CHAPTER VIII

MICRO-HARDNESS STUDIES ON THE $(10\bar{1}0)$ PRISM FACES OF Te CRYSTALS

The results of microhardness studies on prism planes of tellurium whiskers and cleaved surfaces are presented in this chapter. The load variation of hardness at low loads and the directional hardness have been studied. No attempt has been made to study the distribution of dislocations around the indentation mark.

Micro-hardness studies have been applied in recent years to gather information about the plasticity of the crystals and many attempts have been made to relate the hardness number to the tensile properties. In fact, the hardness properties are closely related to the crystal structure. Indentation produces plastic deformation which must be accompanied by dislocation multiplication and movement. Therefore informations about the dislocations associated with this mode of deformation can be collected by etching the indented crystals. Lot of work has been done in this direction. The distribution of dislocations around an indentation in alkali halide crystals and germanium has been investigated by many workers[1-6]. No discussion on this line is made in the text.

According to Mott[7] the hardness properties are basically related to the crystal structure of the material or in other words, the way in which the atoms are packed and the electronic factors operating to make the structure stable. The extreme hardness of diamond can be attributed to the highly stable covalent structure.

The detailed shape of the indentation mark depends on the degree of work-hardening of the material under examination. If the material is highly worked, such that, not much appreciable work-hardening is produced by the indentation process itself the metal behaves as an ideally plastic material. The displaced metal flow up the faces of the indenter and a barrel shaped indent is produced. In the case of annealed metal, the metal is pushed out from the indenter and as a result the indentation mark takes a 'pin-cushioned' shape.

Tolansky and Nickols[8,9] applied the multiple beam interferometric methods to examine the surface flow around Vicker's pyramid indents on single crystals of tin and some alloys. This revealed the existence of curious flow anomalies closely related to the crystallographic directions. The indents instead of having square shapes had two opposite sides convex with the other two concave. There was also a marked pile-up in the direction of the c-axis of the crystal irrespective of the orientation of the indent and sink in a direction perpendicular to the pile-up. The forces acting on the crystal during indentation could be considered to operate normally to the face of the indenter.

The anisotropy of micro-hardness on different planes of a material and at different orientations on the same face have been studied by many workers.

Petty[10] studied the anisotropy of micro-hardness in single crystals of super-purity aluminium using Vicker's and Brinnell indenters. The Vickers hardness indenter showed a slight anisotropic effect in that, the indentations were not a square always. But the actual hardness values at a variety of angles on the three faces (001), (110) and (111) of the superpurity aluminium crystals fell between 14 and 17 U.P.N. with never more than 2 U.P.N. variation between results on any one face. No significant variation in hardness was observed by the above authors when the indentation was carried out at different angles on the same face. Partridge and Robers[11] studied the microhardness anisotropy in zinc and magnesium single crystals. The observed anisotropy in the hardness on the first order prism planes of these crystals was attributed to basal slip and that in the basal plane was attributed to pyramidal slip in zinc and twinning in Magnesium.

The direction al hardness studies on Bi and Sn single crystals were carried out by Tolansky and Williams[12]. Hardness variations were measured over a range of 180° on each surface studied. The variation in hardness depends on the particular crystal surface indented. It was demonstrated by interferometry that slip mechanism plays a primary role in producing directional variations in hardness.

Vahldick et al.[13] observed a variation in the microhardness of MO_2C single crystals on different planes along the same direction. The variation in hardness was attributed to slip mechanism, twinning and the binding mechanism of MO_2C. Tsuya[14] studied the effect of orientation on hardness of Beryllium single crystals in the range of room temperature to 400°C. The anisotrpy of hardness with temperature is related to the alternation of deformation modes. The assymmetry of the indentation mark is explained on the basis of deformation around the impression.

French and Thomas[15] had measured the hardness in WC crystals at loads of 100 and 500 gm on (0001) and $(10\bar{1}0)$ planes. The hardness on basal plane had shown little anisotropy, whereas hardness on prism plane exhibited marked orientation dependence. The micro-hardness was minimum along the c-direction and was maximum normal to the c-axis. The observed anisotropy was explained on the basis of slip and resolved shear stress.

