oration. This work was supported by the University of Trieste.

References

1. V. LONGO and S. ROITTI, *La Ceramica* 29(2) (1976) 9.
2. V. LONGO, F. RICCIARDIELLO and O. SBAIZERO, Paper presented at the 4th CIMTEC, Saint Vincent, Italy, May, 1979.
3. M. DANELON MASTROMONACO, I. BARBARIOL and A. COCCO, *Ann. Chim. (Rome)* 59 (1969) 465.
4. A. COCCO and L. PODDA, *ibid.* 59 (1969) 488.
5. A. COCCO and V. LONGO, *ibid.* 59 (1969) 495.
6. V. LONGO and M. DANELON MASTROMONACO, *ibid* 59 (1969) 502.
7. A. COCCO, V. LONGO and S. ROITTI, *ibid.* 59 (1969) 867.
8. V. LONGO and L. PODDA, *J. Amer. Ceram. Soc.* 61 (1978) 370.
9. V. LONGO, F. RICCIARDIELLO and G. TORRIANO, *La Ceramica* 32(4) (1979) 15.
10. *Idem, ibid.* 32(5) (1979) 1.
11. V. LONGO, S. MERIANI and F. RICCIARDIELLO, Università degli Studi di Trieste, Istituto de Chimica Applicate e Industriale, Pubblicazione No. 85, 1979.
12. V. LONGO, S. MERIANI and F. RICCIARDIELLO, Università degli Studi di Trieste, Istituto di Chimica Applicate e Industriale, Pubblicazione No. 86, 1979.
13. V. LONGO and S. ROITTI, *Ceramurgia* 1 (1971) 4.
14. *Idem, ibid.* 2 (1972) 97.
15. V. LONGO and L. PODDA, *ibid.* 1 (1971) 83.
16. *Idem, ibid.* 1 (1971) 92.
17. *Idem, ibid.* 1 (1971) 11.
18. *Idem, Ann. Chim. (Rome)* 63 (1973) 391.
19. *Idem, La Ceramica* 27(3) (1974) 7.

Received 5 June
and accepted 18 July 1980

VITTORIO LONGO
LILIANA PODDA
Istituto di Chimica Applicata e Industriale,
Facoltá di Ingeneria,
Università di Trieste,
34127 Trieste,
Italy

The influence of CaO-doping on the fracture toughness of hot-pressed Al_2O_3

Only very few investigations on the fracture toughness of hot-pressed alumina have been reported [1, 2]. The influence of impurities was not investigated in these experiments. CaO is a typical impurity frequently found in Al_2O_3. In order to obtain more information on this system Al_2O_3 powders with different CaO-doped levels were hot-pressed. No MgO was added since this dope is not necessary for hot-pressing. The resulting specimens were annealed for various times in order to vary the grain size. The results are discussed and compared with literature data [1–4].

For the experiments a commercial alumina powder, containing no more than 300 ppm impurities was used. The powder was mixed with calcium acetate in ethanol and the mixture was intensively stirred at a temperature of about 80° C. Stirring continued until a reasonably dry powder resulted. The product was dried for 24 h at 120° C. In order to obtain a CaO-dope from the acetate addition, the powder was prefired at 600° C for 4 h in oxygen and afterwards sieved. Continuous hot-pressing [5–7] was carried out at 1400° C, 120 MPa with a feeding rate of 1.25 cm h^{-1}. These being the optimum hot-pressing conditions as described by Peelen [8]. This procedure was carried out for dope levels of 0 to 250 ppm CaO in steps of 50 ppm. The resulting relative densities were all greater than 98.5%.

From the resulting bars, fracture toughness specimens of 1 mm x 3 mm x 15 mm were sawn, in such a way that the fracture planes were perpendicular to the pressing direction. Due to diameter limitations no specimens could be prepared with the fracture planes parallel to the pressing direction. Specimens were annealed at 1550° C, the optimum annealing temperature in the work by Simpson and Merreth [1], for 3, 5 and 8 h in 10^{-5} torr vacuum.* The microstructure of each type of material (characterized by the combination dope level–annealing time) was revealed after polishing by thermal etching at 1450° C at a pressure of 10^{-5} torr for 4 h [9]. Some typical microstructures are given in Figs 1 and 2.

Area distributions of the grains in the scanning

*Astro 1100 V.

