JOURNAL OF MATERIALS SCIENCE 1 (1966) 219–228

Etch-Pit Studies of Dislocations in Indium Antimonide

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Received 1 March 1966

Indium antimonide specimens were plastically bent to introduce an excess of dislocations having either In-atoms at the edge of their extra half-planes or having Sb-atoms there. The lower yield stress for bending at 270°C was dependent on the direction of bend, being greater when specimens were bent to produce excess Sb-dislocations.

Bent specimens were annealed and the etch-pit densities in them compared with theoretical prediction. It was found that a modified CP4 etch containing butylamine gives a reliable estimate of the total dislocation density (i.e. shows up both In- and Sb-dislocations), whilst the modified CP4 etch without butylamine reveals all the In-dislocations and about half the Sb ones.

1. Introduction

The etching properties of InSb have attracted considerable interest since Allen's [1] observation that pits produced with CP4A etchant appear on only four of the eight octahedral faces. This property is consistent with the fact that its sphalerite structure contains no centre of symmetry, and consequently a $(\bar{1}\bar{1}\bar{1})$ plane is not equivalent to a (111) plane. Warekois [2] showed, by means of an X-ray technique, that a face which produced etch pits with a solution containing equal parts of HF, HNO_3 and H_2O was one terminating with either singly-bonded Sb- or triply-bonded In-atoms. It seems reasonable that a face of this kind terminates with In-atoms, since the alternative requires three times as many broken bonds. Such a face, designated $(\bar{1}\bar{1}\bar{1})$ by Warekois, is called an "indium" face in this paper; the (111) type is called an "antimony" face.

Another interesting property of the sphalerite structure is that the structure of a positive dislocation may not be equivalent to a negative one [3]. For example, the dislocation in fig. 1a has rows of In-atoms at the edge of its two extra half-planes, while that of similar character but of opposite sign in fig. 1b has rows of Sb-atoms there. Haasen called the former type an "In-dislocation" and the latter type an "Sb-dislocation". Venables and Broudy [4], who bent specimens to introduce an excess of either the one or the other type of dislocation, concluded that only one type was revealed by their etchant. It cannot be determined from these experiments whether this was the In- or Sb-type. Recently, however, the experiments of Bell, Latkowski, and Willoughby [5] have shown that a modified CP4 etch attacks In-dislocations preferentially, a result which is in agreement with the theoretical predictions of Gatos *et al* [6] and Holt [7].

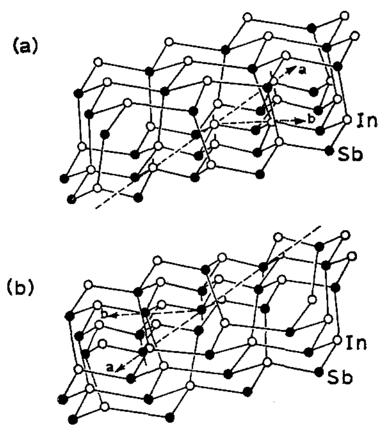


Figure 1 Pure edge dislocations in InSb: (a) In-type; (b) Sb-type.

In all the work mentioned above, etch pits on In-faces have been studied. Gatos and Lavine [8, 9], however, obtained etch pits on both In- and Sb-surfaces by adding "inhibitors", such as stearic acid or primary amines, to a CP4 etch. They suggested that these inhibited etchants revealed both In- and Sb-dislocations on In-faces. More recently, Lavine, Gatos, and Finn [10] have developed an etchant containing butylthiobutane which, they suggested, revealed only Sb-dislocations. They performed a series of bending and etching experiments which appeared to confirm the suggested behaviour for all these etchants, but the evidence is not conclusive.

The purpose of this present series of experiments was to re-examine the behaviour of these etchants on a quantitative basis, making full allowance for the presence of minority sign dislocations. Specimens were plastically bent to various radii of both positive and negative curvature. The dynamics of this process were studied, and the etch-pit configurations of the dislocations introduced were observed both before and after annealing.

2. Experimental Procedure

Single crystals of InSb were supplied by the Royal Radar Establishment. They were all n-type with a carrier concentration of about $10^{14}/cm^3$, and an initial etch-pit density, ρ_0, of 10^3 to $10^4/cm^2$. Samples were prepared in the form of bars oriented as shown in fig. 2 so that

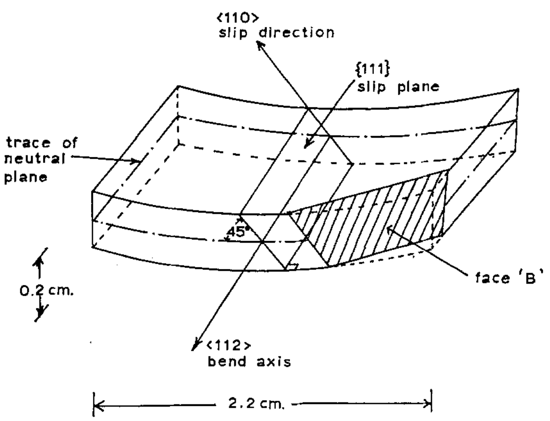


Figure 2 Specimen orientation for plastic bending.

slip occurred on only one system during bending. The polar nature of the etching properties and its calibration by Warekois [2] was utilised to determine the polarity of each sample, in the manner detailed previously [5]. In the rest of this paper, bending to introduce an excess of In-dislocations will be termed "In-bending" and bending to introduce an excess of Sb-dislocations will be termed "Sb-bending".