The microhardness anisotropy of melt grown Te crystals had been investigated by Boyarskaya et al.[16] by Scratch method. They have observed that the hardness along the c-direction is lower than that along a direction perpendicular to c-axis.

Goldenberg and Bychkova[17] investigated the microhardness of NaCl whiskers. The microhardness of the thinnest whiskers decreased very rapidly as thickness increased. But at greater thickness it decreased slowly.

The reported observations[18-21] on the load variation of hardness are somewhat contradictory. Bergsman[18] found that hardness does not vary with load. Rostoker[19] studied the load variation of hardness of copper and concluded that there is a marked decrease in hardness at low loads. Buckle[20] observed a considerable increase in the hardness at low loads. This increase has been attributed to the effect of elastic recovery which results in the reduction of the diagonal length. Grodzinsky[21] in his study of sintered carbides found that the hardness vs load curve shows a peak at low loads. Berzina et al.[22] had studied the variation of microhardness with load of some alkali halide crystals. They have shown that the surface layer of a crystal has a substantial effect on the microhardness in case of ionic crystals at low loads. In view of these contradictory observations, the present author has studied the load variation of microhardness at low loads for tellurium in the form of whiskers and cleaved surfaces of single crystals. The variation in directional hardness on the $(10\bar{1}0)$ prism planes is studied. These studies have enabled the author to compare the hardness of vapour-grown and melt-grown crystals and hence to assess the perfection of the whisker crystals.

The microhardness measurements were carried out with a diamond pyramidal indenter supplied by M/s Cooke, Troughton and Simms Ltd., which can be used with a Vicker's projection microscope. The diagonal lengths were measured with a micrometer eye-piece. In all cases the diagonal length used for calculations was the mean of several measurements.

The whiskers of tellurium and the melt-grown crystals were grown as explained in Chapter IV. The melt-grown crystals were slowly cooled to liquid nitrogen temperature and then cleaved with a knife-edge. The crystals were carefully mounted on glass flats before indenting.

The samples were indented with loads varying from 1 to 100 gm and the Vicker's hardness was calculated. Fig.1 shows the variation of hardness with load on the prismatic plane of whiskers and cleaved crystals. The readings are given in Table 1. From the figure it is seen that there is a peak at 10 gm load for both the curves. For a load above 10 gm slip takes place. Fig. 2 is a photomicrograph of an indentation mark, at 50 gm load.

The tellurium lattice consists of spiral chains each spiral having three atoms per turn. The chains lie parallel to one another with the corresponding

TABLE 1

Variation of Hardness with load.

Obs. No.	Cleaved Specimen		Whiskers	
	Load in gm	Hv	Load in gm	Hv
1	1	17.0	1	19.7
2	2	19.5	2	23.0
3	5	39.5	5	34.8
4	10	40.5	10	83.0
5	20	37.85	20	30.1
6	50	36.7	50	29.8
7	100	36.0	100	18.31

