 77 to 800 K. However, the microhardness anisotropy is clearly revealed for all temperatures

under investigation. Fig. 1 shows the results for the coefficient of microhardness anisotropy, $K(K = (H_{[100]} - H_{[110]})/H_{[100]} = \Delta H/H_{[100]})$, obtained at different deformation temperatures. As seen, the coefficient of anisotropy increases with increasing crystal microhardness when measurements are performed at room temperature; the $K(H)$ dependence is approximately linear. The change in K when passing from soft crystals to hard ones is rather significant: $K = 2.3\%$ for the softest crystal, KCl, and $K = 25\%$ for the hardest crystal, MgO.

The occurrence of such a dependence is entirely natural if the influence of the material hills on the value of the indentation diagonals is taken into account. Indeed, it is known from the literature that increase in crystal microhardness, connected with a lowering of the temperature or with doping, is followed by a decrease in deformation zone size, as the hills of the pressed-out material become nearer to the indentation and as their height increases [3]. Therefore, a
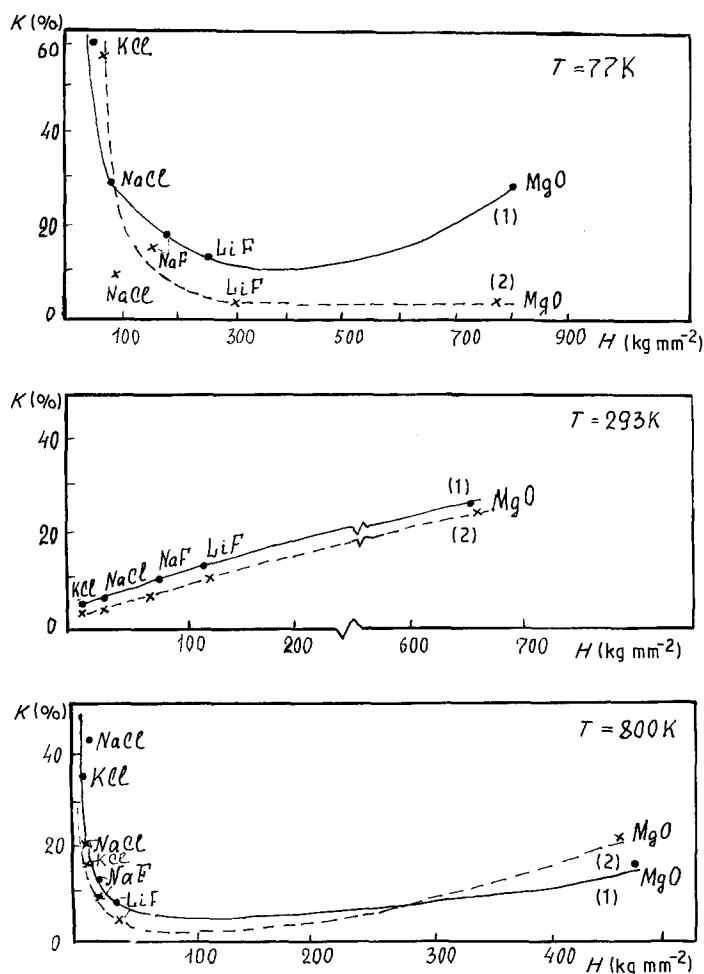

