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Study of Slip Systems in Epidote Single Crystal

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Abstract

Measurements of microhardness anisotropy by Knoop diamond indentations on the prismatic (100) planes of epidote crystals, point group 2/m and space group P21/m, show that microhardness is determined by crystallographic slip on the (100)¡001¿ systems. Microhardness anisotropy is explained in terms of the effective resolved shear stress distribution in these slip systems.

1. Introduction

It is well known that indentation microhardness depends on the crystal plane and the crystallographic direction on that plane [1-4]. The anisotropy in microhardness arises from the crystallographic nature of the deformation process which are initiated and then proceed directly beneath the indenter and around the area of contact between the indenter and the surface. Microhardness anisotropy is a well known phenomenon, and has been reported for many rock salt / fluorite structure crystals, such as NaCl [4], LiF, $MgAl_2O_4$ [5], BaF_2 , HfC and TaC [6]. Li and Bradt [7] have investigated the Knoop microhardness anisotropy in rutile (tetragonal system crystal). Li and Jensen [8] measured Knoop microhardness on the (010) cleavage planes of Bi_2S_3 and Sb_2S_3 single crystals for orientations from the $< 001 >$ to the $< 100 >$ directions. They found that the experimental microhardness versus the azimuthal angle of the indenter was consistent with the calculated effective resolved shear stress for the (010) $< 001 >$ primary slip system.

Epidote has good hardness (7 on the Mohs scale), has a perfect {001} cleavage and possesses transparent colourless, greenish yellow or olive lustre. These feature combined with its high refractive indices (pleochroism: α = 1.715 - 1.751, β = 1.725 - 1.824 and γ = 1.734 - 1.797) would make it an attractive gemstone. The aim of this work was to study the directional microhardness anisotropy in epidote and explain it in terms of the effective resolved shear stress distribution on the slip systems. The relationship between directional microhardness and E.R.S.S. in the monoclinic crystals has not been reported in the literature.

2. Experimental

Natural Epidote ($Ca_2(Al,Fe)_3Si_3O_{12}$) single crystals were obtained from the metamorphic rocks of the Kohistan mountain range, Baluchistan province, Pakistan. (They are also found in the medium grade metamorphic rocks of Untersulzbachtal, in the Austrian Tyrol, and Prince of Wales Island, Alaska). Their chemical composition, in wt. %, is $SiO_2 = 36.92$, $Al_2O_3 = 22.27$, $Fe_2O_3 = 15.2$, MgO= trace, TiO_2 = trace, CaO = 23.11 , $H_2O = 0.1$ and MnO = 0.75.

The pinacoid planes (100) and (001) were well developed and were identified from back reflection X-ray diffraction technique.

Knoop microhardness measurements were made on (100) for test loads of 500 gf with a contact time of 15 s. A Knoop indenter was used to investigate the dependence of hardness on crystallographic directions. Its geometry is shown in Figure 1. Indentations with loads below 500 gf (4.9 N) were found to be load dependent and those with loads above it were accompanied with numerous cracks and, therefore, loads below and above 500 gf were not used. The Knoop microhardness was calculated from the length of the long diagonals of the indentations using the relation:

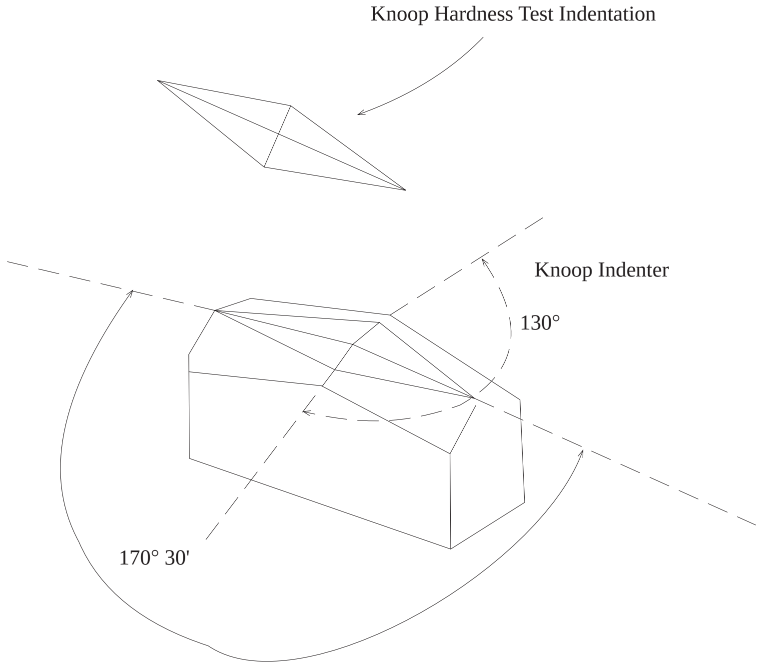

Figure 1. The geometry of the Knoop indenter.

