

Temperature dependence of Vickers microhardness and creep of InBi single crystals

V P BHATT and C F DESAI

Physics Department, Faculty of Science, M S University of Baroda,
Baroda 390 002, India

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Abstract. Variation of Vickers microhardness of InBi single crystals with temperature has been studied. Loading time dependence of the microhardness at different temperatures has been used for the creep study in the temperature range 30° — 85°C. The activation energy for creep has been evaluated and the results are discussed.

Keywords. Vickers microhardness; InBi single crystals; creep characteristics.

1. Introduction

InBi is an intermetallic compound of elements of groups III and V. It belongs to the space group P4/nmm with the axial ratio $c/a = 0.955$ (Binnie 1956). There has been little work done on single crystals of InBi. This may be because its melting point is low ($=109.5^\circ\text{C}$) and it is not a semiconductor unlike the other III – V compounds, such as InSb, InP, GaP, GaAs, InAs etc. This makes the material uninteresting in view of its applicability. However, it is noteworthy that InBi is the most anisotropic substance known so far (Setty and Mungurwadi 1969) and also, it is the most diamagnetic material (White *et al* 1975). Unlike the other III – V compounds, it behaves like a semi-metal. These special features of InBi have attracted some workers to further investigate its properties in detail. Typically, InBi was selected as a member material for crystal growth experiments in space by NASA in its Appollo fly-back missions and the Skylab.

The authors have studied the microhardness property of InBi single crystal and the present paper reports variation of its microhardness and its creep behaviour in the temperature range 30° to 85°C.

2. Experimental

The crystals used in the present study were grown by zone-melting method (Bhatt and Desai 1978). The specimens were in the form of 3 to 4 mm thick cleavage

slices of cross-sectional area about 1 cm^2 obtained by cleaving the crystal-rod along its cleavage plane (001). The cleavage was performed at ice-temperature to minimize deformation. To obtain the hardness values in terms of Vickers hardness, the Vickers diamond pyramidal indenter was used and the indentations and measurements were carried out on the Vickers projection microscope supplied by M/s Cooke, Troughton and Simms Ltd., England. Since this equipment does not provide for high temperature hardness testing, a simple hot stage was prepared which was attached to the hardness tester. The temperature was sensed by a copper-constantan thermocouple. The thermocouple leads were connected to the input terminals on an operational voltage amplifier of gain 100 and the output was read to an accuracy of 1 mV. The microhardness measurements were carried out on the (001) surface of the specimen. Before producing the indentation, the specimen to be indented was held at the desired temperature for at least 30 min to achieve thermal equilibrium.

It is known that microhardness has a complex load-dependent character for small applied loads. In the case of InBi single crystals, we have found this dependence to be confined to loads less than 20 gm. Hence the load of 25 gm was selected to avoid microhardness variations due to fluctuations in the applied load. Data obtained for different indentation times at different temperatures have been used for the creep study.

