TABLE I Core energy of dislocations in some crystals (in electron volts)

| Crystal | $hkl$ | Etchant | $E_t$ | $E_d$ | $E_{co}^{11}$ | Reference | $E_{co}^{13}$ | | $E$ [9] |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Screw | Edge | |
| MgO | 100 | $HNO_3$ | 0.68 | 0.59 | 1.58 | [7] | 3.5 | 4.3 | 41 |
| | | HCl | 0.68 | 0.55 | 1.52 | | | | |
| | | $CH_3COOH$ | 0.81 | 0.66 | 1.55 | | | | |
| $BaF_2$ | 111 | $HNO_3$ | 0.68 | 0.39 | 0.62 | Authors's data | 2.0 | 2.3 | 24 |
| | | HCl | 0.68 | 0.39 | 0.62 | | | | |
| NaCl | 100 | $CH_3COOH$ | 0.67 | 0.37 | 0.37 | Author's estimation according to [8] | 0.56 | 0.75 | 8 |

of $E_{co}^{11}$ and $E_{co}^{13}$ in the series MgO, $BaF_2$, NaCl accords with the decrease in lattice energy of these crystals, $E$ (Table I). Correlation of the values of $E_{co}^{11}$ with $E_{co}^{13}$ and $E$, and the independence of the dislocation core energy on etchant and solvent is evidence of the correct estimation of the dislocation core energy from dissolution and etching data.

## References

1. W. H. ROBINSON, in "Techniques of Metal Research", edited by R. F. Bunshah (Interscience Publishers, New York, London, Sydney, Toronto, 1968) pp. 291–340.
2. E. M. VORONKOVA, B. N. GRECHUSHNIKOV, G. J. DISTLER and J. P. PETROV, "Optical Materials for IR-Technique", (Nauka, Moscow, 1965).
3. V. K. KULIFEEV, V. J. PANSHISHNYI, B. D. ROGOZHIN and V. A. SOKOLOV, *Izv. Vyss. Ucheb. Zaved., Tsvet. Metall.* 1 (1969) 80.
4. B. V. NEKRASOV (Ed.), "Kratkiy Spravochnik Himika", (Goshimizdat, Moscow, 1954) pp. 288–92.
5. A. E. SMIRNOV and A. A. URUSOVSKAYA, *J. Mater. Sci.* 15 (1980) 1183.
6. W. SCHAARWACHTER, *Phys. Stat. Solidi* 12 (1965) 375.
7. K. SANGWAL, *Kristall Technik* 14 (1979) 965.
8. G. K. BARANOVA and E. M. NADGORNYI, *Kristallog.* 17 (1972) 875.
9. K. P. MITSHENKO and A. A. RAVDEL (Eds.), "Kratkiy Spravochnik Fiziko-Himicheskih Velichin", (Himiya, Leningrad, 1974) p. 57.

*Received 28 August
and accepted 10 October 1980*

A. E. SMIRNOV
A. A. URUSOVSKAYA
*Institute of Crystallography
USSR Academy of Sciences
Moscow
USSR*

---

## *A mode of deformation in partially stabilized zirconia*

This work describes the nature of deformation patterns associated with identations and scratches in partially stabilized zirconia. Surface deformation bands could be highlighted by chemical or ion-beam etching of the surface. The nature of the bands has been investigated using optical and transmission electron microscopy.

Partially stabilized zirconias have recently attracted considerable attention as a possible ceramic engineering material because of their exceptional mechanical properties [1–6]. In these materials a two- or three-phase microstructure exists which consists of a cubic stabilized matrix with monoclinic and/or tetragonal zirconia precipitates. It is the metastability of the tetragonal precipitates combined with the volumetric expansion and shear strains associated with their transformation to the monoclinic form that is responsible for the good mechanical properties of the material.

