Acoustic emission from Al_2O_3–Mo fibre composites

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Acoustic emission has been used to investigate the fracture processs in Al_2O_3 and an Al_2O_3-Mo fibre composite. The acoustic emission data indicate extensive micro-fracturing at loads considerably less than that required to cause fracture in Al_2O_3. The acoustic emission emanating from the composite in the early stages of fracture is very similar to that observed in Al_2O_3. However, as final fracture is approached the acoustic emission profiles differ considerably. The acoustic emission profiles are discussed in terms of the fracture mechanisms.

1. Introduction

Considerable effort has been expended in recent years in attempting to overcome the inherent brittleness of ceramics. One of the main approaches has been the incorporation of a ductile second phase either in the form of particles [1] or as fibres [2-4] into the brittle matrix. In spite of this effort the detailed fracture processes and the major factors controlling fracture toughness are still unclear, even though considerable progress has been made [3, 5]. A technique which appears to have considerable potential for elucidating the fracture process is acoustic emission (A.E.) and this has been used in several recent investigations of fracture [6-8].

Acoustic emission refers to the elastic waves produced as a result of the energy released when a material undergoes deformation and fracture. These waves can be detected at the surface of the material with high sensitivity transducers. For a recent review see the paper by Liptai *et al* [9].

The present investigation considers the acoustic emission resulting from the deformation of polycrystalline Al_2O_3 and Al_2O_3 containing Mo fibres, which significantly increases the fracture toughness of Al_2O_3 [4].

2. Experimental procedure

Three-point bend specimens of dimensions $7.4 \times 6.4 \times 25$ mm^3 with a central notch of 1.9 mm depth, were produced by hot-pressing at 1400° C for 1 h under a pressure of 35 MN m^{-2}. Al_2O_3-Mo fibre composite specimens were made by adding Mo wires of length 3.2 and 0.05 mm diameter and hot-pressing under the same conditions as for the Al_2O_3 specimens. A volume fraction of 0.04 Mo wire was used with all specimens having a final theoretical density $> 98\%$. As a result of hot-pressing the Mo fibres tended to be aligned along the long dimension of the specimen. All specimens were deformed at a displacement rate of 0.005 cm min^{-1}.

Acoustic emissions were detected with a lead-zirconate-titanate transducer having a relatively high frequency band of 100 to 300 kHz, which eliminates the necessity of an acoustically silent testing machine. The transducer was coupled to one end of the bend specimen with a thin film of high-vacuum grease to give good acoustic coupling. Signals from the transducer were recorded on magnetic tape and then analysed by playing the signals back through a rate counter. By setting different trigger levels on the rate counter an indication of the distribution of pulse amplitudes involved at various stages of the test could be obtained. A more detailed discussion of the testing technique employed has been given in a previous publication [10].

3. Results

Before considering the acoustic emission results in detail it is necessary to show that the acoustic activity is emanating from the specimen rather

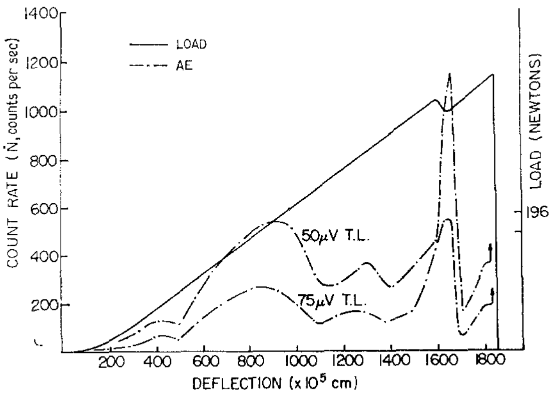

Figure 1 The profile of emission rate (Ṅ) against deflection for unnotched Al_2O_3 at different trigger levels.

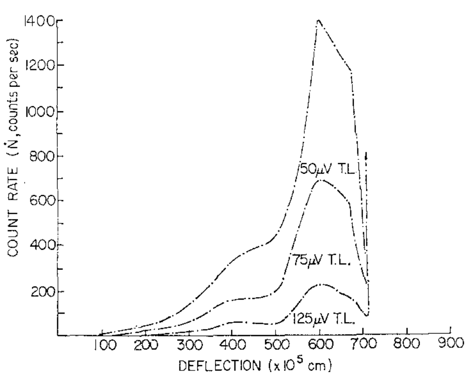

Figure 2 The profile of emission rate (Ṅ) against deflection for a notched Al_2O_3 specimen at different trigger levels.

than the loading device. To check this a specimen was intermittently loaded and unloaded while monitoring the acoustic activity. Kaiser [11] showed that acoustic emission activity is irreversible in the sense that acoustic emissions are not generated during the reloading of a material until the stress level exceeds its previous maximum level. This was the case for the present experiments and, hence, it is reasonable to conclude that the acoustic emissions are emanating from the specimen being strained.