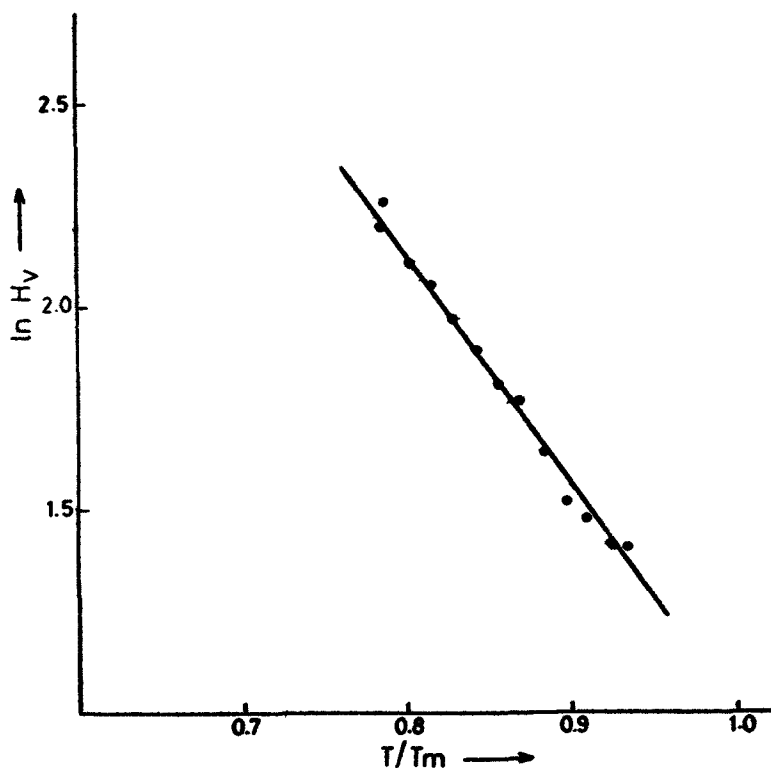


Figure 1. Plot of $\ln H_V$ versus T/T_m .

3. Results and discussion

3.1 Temperature variation of microhardness

For studying the temperature dependence of microhardness, indentations were made at 5°C intervals from room temperature (30°C) up to 85°C on a number of specimens and the results represent the average values. Figure 1 shows the plot of $\ln H_v$ versus T/T_m where H_v is the Vickers hardness and T and T_m , the indentation temperature and the melting point, respectively, both in °K. It can be seen that the plot is a straight line with negative slope indicating a fast decrease in H_v with increasing temperature. It should be noted that the room temperature in the present case corresponds to about 0.8 on the homologous scale, *i.e.* T/T_m . The temperatures used here thus fall in quite a high temperature range above $0.5 T_m$, *viz.* from $0.79 T_m$ to $0.94 T_m$. The steep fall in microhardness with temperature above $0.5 T_m$ has also been observed in a number of cases like Si, Ge and Cu (Atkins 1973) and seems to be a general trend followed by metallic materials.

The dependence of microhardness on temperature is similar to the variation of a number of mechanical properties like flow stress (Holloman and Lubhan 1947). The nature of $\ln H_v$ vs T/T_m graph is in agreement with the hardness – temperature relationship (Ito 1923; Shishokin 1930)

$$H = A \exp -BT \quad , \quad (1)$$

where B is known as the softening coefficient of hardness and A , the extrapolated intrinsic hardness. The slope of the graph in figure 1 yields the value of the softening parameter B to be $150.33 \times 10^{-4} \text{ }^\circ\text{K}^{-1}$.

3.2 Creep and activation energy for flow process

The use of indentation hardness as a means of studying the plastic yield properties of solids is known. The hardness decreases with increasing loading time and the general behaviour may be described by an overall empirical relation which incorporates the earlier relations (Ito 1923; Shishokin 1930). The hardness behaviour closely parallels the creep characteristics of the materials obtained in unidirectional stress tests.

In the present study, a relation based on the kinematic analysis of the creep process during indentation (Atkins *et al* 1966) has been found to be true and it has been used to evaluate the activation energy for creep. The equation is

$$\ln (H^{-3} - H_0^{-3}) = \ln A + \ln (t^{1/3} - t_0^{1/3}) - Q/3RT, \quad (2)$$

where H = the hardness value at time t ; H_0 = the hardness value immediately after attaining the full load at time t_0 ; Q = activation energy for creep; A = a constant; T = absolute temperature; R = the universal gas constant.

The plots of $\ln H_v$ against $\ln t$ obtained at different temperatures are shown in figure 2. It can be seen that $\ln H_v$ varies linearly with $\ln t$ and the slope of the straight lines increases with temperature as predicted by Atkins *et al.* For each

