ON THE ANISOTROPY OF THE MICROHARDNESS OF $NH_4H_2PO_4$ (ADP) SINGLE CRYSTALS

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1. Introduction

It is a well established fact that ionic crystals provide excellent experimental possibilities to investigate processes connected with plastic deformation. This applies especially to NaCl-type ionic crystals with their simple structure and easy manipulation. At the same time, however, for the purpose of investigating more complex plastic deformation mechanisms, ionic crystals of lower symmetry — as for instance $NH_4H_2PO_4$ (ADP)-type crystals — may be expected to yield valuable information. On the other hand, the investigation of these crystals is stressed by being used in a great variety of physical instruments as well as in the laser technique, consequently their mechanical stability against any type of stresses is practically important requirement. In spite of this their mechanical characteristics are little known, including even the very simple macroscopic properties of ADP-type crystals. One of these is the yield point reflecting plastic properties or the microhardness closely related to the former and easy to determine. Since plastic deformation in crystals is connected with definite glide systems, the microhardness of ADP-type crystals may be assumed to show distinct anisotropy. On this assumption microhardness tests were made on various growth faces and cuts of ADP single crystals grown from solution, in order partly to determine the load dependence of microhardness and partly to investigate the relation between the anisotropy of microhardness and the active glide systems.

2. Experimental technique

The investigated crystals were grown by the method of decreasing temperature in the temperature range of 50 °C to 28 °C.

The microhardness measurements were performed on the naturally grown faces or on polished surfaces perpendicular to the Z axis.

The experiments were carried out in a Haneman D 32 Zeiss Neophot equipment. The microhardness values were calculated by the well-known formula [1]

$$H = 1854.0 \frac{P}{d^2} \text{ kp/mm}^2 \tag{1}$$

where P is the force on the diamond indenter and d the diameter of the indentation. The load varied from 5 to 100 ponds, and the load dependence of microhardness was obtained by the statistical evaluation of 30 to 40 indentations. The error of measurements was 0.5 to 1.0%. The distortion of the shape of the indentation by the diamond pyramid on crystal faces in the various crystallographic directions was small enough to permit the use of the relationship $d = (d_{\min} \times d_{\max})^{1/2}$ for average indentation diameters [2].

3. The load dependence of the microhardness of ADP single crystals

The load dependence of the microhardness obtained in our experiments is presented in Fig. 1. Microhardness values vs. indentation diameter are seen to be of the same character on the various faces and Z-cuts of the ADP single crystals. Curve 1 corresponds to values measured on growth face (101), curve 2 to those on (100) ones, and finally, curve 3 to the results on the Z-cut. It is interesting to see the characteristic dependence of the microhardness on the indentation diameter to be independent of the orientation of the indenter with respect to any crystallographic direction, as proved by curve $\bar{2}$ plotted from values obtained on face (100) where the indenter diameter included an angle of 63° with direction [101]. In the region of variation the curves in Fig. 1 are closely approximated by Eq. (1).

Thus the force of indentation (P) and the diameter of indentation (d) of both the ADP and the alkali-halide single crystals are related by

$$P = a \cdot d^n \tag{2}$$

The constants n obtained from the curves are equal and amount to 1.81 ± 0.02. The a values, however, as well as the microhardness values deviate from each other depending upon the orientation of the crystal faces and the direction of the indenter diameter. Presumably the physical interpretation of the constants for ADP crystals is the same as for alkali-halides. Accordingly, the constant n reflects the symmetry of the lattice whereas a is proportional to the surface energy of the crystal face [3]. The correctness of the interpretation of factor n is stressed by the experimental fact that the evaluation of the microhardness values of various ADP-type single crystals (ADP, KDP . . .) has led to identical n values just as was found earlier for the alkali-halides [3]. Of course, the n values obtained for the two systems are not identical, $n_{\text{ADP}} \neq n_{\text{NaCl}}$. The proportionality of factor *a* to the surface energy could not be examined since no surface energy values of ADP crystals are available.

Fig. 1 also demonstrates that the load-dependence of the microhardness of different faces vanishes at nearly the same indentation diameters of $\approx$65 μm. To get some insight into this experimental observation the dislocation densities on various faces were determined by a suitable etching technique

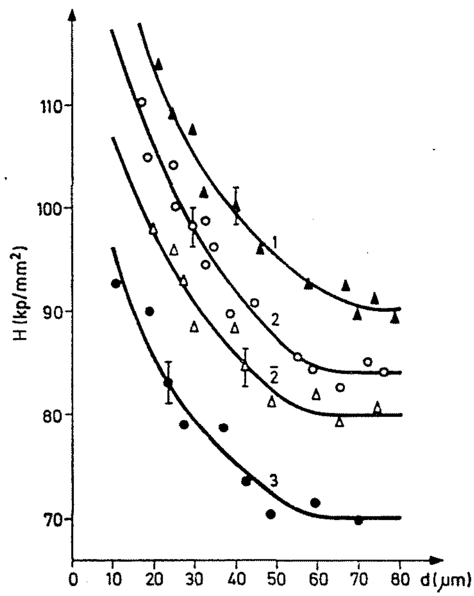

Fig. 1. The indentation-diameter dependence of the microhardness as measured on various faces. 1 — growth face (101), 2 — growth face (100), 3 — face (001) (Z-cut), $\bar{2}$ — growth face (100) using indenter rotated by 63 degrees

[4], to be approximately identical (3.10^4 cm^{-2}) throughout. The average dislocation spacing was calculated from the dislocation density to be approximately identical with the quoted critical indentation diameter ($\approx$ 65 μm). Since in these crystals the dislocations are in very strong interaction one possible explanation of the vanishing diameter — load dependence might be that with greater forces (in case of large indentation diameters) the indenter deforms material which already contains dislocations and the force necessary to this is determined by the dislocations grown into the crystal.

