RELIABILITY OF CHEVRON NOTCH TECHNIQUE FOR FRACTURE TOUGHNESS DETERMINATION IN GLASS COMPOSITES REINFORCED BY LONG FIBRES

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ABSTRACT

The applicability of the chevron-notch technique for changes monitoring in fracture behaviour of fibre/glass matrix composites was investigated. The fracture properties of a commercial available SiC-fibre reinforced glass matrix composite thermally shocked and thermally cycled have been compared with the behaviour in the “as-received” condition. The room-temperature properties have been evaluated by using four-point flexure strength and three-point flexure chevron notch techniques. Specimens with straight sharp notch tested in three point bending have been also followed. The fracture toughness values determined by the chevron-notch tests (the straight notch has been found as less susceptible method) were affected by thermal shocking. For the most thermal shocks applied comparable loss of flexure strength and stiffness of the samples was detected, which has been ascribed to microstructure changes occurred in the material during thermal changes.

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]. Nevertheless, an exact method of fracture behaviour quantification is needed when further development of fibre/brittle matrix composites and assessment of their possible operational degradation is to be made.

The chevron notched specimen technique is a well-established method used to determine the fracture toughness and the work of fracture of brittle materials [2,4-6], including particle reinforced glass matrix composites [7]. As indicated elsewhere [8], however, the technique has not received wide application to measure the fracture toughness of fibre reinforced brittle matrix composites. Other specimen configurations, for example straight notched specimens [9-11], have been more extensively used in these materials. Apart from our own previous research [8,11], few reports are available on the applicability of the chevron notched specimen to fibre reinforced ceramics and glasses. Mecholsky has pointed out, however, that as long as the notch is large enough to cover many fibres at the critical crack length, the chevron notch test represents a useful method to measure the fracture toughness and the work of fracture in ceramic composites [10].

In the work of Ha and Chawla [12], for example, it was shown that using the chevron notched three-point flexure technique the different mechanical behaviour of mullite matrix composites containing mullite fibres with different coatings could be detected. In our previous investigation [8,11], the differences in fracture behaviour of glass matrix composites after different thermal ageing conditions were detected by this technique and reliable fracture toughness data were obtained. On the basis of such previous experiences on chevron notch test, it was thought to be an instructive exercise to extend the investigation to samples thermally loaded under different conditions, i.e. thermal shock and thermal cycling, as done in the present study.

The purpose of the contribution can be seen in the analyses of the suitability of the chevron notch technique for the assessment of the fracture behaviour of fibre/glass matrix composites and the degradation that may occur following thermal exposition or thermal cycling.

MATERIAL AND EXPERIMENTAL METHODS

Materials

The material investigated was a commercially available unidirectional SiC Nicalon (NL202) fibre reinforced borosilicate (DURAN) glass matrix composite fabricated by Schott Glaswerke (Mainz, Germany). Information on the composite constituents is given in **Table 1**. The composite was prepared by the sol-gel-slurry method [13]. The samples were received in the form of rectangular test bars of nominal dimensions 4.5x3.8x100 m^3. The density of the composites was 2.4 g/cm^3 and their fibre volume fraction 0.4. Fairly regular fibre distribution and the absence of porosity were found by microstructural investigations.

TABLE 1
PROPERTIES OF THE PURE GLASS (DURAN) AND COMPOSITE CONSTITUENTS [13]

	Density [g/cm^3]	Young's modulus [GPa]	Poisson's ratio	Thermal exp. coeff. [K^{-1}]	Tensile strength [MPa]
Matrix DURAN	2.23	63	0.22	$3.25 * 10^{-6}$	60
Fibre SiC Nicalon	2.55	198	0.20	$3.0 * 10^{-6}$	2750
Fibre/glass composite	2.40	118	0.21	$3.1 * 10^{-6}$	600-700

Thermal ageing involved heating the samples in air at 600 ºC and 650 ºC for 20 minutes and then dropping them into a stagnant water bath maintained at room temperature. Up to 20 quenches were conducted in each sample. Thermal shock under these conditions has been shown to lead to damage in the form of matrix microcracking in these materials [14]. For the thermal cycling test in air, the samples were alternated quickly between high temperature (700 ºC) and room temperature for different numbers of cycles. The details of procedures have been given elsewhere [15].

