The Acoustic Response of  ${\rm Al}_2{\rm O}_3$ ,  ${\rm Al}_2{\rm O}_3$ .  ${\rm ZrO}_2$  and Reconstituted Brick in Three-Point Bending

A. Fakhr, J.E. Freyre and Rees D. Rawlings Imperial College, London, SW7 2BP

### ABSTRACT

The acoustic response of three ceramics with widely differing microstructures has been monitored during three-point bending fracture toughness testing. The ceramics were single phase  ${\rm Al}_2{\rm O}_3$ , two phase  ${\rm Al}_2{\rm O}_3$ . ${\rm ZrO}_2$  and coarse multiphase reconstituted brick. The acoustic responses of the three ceramics varied greatly and have been correlated with the microstructures. There was no simple relationship between the common acoustic emission and fracture toughness parameters.

### INTRODUCTION

It is surprising that the acoustic response of ceramics, which are generally acoustically noisier than metals, has received so little attention. Some excellent papers (e.g. 1) have been published on single phase ceramics but there is a lack of information on ceramics with more complex microstructures. This paper reports the results of a study of three ceramics (alumina, alumina with ZrO<sub>2</sub> particles and reconstituted bricks) which have markedly different microstructures. The Al<sub>2</sub>O<sub>3</sub>, which was produced by isostatically cold pressing followed by firing, had a mean grain size of 10 $\mu$ m. The Al<sub>2</sub>O<sub>3</sub>.ZrO<sub>2</sub>\* was hot pressed and consisted of 15% of fine ( $1.5\mu$ m) ZrO<sub>2</sub> particles in Al<sub>2</sub>O<sub>3</sub> of mean grain size of 2 $\mu$ m. The reconstituted bricks were manufactured from a predominately limestone aggregate (the particles of which varied in size up to 5 mm diameter) and 10% Portland cement by cold pressing and steam curing.

## EXPERIMENTAL PROCEDURE

The acoustic emission studies were performed on single edge-notch specimens fractured in three-point bending. The specimen dimensions were 72 x 15 x 3 mm (reconstituted bricks) and 30 x 8 x 2.7 mm (Al $_2$ O $_3$  and Al $_2$ O $_3$ . Five specimens of each ceramic were tested. Standard Dunegan-Endevco equipment with a distribution module, was used.

#### RESULTS AND DISCUSSION

Typical load/deflection and ring-down count/deflection curves for the three ceramics are given in Figure 1. For the  ${\rm Al}_2{\rm O}_3$  and  ${\rm Al}_2{\rm O}_3$ .  ${\rm ZrO}_2$  the emissions commenced just prior to failure (at  ${\rm \sim}0.9{\rm o}_{\rm F}$ ) and the emission rate increased with load reaching a maximum at the fracture load  ${\rm o}_{\rm F}$ . The emissions prior to  ${\rm o}_{\rm F}$  are considered to be associated with sub-critical crack growth. The count/deflection curve for  ${\rm Al}_2{\rm O}_3$ .  ${\rm ZrO}_2$  was stepped indicating that the sub-critical crack growth was more discontinuous than in the case of  ${\rm Al}_2{\rm O}_3$ .

The emissions prior to failure in the brick commenced at loads as low as 0.1  $\sigma_{\vec{F}}$ ; this is attributed to the more friable nature of the brick. Furthermore, \*Kindly supplied by Dr. N. Claussen, Max-Planck-Institut fur Metallforschung.

The Acoustic Response of  ${\rm Al}_2{\rm O}_3$ ,  ${\rm Al}_2{\rm O}_3$ .  ${\rm ZrO}_2$  and Reconstituted Brick in Three-Point Bending

the results for the brick were not so consistent as those for the other ceramics e.g. the count-rate sometimes reached a maximum before  $\sigma_{\rm F}$  was reached and the total number of counts to failure showed considerable scatter. The more variable and coarser microstructure of the brick, particularly when it is borne in mind that the fracture path is only 22.5 mm², accounts for the relative lack of reproducibility in the data.

In fracture toughness tests on steel (e.g. 2,3) and in the first stage of stress corrosion cracking of porcelain in water (4) the ring-down count-rate and the stress intensity factor k are related by  $\frac{dN}{dt}$   $\alpha k^{m}$ .

As shown by Figure 2, this relationship was obeyed for  $Al_2O_3$  and  $Al_2O_3$ . ZrO, with exponent m values of 18 and 16 respectively. These values are higher than those obtained in steels (m $\sim$ 5) but less than that for porcelain in water (m = 30). Although there appears to be a linear relationship between log (dN) and log(k)

for brick, the scatter in the data was considered too great to warrant quoting a value for the exponent m.