Figure 1 The dependence of the coefficient of microhardness anisotropy, K, on the crystal hardness at the different deformation temperatures. (1) $d = 30\,\mu$m; (2) $d = 45\,\mu$m.
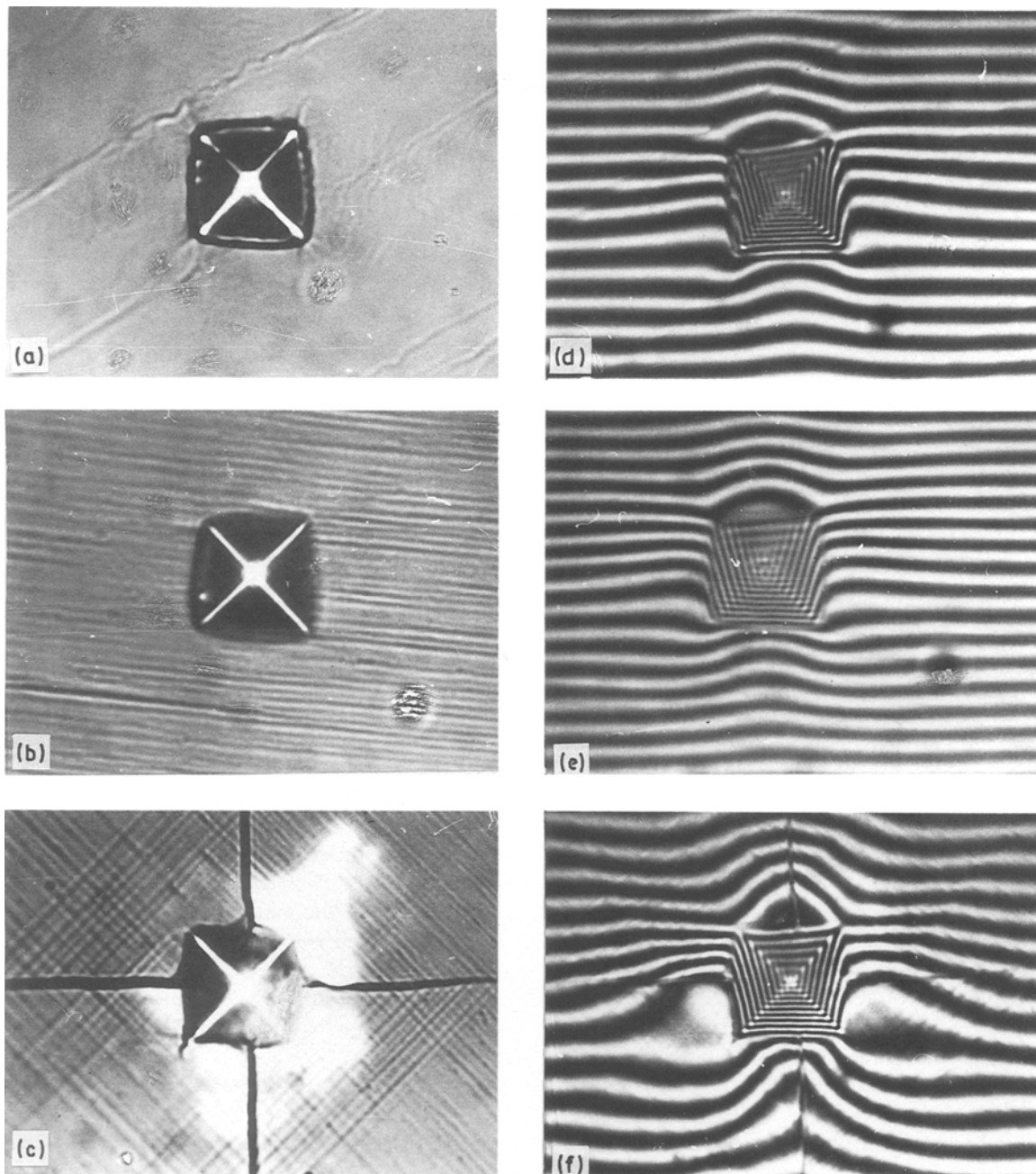
Figure 2 The shape of the indentations (a to c) and their interferogram (d to f) on the (0 0 1) plane of (a, d) NaCl, (b, e) LiF, (c, f) MgO single crystals. $d \parallel \langle 1\,0\,0 \rangle$, $T = 293\,K$.

decrease in the extent of the deformation range and an increase in the hill height are expected to take place with rising hardness in the KCl–NaCl–NaF–LiF–MgO series. This assumption has been confirmed by observing the surface relief near the indentation (Fig. 2). As is seen, the bending of the interference lines near the indentations increases regularly, on passing from softer crystals to harder ones. However, the shape of the $K(H)$ dependence at low $(T = 77\,K)$ and high $(T = 800\,K)$ temperatures is rather unexpected (Fig. 1). In this case the coefficient of microhardness anisotropy is maximum for the softest KCl crystal and decreases regularly in passing from KCl to LiF.

