

## PART - II

MICROHARDNESS OF CRYSTALS (GENERAL)

VARIATION OF LOAD WITH INDENTATION DIMENSIONS

[NaCl, KCl AND KBr]

QUENCH HARDNESS OF ALKALI HALIDES

[NaCl, KCl AND KBr]

HARDNESS ANISOTROPY OF ALKALI HALIDES

[NaCl, KCl AND KBr]

CHEMICAL ETCHING OF ALKALI HALIDES

[NaCl, KCl AND KBr]

CONCLUSIONS AND FUTURE PLAN OF WORK

## CHAPTER - 3

### MICROHARDNESS OF CRYSTALS (GENERAL)

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### 3.1 INTRODUCTION:

Although hardness is one of the most common properties of the materials, it is usually difficult to describe it in a simple way. The reason being that the hardness is greatly influenced by related properties of the material which can contribute to or detract from the basic hardness. It may be broadly defined as the ability of one body to resist penetration by another. It is by definition a relative property of a material and depends on the elastic and plastic properties of both the penetrated body and the penetrator. Hardness, as measured by resistance to abrasion, is also a measure of the wearing quality of a material. When hardness is measured by resistance to cutting, an indication of the machinability quality of a material is obtained. All hardness tests measure some combination of various mechanical properties namely elastic modulus, yield stress (which denotes the onset of plastic behaviour or permanent distortion), physical imperfections, impurities and work hardening 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.

### 3.2 DEFINITIONS AND MEASUREMENTS :

Probably the best general definition had been suggested by Ashby /1/, 'Hardness is a measure of resistance to permanent deformation or damage'. A more

positive definition would be, 'hardness is a combined measure of many complex properties the most direct of which is the resistance of the material to slip or plastic flow. Attempts towards a physical definition of hardness were made by 'Goldschmidt /2/, Chatterjee /3/ and Friedrich /4/. 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. According to Meyer /5/ indentation hardness is the ratio of the load to the projected area of the indentation on the surface under consideration, giving the dimension of stress. Contrary to this, Spaeth /6/ proposed that hardness should not be defined as stress but as the resistance to indentation in the form of the ratio of the specific surface load to the unrecovered deformation.

Chatterjee defined indentation hardness as the work done per unit volume of the indentation in a static indentation test for a definite angle of indentation. On the basis of this definition and Meyer's law  $P = ad^n$  for spherical indenters, he derived a formula for measurement of hardness. According to Plendl and Gielisse /7/ hardness can be defined as pressure or force per square centimeter of indented surface and thus it can be conceived as an energy per unit volume, e.g. the ratio between the input energy and volume of indentation. They have concluded that resistance is a function of the lattice energy per unit volume and called it volumetric lattice energy ( $U/V$ ) having the dimension ergs/c.c.  $U$  is the total cohesive energy of the lattice per mole and  $V$  is the molecular volume defined as  $M/S$  where 'M' is the molecular weight and 'S' is specific heat. The hardness

was thus considered to be the absolute overall hardness. Matkin and Caffyn /8/ 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 indentation. 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 Conral /9/) 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 hardness of materials. They are as follows :

- I. Scratch hardness tester,
- II. Abrasive method,
- III. Dynamic method, and
- IV. Static indentation method.

Several books and review articles are available in which the information on hardness is partly or fully described /10-32/. They are briefly described here.

#### **I. Scratch hardness :**

An early method of measuring scratch hardness, still in wide use today by mineralogists, was developed by Friedrich Mohs in 1822. This gives a relative ranking of minerals based simply on their ability to scratch one another. The Mohs method is not suitable for a general use with materials of hardness greater than 4. Since in

this range the intervals are rather closely and unevenly spaced. The modifications of this method were overshadowed by other sensitive methods and experiments.

## II Abrasive hardness :

Abrasive hardness is defined as the resistance to mechanical wear, a measure of which is the amount of material removed from the surface under specific condition. The hardness may be found by the depth of penetration.

