Letters

Anisotropy and indentation creep in crystals with the rocksalt structure

An earlier review of Knoop hardness measurements, for a very wide range of both non-metallic and metal single crystals, established that the nature of anisotropy was clearly related to the active primary slip systems in a given crystal structure [1]. Most of the results of subsequent work with the Knoop indenter are in accord with those initial observations [2–6]. Indeed, the nature of anisotropy has now been used to study the contribution of plastic flow and to identify the operative slip systems in the deformation of crystals which are predominantly brittle under the experimental conditions which prevail in other methods of measuring mechanical properties. Such studies include measurements on sapphire [5, 7], diamond [8] and the transition metal carbides [3, 4].

There are two models [1, 9], each based on the resolved shear stresses developed on the primary slip systems in the bulk of the crystal beneath the indenter, which are equally successful in explaining and predicting the nature of anisotropy [6]. A number of simplifications are necessarily made in the derivation of both models. For example, they make no attempt to weight the magnitude of the resolved shear stresses on different planes, ignore work-hardening effects, and assume that the contribution of alternative and/or secondary slip systems is negligible. Nevertheless, there is only one category of crystals where the measured anisotropy is at variance with the prediction of the models and within which there appears to be some inconsistency in the nature of anisotropy.

This apparently anomalous group of crystals is based on cubic structures, of the rocksalt type, with $\{1\,1\,0\}\langle 1\,\bar{1}\,0\rangle$ primary slip systems [10, 11]. The purpose of this communication is merely to identify the experimental variables which account for this apparent anomaly – rather than to explain the mechanisms that are thought to be responsible.

Current work has confirmed that indentation creep, i.e. the time dependent penetration of the indenter under a fixed normal load, is observed in the majority of crystalline solids at comparatively

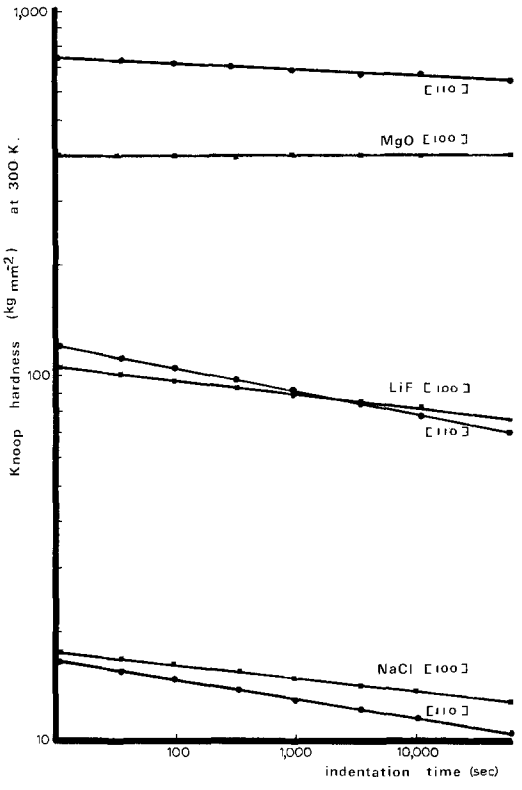

Figure 1 Indentation creep on (0 0 1) planes of MgO, LiF, and NaCl at room temperature – corresponding to 0.150, 0.263 and 0.274 T_m respectively on the homologous temperature scale.

low homologous temperatures [12]. Furthermore, the rate of creep may be markedly anisotropic. For example, log hardness:log time plots, after Atkins *et al.* [13], are shown in Fig. 1 for the (0 0 1) planes of three rocksalt structure crystals. The measurements were made at room temperature and this corresponds to homologous temperatures of 0.274, 0.263 and 0.150 for rocksalt (NaCl), lithium fluoride (LiF) and magnesium oxide (MgO) respectively. In these experiments, the rate of load application was constant but the time of indenter:specimen contact, i.e., the "dwell" time under the full normal load, was varied between 12 and 6×10^4 sec. The anisotropy effect is reflected when the orientation of the long diagonal on the Knoop indenter was made to coincide either with [1 0 0] or [1 1 0] directions. From these results, it is apparent that the [1 1 0]

is harder than the [1 0 0] direction for both LiF and MgO after the standard 12 sec indentation. Conversely, the [1 0 0] is always harder than [1 1 0] for NaCl crystals at this temperature. However the rate of indentation creep is invariably faster in [1 1 0] than in [1 0 0] directions for all these crystals. Consequently, the [1 0 0] yields higher hardness values than the [1 1 0] directions on lithium fluoride after prolonged dwell times > 3000 sec. From the convergence of the two lines, it seems reasonable to anticipate the same effect for MgO after much longer dwell times.

