FRACTURE TOUGHNESS AND WORK OF FRACTURE OF SIC-FIBRE GLASS MATRIX COMPOSITE AGED IN NON-OXIDISING ATMOSPHERE

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The applicability of the chevron-notch technique for changes monitoring in fracture behaviour of fibre/glass matrix composites has been investigated. A commercial SiC-fibre reinforced glass matrix composite was aged in argon at temperatures in the range 500-700 °C for duration of up to 1000 hrs. The mechanical properties of aged samples were evaluated at room temperature by using four-point flexure strength and three-point flexure chevron notch techniques. For identification of the unstable fracture onset an acoustic emission technique was applied. The fracture toughness values in the range 19-26 MPam$^{1/2}$ were little affected by the ageing conditions except for the most severe ageing conditions. The procedure for the work of fracture determination and its dependence on ageing condition is presented.

INTRODUCTION

In ceramics reinforced by brittle fibres, an elastic fibre bridging and a pull-out bridging mainly cause the toughening. Both these mechanisms increase in some extent behind the crack tip along the process zone wake (1-3). The crack growth resistance rises as the crack propagates and leaves the wake. It is difficult to define the intrinsic fracture toughness as a material parameter due to the increasing crack growth resistance curve (2). The work of fracture (WOF) may be a more practical toughness parameter that characterises toughening mechanisms. When the WOF is determined using the chevron notched (CN) specimen technique, it depends on notch geometry. However, it is possible to obtain WOF values in a relative way, i.e. when the same size and geometry of the unnotched ligament is adopted in the specimens. The CN bend specimens thus have some advantages for estimating the fracture resistance of toughened materials, since it is possible to measure simultaneously both the fracture toughness and the WOF from running crack (4,5).

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A key requisite for the glass composites to be useful at high temperatures is the retention of a weak fibre/matrix interface, which is responsible for their flaw-tolerant behaviour (1,2). In silicate matrix containing silicon oxycarbide fibres (Nicalon) (7), the weak interface is provided by a carbon-rich layer around the fibres (8). This interlayer can be degraded when the material is used in oxidising environments at temperatures as low as 400 °C (7,9). In temperature range 500-700 °C, a loss of the "pseudo-ductile" composite behaviour is observed with reduced fibre pull-out effects (9,10). Although considerable research has been conducted to the behaviour of silicate matrix composites in air, much less effort has been paid to the thermal stability of these composites in inert atmospheres.

The purpose of the paper is the verification of the suitability of the CN technique for changes monitoring in the fracture behaviour of fibre/glass matrix composites that may occur following ageing in non-oxidising atmosphere. The application of test techniques and assessment procedures for detection of the microstructural integrity and mechanical property degradation due to ageing treatment are also addressed.

MATERIAL AND EXPERIMENTAL PROCEDURES

Material characterisation

The material investigated was a commercially available unidirectional SiC Nicalon fibre reinforced borosilicate (DURAN) glass matrix composite fabricated by Schott Glaswerke (Mainz, Germany). Information on the composite constituents is given in Table 1. The composites were prepared by the sol-gel-slurry method (11). The samples were received in the form of rectangular test bars of nominal dimensions (4.5x3.8x100 mm^3). The density of the composites was 2.4 g/cm^3 and their fibre volume fraction 0.4.

Tab. 1: Properties of the composite constituents (11)

	Density [g/cm^3]	Young's modulus [GPa]	Poisson's ratio	Thermal expansion coefficient [K^{-1}]	Tensile strength [MPa]
matrix DURAN	2.23	63	0.22	$3.25 \cdot 10^{-6}$	60
fibre SiC Nicalon	2.55	198	0.2	$3 \cdot 10^{-6}$	2750

Thermal ageing involved encapsulating the as-received bars in silica tubes after these had been evacuated and filled with argon. The capsules were aged in a furnace at temperature 500, 600 and 700 °C for 100 hrs. For the 600°C temperature, ageing experiments of 250 and 1000 hs were conducted also. The mass and dimensions of the samples were measured, and the outer surfaces inspected for the appearance of any damage, such as delamination, fibre debonding/protrusion etc. (12).

Mechanical testing

The flexural strength and Young's modulus were determined in four-point bending using 32mm outer span and 16 mm inner span. A cross-head speed of 0.1 mm/s was used.