3.1. Acoustic emission from Al_2O_3

Fig. 1 shows the typical profile of emission rate against deflection for an unnotched Al_2O_3 specimen. It can be seen that considerable acoustic activity begins early in the test and is significant from about 0.2 σ_F where σ_F is the fracture load. By "significant" we mean that even if noise from the loading machine and background noise from the electronics are assumed to be contributing to the measured emissions the detected activity is much above this background effect. The profile shows a peak in activity at a deflection of about 800×10^{-5} cm which was quite reproducible from specimen to specimen although the peak sometimes took the form of a plateau. A second peak in activity occurs just prior to final fracture, the height of this second peak being considerably larger ($\sim \times 2$) than the first peak. Final fracture was accompanied by extremely high activity, often beyond the range of the ratemeter used, together with an audible noise.

In the case of a notched Al_2O_3 specimen the rate profile and the total counts against deflection is as shown in Figs. 2 and 3. It is apparent

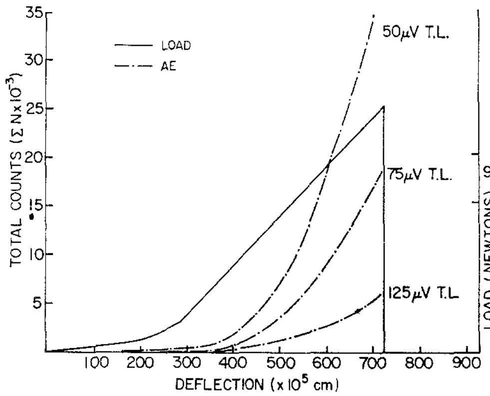

Figure 3 The total number of counts (ΣN) against deflection for a notched Al_2O_3 specimen.

that the profile is very similar to the unnotched specimen but the total deflection involved and hence total number of counts is less.

3.2. Acoustic emission from Al_2O_3-Mo fibre composites

The count rate profile of a notched composite specimen containing Mo fibres is shown in Fig. 4 and its total number of counts with deflection together with the load-deflection plot in Fig. 5. Considerable acoustic activity is observed at small deflections and continues throughout the test, as in the case of Al_2O_3. However, in sharp contrast to the A.E. behaviour of Al_2O_3, the composite shows a considerable period (from a deflection of 1200×10^{-5} to about 1450×10^{-5} in Fig. 4) of very low activity. This behaviour was a characteristic of all the composite samples tested.

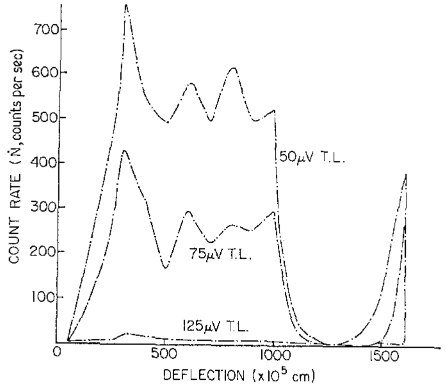

Figure 4 The emission rate against deflection for a notched Al_2O_3-Mo fibre composite.

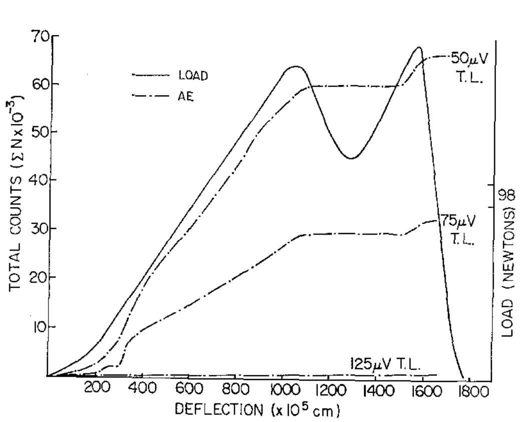

Figure 5 The total number of counts against deflection and the load-deflection plot for notched Al_2O_3-Mo fibre composite.

4. Discussion

4.1. Acoustic emission and fracture processes

The results clearly show that release of elastic strain energy is occurring at loads far below the fracture stress, both in the Al_2O_3 and in the composite. Considering the Al_2O_3, the release of elastic energy could result from several causes, but in the present case only three sources are probable, and these are dislocation motion, twinning and crack phenomena. While plastic flow and twinning have been observed in Al_2O_3 at high temperature they have not been detected at temperatures as low as room temperature. The acoustic emission results however, indicate very extensive activity at room temperature which is incompatible with the plastic flow behaviour of Al_2O_3. In addition, the amplitude of the events are extremely large in comparison with those exhibited by dislocation motion in metals. This leaves "crack phenomena" as the most probable cause of emissions.

