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A simple instrument for relative microhardness measurement

K KISHAN RAO and D B SIRDESHMUKH
Physics Department, Kakatiya University, Warangal 506 009, India

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Abstract. The design of a simple inexpensive instrument which can be used along with a simple laboratory microscope to measure microhardness of crystals is described. The design is based on the fact that the lengths of arms of indentation dislocation rosette (IDR) are related to the hardness. By controlled indentation and subsequent etching of two similar crystals, the microhardness of one can be estimated in terms of that of the other from measurements of the arms of IDR.

Keywords. Mechanical properties; microhardness; indentation dislocation rosette.

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1. Introduction

The hardness of crystals is a property of considerable interest. It is related to the bond strength on the one hand and to the defect structure on the other. It is now accepted that hardness is a measure of the resistance offered by a crystal to the motion of dislocations.

When a crystal is indented and subsequently etched, an array of dislocation etch pits is observed. This array is termed as indentation dislocation rosette (IDR). Hopkins *et al* (1973) observed that the length of the IDR is related to the load on the indenter. Inabe *et al* (1972) observed that the relation between the load and the length of the IDR is exactly similar to that between the load and the diagonal length of the Vicker's indentation impression.

Hardness testing machines are generally expensive. This paper reports the design of a simple inexpensive instrument which facilitates measurement of relative hardness of crystals from measurements of lengths of the IDR.

2. Experimental

2.1 *Principle of the instrument*

Gilman (1975) observed that in crystals with the diamond structure and ZnS structure, the critical shear stress for glide of dislocations is related to the hardness through the relation

$$\tau = K H, \tag{1}$$

where τ is the shear stress, H is the hardness and K a constant. From a study of the IDR in silicon produced by a spherical indenter, Hu (1975) showed that

$$l = C P^{1/2} \tau^{-1/2}, \tag{2}$$

where l is the length of the arm of the IDR, P, the load applied and C a constant. Combining (1) and (2), we get

$$l = A P^{1/2} H^{-1/2}, \tag{3}$$

where A is a new constant. This equation relates the length of the IDR to the hardness. Although (1) and (2) have been proposed for silicon, similar equations should hold for other systems also and hence (3) may have general validity. Equation (3) predicts a $l^2 - P$ relation for a given crystal. Further, if two similar crystals having hardness H_1 and H_2 are indented at identical loads resulting in rosettes of lengths l_1 and l_2, (3) leads to

$$H_2 = (l_1/l_2)^2 H_1. \tag{4}$$

Thus, if H_1 is known, H_2 can be estimated. This is the principle of the design of the instrument.

2.2 *Design of the instrument*

Figure 1(b) shows the line diagram of the instrument. It consists of two pillars A and B. A brass bar F is attached to the pillar A with ball bearings such that it is free to move about it in a vertical plane. D is the sample holder fixed to the pillar B; it can be raised or lowered using a rack and pinion arrangement O. A fine steel pin S is fixed at one end of the bar F, which carries a screw E at its other end with which the bar is balanced. When a load P is applied on the pin, the bar is locked in the horizontal position using a screw G. The crystal X placed on the sample holder is raised till the crystal surface is close to the tip of the pin. Indentation is made by slowly releasing the locking screw till the pin touches the surface of the crystal. An electrical arrangement C, results in the switching off of a bulb when there is a contact between the pin and the crystal. All indentations are made for 5 sec. After indentation, the crystals are etched with suitable etchants and the crystal surface is observed under a microscope. The lengths of the rosette arms are measured with a micrometer eye piece.

