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High-temperature mechanical behaviour of multidirectional Nicalon/CAS-II composite

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High-temperature mechanical behaviour of Nicalon/CAS-II composite has been investigated. Oxidation of the exposed interfaces along matrix cracks at 1000 °C lowered the longitudinal unidirectional strength to the stress level at which matrix cracking began to occur. The strength of cross-plied composites was also severely reduced in 800 °C air. Transverse plies cracked prior to 0° ply matrix cracking. However, embrittlement did not occur until the matrix in the 0° plies cracked. It was established that oxidation does not take part in crack growth parallel to the fibres, except adjacent to exposed edges. Neither does oxygen enter 90° ply cracks in cross-plied composites in sufficient quantity to produce oxidation embrittlement, at least up to the 0° matrix cracking strain.

1. Introduction

Nicalon (a polymer-derived SiC-based fibre [1, 2]) has a unique non-stoichiometric chemistry that makes it particularly suited to the development of high-strength glass-ceramic matrix composites. Chemical reactions can occur between the fibre and matrix during processing of Nicalon-reinforced glass-ceramics, producing the carbon-rich interphase region [3–6]. The strength of the fibre–matrix interphase and the resulting composite mechanical properties are significantly influenced by the formation of such interphase regions [5]. The carbon-rich layer of moderate strength effectively limits load transfer between the matrix and the strong fibre, either by cohesively failing or adhesively separating from the fibre or the matrix. The carbon-rich layer forms a bond strong enough for load transfer, yet weak enough to debond readily and allow fibre bridging during crack propagation [4, 7]. The layer is either non-existent or carbon-poor in composites that exhibit little toughness [7].

Nicalon-reinforced glass-ceramic matrix composites with a well-developed carbon interphase exhibit excellent longitudinal toughness and crack-deflecting ability at room temperature [3, 4, 6, 8]. However, reductions of strength and strain to failure (in the fibre direction) have been observed when testing at temperatures as low as 400 °C [9]. It has been proposed that the transition from tough behaviour at room temperature to brittle behaviour at elevated temperatures is probably due to fibre strength degradation and/or increased fibre–matrix bond strength caused by oxidation effects at interfaces exposed to the environment [4, 10].

The oxidation reaction is not initiated until the matrix cracks upon stressing, allowing penetration of high-temperature air [11, 12]. The air then infiltrates the composite and attacks the low-strength carbon-rich fibre–matrix interphase in such a way as to cause an embrittlement of the fracture process [4, 13]. The interphase carbon layer is removed and a stronger silica bond between the fibre and the matrix is formed [14]. Ingress of oxygen is also possible at cut (or ground) surfaces prior to matrix crack initiation [15, 16]. Oxidation can initiate at cut fibre ends which intersect the composite surface, and then spreads predominantly along the interface from exposed fibre ends rather than through transverse penetration around and between fibres [15, 17].

The objective of this study was to develop an improved understanding of the mechanical behaviour of multidirectional Nicalon/glass ceramic composites at high temperatures. Previous to this study, very little work had been reported on the reasons for observed embrittlement of multidirectional materials at high temperatures. The approach taken here was to focus on the most simple multidirectional laminate, containing 0° and 90° interspersed plies, where 0° is the direction of applied stress.

2. Experimental procedure

The materials used in this study were either unidirectional ($[0]_{16}$) or cross-ply ($[0/90]_{8s}$) Nicalon fibre/calcium aluminosilicate glass ceramic (CAS-II) matrix composites. They were supplied by Corning,

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Inc, in the form of square plates, approximately 15 cm by 15 cm and about 3 mm thick.

A cold-grip test configuration using adhesively bonded aluminium tabs, developed by Schutz [18], was utilized for tensile testing. The tensile behaviour of unidirectional 0° material was first studied to characterize its response in the absence of 90° plies. The cross-plied 0/90 laminate was then characterized to observe the effects of cracking of the 90° plies, the first damage to develop as the stress increases, on the embrittlement of the 0° plies which carry most of the force.

Finally, to understand better the effects of the high-temperature air environment on the 90° plies, direct fracture toughness studies were carried out on unidirectional material with cracks growing parallel to the fibres to simulate cracks in the 90° plies of the cross-plied laminate. The double torsion (DT) test technique [18–33] was used to determine the transverse fracture toughness, G_{Ic}.

The origins and progression of oxygen embrittlement at each stage were investigated through measurement of the bond strengths of individual fibres in the cracking zones, coupled with electron microscopy. Thus, detailed micro-scale measurements of bond properties near or at crack surfaces (and on uncracked controls) have been carried out at each stage. This includes careful mapping of bond-strength distributions for fibres adjacent to the crack surface in transverse fracture tests as a function of position across the specimen thickness. The indentation technique, where individual fibres are compressively loaded on a polished surface to produce debonding [15, 16, 33–40], was used to measure fibre–matrix bond strength. The bond measurements are correlated with scanning electron micrographs of the fracture surfaces to build a more complete picture of the embrittlement process in this class of glass-ceramic matrix composites.

Further details of the experimental methods, equipments and procedures are described elsewhere [33].

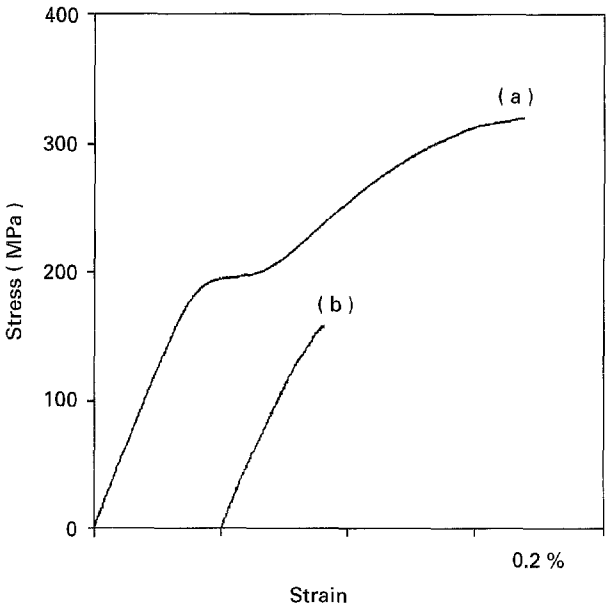

Figure 1 Longitudinal tensile stress–strain curves of unidirectional Nicalon/ CAS-II composites tested (0.7% strain/h) at (a) 20 °C, and (b) 1000 °C.

