

on the other hand, the corresponding data for S, Se, Te and their neighbours indicates that their valency is 4. It may thus be assumed that the valency of the latter group, in their semi-conducting compounds characterized by intensive contraction, is 6. This supposition may probably be verified with the aid of the Mössbauer effect.

References

1. M. V. NEVITT, "Intermetallic Compounds", edited by J. H. Westbrook (Wiley New York, 1967).
2. P. S. RUDMAN, *Trans. Met. Soc. AIME* **233** (1965) 873.
3. I. M. CHAPNIK, *Phil. Mag.* **32** (1975) 673; *Phys. Stat Sol. (a)* **37** (1976) K29.
4. W. B. PEARSON, "A Handbook of Lattice Spacings and Structures of Metals and Alloys", Vol. 2 (Pergamon Press, Oxford, 1967).
5. "Structure Reports", Vols. 31A–39A (Oosthoek, Scheltema and Molkema, Utrecht, 1966–1973).

6. M. W. KING, "Physical Metallurgy", edited by R. W. Cahn (North-Holland, Amsterdam, 1970) p. 33.
7. C. REALE, *Appl. Phys. Letters* **27** (1975) 157.
8. M. H. VAN MAAREN, *Phys. Letters* **29A** (1969) 293.
9. P. C. DONOHUE, W. J. SIEMONS and J. L. GILLSON, *J. Phys. Chem. Solids* **29** (1968) 807.
10. R. G. YAROVAYA, I. YU. RAPP, *Fiz. Met. Metalloved* **31** (1971) 100.
11. J. H. WEAVER, D. W. LYNCH and R. ROSEI, *Phys. Rev.* **B5** (1972) 2829.
12. R. J. BARLETT, D. W. LYNCH and R. ROSEI, *ibid* **B3** (1971) 4074.

Received 25 August
and accepted 10 September 1976

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Non-axial dislocations in reaction-sintered silicon nitride

The exploitation of silicon nitride as an engineering material necessitates an understanding of the deformation, creep and fracture characteristics of both the hot-pressed and reaction-sintered forms of the material. These characterizations obviously require knowledge of the defect microstructures that are likely to occur. This information is most readily obtained by the use of transmission electron microscopy and in the literature there have been several reports [1–4] of examinations of the microstructure using this technique. One common feature of all these reports was the lack of complete unambiguous characterization of the dislocations that are frequently observed. Ambiguity arises because although these workers found it possible to identify dislocations with a Burgers vectors along the *c* axis they could not unambiguously identify those dislocations which also possessed basal plane components in their Burgers vector. The difficulty which was encountered with these non-axial dislocations was the inapplicability of the standard $\mathbf{g} \cdot \mathbf{b} = 0$ invisibility due to the failure to obtain total invisibility of the dislocation image for some reflections. This factor coupled with the frequent observation of multiple images has led to the suggestion by Evans and Sharp [4] that this behaviour may be due to a dislocation

dissociation. Since the Burgers vector for the $\frac{1}{3} \langle 11\bar{2}3 \rangle$ dislocation is large in magnitude, a dissociation would appear to be a probable occurrence but it is also possible that the observed contrast behaviour is related to the complex crystal structure of the material. It is clearly important to establish whether dislocations with a $\frac{1}{3} \langle 11\bar{2}3 \rangle$ Burgers vector are present and if so whether they are dissociated since there have been no reports of the presence of the smaller Burgers vector $\frac{1}{3} \langle 11\bar{2}0 \rangle$. A study has therefore been made of the two possible explanations of the observed contrast behaviour and in this letter we report the results.

The observations in this note were obtained from thin foils of reaction-sintered silicon nitride. Specimens suitable for electron microscopy were obtained by a combination of mechanical grinding and ion-beam thinning. After thinning, the specimens were lightly coated with carbon to minimize charging effects and then examined in a Philips EM300 electron microscope.

