

CHAPTER - 2

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Dislocations which constitute the major part of imperfections are usually created by thermal or mechanical stresses at the time of crystal growth or after it. Plastic deformation introduces new dislocations or moves the ones already present in the crystal. The plastic deformation study has been successful, by large, to understand the fracture mechanics, particularly in ductile metals and alloys. In the case of solid-solution alloys, to accommodate substitute atoms of greater or smaller size, the solvent lattice suffers elastic distortion. The distorted lattice is said to be strained and offers increased frictional stress to the free movement of dislocations when the alloys is sheared.

Deformation in single crystals takes place by slip, twinning, crack and fracture. Though the plastic deformation is said to be the permanent deformation in a body or a single crystal, it is defined as the deformation involving creation or motion of dislocations. The phenomena of crack and fracture are classified as ductile or brittle according to the nature of their nucleation and propagation during the deformation. The other deformation phenomena involve lattice reorientation as in deformation twinning and the occurrence of irregular

inhomogeneous deformations like in irrational twins, kink bands, deformation bands, Brilliantov-Obreimov bands etc. Crocker et al [1] have shown that the kinking and slip phenomena can also be considered as distinct deformation mechanisms. Both of these phenomena have been studied and reported for zinc by Hess et al [2] and for nickel by Flewitt [3]. The factors affecting the occurrence and nature of deformation produced by different mechanisms are crystal structure, nature of atomic bonds or interatomic forces, strain rate, temperature, impurities, method of deformation and crystallographic orientation of the deforming stress axis with respect to the crystal. The general aspects of deformation by slip and twinning have been treated by various authors (Van Bueren, Cottrell, Reed-Hill, Barrett and Reed-Hill et al) [4-8]. The basic theories of crack and fracture have been reviewed by Lawn et al [9] and Taplin [10].

Hardness :

Many definitions have been given for hardness from time to time but none has been found proper with enough quantitative interpretation and understanding for the aim and theme. Tuckerman [11] explained hardness as a hazily conceived aggregate or conglomeration of properties of a materials more or less related to each other. Ashby [12] defined hardness as a measure of resistance to permanent deformation or damage. The general definition of indentation hardness which is

related to the various forms of the indenters, is the ratio of applied load to the surface area of the indentation. Meyer [13] proposed that hardness should be defined as the ratio of load to the projected area of the indentation. Hence the hardness has the dimensions of stress.

Chatterjee [14] further defined indentation hardness as the work done per unit volume of the indentation in a static indentation test for a definite angle of indenter. On the basis of this definition and Meyer's law, $P=ad^n$, for spherical indenters, he derived a formula for calculation of hardness. Plendl et al [15] defined hardness as the pressure or force per square centimeter which can be conceived as the ratio of the input energy and volume of indentation. He further concluded that the resistance itself is a function of the lattice energy per unit volume which is called volumetric lattice energy (U/V), having dimension ergs/c.c. Here "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 the specific heat. Thus hardness was considered to be an absolute property. Matkin et al [16] suggested a correlation of hardness with the dislocation theory. They gave a definition of hardness on the basis of generation and movement of dislocations associated with indentation. Later, Westbrook et al [17] concluded that hardness is not a single property but it is rather a whole complex

of mechanical properties and at the same time a measure of the intrinsic bonding of the material. Gilman[18] defined hardness as the strength determining parameter which gives information regarding elastic, anelastic, plastic, viscous and fracture properties of both the isotropic and anisotropic solids.

From all these definitions, the basic qualitative meaning of hardness turns out to be a measure of resistance to plastic deformation. Practically, it carries different meaning to different people; for a metallurgist it is resistance to penetration, for a lubrication engineer it is resistance to wear, for a minerologist it is resistance to scratching, etc. The hardness of materials can be determined by various methods :

- 1 Scratch method
- 2 Abrasive method
3. Plowing method
4. Rebound method
5. Damping method
6. Erosion method and
7. Static indentation method

These methods are briefly out-lined below :

1. Scratch Method :

This method first developed by Friedrich-Mohs in 1822, is still in

wide use today by mineralogists. In this method, the ability of one material to scratch another is termed as the hardness of that material with respect to the other. The Mohs method is not suitable for general use with materials of hardness greater than 4. Modifications of this method were later made into other sensitive methods and experiments.