The fracture toughness parameters  $K_{LC}$  (critical stress intensity factor) and  $G_{LC}$  (critical elastic strain energy release rate) were determined from the fracture load and are presented in Table 1. It is clear from Table 1 that there is no simple correlation between these parameters and the acoustic response as characterised by the mean value of i) the number of ring-down counts to failure per unit fracture area  $(N_{LC})$ , ii) the number of events to failure per unit fracture area  $(N_{LC})$  and iii) the maximum ring-down count Tate per unit fracture area  $((M_{LC}))$ .

max

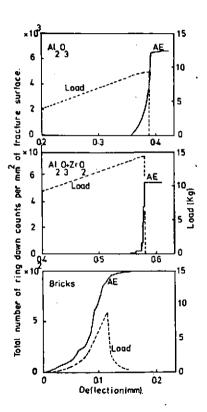


Figure 1. The acoustic response of the three ceramics in three-point bending.

Table 1. Fracture Toughness and Acoustic Emission Parameters

Material	k <sub>1C</sub> (MNm <sup>-3/2</sup> )	Approximate G <sub>1C</sub> (kJm <sup>-2</sup> )	N <sub>T</sub>	N <sub>E</sub>	$(\frac{dN}{dt})_{max}$
A1 <sub>2</sub> 0 <sub>3</sub>	3.93±0.16	38.6	6800	117	15 x 10 <sup>3</sup>
A1707.ZrO,	5.82±0.55	84.7	52	2.2	27
Al <sup>2</sup> O <sub>3</sub> .ZrO <sub>2</sub> Brick	0.39±0.05	8.1	881	4.7	3.8

The Acoustic Response of Al<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>.ZrO<sub>2</sub> and Reconstituted Brick in Three-Point Bending

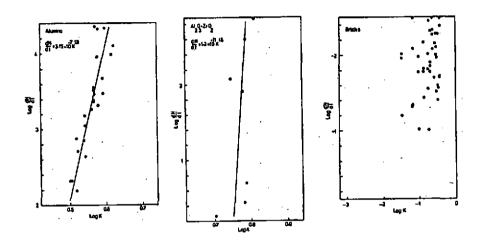


Figure 2. Plots of the log (count rate) against log (stress intensity factor).

Amplitude distribution curves for the three ceramics are given in Figure 3. All distributions conformed to the usual relationship

$$n_{(A)} = (\frac{a}{a_0})^{-1}$$

where  $n_{(A)}$  is the fraction of events whose amplitude exceeds a, a is the lowest detectable amplitude and b is a constant. The brick had a large number of low amplitude events and the maximum amplitude was only 55 dB, consequently the bvalue was high, at 1.75 (see Table 2). This acoustic response is as expected from a poorly bonded, friable material. The Alog had a similar high frequency of low amplitude events but also a greater number of high amplitude events. Hence b was reduced to 0.88, which is of similar magnitude to the values previously obtained for alumina and other single phase ceramics (alumina, 0.8; magnesia, 0.6; spinel, 0.6 (1)). In contrast, the b-value for Al, 0, .Zr0, was only 0.37 and the amplitude distribution was characterised by a small number of events, a significant fraction of which were of high amplitude. The small number of events and the high proportion of high amplitude events are consistent with the stepped ring-down count/deflection curve (Fig. 1). Claussen (5) has attributed the improved fracture toughness of Al<sub>2</sub>O<sub>3</sub>.ZrO<sub>2</sub> compared with Al<sub>2</sub>O<sub>3</sub> to the opening of microcracks in front of the notch tip due to the presence of the ZrO<sub>2</sub> This process should occur frequently and lead to many low amplitude particles. The lack of low amplitude events in the distribution suggests that most of these are below the detection limit of the system. However, the propagation of the main crack should be markedly discontinuous giving a small number of Thus, the emissions from the main crack account events with many of high energy. for the observed amplitude distribution.

The Acoustic Response of  ${\rm Al}_2{\rm O}_3$ ,  ${\rm Al}_2{\rm O}_3$ .  ${\rm ZrO}_2$  and Reconstituted Brick in Three-Point Bending

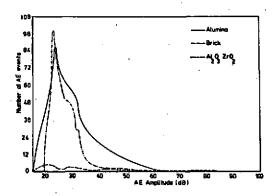


Figure 3. Comparison of the amplitude distributions.