In order to be able to unambiguously identify the presence of dislocations with a non-axial Burgers vector, it is first necessary to eliminate from considerations those dislocations which possess Burgers vectors in the *c* direction. This is most easily achieved by imaging the dislocations using the six basal $\langle 20\bar{2}0 \rangle$ and $\langle 11\bar{2}0 \rangle$ type reflections. The visibilities and invisibilities given

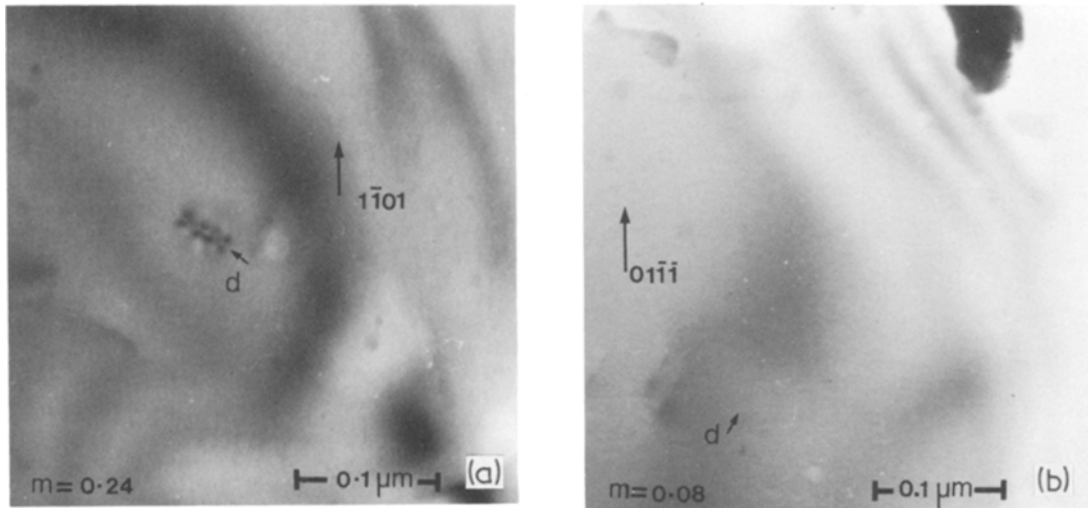


Figure 1 Dislocations with a Burgers vector of $\frac{1}{3} \langle 11\bar{2}3 \rangle$ imaged using $g = \langle 10\bar{1}1 \rangle$ showing examples of (a) weak contrast and (b) invisibility depending on the value of the term $m = \frac{1}{3} \mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u}$.

by these reflections then not only identified the *c* axis dislocations, invisible for all these reflections, but also establishes the basal plane components of the dislocations that remain visible. The complete Burgers vector for these remaining dislocations can now be established by obtaining an invisibility from an appropriate non-basal reflection. In common with previous studies this was found extremely difficult and in the majority of cases weak contrast rather than invisibility was obtained. However, it is a well established feature

of contrast from dislocations that total invisibility may not appear for certain combinations of Burgers vector (*b*), diffracting vector (*g*) and dislocation line vector (*u*). It is generally accepted that the magnitude of the term $m = \frac{1}{3} \mathbf{g} \cdot \mathbf{b} \wedge \mathbf{u}$ gives an approximate indication of the possibility of obtaining complete invisibility for $\mathbf{g} \cdot \mathbf{b} = 0$ high values normally not giving invisibility for $\mathbf{g} \cdot \mathbf{b} = 0$. The factor *m* was therefore determined, using standard stereographic techniques, for those dislocations where weak contrast was observed,