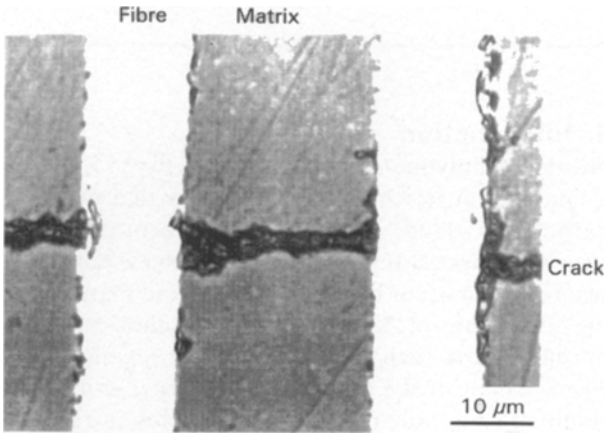

Figure 2 Fibre–matrix debonding prior to crack propagation preventing matrix crack penetration through the fibres.

3. Results and discussion

3.1. Tests on unidirectional Nicalon/CAS-II

A representative room-temperature longitudinal stress–strain curve for unidirectional Nicalon/CAS-II is shown in Fig. 1, curve (a). Initially, the material exhibits linear elastic behaviour with a modulus closely predicted [33] by the rule of mixtures, up to a strain of approximately 0.15%. This linear elastic part is followed by a region over which the response becomes non-linear, usually attributed to the formation of matrix cracks normal to the direction of applied load, resulting in a plateau on the stress–strain curve. At higher strain levels, above approximately 0.25% strain, the material once again approaches linear behaviour dominated by fibre properties until failure. These results are in good agreement with other tensile data previously reported [18].

Extensive matrix cracking and fibre pull-out were characteristic of the room-temperature longitudinal fracture of unidirectional Nicalon/CAS-II. The high matrix crack density observed on the specimen edges examined microscopically after fracture, correlates well with the three-stage stress–strain curve of Fig. 1, curve (a). This indicates that the fibres alone contributed to the strength at high strains. Matrix cracks did not propagate through fibres (Fig. 2) because fibre debonding occurred prior to crack propagation through the fibres and/or matrix cracks deflected parallel to the fibres. The fracture surface displayed extensive fibre pull-out, as in Fig. 3. Fibre pull-out lengths up to 1 mm were commonly observed.

Interfacial bond strength of the unidirectional Nicalon/CAS-II plate (as-supplied) used in the tensile tests was measured to be 381 ± 36 MPa. This value of about 400 MPa for the interfacial shear strength is high enough for load transfer between the fibres and the matrix, yet low enough to produce debonding during matrix cracking, which will prevent the crack from propagating through the fibres. Higher bond strengths were reported to lead to brittle fracture and

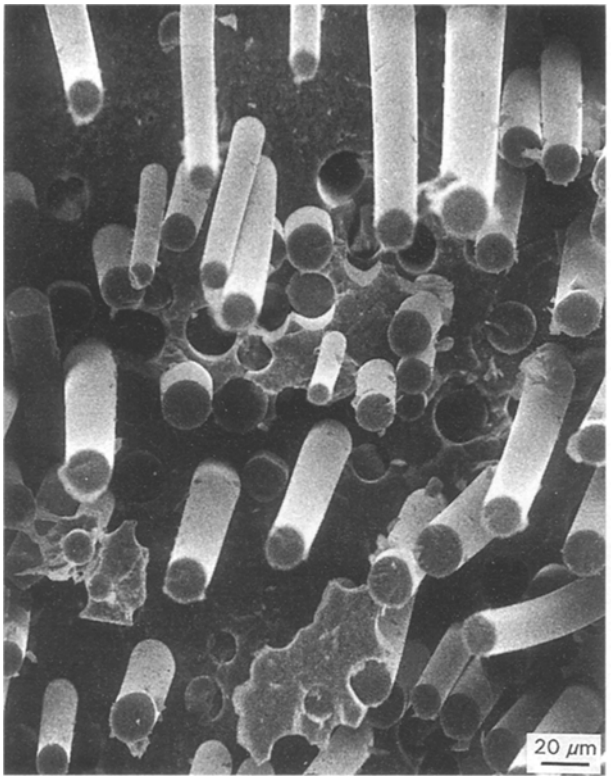

Figure 3 Fracture surface of a unidirectional Nicalon/CAS-II tensile specimen tested at room temperature.

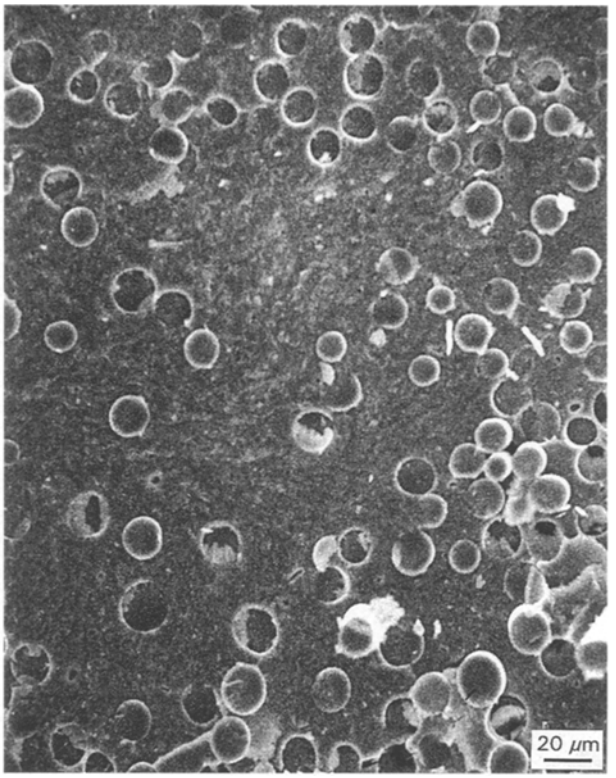

Figure 4 Fracture surface of a unidirectional Nicalon/CAS-II tensile specimen tested in air at 1000 °C (0.7% strain/h).

lower longitudinal strength [15, 34]. The moderate level of bonding between the matrix and the fibre is associated with the interfacial carbon layer which forms upon processing of Nicalon-reinforced ceramics [3–7, 33].