$$H_K = \frac{P}{0.07028d^2} \qquad \mathrm{Kgf/mm^2}$$

where, P is the indentation load (in kilogram-force) and d is the length of the major axis of the indentation (in millimeters). Hardness test indentations were made on five different crystals, with the same load. The indenter was rotated with respect to the [100] direction, from 0° to 360° in steps of 30°. Standard deviation in microhardness was calculated for each orientation.

3. Results

The common forms of the crystal are shown in Figure 2. Orientation relationship of loaded indenter and the slip planes beneath the Knoop indenter is shown in Figure 3. X-ray powder diffraction pattern is shown in Figure 4. The lattice parameters were found as a= 8.8661 Å; b = 5.6006 Å; c= 10.1564 Åand β = 115°25' with point group 2/m and space group P2_1/m. These parameters are very close to those mentioned in the literature [9].

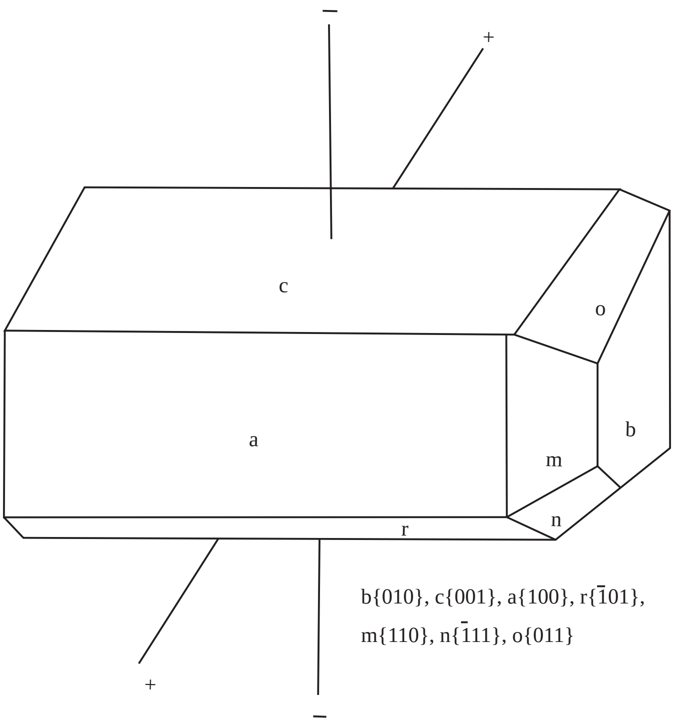

Figure 2. Typical forms in an epidote crystal.

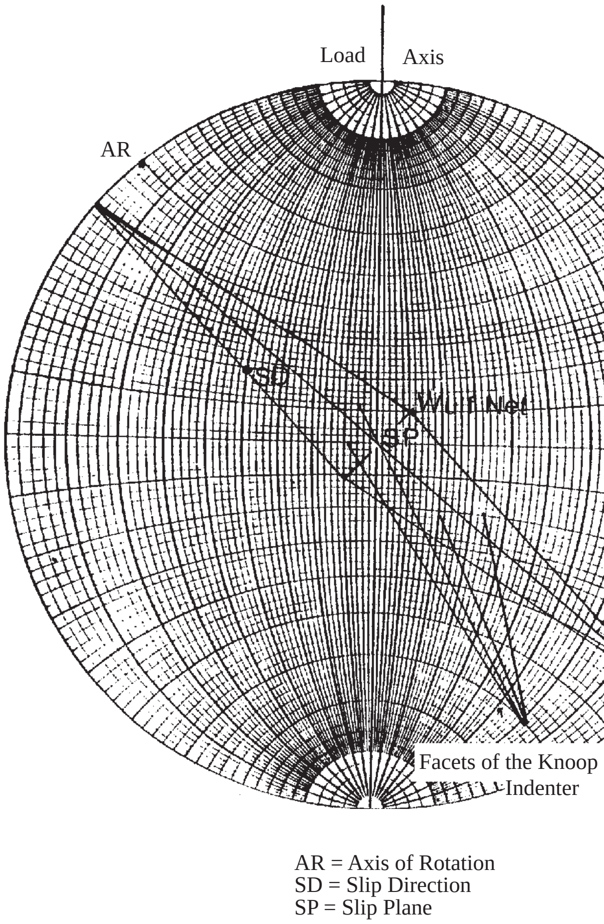

Figure 3. Orientation relationship of loaded indenter and the slip planes beneath the indenter.