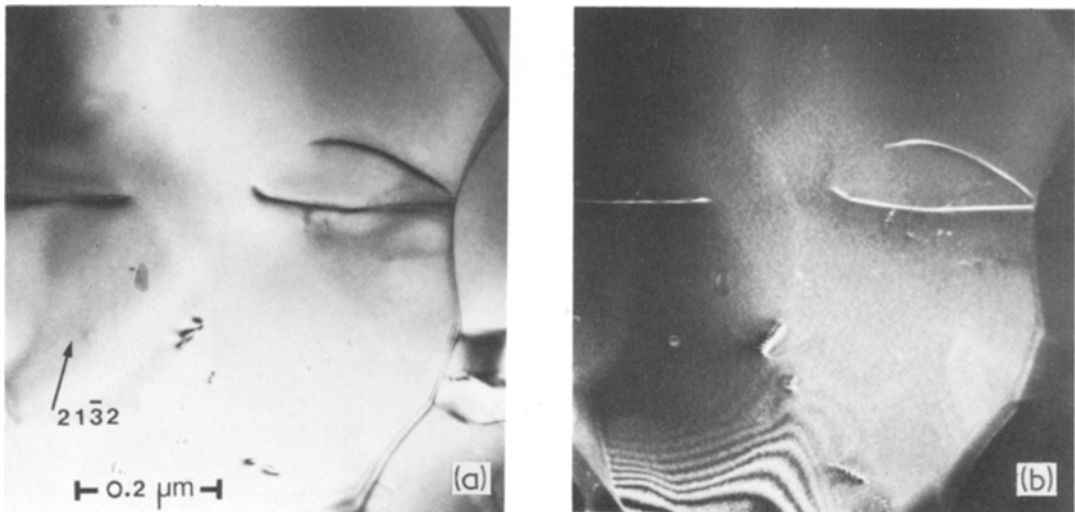


Figure 2 Dislocation with a Burgers vector of $\frac{1}{3} \langle 11\bar{2}3 \rangle$ imaged in (a) bright-field and (b) dark-field, showing the loss of multiple image in dark-field conditions.

assuming that weak contrast was indicative of $g \cdot b = 0$. It was found that in the majority of cases the term m was greater than 0.1 and for those cases where it was lower complete invisibility could be obtained if care was taken to establish the best possible two beam conditions. This is illustrated in Fig. 1 which shows an example of the contrast corresponding to high and low values of m . As long as these precautions were taken, it was found possible to unambiguously identify the presence of the Burgers vector $\frac{1}{3} \langle 1 \ 1 \ \bar{2} \ 3 \rangle$. Although the importance of the term m explains the difficulty that is encountered in obtaining invisibilities, it does not provide a reason for the frequent observation of multiple images. The most common cause of this type of behaviour is the failure to obtain exact two-beam diffraction conditions which causes non-systematic reflections to contribute to the image. The effect of these non-systematic reflections can be eliminated by the use of complementary bright- and dark-field electron microscopy. Of the many instances of multiple images that we have observed, none were indicative of a dislocation dissociation since the multiple image was always lost in dark-field conditions. A typical example of this type of behaviour is shown in Fig. 2a and b which shows the same area imaged for both bright and dark field conditions. It is also worth noting that when the dislocation is imaged close to the Bragg condition it is also quite possible for a symmetrical double image to result from an undissociated dislocation.

From these results it is apparent that the difficulties encountered by other workers in identifying the $\frac{1}{3} \langle 1 \ 1 \ \bar{2} \ 3 \rangle$ Burgers vector is a result of contrast effects caused by the nature of the dislocations. Although the variation of dislocation

image contrast with dislocation character is a well known effect it is worth stressing its special significance to many ceramic materials. This arises from the fact that many ceramics possess large unit cells, often containing several atoms of different elements, which often results in large Burgers vectors. It is therefore obviously necessary when using transmission electron microscopy to determine Burgers vectors to take both full account of the effect that this can have on the dislocation images and to exercise extreme care in the setting of the experimental conditions.

Acknowledgements

The authors wish to thank the Directors of Pilkington Bros Ltd and Dr D. S. Oliver for permission to publish this letter.

References

1. A. G. EVANS and J. V. SHARP, *J. Mater. Sci.* 6 (1971) 1292.
2. E. BUTLER, *Phil. Mag.* 21 (1971) 829.
3. R. KOSSOWSKY, *J. Mater. Sci.* 8 (1973) 1603.
4. A. G. EVANS and J. V. SHARP, "Electron Microscopy and Structure of Materials" (California Press, 1972) p. 1141.

Received 25 August

and accepted 10 September 1976

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Reply to "A discussion of flip-flop decomposition"

In offering a description of a possible mode of decomposition within miscibility gaps, it is my contention that the proposed model is not in agreement with the usual interpretation of Cahn's analysis but this is not intended to imply that the model is not consistent with Cahn's Equation 18 [1]. We did refer to the process as spinodal

decomposition, but with some reservations because this term has been so widely and loosely used as to be almost devoid of meaning. They correctly point out that Hillert [2] had already considered this model, a point which we unfortunately failed to note. While the model would include the case where the initial particles formed during cooling this is, we feel, unnecessarily restrictive.

If, as we believe, Cahn's Equation 18 [1] is applicable to the model in question then its