At 1000 °C, the composite failure strain decreased to the matrix cracking strain and the behaviour was brittle. The stress–strain curve (Fig. 1, curve (b) remained (almost) linear to failure indicating that the matrix cracking strain was not exceeded. The fracture surface was planar with no fibre pull-out (Fig. 4), characteristic of brittle fracture. This planar fracture surface is indicative of a degraded carbon interphase layer, with a subsequent formation of a strong interfacial bond [14, 15, 17].

To confirm the origin of the observed embrittlement, microdebonding tests were performed on fibres directly on the fracture surface. None of the fibres tested could be debonded. The bond strengths were extremely high and either the fibres cracked radially or the measurement capacity of the microdebonding apparatus (> 1000 MPa) was reached before debonding. It should be noted that the fracture surface was exposed to high-temperature air, not only during the test but also after the test while cooling slowly in the furnace. The cooling rate of the furnace is approximately 500 °C h^{-1}. Data from Dannemann [15] show that the tensile strength of Nicalon fibre/glass matrix composite is significantly reduced when the bond strength as measured in the microdebonding test exceeds about 600 MPa. Thus, the control Nicalon/CAS-II used here (about 400 MPa) is already relatively high and the effects of oxidation and subsequent bond formation can easily raise the bond strength to a level where matrix cracks propagate through the fibres.

Post-test microscopic examination of the specimen edges revealed no observable matrix cracking, indicating that specimen failure occurred by the advance of a single matrix crack, probably the first significant matrix crack to form in the gauge section of the specimen.

3.2. Behaviour of cross-plied laminates

A typical tensile stress–strain curve for a 0/90 cross-ply composite is shown in Fig. 5, curve (a). A decrease in the elastic modulus of the composite is observed at about 0.05% strain resulting from the failure of transverse (90°) plies [15]. The curve continues to be approximately linear up to a strain of about 0.15% and then deviates from linearity, as would be expected due to the onset of matrix cracking in the longitudinal (0°) plies. This is followed by the final (almost linear) regime which is governed by fibre properties in the 0° plies.

Matrix cracking both in 0° and 90° plies was extensive. Most cracks in 90° plies propagated into the 0° plies with fibre bridging (Fig. 6). A fibrous, tough composite fracture of the 0° plies was achieved in room-temperature control specimens. Thus, as also observed by others [15], 90° plies do not severely affect the performance of the 0° plies. However, the 90° plies do complicate the failure process in that cracking of the 90° plies occurs at a lower strain than does the first damage (matrix cracking) in unidirectional material. As Fig. 6 indicates, the cracks in the 90° plies can penetrate past the first fibres of the 0° plies, possibly exposing them to environmental attack.

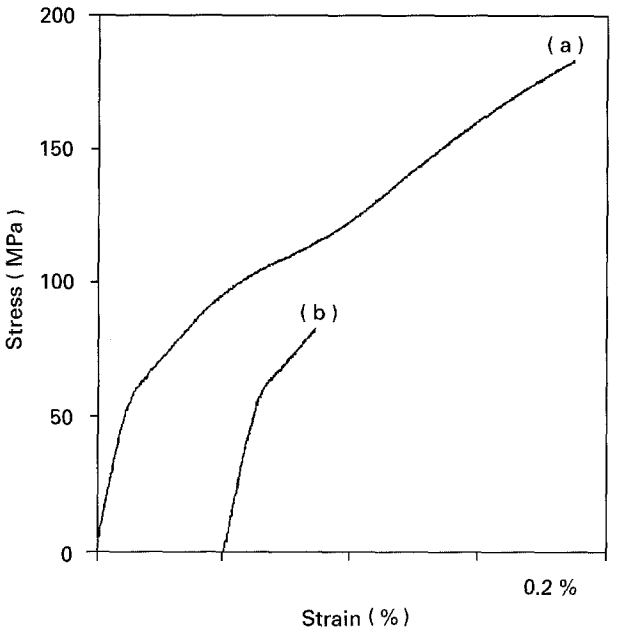

Figure 5 Tensile stress–strain curves of cross-ply Nicalon/CAS-II composites tested (0.7% strain/h) at (a) 20 °C, and (b) 800 °C.

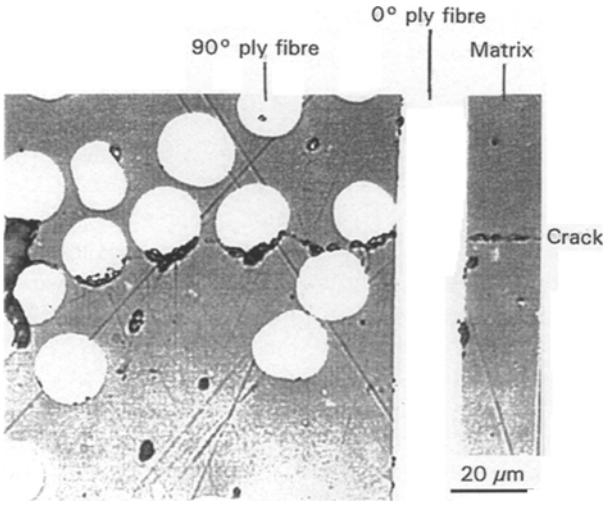

Figure 6 A crack in a cross-ply Nicalon/CAS-II tensile specimen tested at room temperature.

The stress–strain curves for the tensile tests performed at room temperature and 800 °C (Fig. 5) clearly show that strength is again reduced by the presence of high-temperature air, as in the unidirectional case. Specimens failed at 800 °C just as the end of the second linear portion of the stress–strain curve was reached (the point where matrix cracking in the 0° plies begins [15]). The fracture morphology was similar to that of the unidirectional Nicalon/CAS-II specimens tested at 1000 °C.

The fracture surface was planar without any noticeable fibre pull-out as shown in Fig. 7, indicating a higher level of fibre–matrix interfacial strength. Fig. 7 shows that a crack in the 90° ply propagated in a planar fashion through the adjacent 0° ply. The fracture surface of the 90° ply itself is also flatter, compared with the loose fibres on the surface of the room-temperature test in Fig. 8.