## III Dynamic hardness :

The hardness measurement in this method involves the dynamic deformation of specimen under study and is determined by following considerations :

- (a) Here, a steel sphere or a diamond-tipped hammer is dropped from a given height. The ball or hammer rebounds. The height to which it is rebound is read on a scale. This was taken to be the measure of hardness. The kinetic energy of a ball or hammer is used up partly in plastically deforming the specimen surface by creating a slight impression and partly in rebound. This test is sometimes referred to as 'dynamic rebound test'.
- (b) Here, a steel sphere or a diamond-tipped hammer is dropped from a given height, the depth and size of the impression produced and the energy of impact are determined. The ratio of energy of impact to the volume of the indentation mark gives a measure of the hardness.
- (c) Chalmers /33/ assessed the surface hardness in terms of

the reduction in optical reflectivity when a known amount of sand was allowed to impinge on the surface under standard conditions.

#### **(IV) Static indentation hardness :**

The most widely used method of hardness testing is the static indentation method. This is the simplest 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 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 3.1. Diamond indenters must be used for hard materials in order to minimize 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 specimen material. The term connected with static indentation test is microhardness or microindentation hardness as it actually refers to the hardness measurement on the microscopic level. Instead of the above term some authors use low load hardness, leading to the confusion due to incomplete demarcation of ranges of applied loads. Three possible regions can be defined.

- (1) Microhardness : from lowest possible up to maximum of 200 gm.
- (2) Low load hardness : loads from 200 gm to 3 Kg. The most characteristic region comprises of loads from 200 gm to 1 Kg.
- (3) Standard hardness : loads over 3 Kg.

The present study is made in the region of microhardness defined in (1) above. The microhardness is influenced by the microstructures on the as grown, prepared or cleaved surfaces. Further, the experimental errors due to mechanical polishing, preparation of specimen, vibrations, loading rate, non-coincidence of microscopic axis and applied load direction, sharpness of indenter shape, measurement of impression etc. alter the hardness measurements considerably. These errors are minimized as much as possible in the present work.

### 3.3 BRIEF REVIEW OF WORK ON HARDNESS :

A birds-eye-view of the information on hardness upto

the present work is described in this section. The hardness study undertaken so far studying the strength of solids and the effect of various treatments on the hardness of a solid have proved somewhat useful. Most of the work has been reported on alkali halides and metals. Previously hardness studies were made only from the point of view of materials research but as the expansion in the field of scientific research is increased, hardness study helped in understanding various mechanical properties of solids. Gilman and Roberts /34/ 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 indented region during the propagation of stresses, initiating 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 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 both or at times fracture.

(1) Slip is the most common mode of plastic deformation, which is characterised by the displacement of one part of a 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.

(2) 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 definite symmetrical fashion.

Schmidt and Boas /35/ described twinning as simple sliding of one plane of atoms over the next, the extent of movement of each plane being proportional to its distance from the twinning plane. Partridge /36/ studied microhardness anisotropy of magnesium and zinc crystals. He observed twin in above crystals and concluded that the resolved shear stress criterion is insufficient to account for the observed distribution of twins and any analysis which attempts to relate deformation twinning with hardness anisotropy must take into account the dimensional changes which occur during twin deformation. Indenting diamond flats with diamond indenter, Phaal /37/ reported the slip and twinning of diamonds. Vahldick /38/ studied slip system and twinning in molybdenum carbide single crystals with the help of knoop and vickers indenters. Koserich and Bashmakov /39/ studied the formation of twins produced in Bi, Sb, Bi-Sb and Bi-Pb single crystals under the action of concentrated load by diamond pyramidal microhardness tester. 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 coefficient ( $\alpha$ ) in the equation  $l = a + \alpha d$ . The value of ' $\alpha$ ' was more for homogeneous alloys and increased with Sb content and remained constant for higher concentration of Sb and Pb.

Many workers have proposed some or other explanation for the micro-crack formation during indentation of a crystal surface. Smakula and Klein /40/ from the punching

experiments on sodium chloride explained the crack formation on the basis of shear on slip planes. Gilman/41/ attributed these micro-cracks which have a definite crystallographic direction to the piling up of dislocations on the slip plane.

