PART -III

Hardness of d-AHT crystals

General Information on hardness

Hardness of d-AHT crystals

i) Application of Meyer's law/ Kick's law and modified Kick's law

ii) Hardness of d-AHT crystals

iii) Hardness Anisotropy of d-AHT

CHAPTER 6

Hardness (General)

HARDNESS (GENERAL)

6.0 INTRODUCTION

Hardness has long been the subject of discussion among engineers, physicists, metallurgists and mineralogists and there are all sorts of conceptions as to what constitutes hardness. The overwhelming difficulty of defining hardness is that it is not a fundamental property of the material. There is hardness as measured by resistance to cutting, by scratching, by penetration by electrical and magnetic properties. All hardness tests measure some combination of various material properties namely elastic modulus, yield stress (which denotes the onset of plastic behaviour or permanent distortion), physical imperfection, impurities and workhardening capacity. Since each hardness test measures a different combination of these properties, hardness itself is not an absolute quantity and to be meaningful, any statement of hardness of a body must include the method used for measurement.

6.1 DEFINITIONS & MEASUREMENTS:

Attempts towards a physical definition of hardness were made by Friedrich (1) Goldschmidt (2) and Chatterjee (3).

The general definition of indentation hardness which is related to the various forms of the indenters is the ratio of load applied to the surface area of the indentation. Meyer (4) defined it as the ratio of load to the projected area of the indentation on the surface under consideration, giving hardness the dimension of stress. In total disagreement Spaeth (5) suggested hardness to be defined as the resistance to indentation in the form of the ratio of the specific surface load to the unrecovered deformation.

Matkin & Caffyn (6) from their studies on hardness of sodium chloride single crystals containing divalent impurities, correlated hardness with the dislocation theory. They redefined hardness in terms of generation and/or movement of dislocations associated with indentations. It is the measure of the rate at which the dislocations dissipate energy when moving through a crystal lattice. It is now realised that (Westbrook and Conrad (7)) hardness is not a single property but rather a whole complex of mechanical properties and at the same time a measure of the intrinsic bonding of the material.

There are basically four methods to determine the hardness of materials. They are as follows:

1) Scratch hardness test;
2) Abrasive method;
3) Dynamic method and
4) Static indentation method.

Several books and review articles are available in which the information on hardness is partly or fully described (8-30).

Static indentation method is the most widely used method and is of importance to the author.

This is a simple and very sensitive method in which a hard indenter (e.g. diamond) is applied slowly and after a certain time of application, carefully removed, leaving behind a permanent indentation mark on the surface of the specimen. Measurement is made either of the size of the indentation resulting from a fixed load on the indenter or the load necessary to force the indenter down to predetermined depth and the hardness of the material is then defined as the ratio of the load to the area of the indentation mark. The hardness values so obtained vary with the indenter geometry and with the method of calculations.

Many combinations of indenter, load, loading procedure and means of indentation measurement are used among the various tests in order to accommodate various shapes, sizes and hardness of specimens, and this has resulted in a proliferation of hardness scales. The most commonly used indenters are described in Table 6.1.

Diamond indenters must be used for hard materials in order to minimise errors due to elastic distortion of the indenter. In case ball indenters are used, the hardness number will be independent of load only when the ratio of load to indenter diameter is held constant. For cone and pyramidal indenters, hardness number will be independent of load for all loads above a certain minimum value depending upon the specimen material.

6.2 GENERAL INFORMATION ON HARDNESS:

The hardness study undertaken so far for studying the strength of solids and the effect of various treatments on the hardness of a solid, have proved somewhat useful. It improved not only the view of materials research but helped in understanding various other mechanical properties of solids. Gilman and Roberts (31) correlated indentation hardness with the elastic modulus by gathering the data for various materials. Their empirical linear relation shows that elastic modulus is an important factor which determines plastic resistivity against the dislocation motion. The behaviour of the indented region during the propagation of stresses which intiate dislocations and their motion is not yet fully understood. When an indenter is pressed on the surface of a solid, the stresses are not simply tensile or compressive in nature. Stresses in various directions are set-up and one should treat the resultant plastic flow as a result of these combined stresses. It is also observed that the fundamental mechanisms of deformation can be either slip or twin or at times fracture.

i) Slip is the most common mode of plastic deformation, which is characterised by the displacement of one part of the crystal relative to another along certain definite crystallographic planes. The slip planes are usually of low indices and the slip directions are those of closely packed ones in a crystal structure.