4. The anisotropy of the microhardness of ADP single crystals

The anisotropy of the microhardness of ADP crystals is already seen from Fig. 1, where the load curves differ depending upon the growth faces, cuts and indenter orientations.

In order to get better insight in this anisotropy, the so-called rosettes have been determined under constant load on every growth face and Z-cut.

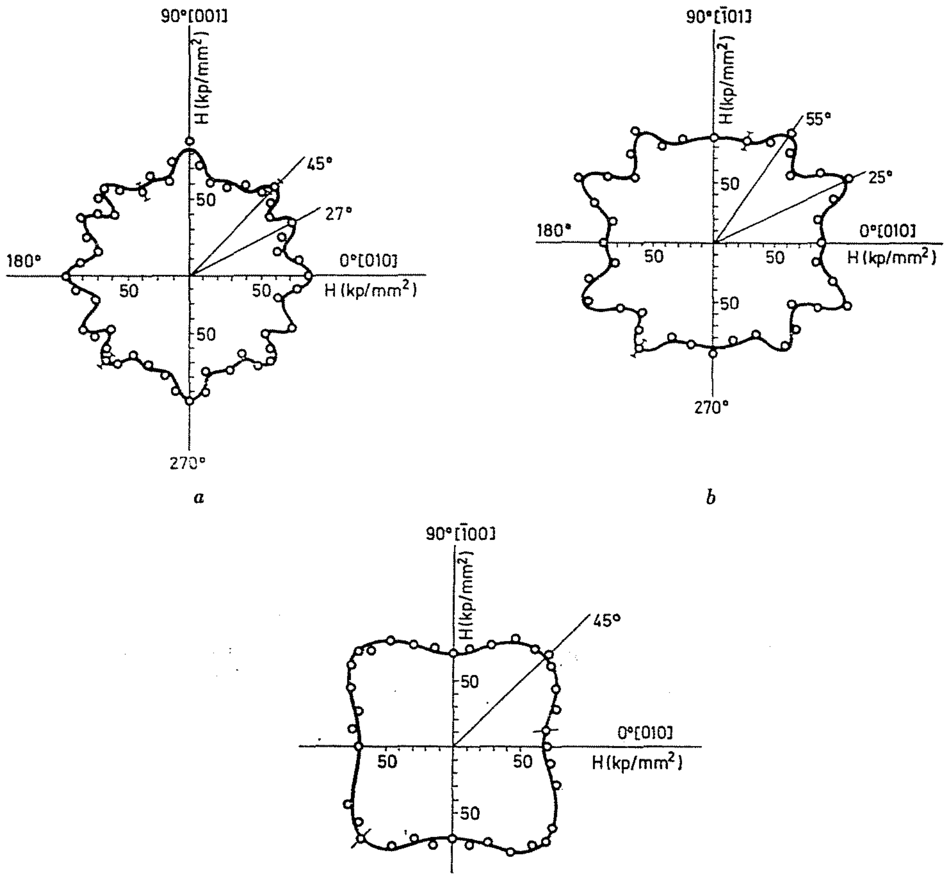

Fig. 2. Microhardness vs. angle between the indenter diameter and direction [010]. a) growth face (100); b) growth face (101); c) face (001) (Z-cut)

Local maxima are shown by centrally-symmetrical rosettes in Fig. 2a, 2b and 2c. Fig. 2a demonstrates the change of the microhardness measured on face (100) to depend upon the angle included between the indentation diameter and the direction [010]. Four, nearly identically located maxima are seen in every plane-quarter (in the first quarter the angles are: 0°, 27°, 45° and 90°). Fig. 2b demonstrates the angle-dependence of the microhardness measured on face (101) (angle included between the indenter diameter and the [010] direction). In every quarter two hardness maxima are obtained, in the first one at angles 25° and 55°. Fig. 2c shows the angle dependence of the microhardness measured on the Z-cut (angle included between the indenter diameter and the [010] direction). Here only one hardness maximum is obtained, in the first quarter, at 45°. The location (angular dependence) of the hardness maxima can be explained by knowing the glide planes (112), (011), (110) and the glide direction [111] as obtained by X-ray diffraction on ADP crystals [4]. According to some simple considerations the angles belonging to the peaks in Fig. 2a, 2b and 2c are those included by the direction [010] and the intersection lines of the glide planes and the crystal face in question. The maxima of microhardness in these directions may be attributed to minima of shear stresses generated in possible glide planes and directions by the indenter adjusted in the given directions. This is the easiest to prove for Z-cut maxima. It is also easy to see that for hardness minima the shear-stress maxima generated by the indenter are responsible.

Summary

The microhardness of the various growth planes and of the Z-cut surface of ADP type crystals was investigated. Depending upon the crystal planes, various microhardness values were obtained although the microhardness dependence on the indentation diameter was found to be of the same type. The microhardness of ADP-type crystals may be characterized with the same two parameters as in the case of the simpler ionic crystals. Some correlation between the anisotropy of microhardness and the possible glide systems was detected.

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