Specimens were deformed in an atmosphere of 90% nitrogen, 10% hydrogen, by means of the four-point bending jig shown in fig. 3.

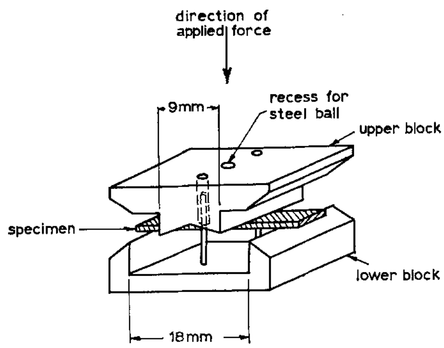


Figure 3 The four-point bending jig.

Loosely-fitting guide rods allowed the upper block a small degree of rotation, so that both inner knife-edges could rest on a specimen even if its upper and lower surfaces were not quite parallel. A ball and socket joint ensured axiality of loading when the knife-edges were driven together by a compression cage fitted to an Instron machine (model TM-M-L). The bending jig and compression cage were surrounded by a resistance furnace fed from a constant voltage transformer. Variations in temperature along the length of the specimen were less than 1° C, and during the course of an experiment the temperature at any point did not fluctuate by more than ±1° C.

From the relations of Timoshenko and Goodier [11] (which strictly apply only to elastic bending), the following formula for the resolved shear stress (σ) was obtained as

$$\sigma = \frac{3\,F(l_0 - l_i)}{2\,bd^2} \qquad (1)$$

where F is the applied force; $2l_0$, span of outer knife-edges; $2l_i$, span of inner knife-edges; b, beam width; and d, beam depth. From the relations of Bruneau and Pratt [12] (again for elastic bending), the glide strain (ϵ) was obtained as

$$\epsilon = \frac{\gamma\, d}{l_i\,(l_0 - l_i) + \frac{1}{3}\,(l_0 - l_i)^2 + \frac{1}{3}\,d^2} \qquad (2)$$

where γ is displacement of the inner knife-edges relative to the outer ones. Using equations 1 and 2 and the imposed deflection rate, the recorded load versus time plots were converted to graphs of shear stress versus glide strain.

After bending, the curvature of specimens was measured by two techniques. First, the silhouette was photographed and the shape-curvature measured by comparison with a series of arcs. The curvature between the inner knife-edges was quite uniform and its radius could be measured by this technique to within 5%. Second, the lattice curvature was measured by a Laue back-reflection X-ray technique. Patterns obtained with a collimated beam, incident at a series of positions across the bend axis, were superimposed on the one film. The lattice curvature between the inner knife-edges was found to be uniform, and the radius of lattice curvature, which could be estimated to within 10%, agreed with the shape-curvature in all cases.

All the bent specimens were sectioned to expose the octahedral face 'B' (fig. 2), and the direction of bend was confirmed. This type of sectioning was used since edge dislocations lying on the primary slip plane should intersect this face at a steep angle. Certain specimens were also sectioned parallel to the primary slip plane in order to reveal dislocations produced by non-primary slip.

The effects of the following etchants were studied.

Modified CP4 etch: 2 parts HNO_3:1 part HF:1 part acetic acid.

Butylamine etch: modified CP4 etch + 0.5% butylamine.

H_2O_2 etch: 1 part H_2O_2:1 part HF:8 parts H_2O + 0.4% butylthiobutane.

No clearly-defined etch pits on Sb-surfaces were obtained with any of these three etchants, and thus all etch-pit measurements given will refer to pits on In-surfaces. Etch-pit densities on the 'B' face were converted to densities on the {112} face perpendicular to the bend axis by dividing by cos 19°. Etch-pit numbers were estimated at a magnification which gave about 200 pits on the projection screen of the microscope; this ensured accuracy of ±7% in the etch-pit density at 95% confidence limits.

3. Results

3.1. Plastic Bending at 270° C

Typical stress-strain curves for the plastic bending of two series of specimens are shown in fig. 4; in one series, the specimens were In-bent and in the other they were Sb-bent. The well-defined yield points are the first reported for the bending deformation of InSb, although similar yield points have been observed in tension (Schäfer *et al* [13]). After the yield drop, flow continued at a constant stress up to the maximum glide strains encountered in these experiments, viz. 5% (specimen curvature of 0.25 cm^{-1}). The etch-pit density on face 'B' at all strains was more than 20 times larger than that on a face parallel to the primary slip plane. This lack of non-primary slip dislocations accounts for the absence of work-hardening.