Briedth et al. /42/ observed crack formation to be less at higher temperature (375°C) than at lower temperature (25°C). The cracks are usually observed to propagate from the corners of the impression. Sugita /43/ while studying indentation hardness of germanium crystal found occurrence of ring cracks and radial cracks and that the load required to produce the observable cracks increased with temperature.

The interferometric studies of indented surface have revealed the nature of deformation and the history of sample under test. Votava et al./44/ were the first to study the deformed region on the cleavage faces of mica and sodium chloride. Tolansky and Nickhols /45/ studied the indented surface of steel, tin and bismuth. They observed maximum distortion along the medians bisecting sides of the square and minimum along diagonals, showing thereby that no distortion projects beyond the diagonal.

Variation of hardness with respect to the impurity content, dislocation density and the change in mobility of dislocation was studied by various workers. Milvidski et al. /46/ observed decrease in hardness with increase in concentration of impurity and dislocation density in silicon single crystals. Kuz'menko et al. /47/ 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 as 'photochemical effect'. Beillin and Vekilov /48/ observed decrease in

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 inturn altered the plasticity. Westbrook and Gilman /49/ studied electrochemical effect in a number of semiconductors. They observed decrease in resistance of semiconducting crystals to mechanical indentations in the presence of a small electric potential (0.05 to 10 V) between the indenters and the crystal surface. Osvenskii et al. /50/ observed decrease in microhardness due to increase in carrier concentration for different contents of donor and acceptor impurities for GaAs and InSb semiconductors. In addition to this they also showed that decrease in hardness was independent of the type of carrier. Smirnov et al. /51/ studied the temperature dependence of carrier density and mobility of Ge crystals after irradiation with electrons and during various stages of annealing. They observed that the microhardness of such crystals did not recover fully their initial value and this was attributed to the interaction between radiation defects and dislocations, which could act as sinks or condensation for compounds of Frankel pairs. Seltzer /52/ who studied the influence of charged defects on mechanical properties of lead sulphide found that the rosette wing length and hardness were nearly independent of concentration of free electrons in n-type, while it had marked dependence on concentration of holes in p-type. For a hole concentration of about  $8 \times 10^{-7} \text{ cm}^{-3}$ , rapid hardening was observed with attendant decrease in rosette size. It was suggested that this behaviour results from an e.s. interaction between charged dislocations and acceptor point defects.

Comparative study of vickers and knoop hardness numbers has been investigated in detail by Mohrnhain /53/ on metallic materials. An analysis of knoop microhardness led Hays and Kendal/54/ to modify Meyer's/55/ law correlating applied load to the longer diagonal by a term which account for the resistance offered by the test specimens. Results were also discussed for use of modified Mayer's law to obtain knoop hardness numbers independent of applied load. Comparitive study of knoop and vickers hardness numbers was reported by Tietz and Troger /56/ on metals, on cleavage faces of calcite by Bhagia /57/, of sodium nitrate by Shah /58/ and on ammonium hydrogen d-tartarate crystals by Patel /59/.

Dislocations are responsible for the plastic deformation of crystalline materials. Excellent books on dislocations are now available /60-67/. There are several mechanisms by which dislocations are multiplied during deformation. As a result their spacing decreases. They interact and impede each others motion leading to work hardening. The strength of dislocation interference depends on the nature of crystal and on the ratio of deformation temperature to the melting point of crystal. In general, hardening of crystals can be accomplished by introduction of any barrier to dislocation motion. This can occur by (a) work hardening (b) impurity hardening (impurities tend to segregate to dislocations and pin them) (c) decreasing grain size in a poly crystal (grain boundaries are barriers to dislocation motion) (d) dispersion of fine particles of second phase in the crystal and (e) phase transformation by quenching.

