Anisotropic studies of quenched and indented potassium bromide cleavages

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Abstract. Kick's law and modified Kick's law for pyramidal indenter were used to study the mechanical response of indented and quenched potassium bromide cleavages. The study has disclosed an unusual interpretation of constants in the above laws. It suggested a pair of anisotropic indices which characterize a crystallographic direction. This study has also shown that there is atleast one crystallographic direction usually termed as homogeneous isotropic direction along which their mechanical properties are same. The implications are discussed in detail.

Keywords. Mechanical response; cleavage plane; pyramidal indenter.

1. Introduction

For studying mechanical response of indented surfaces, two empirical laws viz. Kick's law/Meyers law and modified Kick's law (Kick 1885; Meyer 1956; Hays and Kendal 1973) are available. Detailed study of the literature (Hanemann and Schultz 1941; Onitsch 1947; Grodzinski 1952; Westbrook and Conard 1973) indicates that the physical interpretations of constants in these laws are unsatisfactory. The aim of the present work is to critically re-examine these laws for the physical meanings of these constants by experimentally studying indented potassium bromide cleavages at room temperature, at elevated temperatures for different orientations in a phenomenological manner and by following the well known mathematical and graphical procedure. This is an extension of the earlier work carried out in our laboratory.

2. Experimental

Single crystals of potassium bromide grown from melt by Kyropoulos method (Kyropoulos 1963; Raveendran 1993) were used for indentation. Knoop indenter attached to C Z VERTICAL microscope was used to indent for various applied loads (1.25 to 160 g) and for fixed time of indentation (15 sec). Approximately equal sized samples of freshly cleaved KBr faces—planes—having Miller indices (100) were selected. The indentation marks were produced for different orientations of the longer diagonal of the Knoop indenter with respect to direction [100] on KBr cleavages. These orientations were designated by angle A between the reference direction [100] and the longer diagonal of the impression. For thermal treatment samples were kept in a muffle furnace, the temperature of which was gradually raised to the desired value and maintained at that value for 24 h. It is known that if a brittle crystal is too much quenched to room temperature, it shatters into large number of pieces. To avoid this

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the KBr crystals were quenched to room temperature by keeping the quenching rate as high as possible so that the samples could maintain their shapes and sizes. They were then cleaved at room temperature (303°K). The cleaved specimen was kept on the stage of the vertical microscope. By considering the crystal edge which is along direction [001] as the reference direction, the orientation is determined by measuring the angle A between the longer diagonal of the indentation mark produced by the pyramidal knoop indenter on the cleavage surface and the direction [001]. This is done by carefully adjusting the rotation of the stage. Large number of thermally treated and untreated specimens were indented for orientations 0, 10, 20, 30, 40, 50, 60, 70, 80 and 90° and quenching temperatures 473, 573, 673, 773 and 873°K.

3. Results and discussion

For pyramidal indenters, Kick's law is symbolically represented as

$$p = ad^n, \quad (1)$$

or

$$\log p = \log a + n \log d, \quad (2)$$

where p is the applied load, d the diagonal length of the indentation mark (μm) and a and n are constants of a given material where a is the standard hardness and n measure of the variation in hardness as a function of p or d or indicating capacity for work hardening (Mott 1956). The graphical analysis of (2) clearly indicated splitting of single straight line into two straight lines with slopes n_1 , n_2 and intercepts a_1 , a_2 for different crystals. The first line with n_1 and a_1 corresponds to low load region (LLR) and the second one with n_2 and a_2 to high load region (HLR) (Joshi and Pandya 1991; Jani *et al* 1994).

The present work also found the splitting of the above plot into two straight lines with n_1 , a_1 and n_2 , a_2 for cleavage faces of KBr crystals (Raveendran 1993). Due to visible scattering in n and a values, the necessity of application of modified Kick's law arises.

The modified Kick's law for pyramidal indenter is

$$P - w = bd^2,$$

or

$$\log P - w = \log b + 2 \log d.$$

Where b is the intercept and w the newtonian resistance pressure (Hays and Kendal 1973; Pratap and Hari Babu 1980; Joshi and Pandya 1991). The graphical plot of modified Kick's law also shows splitting of $\log (P - w)$ vs $\log d$ into two recognizable straight lines with intercepts b_1 , b_2 and newtonian resistance pressures w_1 and w_2 . It is well recognized that for single crystals of different types (Berzina *et al* 1965; Joshi and Pandya 1993) in HLR hardness is constant and independent of applied load. Hence in the present work for Kick's law and modified Kick's law, only HLR is considered for straight line plots with n_2 , a_2 and b_2 , w_2 parameters vary with ' A ' for different temperatures (table 1).

3.1 Variation of anisotropic indices with orientation

A careful study of plots of n_2 vs A , a_2 vs A and b_2 vs A for room temperature and different quenching temperatures (figures 1–3) reveals the following: (i) all these plots

Table 1. Angle A slopes $n_{2\text{obs}}$, $n_{2\text{mod}}$, intercepts a_2 , b_2 and newtonian resistance pressure w_2 of the plots of $\log d$ vs $\log P$ for KBr at room temperature 303°K and quenching temperatures 673°K and 873°K.

