APPENDIX

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APPENDIX - I

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MICROINDENTATION HARDNESS OF

INDIUM ANTIMONIDE CRYSTALS

APPENDIX - 1

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MICROINDENTATION HARDNESS OF

INDIUM ANTIMONIDE CRYSTALS

PAGE

A.1	Introduction	I
A.2	Experimental	vı
A₀ 3	Variation of hardness with load	ν
	A.3.1 Observations	VI
	A.3.2 Results and Discussion	VI
	A.3.3 Relation between hardness and temperature of quenching	VIII
A.4	Graphical analysis of observations $$	XVIII
A.5	Conclusions	XX

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REFERENCES

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A.1 INTRODUCTION

Of all the III - V compound semiconductors, Indium antimonide is easy to grow and its technology is advanced to the state that it could compare well in impurity content with the purest germanium and silicon. It is an almost ideal vehicle for conducting work in the study of band structure, effective masses, crystal binding and transport properties. Since the discovery of the semiconducting properties of InSb in 1950, a large amount of information is now available.¹⁻⁴ With the invention of the semiconductor laser and discovery of the Gunn effect, there is now an increased prospect of the industrial application of III - V compounds.

Indium antimonide is a stoichiometric compound with a zinc blende structure and chemical formula InSb. Some important properties and information about it is collected in table Ao. The hardness formulae derived in a phenomenological manner by a systematic detailed study of hardness of single crystals of alkali halides (KCl^5 , KBr^6), of rhombohedral crystals (Calcite^{5,6,7}, sodium nitrate in the present work), of metallic crystals (Zinc⁶) and ferro electric crystals (TGS⁶) are now firmly established for predicting quench hardness of single crystals of Indium antimonide is to verify the hardness formulae derived for single crystals of above materials.

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Table Ao

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GENERAL INFORMATION OF INDIUM ANTIMONIDE

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Chemical formula	:	InSb
Crystal structure	:	Zinc blende type, Layered crystal,Lattice constant 6.5 A at 300 ⁰ K.
Melting point	:	535 [°] C (under atmospheric pressure)
Crystal Growth	:	CZochralski method (Gatos 1960)
Growth Direction	:	[111] and [112]
Electron Density	:	10^{13} to 10^{16} per cm ³
Hardness of Indium	:	Between 3 and 3.5 on Moh's scale.
Hardness of Antimony	:	1.2 on Moh's scale
Mechanical property	:	Soft, Brittle, cleavage (111)
Valence electrons of Antimony atom	:	5
Valence electrons of Indium atom	:	3
Electron density	:	10 ¹³ to 10 ¹⁶ per cm ³
Band gap at 77°K	:	0.43 ev
Effective mass of electron	:	0,013 m ⁺

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Effective mass of Hole : 0.4 m^4 Effective charge : $0.42 \text{ e}, -0.45 \text{ e}, -0.16 \text{ e}^{++}$ Spin-orbit splitting : 0.81 evElectron mobility at : $7 \times 10^4 \text{ cm}^2/\text{V}-\text{S}$ Hole mobility at : $7 \times 10^4 \text{ cm}^2/\text{V}-\text{S}$ Ratio b $(=/\mu_n//\mu_p)$: 100

Refractive Index at $\lambda = 0.620 \text{ micron} = 4.29$ Extinction
Coefficient at $\lambda = 0.620 \text{ micron} = 1.83$ Reflectance
Coefficient at $\lambda = 0.620 \text{ micron} = 0.453$

+ m = Mass of free electron

e = Charge of free electron

+ from other method

++ The Callen effective charge

A.2 EXPERIMENTAL

Small crystal cleavages from one big single crystal were used in the present study. Every time freshly cleaved blocks of appropriately 8 mm x 5 mm x 1 mm size were used for hardness studies. Precautions such as use of almost identical sizes of treated and untreated samples, fixing and levelling of cleaved block on glass plate, spacing between two consecutive indentation marks on cleaved surface, indentation time, measurement of dimensions of indentation marks, gradual heating of a sample to a desired temperature and quenching it to room temperature etc. (cf. Chapter V) were scruplously observed in the present work. The maximum temperature for quenching experiments for this crystal was 633°K. Since InSb gets oxidised in air ; experiments on thermal treatment were carried out by keeping sample in inert (Hydrogen) atmosphere (Worth 1962) or in vacuum. The indentation marks for applied loads varying from 1.25 gm to 120 gm were produced by square based Vickers pyramidal indenter on the freshly cleaved surface in such a manner that one of the diagonals of the mark was always parallel to crystal edge.

The study of the variation of diagonal length of indentation mark with applied load on cleavage faces of indium antimonide (Panchal 1981) has led to conclusions

II

which are almost identical with those obtained from a corresponding study of synthetic single crystals of sodium nitrate (cf. Chapter V). The empirical equations used in the above analysis is Meyer's law

$$P = a d^{n} \dots (1)$$
or log P = n log d + log a \dots (2)

where d is diagonal length of indentation mark produced by an applied load P ; a is standard hardness and the exponent n is a constant ; both are characteristics of material.

A.3 VARIATION OF HARDNESS WITH LOAD

It is now well established from a study of hardness of synthetic single crystals of sodium nitrate and other crystals that 'standard hardness', 'a' is a function of quenching temperature ; 'a₁' and 'a₂' in general vary with quenching temperature (T_Q) . It is now interesting and useful to study in detail how hardness changes with quenching temperature for InSb crystals.

The Vickers hardness number is defined by equation, (Mott, 1956)

$$H_v = 1854.4(P/d^2)$$
 (3)

where load P is measured in grams and diagonal length d,

 $\overline{\mathbf{V}}$

of the indentation mark in microns. The hardness number is not an ordinary number, but a constant having dimensions and a deep, but less understood physical meaning. The present work is a quantitative study of effect of quenching temperature on hardness. It is basically a phenomenological study of the problem.