In order to understand the reason for this dependence it is necessary to consider the changes in K with temperature. It may be expected that increasing microhardness of the crystals under investigation on lowering the temperature from 293 to 77 K must be

followed by an increase in the anisotropy coefficient, but the temperature softening in the interval 293 to 800 K must be followed by a decrease in K.

As seen from Table I, the transition in the deformation from room temperature to 77 K is indeed followed by a marked enhancement of the anisotropy coefficient for all the crystals under investigation. However, in the temperature interval 293 to 800 K, an increase in K is seen, but no decrease (MgO crystals are an exception). The change in K with temperature is schematically shown in Fig. 3. It is seen that the regions of the curve to the left of 293 K are a rising branch, and the softer the crystals the steeper the shape of the curve becomes.

Measurements of the height and spread of the hills, arranged along the $\langle 1\,1\,0 \rangle$ directions, were made, using the interferometry, to explain this result. Fig. 4 shows that the influence of changes in the deformation
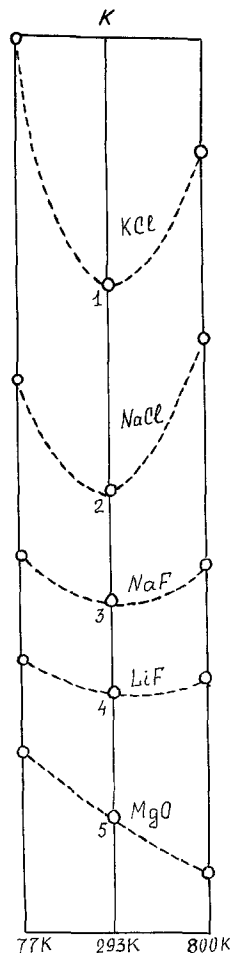
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Figure 3 Schematic illustration of the temperature dependence of the coefficient of microhardness anisotropy.

material are already positioned very close to the indentation for MgO crystals at room temperatures (Fig. 4, curve 4); therefore, it seems natural that the decrease in temperature in the interval 293 to 77 K does not have a very significant influence on K.

So the enhancement of the coefficient of microhardness anisotropy on passing from room temperature to the liquid nitrogen temperature may be explained by the influence of the hills on the size of the indentation diagonals.

Let us consider the change in the coefficient of microhardness anisotropy on increasing the deformation temperature from 293 K to 800 K. An increase in K in this temperature region occurs for all the crystals under investigation, except MgO (Table I, Fig. 3). It is possible that the transition from the "descending" branch of the $K(T)$ dependence to the "rising" branch for MgO occurs in a higher temperature region in comparison with that for the other crystals.

The rise in K on increasing the temperature from 293 to 800 K can be explained as follows. A change in the mechanism of surface relief formation takes place as the temperature rises, and it is known from the literature [23] that the hardness of the material and its capacity for strain hardening have a significant influence on the peculiarities of surface relief formation near the indentations. If the indentor penetrates the material with a small strain hardening capacity, the formation of hills occurs. In contrast, a depression of the surface near the indentation arises when penetrating a soft material. Therefore, one can assume that this factor, and not the material hills, must be the main influence on microhardness anisotropy at elevated temperatures.

This assumption was confirmed by studying the change of the indentation shape with increasing temperature. It was shown, as is the case with the hills of a material, that the dips in a material were arranged along definite crystallographic directions; these are $\langle 1 0 0 \rangle$ [3, 21]. The occurrence of a surface depression near the indentation results in a shortening of its diagonals, therefore $H_{\langle 100 \rangle} > H_{\langle 110 \rangle}$. This difference increases with increasing temperature because in this case the degree of surface lowering also increases. The temperatures used in the present investigation were apparently insufficiently high to operate such a mechanism for the hardest MgO crystal.