In view of the results of Peissker *et al* [14], who found that, in creep experiments, the constant creep rate was higher for In-bending than for Sb-bending, it is interesting to compare the stress-strain curves for In- and Sb-bending. In the case of the two specimens C3 and C4 of fig. 4, the lower yield stress for Sb-bending is about 34% greater than that for In-bending. This difference is quite typical, as shown by the collected results in table I. In this table, specimens C1 to C8 were all taken from crystal C ($\rho_0 = 2.1 \times 10^3/cm^2$), whilst specimens E1 to E4 were taken from crystal E ($\rho_0 = 1.6 \times 10^4/cm^2$). In view of the difference in initial etch-pit densities, it is not surprising that the values of the lower yield stresses for a given sign of bending are different for the two crystals. The higher yield stress is associated with the lower ρ_0, as observed by Bell and Bonfield [15] for the case of Ge.

After bending, the etch-pit distributions in these specimens were investigated. Fig. 5 shows the way the etch-pit densities (with modified CP4) on a face 'B' varied with distance perpendicular to the neutral plane. At the neutral plane, the Sb-bent samples had a very low etch-pit density which did not increase as the curvature increased, and the pits were either aligned along the trace of the primary slip plane, or randomly distributed. In-bent samples, however, had a higher etch-pit density near the neutral plane than the corresponding Sb-bent samples, and the density in this region did increase as the curvature increased. Furthermore, near the neutral plane, the pits were aligned *perpendicular* to the trace of the primary slip plane. Fig. 6a contrasts this polygonised structure with that in an Sb-bent sample in fig. 6b.

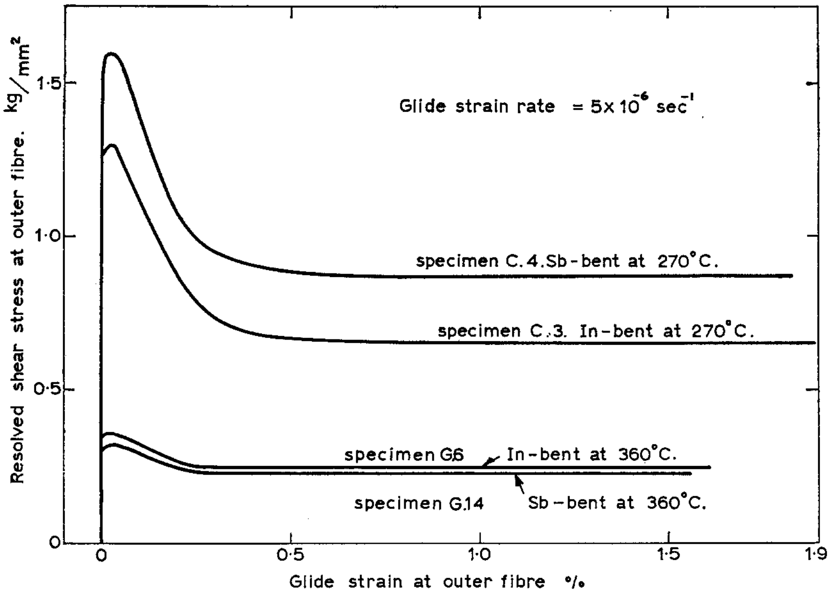


Figure 4 Typical stress-strain curves obtained on bending.

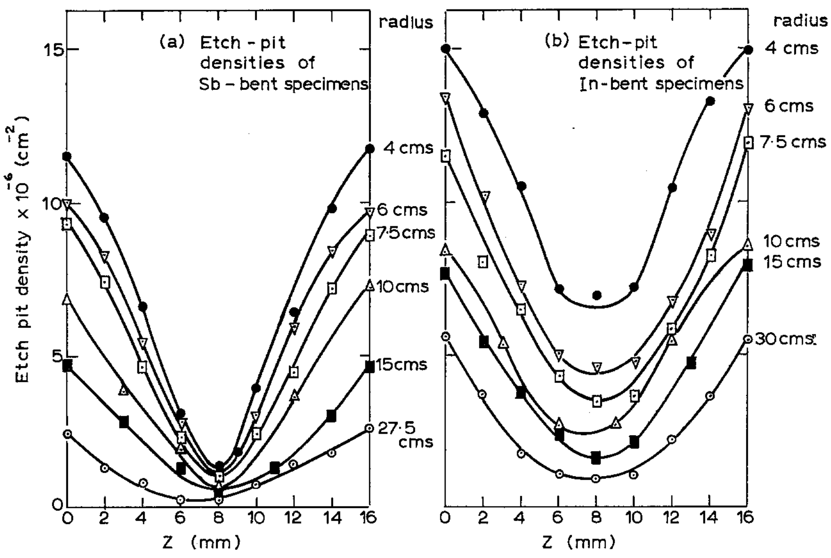


Figure 5 Etch-pit densities of In- and Sb-bent specimens (modified CP4 etch), shown as a function of *Z*, distance perpendicular to the neutral plane. These specimens were bent at 270° C.

