

Notch-Sensitive Fracture Behavior of a Silicon Carbide Fiber-Reinforced Glass-Ceramic at Elevated Temperatures

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The effect on high-temperature embrittlement of introducing a through-thickness notch in a multidirectional silicon carbide fiber-reinforced calcium-aluminosilicate glass-ceramic composite was investigated through tensile testing, microdebonding, and light and scanning electron microscopy techniques. The fracture mechanism of the composite changed from notch insensitive at room temperature to notch sensitive at elevated temperatures due to increased fiber-matrix bond strength caused by oxidation effects at interfaces exposed to the oxidative environment. Stress concentration and bending effects at the notch tip resulted in growth of the notch through fibers in a planar fashion covering the entire fracture surface. This was contrary to the case of an unnotched composite, for which two distinct fracture surface regions were observed as planar embrittlement zones at the periphery and fibrous at the center. Cracks in the notched composite were more closed relative to those in the unnotched one, except at the notch tip. Concentration of the stress at the notch tip increased the high-temperature embrittlement effect on the composite.

Keywords	ceramic matrix composites, embrittlement, high temperature properties, interface, notch sensitivity, oxidation

1. Introduction

In recent years, Nicalon (a polymer-derived SiC-based fiber (Ref 1, 2))-reinforced calcium-aluminosilicate glass-ceramic composite (Nicalon/CAS-II) has received considerable attention because it exhibits excellent longitudinal toughness and crack-deflecting ability at room temperature (Ref 3-5) through the development of a low-strength carbon interphase during processing (Ref 3, 6). However, reductions of strength and strain to failure (in the fiber direction) have been reported when testing in air at elevated temperatures (Ref 3, 5), probably due to strengthening of the fiber-matrix bond (Ref 3) caused by oxidation effects at interfaces exposed to the oxidative environment (Ref 7-9).

The performance of Nicalon/CAS-II composite laminates with notches is important because composite components need to be joined to other components in most engineering applications. Stress concentrations arise at these attachments and dominate the design and reliability. Previous studies have concentrated on the unnotched behavior of this composite, notably on the problem of matrix cracking and the associated mechanics of fiber bridging (Ref 3, 10) . The objective of this study was to investigate the high-temperature performance of multidirectional (cross-ply) Nicalon/CAS-II composite (notched vs. unnotched) through microdebonding and microscopic analysis of the fracture surfaces in addition to characterization of mechanical properties.

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2. Experimental Procedure

The materials used in this study were cross-ply ($[0/90]_{8s}$) Nicalon-fiber/CAS-II composites. They were supplied by Corning, Inc., in the form of 3 mm thick square plates.

A cold-grip test configuration using adhesively bonded aluminum tabs (Ref 3, 5) was used for tensile testing. Unnotched specimens were prepared by machining coupons of 150 by 10 by 2.8 mm from Corning-supplied composite plates. A schematic of them is shown in Fig. 1. Notched specimens, on the other hand, were prepared by introducing a 10 mm wide through-thickness notch into one side of the 150 by 10 by 2.8 mm coupons. Aluminum tabs with a 15° taper were bonded to the ends. Tests were conducted on an Instron servoelectric universal testing machine with hydraulic grips and a slot-type furnace mounted on rails. Strain measurement was provided by an Instron capacitance extensometer.

Fiber-matrix bond strength measurements were performed using the indentation technique, where individual fibers are compressively loaded on a polished surface to produce debonding (Ref 3, 11) . The microdebonding technique was coupled with light and electron microscopic analysis of the fracture surfaces to build a more complete picture of the high-temperature notch sensitive fracture behavior of the cross-ply Nicalon/CAS-II system.

3. Results and Discussion

Figure 2 shows the stress-strain curves for unnotched and notched cross-ply Nicalon/CAS-II obtained at room temperature and at 800 °C. The general features of the curve for the unnotched specimen tested at room temperature (curve a) were as expected. The material was linear elastic at up to about 0.05% strain, at which point the failure of transverse (90°) plies resulted in a decrease in the elastic modulus of the composite. At about 0.15% strain, the material deviated from linearity due to matrix cracking in the longitudinal (0°) plies (Ref 5, 10). Then the material again approached linear behavior governed by fiber properties in the 0° plies until failure at about 0.65% strain.