A.3.1 Observations

The observations which were recorded for studying the equation, $P = a d^n$ are used in the present investigation (Table A-1,2) for thermally treated and untreated samples. The observations are graphically studied by plotting the graphs of hardness number versus load P (Fig. A-1, 2, 3, 4, 5, 6). In what follows the hardness and hardness number will be used to indicate same meaning.

A.3.2 Results and discussion

It is clear from the graphs of hardness number (H) versus load (P) that contrary to theoretical expectations (cf. Chapter IV), the hardness varies with load. The hardness at first increases with load, reaches a maximum value then gradually decreases, and attains a constant value for all loads. This behaviour is also found for both types of hardness number viz. Knoop hardness number (H_k) and Vickers hardness number (H_v) in case of sodium nitrate crystal (cf. Chapter VI) and calcite crystal

VI

Table A-1

969 0.7078 0.6978 $ 0.7497$ 0.7497 0.7497 0.7497 0.7497 0.7497 0.7835 0.8062 0.8347 1740 0.81746 0.7971 0.7971 0.7971 0.8062 0.8347 1740 0.8746 0.7947 0.7971 0.8115 $-$ 1740 0.8746 0.7947 0.7971 0.8115 $-$ 1751 0.9297 0.8746 0.86122 0.8633 0.8633 0.8687 1751 0.9297 0.8746 0.9360 0.9148 0.96333 0.9424 0.9235 0.92424 0.92365 0.9148 0.96518 0.9424 0.9142 0.9235 0.9360 0.93311 0.9518 0.9424 000 1.0335 1.0172 1.0172 1.0172 1.0172 1701 1.0335 1.0172 1.0172 1.0172 1.0172 1771 1.2004 1.1036 1.1038 1.0172 1.0036 1771 1.2757 1.2595 1.2551 1.2557 1.2556 1.2501 1.2561 1.2561 1.2561 1.2557 1.2556 1.2757 1.2563 1.2412 1.4150 1.4134 1.4102 1722 1.2561 1.2561 1.4526 1.4506 1.4506 1.471 1.4729 1.4729 1.4729 1.4526 1.4506 1.471 1.4729 1.4729 1.4729 1.4506 1.4506 1.471 1	IG Log P	306	375	444 Log	495 đ	578	633
1979 0,8047 0,7947 0,7947 0,7835 0,8062 0,8347 740 0,8746 0,7971 0,7971 0,8115 - 740 0,8746 0,8613 0,7971 0,8115 - 751 0,8746 0,8612 0,8115 - - 751 0,8746 0,8746 0,8512 0,8633 0,8687 0,8919 751 0,8746 0,8746 0,8746 0,8746 0,9148 0,9618 0,9424 7000 0,9424 0,9205 0,9311 0,9518 0,9424 0,9424 701 1,0172 1,0172 1,0172 1,00172 1,0000 1,0172 1,0000 771 1,0000 1,0172 1,0172 1,0172 1,0172 1,0172 1,0000 1771 1,0172 1,0172 1,0172 1,0172 1,0172 1,0000 1771 1,0204 1,0172 1,0172 1,0172 1,0172 1,0212 1,0172 1771 1,2204 1,0173 1,0172 1,0172 1,02557 1,0172<	396	59 0.70 78	0.6978	ł	0.7497	1	1
740 0.8746 0.7854 0.8013 0.7971 0.8115 $-$ 790 0.8853 0.8282 0.8512 0.8597 0.8633 0.8687 751 0.9297 0.8746 0.8512 0.8597 0.8633 0.8687 761 0.9297 0.8746 0.9360 0.9148 0.9424 700 0.9424 0.9207 0.8746 0.9360 0.9148 0.96333 701 1.0335 1.0460 1.0382 1.0172 1.0212 1.0000 761 1.0335 1.0172 1.0172 1.0212 1.0000 701 1.0335 1.0172 1.0172 1.0212 1.00664 771 1.0000 1.1243 1.1179 1.1118 1.1038 1.00664 771 1.2757 1.2595 1.1179 1.1188 1.1038 1.0264 771 1.2757 1.2595 1.2561 1.2557 1.2556 1.2556 782 1.2561 1.2561 1.2557 1.2556 1.3463 782 1.4720 1.4211 1.4150 1.4134 1.4102 792 1.4729 1.4729 1.4625 1.4506 1.4506 792 1.5566 1.5556 1.4526 1.4625 1.4506 792 1.5566 1.4222 1.4212 1.4150 1.4134 1.4102 792 1.55068 1.55115 1.55259 1.4506 1.4910	397	0.8047	0.7497	0.7947	0,7835	0,8062	0.8347
(990 0.8853 0.8282 0.8512 0.8597 0.8633 0.8633 0.8687 (751 0.9297 0.8746 0.8948 0.9148 0.9865 0.8919 (761 1.0335 1.0460 1.0382 1.0172 1.0212 1.0000 (701 1.0335 1.0460 1.0382 1.0172 1.0212 1.0000 (771 1.0335 1.0172 1.0172 1.0212 1.00644 (771 1.2004 1.1243 1.1179 1.1118 1.1038 1.0864 (771 1.2757 1.2564 1.2561 1.2555 1.0172 1.0722 (301) 1.2757 1.2564 1.2564 1.2555 1.2555 1.2555 (302) 1.2577 1.25561 1.25561 1.2555 1.2555 1.2555 (302) 1.4710 1.2555 1.2636 1.2555 1.4136 1.4102 (302) 1.4710 1.4270 1.4222 1.4256 1.4134 1.4102 (302) 1.4711 1.4720 1.4722 1.4126 1.4625 1.4506 (302) 1.4711 1.4720 1.4722 1.4720 1.4625 1.4506 (302) 1.4712 1.4722 1.4729 1.4625 1.4508 1.4506 (322) 1.4522 1.55252 1.4526 1.4506 1.4506 1.4506 (322) 1.4722 1.5215 1.4526 1.4506 1.4506 1.4506	574	40 0 . 8746	0.7854	0°8013	0.7971	0,8115	1
(751 0.9297 0.8746 0.8948 0.9148 0.8865 0.8919 000 0.9424 0.9205 0.9360 0.9331 0.9518 0.9424 761 1.0335 1.0460 1.0382 1.0172 1.0212 1.0000 1.0010 1.0030 1.0172 1.0172 1.0212 1.0000 1.0020 1.0266 1.0382 1.0172 1.0212 1.0064 1.771 1.2004 1.1243 1.1179 1.0172 1.0212 1.0064 1.771 1.2004 1.1243 1.1179 1.1118 1.0038 1.0864 1.771 1.2004 1.12656 1.2153 1.0172 1.0264 1.771 1.2004 1.12636 1.26561 1.2557 1.2556 1.2757 1.2563 1.2636 1.2561 1.25557 1.2556 1.2671 1.2561 1.2561 1.2553 1.4122 0031 1.4711 1.4270 1.4222 1.4211 1.4150 1.4134 1.4711 1.4930 1.4729 1.4625 1.4506 1.4506 7722 1.5502 1.55034 1.4506 1.4911 1.4506	669	0,8853	0.8282	0.8512	0.8597	0,8633	0.8687
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7611.03351.04601.03821.01721.02121.000610101.09001.12431.11791.11181.10381.086417711.20041.19561.11791.118751.17221.27571.25951.26361.18531.18751.17221.27571.25951.26361.25611.25571.25581.27571.25951.26361.25611.255611.25581.36711.35471.36041.36111.35391.34631.42701.42221.642111.441501.441341.44020001.47111.49301.47291.46251.45981.45067921.55681.55151.55151.553391.4506	8	0.9424	0, 92 05	0.9360	0,9831	0,9518	0.9424
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1771 1,2004 1,1936 1,1958 1,1853 1,1875 1,01722 1,021 1,2757 1,2595 1,2636 1,2561 1,2557 1,2558 1,021 1,2757 1,2595 1,2636 1,2561 1,2557 1,2558 1,782 1,3671 1,3547 1,3604 1,3611 1,3539 1,3463 1,782 1,4711 1,4270 1,4222 1,44150 1,44134 1,4102 0000 1,4711 1,4930 1,4729 1,4625 1,4598 1,4506 0792 1,5068 1,5215 1,5115 1,5259 1,5034 1,4911	ы Б	1,0900	1.1243	1.1179	1.1118	1.1038	1.0864
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782 1.3671 1.3547 1.3604 1.3611 1.3539 1.3463 0031 1.4270 1.4222 1.4211 1.4150 1.4134 1.4102 0000 1.4711 1.4930 1.4729 1.4625 1.4598 1.4506 0792 1.5068 1.5252 1.5115 1.5259 1.5034 1.4911	602	1,2757 1,2757	1.2595	1.2636	1.2561	1.2557	1.2558
1031 1.4150 1.4134 1.4102 1000 1.4711 1.4930 1.4729 1.4625 1.4598 1.4506 1002 1.5068 1.5252 1.5115 1.5259 1.5034 1.4911	778	1.3671	1.3547	1°3604	1.3611	1.3539	1.3463
0000 1.4711 1.4930 1.4729 1.4625 1.4598 1.4506 0792 1.5068 1.5252 1.5115 1.5259 1.5034 1.4911	903	1.4270 [.]	1°4222	1 °421 1	1.4150	1.4134	1.4102
792 1.5068 1.5252 1.5115 1.5259 1.5034 1.4911	80	1.4711	1.4930	1.4729	1.4625	1.4598	1.4506
	610)2 1.5068	1.5252	1,5115 1	1,5259	1°5034	1.4911