The microhardness varied with the crystallographic orientation. Microhardness anisotropy $(H_{\max} - H_{\min}/H_{\max})$ of 25% has been observed on this plane, the hardness is found to be maximum in directions parallel to $< 100 >$ and minimum parallel to $< 001 >$. Figure 5.(a) shows the microhardness profile on the (100) plane.

The effective resolved shear stress, E.R.S.S., on the slip planes of a crystal correctly identifies the slip system beneath an indenter. Several investigators [e.g. 7] have successfully explained the microhardness anisotropy on the basis of Daniels and Dunn's (D.D) model [1], which relates the microhardness to an effective resolved shear stress distribution in slip systems when an indentation made by a conical indenter is considered. It is assumed that the material beneath the indenter is deformed by a tensile force parallel to the steepest slope of the individual facets of the indenter. The rotation of the element of the material close to the facets of the indenter is taken into account. A slip system which allows rotation of slip planes about an axis parallel to the indenter facets will be favoured. The effective resolved shear stress σ according to the D.D. Model:

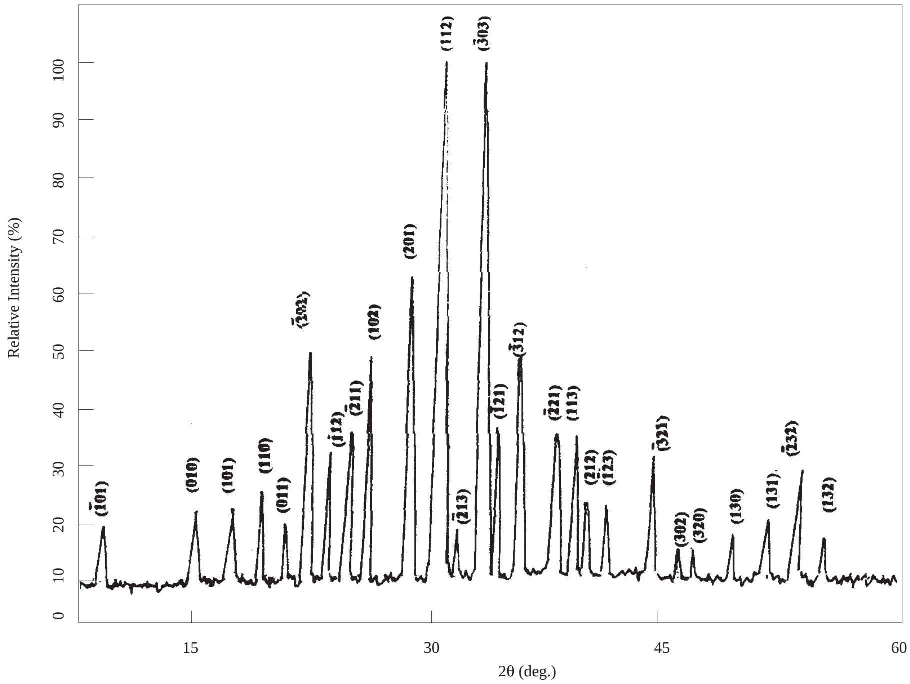

Figure 4. X-ray powder diffraction pattern of Epidote.

$$\sigma = \frac{F}{A} \cos\theta . \cos\phi . \cos\varphi,$$

where, F is the applied force, A is the area supporting F, θ is the angle between the axis of F and slip direction, ϕ is the angle between the axis of F and normal to the slip plane and ψ is angle between each facet of the indenter and axis of rotation for a given plane. The orientation relationship is demonstrated in Figure 3. The term cosy in the above equation is called the *constraint term* and is the measure of the ease with which a slip system can rotate to allow the penetration of the indenter. The maximum constraint to slip plane occurs when the angle ψ is 90°. According to Brooks [8], this term is incompletely defined. According to him, the constraint term should $be = (\cos\psi + \sin\gamma)/2$, where γ is the *modifying angle* that is the angle between the slip direction and the major axis of the indenter. The revised expression for E.R.S.S is, therefore:

$$E.R.S.S. = \frac{F}{A} \cos\theta . \cos\phi \frac{\cos\phi + \sin\gamma}{2}.$$

Slip occurs in the slip systems with the maximum E.R.S.S. and in the direction corresponding to those in which microhardness is minimum.

