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Studies on microhardness of quenched mesolite crystals

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Observations and results on studies of the microhardness of {1 0 1} and {1 0 $\bar{1}$} faces of natural mesolite crystals are illustrated and explained. Variations in Vickers hardness number (VHN) with temperature of quenching and also with the applied load are discussed.

1. Introduction

Point indentation techniques used for hardness testing find wide applications in the study of mechanical properties of solids. Buckle [1] has pointed out the possibility of investigating various properties by means of measurements of microhardness. In general, indentation is carried out using sharp indenters, such as cones or pyramids, since their residual impressions have geometrical symmetry. With such geometry, the contact pressure is independent of indent size, and thus offers a convenient measure of hardness [2]. To the best of our knowledge, study of hardness has rarely been carried out on crystals of the zeolite family. Hence, it was thought worthwhile to investigate the hardness of natural mesolite, a zeolite family crystal.

Mesolite belongs to the family of crystals of the natrolite group of zeolite crystals. It has an orthorhombic structure with the unit cell formula

$$Na_{16}Ca_{16}[(AlO_2)_{48}(SiO_2)_{72}] \cdot 64H_2O$$

The present paper reports observations and results of our studies on the hardness of natural mesolite crystals. Observations in this connection were made on {101} and {10$\bar{1}$} faces. The effect of quenching on the hardness of mesolite crystals is also described and explained.

2. Experimental details

Natural mesolite crystals from Poona, India were examined under an optical microscope. Crystals with highly reflecting, planar faces were selected for the present study. The crystals were placed in an electric oven, and maintained at a predetermined constant temperature, for 12 h. They were then quenched in acetone (Analar grade). The quenched crystals were used for hardness measurements. Static indentations were made with a pyramid diamond indenter, using Vickers microhardness tester, MO 6270. For a fixed load, indentation marks were made for different indentation periods, 5 to 30 sec, from which it was found that the Vickers hardness number (VHN) is independent of dwell time beyond 30 sec. For a fixed indentation time, greater than 30 sec, the load P was varied from 5 to 70 g. This procedure was carried out for different fixed indentation periods. Square indentation impressions were obtained. For each load, indentation marks were made for different indentation periods of 30 to 50 sec. From ten indentation tests for each load, the mean value of the diagonals of the square indentation marks was determined. The diagonals of the indentation marks were measured using a filar micrometer of the Vickers projection metallurgical microscope. The VHN was calculated using the formula [3]

$$\text{VHN} = 1.854 \frac{P}{d^2}$$

where P is the load applied (kg), and d is the mean value of length of the diagonal of the indentation figures (mm).

Necessary precautions were taken to minimize error due to misorientation of the indenter with respect to the crystal surface. The VHN was calculated for different loads, and for different indentation periods, for mesolite crystals quenched at different temperatures, room temperature (30° C) to 140° C. VHN against load plots were constructed for different quenching temperatures for the {1 0 1} and {1 0 $\bar{1}$} faces of the crystals.

3. Observations, results and discussion

Sharp square indentation marks were obtained on the {1 0 1} and {1 0 $\bar{1}$} faces. Impressions for loads exceeding 60 g were accompanied by cracks around them. The material of the crystal surface is observed to be chipped off around the indentation mark. This occurs at intermediate and high loads, the degree of chipping depending on the load. Material is chipped off non-uniformly from different sides of an indentation mark.

The load, P, and the corresponding length, d, of the diagonal of the indentation mark are related as follows:

$$P = ad^n$$

Here, a and n are constants for a given material. The value of n represents the capacity for work-hardening. Fig. 1 shows plots of log d against log P, at room temperature (30° C) for {1 0 1} and {1 0 $\bar{1}$} faces. From these graphs, values of n for {1 0 1} and {1 0 $\bar{1}$} were calculated as 2.02 and 2.38, respectively.

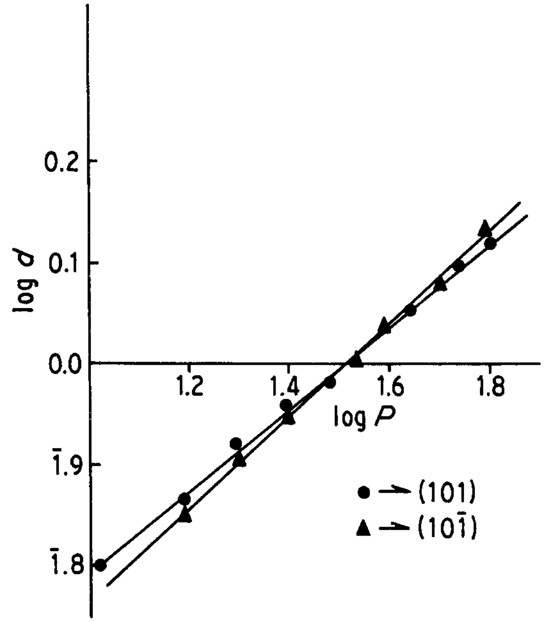

Figure 1 Plots of log d against log P for {1 0 1} and {1 0 $\bar{1}$} faces at room temperature.