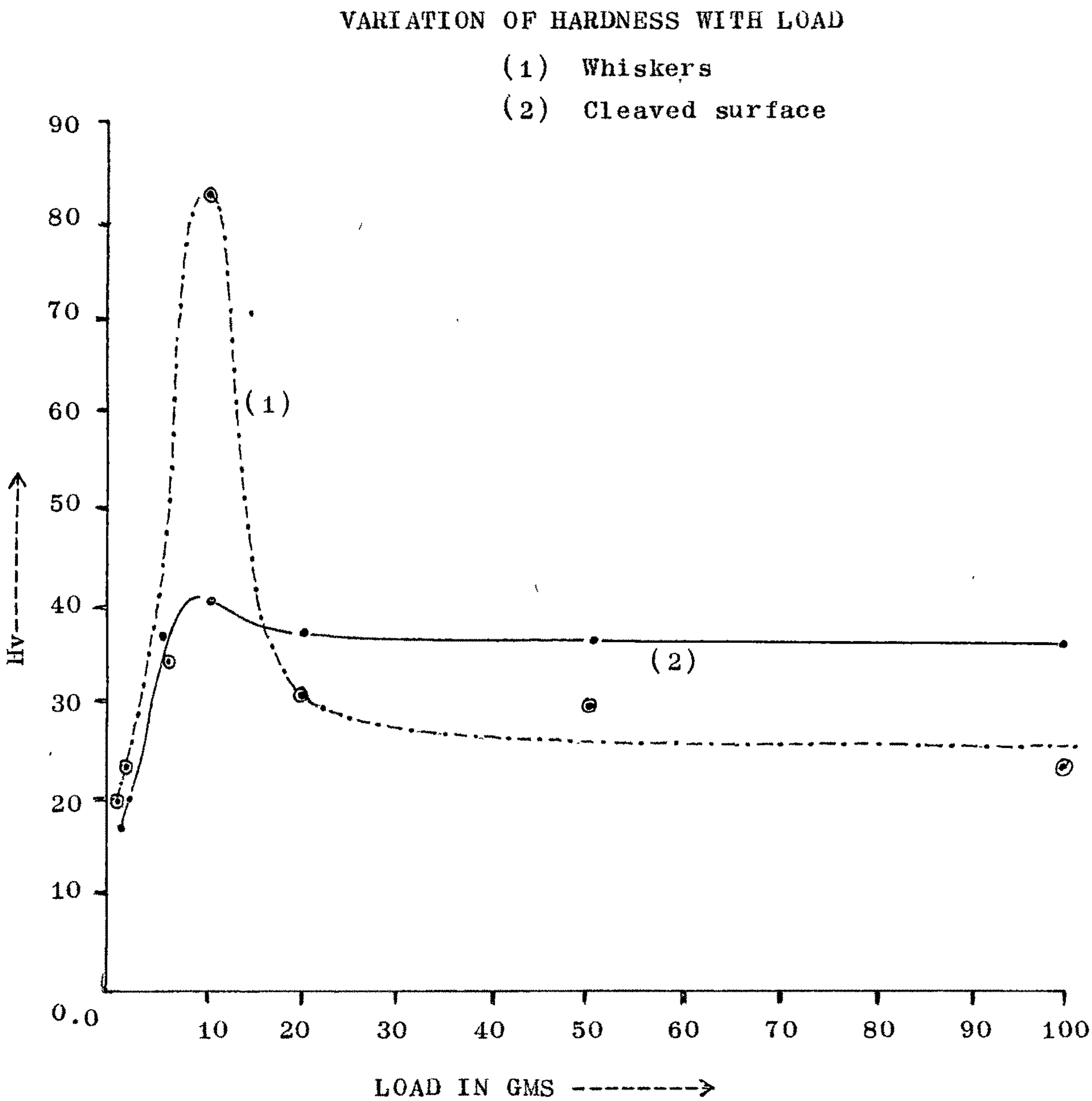
VARIATION OF HARDNESS WITH LOAD
(1) Whiskers
(2) Cleaved surface
90
80
70
60
50
40
30
20
10
0.0
Hv
(1)
(2)
10
20
30
40
50
60
70
80
90
100
LOAD IN GMS