The CN specimen technique was employed for fracture toughness determination. Chevron notches with angles of 90° were cut using a thin diamond wheel. A three point bending (with span of 16 mm) at a constant cross-head speed of 0.1 mm/min was employed. The fracture toughness was calculated from the maximum load (F_{max}) and the corresponding minimum value of geometrical compliance function (Y^*_{min}) using eq.

$$K_{IC} = \frac{F_{max} \cdot Y^*_{min}}{B.W^{1/2}}$$

where B and W are the thickness and height of the specimens respectively. The calculation of the geometric function Y^*_{min} for CN bend bars was based on the use of Bluhm's slice model and a simplified solution (13,14) of normalised stress intensity factors for chevron-notched specimens. The chevron-notch depth a_0 was measured after testing from SEM micrographs of fractured specimens.

The acoustic emission technique (AE) was used during the test. Traces of cumulative number of AE events were obtained in the same time scale as the load vs time plots. This technique allows for an accurate detection of the microcrack initiation onset at the CN tip, which occurs when a sharp increase in the number of AE events is observed.

RESULTS AND DISCUSSION

Fracture toughness K_{IC}

Table 2 summarises the results obtained from the mechanical tests performed. The data for the Young's modulus and the flexure strength for the as-received and heat-treated material correspond closely to values reported in the literature for similar composites [11], except for the two more extreme ageing conditions of 600°C/1000 hs and 700°C/100 hs.

Table 2. Summary of mechanical properties

Ageing condition temp. / duration	Young's modulus [GPa]	Flexure strength [MPa]	Fracture toughness [MPam$^{1/2}$]	Work of crack initiation [Jm^{-2}]
as-received	122	688		
500°/ 100 hrs	128	603	24.0 (0.9)	6990
600°/ 100 hrs	123	691	23.9 (1.4)	7550
600°/ 250 hrs	126	722	25.7 (1.3)	8310
600°/1000 hrs	107	253	20.8 (0.7)	6450
700°/ 100 hrs	100	184	19.2 (1.1)	4420

The fracture toughness determined by the CN specimen technique seems to be little dependent on the ageing condition. The material exhibits average K_{IC} values in the range 19-26 MPam$^{1/2}$, which are similar to data quoted in the literature (16). Typical load-displacement traces obtained in CN tests are shown in **Fig. 1** for samples aged at different temperatures for 100 hrs and for different durations at 600 °C. The poorest mechanical response of the samples aged at the most severe conditions, 100 hrs at 700 °C and 1000 hrs at 600 °C, is evident. See also **Fig. 2**.

Fracture surfaces exhibit the extensive fibre pull-out. Assessment of the micrographs have shown that the average pull-out lengths in both samples is similar, independently of the ageing condition. This is consistent with the little variation of fracture toughness, K_{IC}, and work of fracture initiation, W_i, data measured by the chevron notch technique, as shown in Table 2. Furthermore, the significant fibre pull-out effect exhibited by the composites is indicative of a weak interfacial bond (and thus a qualitative confirmation of the results of the push-out indentation measurements (12)).

The acoustic emission technique showed that individual fracture events started at about 1/2 to 2/3 of the maximum load (a smooth increase in the cumulative number of AE events). Microstructural changes corresponding to this acoustic emission should be matrix microcracking and partial local interfacial decohesion. These microfracture events, however did not result in a departure from linearity of the load - displacement trace. The first nonlinearity observed in all samples tested occurred at loads close to the maximum load. Crack propagation trough the glass matrix and fibre debonding and fracture only could be responsible for the strong increase in the cumulative number of AE events.

An increase in the number of AE events indicates that the actual crack is developed at the CN tip and the unstable fracture occurred from propagating crack perpendicular to the fibre axis. The actual onset of crack growth initiation at the CN tip was possible to be identified and valid conditions for obtaining fracture toughness data were thus met.

Work of fracture

The work of fracture is one of most usefull non-linear toughness characterisation parameters. In our paper, the work of crack initiation (W_i) was evaluated from the area under the load-displacement curves limited by the load point at unstable fracture initiation. This substantial portion of WOF was determined as the deformation work to the doubled area of fracture surface. Arising from W_i an energy criterion for crack initiation can be adopted arising from equation valid for straight notch/crack:

$W_i = K_{IC}^2 (1-\nu^2) / 2E$, or

$(2E.W_i)^{1/2} = K_{IC} (1-\nu^2)$.

For a first approximation this relation can be used as basis for correlation of work of crack initiation with fracture toughness. The values of W_i are given in **Fig. 3** as value of expression $(W_i{*}2E)^{1/2}$, i.e. in quantity comparable with fracture toughness values. Because the value of expression $(1-\nu^2)$ on the right side of above mentioned equations is equal nearly to 1, some overestimation can be observed as far as real values of W_i is observed. This aspect may be connected with uncertainty of Y^*_{min} determination (and K_{IC} underestimation) for CN specimen and materials with rising crack resistance curves mentioned in introduction and needs further investigation. Nevertheless, the good correlation between the values of W_i and K_{IC} is giving evidence about a application possibility of CN technique for toughness assessment of fibre/glass matrix composites.

