70. M. P. VICTORIA and A. E. VIDOZ, *Phys. Stat. Sol.* **28** (1968) 131.
71. A. E. VIDOZ, *ibid* 145.
72. P. MORGAND, P. MOUTURAT, and G. SAINFORT, *Acta Met.* **16** (1968) 867.
73. G. SCHOECK, *Scripta Met.* **2** (1968) 283.
74. E. A. STARKE JR, J. C. OGLE, and C. J. SPARKS JR, in "Advances in X-ray Analysis" Vol. 12 (Plenum Press, New York, 1969) p. 372; *Trans. Met. Soc. AIME*, **245** (1969) 169.
75. J. J. SVITAK and R. M. ASIMOW, *Trans. Met. Soc. AIME* **245** (1969) 209.
76. R. A. KLOSKE and M. E. FINE, *ibid* 217.
77. G. SCHOECK, *Acta Met.* **17** (1969) 147.
78. G. E. POQUETTE and D. E. MIKKOLA, *Trans. Met. Soc. AIME* **245** (1969) 743.
79. J. R. BEELER JR, in "Int'l. Conf. on Phys. Met. of Ti" (1968), to be published.

Internal Friction

80. C. Y. LI and A. S. NOWICK, *Acta Met.* **9** (1961) 49.
81. A. S. NOWICK and D. P. SERAPHIM, *ibid* 40, 85.
82. D. O. WELCH and A. D. LECLAIRE, *Phil. Mag.* **16** (1967) 981.

Electrical Resistivity

83. J. B. GIBSON, *J. Phys. Chem. Solids* **1** (1956) 27.
84. A. E. ASCH and G. L. HALL, *Phys. Rev.* **132** (1963) 1047.
85. S. MATSUO and L. M. CLAREBOUGH, *Acta Met.* **11** (1963) 1195.
86. A. A. KATSNEL'SON and L. M. SCHEVCHUK, *Fiz. Met. Metall.* **24** (1967) 97.

Magnetic Behaviour

87. H. J. BUNGE and H. G. MÜLLER, *Z. Metallk.* **48** (1957) 26.
88. S. CHIKAZUMI, K. SUZUKI, and H. IWATA, *J. Phys. Soc. Japan* **12** (1957) 1259; **15** (1960) 250.
89. A. ARROTT and H. SATO, *Phys. Rev.* **114** (1959) 1420, 1427.
90. N. TAMAGAWA, Y. NAGAKAWA, and S. CHIKAZUMI, *J. Phys. Soc. Japan* **17** (1962) 1256.
91. G. E. BACON and J. CRANGLE, *Proc. Roy. Soc.* **A272** (1963) 387.
92. P. GEHLEN and J. B. COHEN, *Phys. Rev.* **139** (1965) A844.
93. G. Y. CHIN, *J. Appl. Phys.* **36** (1965) 2915; *Materials Science and Eng.* **1** (1966) 77.
94. G. P. HUFFMAN and R. M. FISHER, *J. Appl. Phys.* **38** (1967) 735.
95. G. Y. CHIN, E. A. NESBITT, J. H. WERNICK, and L. L. VANSKIKE, *ibid* 2623.
96. B. MOZER, D. T. KEATING, and S. C. MOSS, *Phys. Rev.* **175** (1968) 868.
97. A. KIDRON, *Phys. Rev. Lett.* **26a** (1968) 593; but see comments on this work by S. C. MOSS, *ibid*, in press.
98. T. J. HICKS, B. RAINFORD, J. S. KOUVEL, G. G. LOW, and J. B. COMLY, *ibid* **22** (1969) 531.
99. C. G. ROBBINS, H. CLAUS, and P. A. BECK, *ibid* **22** (1969) 1307.

Corrosion

100. G. TAMMAN, *Z. anorg. Chem.* **107** (1919) 1.
101. D. L. DOUGLASS, G. THOMAS, and W. R. ROSER, *Corrosion* **20** (1964) 15.

Letters

An Electron Microscope Examination of Deformed Polycrystalline Magnesium Oxide

Metallic oxides have a wide potential in many high-temperature applications, and it is therefore important that methods are developed for the microstructural examination of these materials in the deformed state. Transmission electron microscopy has been widely used to observe dislocation configurations in ceramic single crystals; only a few reports have been published on the application of this technique to polycrystals, however, since the production of suitable thin foils is often difficult under such conditions due to substantial porosity and/or weak second phases. Argon-ion bombardment has been employed to thin some polycrystalline ceramics [1, 2] but, whilst this only slightly enlarges any pores, a recent study suggests that it introduces some radiation damage [3].