Perinova and Urusovskaya /68/ studied hardening of single crystals of NaCl by X-ray and found the increase in microhardness by irradiation due to pinning of dislocations in irradiated samples and that the pinning was not destroyed by illumination. The effect of impurity on hardness was studied by various workers. Dryden et al./69/ studied hardness of alkali halides when low concentrations of divalent cations are incorporated in the crystal lattice on the basis of dielectric measurements of doped crystals. Urusovskaya et al. /70/ investigated the influence of impurity on the strength of crystals, microhardness, length of dislocation rosette rays and velocity movement in cesium chloride crystals. Takenchi and Kitano /71/ reported the softening of sodium chloride crystals due to introduction of water molecules.

The plastic resistance was almost independent of dislocation velocity except at very high velocities. It was however strongly influenced by temperature, impurities, radiation damages and structure of core of dislocation. Gilman /72/ observed a sharp drop in plastic resistance of covalent crystals at roughly about two-third of the melting temperature and suggested that the drop was because of cores of dislocation in covalent crystal 'melt' at this temperature.

When indented crystal was etched by a dislocation etchant, rosettes around indentation were formed on some crystals, indicating formations of dislocation loops /73-89/. Because of substantial effect of surface layers on the microhardness, the increase in microhardness was observed when applied load was reduced (Upit et al./90/). It was shown that for a load 'p'

and the length 'l' of rays in dislocations around indentation, the ratio  $p/l^2$  was not constant at low loads due to retarding influence of the surface on the motion of dislocations. Further they estimated the change of the mechanical properties of a crystal as the indentation depth decreased on the basis of correlation between size of an indentation and the length of the dislocation beam. Detailed study of dislocation rosette structure on various crystallographic planes and determination of microhardness at high temperatures (1200°C, 1600°C and 1800°C) of  $Y_3 Al_5 O_{12}$  revealed local plastic deformation around indentation mark. (Voinova, N. N. and Bereehkova, G.V./91/). They observed that (112) plane of  $Y_3 Al_5 O_{12}$  exhibits highest value of microhardness. Temperature dependence of microhardness was reported by Sarkozi and Vannay /92/. They concluded that besides thermal stress the observed hardening may be due to dislocation piled up at various impurities, to complexes in solid solution and vacancy clusters which were developed at high temperatures. By quenching, the clusters become distributed in the crystals as fine dispersion. Edelman /93/ showed that microhardness of InSb and GaSb single crystals decreased exponentially with temperature. The presence of deflation point on the curves at  $0.50 T_m$  indicates the deformation by slip ( $T_m$  is the melting point of crystal). The decrease in hardness with decrease in carbon content in titanium carbide was confirmed by Samsonov et al./94/.

Temperature dependence of microhardness was also studied by Shah /58/, who showed that hardness of calcite cleavage faces increases with quenching temperature. The microhardness of Zn and KBr (Acharya /95/) and sodium nitrate (Joshi /96/) decreases with increasing quenching

temperature while converse is the case for TGS and InSb. They obtained an empirical formula connecting hardness with quenching temperature which was successfully applied to study quench hardness of even non-crystalline materials such as Glass Fibre Reinforced Polymer (GFRP) composites (Dubey /97/). Microindentation studies were performed on  $\text{CuInSe}_2$  /98/, super conducting material  $\text{Y.BaCuO}$  /99/, rubidium hydrogen tartarate /100/, mercuric iodide /101/,  $\text{Ba}_{1-x}\text{K}_x\text{BiO}_3$  /102/ and  $\text{BaFCI}$  /103/. Vickers indenter was used by many workers in recent years /104, 105, 106/, and a relation was obtained between Vickers hardness number and Universal hardness from a specimen's elastic characteristics /107/. J. Benet et al. /108/ observed that hardness is decreasing with increasing load and the radical crack lengths were used to calculate the fracture toughness and brittleness index.

It can be seen from the 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.

The present work is centered on the study of microhardness of cleavage planes of synthetic melt grown single crystals of Sodium Chloride, Potassium Chloride and Potassium Bromide by using Knoop indenter. The study includes the following :

- i) Variation of load with diagonal length of indentation mark.

- ii) Variation of hardness with applied load for different orientation and
- iii) Quench hardness.

This work is reported in the ensuing chapters.



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