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		Т. Т	ble A-2			
QUENCHING TEMPERATURE T _û K	306	375	444	495	578	633
LOAD P gm		VICKERS HARDN	ess number h _v	= 1854.4 P/d ²	Kg - mm ⁻²	,
1.25	88 . 66	93•23	ł	73.41	9	I
2,50	113,94	146.81	119.29	125.62	113.18	99.25
3.75	123.88	186.87	173.65	177.04	165.51	
5.0	157.27	204 ° 52	183,93	176,92	173.99	169.72
7.50	192°27	247.75	225.69	2.05,93	234.57	228.83
10.0	241 °76	267 _° 39	249,00	2 00° 44	231 50	241.76
15.0	238,38	225.06	233,26	257 . 01	252 . 29	277.05
20.0	245.07	209.21	215.45	221.68	229 . 94	249.15
30°0	221 °09	228,08	225.69	236 . 95	234.57	251.70
40°0	208.40	224 . 56	22D.20	228.05	228,20	228.37
60° C	205.16	217.27	211.50	210,94	218,03	225.78
80°0 .	207.63	212.27	213,30	219,38	221.12	224.30
100.0	211 °79	207.39	210,00	220,38	223.01	232,80
120.0	215.67	198.12	211.05	197.53	219.10	231.82

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H^ Kg-mm²

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(Bhagia 1983). The theoretical conclusion that hardness is independent of load is true only at higher loads. The maximum value of hardness corresponds with a load which is nearer the value of the load at which the kink in the graph of log P versus log d is observed (Panchal 1981). The graph of H_v versus P can be conveniently divided into three parts OA, AB and BC where the first part represents linear relation between hardness and load, the second part, the non-linear relation and the third part the linear one. It should be noted that there is a fundamental difference between linear portions OA and BC of the graph OABC. This possibly reflects varied reactions of the cleavage surface to loads belonging to different regions. Besides it supports, to a certain extent, the earlier view about the splitting of the graph of log P versus log d into two recognizable lines (Panchal 1981).

The complex behaviour of microhardness with load can be explained on the basis of the depth of penetration of the indenter. At small loads the indenter penetrates only surface layers, hence the effect is shown more sharply at those loads. However, as the depth of impression increases, the effect of surface layers becomes less dominant and after a certain depth of penetration, the effect of inner layers becomes more and more prominent than those of surface layers and ultimately there is practically no change in value of hardness with load.