0022–2461/81/030841–04$02.40/0

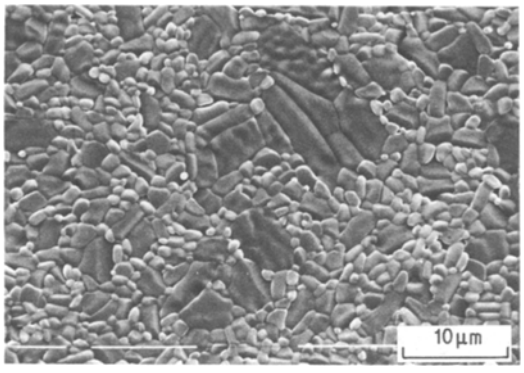

Figure 1 Microstructure of the "as hot-pressed" alumina containing 250 ppm CaO.

electron micrographs were determined with a digital planimeter.† From these data the volume grain size distributions were calculated using the Saltikow–Johnson [10] transformation and fitted to a log-normal distribution. The resulting mean grain sizes (D_A) are shown in Fig. 3.

In each fracture toughness specimen a notch of width $\approx 100\,\mu m$ and depth $\approx 500\,\mu m$ was made. The notched specimens were precracked by a Vickers hardness indentor (1 N load) just below the notch root on both sides of the specimens. The fracture toughness was measured in a three-point bending set-up using a test machine‡ crosshead speed of $4.3\,\mu m\,sec^{-1}$. In order to prevent slow crack growth all experiments were done in a dry N_2 gas atmosphere. The value of the compliance

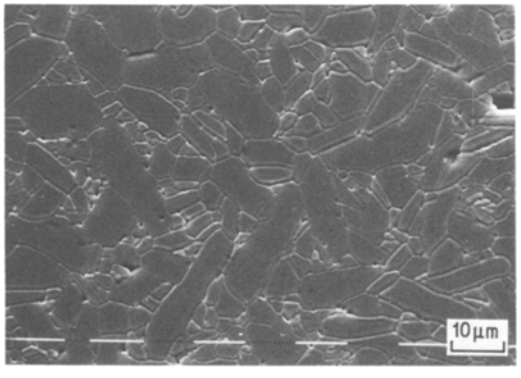

Figure 2 Microstructure of hot-pressed alumina after annealing for 8 h at 1550° C containing no CaO-dope.

†Kontron-MOP-AM-03.
‡Tinius Olsen Electomatic.

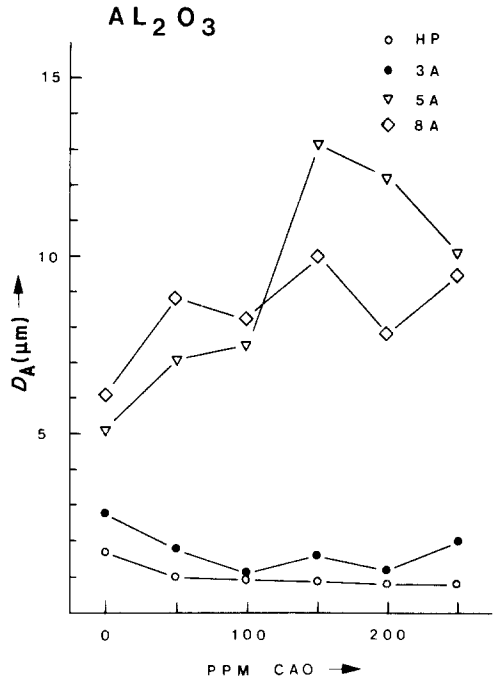

Figure 3 Mean grain size D_A of the various aluminas. HP denotes "as hot-pressed", while 3A, 5A and 8A denote 3, 5 and 8 h annealing at 1550° C respectively.

factor in the calculation of the fracture toughness from the geometric data and the fracture load was taken from the work by Brown and Srawley [11]. The resulting fracture toughness (K_{IC}) values are shown graphically in Fig. 4. For each type of material at least 7 specimens were used. The standard deviation in K_{IC} for each material was 8% on average.

The fracture surfaces were examined with a scanning electron microscope (SEM). Prior to SEM examination, the fracture surfaces were shadowed with gold to eliminate charging effects. Some typical fracture surfaces are given in Figs 5 and 6.

The grain size is an important parameter in fracture mechanics studies of materials. Upon hot-pressing the different powders a fairly constant grain size is obtained (Fig. 3). The shape of the grains is rather elongated (Figs 1 and 2).