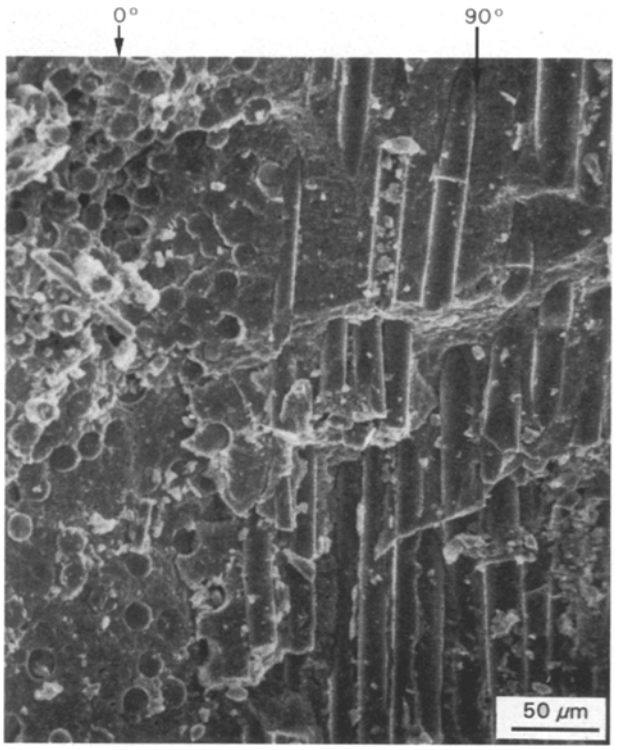

Figure 7 Fracture surface of a cross-ply Nicalon/CAS-II tensile specimen tested in air at 800 °C (0.7% strain/h).

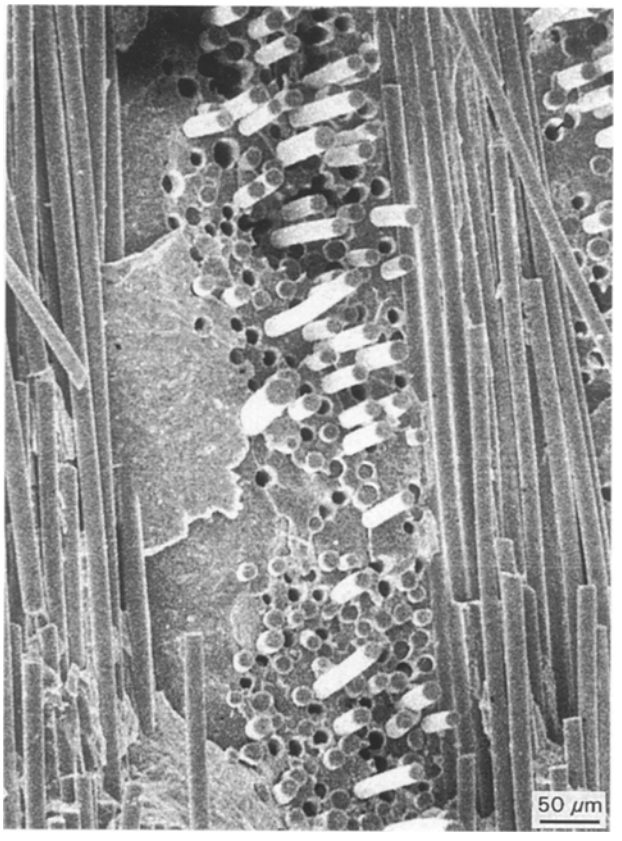

Figure 8 Fracture surface of a cross-ply Nicalon/CAS-II tensile specimen tested at room temperature.

Microdebonding tests were also performed on the fracture surface to confirm the increase in the interfacial bond strength. Most fibres broke before debonding because of extremely high fibre–matrix bond strength. Two fibres which could be debonded

revealed interfacial bond strength values of 806 and 1434 MPa, which are significantly higher than the fibre–matrix bond strength (430 ± 24 MPa) measured on the control material. Thus, the 0° ply suffered the same increase in bond strength, and consequent embrittlement, as discussed for unidirectional material.

Examination of the stress–strain curve obtained from the tensile test performed on cross-ply Nicalon/CAS-II at 800 °C indicates that composite strength is limited by the 0° ply proportional limit point (matrix cracking) and not the 90° ply failure point. Prewo [3] observed a similar tensile behaviour for cross-ply Nicalon/LAS system at 1000 °C in air, including 100 h stress-rupture tests. The embrittlement of the fracture process is apparently not initiated until the matrix in 0° plies cracks upon stressing, allowing penetration of high-temperature air into the interior of the 0° plies.

Microscopic examination and microdebonding tests were performed on an edge of a cross-ply composite tensile specimen fractured at 800 °C. The polished edge (at about 0.5 mm depth in from the as-tested edge) of the fractured specimen revealed extensive cracking in the 90° plies but not in the 0° plies (as opposed to extensive matrix cracking of the 0° plies at room temperature). Most of the cracks in the 90° plies stopped or deflected in the 0° direction upon reaching the fibres of 0° plies. A few of the cracks in the 90° plies propagated to the closest 0° fibres (Fig. 9), interestingly with bridging fibres as in the case of room-temperature fracture.

Microdebonding tests were performed on the fibres in 90° plies close (within a fibre diameter distance) to the fracture surface and also within a fibre diameter distance to the 90° ply cracks in the interior of the specimen (0.5 mm or more away from the fracture surface). While the fibres next to the fracture surface were strongly bonded to the matrix (with bond strengths of greater than 812 MPa), and most of them broke before debonding, the fibre–matrix bond strength obtained by testing the fibres next to the 90° ply cracks in the interior was 462 ± 61 MPa, which is not significantly higher than that of the control material (430 ± 24 MPa).

These microscopy and microdebonding test results indicate that cracks in the 90° plies of the cross-ply Nicalon/CAS-II composites might not be sufficiently open prior to fracture for oxygen to reach the interphase in sufficient quantity to cause the oxidation-induced bond-strength increase. However, the planar fracture surface of 90° plies (Fig. 7) compared to the rougher surface with loose fibres at room temperature (Fig. 8), indicates a significant increase in the interfacial bond strength of the 90° plies on the fracture surface of the specimen tested at 800 °C. This observation, combined with the above microdebonding examination near the fracture surface and also in the interior of the specimen, suggests that oxidation of the interphase in 90° plies occurs only when the cracks open sufficiently for the high-temperature air to penetrate in, or on, the fracture surface after fracture.