Mechanisms of plastic deformation are most likely to control this process of indentation creep. Hence, these effects and apparent anomalies should be both time and temperature dependent. Therefore, two further sets of experiments were carried out. In the first set, the effect of dwell time on the hardness of MgO was measured at homologous temperatures closer to those of the NaCl when deformed at room temperature. The results are shown in Fig. 2. These confirm a greater rate of creep in [1 1 0] than in [1 0 0] directions and that, at a sufficiently high temperature, the [1 0 0] is harder than [1 1 0] for all indentation

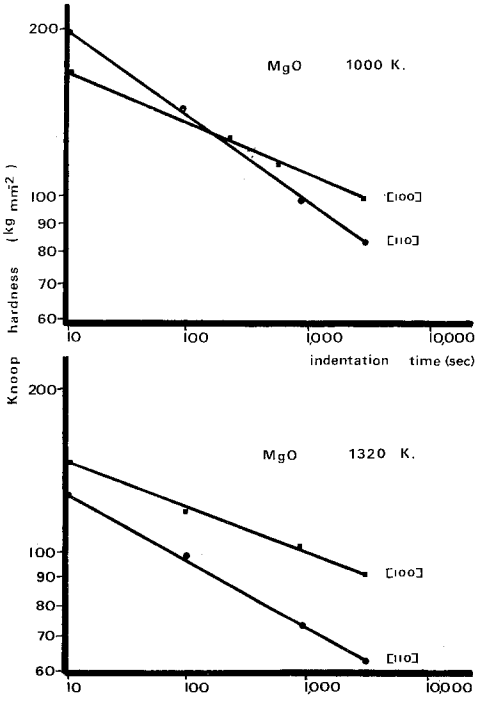

Figure 2 Indentation creep on (0 0 1) plane of MgO at temperatures of 1000 K (0.325 T_m) and 1320 K (0.430 T_m).

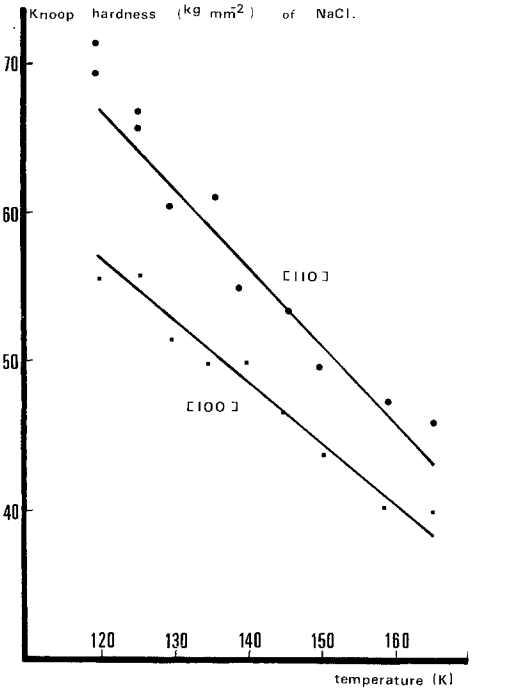

Figure 3 The effect of temperature, in the range 120 to 160 K (0.122 to 0.149 T_m), on the Knoop hardness of (0 0 1) NaCl.

dwell times. In the second set, the standard 12 sec indentation hardness of NaCl was determined at experimental temperatures in the range of 120 to 160 K. The results shown in Fig. 3 now confirm that, under these experimental conditions, the nature of anisotropy for NaCl is typical of the general category of cubic crystals with $\{1\,1\,0\}\langle 1\,\bar{1}\,0\rangle$ slip systems, i.e. [1 1 0] directions are harder than [1 0 0].

