## *Deformation and fracture of indium antimonide crystals*

Several papers [1–3] and a review [4] have recently been devoted to the study of the deformation and cracking of semiconductor crystals having the diamond structure, at temperatures where they are conventionally regarded as brittle. The experimental method used was based on indentation; this is simple in practice, but in view of the complexity of the deformation under the indenter it may lead to difficulties in the interpretation of the results.

However, in the light of recent studies of indentation of brittle materials [2, 5–7], it has become a method of considerable potentialities in investigations of their plastic response and fracture characteristics. We were, therefore, prompted to examine in more detail than in our work on germanium crystals [1] the unusual dependence of the Vickers hardness (VHN) on the load ($L$) acting on the indenter, and the significance of this effect in relation to the mode of deformation in the material surrounding the indentation.

In order to facilitate indentation at temperatures within the "brittle" as well as the "ductile" regions without exceeding the range −70 to 200° C, readily attainable by simple means, e.g. solid carbon dioxide and heated silicon oil, we chose indium antimonide crystals. These have a substantially lower melting point (523° C) than the germanium (937° C) previously used, but a similar crystal structure, and we expected the brittle-to-ductile transition to occur in the range 0 to 100° C rather than close to 350° C as is the case with germanium. The bath heater was similar to that used by Sumino and Hasegawa [8] in their work on the hardness of Ge. The crystals, measuring $4 \times 4 \times 10\,mm^3$, supplied by "MCP Electronics" Wembley, Middlesex, were of N-type with a carrier concentration of about $10^{14}\,cm^{-3}$, a resistivity of $0.1\,\Omega\,cm$ at −200° C, and a dislocation density, as determined by etch-pit counts, of about $3 \times 10^3\,cm^{-2}$. Before indentation crystals were mechanically polished to a mirror finish, and about 1 $\mu$m of the surface was then removed by polishing at −25° C for 20 min in a solution containing 70, 28 and 2 volume fractions respectively of $CH_3OH$, $HNO_3$ and HF. Loads used ranged

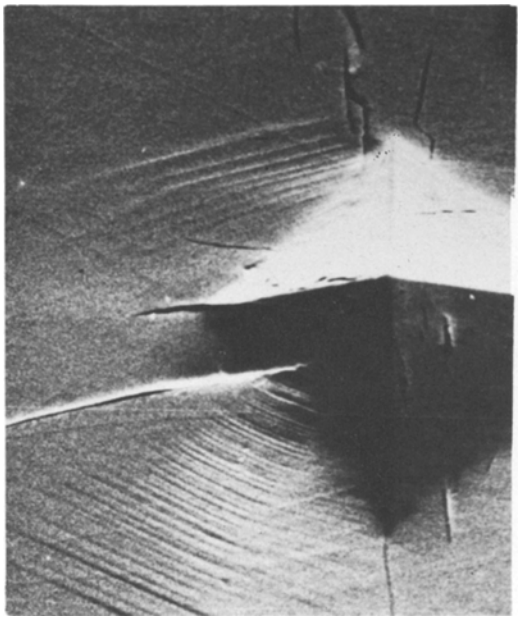

*Figure 1* Slip bands near a diamond-pyramid indentation made on a $\{1\,1\,2\}$ surface of an InSb crystal with a 30 g load at 150° C. The length of side of the square base is 20 $\mu$m.

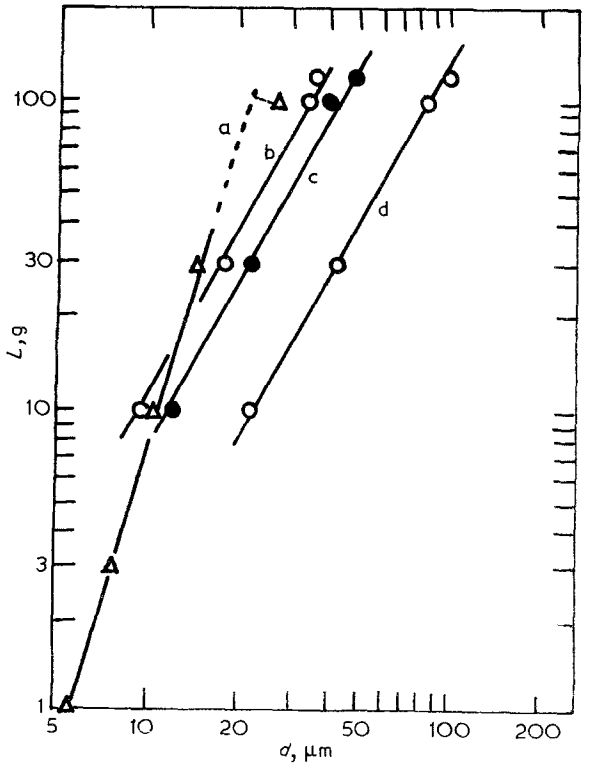


*Figure 2* The relation between the load ($L$) and the length ($d$) of the projected indentation diagonal at (a) −65° C, (b) 60° C, (c) 110° C and (d) 200° C.

from 1 to 100 g: of the three faces indented, i.e. (1 1 1), (1 $\bar{1}$ 0) and (1 1 $\bar{2}$), the second was found to be somewhat softer than the others, but the anisotropy of the VHN was not sufficiently pronounced to warrant discussion of details here.