Influence of ageing

Summary of fracture toughness data in dependence on ageing condition are shown in **Fig 2.** The results obtained are in broad agreement with data in the literature, in that thermal exposures at temperatures below 1000 °C in inert atmospheres do not have deleterious consequences on the properties of the SiC-Nicalon fibres and on the interface in silicate matrix composites (12). Thus, the temperature capability of the matrix is the limiting factor for applications involving non-oxidising environments. For the borosilicate glass matrix investigated here the limiting temperature is in the range 600 °C-700 °C, depending on the duration of the exposures, since at this temperature range, the matrix softens leading to microstructural damage that results in loss of mechanical properties. Short excursions at these temperatures may be possible, however.

CONCLUDING REMARKS

The present results have demonstrated that the chevron-notch technique can be a reliable method to assess fracture properties in brittle matrix composites reinforced by brittle fibres. The method has some advantages for estimating the fracture resistance of toughened materials, since it is possible to measure simultaneously both the fracture toughness and the work of fracture from the running crack. Because of dependance of work of fracture on the chevron geometry this can be used in a relative way by comparing values from the same CN specimen geometry.

The chevron-notch test measurements for different ageing conditions were consistent with the results of interfacial properties determined by a push-out indentation method [12]. The K_{IC} values determined using the chevron-notch technique (in the range 19-26 $MPam^{1/2}$) are comparable to data obtained in similar materials using other techniques. For the materials investigated, it was found that exposures to inert atmospheres at temperatures in the range 500 – 700 °C do not have deleterious consequences on the properties of the SiC-Nicalon fibres and on the interface.

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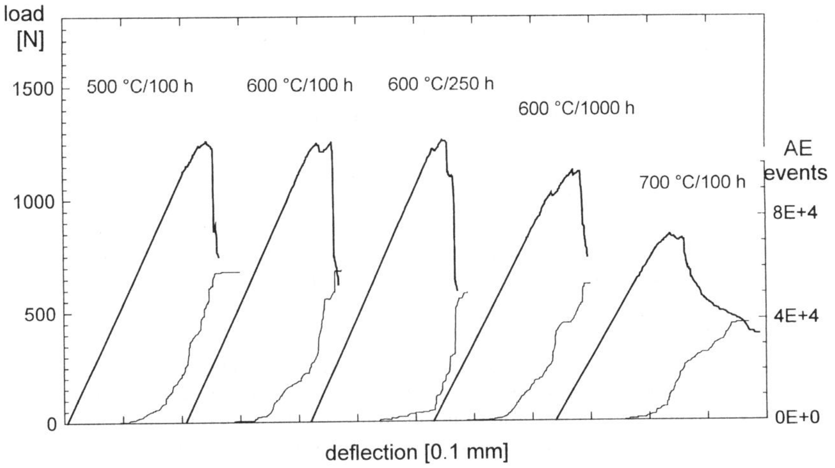

Figure 1. Examples of load-deflection curves and cumulative number of AE events for selected ageing temperatures

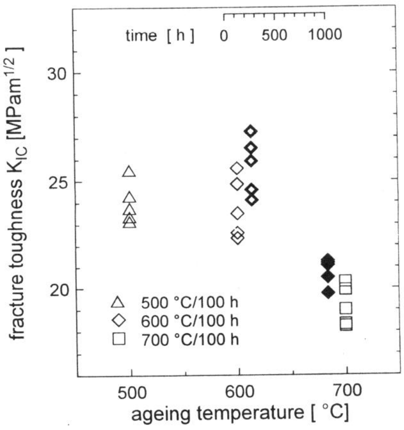

Figure 2. Fracture toughness data in dependence on ageing condition

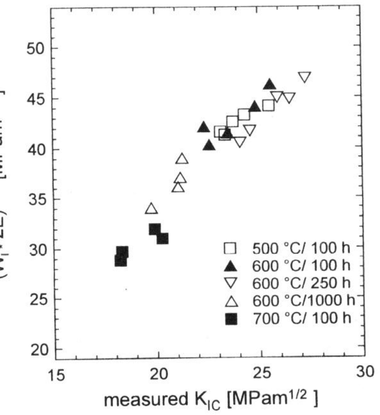

Figure 3. Correlation of work of fracture and fracture toughness values