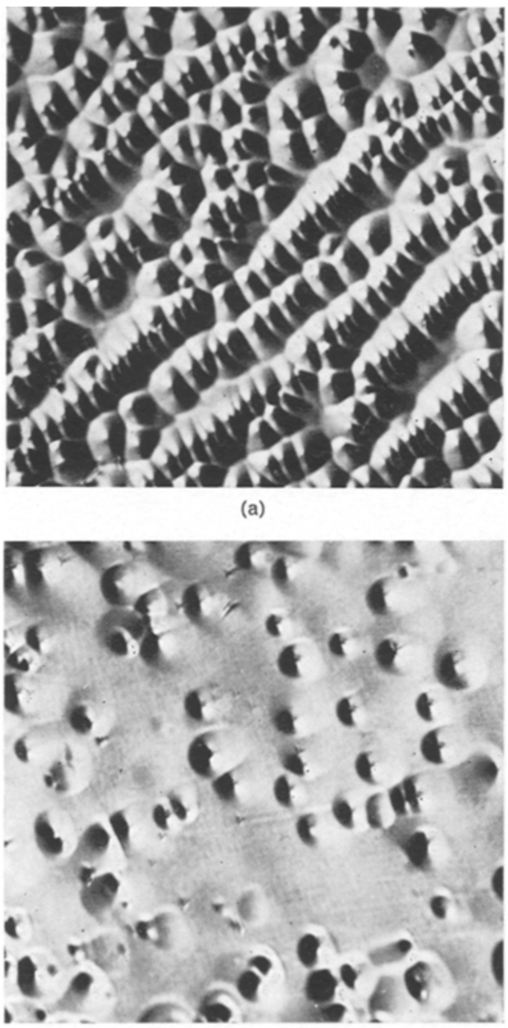
Fig. 7 shows the etch-pit densities, in one In-bent and one Sb-bent sample, as a function of distance perpendicular to the neutral plane, as revealed by: (a) the CP4 etch; and (b) the butylamine etch. For both samples, the butylamine etch produced more pits than the CP4 etch; but the difference was much greater in the Sb-bent sample. This is qualitatively consistent with the behaviour predicted by Lavine *et al* [10], viz. that the butylamine etchant reveals both In- and Sb-dislocations and the CP4 etchant reveals only In-dislocations.

3.2. Plastic Bending at 360° C

Stress-strain curves for an In-bent and an Sb-bent sample deformed at 360° C are shown in fig. 4, beside those of two specimens bent at 270° C. The samples bent at 360° C also show

TABLE I Lower yield stresses σ_{ly} of specimens bent at 270° C.

In-bent specimens		Sb-bent specimens	
Specimen no.	σ_{ly} (kg/mm²)	Specimen no.	σ_{ly} (kg/mm²)
C1	0.6566	C2	0.850
C3	0.650	C4	0.87
C5	0.605	C6	0.950
C7	0.572	C8	0.801
E1	0.475	E2	0.562
E3	0.445	E4	0.543



(a)

(b)

Figure 6 Micrographs of etch pits near the neutral axis in samples bent to a radius of 6 cm at 270° C and etched with the butylamine etch: (a) In-bent; (b) Sb-bent (×800).

yield points, but these are less pronounced than for those bent at 270° C. The etch-pit density on a section parallel to the primary slip plane was again small, indicating that non-primary slip was insignificant.

In contrast with the specimens bent at 270° C, the lower yield stresses for In- and Sb-bending at 360° C were not significantly different (see, for example, the two specimens G6 and G14 of fig. 4, whose initial etch-pit densities were 2.5×10^3 and 3.0×10^3/cm² respectively). Etch-pit distributions in a pair of bent samples are shown in fig. 8. In all cases, the pit density (with a given etchant) varies by only about 15% with distance perpendicular to the neutral plane. Average values of the etch-pit density in samples bent to a range of curvatures are given in table II. On a microscopic scale, the pits were aligned in discrete walls perpendicular to the slip plane in both In-bent and Sb-bent samples.

3.3. Annealing of Bent Specimens

Following the derivation of Nye [16], it may be shown that, in a bent crystal containing dislocations of opposite sign, lying parallel to the bend axis,

$$\rho_{\text{maj}} - \rho_{\text{min}} = \frac{1}{R\mathbf{b}\cos\theta} \tag{3}$$

where ρ_{maj} is the density of edge dislocations of the sign accommodating the curvature; ρ_{min}, the density of edge dislocations of the opposite sign; R, the radius of curvature; and θ, the angle between slip plane and neutral plane. If a sample is annealed at a temperature near the melting point, the density of minority sign dislocations will tend to zero as annihilation proceeds. Thus, a bent and annealed crystal provides a sensitive test of the ability of an etch to reveal dislocations of a particular sign.