The non-monotonic shape of the $K(H)$ curves (Figs 1a, c) can be explained by the common influence of the two facts shown: the increase in the coefficient of microhardness anisotropy in the region of small H values at 77 K (Fig. 1a) is connected with the influence of the material hills, but a similar shaped curve at 800 K (Fig. 1c) is found due to the action of the material depressions. The results obtained show the importance of the dislocation mechanism of indentation formation for the crystals under investigation in the temperature region 77 to 800 K. One further factor influencing the microhardness anisotropy was shown by Roberts [24]: the dependence of strain hardening in the region near the indentation on the indetor orientation. This is connected with the change in the activity of different slip planes due to the indentor turn relative

temperature is more significant for the soft crystals. The top of the hill on KCl is positioned so far from the indentation at 293 K that it has practically no influence on the diagonal value. However, the hill is close enough to the indentation edges at 77 K to have an influence (Fig. 4, curve 1). In addition, the height of the hill in this temperature region increases almost by a factor of two. A similar increase in hill height also takes place for LiF; however, the arrangement of the hills changes insignificantly (Fig. 4, curve 3). Therefore, the degree of microhardness anisotropy for LiF undergoes less significant changes. The hills of the
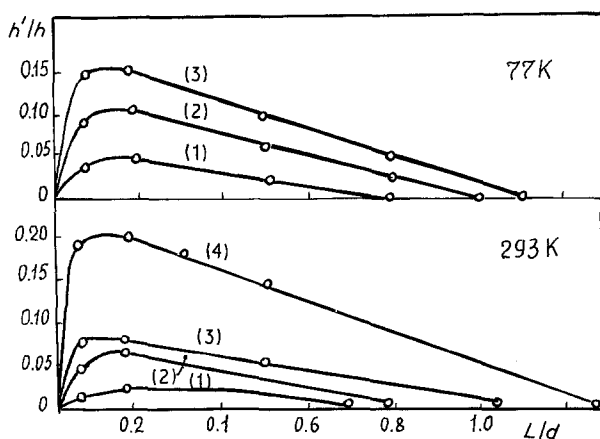

Figure 4 The profiles of the hills on the pressed-out material near the indentations on the (0 0 1) plane of (1) KCl, (2) NaCl, (3) LiF, (4) MgO, crystals. L = the distance from the edge of the indentation; h = the depth of the indentation; h' = the height of the hill.

to the crystallographic directions of the sample. However, for the crystals under investigation, it is shown that the plastic flow of the material may be described by using the same model for the indentor orientations $d \parallel \langle 1\,0\,0 \rangle$ and $d \parallel \langle 1\,1\,0 \rangle$ and for the different deformation temperatures, i.e. the same slip planes are active mainly for all cases [3, 25]. Therefore, the factor shown by Roberts [24] has practically no influence on the microhardness anisotropy of the crystals considered in the present paper.

5. Conclusions

1. The microhardness anisotropy is clearly revealed on the (0 0 1) cleavage plane of ionic crystals with NaCl lattice (KCl, NaCl, NaF, LiF, MgO) at the deformation temperatures 77, 293 and 800 K; $H_{[1\,0\,0]} > H_{[1\,1\,0]}$ ($H_{[1\,0\,0]}$ is the microhardness for the orientation of the indentation diagonals $d \parallel \langle 1\,0\,0 \rangle$, $H_{[1\,1\,0]}$ corresponding to the orientation $d \parallel \langle 1\,1\,0 \rangle$).

2. The coefficient of microhardness anisotropy, K, ($K = (H_{[1\,0\,0]} - H_{[1\,1\,0]}/H_{[1\,0\,0]})$ increases more or less linearly with increasing crystal microhardness in the series KCl–NaCl–NaF–LiF–MgO crystals when measurements are performed at room temperature.

3. A non-monotonic $K(H)$ dependence was revealed for deformation at low and high temperatures; a sharp increase in K with decreasing H was observed in the region of small H values.

4. The $K(H)$ dependence observed experimentally was mainly due to the distortion of the surface relief near the indentation and its change with changing temperature, and on passing from one crystal of the KCl–NaCl–NaF–LiF–MgO series to another one.

5. The results obtained in the present paper indicate the dislocation mechanism of indentation formation for the crystals investigated in the temperature interval 77 to 800 K.

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