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CHAPTER - VII

VARIATION OF LOAD WITH DIAGONAL LENGTH OF INDENTATION MARK.

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7.1 INTRODUCTION :

A variety of useful tests have been devised wherein some kind of mechanical operation is performed on the surface of the specimen. Quantities measured by these surface tests are generally associated with the term 'hardness'. Hardness as applied to amorphous and crystalline materials has long been the subject of discussion amongst engineers, physicists, metallurgists and minerologists and there are all sorts of conceptions as to what constitute hardness. The overwhelming difficulty of defining hardness is that it does not appear to be fundamental property of material. There is no universally accepted single test for hardness applicable to all materials. Thus there is hardness as measured by resistance to cutting, by scratching, by penetration, by electrical and magnetic properties (Mott, 1956). The fundamental physics of hardness is not yet clearly understood. The present work is taken up with the express purpose of critically reexamining the various formulae connected with hardness by systematically studying 'microhardness' of natural calcite crystals. It is an extension of the work reported earlier (Shah 1976, Acharya 1978). Further, it also aims at comparing the hardness values and behaviour using two types of diamond pyramidal indenters viz. Vickers and Knoop indenters. As far as the author is aware no such systematic work on ionic crystals is reported so far. Shah (1976) reported the systematic work on calcite cleavage faces using Vickers diamond indenter alone. No attempt has yet been made to compare hardness values of crystals (pre-treated and heat-treated) using both types of indenters. In what follows the terms 'hardness' and 'microhardness' of crystals are used to indicate the same meaning.

7.2 EXPERIMENTAL :

Natural crystals of calcite obtained from different localities such as Pavagarh, Chhota Udaipur (Gujarat State) and Rajasthan were used for the purpose of present study. Since the crystals from different localities contained different types and concentration of impurities, small crystal cleavages from a big block of rhombohedral calcite were used in the present investigation. Every time freshly cleaved surfaces were used for hardness studies. Further, cleaved crystals of approximately equal sizes are used so that a comparision of treated and untreated samples can be easily made without introducing other factors. Freshly cleaved blocks having dimensions 10 mm x 10 mm x 2 mm were fixed on glass plates with an adhesive. The levelling of the specimens was tested by using a table microscope. The hardness tester described in Chapter II was used to produce indentations on cleaved surface by using square-based vickers pyramidal and rhomb-based knoop pyramidal indenters. The filar micrometer eyepiece was used to measure the surface dimensions of the indentation marks. In order to avoid the influence of one indentation mark on the other, the distance between two consecutive indentations was maintained at a minimum of eight times the diagonal length of the mark and the indentation time for all specimens was kept 15 seconds. The load was varied from 2.5 gms. to 160 gms. for all temperatures. Care was taken to see that errors introduced during the work of indentation and measurements of the dimensions of the indentation marks are avoided or minimized. The indentation marks were produced by diamond indenters on the surface in such a way that one of their diagonals always remained parallel to [110] direction on the crystal surface. It should be mentioned here that the indentations were produced by knoop and Vickers indenters on the same sample to facilitate comparision. Due to non-availability of a hot stage and optical components of microscope to be used with hot stage in hardness tester, the indentation work was carried out at room temperature for annealed and/or quenched crystals for studying the variations of hardness with temperature. For these experiments, crystals of approximately equal sizes were used. They were gradually raised to a desired temperature and kept at this temperature for identical periods running into a few hours (24 hours in the present case). They were then quenched to room temperature. The quenching rates were made as high as possible and were adjusted so that the quenched crystals maintained their shapes. In the present case the rate of quenching varied from 1.6°C/sec. to 11.6°C/sec. These experiments were conducted upto a temperature of 500°C because beyond this temperature calcite begins to decompose into CaO and CO_2 as shown by work on thermal etching of calcite cleavages (Mehta, 1972) and on electrical conductivity of calcite (Shah, 1976).