In the present work, polycrystalline magnesium oxide, available from a previous investigation [4], was produced by hot-pressing MgO powder with 3 wt % LiF for 3 h at 1000° C and annealing for 3 h at 1300° C. The average grain size was 12μm, the density was $> 99.8\%$ theoretical, and the purity was 99.98 % of which the major impurity was 75 ppm Li resulting from the use of LiF as a densifying additive. To permit a study of the dislocation configurations in the deformed state, three specimens were tested in compression at temperatures of 600, 1000 and 1400° C respectively, under a constant force rate of 1.38×10^6 dynes/cm^2 sec. The deformed and fractured specimens were sectioned on a diamond saw into strips of $\sim$ 0.5 mm thickness, and discs of 2.5 mm diameter were cut out ultrasonically.

A flat-bottomed 0.9 mm diameter ultrasonic drill was used to "dish out" the specimens on one side, to a central thickness of ~ 0.25 mm. They were then thinned chemically by immersing in orthophosphoric acid at 150° C and withdrawn at the first sign of perforation. The thinned discs were inserted directly in the electron microscope, thereby avoiding the possibility of introducing mechanical damage when cutting thin pieces from larger strips [5]. The foils were examined in an AEI EM6 electron microscope operating at 100 kV.

The majority of discs prepared in this way showed large areas suitable for transmission around the small central hole. Careful examination revealed no damage in the specimens due to the ultrasonic cutting or "dishing" operations, either in the EM6 electron microscope or in thicker sections of two discs examined in the Cavendish Laboratory high voltage microscope operating at 700 kV.

An example of the grain configuration is shown in fig. 1 for the specimen tested at 600° C. At this temperature, no measurable plastic strain occurred, and the majority of grains were entirely free of dislocations; the few isolated dislocations occasionally observed were probably grown-in. In contrast, in the specimen deformed at 1000° C, which fractured at a plastic strain of ~ 0.001, mobile dislocations were observed in most of the grains, and there were many dislocation loops of the type examined in some detail in deformed MgO single crystals [6, 7].

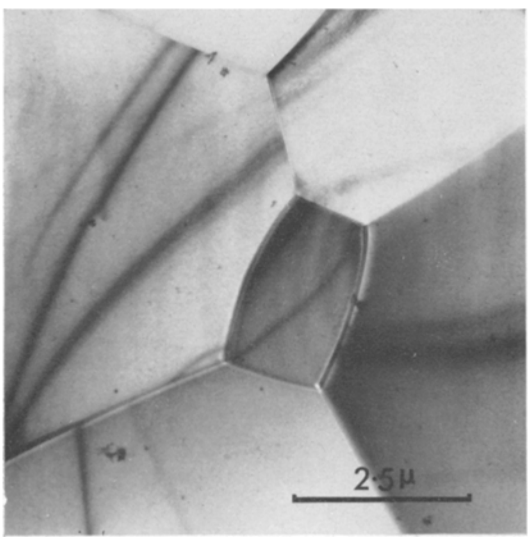


Figure 1 Grain configuration in a specimen tested at 600° C The material is brittle at this temperature, and the majority of grains are entirely free of dislocations.

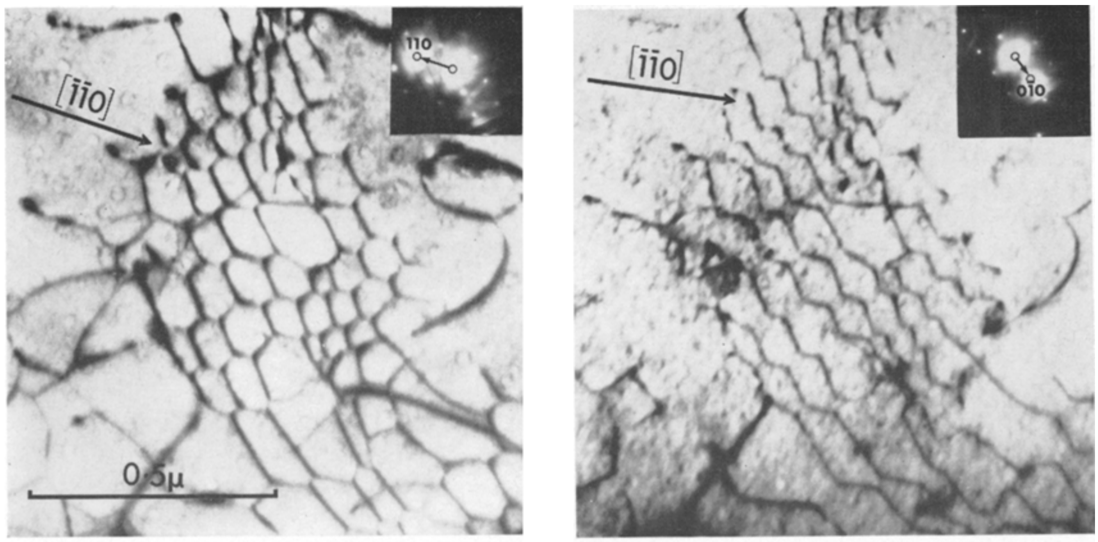
Substantial ductility is possible in polycrystalline MgO at high temperatures, since slip then takes place on both the {110} ⟨110⟩ and {100} ⟨110⟩ systems, thus providing the five independent slip systems necessary for polycrystalline plasticity [8]. Furthermore, tension tests on MgO single crystals have shown that 90° intersections of the {110} ⟨110⟩ slip systems are possible at temperatures of 1300° C and greater [9]. An examination of the specimen tested at 1400° C, which fractured at ~ 0.1 plastic strain, revealed an extensive dislocation structure in all grains, with much evidence of the initial stages of polygonisation and cell formation. It was a general impression from many observations that cell structures were more advanced in grains of particular orientations, but no quantitative measurements were taken to substantiate this because of the uncertainty in relating observed orientations in the plane of the foil to orientations with respect to the loading axis.