Graphs of d^2 against P are shown in Fig. 2 for {1 0 1} and {1 0 $\bar{1}$} faces, at room temperature. From these graphs it can be concluded that d^2 is directly proportional to P. Figs. 3a and b show plots of VHN against load, P, for {1 0 1} and {1 0 $\bar{1}$} faces, respectively, at different quenching temperatures. From these graphs it is clear that VHN varies with load in a complex manner, for all quenching temperatures. These graphs also indicate that, for a given load, VHN increases with increase in quenching temperature. Variation of VHN with load, for a given quenching temperature, can be broadly divided into three regions (see Figs. 3a and b):

1. linear region AB, low load region (below 30 g);
2. non-linear region BB′C, intermediate load region (between 30 and 60 g);
3. linear region CD, high load region (above 60 g).

These three regions suggest prominent parameters operating in the three different ranges of applied load. Variations of the microhardness with load can be qualitatively explained on the basis of depth of penetration of the indenter. For small loads, the indenter penetrates surface layers only, and hence VHN increases slowly in the low load region. With increase in load, penetration depth also increases. Here, the overall effect is due to

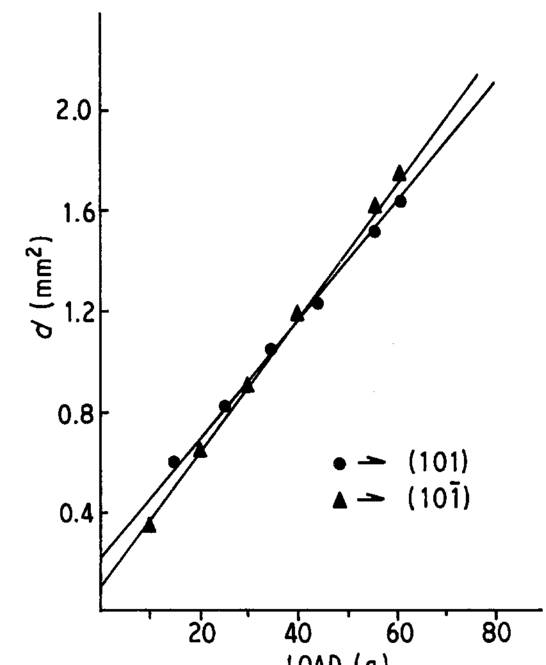

Figure 2 Plots of d^2 against P for {1 0 1} and {1 0 $\bar{1}$} faces at room temperature.

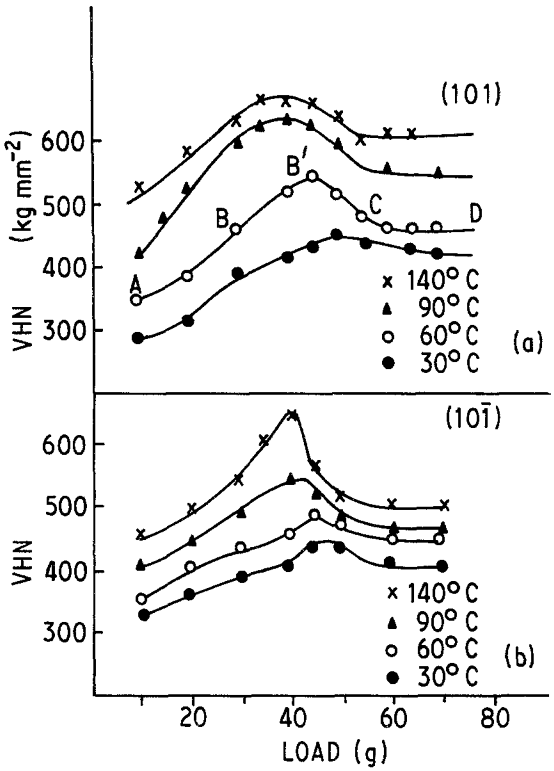

Figure 3 VHN plotted against load P for $\{101\}$ and $\{10\bar{1}\}$ faces.