7.3 OBSERVATIONS :

The diagonals of the indentation marks produced by various loads were measured. Several sets consisting of a large number of observations on freshly cleaved surfaces of calcite indented by various loads at room temperature were taken and a typical set of observations, recorded in Table 7.1, was studied graphically by plotting log P versus log d. P is the load in grams and d is the average value of the diagonal of the indentation mark in microns (Fig. 7.1), 7.2, 7.3, 7.4 and 7.5). It should be noted that irrespective of the magnitude of the load the impressions of the indent marks on cleavage surface of calcite are geometrically similar. (Fig. 7.6 and 7.7).

TABLE - 7.1

log P	log d					
	303°K		573°K		623°K	
	log dk	log dv	log dk	log dv	log dk	log dv
0.3979	1.3791	0.9849	1.4014	0.9599	1.3791	0.9294
0.5740	1.4500	0.9731	1.9572	1.0112	1.4424	1.0001
0.6989	1.5097	1.0546	1.4745	1.0414	1.4983	1.0253
0.8750	1.5153	1.1003	1.5317	1.1089	1.5208	1.1089
0.9420	1.5420	1.1311	1.5411	1.1511	1.5326	1.1346
1.0000	1.6009	1.1673	1.5775	1.1583	1.5677	1.1464
1.0969	1.6009	1.1914	1.5963	1.2041	1.5775	1.1821
1.1761	1.6232	1.2360	1.6055	1.2482	1.6055	1.2175
1.3010	1.6951	1.3015	1.7001	1.2811	1.6800	1.2787
1.4771	1.7816	1.3636	1.8327	1.3692	1.7993	1.3804
1.6020	1.8736	1.4621	1.8951	1.4771	1.8574	1.4329
1.6989	1.9454	1.5097	1.9754	1.5024	1.9264	1.4998
1.7781	1.9754	1.5775	1.9849	1.5502	1.9597	1.5453
1.8450	2.0142	1.5917	2.0124	1.5775	2.0106	1.5776
1.9031	2.0497	1.6361	2.0497	1.6157	2.0297	1.6042
2.0000	2.1018	1.6877	2.0945	1.6734	2.0781	1.6675
2.0792	2.1391	1.7236	2.1351	1.7275	2.1351	-
2.1461	2.1759	1.7631	2.1673	1.7430	2.1635	1.7403
2.2041	2.2007	1.8107	2.2253	1.7806	2.1758	1.7789

TABLE 7.1 (Cont..)

log P	log d			
	673°K		773°K	
	log dk	log dv	log dk	log dv
0.3979	1.5097	0.9020	1.4493	0.9299
0.5740	1.4117	0.9724	1.4635	1.0000
0.6989	1.5097	1.0212	1.4683	1.0212
0.8750	1.5263	1.1146	1.5021	1.1075
0.9420	1.5430	1.1236	1.5011	1.1211
1.0000	1.5475	1.1417	1.5153	1.1461
1.0969	1.5627	1.1700	1.5775	1.1764
1.1761	1.6009	1.2000	1.6232	1.2148
1.3010	1.6990	1.2700	1.7438	1.2767
1.4771	1.7785	1.3666	1.8511	1.3804
1.6021	1.8880	1.5200	1.8638	1.4317
1.6989	1.9133	1.4986	1.9328	1.4996
1.7781	1.9536	1.5423	1.9637	1.5440
1.8450	1.9849	1.5776	1.9735	1.5774
1.9031	2.0070	1.6011	2.0297	1.6022
2.0000	2.0719	1.6647	2.0781	1.6672
2.0792	2.1215	1.7372	2.1004	-
2.1461	2.1364	-	2.1521	1.7404
2.2041	2.1700	1.7694	2.1673	1.7782