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.

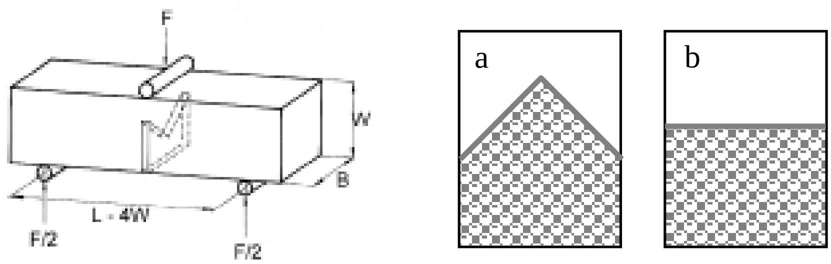

Figure 1: Schematics showing the loading geometry and fracture plane for (a) chevron notch and (b) straight notch technique

The chevron notch (CN) specimen technique was employed for fracture toughness determination (**Figure 1**). 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. Graphs of load versus deflection were recorded and the maximum force was determined from each trace. The fracture toughness value was calculated from the maximum load (F_{max}) and the corresponding minimum value of geometrical compliance function (Y^*_{min}). The calculation of the function Y^*_{min} for chevron notch bend bars was based on the use of Bluhm's slice model. The calculation procedure used for the purposes of this investigation has been described elsewhere [6]. The chevron-notch depth a_0 was measured from SEM micrographs of fractured specimens.

Additionally, straight-notched specimen technique (Figure 1 b), recommended as a standard procedure [17], was also employed. The straight notch having width of about 150 μm and depth of about 1.5 mm has been ground by thin diamond wheel. Then the tip was sharpened by hand by polishing with a conventional razor blade and diamond paste of size of 0.1 μm. Testing conditions were the same as in case of CN technique.

The acoustic emission technique (AE) was used during the test. Traces of cumulative number of counts (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 chevron notch, which occurs when a sharp increase in the number of AE events is observed. Valid measurements for computing K_{IC} are those in which this increase of AE events coincides with the end of the linear part of the force versus time trace, as explained below.

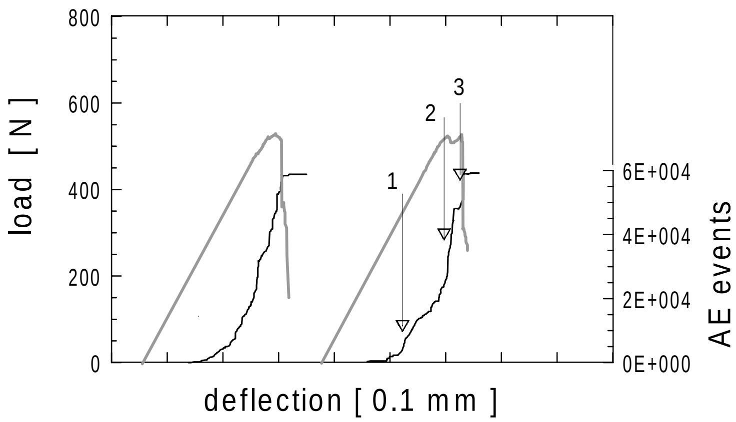

Figure 2: Load deflection curves obtained from 3-point bend tests of CN specimens with cumulative curve of AE events

RESULTS AND DISCUSSION

Correlation of chevron notched and straight notched specimens

Fracture behaviour of pure glass

For pure glass the load deflection traces have been found to be qualitatively comparable. The typical examples of load - deflection records are shown in **Figure 3**. Summary of primary data is supplied in **Table 2**. For specimen with straight notch the load is increasing to a maximum, then the crack is initiated below the notch root running more or less by unstable manner. For CN specimen the crack was initiated at about 1/2 to