A.3.3 Relation between hardness and temperature of

quenching

It is clear from the observations of hardness of quenched and unquenched samples (Table A-2) that hardness depends upon the quenching temperature (T_Q) . Hardness in high load region (HLR) is independent of load. Hence average values of hardness (\overline{H}) in high load region are computed and are recorded in Table A-3. Fig. A-7 shows the plot of log (\overline{H} T_Q) versus log T_Q. The plot is a straight line. The straight line follows the equation,

$$\log \widehat{H} T_{O} = m \log T_{O} + \log C$$
 (4)

where m is the slope and C is a constant. Therefore,

$$\frac{1}{H} T_{\mathbf{Q}} = C \qquad \dots \qquad (5)$$

or,

$$\overline{H} \mathbf{T}_{\mathbf{Q}}^{k} = C \qquad \dots \qquad (6)$$

where k = 1 - m. The value of k is - 0.11 for Indium Antimonide (Table A-3). It is clear from table that Vickers hardness number in HLR increases with temperature of quenching. However the percentage increase in hardness with respect to hardness at room temperature (306°K) is quite small. TABLE A-3

QUENCHING TEMPERATURE TOK	Log T _{&}	AVERAGE HARDNESS IN HLR Ĥ kg. mm ⁻²	Log H T _e	$H T_{\alpha}^{-0.11} = C$	% DEVIATION OF C FRCM MEAN VALUE	% DEVIATION OF C FROM VALUE OF C FROM GRAPH
306	2.4857	209.73	4 . 8073	01.111	+ 1,230	+ 0•234
375	2.5740	211.92	4,9000	109.76	600°0 +	- 0.974
444	2.6474	213.21	4 . 976 1	108,38	- 1.240	- 2.219
495	2.6946	215.25	5 ° 0275	108.10	- 1,503	- 2.472
578	2.7619	221.80	5.1071	109.49	- 1.237	- 1.217
633	2.8014	228.61	5.1605	111.72	+ 1.790	- 0°794
		<u>.</u>				

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It is desirable to ascertain how far the relation $H T_Q^k$ = constant is true for individual observations on quench hardness. This constant is designated by C (Table A-3). The percentage debiate of C from mean value and from graph are small. From empirical formula for Vickers hardness number it is obvious that hardness number, inversely proportional to square of the diagonal of the indentation mark for a constant load. Since hardness depends on temperature of quenching, the diagonal length of the indentation mark would also depend on the temperature of quenching. Thus

$$H = R (P/d^2)$$
 (7)

where R is a constant depending upon the geometry of the indenter. Further,

$$H T_Q^k = C \qquad \dots \qquad (8)$$

Combination of above equations gives,

$$\frac{\mathbf{P} \mathbf{T}_{\mathbf{Q}}^{\mathbf{k}}}{d^2} = \frac{C}{R} = \text{Constant} = S \dots (9)$$

or,

$$\frac{T_Q}{d^2} = S \dots (10)$$

$$\frac{T_{Q}}{d^{2}} = \frac{s}{p} - T_{Q}^{1-k}$$
 (11)

or

$$\log \frac{T_Q}{d^2} = \log (S/P) + (1 - k) \log T_Q \dots (12)$$

Let
$$S/P = A$$
, $m_1 = (1 - k)$ (

Hence equation (12) becomes

$$\log \frac{T_Q}{d^2} = m_1 \log T_Q + \log A \dots (13)$$

$$\frac{10}{d^2} = S/P = A$$
 (14a)

Utilization of equations (7), (8) and (14) gives,

$$\frac{T_0}{d^2} = \frac{C}{RP} \qquad \dots \qquad (14b)$$

It is obvious from the above equation 13 that for a given applied load if a graph of log (T_Q/d^2) is plotted against log T_Q , the slope of graph will be (1 - k). However if this is repeated for several applied loads, it is evident from the above equation that graph of log (T_Q/d^2) versus log T_Q should consist of straight lines parallel to one another having slope (1 - k) and different intercepts. Further, the slope of plot (Fig. A-8 ; Table A-4) is 1.11

$$i_{\circ}e_{\bullet} = 1 - k = m_1 = 1.11$$

Hence the value of k is - 0.11 which is identical with the value of the exponent k in the equation (8) connecting hardness number and quenching temperature.

It was reported that'a' and 'n' are constants and the straight line represented by the plot of log P versus log d consisted of two straight lines with slopes n_1 and n_2 and intercepts 'a₁' and 'a₂' respectively (Panchal 1981 Table A-5). The combination of equations 1 and 9 yields

$$a_2 a^{n_2} T_Q^{k} = S \dots (15)$$

substituting

in eq. 14a, one gets

Since n_2 is not having an integral value, it is necessary to have a different approach. If graph of

		·	Table A .4			
QUENCHING TEMPERATURE T ^Q K	306	373	444	495	578	633
LOAD P gm				Log (T_Q/d_v^2)		
40	1.9342	0,0555	0,1201	0.1823	0.2504	0,2890
60	<u>1</u> ,7518	1.8650	1.1265	0,7175	0,0541	0,1087
80	1.6316	1.7318	Ĩ.8051	<u>1</u> .9723	Ĩ.9350	<u>1</u> ,9809
100	Ĩ.5434	I.•5879	1.7016	<u>1</u> ,8646	I.8429	Ĩ.9002
120	I.4722	Ĩ . 5236	1.6244	1.7698	1.7551	1.8192
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Table A-5