Smakula and Klein (32) from their punching experiments on sodium chloride explained the crack formation on the basis of shear on slip planes. Gilman (33) attributed these microcracks which have a definite crystallographic direction to the piling up of dislocation on the slip plane.

In crystalline materials plastic deformation or slip occurs through the movement of line imperfections called dislocations. As dislocations are multiplied (by one of the several mechanisms) during deformation, their spacing decreases and they interact and impede each others motion, thus leading to workhardening. The strength of dislocation interference depends on the nature of the crystal and on the ratio of temperature of deformation to the melting point of the crystal.

ii) Certain crystals may also deform by twinning, a mechanism by means of which a portion of a crystal may change lattice orientation with respect to the other in a definite symmetrical fashion. Schmidt and Boas (34) described the twinning as the simple gliding of one plane of atoms over the next, the extent of the movement of each plane being proportional to its distance from the twinning plane. Partridge (35) studied the microhardness anisotropy of magnesium and zinc crystals. He observed twin in the above crystals and concluded that the resolved shear stress criterion is insufficient to account for the observed distribution of twins and that the dimensional changes which occur during twin deformation must also be taken into account. Indenting diamond flats with diamond indenter Phall

(36) reported the slip and twinning of diamonds. Vahldick (37) studied the slip system and twinning in molybdenum carbide single crystals with the help of knoop and vickers indenters. When the indented crystal was etched by a dislocation etchant, rosettes were formed on some crystals (usually alkali halides) indicating the dislocation distribution around an indentation. Dislocation loops are also formed around the indentation mark in cesium iodide and sodium chloride (38, 39).

The interferometric studies of indented surfaces have revealed the nature of deformation and the history of the sample under test. Votava et al (40) were the first to study the deformed region on the cleavage faces of mica and sodium chloride. Tolansky and Nickols (41) studied the indented surfaces of steel, tin and bismuth. They observed maximum distortion along the medians bisecting the sides of the square and minimum along diagonals, showing thereby that no distortion projects beyond the diagonal.

In general, hardening of crystals can be accomplished by introduction of any barrier to dislocation motion. This can occur by :

a) workhardening b) impurity hardening (impurities tend to segregate dislocations and pin them), c) decreasing grain size in a polycrystal (grain boundaries are barriers to dislocation motion), d) dispersion of fine particles of second phase in the crystal and e) phase transformations (by quenching).

It can be seen from this brief review that the amount of plastic deformation induced in a material by an indenter under load depends in a complicated way on a variety of factors which defy simple analysis.

6.3 VARIATION OF HARDNESS WITH LOAD:

For geometrically similar shapes of the indent marks for all loads, it can be shown that hardness is independent of load (16). However, this is experimentally incorrect for certain ranges of applied loads. It is clear that during a hardness test the formation of indentation mark leads to an increase in the effective hardness of the material and so the hardness number obtained is not the actual hardness of the material in the initial state. This is mainly due to workhardening of the substance during the process of indentation which will be varying with the load. Attempts have been made to determine the absolute hardness by eliminating workhardening. This can be done only if the method does not appreciably deform the substance plastically. Absolute hardness was found to be one third of the normal hardness by Harrise (42).

A large number of workers have studied the variation of hardness with load and the results given are quite confusing. Knoop et al (43), Bernhardt (44), observed an increase in hardness with the decrease in load whereas Campbell et al (45) Mott (13) observed a decrease in hardness with decrease in load. Some authors e.g. Taylor (46), Bergsman (47) reported no significant change of hardness with load. In view of these different observations it has become rather difficult to establish any definite relationship of general validity between microhardness values and applied load.

There are two ways of studying the relation between hardness (H) and applied load (P) or relation between load and diagonal (d) of the indentation mark. An empirical formula given by the equation,

$$P = a\,d^{n} \quad \text{.....................(6.1)}$$

where "a" and "n" are constants of the material under test.