TABLE 2

PRIMARY DATA FROM BEND TESTS CARRIED OUT WITH PURE GLASS

STRAIGHT NOTCH				CHEVRON NOTCH			
Nr.	Fracture load F_{max} [N]	Notch depth [mm]	Fract. toughness K_{IC} [MPam$^{1/2}$]	Nr.	Fracture load F_{max} [N]	Notch depth [mm]	Fract. Toughness K_{IC} [MPam$^{1/2}$]
2/1	16,50	1,577	0,69	3/1	13,39	1,390	1,23
2/2	13,06	1,914	0,67	3/2	9,34	1,390	0,79
2/3	13,24	2,077	0,69	3/3	9,85	1,400	0,82
2/4	19,03	1,660	0,83	3/4	9,38	0,978	0,59
2/5	26,86	1,639	1,16	3/5	13,47	0,925	0,81
2/6	17,92	1,667	0,79	3/6	9,99	0,988	0,64
2/7	17,19	1,618	0,75	3/7	11,33	0,990	0,73
2/8	16,86	1,534	0,70	3/8	12,51	1,020	0,84

2/3 of maximum force, than is running in stable regime (i.e. the crack driving force being controlled by increasing length of crack front). The onset of unstable fracture (necessary for K_{IC} determination) corresponds to maximum force. Fully compatible fracture behaviour can be observed when comparing both specimen techniques based on load deflection traces. When comparing the data for pure glass nearly comparable K_{IC} values have been gained from both specimen techniques (0.75 $MPam^{1/2}$) those for CN specimens and (0.73 $MPam^{1/2}$) for specimens with straight notch. As shown in Figure 4 the scatter characteristics are slightly different. This is explained by different sizes of highly stressed zones (the higher volume stressed the lower scatter - specimens with straight notch).

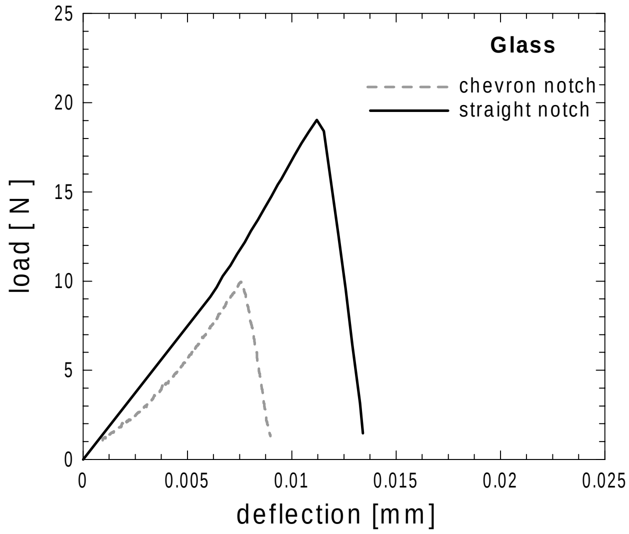

Figure 3: Load deflection traces obtained at 3-point bend test for pure glass

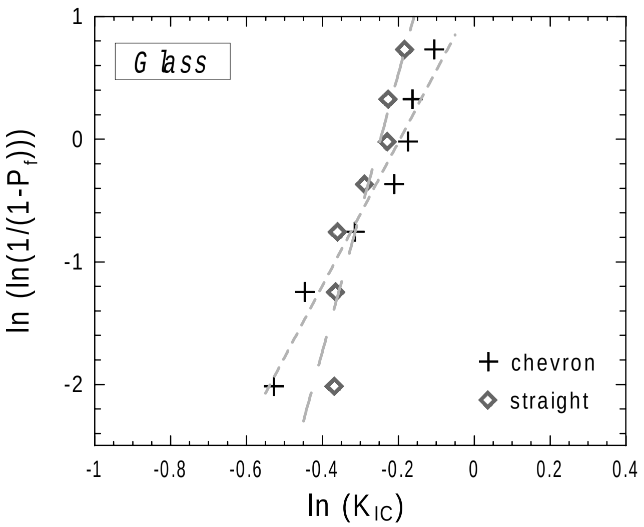

Figure 4: Weibull plot of fracture toughness data for pure glass

Fracture characteristics for composite material

For the composite, the record from specimens with straight notch is typical by pop-in effects that can be observed at maximum load (**Figure 5**). The pop-ins have been assigned to the stopping of major crack propagation at fibre/matrix interfaces followed by the interface delamination. Fractographic observations have supported this explanation. The delamination is observed in fracture surfaces very near the notch root and in almost cases also on specimen side surfaces. For the chevron notch specimen the crack is developed at about 1/2 to 2/3 of maximum load and is smoothly running up to maximum load after which unstable fracture occurs.