Fig. 7 shows the effect of annealing specimens bent at 270° C. After 7 days at a temperature within 15° C of the melting point, the etch-pit density was invariant with distance perpendicular to the neutral plane. The high density in the outer regions had fallen, whilst that in the region of the neutral plane had actually increased. Etch pits were aligned in polygon walls perpendicular to the slip plane. In the case of the annealed In-bent specimen, both the modified CP4 and butylamine etchants gave etch-pit densities which were close to the theoretical dislocation density. In the case of the annealed

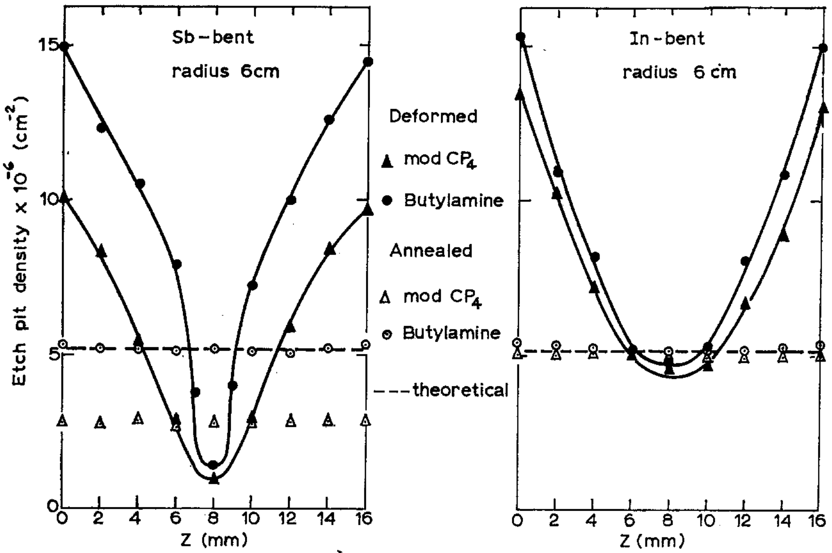


Figure 7 Etch-pit densities shown as a function of distance perpendicular to the neutral plane in an In-bent and an Sb-bent sample before and after annealing. Specimens were bent at 270° C and subsequently annealed at 512° C for 7 days.

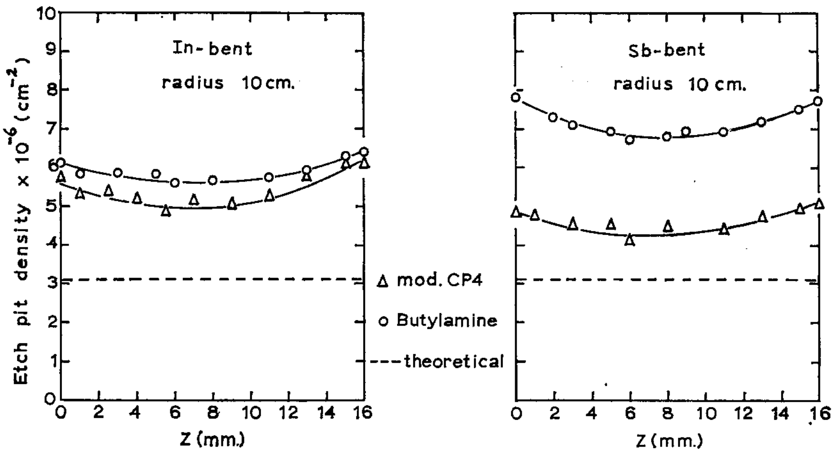


Figure 8 Etch-pit densities as a function of distance perpendicular to the neutral plane in specimens bent at 360° C.

Sb-bent specimen, however, the butylamine density was close to the theoretical value, while the modified CP4 density was about half this number. Specimens bent to curvatures from 4 to 30 cm, and annealed, showed a similar behaviour, as shown by the collected results in fig. 9. The micrographs in fig. 10 show typical areas of annealed Sb-bent and In-bent samples after treatment with each etchant. These structures indicate that In-bent samples have a greater tendency to polygonise than the Sb-bent samples.

The behaviour of the H_2O_2 etch (section 2) was examined on these same samples bent at 270° C and annealed at 512° C. The results did not substantiate the suggestion of Lavine *et al* [10] that this etch is specific to Sb-dislocations. Rather did it appear to show a preference for the In-dislocations.

When specimens which had been deformed at 360° C were annealed, there was no significant change in etch-pit density, although the average density was in all cases higher than that predicted from the bend radius by equation 3. It seems, therefore, that minority sign dis-

TABLE II Etch-pit densities on samples bent at 360° C.

In-bent specimens

Specimen no.	ρ_{CP4} (cm^{-2})	ρ_{but} (cm^{-2})	ρ_{Sb} (cm^{-2})	ρ_{In} (cm^{-2})	R (cm)	ρ_{theor} ($1/R\mathbf{b}\cos\theta$) (cm^{-2})	$\rho_{In} - \rho_{Sb}$ (cm^{-2})
G6	5.2×10^6	5.82×10^6	1.24×10^6	4.58×10^6	10.0	3.09×10^6	3.34×10^6
G3	2.9×10^7	3.30×10^7	8.0×10^6	2.5×10^7	2.5	1.24×10^7	1.7×10^7
G1	1.25×10^6	1.40×10^6	0.30×10^6	1.10×10^6	30.0	1.06×10^6	0.8×10^6
G11	6.3×10^5	7.25×10^5	1.90×10^5	5.25×10^5	>50	$<6.2 \times 10^5$	3.35×10^5
G12	2.5×10^6	2.88×10^6	0.76×10^6	2.12×10^6	22.0	1.40×10^6	1.36×10^6
G2	2.8×10^6	3.18×10^6	0.76×10^6	2.32×10^6	20.0	1.55×10^6	1.56×10^6
G10	1.2×10^7	1.35×10^7	3.0×10^6	1.05×10^7	4.0	7.75×10^6	7.5×10^6