2. Abrasive Method :

In this method the measure of resistance to mechanical wear is taken to be the amount of material removed from the surface under specific condition, where a specimen is loaded against a rotating disc. The rate of wear is taken as the hardness measure.

3. Plowing Method :

In this method, a blunt element such as diamond is moved across a surface under controlled conditions of load and geometry and the width of the groove produced is taken as the measure of hardness.

4. Rebound Method :

In this method, an object like steel ball having a standard mass and dimension, is bounced from the surface and the height of rebound is taken as a measure of hardness.

5. Damping Method .

In this method, the change in amplitude of a pendulum having a hard

pivot resting on the surface of the material is taken as a measure of hardness.

6. Erosion Method :

Here, sand or abrasive grain is caused to impinge upon the surface under standard conditions and loss of material in a given time is taken as a measure of hardness.

STATIC INDENTATION METHOD :

In this method, a steel ball, a pyramid or a cone is forced into the surface and the load per unit area of the permanent impression formed is taken as the hardness measure. The Brinell, Vickers, Rockwell and Knoop tests are of this type. A hard indenter of specific geometry is slowly pressed under a load into the surface to be examined and after a certain time of application, it is carefully removed leaving behind a permanent indentation mark on the surface. The ratio of applied load to the area of the mark is termed as the hardness of the specimen indented. The hardness value, apart from other factors, also depends on the geometry of the indenter and if the specimen is anisotropic, complicated effects like ridging and sinking, especially with pyramidal indenters [19], occur, requiring correction in the formula used to calculate hardness. The indenter is made up of a very hard material most usually diamond, to eliminate errors due to

elastic distortion. Vickers square based pyramidal diamond indenter is employed wherever geometrically similar impressions are needed. To study hardness anisotropy of a crystal, usually Knoop indenter, which is rhomb-based diamond pyramid is employed and to eliminate anisotropy effects, pentagonal indenter as designed by Brookes and Moxley [20] is used. The static indentation test appears to be simple but the stress fields developed in crystalline materials are extremely complex.

Hardness anisotropy is an important field of study Hannink et al [21] studied and reported the slip behaviour and slip systems in cubic carbides by the method of hardness anisotropy measurement. The shape of the indentation mark was found to depend on the orientation of the indenter with respect to the indented surface in cubic crystals [22] The non-square shape of the indentation mark is related to the anisotropic elastic properties of the crystals as per Boyarskaya [23], whereas Petty [24] related the non-square shape to the anisotropy of plastic properties in the case of aluminium single crystals. A mathematical expression for Knoop hardness anisotropy of cubic crystals has been given by Shimotori [25]. Brookes et al [26] have given an excellent review of the effect of plastic anisotropy and have established a relation between effective resolved shear stress just below the indenter and the observed hardness.

Murphy [27] measured hardness anisotropy in copper crystals. The anisotropy variation of hardness and hence the plastic deformation was explained as due to escape of primary edge dislocations. Anisotropy in microhardness on (111) plane of Bi single crystal has been studied by various techniques [28]. Interestingly, Gilman,[18] correlated indentation hardness with various physical properties. He has shown by giving graphical correlations, that apart from stress, other physical properties which are fundamental in nature also play an important role in determining the hardness. These are, elastic modulus, for metals and covalent crystals and average band gap, energy gap density, optical energy gap, homopolar energy gap and interatomic distance for covalent crystals.