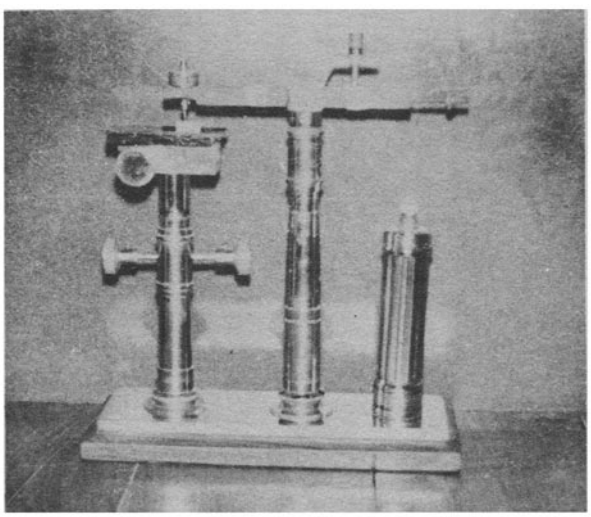

Figure 1(a). Photograph of the fabricated instrument.

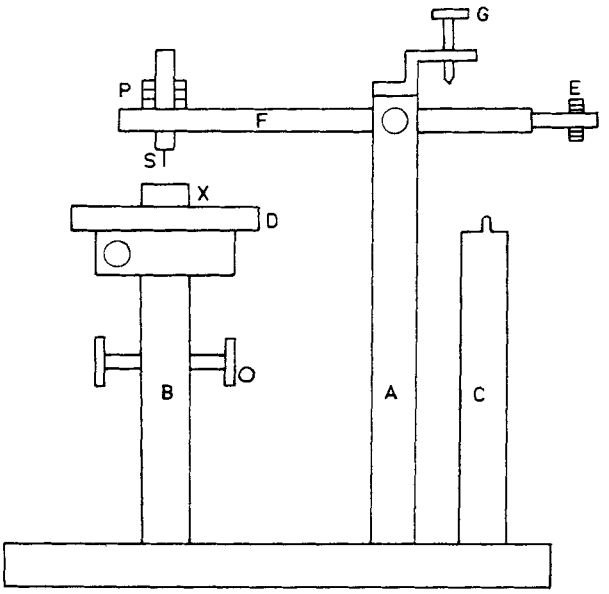

Figure 1(b). Line diagram of the indenter.

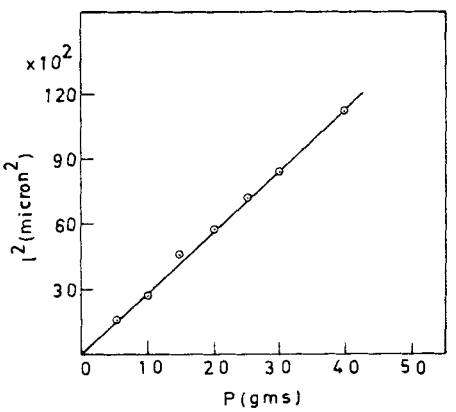

Figure 2. Plot of square of rosette length (l^2) against load (P).

3. Results and discussion

Using the instrument, indentations have been made on freshly cleaved samples of pure NaCl, doped NaCl, KCl, KBr and LiF. The crystals have been etched with known etchants (Raju *et al* 1972; Gilman *et al* 1958). The l^2 values are plotted against the load P for pure NaCl (figure 2). A linear relationship is seen as predicted by (3). Hu (1975) observed a similar relationship in silicon indented with a spherical indenter. The present results show that the relationship is independent of the shape of the indenter or the crystal system.

Figure 3 (a, b) show the IDR pattern for pure NaCl and NaCl doped with $SrCl_2$. The increase in hardness on doping is clearly reflected in the reduced size of the IDR.