Sb-bent specimens

Specimen no.	ρ_{CP4}	ρ_{but}	ρ_{Sb}	ρ_{In}	R	ρ_{theor}	$\rho_{Sb} - \rho_{In}$
G4	1.1×10^6	1.71×10^6	1.21×10^6	0.50×10^6	30.0	1.06×10^6	0.71×10^6
G5	9.00×10^6	1.45×10^7	1.10×10^7	0.35×10^7	4.0	7.75×10^6	7.5×10^6
G8	2.0×10^6	3.1×10^6	2.2×10^6	0.9×10^6	22.0	1.40×10^6	1.30×10^6
G7	1.30×10^6	2.02×10^6	1.44×10^6	0.58×10^6	30.0	1.06×10^6	0.86×10^6
G13	3.26×10^6	5.05×10^6	3.58×10^6	1.47×10^6	13.0	2.38×10^6	2.11×10^6
G14	4.68×10^6	7.27×10^6	5.18×10^6	2.09×10^6	10.0	3.09×10^6	3.09×10^6
G9	6.18×10^6	9.6×10^6	6.84×10^6	2.76×10^6	6.5	4.75×10^6	4.08×10^6

locations were present, but that the stable, polygonised arrangement of the discolations somehow prevented annihilation.

4. Discussion

4.1. The Reliability of the Etch-Pit Techniques

The annealing experiments described in section 3.3 permit a quantitative assessment of the behaviour of two etchants.

It is simplest to assume that no minority sign dislocations remained after specimens bent at 270° C had been annealed within 15° C of the melting point for 7 days. It then follows that the butylamine etch revealed both In-dislocations and Sb-dislocations with equal efficiency, since the theoretical density was revealed in annealed In-bent and annealed Sb-bent samples. The CP4 etch must have revealed all the In-dislocations, since the theoretical density was revealed in annealed In-bent samples, but it does not seem to be entirely specific to In-dislocations, since it gave values about half that predicted from the curvature in annealed Sb-bent samples. It follows that the CP4 etch reveals all the In-dislocations present and about half of the Sb-dislocations.

Using this conclusion, it is possible to estimate the number of majority and minority dislocations in the samples bent at 360° C.

We have $$\rho_{but} = \rho_{In} + \rho_{Sb} \qquad (5)$$

and $$\rho_{CP4} \simeq \rho_{In} + \tfrac{1}{2}\rho_{Sb} \qquad (6)$$

Thus the densities of In- and Sb-dislocations can be obtained separately when the two etch-pit densities are available. Table II lists values of ρ_{In} and ρ_{Sb} calculated in this way. In the case of In-bent samples, ρ_{In} is about a factor of 3 to 4 greater than ρ_{Sb}, whilst in Sb-bent samples ρ_{Sb} is only a factor of 2 to 3 greater than ρ_{In}. The excess density of majority dislocations was then obtained by subtraction and is compared with that predicted by the Nye relation. This relation gives the excess density of majority dislocations in the absence of macroscopic elastic stresses, and thus strictly can only be applied to specimens which have been annealed. However, since the specimens bent at 360° C showed a uniform etch-pit distribution, the residual elastic stresses must have been low. Table II shows that the excess density of majority dislocations from etch-pit measure-

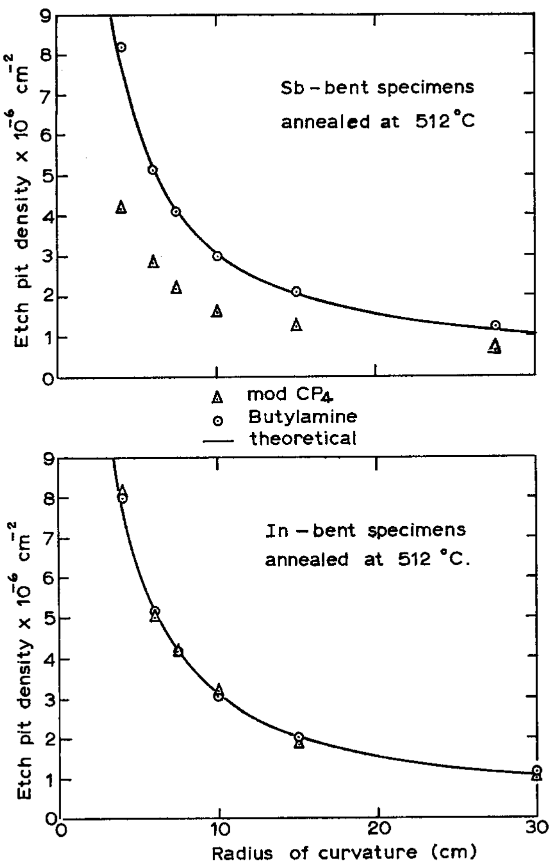


Figure 9 Etch-pit densities as a function of curvature in annealed samples.

ments agrees very well with that predicted by equation 3. In all cases, the difference between the etch-pit estimate and the theoretical estimate of the dislocation density is less than that which would arise from the maximum experimental errors. This correlation, therefore, gives confidence in the validity of the assumptions made.