3.3. Transverse fracture toughness test results

Transverse fracture toughness, G_{Ic} versus time to failure (testing time) plot of Nicalon/CAS-II specimens for room temperature, 800 and 1000 °C is shown in Fig. 10. There seems to be no significant effect of test rate on transverse fracture toughness at room temperature. The G_{Ic} data scatter in the range 38–61 J m^{-2} with an average value of 48 J m^{-2}. On the other hand, the transverse fracture toughness of Nicalon/CAS-II composite was determined to be lower than 38 J m^{-2} for the fracture tests performed at 800 and 1000 °C. Also, the test rate had some effect on the composite toughness at 1000 °C. The average G_{Ic} measured on the samples fractured in 9–10 h was about 20% lower than that of the ones fractured in less than 2 h. Overall, there is a reduction in G_{Ic} from about 48 J m^{-2}

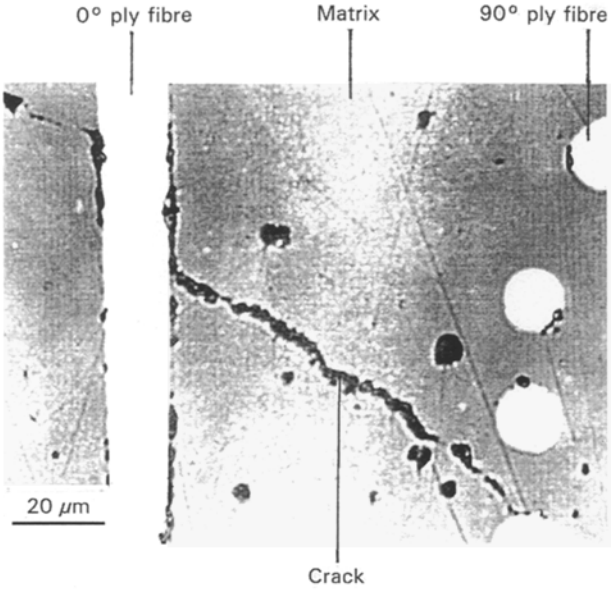

Figure 9 A crack at a depth of about 0.5 mm below an edge of a cross-ply Nicalon/CAS-II tensile specimen fractured at 800 °C (0.7% strain/h).

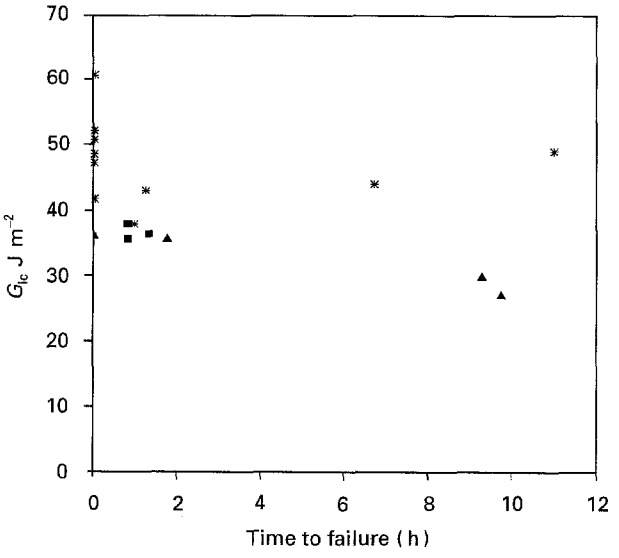

Figure 10 Transverse fracture toughness versus time to failure plot of Nicalon/ CAS-II composite. (*) 20° C, (■) 800 °C, (▲) 1000 °C.

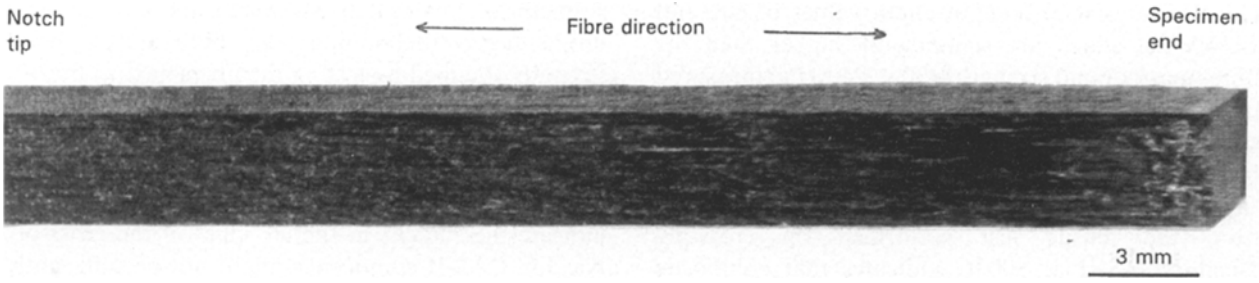

Figure 11 Fracture surface of a Nicalon/CAS-II double torsion specimen tested at 1000 °C in air (time to failure 10 h).

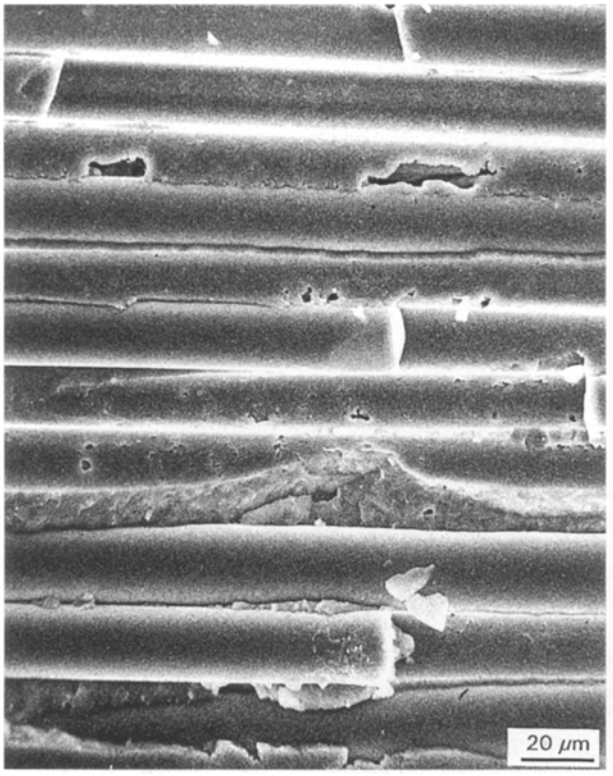

Figure 12 Scanning electron micrograph taken on the planar part in the first half of the fracture surface of a Nicalon/CAS-II double torsion specimen tested at 1000 °C in air.