After annealing for 3 h at 1550° C the grain size still remains fairly constant. However, after the longer annealing times the grain growth is quite different for materials with different CaO

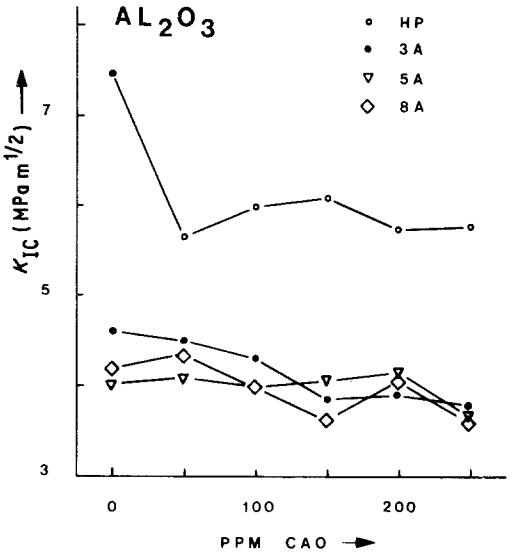

Figure 4 Fracture toughness K_{IC} of the various aluminas. HP denotes "as hot-pressed", while 3A, 5A and 8A denote 3, 5 and 8 h annealing at 1550° C respectively.

levels (Fig. 3). After 8 h annealing the grain size is difficult to quantify because a bimodal distribution seems to occur. The quoted numbers are therefore rather unreliable.

The fracture toughness values K_{IC} of the specimens labelled "as hot-pressed" are high, averaging about 6.1 MPa $m^{1/2}$ (Fig. 4). This high value may be due to compressive stresses resulting from the continuous hot-pressing technique, but the situation is not clear at all. After annealing, the K_{IC} value drops to a fairly constant average of 4.0 MPa $m^{1/2}$ (Fig. 4), which is independent of grain size and CaO content.

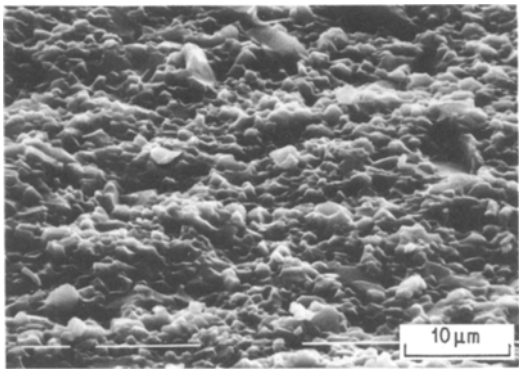

Figure 5 Fracture surface of the "as hot-pressed" alumina containing 250 ppm CaO.

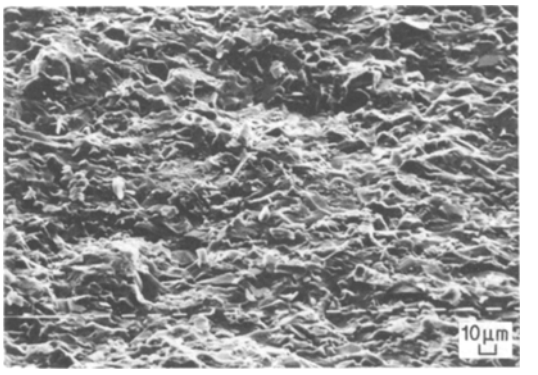

Figure 6 Fracture surface of hot-pressed alumina after annealing for 8 h at 1550° C containing no CaO-dope.

The SEM photographs of the fracture surfaces show a rather irregular picture of intergranular and transgranular fracture for the "as hot-pressed" materials (Fig. 5) while after annealing nearly 100% transgranular fracture occurs (Fig. 6).

In 1974 Simpson and Merreth [1] carried out some fracture toughness measurements on hot-pressed alumina, also not containing any MgO-dopant. The grain size was between 1.5 and 15 μm, depending on annealing time and temperature. For K_{IC} a value of 3.5 to 4.5 MPa $m^{1/2}$ was reported. The fracture mode was mainly intergranular, however. The direction of the fracture plane with respect to the pressing direction was not stated although "uniaxial" die-pressing was used.

Bansal [2] has also reported work on the fracture toughness of hot-pressed alumina. However, his material contained 0.2 wt% "grain growth inhibitor" (MgO?). The grain size was approximately 2 μm. Although double torsion and notched bend tests were carried out, Bansal presented his data in the form of fracture energy γ. For a fracture plane parallel to the pressing direction he reports $\gamma = 42.2\,J\,m^{-2}$. Taking Young's modulus to be $E = 413$ GPa [2] yields a value of 5.9 MPa $m^{1/2}$ for the fracture toughness K_{IC}. The fracture was mainly intergranular and no annealing was carried out. On the other hand for fracture planes perpendicular to the pressing direction he reports a mainly transgranular fracture with a γ of $21.2\,J\,m^{-2}$. This corresponds to a value of 4.2 MPa $m^{1/2}$ for K_{IC}. Thus, our results for transgranular fracture agree nicely with those of Bansal.

