Fracture 1977, Volume 3, ICF4, Waterloo, Canada, June 19 - 24, 1977

GRAIN SIZE AND FRACTURE TOUGHNESS OF ALUMINA

P. L. Pratt*

INTRODUCTION

For a number of years the influence of grain size upon the fracture toughness of polycrystalline Al_2O_3 has been unclear, some sources suggesting an increase of toughness with grain size [1], [2], and others a decrease [3], [4]. A number of factors may contribute to this conflict of findings and it is becoming increasingly important to decide which of them are most significant in determining the measured fracture toughness. Different aluminas, or different methods of preparation, might well be expected to give different values of the toughness as a function of grain size, but it was the work of Simpson [5] which first suggested that the type of test used might be an important factor also. For fine-grained Al_2O_3 prepared by cold-pressing and sintering he found comparable results using single edge-notch bend (S.E.N.B.) and double cantilever beam (D.C.B.) test pieces, whereas for a similar coarse-grained Al_2O_3 the D.C.B. results were almost twice those from S.E.N.B. specimens. Recently Simpson, Ritchie and Lloyd [6] have suggested that "the geometry of the D.C.B. specimen, with its large initial crack size and ease of pre-cracking, is more reliable than that of the S.E.N.B. specimen for the purpose of comparing the effect of several microstructures". This conclusion was based on the observation of slow crack growth in coarse-grained S.E.N.B. test pieces that had not been precracked, when held for several minutes at 90% of their fracture load. The suggestion was made that in blunt-notched S.E.N.B. specimens of coarse grain size there is a weakened zone of material at the notch-root, and that during a normal test the crack length could become much longer than the initial value, by spreading through the weakened zone during loading, before fracture of the test-piece. If this were so, the validity of the blunt-notched S.E.N.B. test would be seriously in doubt. Furthermore, despite counter-claims [7], there are those who believe that, even for ceramics, pre-cracking of fracture mechanics test-pieces is essential if the results are to be valid, [8], [9], [10]. The purpose of this paper is to support the need for pre-cracking both D.C.B. and S.E.N.B. test pieces, to demonstrate the value of the S.E.N.B. test and to raise doubts about the validity of the D.C.B. test, especially when it gives results for coarse-grained alumina almost twice those of the S.E.N.B. test.

EXPERIMENTAL METHODS

Details of the characterization of the materials used in this work are given in Dalgleish, Pratt and Sandford [10]. The aluminas were isostatically cold-pressed and fired on commercial schedules, repeated firings being used to obtain the coarser grain sizes. A more substantial programme of work is in hand at the present time on a wider range of both materials and of grain sizes.

*Imperial College, University of London, United Kingdom

S.E.N.B. tests were carried out on specimens blunt-notched with a diamond wheel 0.3 mm thick, and on pre-cracked specimens cut from plates which had been centre-drilled, notched and compressed with the notches parallel to the loading axis. D.C.B. tests were carried out on side-grooved specimens which had been pre-cracked, some being broken instantaneously and others loaded discontinuously to give several toughness values with repeated crack propagation. Double torsion (D.T.) specimens were grooved on one side only and broken instantaneously.

RESULTS AND DISCUSSION

The influence of grain size on fracture toughness, K_{IC}, for the early part of this work, is shown in Figure 1. Mean grain sizes ranged from 4 to 20 µm as in the work of Simpson, Ritchie and Lloyd [6]. However, in contradiction to their results, K_{IC} decreases with increasing grain size for those D.C.B. tests in which the specimen was fractured completely at the first loading, in line with the S.E.N.B. results. The lower line in Figure 1 for S.E.N.B. is for the blunt-notched specimens and the upper line is for pre-cracked specimens. In this case the agreement between pre-cracked D.C.B. and S.E.N.B. is within the limits of experimental scatter for the whole range of grain sizes. An increase of K_{IC} with grain size was found only with those D.C.B. specimens which were loaded so as to give repeated propagation of the crack. For these specimens the loading was stopped as soon as the load-extension curve started to deviate from linearity, and the new length of the crack was measured before loading was continued. These values of the fracture toughness clearly correspond to slow crack growth rather than rapid fracture. At 20 µm grain size the increased value of K_{IC} for repeated propagation corresponds to an increase of about a factor of two in fracture surface energy, γ_i, assuming linear elastic fracture mechanics applies so that $K^2_{IC} \propto G_{IC} \propto 2\gamma_i$. This increase is close to that reported by Simpson [5], and it would be interesting to know if his measurements were based on repeated crack propagation involving slow crack growth. The method of repeated crack propagation is economical in its use of D.C.B. specimens, but could well involve an increased amount of multiple cracking in the coarse-grained material.

While these results do in fact support the findings of Simpson [5] in that the D.C.B. test can give higher toughness values at coarse grain size, nevertheless they contradict the suggestion of Simpson, Ritchie and Lloyd [6] that the D.C.B. test is reliable and that the S.E.N.B. test is suspect. Simpson, Ritchie and Lloyd [6] state that double-torsion (D.T.) tests for their two grain sizes "yielded fracture energies which were in agreement with the D.C.B. data", supporting their hypothesis that the S.E.N.B. data are suspect. On the other hand the D.T. measurements in Figure 1 show little change with grain size and are closer to the D.C.B. results for instantaneous fracture than to those for repeated crack propagation. In view of this direct conflict of experimental evidence it seems dangerous to dismiss the S.E.N.B. tests out of hand. Blunt-notched S.E.N.B. specimens can give lower fracture toughnesses than precracked specimens, as shown in Figure 1, and this suggests that damage in the material near the notch root can be severe. Other workers [8] have found similar low values for blunt-notched S.E.N.B. specimens compared with precracked specimens, and this could perhaps account for part of the difference found by Simpson [5] between S.E.N.B. and D.C.B. data, since he did not precrack his specimens. However, some blunt-notched specimens have shown higher fracture toughness

than precracked specimens of the same material [10], and these presumably were notched more carefully so that pre-cracking served to sharpen the blunt notch, as might be expected. Careful study of the fracture surfaces near the root of the notch in the low toughness material, has shown a region some 10 - 20 µm deep whose appearance differs from that further away from the notch. This might correspond to a region of damage caused by thermal stresses during notching. Certainly these particular specimens were notched rather quickly, compared with those which showed a higher toughness in the blunt-notched state. A similar damaged zone under the root of sawn notches in Si_3N_4 has been reported by Rowcliffe [11].