QUENCHING			LOW LOAD REG.	HIGH TO	AD REG.	1	-0.08	
TEMPERATURE Tor Tor	Log T	'n	a ₁ x 10 ⁻³	2 2	3 3 3	$\operatorname{Log}(a_2 \Re)$	a2 ^T a = D.	LOAD AT KINK P gm
306	2.4857	3.75	2。44	1.9	0.1555	1.6774	0•09837	10
375	2.5740	3.42	7.08	1.9	0.1558	1.7748	0, 09883	10
444	2.6474	3,52	5.14	1.9	0.1596	1.8504	00 860 °C	10
495	2.6946	3,33	6.94	1.9	0.1639	1,9092	0, 09977	IO
578	2,7619	3,87	2°27	1.9	0.1650	1.9794	0*09920	15
633	2 , 8014	4 •8	0.29	1.9	0.1674	2,0251	16660*0	15
·								

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log $(a_2 d^2/T_q)$ versus log T_q is plotted, it consists of a series of parallel lines corresponding to different intercepts (Table A-6; Fig. A-9). Thus each straight line follows the general equation,

$$\log \frac{a_2}{T_0} d^2 = m_2 \log T_0 + \log B \dots (18)$$

Slope of each straight line is $m_2 = -1.031$. Simplification of the above equation yields,

$$a_2 a^2 T_Q^{0.031} = B$$
(19)

Combination of above equation with eqn. (1) gives

$$P d^2 - n_2 T_0^{0,031} = B$$
 (20)

Comparison of Meyer's law (eqn. 1) with formula for hardness (eqn. 3) clearly suggests that the constant 'a' and hardness number are related. Inspection of the variation of various functions involving H, a_2 and T_Q indicates that graph of log ($\overline{H} T_Q/a_2$) versus log T_Q would be a straight line following the equation,

 $\log \frac{\overline{H} T_{Q}}{a_{2}} = m_{3} \log T_{Q} \neq \log E \qquad \dots \qquad (21)$

where slope is given by $m_3 = 1.022$ and E is constant. This plot for Vickers hardness numbers is presented in fig. A.10 (Table A.7).

enching Iperature T _r ok	306	373	444	495	578	633
coat) gm			Log (a ₂	d_v^2/T_{λ}		
40	<u>1</u> ,2573	í.1433	1.0812	í "0324	2,9669	2.9327
60	I.4398	Ĩ, 3335	<u>1</u> 。2748	1• 2425	1.1630	<u>1</u> .1136
80	<u>1</u> .5598	1.4668	<u>1</u> ,3962	<u>1</u> .3501	1 .2824	<u>1</u> ,2418
00	1 •6481	Ĩ •6105	Ĩ.4998	$\overline{1}_{\bullet}4450$	<u>1</u> 。3751	<u>]</u> 。3224
20	1017.1	1.6749	1 °5769	<u>1</u> .5718	1.4622	1.4034

Table A-6

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Table A-7

QUENCHING TEMPERATURE T _Q ^O K	$\mathbf{rog} \ \mathbf{T}_{\mathbf{Q}}$	Log H	$\log (a_2^{H})$	$\operatorname{Log} \left(\frac{\overline{H}}{a_2} = \frac{\underline{T}_2}{2} \right) = E$	$= (\bar{H} \bar{T}_{Q}/a_{2})$
306	2.4857	2,3216	1 . 5134	5.6157	1185.09
375	2,5740	2 • 32 61	1.5270	5*7 015	1167.21
444	2.6474	2 • 3 2 8 8	1.5318	5 °7748	1163 。 97
495	2.6946	2.3329	1 •5475	5.8127	1141.47
578	2.7619	2 °3459	1,5634	5 . 8905	1164.28
633	2,8014	2,3591	1,5828	5 . 9378	1180,31
,					



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Substitution of
$$a_2 = (P/d^2) d^2 - n_2$$
 and

$$H = R(P/d^2)$$
 in eqn. (21)

yields,

$$E = R T_Q \qquad d^2 \qquad \dots \qquad (22)$$

simplifying eqn.21,gets

$$\frac{\overline{H} \quad T_{Q}}{a_{2}} = E \quad \dots \quad (23)$$

i.e.

,

$$\frac{\tilde{H} T_{0}}{a_{2}} = E \dots (24)$$

multiplication of eqn.17 with eqn.20 gives

,

$$\mathbf{T}_{Q}^{\mathbf{k}} \cdot \mathbf{a}_{2}^{\mathbf{n}} \mathbf{d}^{\mathbf{2}} \cdot \mathbf{P} \mathbf{d}^{\mathbf{2}} \cdot \mathbf{T}_{Q}^{\mathbf{0} \cdot \mathbf{0} 3 \mathbf{l}} = \mathbf{A} \mathbf{B} \times \mathbf{P}$$

or,

$$(k + .031)$$

$$= AB = Constant \dots (25)$$

Thus the intercepts a_2 could be associated with the quenching temperature. This can also be understood from a graphical plot of log $(a_2 T_Q)$ versus log T_Q which follows the equation,

$$\log (a_2 T_Q) = m_4 \log T_Q + \log D \qquad \dots (26)$$

The graph of log $(a_2 T_Q)$ versus log T_Q is shown in figure A-11 (Table A-5).

Thus,

$$a_2 T_Q = D$$
 (27)

where m_A is the slope of fig. A-ll and equals 1.08.

Hence the above equation becomes

$$a_2 T_0 = D$$
 (28)

substitution of

$$a_2 = P/d^2 \cdot d$$
 in equation (27)

yields,

$$-\frac{P}{d^2} \cdot \frac{2 - n_2}{d} \cdot \frac{1 - m_4}{T_Q} = D \dots (29)$$

Slight dependence of a_2 on quenching temperature can be expected because value of a_2 (Table A-5) is quite small. It is suggested from the form of eqn. (1) and eqn. (7) that there must be some relation between hardness number and a_2 . After considering several functions containing H and a_2 it was found that the plot of log (a_2 \overline{H}) versus



log \widehat{H} gives a better straight line, obeying the general equation

 $\log (a_2 \tilde{H}) = m_5 \log \tilde{H} + \log F$ (30)

Hence,

$$a_2 H^{-1} = F$$
 (31)

slope of the above plot (fig. A-12) is given by $m_5 = 1.779$. Substitution of value of m_5 in eqn. 31 gives,

$$a_2 = F$$
 (32)

This shows very clearly that hardness number and the intercept of the straight line (cf. fig. A-12) corresponding to HLR are intimately connected, thereby indicating connection Meyer's law with hardness number.