The general definition of hardness number is

$$H_i = rP/d^2 \quad \text{.....................(6.2)}$$

where "r" is a constant and depends upon the geometry of the indenter and "i" indicates the indenter e.g. i=v for Vickers hardness number. The combinations of the above equations yield,

$$H = a_1 d^{n-2} \quad \text{.....................(6.3)}$$

$$H = a_2 P^{(n-2)/n} \quad \text{.................(6.4)}$$

Where $a_1 = ra$ ....(6.5)

$$a_2 = ra^{2/n} \quad \text{.............(6.6)}$$

It has been shown that in case of Vickers hardness number, the value of the exponent "n" is equal to 2 for all indenters that give geometrically similar impressions. This implies a constant hardness value for all loads.

Hanemann and Schulz (48) from their observations, concluded that in the low load region "n" generally has a value less than two. Onitsch (49) found such low values of n (1 to 2) by observing variation of hardness with load while Grodzinski (50) found variation of n values from 1.3 to 4.9. Due to this variation in the results, a high load region was selected which led to the definition of microhardness independent of applied load. The hardness values so obtained for this region again showed scattered results even though the apparatus had good mechanical precision. The scattered observations may be attributed to the following reasons:

1) Equation i.e. $P = ad^n$ is not completely valid.
2) Microstructures exercise a considerable influence on measurements involving very small indentations.

3) The experimental errors due to mechanical polishing, preparation of specimen, vibrations, loading rate, non-coincidence of microscopic axis and applied load direction, shape of indenter, measurement of impression, etc. affect the hardness measurements considerably.

The term connected with the above test, microhardness means microindentation hardness, as it actually refers to the hardness measurement on the microscopic scale. Instead of the above term, some authors use low-load hardness. Three possible regions can be defined:

1. Microhardness: from lowest possible loads upto maximum of 200 grms.

2. Low load hardness : Loads from 200 grms to 3 kg. The most characteristic region comprises of loads from 200 grms to 1 kg.

3. Standard hardness: Loads over 3 kgs.

Since the present study is made in the region of microhardness as defined in (1) above, the following presents a brief review of the work reported on microhardness of various crystals.

Many workers (from 1960 onwards) reported an increase of hardness with load at low loads, attaining a maximum value at a certain load, decreasing and remaining constant for a range of higher loads. Murphy (51) explained the variation in hardness in copper crystals, due to the escape of primary edge dislocations.

Sugita (52) found ring cracks and radial cracks in Germanium crystal and the load required to produce them increased with temperature.

The formation of twins in Bi,Sb, Bi-Sb, Bi-Sn and Bi-Pb were studied by Koserich and Bashmakou (53). They showed that the length (l) of twins was proportional to the diagonal (d) of the indentation and the intensity of twinning is given by the coefficient "a" in the equation:

$$l = a + \alpha d$$

The value of 'α' was more for homogeneous alloys and increased with Sb content and remained constant for higher concentration of Sn and Pb.

Shah and Mathai (54) explained hardness in terms of slip taking place due to deformation in the crystal (Tellurium). Edelman (55) showed that microhardness of InSb and GaSb single crystals decreased exponentially with temperature. The presence of deflection points on the curves at 0.45 - 0.50 Tm indicate the deformation by slip.

The hardness decreased with decrease in carbon content in titanium carbide was confirmed by Samsonov et al (56).

Milvidski et al (57) observed decrease in hardness with increase in concentration of impurity and dislocation density in silicon single crystal. Kuzmenko et al (58) showed decrease in hardness due to change in mobility of dislocations as a result of excitation of electrons during lighting and their transition to higher energetic zone in titanium iodide and termed this a photochemical effect. Beillin and Vekilov (59) observed a decrease in the hardness upto 60% illumination in Ge and Bi. Decrease in hardness was attributed to the induced photoconductivity, which altered the widths of the dislocation cores at the sample surface and in turn altered the plasticity.

Westbrook and Gilman (60) studied electrochemical effect in a number of semiconductors. They observed a decrease in resistance of semiconducting crystals to mechanical indentations in the presence of a small electric potential (0.05 to 10 v) between the indenter and crystal surface.