The materials used in this study are magnesia and calcia partially stabilized zirconia (Mg- and Ca-PSZ respectively). Previous studies of these materials have shown that the mechanical properties may be optimized by choosing suitable com-

0022–2461/81/051428–04$02.40/0

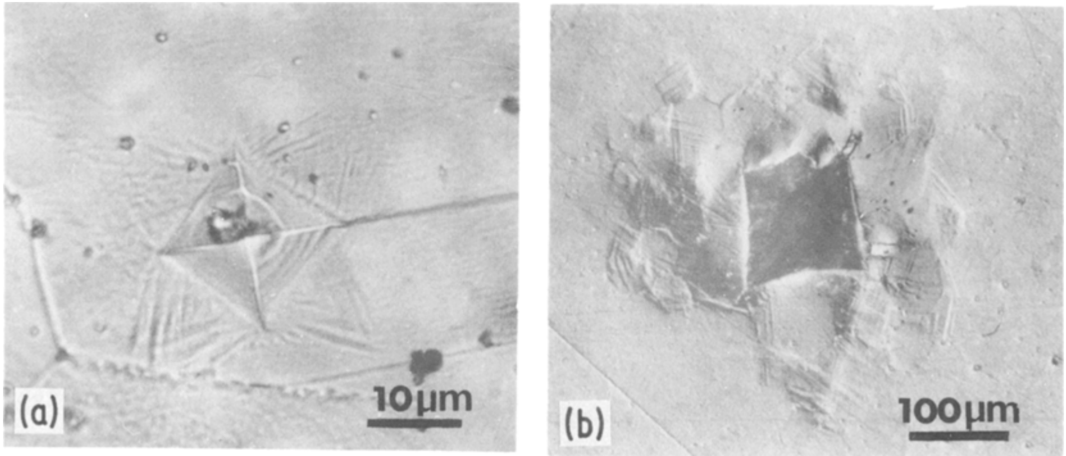

*Figure 1* **Optical micrographs of indentations, using Nomarski illumination. (a) Ca–PSZ ion etched and thinned foil, indent produced using 3 N load. (b) Mg-PSZ indented with 100 N load on unetched surface.**

positions and heat treatments. For a given composition the heat treatment required to give optimal properties is referred to as peak ageing. The present specimens were in various stages of under-ageing. This condition was chosen to ensure that the transformed precipitates observed were a result of the test programme and not of the thin-foil preparation process. Specimen surfaces were prepared using standard metallographic techniques and were polished to one micrometre finish. Indentations were made with a Vickers pyramid indenter using various loads between 3 and 200 N. In early experiments, indented surfaces were etched in a hydrofluoric acid solution. However, subsequent work showed that ion etching for one to five hours with 5 kV Ar ions revealed the deformation patterns better.

A deformation pattern about an indentation in Ca-PSZ is shown in Fig. 1a. The characteristic aspects of the deformation are the "slip-band"-like features emanating from the faces of the pyramidal impression. Closer inspection of these bands, near to an indentation and within a single grain, reveals that they are not strictly crystallographic and are indeed slightly curved. However, the features are almost always symmetric about the faces of the impression. The bands follow very closely the slip line pattern predicted from slip line field theory for deformation beneath a punch or very blunt cone [7]. With heavier indentation loads slip lines may be observed in grains positioned at three or four grain diameters from the impression, as shown for Mg-PSZ in Fig. 1b.

Generally, the slip line regions are situated in a triangular manner about the faces of the impression with little or no slip behaviour about the corners, as shown in Figs 1a and 2. Slip bands in grains not adjacent to the indentation site tend to be continuous through the grain and highly crystallographic, although the orientation of the slip traces have not been ascertained. It appears, however, that the traces occur preferentially in those grains where slip bands can orientate close to 45° to radial directions from the indentation; i.e. in the directions of maximum shear. A good example of this is shown in Fig. 1b.

After indentation only minor cracking was observed; on subsequent ion thinning cracks connecting closely spaced indentations appeared. In some of our peak-aged Mg-PSZ materials little

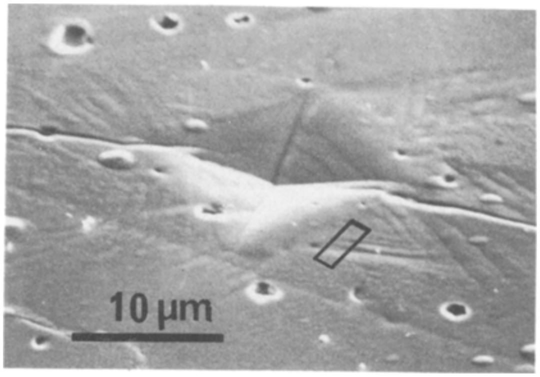

*Figure 2* Scanning electron micrograph of indent in Ca-PSZ (3 N load) showing region of TEM in Fig. 3.