It is interesting to examine the accuracy of each observation in the above plots by considering the coefficient of variation for different constants associated with different equations mentioned above.

The values of A, B, E and D are compared for each observation using equations 17, 19, 22, 29 respectively and are presented in Tables A-8.1 to A-8.6. For these



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D_{m} $A \times 10^{-3}$ B $AB \times 10^{-2}$ $D \times 10^{-2}$ P/B $AF \times 10^{-2}$ E $(DE/1854.4) \times 10^{-1}$ 0 1.544 64.07 9.892 9.536 0.6243 6.176 1214.6 6.246 0 1.008 98.15 9.892 9.588 0.6113 6.047 1189.3 6.149 0 0.745 132.1 9.891 9.838 0.6029 5.964 1173.1 6.309 0 0.745 132.1 9.892 10.14 0.5968 5.964 1173.1 6.323 0 0.590 167.6 9.892 10.41 0.5919 5.856 1151.7 6.323 0 0.488 202.7 9.8928 10.41 0.5919 5.856 1151.7 6.347 M - - 9.8928 10.41 0.50919 5.8994 1177.98 6.2548					ROOM TEMPERAT	URE 306	×		
0 1.544 64.07 9.892 9.536 0.6243 6.176 1214.6 6.246 0 1.008 98.15 9.892 9.588 0.6113 6.047 1189.3 6.149 0 0.745 132.1 9.891 9.638 0.6013 6.047 1189.3 6.149 0 0.745 132.1 9.891 9.638 0.6029 5.964 1173.1 6.309 0 0.590 167.6 9.892 10.14 0.5968 5.904 1161.2 6.233 0 0.590 167.6 9.892 10.141 0.5919 5.856 1151.7 6.233 0 0.488 202.7 9.892 10.41 0.5919 5.856 1151.7 6.347 M - - 9.8918 9.9024 0.6054 5.9894 1177.98 6.347	Ő E	A × 10 ⁻³	Ø	AB x 10 ⁻²	D x 10 ⁻²	P/B	AP x 10 ⁻²	E E	(DE/1854.4) x 10
0 1.008 96.15 9.892 9.588 0.6113 6.047 1189.3 6.149 0 0.745 132.1 9.891 9.838 0.6029 5.964 1173.1 6.309 0 0.590 167.6 9.892 10.14 0.5968 5.904 1161.2 6.309 0 0.590 167.6 9.892 10.14 0.5919 5.856 1151.7 6.347 0 0.468 202.7 9.892 10.41 0.5919 5.856 1151.7 6.347 0 0.468 202.7 9.892 10.41 0.5919 5.856 1151.7 6.347 M - - 9.8918 9.9024 0.6054 5.9894 1177.98 6.2548		1.544	64.07	9,892	9•536	0.6243	6.176	1214.6	6 • 246
0 0.745 132.1 9.691 9.636 0.6029 5.964 1173.1 6.309 0 0.590 167.6 9.892 10.14 0.5968 5.904 1161.2 6.323 0 0.468 202.7 9.892 10.14 0.5919 5.856 1151.7 6.347 0 0.468 202.7 9.892 10.41 0.5919 5.856 1151.7 6.347 1 - - 9.8918 9.9024 0.6054 5.9894 1177.98 6.2548	0	1,008	98.15	9 . 892	9 • 588	0.6113	6.047	1189.3	6•149
0 0.590 167.6 9.892 10.14 0.5968 5.904 1161.2 6.223 0 0.488 202.7 9.892 10.41 0.5919 5.856 1151.7 6.347 M - - 9.8918 9.9024 0.6054 5.9894 1177.98 6.2548	0	0 . 745	132,1	168.6	9.838	0.6029	5.964	1173.1	6•309
0 0.488 202.7 9.892 10.41 0.5919 5.856 1151.7 6.347 M - - 9.8918 9.9024 0.6054 5.9894 1177.98 6.2548	۰ ۲	0*590	167.6	9,892	10,14	0.5968	5.904	1161.2	6.223
M 9,8918 9,9024 0,6054 5,9894 1177,98 6,2548		0 . 488	202.7	9.892	10,41	0*5919	5.856	1151.7	6.347
	NN	1	B	9,8918	9.9024	0.6054	5,9894	1177.98	- 6•2548
							,		

		•4) x 10 ⁻²	. 06	77	30	21	15 .	220
-		(DE/1854	Q.	6. 3	6.2	0°. 0	. 8 .0	
		臼	1213.6	1187.3	1168.9	1150.1	1141.6	1172 2
	375°K	AP x 10 ⁻²	6.188	6.056	5.962	5 5 . 866	5.822	5 0700
Б А.8 .2	nperature	₽/B	0.6226	0.6091	0.5997	0.5900	0.5856	7 10 9 0
TABL	QUENCHING TO	D x 10 ⁻²	10.01	9.961	9,884	9.064	9.447	0 2050
_ 1		AB x 10 ⁻² .	9.938	9441	9.941	9.941	9.941	
		ф,	64 . 24	9 8 . 49	133.4	169.5	204.9	
		A x 10 ⁻³	1.547	1.009	0.745	0.587	0.485	1
•		LOAD P gm	40	60	80	100	120	MEAN

