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Microhardness of rhombohedral crystals : Calcite and sodium nitrate

J R PANDYA, L J BHAGIA and A J SHAH
Applied Physics Department, Faculty of Technology and Engineering, M.S. University of Baroda, Baroda 390 001, India.

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Abstract. The variation of hardness of rhombohedral single crystals of calcite and sodium nitrate with quenching temperature is studied. Vickers and Knoop hardness numbers are determined from the indentations produced on freshly-cleaved crystal surfaces for various loads. The variation of hardness number with quenching temperature can be represented by HT_q^k = constant where the exponent k is less than unity and its sign determines the nature of material.

Keywords. Microhardness; hardness numbers; quenching temperature; calcite; sodium nitrate.

1. Introduction

Among the mechanical properties of materials, hardness is less understood. The present work aims at reexamining in a phenomenological manner the empirical formulae for obtaining Vickers and Knoop hardness numbers (H_v and H_k respectively) by studying the hardness of natural crystals of calcite and synthetic single crystals of sodium nitrate at different quenching temperatures (T_Q°K). It is an extension of the work reported earlier (Mehta 1972; Shah 1976; Acharya 1978).

2. Experimental

Natural crystals of calcite were obtained from different parts of the country. A large transparent block of calcite free from twinning was selected. Small pieces of cleaved crystals obtained from the transparent block were cleaved and used. Single crystals of sodium nitrate (GRSM quality) were grown from the melt by following the floating zone technique (Komnik and Startsev 1969). The crystals grown were annealed for sufficiently long time to minimise the thermal stresses and imperfections. Vickers and Knoop indenters attached to a vertical microscope were used to indent freshly-cleaved surfaces of thermally-treated and untreated calcite and sodium nitrate crystals for various applied loads. For hardness studies at different quenching temperatures, the specimen was gradually raised and maintained at the desired temperature for a fixed time (more than 24 hr) and quenched to room temperature. The specimen was then cleaved and indented by applying various loads to the indenter. The dimensions of the indentation marks were measured along direction [100] by a filar micrometer eyepiece with least count 0.2m.. The loads applied ranged from 2.5 to 160 g. The hardness number was calculated using the formulae (Mott 1956)

$$H_v = 1854.4\, P/d^2 \text{ kg mm}^{-2}, \quad (1)$$

$$H_k = 14230\, P/d^2 \text{ kg mm}^{-2}, \quad (2)$$

where P is the applied load in g and d the average diagonal length of the indentation mark in microns. The Vickers diamond pyramidal indenter has a square outline whereas the Knoop indenter has an elongated rhombic outline. The average value of the longer diagonal of the Knoop indentation mark is taken as in (2). In what follows the term hardness will be used to mean hardness number (H).

3. Results and discussion

Diagonal lengths were measured for various applied loads for both indenters at different quenching temperatures and the hardness values determined using (1) and (2). Typical observations at different loads and quenching temperatures presented in figure 1 show a plot of H_k versus load on calcite cleavage surface for quenching temperatures 303° and 623°K. For sodium nitrate crystals H_v is plotted against load for quenching temperatures 303 and 533°K (figure 2). It is clear that each consists of three clearly recognizable portions AB (linear part), $BB'C$ (non-linear part) and CD (linear part) corresponding to low-load region (LLR), intermediate-load region (ILR) and high-load region (HLR) respectively. The three regions also suggest prominent factors operating in the different ranges of applied load. This behaviour of microhardness with load can be qualitatively explained on the basis of the depth of penetration of the indenter. At small loads the indenter penetrates only the surface layers and therefore the effect is shown sharply at these loads (LLR region). The penetration depth increases with applied loads and the overall effect is due to surface and inner layers. This complex effect appears to be responsible for the non-linear portion of the plot (ILR region). After a certain penetration, the effect of inner layers becomes more and more prominent than that of surface layers and ultimately no change is observed in the value of hardness with load corresponding to the HLR region of the graph.

In the HLR , the hardness is constant and independent of load. This region is studied in the present investigation. The hardness in this region is slightly temperature-dependent. Similar curves (not shown) are also obtained for the H_v value of calcite with load and for the H_k value of sodium nitrate with load for different quenching temperatures.

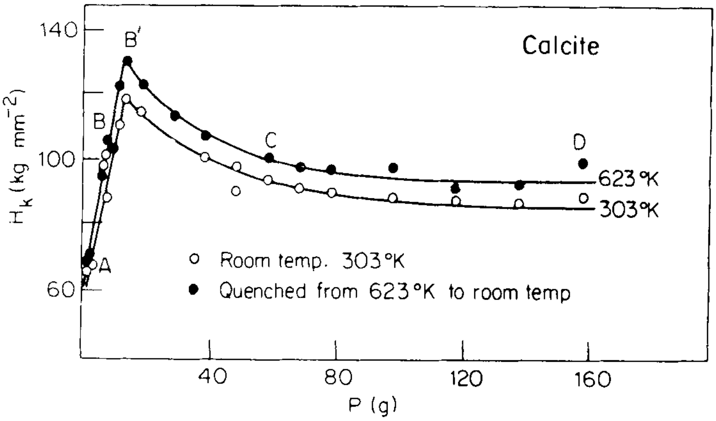

Figure 1. Plot of Knoop hardness number *vs* load on calcite cleavage surface for quenching temperatures 303 and 623°K, consisting of three distinct parts *AB, BB'C and CD.*