The acoustic emission results therefore indicate considerable sub-critical crack growth prior to final fracture and increasing significantly just prior to macroscopic fracture. A crack only propagates after satisfying the energy condition

$$-\frac{dU}{dc} = \frac{dW}{dc} \tag{1}$$

where U and W are the stored elastic energy and the energy dissipated irreversibly, respectively, and $2c$ is the crack length. For propagation of an elastic crack, where the only dissipative force is the energy of new surfaces, Equation 1 reduces to the Griffith equation

$$\sigma = \left(\frac{2E\gamma}{\pi c}\right)^{1/2} \tag{2}$$

for an elliptical crack normal to a tensile stress σ. Satisfying Equation 2 immediately leads to instability, but as pointed out by Glucklich [12] satisfying Equation 1 does not *a priori* result in instability. For example, the Griffith criteria assumes that dW/dc is a constant, while it may, in fact, increase with c to create the inequality

$$-\frac{dU}{dc} < \frac{dW}{dc}\,. \tag{3}$$

This condition would be satisfied if crack blunting resulting from plastic flow occurred. In the case of Al_2O_3 it is unlikely that sufficient plastic flow occurs to achieve this. Limited plastic flow, if any, is supported by the fact that fracture toughness test on Al_2O_3 indicate a surface energy of about 3×10^4 erg cm^{-2}, which is only a factor of ten higher than the theoretical value. In metals, on the other hand, where extensive plastic flow occurs, theoretical and experimental values often differ by 10^5 times. It should be noted that acoustic emission itself is an energy dissipation mechanism which would contribute to the apparent surface energy. An alternative mechanism for producing stability is for dU/dc to decrease with increasing crack length rather than increasing as in the Griffith theory. This can be achieved by the crack propagating from a high tensile stress region into one of lower stress such as might occur in a matrix containing varying residual stresses. Since the specimens are produced by hot-pressing, non-uniform residual stresses are a distinct possibility. A similar suggestion has been made by Scholz [13] in reference to the fracture of rocks, which also show extensive acoustic activity at stresses far less than the fracture stress. Fracture of extremely brittle particles in the Al_2O_3 matrix could also achieve this effect and give rise to the A.E. observed.

The A.E. results indicate, however, that sub-critical stable crack growth occurs quite extensively prior to fracture in both the Al_2O_3 and the composite. This activity increases significantly as the fracture stress is approached. This would be expected, since as the stress is increased one is getting closer and closer to the instability criteria which is satisfied at the fracture stress. The similarities between the acoustic emission profiles obtained from Al_2O_3 and Al_2O_3-Mo composite at comparable deflections suggests that the fracture processes are essentially similar in the early stages. If this is the case, one would expect

$$\frac{\Sigma N}{D} \simeq \text{constant}$$

where ΣN is the total number of counts, D is the deflection, for both Al_2O_3 and Al_2O_3-Mo fibres. Considering the notched Al_2O_3 data in Fig. 3 we obtain for a 50 μV trigger level

$$\frac{\Sigma N}{D} = \frac{35 \times 10^3}{700 \times 10^{-5}}$$

$$= 5 \times 10^6 \text{ counts per cm deflection.}$$

For the composite in Fig. 5 we obtain,

$$\frac{\Sigma N}{D} = \frac{60 \times 10^3}{1200 \times 10^{-5}}$$

$$= 5 \times 10^6 \text{ counts per cm deflection.}$$

Of course this exact agreement is fortuitous, however the similarity in the A.E. behaviour is clearly established. Notice that we have only considered the A.E. behaviour in the composite up to the period of very low activity prior to final fracture. The later stages of fracture in the composite differ significantly from the Al_2O_3 as will be shown in the next section.

4.2. Fracture process

The fracture process in Al_2O_3 is the normal one from brittle materials. Although sub-critical stable crack growth does occur prior to the final fracture as shown by its A.E. behaviour, the instability condition, i.e. $-(\mathrm{d}U/\mathrm{d}c) > (\mathrm{d}W/\mathrm{d}c)$ is reached very rapidly.

In the case of the Al_2O_3-Mo composite we have a similar situation in the initial stages of fracture. Crack initiation occurs at the notch and propagates through the Al_2O_3. In all specimens tested this initial crack immediately extended through at least 80% of the thickness of the specimen and in several cases through over 90% of the thickness. There is a tendency for crack branching and deflection at fibres as shown in Fig. 6, where the white areas are the fibres. The crack by-passes the fibres by a process of occlusion, however, the specimen does not split in two since the fibres cross the crack, linking the matrix on either side of the crack. This crack process is in agreement with the acoustic emission results in that the acoustic profiles are essentially the same in Al_2O_3 and in the composite, which is expected since only fracture of Al_2O_3 is occurring in this initial period.