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				DUENCHIN	G TEMPERAT	URE 444 ⁰ K		
P gm	$\lambda \times 10^{-3}$	'a	ÅB x 10 ⁻²	$\dot{D} \times 10^{-2}$	₽,⁄В	AP x 10 ⁻²	, 업	(DE/1854.4) x 10 ⁻²
40	1.525	64 .648	9.859	9.757	0.6188	6.101	1207.8	6 . 355
60	0.994	99.14	9.859	9,585	0.6052	5.967	1181.2	6.1.05
80	0.736	134.1	9.859	661.6	0.5968	5.884	1164.8	6.155
100	0.581	169.6	9.859	9.765	0.5897	5.814	1151.0	6.061
120	0.480	205.3	9.858	9*898	0.5845	5.762	1140,8	6•089
MEAN			9.8588	9.7608	0.599	5.9056	1169.12	6.153

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	And the second			DENCH11	NG TEMPERA	TURE 495 ⁰ K		
СКО Шб	A x 10 ⁻³	i i i i i i i i i	ÁB x 10 ⁻²	D x 10 ⁻² ,	₽/B	áp x 10 ⁻²	·臼	`(DE∕1854.4) × 10
40	1.550	64.74	10,03	966*6	0.6178	6.200	1206.8	6.505
60	1.009	96°38	9.987	9.472	0.6062	6.054	1178.1	6.017
80	0.747	134.32	10.04	9.975	0.5956	5.978	1163.6	6.259
oa	0.591	169.7	10,05	10.13	0.5891	5.914	1150,9	6.287
20	0 . 486	206.7	10,04	9.213	0.5806	5.827	1134.3	5.635
EAN		Na - Serriger Berger gergen Berger	10.0274	9.7572	0.5978	5.9946	11.66.74	6.1406

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Ì				QUENCHIN	G TEMPERA	rure 578°K		
A	x 10 ⁻³	μ	AB x 10 ⁻²	D x 10 ⁻²	₽/B	ÅP x 10 ⁻²	្មជ	(DE/1854.4) x 10 ⁻²
-	•535	65.05	9,983	9.888	0.6149	6 .1 38	1202 .8	6.413
	• 000	99,81	9.982	9.654	0.6012	100*9	1175.9	6.122
0	0.740	134.9	9,982	9,921	0.5929	5.919	1159.9	6 • 2 ° 5
Ŷ	0.586	170:4	9 ° 98 3	10 °12	0,5866	5.856	1147.6	6.252
0	• 483	206.6	9,983	10,04	0.5808	5.798	1136.2	6.151
	I	n ann Mar Briddan Ain An Mar An Mar B	9,9822	9.9246	0.59528	5.9424	1164.48	6.2306
			والمتعاونين والمعارفة المحادثة المحادثة والمحادثة والمحادثة والمحادثة والمحادثة والمحادثة والمحادثة والمحادثة	ter men signer, Striv Statementers des anderes Store Store Store				

				QUENCHI	ING TEMPER	ATURE 633 ⁰ K		
ьодр Р дт	A x 10 ⁻³	ра,	AB x 10 ⁻²	рх 10 ⁻²	P/B	.AP x 10 ⁻²		(DE/1854.4) x 10 ⁻²
40	1.541	65 • 23	10.05	9 . 813	0.6132	6.165	12.00.3	6.357
60	1 •006	99. 91	10 , 05	1 06°6,	0.605	6 • 038	1175.6	6.281
80	0.744	135.2	10,05	0 9 •86	0.5918	5.949	1158.4	6.239
100	0,589	170.6	10,05	1.0,46	Ò •5863	5.895	1147.7	6.473
120	0.487	206.6	10,05	10.51	0,5808	5.840	1137.1	6.444
MEAN	1		10.05	10.1356	0.5954	5.9774	1163.82	6 . 3588

			TABLE A.9				
× 10 ⁻² D ×	10-2	AP x 10 ⁻²	(DE 1854.4) x10 ⁻²	P/B	E	U	a⁄o
8918 9.9	9024	5.9894	6.2548	0.6054	1177.98	111.10	1121.95
9404 9.6	6852	5.9788	6.1266	0.60 1 4	1172°30	109 ¢38	1138.36
.8588 9.7	76.08	5.9056	6 .1 530	0.5990	1169°12	108,38	1110.36
.0274 9.7	7572	5,9946	6.1406	0.5978	1166.74	108.10	1107 _{.89}
.9822 9.5	9246	5 9 424	642306	0.5953	1164.48	109.49	1103.22
• 05 10.1	1356	5.9774	6 . 3588	0.5954	1163 。82	111.72	1102.25
	1999 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 - 1997 -	a se	والمحافظ والمحافظ المحافظ المحافظ والمحافظ والمحافظ والمحافظ والمحافظ والمحافظ والمحافظ والمحافظ	an nya Sundana Arts Airi Sundan Sustainin a	allen dies en gleen die Volle zur Gitt Alle de Kinze Gitte. Some China die		
•9584 0 9.6	8609	5 • 9647	6.2107	0 . 5991	1169.07	109.758	1113.15
.737 1.5	553	1.291	1.3494	0.6103	0.4821	1.2229	1.0529
893 <u>8</u> 9.6	862	5 • 8954	6 • 208	0.5958	1167.4	110.84	1129.0

tables the above equations are rewritten here in sequence with new equation numbers.

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$$A = -\frac{1}{P} - \frac{1}{P} - \frac{n_1}{a_2 d} - \frac{n_2 - 2}{a_2 d}$$
 (33)

$$B = P \cdot d \cdot T_Q^{0.031} \cdot \dots \cdot (34)$$

$$-\frac{P}{B^{-}} = -\frac{\frac{n_2}{2} - 2}{\frac{1}{\sqrt{0.031}}}$$
 (35)

$$AB = \frac{P \mathbb{T}_{Q}^{k} + 0.031}{d^{2}} \dots \dots (36)$$

$$AP = T_{Q} \cdot a_{2} \cdot d^{2} \cdot \dots \cdot (37)$$

$$D = -\frac{P}{d^2} \cdot \frac{2 - n_2}{d} \cdot \frac{1 - m_4}{T_Q} \quad \dots \quad (38)$$

$$E = R T_Q \qquad \frac{1 - m_3}{d^2} \qquad \frac{n_2 - 2}{d^2} \qquad \dots \qquad (39)$$

$$DE = -\frac{RP}{d^2} - \frac{T_Q}{T_Q}$$
 (40)

The mean values of constants are summarised in Table A-9.