The distribution of dislocations around an indentation mark was studied using chemical etch pit technique by Urusovskaya and

Tyagaradzhan (61). They found larger number of prismatic loops. Shukla and Murthy (62) found an increase in the distance travelled by leading dislocations with increase in load in NaCl single crystals.

The effect of impurity on hardness was also studied by various workers. Dryden et al (63) studied the hardness of alkali halides when low concentration of divalent cations are incorporated in the crystal lattice on the basis of dielectric measurement of doped alkali halide crystals.

Temperature dependence of microhardness was performed by Sarkozi and Vannay (64). They concluded that besides thermal stress the observed hardening may be due to dislocations piled up at various impurities, to complexes in solid solution and vacancy clusters which were developed at high temperature. By quenching the clusters become distributed in the crystals as fine dispersions.

Temperature dependence of microhardness was also studied by Shah (65) who found that hardness of calcite cleavage faces increases with the temperature. Acharya (66) found that the hardness of Zn and KBr decreases with the quenching temperature while the hardness of TGS increases with quenching temperature.

An analysis of Knoop microhardness led Hays and Kendall (67) to modify Meyer's (68) law correlating applied load to the long knoop diagonal by the term which accounted for the resistance offered by the test specimens. Results were also discussed for usage of modified Meyer's law to obtain knoop hardness numbers independent of applied load. Comparative study of knoop and Vickers hardness numbers was also reported by Tietz and Troger (69).

6.4 HARDNESS ANISOTROPY:

If, the material under the hardness test has planar anisotropy that is, the strength varies along different directions in a given plane, symmetrical indenters like the pyramidal and spherical indenters cannot be used to distinguish such property variations. One exception is the knoop indenter: this was extensively used by Joshi (70) to study the hardness anisotropy of $NaNO_3$ and $CaCO_3$ cleavages. It was also used by the author to study the hardness anisotropy of d-AHT single crystals.

Knoop and his associates originally developed the four-sided pyramid indenter for determining the hardness of semibrittle materials (71). It was the unique geometry that offered several advantages over the conventional and symmetric indenters. For this reason, the Knoop hardness testing method has been extensively examined and put to use in different applications since its development in 1939. One of the features is that, because of its shallow depth of penetration, brittle materials like glass or minerals could be indented without causing premature fracture. Another feature is that, due to the nonsymmetric indenter shape, the variations in hardness along different directions in a given surface can be determined.

The geometry of the indenter is shown in Fig.6.1, where the included conical angles extending along the major and minor axes of the indenter are $172^{\circ}30'$ and 130° respectively. It is generally assumed that there is negligible elastic recovery in the major diagonal direction compared to the minor diagonal direction when the indenter is removed (72). Based on this assumption the Knoop hardness number (KHN) is given as the indenter load divided by the indentation area projected on the original undisturbed surface in terms of the length of the major diagonal and in units of Kg/sqmm.

In addition to the problems associated with elastic recovery, the material near the indenter surface is known to pile up or sink in, depending on the interfacial frictional conditions and the material properties, such as the strain-hardening capacity. This phenomenon also tends to change the mode of deformation near the indenter surface and therefore the hardness of the material.

6.5 ORIENTATION DEPENDENCE OF HARDNESS:

An important feature of the Knoop hardness test is that the hardness value is dependent on the orientation of the major axis of the indenter in a given plane, as well as on the orientation of the plane itself with respect to the principal axis of anisotropy (3,6,73,74,75). Single crystals therefore can serve as ideal materials to establish the orientation dependence of hardness values.

The degree of hardness anisotropy was found to be increasing with increase in temperature on (001) faces of n-type, p-type and intrinsic Ge and GaAs (76) and decreasing as temperature increases for Bi and $LiNbO_3$ (77).

Comparisons were made between the scratch hardness (Hs) as determined by the width of a groove formed under conditions of plastic deformation and indentation hardness (H) of crystalline materials (78) and it was concluded that anisotropy measurements allow the slip systems of a given crystal to be identified.

Hardness anisotropy was studied by many workers (79)(80) and Knoop microhardness anisotropy was related to the slip systems as they determined the corresponding effective resolved shear stress (86).