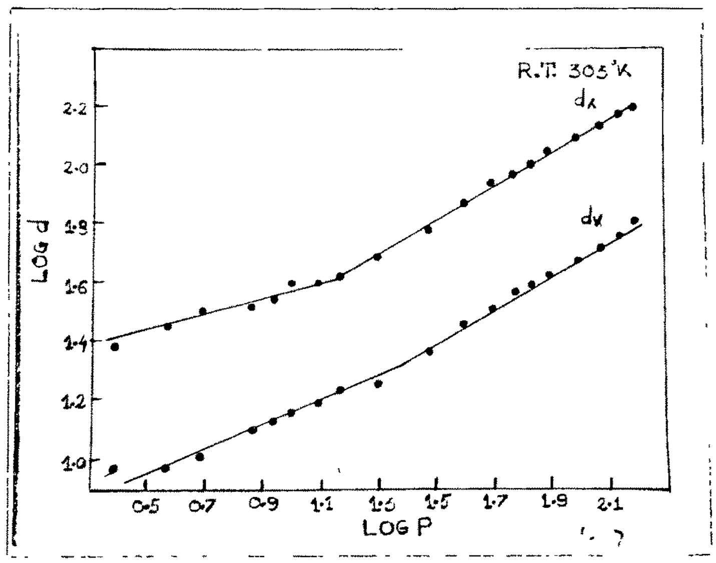

Fig. 7.1 Plot of log P versus log d for Knoop and Vickers diagonals at 303^{O}K

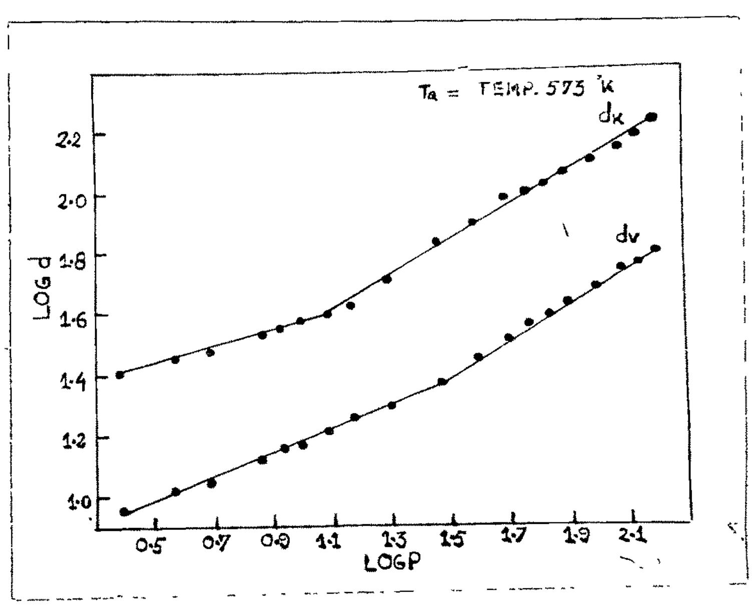

Fig. 7.2 Plot of log P vs log d (Knoop and Vickers) for the Samples quenched from 573^{O}K.

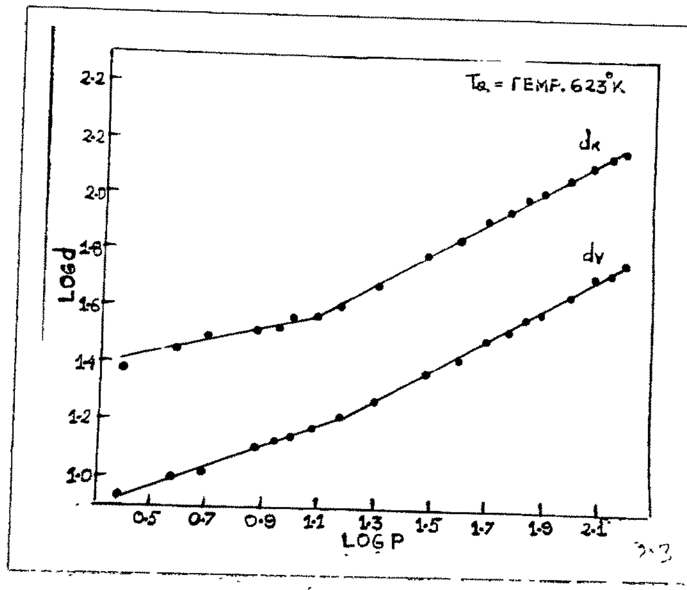

Fig. 7.3 Plot of log P vs log d (Knoop and Vickers) for the samples quenched from 623°K