Acoustic emission, a technique that has gained application for detecting the onset of microcracking when testing composite materials [17], was used to assess whether valid conditions for obtaining fracture toughness data from the CN test were met. When a mechanical stress is applied to a composite, several fracture phenomena can occur in the material, including matrix microcracking, fibre-matrix debonding, delamination and fibre failure. 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 point labelled by arrow 1 in Figure 2). 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 non-linearity observed in all samples tested occurred at loads close to the maximum load applied, and it was associated with a strong increase in the cumulative number of acoustic emission events. This increase is so high that the crack propagation trough the glass matrix and fibre debonding and fracture only could be responsible for this significant effect. An increase in the number of AE events observed at the end of the linear part of the load-deflection trace (arrow 2 – Figure 2), indicates that the actual crack is developed at the chevron-notch tip. In these cases, the measurements of K_{IC} can be taken as valid, since the unstable fracture (at maximum force) occurs from a propagating crack perpendicular to fibre axis.

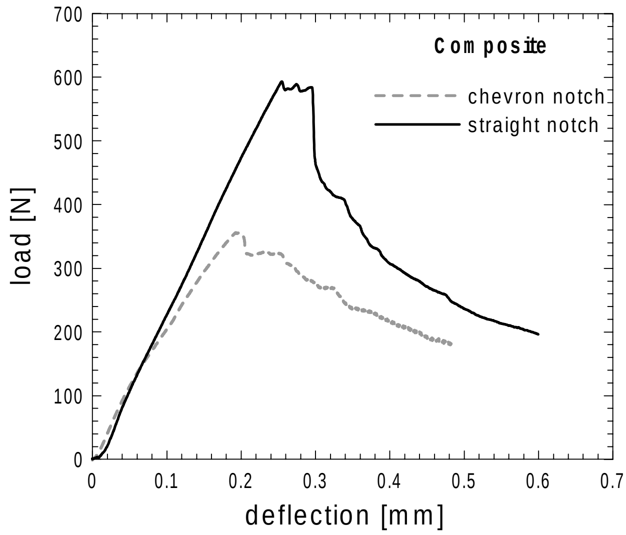

Figure 5: Load deflection traces obtained at 3-point bend test for composite material

TABLE 3

PRIMARY DATA FROM BEND TEST OBTAINED FOR SiC FIBRE-GLASS COMPOSITE

STRAIGHT NOTCH				CHEVRON NOTCH			
Nr.	Fracture load F_{max} [N]	Notch depth [mm]	Fract. toughness K_{IC} [MPam$^{1/2}$]	Nr.	Fracture load F_{max} [N]	Notch depth [mm]	Fract. toughness K_{IC} [MPam$^{1/2}$]
8/1	593,12	2,117	25,19	9/1	295,32	1,770	24,31
8/2	563,47	2,082	23,56	9/2	296,45	1,770	24,60
8/3	642,53	2,148	28,01	9/3	279,91	1,790	24,08
8/4	656,15	2,058	26,93	9/4	303,45	1,770	26,10
8/5	663,23	1,912	24,77	9/5	285,54	1,800	25,25
8/6	653,93	2,056	26,81	9/6	283,39	1,800	24,18
8/7	678,25	2,069	28,04	9/7	297,22	1,760	23,64

Differences have been found when comparing the fracture toughness characteristics determined by using the straight notch bend specimens with those obtained from chevron notched specimens. Typical examples of load deflection traces are shown in **Figure 5** and **s**ummary of primary data is given in **Table 3**. In Weibull plot the differences are evident from the shift of prime along K_{IC} axis (**Figure 6**) showing the overestimation of toughness characteristic for specimen with straight notch (26.19 MPam$^{1/2}$ for straight notch technique against 24.59 MPam$^{1/2}$ for CN technique). Not only the average K_{IC} values but also the scatter characteristic differ in remarkable extent. Only the data obtained from load - deflection traces having no more than one pop-in are on level fully comparable with data from CN specimens.