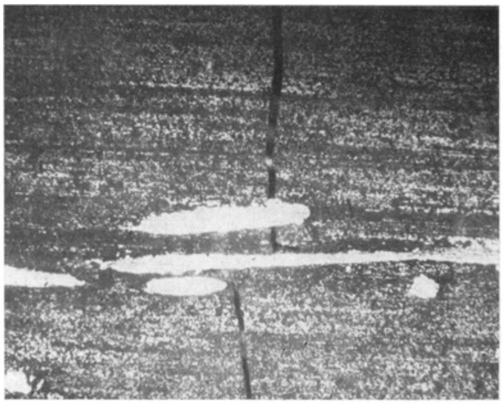

Figure 6 Crack branching and deflection at fibres.

In previous work on Al_2O_3-Mo composites, Tinklepaugh [2] detected microcracking in the composite immediately after hot-pressing, attributing this to differential thermal expansion between Al_2O_3 and Mo. In the present specimens no initial microcracks could be detected, either metallographically or with dye penetrant and the initial acoustic emission activity in terms of rates, amplitudes, etc, were very similar in Al_2O_3 and Al_2O_3-Mo. Hence, initial microcracks, if present, do not appear to be particularly influential in the present case.

Further deflection of the specimen after the initial crack has been formed, results in opening up of the crack and the gradual pulling out of the fibres. The acoustic activity during this stage of the fracture process increases again and with the means of analysis available during the present study, is very similar to the activity observed prior to initial crack propagation. This suggests that the fracture mechanisms are the same in both cases, i.e. pull out of the fibres involves brittle fracture of the Al_2O_3 matrix in the neighbourhood of the fibres. It should be noted, however, that some additional macroscopic cracking of the Al_2O_3 matrix also occurs during this period and this is accompanied by extremely large bursts of activity.

In the very last stages of fracture, necking down of the fibres occurs and final fracture usually corresponds to ductile failure of the Mo fibres, as was also observed by Simpson and Wasylyshyn [4] in scanning microscopy photographs. This necking down of Mo fibres corresponds with a very low acoustic emission activity which supports the initial contention that the early acoustic emission activity is associated with the failure of Al_2O_3.

No detailed measurements of fracture toughness were made as these have already been reported [4], but an increase in fracture toughness was observed in the composite. This appears to be due to the extra energy necessary for crack branching, fibre pull-out and necking down of the fibres to produce final fracture.

5. Conclusions

Acoustic emission has been used to follow the fracture process in Al_2O_3 and Al_2O_3-Mo fibre composites. The acoustic emission indicates extensive microcracking prior to final failure in both Al_2O_3 and the composite. In the composite the acoustic emission indicates that fibre pull-out is accompanied by microcracking of the Al_2O_3 matrix, similar to that observed prior to initial macroscopic cracking.

Acknowledgements

The authors wish to thank the National Research Council of Canada for financing this work, Dr L. A. Simpson of Canadian Atomic Energy Limited, Pinawa, for providing the samples used, and Mr D. Mardis for assistance with some of the testing.

References

1. P. HING and G. W. GROVES, *J. Mater. Sci.* **7** (1972) 422.
2. J. R. TINKLEPAUGH, "Strengthening Mechanisms – Proceedings of the 12th Army Materials Research Conference" (Syracuse University Press, Syracuse, New York, 1966) p. 579.
3. P. HING and G. W. GROVES, *J. Mater. Sci.* **7** (1972) 427.
4. L. A. SIMPSON and A. WASYLYSHYN, *J. Amer. Ceram. Soc.* **54** (1971) 56.
5. D. C. PHILLIPS and A. S. TETELMAN, *Composites* **3** (1972) 216.
6. H. L. DUNEGAN, D. O. HARRIS and C. A. TATRO, *Eng. Fract. Mech.* **1** (1968) 105.
7. J. C. RADON and A. A. POLLOCK, *ibid*, **4** (1972) 295.
8. J. FITZ-RANDOLPH, D. C. PHILLIPS, P. W. R. BEAUMONT and A. S. TETELMAN, *J. Mater. Sci.* **7** (1972) 289.
9. R. B. LIPTAI, D. O. HARRIS and C. A. TATRO, "Acoustic Emission" (ASTM publication, 1971) p. 3.
10. K. TANGRI, J. P. TORONCHUK and D. J. LLOYD, *Trans. Indian Inst. Met.* **25** (1972) 33.
11. J. KAISER, *Arch. Eisenhüttenw.* **25** (1953) 43.
12. J. GLUCKLICH, *Eng. Fract. Mech.* **3** (1971) 333.
13. C. H. SCHOLZ, *J. Geophys. Res.* **73** (1968) 1417.

Received 30 July and accepted 21 September 1973.