Fig. I

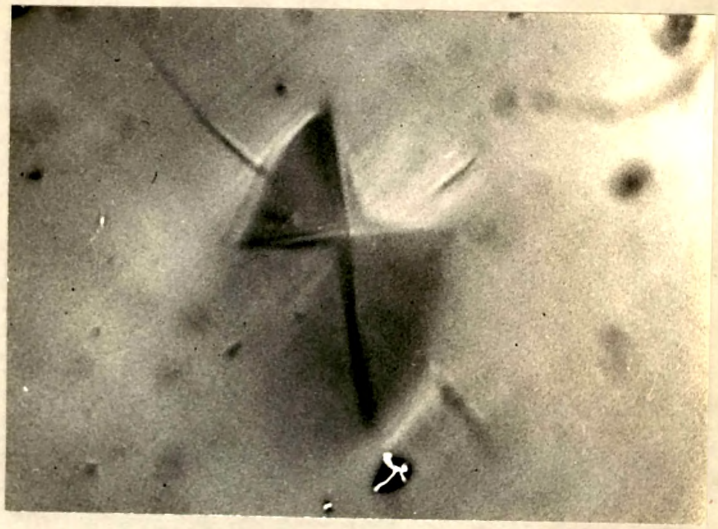

Fig.VIII-2 X940

atoms in each chain forming hexagonal nets. The bonds between atoms on each chain are covalent whereas between chains they are a weak mixture of electronics and Van der Walls binding. Because of the anisotropy of the atomic arrangement, the effect of the applied stresses is to cause the chains not to bunch together but to extend in length, slip occuring in the $\langle\bar{1}2\bar{1}0\rangle$ directions. The initial increase in hardness may be due to the elastic recovery in the diagonal length. Once slip starts above 10 gm load the hardness value decreases suddenly due to deformation occuring by slip. The comparatively higher hardness value or the shift in the curve in case of the whisker is due to the high perfection of the crystals.

The directional hardness on the prism plane was studied by rotation of the indenter over a range of 180°. Micro-indents with 10 gm load, applied, ~~applied~~ for 20 seconds, were made. The first indent was made with one of the diagonals of the pyramid indenter parallel to the direction of the c-axis. A succession of indents were made with the angle between the indentation diagonal and the c-direction increasing by 15° upto 180°.

The directional hardness variation observed for $(10\bar{1}0)$ faces of the whiskers and cleaved specimens is presented in Fig. 3. The hardness values obtained were as given in Table 2. Two peaks are observed at 45° and 135° for whiskers and at 50° and 150° for cleaved specimens. The minima are at 0°, 90° and 180°. The hardness, as seen from the graph, is a maximum when one of the indentation diagonals is at 45° to the c-direction, and is a minimum when the diagonal is parallel to the c-direction. The ratio of the maximum and minimum values of hardness is 2.3:1 for whiskers and 1.8:1 for cleaved crystals.

The slip mechanism plays a primary role in leading to the observed variation hardness with load and the variation in directional hardness. The observed directional anisotropy in hardness is mainly due to the change in the orientation of the indenter relative to the active slip planes and slip directions. The forces acting on the crystal during indentation can be considered to operate normally to the face of the indenter. The length of the diagonal can thus be regarded as a measure of the ease of slip, a larger

TABLE 2

Directional Variation of hardness.

Obs. No.	Cleaved Specimen		Whiskers	
	Angle of rotation in degrees	Hv	Angle of rotation in degrees	Hv
1	0	23.4	0	34.0
2	15	27.0	15	43.3
3	30	38.2	30	57.0
4	45	40.1	45	80.0
5	60	31.5	60	44.8
6	75	25.3	75	36.6
7	90	25.0	90	34.0
8	105	24.0	105	39.7
9	120	29.9	120	51.6
10	135	38.4	135	78.9
11	150	41.9	150	50.3
12	165	31.4	165	42.5
13	180	26.5	180	37.7