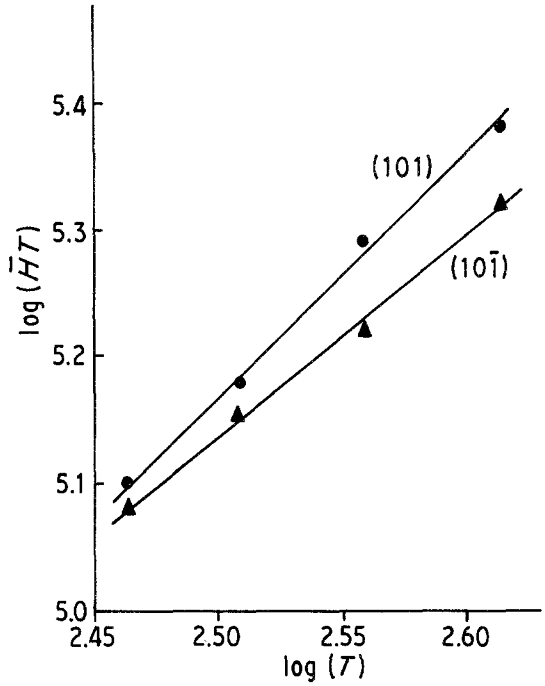

Figure 4 Log ($\bar{H}T$) plotted against log (T) for $\{101\}$ and $\{10\bar{1}\}$ faces.

surface as well as inner layers of the specimen. This complex effect seems responsible for the non-linear part of the plot (more rapid increase, reaching a maximum, and decrease) for intermediate loads. Beyond a certain penetration, the effect of inner layers becomes more prominent than that of the surface layers, so much so that finally the inner layers alone become effective. In consequence, VHN does not vary with further increase in load. This accounts for the flat portion, CD, of the graph in the high load region. Here, VHN is independent of load. It is interesting to note that, for a given load, VHN increases with increase in quenching temperature. Furthermore, the plots in Fig. 3 clearly show that the maximum value of VHN in the intermediate load region, shifts towards the low load region, with increase in the temperature of quenching, for both the faces $\{101\}$ and $\{10\bar{1}\}$.

Table I shows constant values of VHN, in the high load region, for different temperatures. It may be noted that, at a given temperature, the value of VHN for $\{10\bar{1}\}$ is smaller than that for $\{101\}$. Indentation is invariably associated with surface cracks. The dimensions of the cracks are directly proportional to the size of the indentation marks [4].

The microhardness of the crystals under study was measured up to a quenching temperature of 140° C. Beyond this temperature, the crystals became quite brittle. This is attributed to the breaking of bonds betwen neighbouring layers. Fig. 4 shows a plot of $\log(\bar{H}T)$ against $\log(T)$. Here, $\bar{H}$ is the average value of VHN in the load-independent region, and T is the quenching temperature. Hence, one can write

$$\log(\bar{H}T) = m \log T + \log A$$

$\log(\bar{H}T)$ is a function of quenching temperature T.

$$\bar{H}T^{1-m} = \text{constant } C$$

$$\bar{H}T^{K} = \text{constant } C$$

This constant C is independent of quenching tem-

TABLE I VHN in the high load region

Temperature (° C)	VHN (kg mm^{-2})	
	for $\{101\}$	for $\{10\bar{1}\}$
30	415	404
60	456	444
90	544	460
140	604	496

perature. The value of m is computed from the graphs in Fig. 4. Hence, values of K are found to be -0.90 and -0.58 for the $\{101\}$ and $\{10\bar{1}\}$ faces, respectively.

$$\bar{H}(T)^{-0.90} = \text{constant for the } \{101\} \text{ face}$$

$$\bar{H}(T)^{-0.58} = \text{constant for the } \{10\bar{1}\} \text{ face}$$

For both the faces, the exponent of temperature is negative. This implies that load-independent hardness increases with the quenching temperature. This is also evident from Fig. 3. The constants K and C in the equations are assumed to be independent of load and quenching temperature.

4. Conclusions

1. For a given temperature of quenching, with increase in load the VHN (a) increases slowly in the low load region, (b) increases, reaches a maximum and then decreases in the intermediate load region, and (c) remains practically constant in the high load region.

2. With the increase in quenching temperature the maximum value of VHN (in the intermediate load region) shifts towards the low load.

3. In the high load region, the relation $\bar{H}T^K =$ constant holds good. Average value ($\bar{H}$) of VHN, in the high load region, increases with increase in quenching temperature.

4. Diagonals of the indentation marks are found inclined at 45° to the c-axis for both the faces $\{101\}$ and $\{10\bar{1}\}$. This was verified from etch patterns on these faces.

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