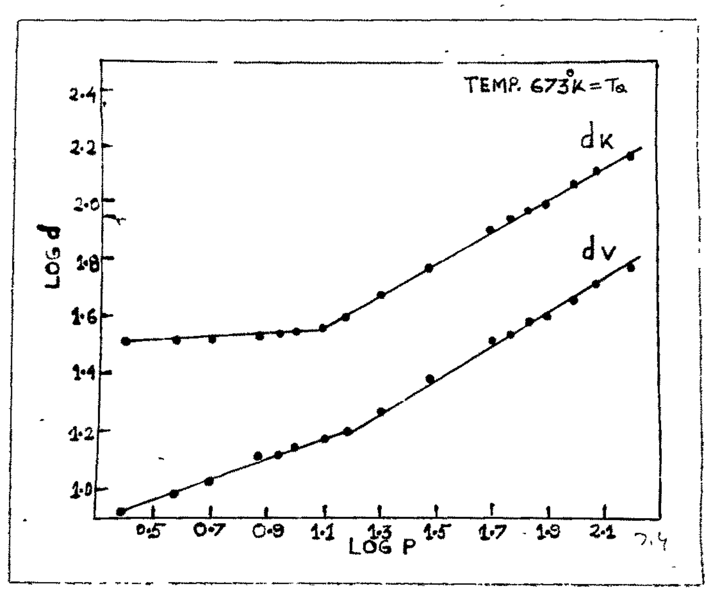

Fig. 7.4 Plot of log P vs log d (Knoop and Vickers) for the samples quenched from 673°K

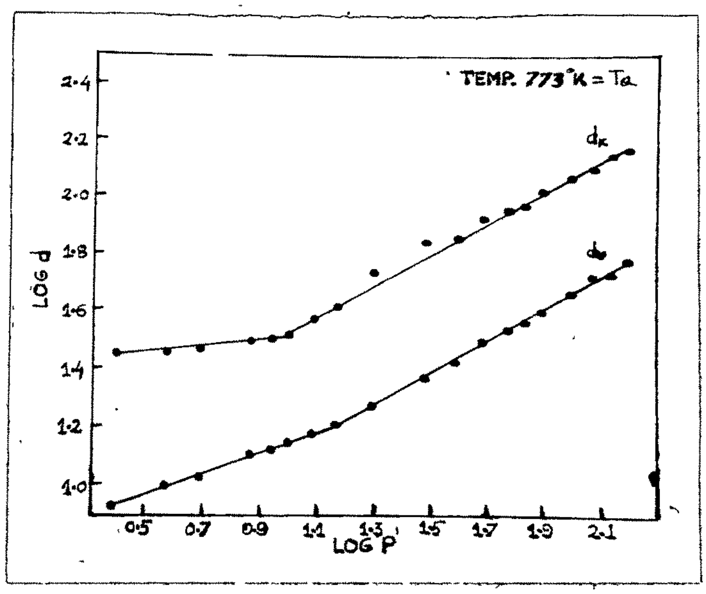

Fig. 7.5 Plot of log P versus log d for samples quenched from 773°K

Fig. 7.6 Knoop indentation mark along ~~on~~ [100] on calcite cleavage face. (P = 80 gm.)

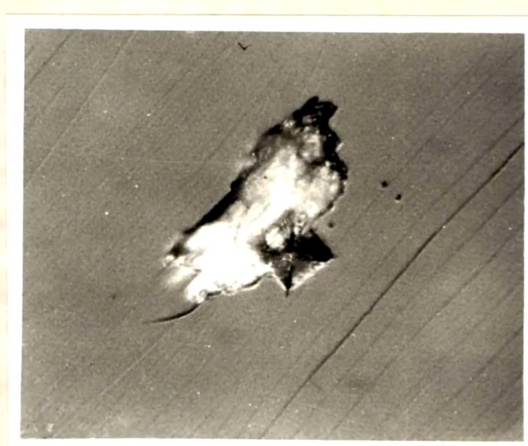

Fig. 7.7 Vickers indentation mark along [100] on calcite cleavage (P = 80 gm.)

relation between log P and log d is linear, the graph is a straight line, the slope of this line gives the value of 'n' and the intercept on log P axis gives the value of log a and hence 'a'. For all indenters that give geometrically similar shapes (impressions), Kick's law postulates a constant value of 'n' viz. n = 2. This implies a constant hardness value for all loads according to the definition of Knoop and Vickers hardness numbers (KHN and VHN).