The effect of polishing (82), radiation and magnetic fields (83) doping of crystals (84) on anisotropy was studied, and the anisotropy was explained to be a result of structural changes and internal stress redistribution in commercially pure beryllium induced by thermoelastic stresses due to thermocycling (85). It was shown that in addition to the expected anisotropy in the continuous material, there was significant anisotropy in the carbon fibre reinforced injection moulded thermoplastic (86).

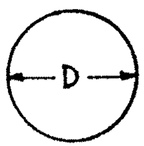
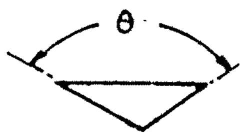
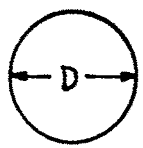
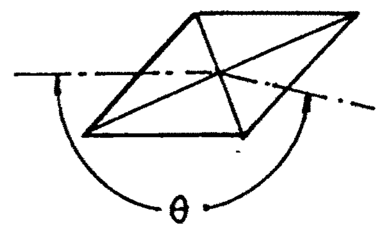
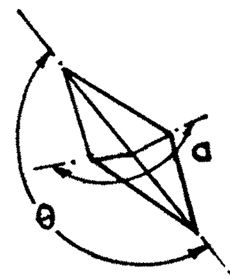
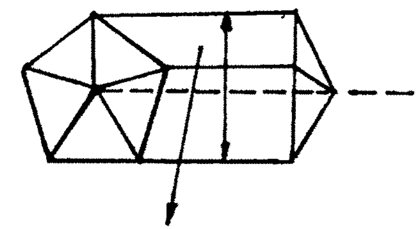
Microindentation studies were performed on $CuInSe_2$ (87), superconducting material Y BaCuO (88), rubidium hydrogen tartrate (89), mercuric iodide (90), $Ba_{1-x}K_xBiO_3$ (91), NaCl (92) and BaFCl (93).

Vickers indenter was used by many workers in the recent years, (94),(95),(96), and a relation was obtained between Vickers hardness number and universal hardness from a specimen's elastic, characteristics (97).

It was interesting to note that microhardness decreased with increase of load and the radial crack lengths were used to calculate the fracture toughness and brittleness index (98).

The above represents a brief work done on hardness of various crystals. The present work is centered on the study of variation of load with diagonal length of indentation mark, variation of directional hardness with applied load on as-grown and cleavage faces of gel grown ammonium hydrogen d-tartrate single crystals by using Knoop pyramidal indenter.

TABLE 6.1

	Brinell	Rockwell		Vickers	Knoop	Brookes & Moxley
Material of which indenter is made	Hardened steel or tungsten	Diamond	Hardened steel	Diamond	Diamond	Diamond
Shape of indenter	Sphere	Cone	Sphere	Square based pyramid	Rhomb based pyramid	Pentagonal
Dimensions of indenter	 $D=10$ mm	 $\theta=120°$	 1/16 in. 1/8 in. 1/4 in. 1/2 in.	 $\theta=136°$	 $a=130°$ $\theta= 172°30'$	 Zero azimuth
Characteristics	1. Geometrically similar impressions are not obtained	1. Prepares the surface upon which the further penetration due to major load is based. 2. Hardness is read directly on the dial gauge. 3. Hardness value may be appreciable in error due to large amount of recovery along depth.		1. Geometrically similar impressions are obtained.	1. Hardness of upper most surface layers can be found. 2. Sensitive to anisotropy of crystals 3. Shorter diagonal undergoes recovery	1. Eliminates the anisotropy normally observed in hardness with other indenters.