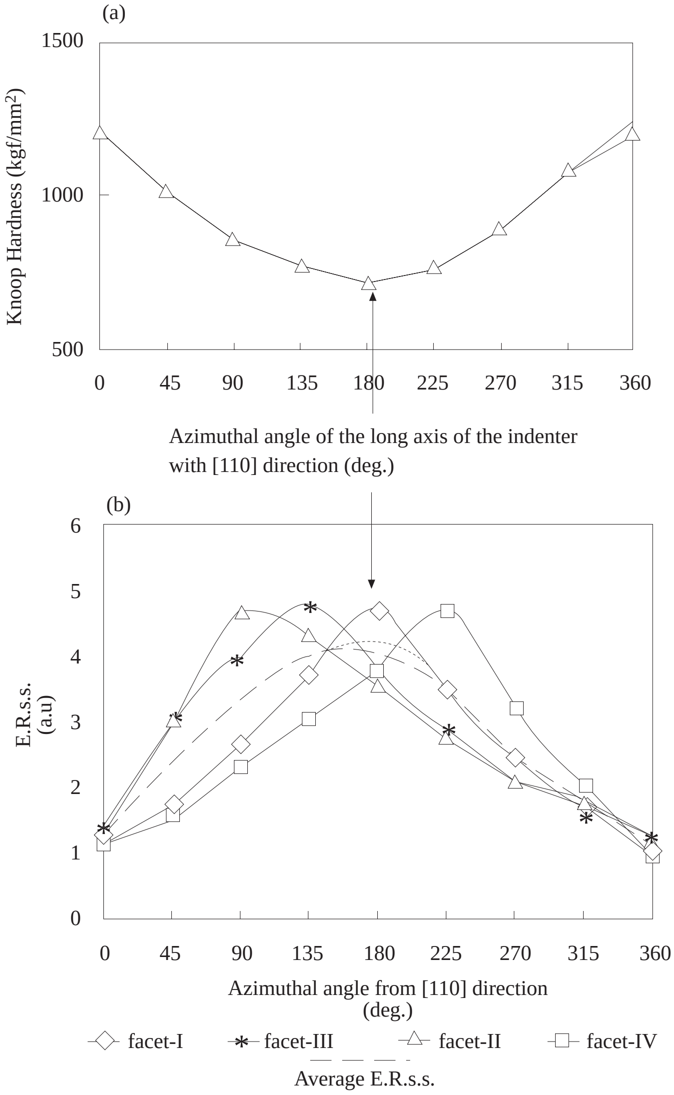

Figure 5. (a) Knoop microhardness profile on the (100) plane. (b) The E.R.S.S. plots (for each of the four facets of the Knoop indenter) for the (100) plane of the epidote crystal assuming (100)$< 001 >$ slip system.

In order to predict the primary slip system with the aid of E.R.S.S. diagram, a computer program for solving the above equation was written which generated data for the

E.R.S.S. values for each facet of the Knoop indenter. All possible slip systems were considered. Since the E.R.S.S. profiles and the microhardness are expected to be reverse of each other, it was easy to establish the operating slip system. Figure 5(b) shows E.R.S.S. curves for (100) assuming (100) < 001 > slip systems. On comparing Figure 5.(a) and Figure 5(b), we note that minima point in the E.R.S.S. plot at < 100 > directions corresponds to the maxima point in the microhardness plot. Microhardness anisotropy on other planes of the crystals is expected. Other slip systems are being investigated.

4. Conclusions

The results of this study show that the microhardness of epidote is highly anisotropic on the (100) plane. On comparing the microhardness profiles with computed E.R.S.S. plots, it is obvious that the microhardness anisotropy is controlled by the E.R.S.S. on the slip planes beneath the Knoop indenter. The slip system proposed on the basis of microhardness studies is the system (100) < 001 >.

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