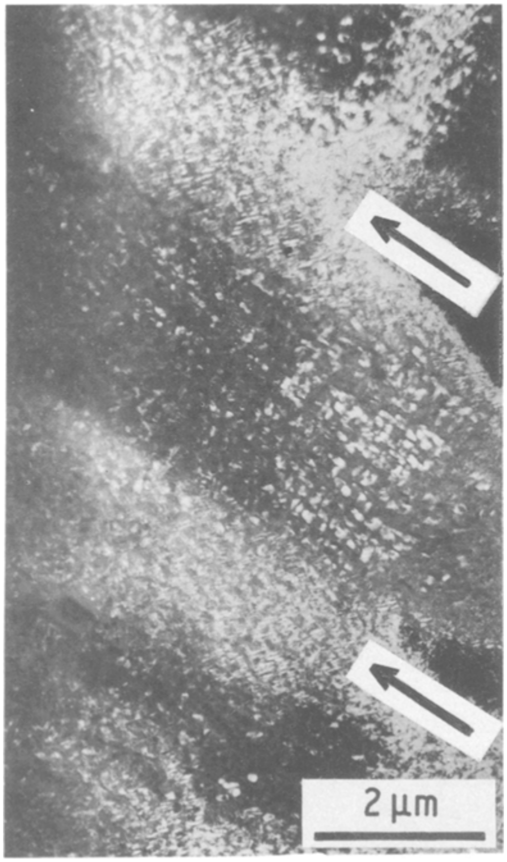

*Figure 3* Dark-field TEM of slip bands adjacent to an indentation. For description see text.

or no radial or Palmquist cracking was observed, even at loads of 200 N (see Fig. 1b).

Transmission electron microscopy (TEM) of the indented region failed to reveal the presence of dislocations. The most commonly observed feature outside the intense inner deformation zone was regularly spaced bands of transformed precipitates. Fig. 2 shows a scanning electron micrograph of the slipped regions about an indentation in underaged Ca-PSZ. A TEM of an area similar to that indicated by the rectangle in Fig. 2 is shown in Fig. 3. This dark-field TEM image shows regularly spaced bands of coarsely twinned monoclinic precipitates (arrowed) separated by regions of very finely twinned monoclinic or untransformed tetragonal precipitates. The coarsely twinned monoclinic precipitates appear to have formed from favourably orientated tetragonal precipitates by a cooperative shear mode.

Similar bands of transformed precipitates were observed in the base of scratches resulting from the surface preparation process. This banded structure was observed for both Mg-PSZ and Ca-PSZ materials, as shown in Figs 4a and b. There was no evidence of cracking in the banded regions which might have iñitated transformation of favourably sized precipitates close to a crack tip.

The TEM observations support the optical observations of localized bands in which considerable shear strain occurs. The co-operative transformation of the precipitates and the shear strains associated with the transformation appear to be responsible for the observed shear bands. The curvature of the shear bands close to low-load indents indicates that the propagation of these bands is along maximum shear trajectories. This may occur due to the discrete shear associated with the transformation of a tetragonal precipitate and thus not be limited to particular crystallographic directions as for instance occurs with twinning. However, on the grounds of symmetry it would be favourable for slip bands to form in particular crystallographic directions, as shown in Fig. 4a. Further work is currently in hand to ascertain the mechanism of slip band initiation and propagation.

Finally, mention should be made concerning the lack of dislocation contrast in PSZ materials. A number of papers have been written describing the microstructure of PSZ materials [2, 3, 8], no observations of line defects have ever been reported, resulting either from "grown-in" or deformation processes. Similarly, in the present study no dislocation-type contrast was observed. However, in a recent study, Mecartney *et al.* have observed dislocations as a result of high-temperature deformation in a calca-stabilized zirconia [9]. In the present work, the reason for the lack of dislocation contrast, as a result either of the sintering or deformation process, is primarily due to the already highly strained precipitate-rich material. In such a material it has not been possible to establish the critical diffraction conditions necessary to observed dislocations.

## Acknowledgements

The authors wish to thank M. J. Bannister, N. Gane, N. A. McKinnon and M. J. Murray for comments and discussion.