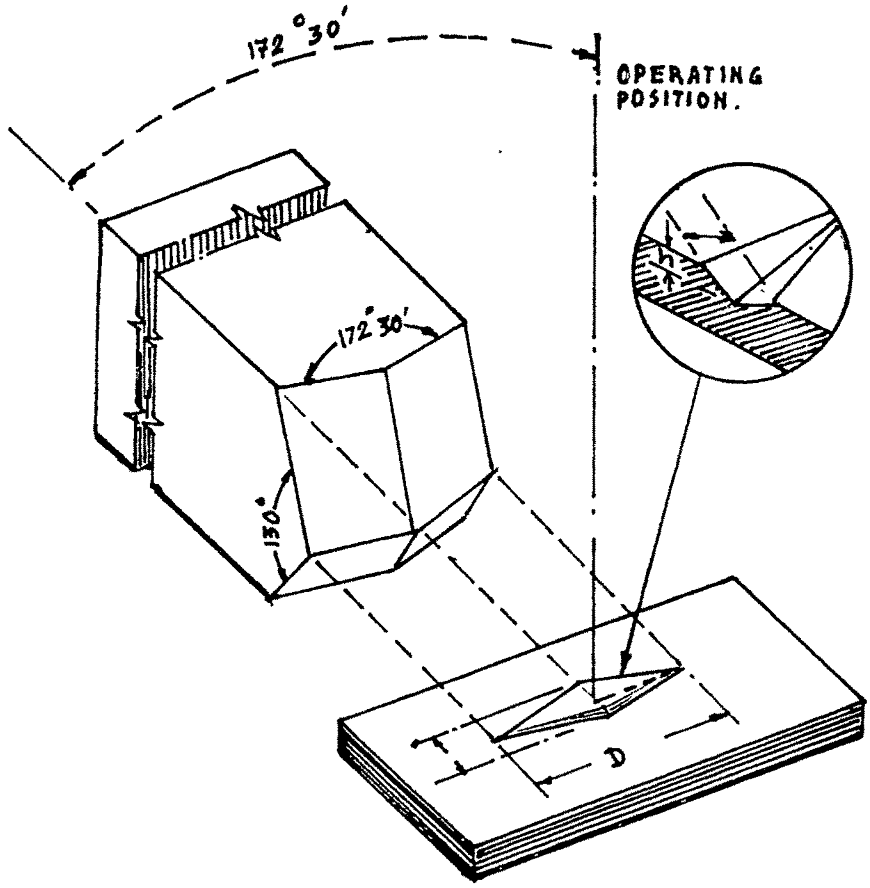


Fig. 6.1 a

Fig. 6.1 a	*Some of the details of the Knoop indenter, together with its impression*
Fig. 6.1 b	*Schematic diagram of the Knoop indenter and cylinder of deformation showing positions of force (F), slip direction (SD), slip plane (SP) and axes of rotation (AR and H)*

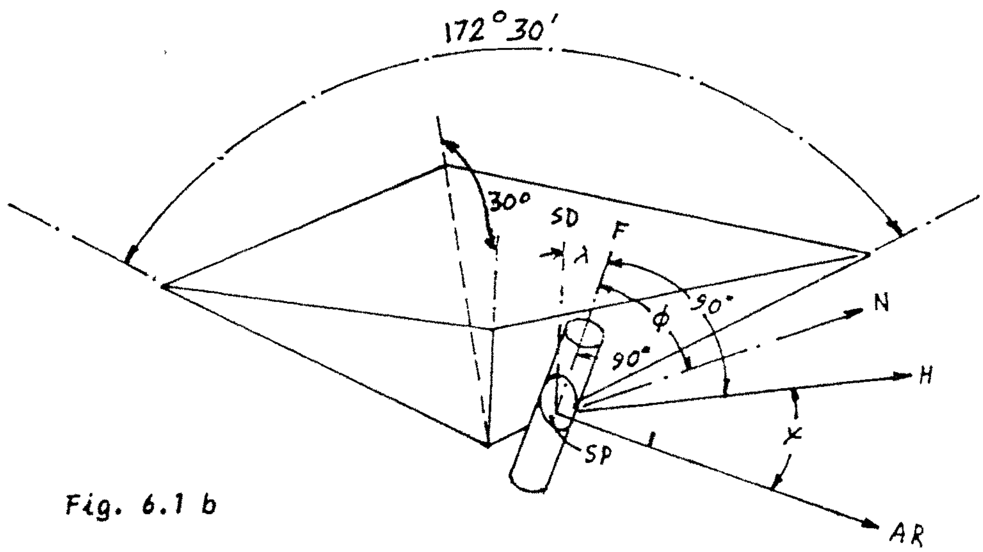


Fig. 6.1 b

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CAPTIONS TO FIGURES.

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