Table 2. Amplitude Distribution Data (A<sub>M</sub> = maximum amplitude, A<sub>F</sub> = most frequent amplitude)

	,			
Material	N <sub>E</sub>	Ъ	A <sub>M</sub> (db)	A <sub>F</sub> (db)
A1,0,	117	0.88	80	24
$A1_{2}^{2}0_{3}^{3}.Z_{7}0_{3}$	2.2	0.37	85	20
A1 <sub>2</sub> 0 <sub>3</sub> .Zr0 <sub>2</sub> Brick	. 4.7	1,75	55	23

## CONCLUSIONS

- 1) Acoustic emissions, associated with sub-critical crack growth, were detected in all ceramics at loads less than the fracture load. These emissions occurred at particularly low loads in the more friable reconstituted brick.
- 2) For Al $_2$ O $_3$  and Al $_2$ O $_3$ .ZrO $_2$  the ring-down count rate was proportional to the stress intensity factor to the power of 18 and 16 respectively.
- 3) The amplitude distributions for the three ceramics conformed to the equation  $n_{(A)} = (\frac{a}{a_0})$ . The b-values so obtained were correlated with the microstructural sensitive fracture processes in the ceramics.

#### REFERENCES

(1) A.G. Evans and L.J. Graham, Acta Met. (1975) 23 1303; (2) H. Dunegan and A.S. Tetelman, Engineering Fracture Mechanics (1971) 2 387; (3) H. Dunegan, Met. Eng. Quart. (1975) Feb. 8; (4) A.G. Evans and M. Linzer, J. Amer. Ceram. Soc. (1973) 56 575; (5) N. Claussen, J. Amer. Ceram. Soc. (1976) 59 49.

STABILITY OF COMPOSITE MATERIALS

A.R. Bunsell, D. Laroche

Ecole des Mines de Paris, B.P. 87, 91003 Evry Cedex (France)

Little attention seems to have been given to the long term behaviour of high modulus composite materials. This is surprising as these materials are being used increasingly in primary load supporting roles where failure could have serious consequences. A composite material by definition consist of at least two dissimilar materials which respond to loadings differently. Repeated loadings or a constant load may lead to eventual failure after a considerable length of time if plastic deformation or relaxation of the matrix leads to an increase in stress on the fibres. In this paper studies which are still continuing will be described in which the monitoring of acoustic emission is used to determine whether progressive damage is occurring in c f r p or if it has ceased.

Carbon fibre reinforced epoxy resin consists of elastic fibres in an anelastic and viscoelastic matrix. The emissions recorded from the materials under load have been shown to be definite indications of internal damage and most probably of fibre failure (1). In this study three types of loading conditions are being considered and the same form of specimen shape is used throughout except when the results have been applied to more complicated structures. The specimens consist of either high modulus or high strength carbon fibres aligned unidirectionally in an epoxy resin. The forces experienced by the material in all of the tests are in a direction parallel to the fibre alignment direction. The tests involve either a constant applied load, a constant deformation or a cyclic loading to a constant maximum level.

With a constant deformation imposed on the cfr parelaxation is found to occur and the load on the material falls. The load trace appears to tend asymptotically with time to a constant load level indicating that after the initial deformation there is a limited period of readjustment in the material. There have been examples of specimens breaking during this relaxation period when the load supported was below the maximum load experienced. The rate of acoustic emission recorded during this period is found to miror the rate of relaxation. Most emissions are recorded immediately after the maximum deformation is attained and the rate of emission falls with time until, after typically ten minutes the emission ceases.

If the load is maintained constant as in a creep test a rapid increase in emission occurs on loading and in the period immediately following loading. The rate of emissions quickly and steadily decreasehowever and tends towards zero although occasional emissions may still be recorded for several days if the load is sufficiently high. Those specimens which have been found to fail during creep loading have in every case still been emitting and had therefore not completely stabilised.

#### STABILITY OF COMPOSITE MATERIALS

During cyclic loading the "Kaiser effect" is not seen to be completely obeyed and on reloading emission recommences just before the previous maximum load is attained. Subsequent cycling to the same load level indicates that the load at which emission recommences moves closer to the maximum load, as cycling progresses, and finally ceases. Failure of specimens subjected to cyclic loading have again only been obtained whilst emissions were still being recorded.

These type of tests have also been conducted on filament wound ring specimens and pressure vessels with similar results being obtained. A tentative acoustic emission proof testing technique has been proposed for these type of structure (2) based on the earliest work with the plane specimens.

The physical mechanisms involved in the stabilisation seem to concern the anelastic and viscoelastic properties of the matrix combined with the distribution of fibre properties to be found in any fibre bundle. We are at present developing a mathematical model to attempt to explain the observed behaviour. This if it succeeds should give us a better understanding of the long term behaviour and stabilisation of c f r p .

#### REFERENCES

- Fuwa M., Bunsell A.R., Harris B., J. Phys. D. 9 (1976) 353.
  An evaluation of acoustic emission techniques applied to carbon-fibre composites.
- Fuwa M., Bunsell A.R., Harris B., J. Strain Analysis 11 (1976) 97.
  Acoustic emission studies of filament wound carbon fibre reinforced rings and pressure vessels.