Among the factors not inherent to the materials, which can influence resistance to dislocation motion and in turn the observed hardness value, the main ones are,

- 1) Work hardening
- 2) Impurity hardening
- 3) Variation of grain size in polycrystalline materials
- 4) Dispersion of second phase particles and
- 5) Phase transformation

The hardness dependence on surface treatment, dopant and orientation of crystal has been established [29]. Gilman [18] has observed, in the case of CdS crystals, that the local pressures created below the indenter may induce phase change of the test material and can affect the measured value of hardness. Hardness variation with respect to impurity content, dislocation density and change in mobility of dislocation has been studied by various workers. In Si single crystal, hardness was found to decrease with increase in concentration of impurity and dislocation density [30]. Many workers have studied the Vickers micro-hardness of $\text{Cd}_x\text{Hg}_{1-x}\text{Te}$ alloy at room temperature as a function of x and their findings are as follows :

- 1) The hardness increases as a function of composition up to $x = 0.75$ [31]
- 2) Increase in hardness with increase in x from 20MPa at $x = 0$ to 440MPa at $x = 1$, exhibiting a maximum of about 850 MPa at $x = 0.75$ Hardening rate dH/dt depends on the composition [32].
- 3) The same results of increase in hardness in the composition range $0.6 < x < 0.8$ for $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ alloys [33].

In InBi:Te as concentration of tellurium increases the hardness increases [34].

The materials with high dislocation mobility are harder than those with low dislocation mobility. For example, it has been found that the semimetals have small microhardness and low dislocation mobility [35].

VARIATION OF HARDNESS WITH LOAD :

From the geometrically similar shapes of the indentation marks at various loads, it can be shown that the hardness is independent of load, though it is not true experimentally for certain ranges of applied load. The hardness obtained by the indentation tests is not the actual hardness prior to indentation. This is so because the indentation process deforms the indented region of the sample. This deformation has to bear its effect in responding to the progressive penetration of the indenter. Usually at low applied loads, the deformation causes work hardening of the surface layers. Hence, the measured hardness is more than the actual. The main findings in this respect are briefly given below.

The variation of hardness with load was explained in terms of slip in Te crystals [36]. Knoop [37] and Bernhardt [38] observed increase in hardness with decrease in load. Campbell et al [39] and Mott et al [40] observed decrease in hardness with decrease in load. Taylor[41] and Bergsman [42] observed no significant change in hardness by varying load. A relationship between microhardness and applied load has been given by Meyer [13], viz., $P = ad^n$ where P = applied load, d = diagonal of the indentation mark and a and n are constants. In the case of Vickers microhardness, the value of the exponent n is equal to 2 for all indentation marks. It implies a constant hardness value for all

loads. Hanemann [43] concluded that in the low load region, n has a value less than 2. While Onitsch [44] found the value of n between 1 and 2. Grodzinski [45] found variation of n from 1.3 to 4.9. However, most of the values of n were found to be 1.8. Though hardness would be expected to be constant, the actual results obtained by different workers, revealed disparities amounting to 30 to 50%. In the case of SnSe , SnSe_2 and $\text{Bi}_2\text{Te}_3:\text{Sb},\text{Sn},\text{Se}$ crystals, the hardness behaviour at low loads has been attributed to the deformation induced coherent region below the indented surface [46,47]. Due to this variation, a high load region has to be selected which leads to the definition of a load independent value of microhardness. The scattered results may be attributed to the following reasons :

- 1) Meyer's law is not valid
- 2) Microstructures exercise a considerable influence on measurements involving very small indentations.
- 3) Experimental errors due to mechanical polishing, penetration of specimen, vibrations, loading rate, shape of indenter and measurement of impression affect the hardness value considerably.

Though the range of macro and micro indentations are not principally definable, in practice, two possible regions can usually be recognized :

(1) Microhardness :-

From the lowest possible loads up to a maximum, which may be around 50 gms.

(2) Macro or Standard hardness .-

For loads over a high value, which may be around 500 gm or more.

A detailed account of work carried out by the present author on microhardness of $\text{In}_{0.1}\text{Bi}_{1.9}\text{Te}_3$ $\text{In}_{0.2}\text{Bi}_{1.8}\text{Te}_3$ $\text{In}_{0.5}\text{Bi}_{1.5}\text{Te}_3$ and pure Bi_2Te_3 is given in Chapter – VII

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