The indenter was held under load for 10 sec in all cases; an investigation of the effect of holding-time on the VHN, using a 100 g load, showed that transient "indentation creep" [9] was insignificant except above 120° C, when the hardness dropped a few percent if the load was applied for 1 to 2 min. With a 100 g load the VHN was found to be effectively constant below 0° C at a value of 280, falling to about 250 at 20° C, 165 at 60° C, 110 at 110° C and to about 35 at 200° C.

Definite evidence of "ductility", i.e. slip bands near the indentation, was found at and above room temperature (Fig. 1), but not at significantly lower temperatures. With the present method of deformation the ductile-to-brittle transition appears to begin at about 0° C, as may also be inferred from the foregoing hardness data.

Fig. 2 shows the relation between the load and $d$, the length of the indentation diagonal as projected onto the crystal surface, at four temperatures. Each point is based on at least three measurements, made by optical and scanning electron-microscopy. In the "brittle" range, i.e. line (a) referring to −65° C, one has $L \propto d^{3.5}$ up to $L \simeq$ 30 g, which implies that the VHN $\propto L^{0.57}$, in reasonable agreement with the relation VHN $\propto L^{0.50}$ valid to about the same load level in germanium at room temperature [1]. Again, as in germanium, at higher loads the dependence of the VHN on load becomes less pronounced.

The VHN, evaluated as the ratio $2L/d^2$ kg mm$^{-2}$, for the points on the line (a), i.e. for loads of 1, 3, 10 and 30 g, is 64, 102, 163 and 265 respectively. With the 100 g load the measured VHN (275) is substantially less than the corresponding value obtained by extrapolation of the power-law relation (450). The lines (b), (c), (d), referring to 60, 110 and 200° C, in the order given, comply with the relation $L \propto d^2$, i.e. at these temperatures the VHN is no longer discernably load-dependent in the $L$-range investigated. This "conventional" behaviour can almost certainly be ascribed to the relative ease of slip and the concomitant, reduced, propensity to cracking.

## References


1. R. K. BANERJEE and P. FELTHAM, *J. Mater. Sci.* **9** (1974) 1478.
2. M. J. HILL and D. J. ROWCLIFFE, *ibid* **9** (1974) 1569.
3. M. V. SWAIN, B. R. LAWN and S. J. BURNS, *ibid* **9** (1974) 175.
4. R. K. BANERJEE and P. FELTHAM, *Rev. Deform. Beh. Mater.* **1** (4) (1975) in press.
5. B. J. HOCKEY and B. R. LAWN, *J. Mater. Sci.* **10** (1975) 1275.
6. B. R. LAWN and M. V. SWAIN, *ibid* **10** (1975) 113.
7. B. R. LAWN and E. R. FULLER, *ibid* **10** (1975) 2016.
8. K. SUMINO and H. HASEGAWA, *Trans. Jap. Inst. Met. (Suppl)* **9** (1968) 749.
9. C. A. BROOKES, R. P. BURNARDS and J. E. MORGAN, *J. Mater. Sci.* **10** (1975) 2171.


*Received 14 January*
*and accepted 3 February 1976*

R. K. BANERJEE
P. FELTHAM
*Brunel University,*
*Uxbridge,*
*London, UK*

## *The effect of thermal cycling on the microstructure of the Pb–Ag eutectic*

An important problem associated with the use of *in situ* composite materials at high temperatures is the prevention of microstructural coarsening. The stability of eutectic composites at uniform high temperature has been studied extensively (see [1]). In contrast, the thermal stability of eutectic alloys under more realistic conditions (e.g. in fluctuating temperatures [2–4], or in temperature gradients [1, 5–7]) has been comparatively neglected.

The present communication describes the results of an investigation of the thermal stability, under conditions of cyclically fluctuating temperature, of the fibrous Pb–Ag eutectic composite. This alloy has been shown [5] to exhibit unusual morphological changes after heating in a temperature gradient; and it was considered pertinent to investigate the effects on Pb–Ag of other non-isothermal heat-treatments.