CONCLUSIONS

These results suggest that both the S.E.N.B. and the D.C.B. tests should be treated with some caution before accepting them as reliable, that under certain conditions D.C.B. and S.E.N.B. test results appear to agree closely over a range of grain sizes, and that controlled precracking of test-pieces is essential for valid fracture toughness measurements. A more careful study of the mechanics of the S.E.N.B., D.C.B. and D.T. tests, and of the mechanisms of fracture of alumina in those tests, is essential before the differing results obtained from them can be properly understood. Such a study on alumina, and on a cubic spinel for comparison, should be completed by the time of the Conference. At this moment it is not clear why the intrinsic fracture surface energy of alumina should double on going from 3 µm grain-size to 20 µm grain-size, as Simpson, Ritchie and Lloyd have suggested [6]. In view of the different stress fields in the neighbourhood of the crack tip in the D.C.B. and S.E.N.B. specimens, it is certainly possible [12] that cleavage and intergranular fracture mechanisms, as well as multiple fracture, may be involved to different degrees in the two tests at different grain sizes. If this is the case, the intrinsic fracture toughness of alumina may vary but little with grain size, and the correct choice of fracture mechanics test for assessing the development of tougher ceramics becomes very important.

REFERENCES

1. GUTSHALL, P. L. and GROSS, D. E., Eng. Fract. Mech., 1, 1969, 463.
2. SWANSON, G. D., J. Am. Ceram. Soc., 55, 1972, 48.
3. SIMPSON, L. A., J. Am. Ceram. Soc., 56, 1973, 7.
4. PERRY, J. R. and DAVIDGE, R. W., Ceramurgia, 3, 1973, 22.
5. SIMPSON, L. A., J. Am. Ceram. Soc., 56, 1973, 610.
6. SIMPSON, L. A., RITCHIE, I. G. and LLOYD, D. J., J. Am. Ceram. Soc., 58, 1975, 537.
7. CLAUSSEN, N., PABST, R. and LAHMANN, C. P., Proc. Brit. Ceram. Soc., 25, 1975, 139.
8. EVANS, A. G., LINZER, M. and RUSSELL, L. R., Materials Science and Engineering, 15, 1974, 253.
9. EVANS, A. G., "Fracture Mechanics of Ceramics", Plenum Press, 1974, 17.
10. DALGLEISH, B. J., PRATT, P. L. and SANDFORD, J., Science of Ceramics, 8, 225, Brit. Ceram. Soc., 1976.
11. ROWCLIFFE, D. J., Private Communication.
12. PRATT, P. L., "Mechanics and Physics of Fracture", Paper 22, Institute of Physics, 1975.

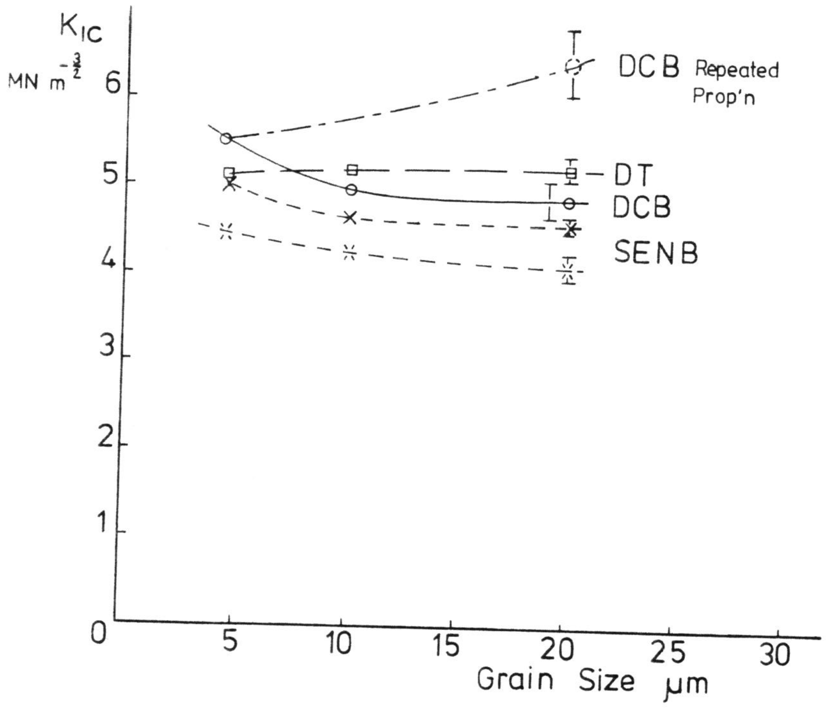

Figure 1 Comparison of the Fracture Toughness K_{IC} Measured as a Function of Grain-Size, for Polycrystalline Al_2O_3 Using the S.E.N.B., D.C.B. and D.T. Tests