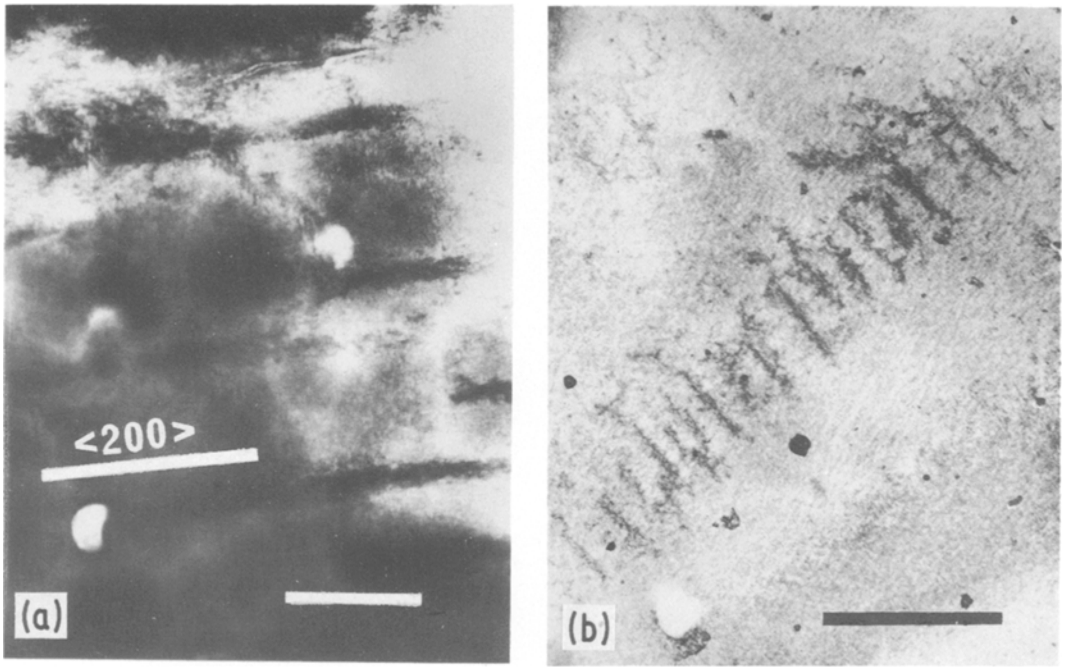

*Figure 4* Bright-field TEM showing the base of scratches in (a) Mg-PSZ and (b) Ca-PSZ. Bar length equals five micrometer.

## References

1. R. C. GARVIE, R. H. J. HANNINK and R. T. PASCOE, *Nature* **253** (1975) 703.
2. R. T. PASCOE, R. H. J. HANNINK and R. C. GARVIE, *Sci. Ceram.* **9** (1977) 447.
3. D. L. PORTER and A. H. HEUER, *J. Amer. Ceram. Soc.* **60** (1977) 183.
4. *Idem, ibid.* **62** (1979) 298.
5. S. T. GULATI, J. D. HELFINSTINE and A. D. DAVIS, *Ceram. Bull.* **59** (1980) 211.
6. R. C. GARVIE, R. H. J. HANNINK and C. URBANI, *Ceramurgica* **6** (1980) 19.
7. R. HILL, "The Mathematical Theory of Plasticity" (Clarendon Press, Oxford, 1950).
8. R. H. J. HANNINK, *J. Mater. Sci.* **13** (1978) 2407.
9. M. L. MECARTNEY, W. T. DONLON and A. H. HEUER, *ibid.* **15** (1980) 1063.

*Received 1 September*
*and accepted 10 October 1980*

R. H. J. HANNINK
M. V. SWAIN
*CSIRO Division of Materials Science*
*Melbourne*
*Australia*

---

## *Optical properties of palmitic acid thin films*

Long-chain fatty acid films find numerous applications in optical devices [1–5]. The optical properties of thin films of palmitic acid formed by the Langmuir and Blodgett technique [6–9] and by vacuum evaporation [10] have been studied by various workers. In the present investigation the ion-plating technique [11] has been adopted to obtain the palmitic acid thin films and their optical properties are studied in detail.

Pure palmitic acid (99.5%; Eastmann, Kodak, New York) was evaporated from a molybdenum boat using an ion-plating technique in the presence of r.f. glow and deposited on to well-cleaned glass slides. The vacuum was maintained at about $2 \times 10^{-2}$ Torr. The r.f. power was kept at 150 W