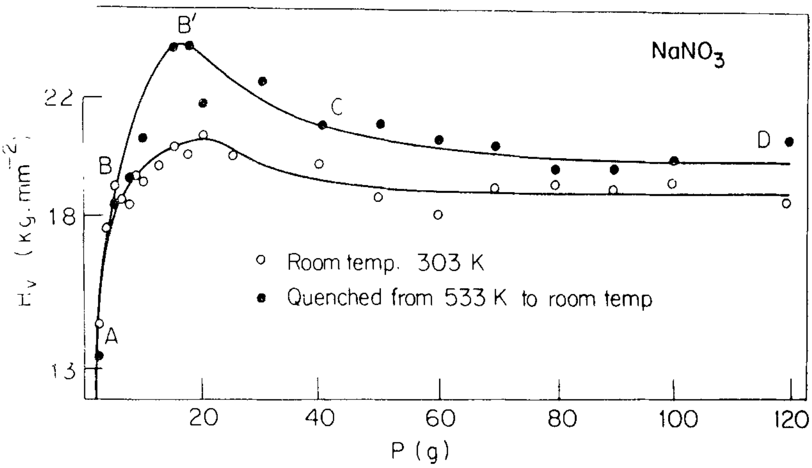

Figure 2. Plot of Vickers hardness number *vs* load on sodium nitrate cleavage surface for quenching temperatures 303°K and 533°K consisting of three recognisable regions; linear portion *AB*, non-linear portion *BB'C* and linear portion *CD*.

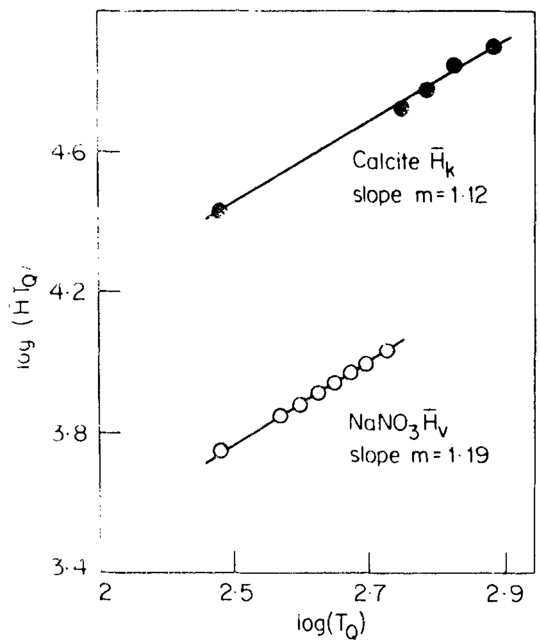

Figure 3. Plot of log $\bar{H}T_Q$ *vs* log T_Q for sodium nitrate and calcite crystals.

The relation between quench hardness and temperature could be successfully evolved by plotting log $\bar{H}T_Q$ against log T_Q (figure 3) for calcite and sodium nitrate crystals; $\bar{H}$ is the average hardness value in the HLR. From the straight line graph the following relation between hardness and temperature can be obtained

$$\log \bar{H}\, T_Q = m \log T_Q + \log A \tag{3}$$

where m is the slope and $\log A$ is the intercept on the axis of $\log \bar{H}T_Q$ when $\log T_Q$ is zero. Simplifying the above equation yields

$$\bar{H}T_Q^{l-m} = HT^k = A = \text{constant} \quad (4)$$

where $k = l\text{-}m$. From the plots of sodium nitrate and calcite (figure 3) the m values are found to be 1.19 and 1.12 respectively. Substitution of these values in (4) gives

$$HT_Q^{-0.19} = \text{constant}, \quad (5)$$

$$HT_Q^{-0.12} = \text{constant}, \quad (6)$$

These equations indicate that hardness at high loads is dependent on quenching temperature. The constants in the equations are assumed to be independent of load and quenching temperature. This may not be exactly true due to several factors such as incomplete quenching from elevated temperatures to room temperature, complicated interactions of similar and different types of imperfections etc. For single crystals of calcite and sodium nitrate the slopes of the plots (figure 3) are slightly different is probably because these crystals though isostructural show fine differences like NO_3^- group being more planar than the CO_3^- group in the crystal structure. Further, calcite is chemically more active than sodium nitrate. However, it is difficult to predict the extent to which these differences alter the slopes. The exponent values (k) in (5) and (6) are -0.19 and -0.12 respectively and are numerically less than unity implying that hardness increases with quenching temperature. By implication k should be positive for those crystals for which hardness decreases with increasing temperatures. This is indeed found to be true for other crystals (Acharya 1978; Panchal 1981).

4. Conclusions

The temperature-dependent studies of quench hardness numbers obtained by employing Knoop and Vickers hardness indenters for sodium nitrate and calcite crystals show that (i) in the low and intermediate load regions, hardness depends on load whereas for the high-load region hardness is constant and independent of load. (ii) The quench hardness increases with temperature. (iii) The sign of the exponent of quenching temperature indicates the nature of the crystalline material.

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