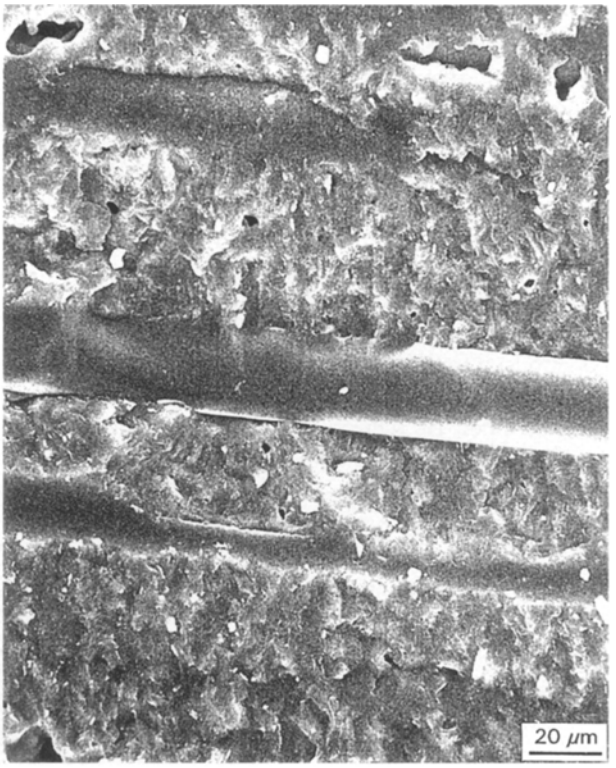

Figure 13 Scanning electron micrograph taken near the end of the fracture surface of a Nicalon/CAS-II double torsion specimen tested at 1000 °C in air.

down to under 38 J m^{-2} as the temperature increases from room temperature to 800 or 1000 °C.

The fracture surfaces of the DT specimens at 1000 °C were examined and found to have three distinct regions, as shown in Fig. 11. The fracture surface near the notch tip was planar, with no noticeable fibre pull-out (Fig. 12). Crack propagation was mainly through the fibre–matrix interface, breaking the fibres bridging the crack surface but without pulling the broken fibres out of the matrix. The profile of the planar fracture surface front was curved and extended further along the bottom face of the double torsion specimen. Also, the planar fracture extended only a little more than half way through the thickness from the bottom side of the specimen.

The end of the fracture surface was again planar without any fibre pull-out to a distance of about 1.5 mm from the cut end of the specimen. However, the fracture process was more brittle and the cracking was through the matrix, interphase and also through the fibres, as shown in Fig. 13.

The rest of the fracture surface was rough, with debonded loose fibres (Fig. 14), similar to fracture surfaces at room temperature.

Microdebonding measurements were performed at the end of the specimen at specific positions along the fibre direction below the cut surface (sketched in Fig. 15). The surface had been exposed to high-temperature air before, during and after the fracture (for over 10 h). The purpose was to investigate the effect of oxidation penetration from the exposed fibre ends. The tests were performed in the central region of the sample (i.e. away from the fracture surface and the other exposed edges).

A significant increase in the interfacial bond strength was observed at depths up to 1.4 mm. The bond strengths were extremely high and either the tested fibres cracked before being debonded or the bond strength exceeded the measurement capacity of the microdebonding apparatus (> 1000 MPa). At a depth of about 1.8 mm beneath the surface, the bond strength reverted to that of the unexposed material

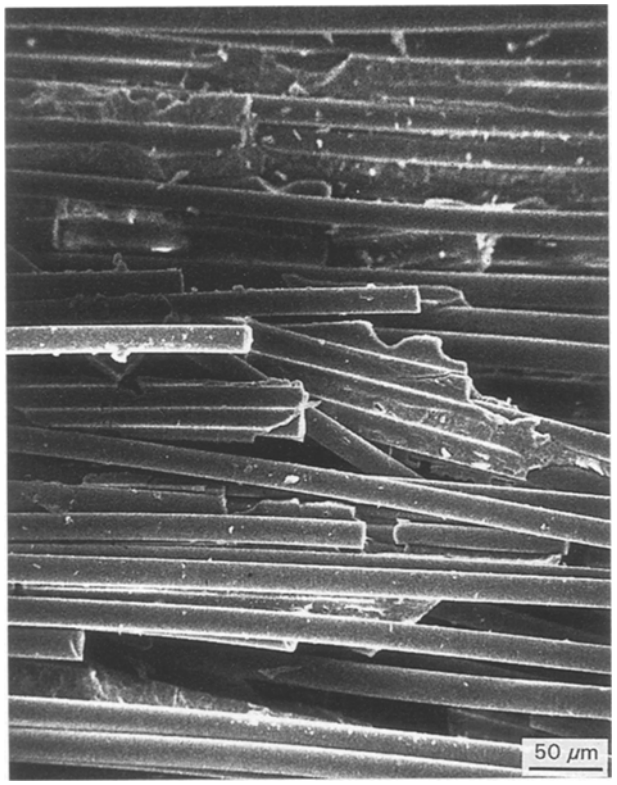

Figure 14 Scanning electron micrograph taken on the rough part (with debonded fibres) in the second half of the fracture surface of a Nicalon/CAS-II double torsion specimen tested at 1000 °C in air.

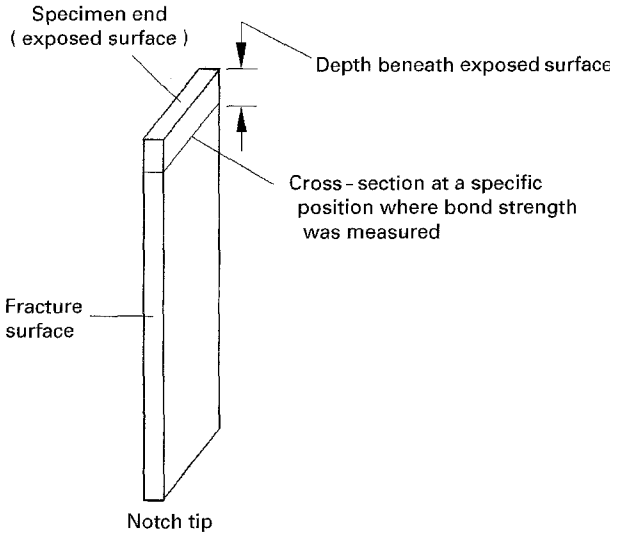

Figure 15 Diagram showing composite specimen fractured in air, test duration: for 10 h at 1000 °C, showing where the microdebonding measurements were performed.

and the oxidation process was apparently sealed off. So, the brittle fracture at the end of the specimen (fractured in air for 10 h at 1000 °C) to a depth of about 1.5 mm corresponds to the oxidation penetration depth along the fibre–matrix interface from the cut fibre ends which intersect the composite surface.