The higher K_{IC} values obtained from specimens with straight notch can be explained by premature localisation of fracture events to the stage of delamination. This is known phenomena typical also for long fibre/glass composites that delamination may occur also in case the crack is already running perpendicularly to fibres. The standard matrix cracking preceding the unstable crack formation is more or less suppressed and/or it is strongly affected by delamination. The pop-ins observed correspond well to this phenomenon even when they are accompanied by strong increase of AE events (arrow 3 in figure 2) – after delamination some time is needed for further macrocrack development. The microcracking localisation is connected with larger energy dissipation and causes movement of unstable (macro)crack initiation to later stages of loading. Because of impossibility of crack length measurement during test an uncertainty arises how to obtain the real crack length values corresponding exactly to the critical conditions of load, both quantities being necessary for K_{IC} values calculation.

For chevron notch the failure is initiating in relative small volume near the chevron notch tip and condition for crack propagation without premature localisation (to fibres/matrix interfaces) are more advantageous. The crack tip driving force is higher than in case of straight notch. This is important feature of chevron notch that condition for unstable crack propagation are formed during running crack whereas as the critical condition (for K_{IC} determination) the initial notch (depth) is taken.

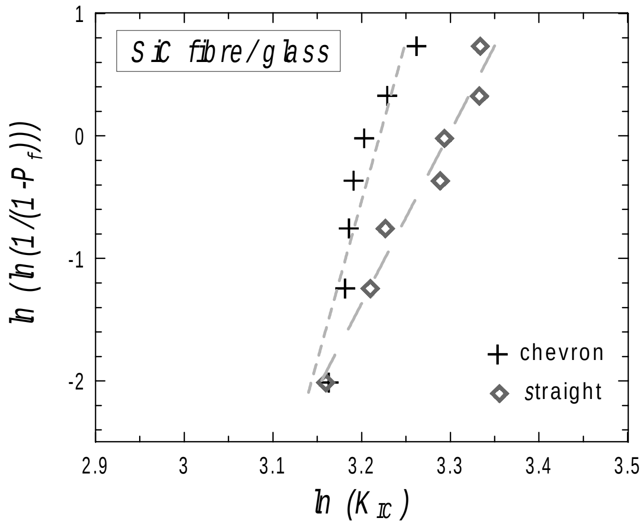

Figure 6: Weibull plot of K_{IC} data for composite material

The fracture initiation in CN specimens is thus connected with comparably lower probability than for specimen with straight notch that major crack tip meat the weak matrix-fibre interface and change the trajectory along the interface. The differences observed and the evidently more advantageous condition for the exact measurement led to preferable use chevron notch technique for fracture toughness determination in long fibres/brittle matrix composites.

Fracture toughness of thermally aged and shocked materials

Figure 7 summarises the results obtained from the mechanical tests performed. Except for the sample thermally cycled from 700 °C for a high number of cycles (821), for which a strong loss of flexure strength was measured (422 MPa against 688 MPa of as rec state), the data seem not to be significantly influenced by the thermal loading conditions investigated. The material exhibits average K_{IC} values in the range 18-26 $MPam^{1/2}$, which are close to data quoted in the literature for similar fibre reinforced glass and glass-ceramic composites [9] and for SiC/SiC composites [17]. Observation of fracture surfaces by SEM revealed extensive fibre pull-out, for thermally shocked samples and for the sample thermally cycled for 466 cycles. Comparison of fracture surface micrographs for different conditions showed that the average pull-out lengths were similar to that of the as-received material. This is consistent with the little variation of fracture toughness, K_{IC}, and work of fracture initiation, W_{in}, (as shown below in Figure 8). Furthermore, the significant fibre pull-out effect exhibited by the composites is indicative of a weak interfacial bond remaining after the thermal loading. The interfacial bond in the present material before and after thermal shock loading was measured using a push-out indentation technique and the results were reported elsewhere [8]. Those measurements confirmed that under the conditions of thermal loading investigated in the present study, the interfacial shear stresses, e. g. frictional shear strength (τ_{fr}) and debunking shear strength (τ_{db}), remained nearly constant and were closed to the as-received values. Thus, thermal shock and cycling at the temperatures investigated did not lead to a degradation of the carbonaceous interface.