A prominent feature in the specimen tested at 1400° C was the widespread occurrence of hexagonal networks, as shown in fig. 2a; these have been reported in MgO single crystals after deformation to high strains (> 0.3) at 1800° C or greater [10, 11] but not apparently at temperatures as low as 1400° C. Stable networks form on (110) planes due to the interaction of the slip vectors of dislocations on oblique (i.e. at 60°) planes, according to the standard reaction:

$$\frac{a}{2}\left[110\right] + \frac{a}{2}\left[\bar{1}01\right] \rightarrow \frac{a}{2}\left[011\right]$$

When viewed in a (001) plane, as in fig. 2a, the hexagons are elongated so that the shortest side is in a ⟨110⟩ direction. Theoretically, the ratio of "height" to "shortest side" is 3, but this is not entirely fulfilled in the example shown here because the network is still in the early stages of formation. The dislocation contrast in fig. 2a is produced by a (110) reflection and the same network is shown in fig. 2b with a $(0\bar{1}0)$ reflection. Using the **g.b** = 0 criterion, where **g** is the reciprocal lattice vector for the operating reflection, the Burgers vector **b** of the extinguished dislocation is $a/2$ $[\bar{1}01]$.

These results show that a chemical technique, which avoids the possibility of introducing mechanical damage, may be used to prepare thin foils of polycrystalline magnesium oxide approximating to theoretical density and it seems likely



(a) (b)

Figure 2 Hexagonal dislocation network in a specimen deformed at 1400° C to 0.1 plastic strain. (a) with **g** = (110) (b) with **g** = ($0\bar{1}0$). (Diffraction patterns corrected for image rotation.)

that a similar method is also applicable to other fully-dense polycrystalline ceramics.

Acknowledgement

We thank Dr D. Tabor, for provision of research facilities, Dr V. E. Cosslett for use of the high voltage electron microscope, and the Ministry of Technology and Science Research Council for financial support.

References

1. M. PAULUS, *Mat. Sci. Res.* **3** (1966) 183.
2. M. PAULUS and F. REVERCHON, *J. Phys. Radium* **22** (S6) (1961) 103A.
3. N. J. TIGHE and A. HYMAN, "Anisotropy in Single-Crystal Refractory Compounds" Vol. 2 (Plenum Press, New York, 1968) p. 121.
4. T. G. LANGDON and J. A. PASK, "Ceramic Microstructures" (John Wiley and Sons, New York, 1968) p. 594.
5. T. G. LANGDON, *Rev. Sci. Instr.* **38** (1967) 125.
6. G. W. GROVES and A. KELLY, *Proc. Roy. Soc.* **275A** (1963) 233.
7. J. WASHBURN and T. CASS, *J. de Physique* **27** Colloque C3 (1966) 168.
8. G. W. GROVES and A. KELLY, *Phil. Mag.* **8** (1963) 877.
9. R. B. DAY and R. J. STOKES, *J. Amer. Ceram. Soc.* **47** (1964) 493.
10. R. J. STOKES and C. H. LI, *Discuss. Faraday Soc.* **38** (1964) 233.
11. R. B. DAY and R. J. STOKES, "Anisotropy in Single-Crystal Refractory Compounds" Vol. 2 (Plenum Press, New York, 1968) p. 267.

14 May 1969

R. H. J. HANNINK
T. G. LANGDON*
Surface Physics, Cavendish Laboratory
University of Cambridge, UK

*Now at: Department of Metallurgy, University of British Columbia, Vancouver, Canada.

A Note on the Fracture of Polyester Resin

Armed with the ideas of fracture mechanics, designers are able to approach with more equanimity than hitherto the problems of designing with brittle and potentially brittle materials. Conventional methods of studying ductile/brittle behaviour – Charpy and Izod tests for example – have always been regarded with some suspicion, and these are being relinquished without regret in favour of tests based on the more formal stress-intensification and crack propagation notions. In comparison with the old impact tests, which are mechanistically more complex, the newer methods are certainly more easily related to physical processes. Charpy tests and the like are notorious for their unreliability and for the fact that the properties measured by them