The effect of exposure time on the oxidation penetration depth (along the fibre axis) from the exposed fibre ends in Nicalon/CAS-II at 1000 °C was investigated by determining fibre–matrix bond strength variations at specific depths below an oxidized surface for

TABLE I Fibre–matrix bond-strength measurements on Nicalon/CAS-II samples, exposed to air at 1000 °C for several exposure times, at various depths below the exposed surface

Exposure time (h)	Depth beneath exposed surface (mm)	Fibre–matrix bond strength (MPa)
1	0.3	> 1245[a]
	1.0	433 ± 30
5	1.0	> 1300[a]
	1.4	412 ± 26
10	0.3	> 1275[a]
	1.0	> 1250[a]
	1.4	> 1220[a]
	1.8	408 ± 41
50	1.8	400 ± 32

[a] Bond strengths exceeded the measurement capacity of the microdebonding apparatus.

exposure times of 1, 5, 10 and 50 h. The bond-strength results are compiled in Table I.

The longitudinal oxidation penetration depth increased considerably for a 10 h exposure to air at 1000 °C (1.4–1.8 mm deep) relative to an exposure of 1 h (0.3–1.0 mm deep). Increase of total oxygen penetration with exposure time occurs for exposure times up to about 10 h. However, longer exposure times do not appear to enhance embrittlement. The oxidation penetration depth following 50 h exposure was not significantly greater than that of 10 h exposure. Creation of another interfacial region (formation of a silica bond between the fibre and the matrix [14]), filling in the gap between the fibre and matrix caused by oxidative removal of the carbon layer, is believed to thwart further oxidation down the walls of the fibres [15].

As noted, high-temperature oxidation of the fibre–matrix interphase causes a planar fracture during crack propagation parallel to the fibres, away from the cut ends. This might be occurring after the crack forms, so that cracking does not propagate through the fibres (Fig. 12), contrary to the fracture close to the cut specimen end at which cracking propagates through the fibres as well as through the matrix and the interphase (Fig. 13). This was also the case for the cracking in 90° plies during tensile testing of cross-ply composites at 800 °C.

Microdebonding tests were also performed along the crack on cross-sections of double torsion specimens fractured at temperatures from 20–1000 °C (1–2 h long tests). One such cross-section is shown in Fig. 16. The fibres bridging the crack surface caused the two arms of the double torsion specimens to remain intact even after the failure. The crack is more opened on the bottom side of the specimen and less opened on the top side (due to the nature of the loading [20, 22, 23]). Only the fibres very close (within less than a fibre diameter distance) to the crack were tested.

Fibre–matrix bond strength distributions along the crack between the two (top and bottom) surfaces of the DT specimens tested at temperatures of 20, 600, 800 and 1000 °C are given in Fig. 17. No significant change in fibre–matrix bond strength was

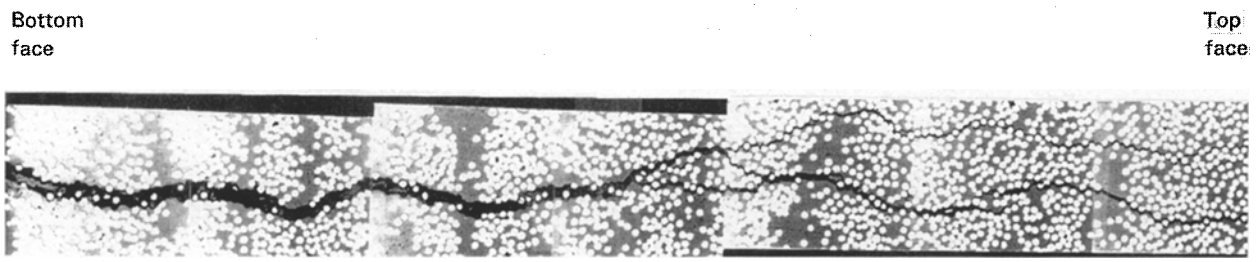

Figure 16 Cross-section of a Nicalon/CAS-II double torsion specimen fractured at 800 °C.

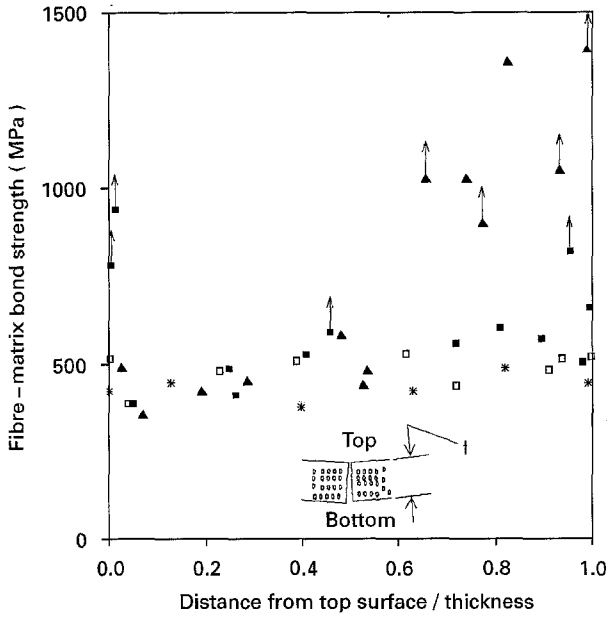

Figure 17 Interfacial bond strength distribution along the crack from top to bottom face for DT specimens fractured (1–2 h long tests) at different temperatures (bond strengths with arrows exceeded the measurement capacity of the apparatus). (*) 20 °C, (□) 600 °C, (■) 800 °C, (▲) 1000 °C.

observed from 20–600 °C. The most significant increase in the interfacial strength was observed at 1000 °C. However, a critical observation is that oxidation was effective only half way across the fracture surface from the bottom side, while the bond strengths on the other half were similar to those of control composites (except for the oxidized fibres very close to the top surface exposed to the environment). The results for the fracture at 800 °C were similar in nature but with lower increases in bond strength.

The explanation for this selective oxidation may be drawn from the micrograph of the crack in Fig. 16. The crack is tightly closed near the top side. Crack opening near the top side is not more than a few micrometers even after handling between the test and the mounting of the sample (which was cut from the cracked DT specimen). The crack opening, which was probably much smaller than that during and after the failure of the DT specimen, might not have allowed air to penetrate through to the top side.

This finding might also be the reason that the fracture surface close to the top side of the double torsion specimen seems more rough with debonded fibres, just as in room-temperature tests.