A careful study of the graphs (log P vs log d) shows that there are two clearly recognisable straight lines of different slopes meeting at a kink which is obtained at a load of 15 grams for Knoop diagonals (d_k) and about 30 grams for vickers diagonals (d_v) at room temperature. As the quenching temperature increases, the load at kink shifts towards lower loads. Further the first part of ~~of~~ the straight line corresponding to observations taken at low loads upto 30 grams for vickers diagonals (d_v) and 15 grams for Knoop diagonals (d_k) at room temperature has slope(n_1) of higher value whereas for the second part of straight line for higher loads, the slope (n_2) has values less than 2. Since n values are different in different regions of the graphs of log P versus log d, being greater in first region, the 'a' values also vary in two regions being less in first region of low loads and more in second region of high loads.

7.4 RESULTS AND DISCUSSION :

There are two ways of studying the relationship between microhardness and applied loads. One way of studying this relationship was given by Hanemann (1941) in the form of an empirical rule that was believed to permit the intercomparision of microhardness values. This rule states that the load P is related to diagonal length of an indentation mark by the expression,

$$P = a\ d^{n} \quad \text{-------------} \quad (7.1)$$

where 'a' and 'n' are constants of the material under test ; 'a' represents the 'standard hardness' for an indenter of fixed diameter and 'n' giving the measure of the variation in hardness as a function of 'P' or 'd'. The other way is to study the variation of hardness (Knoop and Vickers hardness numbers) directly with load. In what follows, the detailed study on the variation of load with average diagonal length will be presented.

The equation 7.1 is also known as Kicks law. Taking logarithms of both sides yields

$$\log P = \log a + n \log d \quad \ldots\ldots \quad (7.2)$$

The values of constants 'a' and 'n' can thus be determined from a graph of log P versus log d. Since the

The values of n_1, n_2 and corresponding intercepts a_1 and a_2 for Knoop and vickers indentations are recorded in tables 7.2 and 7.3 respectively. Tables 7.2 and 7.3 also show$ the load at kink for various quenching temperatures.

It may be remarked in passing that several workers have reported visible scattering in 'n' values. e.g. see Hanemann and Schultz (1941), Onitsch (1947), Grodzinski (1952) . However, none has reported the splitting of graphs into two straight lines and on its characteristics. The study of variation of load with diagonal length of vickers indentation mark on faces of different types (c-, m-, d- and o-faces) of natural and synthetic barite crystals (Saraf, 1971) has shown very clearly the existance of two clearly recognisable straight lines of the graph of log P versus log d. Later, Shah(1976) and Acharya (1978) verified the splitting of graph of log P versus log d on calcite, zinc, TGS and KBr crystals. This behaviour of log P vs log d graphs is also observed on InBi alloy crystals (Desai, 1980), InSb (Panchal, 1981) using vickers pyramidal indenter. In the present investigation, for the first time the author has verified the splitting of the graph in two regions using knoop pyramidal indenter also. It is thus certain that the splitting of the graph into two straight lines is natural and is due to varied reactions of the crystal surfaces under indentations.

TABLE 7.2 (Knoop indenter)

Quenching temp. T_a (^oK)	n_1	n_2	a_1	a_2	load at Kink (gm)
303	3.70	1.80	1.77×10^{-5}	1.77×10^{-2}	15
573	3.70	1.80	1.66×10^{-5}	1.77×10^{-2}	12.5
623	4.20	1.80	3.16×10^{-6}	1.82×10^{-2}	12.5
673	9.10	1.80	7.58×10^{-14}	1.88×10^{-2}	12.5
773	9.10	1.80	2.18×10^{-13}	1.82×10^{-2}	10.0

TABLE 7.3 (Vickers indenter)