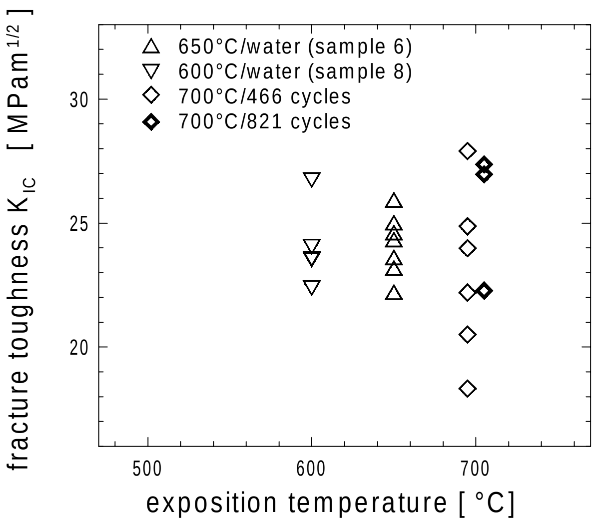

Figure 7: Fracture toughness of thermally shocked and cycled samples

The material aspects have been analysed in detail elsewhere [18], the supplied data should evidence the high susceptibility of chevron notch technique for identification of very subtle differences in fracture behaviour and extent of toughening mechanisms in composites.

Work of fracture initiation

The work of crack initiation (W_{in}) was evaluated from the area under the load-displacement curves limited by the load point at unstable fracture initiation. This substantial portion of the work of fracture was determined

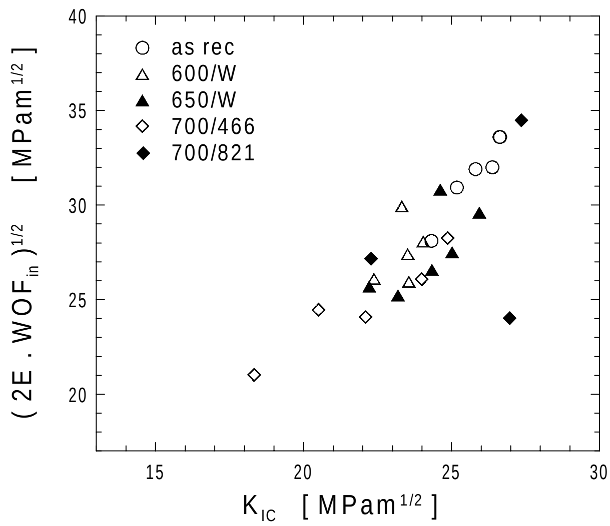

Figure 8: Correlation of work of crack initiation with fracture toughness for CN specimens

as the deformation work related to the doubled area of fracture surface. Arising from W_{in}, an energy criterion for crack initiation can be adopted by considering the following equation, valid for straight notch/crack:

$$W_{in} = K_{IC}^2 (1-\nu^2) / 2E \qquad (1)$$

or

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

where E, ν are, respectively, the Young's modulus and the Poisson's ratio of the material. To a first approximation these relations can be used as a basis for correlating the work of crack initiation with fracture toughness. The values of W_{in} are given in Fig. 8 as value of expression $(W_{in}*2E)^{1/2}$, i.e. in a quantity comparable with fracture toughness values. Because the value of $(1-\nu^2)$ on the right-hand side of Eqs. (1) and (2) approximates to 1, some overestimation can be observed in comparison to the real values of K_{IC}. This aspect may be also connected with the uncertainty of Y^*_{min} determination for CN specimen in materials with rising crack resistance curves and it needs further investigation. Nevertheless, the good correlation between the values of W_{in} and K_{IC} is giving support about the suitability of the CN technique for toughness assessment in this kind of fibre reinforced brittle matrix composites.

CONCLUSION

The work has demonstrated that the chevron-notch technique can be a reliable method to assess fracture properties in brittle matrix composites reinforced by long brittle fibres. The data obtained have shown the high susceptibility of CN technique for identification of very subtle differences in fracture behaviour and extent of toughening (degradation) mechanisms in composite. Because of the dependence of work of fracture on the chevron notch geometry, the technique can be used in a relative way to compare values from the same CN specimen geometry.

The K_{IC} values determined using the CN technique on the SiC-fibre reinforced glass matrix composite investigated (in the range 18-26 $MPam^{1/2}$) are comparable to data in the literature obtained in similar materials using other techniques. The fracture toughness and work of fracture values determined by the CN specimen method confirm previous findings regarding the retention of "composite" fracture behaviour of the investigated materials after thermal shock and thermal cycling under the conditions investigated.

ACKNOWLEDGEMENTS

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