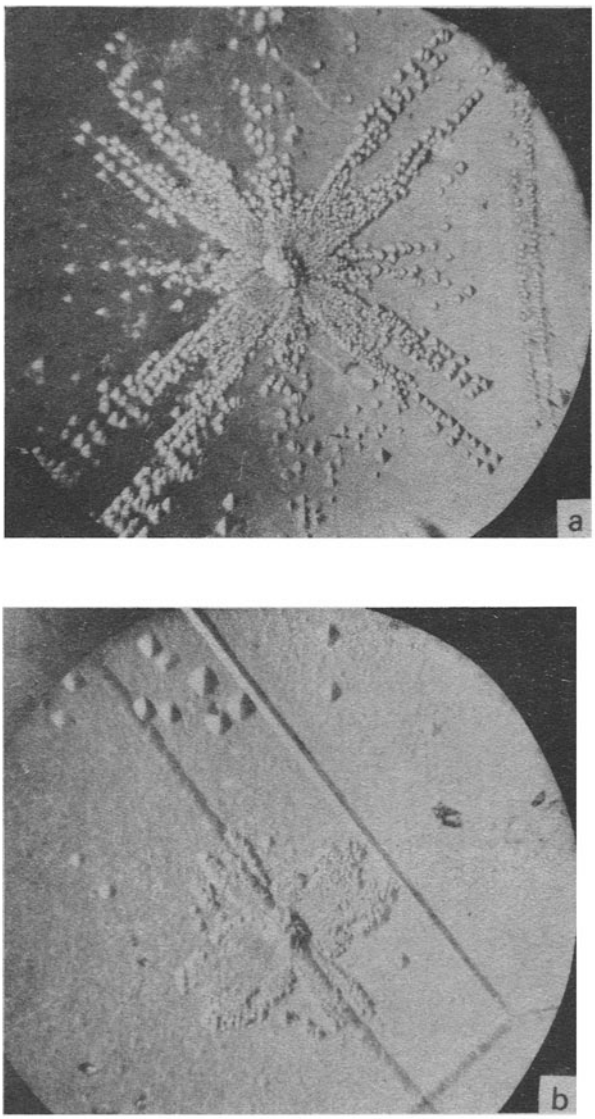

Figure 3. (× 300) Indentation dislocation rosette pattern on (**a**). Pure NaCl (**b**). Sr-doped NaCl.

Using the instrument and the method described, the microhardness of the crystals was estimated in terms of the hardness of pure NaCl for which a value of 22·1 kg/mm^2 (Berzina *et al* 1965) was used. The results are given in table 1. The values obtained agree well with those obtained by others using sophisticated instruments; the gradation in the hardness values of LiF, KCl and KBr is the same in the data obtained in the present work and in data from literature. The hardness values of Sr-doped NaCl measured with the present instrument and a Vicker's instrument agree closely. The instrument has been tested, for the present, for the alkali halides for hardness values up to 100 kg/mm^2.

Table 1. Relative hardness values (kg/mm^2).

Crystal	Hardness	
	Present method	Other values
LiF	99·8	103[a]
KCl	8·4	10·2[b], 13·1[a]
KBr	7·6	7·4[c], 8·7[d], 10[a]
NaCl doped with $SrCl_2$	36·6	35·1[e]

[a] Berzina *et al* (1965), [b] Rao and Babu (1978), [c] Chin *et al* (1972), [d] Sirdeshmukh and Shah (1965), [e] Direct measurement with Vicker's hardness tester for the same sample.

Efforts are on hand to extend its use to crystals with other structures and to crystals with higher hardness values.

For accurate results, the indentation of the standard crystal (NaCl) and the sample crystals has to be made under identical loading conditions. Care has also to be taken to see that the indenter needle is exactly normal to the crystal surface at the position of contact. Otherwise, the rosette develops asymmetry and consequently the results will be in error. If these precautions are taken, the accuracy of the results depends on the accuracy of measurement of rosette arms and the value of the hardness of the standard. Keeping these factors in view and the scatter existing in the hardness values of NaCl, the errors in the hardness values now obtained are estimated to be $\pm 5\%$.

4. Conclusions

The results obtained with this instrument compare well with those obtained with commercial, sophisticated hardness testing machines. This method is limited to crystals which are isomorphous and for which etchants are well established. The method should prove useful for measurement of changes in hardness of a crystal, due to any treatment given to the crystal. Some possible areas of applications are: (i) temperature variation of hardness, (ii) radiation hardening, (iii) impurity hardening and (iv) annealing.

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