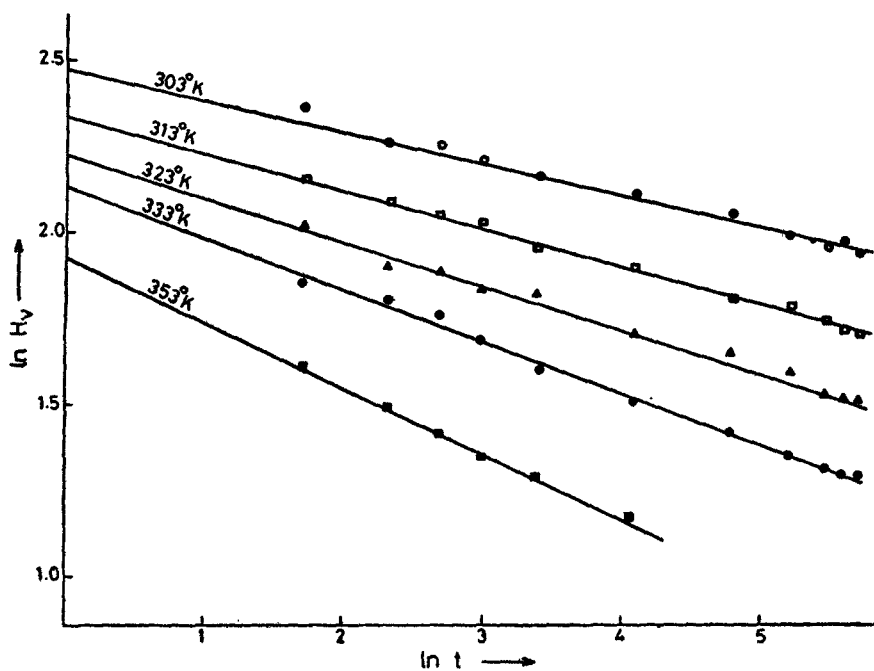


Figure 2. Plots of $\ln H_v$ against $\ln t$ at different temperatures.

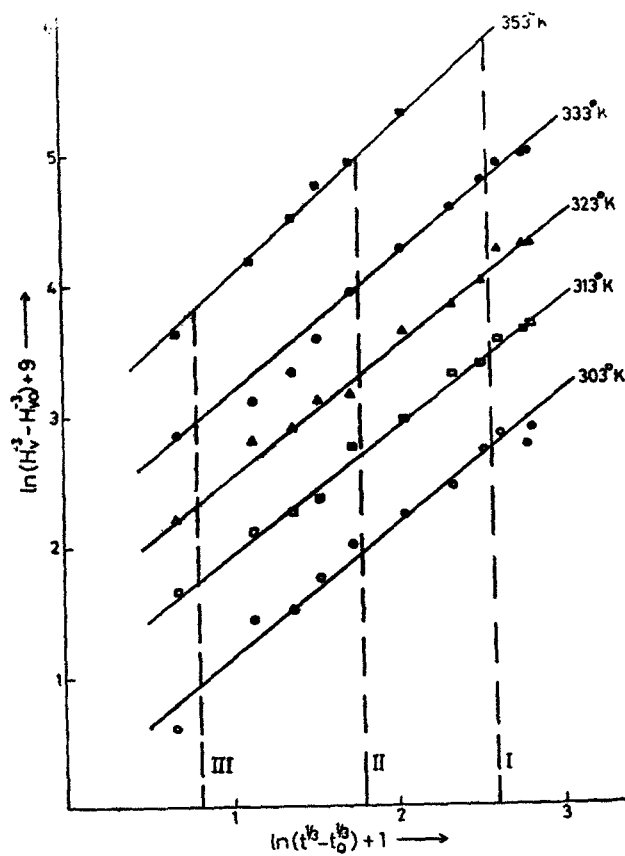


Figure 3. Plots of $\ln (H_v^{-3} - H_{v0}^{-3})$ against $\ln (t^{1/3} - t_0^{1/3})$ at different temperatures.