The value for K_{IC} is nearly independent of the grain size. This is also the case for sintered Al_2O_3 with MgO-doping, see for instance [12–14].

The fracture toughness is also independent on the CaO content. Ca, however, is a well-known segregant on grain boundaries in ceramic materials [3, 4, 15]. Jupp *et al.* [3] measured a drop in fracture toughness for intergranular fracture in commercial opaque alumina with increasing Ca content in the grain boundaries (as measured by Auger spectroscopy). In the case of transgranular fracture, as we have observed, one would expect no (if only a small) influence of the CaO-dope on K_{IC}. This is confirmed by our experiments.

References

1. L. A. SIMPSON and G. J. MERRETH, *J. Mater. Sci.* 8 (1974) 685.
2. G. K. BANSAL, in "Ceramic Microstructures '75", edited by R. M. Fulrath and J. A. Pask (Westview Press, Boulder, Colorado, 1970) p. 860.
3. R. S. JUPP, D. F. STEIN and D. W. SMITH, *J. Mater. Sci.* **15** (1980) 96.
4. A. W. FUNKENBUSCH and D. W. SMITH, *Metall. Trans.* **6A** (1975) 2259.
5. G. J. OUDEMANS, *Philips Technical Rev.* **29** (1968) 45.
6. *Idem, Proc. Brit. Ceram. Soc.* **12** (1969) 83.
7. R. J. KLEIN-WASSINK, *High Temp.–High Press.* **3** (1971) 411.
8. J. G. J. PEELEN, *Ceramurgia Int.* **5** (1979) 70.
9. G. WILLMAN and G. HEIMKE, *Prakt. Metall.* **15** (1978) 11.
10. E. E. UNDERWOOD, in "Quantitative Microscopy", edited by R. T. Dehoff and F. N. Rhines (McGraw-Hill, New York, 1968) p. 149.
11. W. F. BROWN and S. E. SRAWLEY, *ASTM-STP-410.*
12. J. D. B. VELDKAMP and N. HATTU, *Philips J. Res.* **34** (1979) 1.
13. P. L. PRATT, *Met. Sci.* **16** (1980) 363.
14. R. W. RICE, *Treat. Mater. Sci. Tech.* **11** (1977) 200.
15. M. PAULUS, *Mater. Sci. Res.* 3 (1966) S31.

Received 12 June
and accepted 18 July 1980

G. DE WITH
N. HATTU
Philips Research Laboratories,
Eindhoven,
The Netherlands

Strengthening of an austenitic stainless steel alloy by cryoforming

Cryoforming involves strengthening by transformation during plastic deformation at cryogenic temperatures. Since the process involves deformation at temperatures below ambient temperatures, it is most usefully applied to materials with M_s and M_d temperatures below room temperature. M_s is the temperature at which spontaneous allotropic transformation would occur and M_d is the highest temperature at which allotropic transformation can be induced by plastic deformation.

Several investigators [1–6] have studied the improvement of the mechanical properties produced in various materials by cryoforming. Prominent among the materials known to be favourably affected by cryoforming are the austenitic stainless steels. Deformation at cryogenic temperatures induces martensitic transformation in these materials, leading to improved strength. Foster Wheeler Corporation [7] and Arde Inc. [8] have successfully applied the process to the manufacture of stainless steel pressure vessels. A large proportion of previous investigations into the effects of cryoforming have involved evaluation of room-temperature properties. Since these materials also find application at cryogenic temperatures, in this study, the effects of cryoforming on the cryogenic mechanical properties of an austentic stainless steel alloy were investigated by testing the cryoformed material at a temperature of -196° C.

Qualitative and quantitative correlations have been attempted between the extent of martensitic transformation and the cryogenic yield strength of the cryoformed material and a mechanism of strengthening has been proposed. A low alloy austentic stainless steel was selected for this study since alloys of this grade have been observed to be most favourably affected by cryoforming. The composition and tensile properties of the alloy are shown in Tables I and II, respectively. Magnetic measurements, X-ray diffraction techniques and optical metallography were used to ascertain that

 0022–2461/81/030844–06$02.60/0