These preliminary experiments indicate that it may well be advantageous to consider the indentation process in two parts. First, there is the initial penetration which occurs at comparatively high rates of strain and is accommodated by slip on the primary slip system. Thus, the initial hardness is governed by $\{1\,1\,0\}\langle 1\,\bar{1}\,0\rangle$ slip in rocksalt crystal structure. Secondly, there is the continued (time-dependent) penetration of the indenter that is controlled by creep mechanisms at lower rates of strain. The availability of secondary/alternative slip systems, such as $\{0\,0\,1\}\langle 1\,1\,0\rangle$ in rocksalt crystal structures, might be particularly important in this part. Nevertheless, the predominance of one mechanism over the other would depend on the homologous temperature. Primary slip should be

more important than indentation creep at low homologous temperatures, whilst the converse should hold at higher homologous temperatures. These results indicate that the critical transition temperature is in the region of 0.25 to 0.3 T_m for the standard (12 sec) indentation measurement on rocksalt type crystals.

It is clear that, whatever more detailed analyses are subsequently developed, the apparently anomalous results reported for anisotropy in the hardness of rocksalt crystal structure are due to indentation creep phenomena. Moreover, this work emphasizes the need to control both the indentation time and homologous temperature for accurate comparisons of hardness data in crystalline solids.

References

1. C. A. BROOKES, J. B. O'NEILL and B. A. W. REDFERN, *Proc. Roy. Soc.* **A322** (1971) 73.
2. J. B. O'NEILL, B. A. W. REDFERN and C. A. BROOKES, *J. Mater. Sci.* 8 (1973) 47.
3. D. J. ROWCLIFFE and G. E. HOLLOX, *ibid* **6** (1971) 1303.
4. R. J. HANNINK, D. KOHLSTEDT and M. J. MURRAY, *Proc. Roy. Soc.* **A326** (1972) 409.
5. R. P. BURNAND, Ph.D. Dissertation, Exeter University (1974).
6. B. MOXLEY, Ph.D. Dissertation, Exeter University (1974).
7. B. J. HOCKEY, *J. Amer. Ceram. Soc.* **54** (1971) 223.
8. C. A. BROOKES, *Nature* **228** (1970) 660.
9. J. POSPIECH and J. GRYZIECKI, *Arch. wun Hutnictlia* **XV** (1970) 267.
10. L. S. COMBES, S. S. BALLARD and K. A. MCCARTHY, *J. Opt. Soc. Amer.* **41** (1951) 215.
11. G. Y. CHIN, L. G. VAN UITERT, M. L. GREEN and G. ZYDZIK, *Scripta Met.* **6** (1972) 503.
12. J. E. MORGAN, unpublished work.
13. A. G. ATKINS, A. SILVERIO and D. TABOR, *J. Inst. Met.* 94 (1966) 369.

Received 28 April
and accepted 27 May 1975

C. A. BROOKES
R. P. BURNAND*
J. E. MORGAN
Department of Engineering Science,
University of Exeter,
Exeter, Devon, UK

*Present address: De Beers Diamond Research Laboratory, P.O. Box No. 916, Johannesburg, South Africa.

Complex faults in sodium beta alumina

Sodium beta alumina is a ceramic oxide that is currently under intense investigation for its application as a solid electrolyte membrane in the sodium-sulphur battery [1]. At Cornell we are investigating the relation between microstructure, impurity content, and ionic conductivity in this material. Characterization of the microstructure is necessarily an important part of such a project. In this letter we report on the nature of some planar faults we observed in sodium beta alumina.

Polycrystalline solium beta alumina samples were prepared from commercial powder* by sintering in an argon atmosphere at temperatures between 1700 and 1750° C. The samples were packed in β-alumina to minimize soda loss during sintering. Sintering times were typically 45 min. Transmission electron microscope samples were prepared by ion-milling. The thin foils were observed in a Siemens 101B electron microscope.

Frequently, networks of planar faults can be observed, as shown in Fig. 1. These faults are probably similar to those reported by LeCars *et al.* [2], but these authors did not perform an analysis. The trace analysis showed that the planar faults were contained in the $\{2\bar{1}\bar{1}0\}$ type planes of the hexagonal beta alumina. This corresponds to the $\{1\,1\,0\}$ type planes in the "spinel block" [3] of the crystals. It was found that the faults were never observed when the $[0\,0\,0\,1]$ direction was perpendicular to the electron beam, indicating that the fault displacement vectors have to be contained in the basal plane. Since the faults extend over large distances, they must be of low energy, ruling out displacements that would produce oxygen stacking faults in the "spinel blocks". This is a reasonable observation, since to our knowledge no widely dissociated $\frac{1}{4}\langle 1\,1\,0\rangle$ dislocations have been observed in spinel type crystals, indicating indeed that anion stacking faults with $R = (1/12)\langle 1\,1\,2\rangle$ in spinel type

* Alcoa XB2 "superground".