4.2. Polygonisation Mechanisms

Duga and Maringer [17] reported that InSb does not polygonise even when heated within a few degrees of the melting point. However, this appears to be true only for specimens deformed by multiple slip. In the present experiments, samples bent at 270° C deformed on one set of slip planes only and showed little polygonisation, but after annealing close to the melting point it was very marked (see fig. 10). Vogel [18] found similar effects in Ge.

It is interesting to consider whether the sharply polygonised dislocation structures obtained in samples bent at 360° C (but not annealed) were a result of glide processes alone, or of a combination of glide and climb. Livingston [19] was able to show that glide polygonisation, in which edge dislocations of like sign stick in minimum energy positions when they attempt to pass on parallel slip planes, can occur in copper at temperatures well below those necessary for climb. The mechanism of polygonisation in InSb at 360° C, however, is not so clear. The small applied stress (a consequence of the small applied strain rate) would favour the formation of polygon walls by a glide process. Nevertheless, following the arguments of Bell *et al* [5], it seems very likely that climb, rather than double cross-glide, played a part in the transfer of slip from one plane to another, and possibly, therefore, in the formation of polygon walls too.

4.3. The Differences between In-Bending and Sb-Bending

The etch-pit distribution in samples bent at 270° C point to a simple explanation of the observed difference between the yield stresses for In- and Sb-bending.

In a bending experiment, the first dislocation sources to operate must be at or near the surface where the stresses are greatest, and dislocations of the sign required to accommodate the curvature will move towards the neutral axis. The stress will fall off as the neutral axis is approached, and so the closest distance of approach of the dislocations to the neutral axis gives an indication of the minimum stress required to move them. In specimens bent to the same lattice curvature, the etch-pit measurements showed that the dislocations moved much closer to the neutral axis on In-bending than on Sb-bending, even though the applied load was greater in the latter case. Thus, one simple explanation is that In-dislocations glide more readily than Sb-dislocations.

An alternative can be obtained in terms of their different tendencies for climb. The annealing experiments of section 3.3 indicated that In-dislocations climb more readily than Sb-dislocations. Different tendencies for climb (leading to the propagation of slip from one plane to another) could result in differences in the lower yield stress for In- and Sb-bending.