The effect of high-temperature interphase oxidation on cracking parallel to the fibres close to the exposed edges in Nicalon/CAS-II is evident by propagation of cracks through fibres. Cracking through fibres, not just through the interphase and/or matrix, is an indication of a significant increase in the interfacial bond strength. The penetration of oxygen through the edges results in the high bond strength along the fibres, inward from their exposed ends.

Away from the exposed edges, cracks do not propagate through fibres. This suggests that cracks grow in the absence of interphase oxidation in the interior of the composite. However, permeation of oxygen through the crack, after it initiates, seems to cause the oxidation of the interphase, again increasing the fibre–matrix bond strength. The crack then opens, breaking the fibres bridging the crack surface without pulling them out of the matrix. The increased interfacial bond strength results in a planar fracture surface without noticeable fibre pull-out, lowering the transverse fracture toughness of the composite. This is evident near the notch, along the more open side (Figs 11 and 12). This process is test-time dependent, i.e. the slower the fracture (test) the more effective the oxidation process (the wider the planar part).

The decrease in the transverse composite fracture toughness at 1000 °C might also be caused by lower matrix fracture toughness at 1000 °C relative to that at room temperature [41].

The possible occurrence of high-temperature stress corrosion crack growth was also explored by performing two double torsion tests holding the load constant, to give G_I of about 20 and 25 J m^{-2} for about 3 h at 800 °C. Examination of the tested specimens under the light microscope did not reveal any crack growth. Cracks grow only when G_I is close to the critical value, G_{Ic}, indicating that no significant stress corrosion crack growth parallel to the fibres is observed in the Nicalon/CAS-II composite at lower G_I, at least for the experiments carried out in this study.

Some environmental stress cracking might be expected by oxidative removal of the interfacial carbon layer at high temperatures. However, this seems to be prevented by subsequent replacement of the oxidized carbon layer with a new interfacial phase closing the gap between fibre and matrix, and sealing the oxidation process off, as described elsewhere [10].

Cracking parallel to the fibres in unidirectional Nicalon/CAS-II simulates 90° ply cracking in 0/90 laminates (discussed earlier). In both cases, crack initiation occurs without any significant effect of

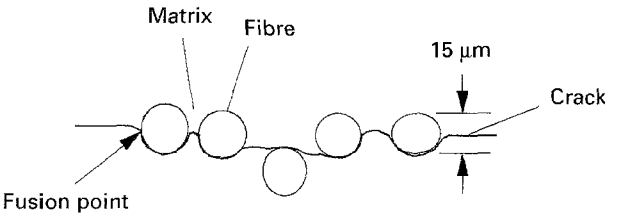

Figure 18 Diagram showing the contacts between fibres and matrix along the fibre axis as the fibres are slightly extracted from the matrix.

high-temperature interphase oxidation in the interior of the composite. Cracks propagate through the interphase and matrix, but not through the fibres.

Tensile and double torsion test results show that sufficient oxygen for interphase oxidation does not penetrate into cracks running parallel to the fibres. As shown in Fig. 6, the cracks grow predominantly in the fibre–matrix interface. Fig. 18 illustrates that contacts between fibres and matrix must exist along the fibre axis as the 15 μm diameter fibres are only slightly extracted from the matrix. These points would be expected to oxidize and fuse near the exposed edges, greatly limiting the ingress of oxygen, much as the oxidation tunnelling process along exposed fibres was shown to seal-off.

Interphase oxidation and rebonding become effective after the crack opens sufficiently for the oxidative environment to permeate, preventing fibres from pulling out of the matrix during further opening of the crack. This results in a planar fracture without a significant amount of loose (debonded) fibres on the surface, even in the absence of crack propagation through fibres.

Cracking along the fibres in unidirectional Nicalon/CAS-II near the exposed edges (within the oxidation penetration depth from the exposed edge) is also expected to be similar to 90° ply cracking in cross-ply laminates at similar positions. Cracks are expected to propagate not just through the interphase and matrix, but also through the fibres in this oxidation region. This was shown, above, to be the case in cracking parallel to the fibres in unidirectional laminates. However, badly damaged fracture surfaces of 0/90 composite tensile specimens made it impossible to explore the propagation of cracks near the edges of 90° plies in these laminates.

4. Conclusion

The results of this study provide a more complete picture than previously existed of the oxidation embrittlement process in Nicalon/CAS-II composites. As generally accepted, the oxidation embrittlement process is ultimately the result of the oxidation of the carbon interphase between the fibres and the matrix, followed by the formation of a strong bond (probably a silica bond) in place of the carbon. The bond-strength data developed in this work support this already well-established view. When the bond strength exceeds a certain value in the range of 600 MPa, matrix cracks in 0° plies then can penetrate through the fibres, yielding a material nearly as brittle as the ceramic matrix without fibres.

Tensile testing at 1000 °C in air lowers the longitudinal unidirectional strength to the stress level at which matrix cracking began to occur. Interfaces exposed along matrix cracks increase the bond strength, resulting in brittle composite fracture. The strength of cross-plied composites is also severely reduced in 800 °C air. Transverse plies crack prior to 0° ply matrix cracking. However, embrittlement does not occur until the matrix in the 0° plies cracks.

Interphase oxidation does not appear to play a significant role in crack growth parallel to the fibres of transverse plies except near exposed edges. Oxygen does not appear to have penetrated the transverse cracks except directly on the specimen fracture surface. Evidence for this was lack of bond-strength changes along the transverse ply cracks.

Transverse fracture toughness, G_{Ic}, of unidirectional materials does decrease moderately with increasing temperature (as does the bulk matrix), but no evidence of an interphase oxidizing effect on crack growth could be found. Cracks would not grow in the oxidizing environment at G_I values slightly below G_{Ic}, and oxidation did not occur on that part of the fracture surface which was cracked but not widely opened during the test.

These results establish that oxidation does not take part in crack growth parallel to the fibres, except adjacent to exposed edges. Neither does oxygen enter 90° ply cracks in cross-plied composites in sufficient quantity to produce oxidation embrittlement, at least up to the 0° matrix cracking strain. Matrix cracks in the 0° plies at higher strains do allow oxidation embrittlement of the 0° plies in unidirectional and cross-plied composites. Contacts between fibres and matrix along the 90° ply cracks may locally seal-off and prevent further spread of oxygen. No such contacts and possible sealing-off occur along matrix cracks in the 0° plies, and oxygen is free to spread throughout the opened matrix crack to reach the carbon interphase regions.

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