Quenching temp. T_Q (^oK)	n_1	n_2	a_1	a_2	load at Kink (gm)
303	2.35	1.82	1.99×10^{-2}	1×10^{-1}	30
573	2.60	1.80	0.95×10^{-2}	1×10^{-1}	30
623	2.74	1.80	0.70×10^{-2}	1.03×10^{-1}	20
673	2.80	1.78	0.66×10^{-2}	1.06×10^{-1}	15
773	2.86	1.80	0.51×10^{-2}	1.06×10^{-1}	15

7.4.1 Characteristics of two straight line regions in the graph :

The separation of the straight graph into two regions with different slopes indicates that in the first region of low loads, the value of hardness is strictly dependent on load and in the second region of high loads this dependence on applied loads is relatively reduced. It appears that besides this dependence on load, there could also be other factors contributing to this behaviour.

In order to determine the relative importance of these factors affecting the values of 'a' and 'n', the study was carried out on crystal surfaces which were quenched from high temperatures to room temperature. It is obvious from tables 7.2 and 7.3 that the values of 'a_1' and 'n_1' for low load region show comparatively large differences at all quenching temperatures whereas for the second part of the graph there are less difference in 'a_2' and 'n_2' values. Further, the values 'n_1' increase with the temperature in both cases (Knoop and Vickers) whereas 'a_1' values decrease with the temperature. This clearly indicates that 'n_1' and 'a_1' values are dependent on the previous history of the sample, where as the second part of the graph giving 'n_2' and 'a_2' values remain comparatively less affected by the previous history of the sample.

It should be remarked here that n_1 values in case of vickers indenter (Table 7.3) show less variation with quenching temperature where as for knoop indenter marked variation of n_1 values with quenching temperature are noticed (Table 7.2). Hence the two regions correspond in general with the structure sensitive and structure insensitive properties of the crystal. They can roughly correspond with extrinsic and instrinsic properties of the crystals. Further, the initial indentation under low loads i.e. initial plastic deformation produces cold working of the crystal. There will also be certain amount of recovery from this deformation. As a result the degree of hardening of crystal surface should increase. This is more true for low loads near kink. Hence with an increase in applied load the surfaces should offer high resistance to the indentation. The hardness in this region will therefore be lower than that in first region, mostly near kink. The surface is likely to follow Kick's law and the value of 'n_2' will be nearly equal to 2. In addition to the cold working and recovery of strained crystals, several factors such as surface energy, concentration of different types of imperfections, interactions, effect of penetration of indenter etc. are also operating in a way unpredictable at present. The experimentally observed deviations from the above remarks are therefore likely to be due to these factors which are not yet clearly understood. It is therefore difficult to conjecture conclusively from 'n_1' and 'a_1' values only the behaviour of crystal surface. The marked variation of n_1 with the temperature in case of knoop indenter indicates that surface layers of the specimen are more susceptible to changes in quenching temperature because the knoop pyramidal indenter, in general, measures the hardness of surface layers. It should be mentioned here that although the indentation work was carried out on freshly cleaved surfaces of quenched crystals with the intention of removing surface hardening of quenched specimens, the hardness study of the cleaved surfaces which were once the inner parts or interiors of quenched crystal has shown a noticeable change with quenching temperature i.e. 'body' hardness is affected by heat treatment, of course this change is obviously smaller than that of surface hardening of the quenched specimens.

7.5 CONCLUSIONS :

The following conclusions are drawn from the above discussion :-

(i) The graph of log P versus log d consists of two clearly recognisable straight lines having different slopes and intercepts on the axes.

(ii) The indenter load corresponding to kink representing a transition from one straight line to another depends upon quenching temperature.

(iii) The slope of first part corresponding to low load region of the graph is greater than that of the second part. The intercept made by the first line has less value than that made by second line.

(iv) The slopes 'n_1' corresponding to low loads are more susceptible to quenching temperature. In particular for knoop diamond pyramidal indenter 'n_1' is more susceptible to quenching temperature than for vickers pyramidal indenter.

(v) The defect structures operate differently in low load and high load regions corresponding to two parts of the graph of log P vs log d.