5. Conclusions

(a) It has been shown that etch-pit techniques

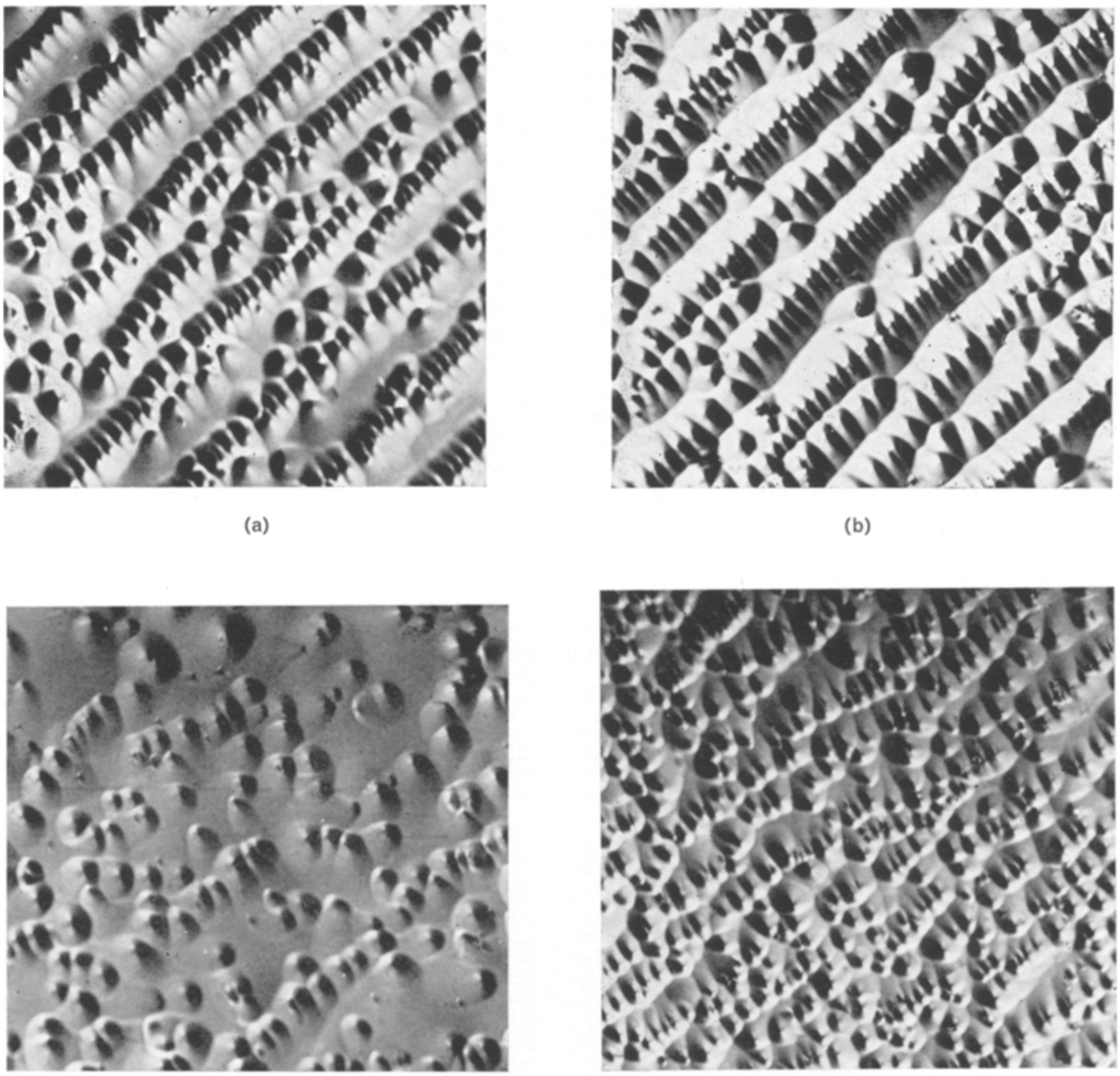


Figure 10 Micrographs of etch pits in deformed and annealed specimens (×800). Both specimens were bent to a radius of 6 cm and annealed a 512° C for 7 days. In-bent specimen: (a) modified CP4 etchant; (b) butylamine etchant. Sb-bent specimen: (c) modified CP4 etchant; (d) butylamine etchant.

give a reliable estimate of the dislocation density in InSb. The butylamine etch reveals In- and Sb-dislocations with equal efficiency, whilst modified CP4 reveals all the In-dislocations and about half the Sb ones.

(b) Bending at 270° C involved a higher value of the lower yield stress for Sb-bending than for In-bending. This may be due either to the glide mobility of In-dislocations being greater than that of Sb-dislocations, or to the more rapid climb of the former.

Acknowledgements

This work was carried out in the Department of Metallurgy, Imperial College, and the authors are indebted to Professor J. G. Ball for provision of laboratory facilities. Financial support was provided by the Science Research Council, the Ministry of Defence (Navy), and the Trustees of the Beit Fellowship for Scientific Research. Grateful thanks are extended to these bodies, and also to Drs J. B. Mullin, K. F. Hulme, and colleagues at R R E Malvern, who supplied the crystals, and also took part in many useful discussions. Dr D. B. Holt kindly read the manuscript and made useful comments. The paper is published by permission of the Controller, HM Stationery Office.

References

1. J. W. ALLEN, *Phil. Mag.* **2** (1957) 1475.
2. E. P. WAREKOIS, Lincoln Lab. Quarterly Prog. Report (15 July 1959), p. 45.
3. P. HAASEN, *Acta Met.* **5** (1957) 598.
4. J. D. VENABLES and R. M. BROUDY, *J. Appl. Phys.* **29** (1958) 1025.
5. R. L. BELL, R. LATKOWSKI, and A. F. W. WILLOUGHBY, *J. Matls. Sci.* **1** (1966) 66.
6. H. C. GATOS and M. C. LAVINE, *J. Electrochem. Soc.* **107** (1960) 427.
7. D. B. HOLT, *J. Appl. Phys.* **31** (1960) 2231.
8. H. C. GATOS and M. C. LAVINE, *J. Appl. Phys.* **31** (1960) 743.
9. H. C. GATOS and M. C. LAVINE, *J. Phys. Chem. Solids* **14** (1960) 169.
10. M. C. LAVINE, H. C. GATOS, and M. C. FINN, *J. Electrochem. Soc.* **108** (1961) 974.
11. S. TIMOSHENKO and J. N. GOODIER, "Theory of Elasticity" (McGraw-Hill, 1951).
12. A. A. BRUNEAU and P. L. PRATT, *Phil. Mag.* **7** (1962) 1871.
13. S. SCHÄFER, H. ALEXANDER, and P. HAASEN, *Phys. Stat. Sol.* **5** (1964) 247.
14. E. PEISSKER, P. HAASEN, and H. ALEXANDER, *Phil. Mag.* **7** (1961) 1279.
15. R. L. BELL and W. BONFIELD, *Phil. Mag.* **9** (1964) 9.
16. J. F. NYE, *Acta Met.* **1** (1953) 153.
17. J. J. DUGA and R. E. MARINGER, *Bull. Am. Phys. Soc.* **5** (1960) 375.
18. F. L. VOGEL, *J. Metals* **8** (1956) 946.
19. J. D. LIVINGSTON, *J. Austr. Inst. Metals* **8** (1963) 15.