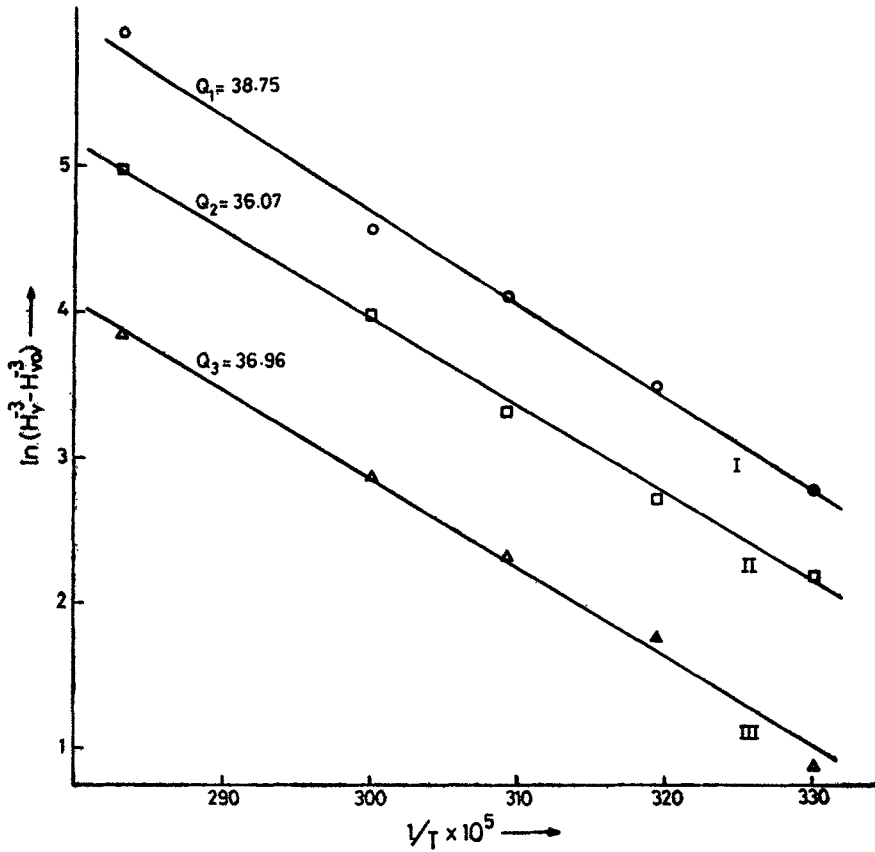


Figure 4. Plots of $\ln(H_v^{-3} - H_{v0}^{-3})$ versus $1/T$ for different values of $\ln(t^{1/3} - t_0^{1/3})$.

temperature, H_{v0} was obtained for $t_0 = 1$ sec from these plots. Figure 3 shows the plots of $\ln(H_v^{-3} - H_{v0}^{-3})$ versus $\ln(t^{1/3} - t_0^{1/3})$ at different temperatures. These are straight lines, all of almost equal slope close to unity in accordance with (2).

To evaluate Q , the usual method is to find difference between intercepts at two temperatures T_1 and T_2 in these graphs (figure 3) and to equate it to $Q(1/T_2 - 1/T_1)/3R$ under the assumption that Q remains constant with temperature. However, in many cases Q has been found to be different in different temperature ranges. Typical examples are aluminium (Lytton *et al* 1958) and tungsten carbide (Atkins 1973).

Moreover, we have found that this method of evaluating Q may sometimes lead to confusing results. Instead, an alternative method which does not assume Q to be constant with temperature has been used. From the plots of figure 3, three sets of $\ln(H_v^{-3} - H_{v0}^{-3})$ values at different temperatures were obtained for three different values of $\ln(t^{1/3} - t_0^{1/3})$ indicated by vertical broken lines. Selection of such widely separated different ordinate values would naturally average out small deviations of the straight line slopes from unity in the final

results. These values of $\ln (H_v^{-3} - H_{v0}^{-3})$ were plotted against the inverse of corresponding temperatures (figure 4). Again these curves are straight lines with approximately equal slope according to (2). The slope is given as $-Q/3R$. In case Q were different in different temperature ranges, the curve would split up into more than one straight line with different slopes yielding different Q values. The fact that the curves, here, are single straight lines indicates that Q is indeed constant in the temperature range used. The average value of the activation energy Q obtained from the three data-lines has been found to be $37.27 \text{ kcal-mole}^{-1}$. In a large number of metals, the activation energy for creep has different values for low ($<0.5T_m$) and high ($>0.5 T_m$) temperature ranges, indicating different creep controlling mechanisms. Since in the present case the temperature range used is well above $0.5T_m$, the value of Q remaining constant is to be expected. It may have a different value for temperatures below $0.5 T_m$ viz temperatures less than -81°C . However, to obtain data for temperatures less than the room temperature was not possible and the overall behaviour of Q with temperature remains obscure.

In a number of metals like In, Cd, Pb, Cu, Au, Ni, α -Fe and γ -Fe (Hargreaves 1928), the activation energy for creep at high temperatures has been found to be close to the activation energy for self-diffusion and it has been suggested that the creep rate is determined by diffusion - controlled mechanisms. The temperatures used in the present creep study are well above half the melting point of the crystal and it is quite probable that the creep at these temperatures is controlled by a diffusion activated process like dislocation climb.

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