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A careful study of mean values of 'constant' and

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their deviations from the corresponding observations clearly indicates that the deviations are within experimental errors. A glance at Table A-9 shows that

$$D = AB (41)$$

$$AP = DE/R (42)$$

$$E = C/D (43)$$

Thus for all loads in HLR, the variation of hardness number H and the variation of hardness constant a_2 with temperature of quenching and also with each other follows the equation,

$$H T_{Q}^{k} = C = Constant \dots (44)$$

$$a_{2} T_{Q}^{r} = D = Constant \dots (45)$$

$$a_{2} H^{S} = F = Constant \dots (46)$$

where k, r and s are numbers numerically less than unity. The signs for these constants decide the nature of the crystal. For Indium Antimonide they are negative as shown above. The constants in above equations have different values. Further quenching can also be carried out by bringing a crystal from very low temperature to room temperature. Thus for $T_{\Omega} = 1^{O}K$,

XVIII

Н		Constant	* * * * *	(47)
a ₂	Ħ	Constant	••••	(48)

These values can be considered to characterize a crystalline material. Thus for Indium Antimonide, the quench hardness number and quench hardness constants are given by

> H = $109.758 \text{ kg} - \text{mm}^{-2}$ (49) a = $9.95 \text{ kg} - \text{mm}^{-2}$ (50)

A.4 GRAPHICAL ANALYSIS OF OBSERVATIONS

It is clear from the general information on graphical analysis (cf. Chapter III) that there are basically 5 methods to obtain the estimation of the best straight line. In the present work the calculated values of hardness (represented by hardness number) in the high load region are combined with those of observed values of quenching temperature T_Q for obtaining a straight line plot between the variables $\log H T_Q = y$ and $\log T_Q = x$ (cf. Table A.3 and fig. A.7). The total number of observations in this table is six. The mean values of x ana y are given in table A.0, The two pairs of extreme observations alongwith their spread from the corresponding mean values are given in this table. Further while drawing a graph by visual estimation the values of the pairs of extreme observation slightly change. These are also recorded alongwith a percentage change in their values from the slope of the straight line plot (fig. A.7). It is clear from this percentage change in the slope value that the quenching temperatures viz. 306 and 633° K corresponding to those extreme observations have a significant effect on the slope value. This also suggests that these observations are unusual in the sense that inspite of taking all possible precautions in experimental work the same set of observations are obtained. The reasons are not yet clear.

Zero sum method is used to calculate the slope (cf. equation 3.3, Chapter III). It is noticeable that percentage change in the value of the slope from the one obtained from the plot (fig. A.7) is zero (Table B). Centroid methods are used to determine the slope for comparing it with the one obtained from the actual straight It should be remarked that the conditions viz. line. total number of observations and equal spacing of actually known variable which form the basis for calculating the slope, are not fully satisfied. However the percentage change in the value of the slope from the one actually determined from the graph is within experimental errors (Table C). In the method of estimation of best straight line by data used in specific manner, namely equal spacing

of independent variable and multiplication of each straight line equation by the first 6 natural numbers in the sequence 1, 2, 3 6, the conditions are not fully satisfied. However the difference between calculated value and graphical value of the slope is zero (Table, D). The statistical estimation of best straight line gives the regression coefficients (cf. equations 3.30 and 3.35, Chapter III) which are the calculated values of slopes. The percentage change of these values from the actual A^{10} value of slope (Table, E) are small and within experimental errors.

The results of all these methods indicate that the experimental observations are reliable for carrying out the analysis. However the results of the first method, namely visual estimation of best straight line presents doubts about the reliability of the observations corresponding to quenching temperatures (306 and 633°K). Since the author is confident about the observations presented here, it is likely that these unusual observations may be due to some unknown factor(s) operating in the physical processes of InSb or inadequacy of the empirical formulae for hardness used in the present work or conceptual and theoretical deficiencies about hardness.

A.5 CONCLUSIONS

(i) The defect structures operate differently in low and high load regions.

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- (ii) Hardness depends upon quenching temperatures.
 Relations between hardness and quenching temperature in the high load region is given by
 - (a) $H T_Q^k$ = Constant, where k = -0.11 for InSb crystal
 - (b) $a_2 T_Q^r$ = Constant, where r = -0.08 for InSb crystal
 - (c) $a_2 H^S = Constant$, where s = -0.779 for InSb crystal.
- (iii) The above conclusions are in general agreement with those obtained for single crystals of KCl, KBr, CaCO₃, NaNO₃, Zn and TGS.

REFERENCES

1.	R.K.	Willa	rdson	anđ	S	emi	ico	nd	luctor	cs a	and	Semi-	
	A.C.	Beer	(Eds)		m	ieta	ls	5,	Vol.	1,	Phy	sics	of
					I	II		A	compo	ound	ds,	1966,	,

Academic Press.

- 2. R.K. Willardson and A.C. Beer (Eds) Semiconductors and Semi metals, Vol. 2, Physics of III - V Compounds, 1966, Academic Press.
- 3. R.K. Willardson and A.C. Beer (Eds) Vol. 3, Optical properties of III - V Compounds, 1967, Academic Press.
- 4. R.K. Willardson and Vol. 4, Physics of III V A.C. Beer (Eds) Compounds, 1968, Academic Press.
- 5. R.T. Shah
 6. C.T. Acharya
 Ph.D. Thesis, M.S. Univ. of Baroda, Baroda, 1976.
 Ph.D. Thesis, M.S. Univ. of Baroda, Baroda, 1978.
- 7. L.J. Bhagia Ph.D. Thesis, M.S. Univ.

Ph.D. Thesis, M.S. Univ. of Baroda, Baroda, 1982.