Angle A	$n_{2\text{obs}}$	$n_{2\text{mod}}$	w_2	$b_2 \times 10^{-3}$	$a_2 \times 10^{-3}$
<i>Room temperature (303°K)</i>					
0	1.8500	2.1460	1.6167	0.5939	1.5759
10	1.8000	2.0000	1.9975	0.5635	2.0345
20	1.8400	2.0000	1.5165	0.5314	1.5028
30	1.7910	1.9985	3.0851	0.4860	1.7740
40	1.7516	1.9900	2.6160	0.4411	2.2030
50	1.7476	2.0002	2.1178	0.4452	2.2045
60	1.7756	1.9817	2.3431	0.4920	2.9625
70	1.8661	1.9990	2.6178	0.5353	1.2705
80	1.8674	2.0102	3.1518	0.5662	1.3822
90	1.8644	2.0000	1.6597	0.6055	1.4473
<i>Quenching temperature (673°K)</i>					
0	1.8310	2.0000	1.1585	0.5620	1.7128
10	1.8500	2.0101	1.3900	0.5142	1.4196
20	1.8308	1.9910	1.8518	0.5081	1.5305
30	1.8000	2.0000	2.0180	0.4311	1.6943
40	1.7791	2.0000	2.5000	0.4296	1.7284
50	1.7791	2.0110	2.1916	0.4042	1.7301
60	1.8010	1.9900	2.7500	0.4311	1.6422
70	1.8410	2.1040	1.9516	0.4861	1.4504
80	1.8586	2.0000	1.8616	0.5190	1.3394
90	1.8310	2.0000	1.1690	0.5541	1.7128
<i>Quenching temperature (873°K)</i>					
0	1.8715	2.0000	1.3251	0.7460	1.1555
10	1.8281	2.0000	2.0500	0.7254	2.0927
20	1.7917	1.9990	2.1600	0.6920	2.4482
30	1.7784	2.0000	2.8500	0.6395	2.5604
40	1.7680	2.0101	2.9000	0.6017	2.6600
50	1.7680	2.0101	3.0030	0.6017	2.6600
60	1.7891	2.0000	2.0850	0.6491	2.4558
70	1.7990	1.9990	2.1050	0.7248	2.3535
80	1.8391	1.9616	1.9616	0.7410	1.9925
90	1.8685	2.0000	2.0000	0.7460	1.7836

have one common feature, viz. n_2 , a_2 and b_2 have extremum values for orientation 45° and for all temperatures. This orientation corresponds to direction [110] on the surface, (ii) these values are different for room temperature and different quenching temperatures and (iii) n_2 has a minimum value for $A = 45^\circ$ (figure 1) whereas a_2 has a maximum value for the same orientation (figure 2).

This is understandable as in HLR hardness is constant and independent of applied load. Hence with a decrease of n_2 , there is a corresponding increase for a_2 . Similarly the same relation holds for plot of b_2 vs A (figure 3) and approximately for w_2 vs A (not shown). The approximation in the plot of w_2 vs A is due to the fact that in (3) w is assumed to be newtonian resistance pressure which does not allow plastic deformation (Hays and Kendal 1973). It has units whereas n_2 is a slope and a pure number.

Figure 1. Plot of n_2 vs A for KBr cleavage plane (100) at different temperatures.

Figure 2. Plot of a_2 vs A for KBr cleavage plane (100) at different temperatures.

Figure 3. Plot of b_2 vs A for KBr cleavage plane (100) at different temperatures.

It is thus clear from the above analysis that the direction $[110]$ corresponding to 45° orientation on cleavage faces of this crystal occupies an important position in the study of Kick's law and modified Kick's law.

3.2 Variation of anisotropic indices with temperature

It is found that for this crystal, the plots of $\log a_2 T_q$ vs $\log T_q$ (figure 4) and $\log b_2 T_q$ vs $\log T_q$ (not shown) for different orientation consists of a series of almost parallel straight lines with almost equal slopes and different intercepts corresponding to different orientations. The plots follow general equation

$$\log g T_q = m \log T_q + \log Cr. \quad (3)$$

Where the variable g may be a_2 or b_2 and the straight line has slope m and intercept Cr where r denotes a given orientation. Different values of r gives a series of parallel lines.

Simplification of the above yields

$$g T_q^{1-m} = Cr. \quad (4)$$

Replacement of g by a_2 and b_2 in the above yields two equations

$$a_2 T_q^{1-m_2} = Cr_2, \quad (5)$$

and

$$b_2 T_q^{1-m_3} = Cr_3. \quad (6)$$

The relation between w_2 and T_q is not as simple as the one given above between a_2 (or b_2) with T_q due to the reasons mentioned above.

It should be noted that K_2 and K_3 are independent of quenching temperatures but depend on orientation A . K_2 and K_3 are less than unity and negative and they vary

Figure 4. Plot of $\log a_2 T_q$ vs $\log T_q$ for different orientations on KBr (100) cleavages.

Table 2. Values of slope m_2 , intercept Cr_2 and $K_2 (= 1 - m_2)$ from the plot of $\log 'a_2' T_q$ vs $\log T_q$.