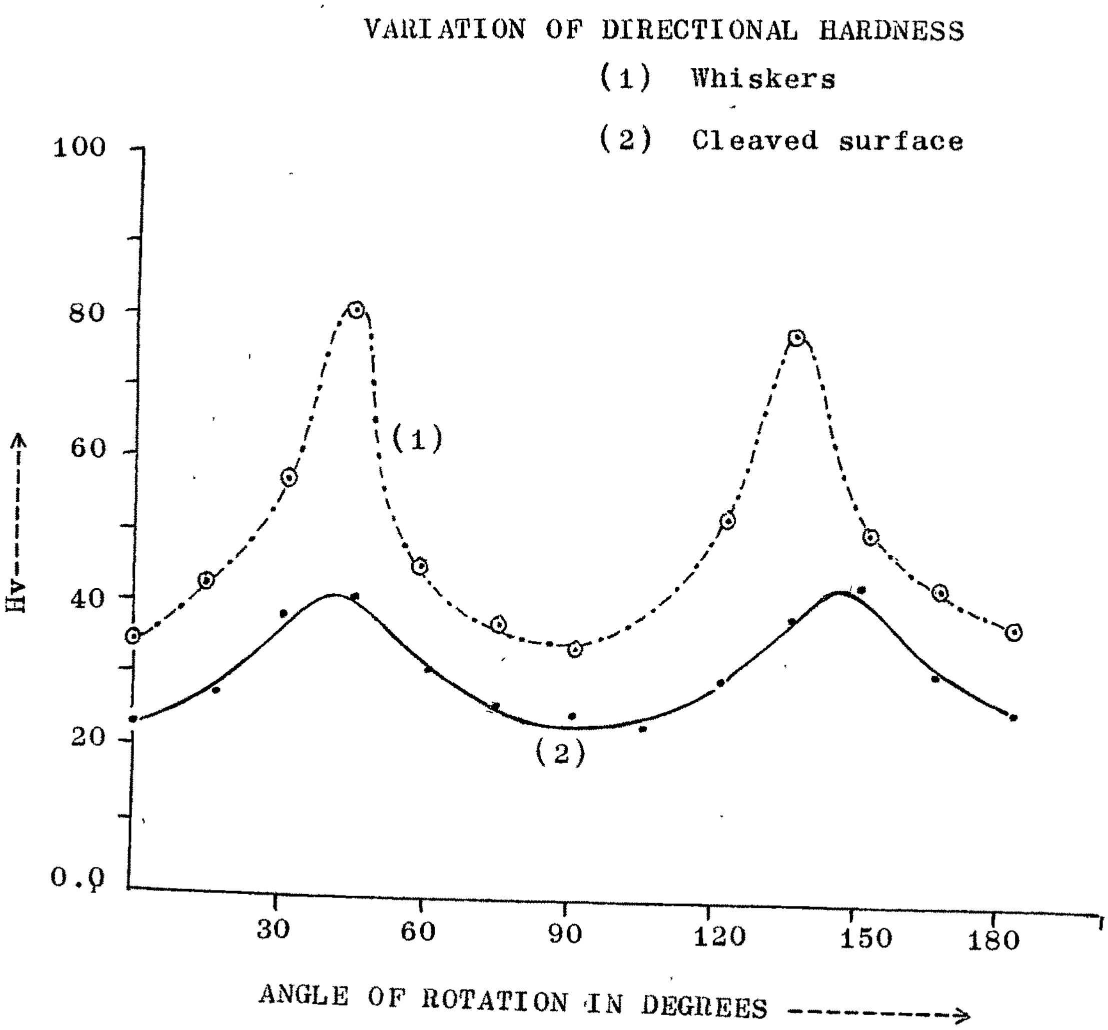
VARIATION OF DIRECTIONAL HARDNESS
(1) Whiskers
(2) Cleaved surface
100
80
60
40
20
0.0
Hv
(1)
(2)
30
60
90
120
150
180
ANGLE OF ROTATION IN DEGREES

Fig. 3

indentation appearing when the resolved shearing stress is high, a smaller indentation when it is low. When the slip plane is most suitably oriented the yield stress or the applied stress required is a minimum. The slip plane is most suitably oriented for slip during indentation when the hardness value is minimum, that is in the present case when the indentation diagonal is parallel to the c-axis. Maximum hardness occurs when the crystal is least suitably oriented for slip or when the resolved shear stress on possible slip planes is least. Hardness of a crystal may therefore be considered as a measure of yield stress of the crystal in different directions.

Results and Conclusions

At low indentation loads the microhardness varies with load. In Fig. 1 a peak is observed at 10 gm load for both whiskers and cleaved surfaces. The initial rise in hardness may be due to the elastic recovery of the diagonal lengths. The sudden fall in hardness value after 10 gm load is due to the fact that slip occurs above 10 gm load and the hardness decreases due to deformation by slip.

Marked anisotropy is observed in directional hardness of whiskers and cleaved crystals. The hardness is a minimum when the diagonal of the indent is parallel to the c-direction, and it is a maximum when the diagonal is at 45° to the c-direction. The variation in directional hardness is due to the change in orientation of the indenter with respect to the active slip directions of the crystal.

Both in Fig.1 and Fig.3 the curve for whiskers exhibits a marked shift. This higher value of hardness for whiskers than that of cleaved surfaces is due to the high perfection of the whisker crystals.

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