Orientation A(deg)	Slope m_2	Intercept Cr_2 ($\times 10^{-3}$)	$1 - m_2$ $= K_2$
0	1.125	0.750	-0.125
10	1.071	1.311	-0.071
20	1.071	1.311	-0.071
30	1.000	2.290	+0.000
40	1.000	2.290	+0.000
50	1.000	2.290	+0.000
60	1.000	2.290	+0.000
70	1.071	1.311	-0.072
80	1.071	1.311	-0.071
90	1.125	0.750	-0.125

from -0.125 to 0 and -0.25 to 0 respectively. Similarly the values of Cr_2 and Cr_3 are also independent of temperature but depends on orientation. They also vary from 0.75 to 2.29 and 1.33 to 5.62 respectively. The observation in tables 2 and 3 indicate that the direction [110] is important. The present analysis has convincingly shown that n and a in Kick's law or b and w in modified Kick's law are not simple constants characteristic of the materials but are constants with a deep meaning. They depend on orientation of the pyramidal indenter with respect to a chosen direction on a crystal face and temperature.

Table 3. Values of slope m_3 intercept Cr_3 and $K_3 (= 1 - m_3)$ from the plot of $\log b_2 T_q$ vs $\log T_q$.

Orientation A(deg)	Slope m_3	Intercept Cr_3	$1 - m_3$ $= K_3$
0	1.25	1.33	-0.25
10	1.20	1.73	-0.20
20	1.14	2.26	-0.14
30	1.00	5.62	+0.00
40	1.00	5.12	+0.00
50	1.00	5.12	+0.00
60	1.00	5.62	-0.00
70	1.14	2.26	-0.14
80	1.20	1.73	-0.20
90	1.25	1.33	-0.25

3.3 Importance of direction [110]

Interesting and useful conclusions drawn from the above analysis is that the direction [110] for cleavage face of KBr in particular and the family of direction $\langle 110 \rangle$ in general is the one along which the quantities characterizing straight lines viz. (n_2, a_2) and (b_2, w_2) have extremum values. The orientation of the indenter corresponding to this direction is 45° with respect to reference direction [110] in particular and $\langle 110 \rangle$ in general. From the crystal structure of KBr (not shown), it is clear that the projection of direction [111] on KBr cleavage surface (100) is [110]. Further the arrangements of ions along this direction and direction [111] are the arrays of like ions separated by a constant distance and facing arrays of unlike ions on all sides. Since the ionic charges are identical (positive or negative) there is repulsion among them along these directions. If this is the only factor in the lattice, the repulsive force will destroy it. However, there are attractions due to the proximity of ions of opposite charges. Hence on the whole the lattice is maintained. However, the value of a property along this direction severely affected due to the arrays of like charges. According to Kick's law for a constant P and d , if n decreases a must increase so that the product ad^n has a constant value (P) provided P and d are kept constant. However the physical meaning of a is much different from n . In the literature a is described as standard hardness. On the basis of the current analysis it is felt that the use of the above phrase is a misnomer. It should be considered as a constant dependent on direction and temperature and the minimum value of n and maximum value of a are independent of direction and temperature. In the HLR along direction [110] the minimum value of n_2 corresponds with a maximum value of a_2 at all quenching temperatures and room temperature. In the present analysis weightage is given to HLR region in view of the fact that hardness is constant and independent of applied loads.

In the modified Kick's law pair of important quantities are b and w . The meaning given by Hays and Kendal (1973) for these quantities are different. b is considered as standard hardness and w the Newtonian resistance pressure responsible for the elastic deformation. However the present analysis shows that these quantities are dependent on the direction along which they are determined i.e. the crystal structure. Further only along direction [110], b_2 and w_2 have extremum values and are independent of

temperature and direction. Since along this direction the quantities (n_2, a_2) and (b_2, w_2) are independent of direction, this direction can be considered as homogeneous and isotropic direction. Hence it should be taken as reference direction and not the direction [100]. Further, it is well known that all real crystals contain imperfections. The above analysis has taken into account only crystal structure and not impurities and imperfections of different types in the crystals.

4. Conclusions

The following conclusions are drawn from the experimental study of the variation of applied load with longer diagonal of the Knoop indentation mark for different orientations and quenching temperatures.

- (I) The plots of $\log d$ vs $\log p$ consist of two clearly recognizable straight lines having different slopes (n_1 and n_2) and different intercepts (a_1 and a_2).
- (II) The variation in the exponents of d can be eliminated by employing modified Kick's law, where the exponent is nearly equal to 2 and n_2 and a_2 is replaced by b_2 and w_2 . This law is applicable only in HLR.
- (III) (n_2, a_2) and (b_2, w_2) have clear relation with orientation A and quenching temperatures and room temperature. Along [110] or in general $\langle 110 \rangle$ for KBr crystals extremum values which are independent of orientation and temperature.
- (IV) a_2 and b_2 depend on quenching temperature and in HLR follow the relations

$$a_2 T_q^{1-m_2} = \text{constant},$$

and

$$b_2 T_q^{1-m_3} = \text{constant}.$$

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