A fibrous, tough composite fracture was achieved with extensive matrix cracking in both 0° and 90° plies. Most cracks in 90° plies propagated into the 0° plies with fiber bridging (Fig. 3). The strong and tough behavior of the cross-ply Nicalon/CAS-II composite was due to its low fiber-matrix bond strength (about 430 MPa, as shown in Table 1), which produced debonding during matrix cracking perpendicular to the fibers, preventing the crack from propagating through the fibers in 0° plies (Ref 3, 10).

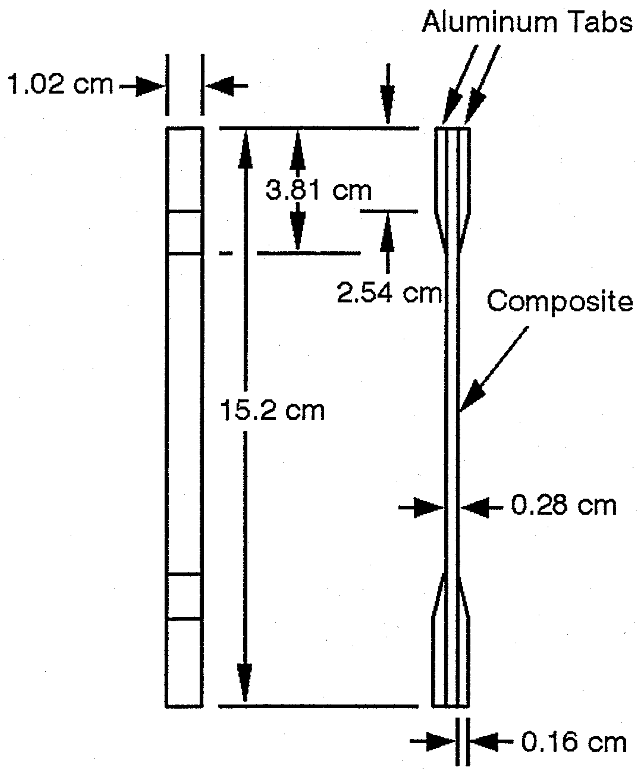

Fig. 1 Schematic of the unnotched tensile specimen. Source: Ref 12

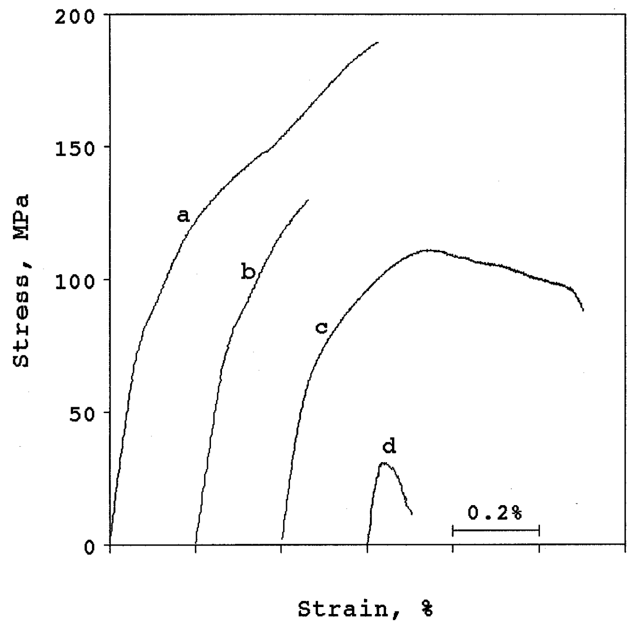

Fig. 2 Tensile stress-strain curves of cross-ply Nicalon/CAS-II composites. (a) Unnotched, 20 °C. (b) Unnotched, 800 °C. (c) Notched, 20 °C. (d) Notched, 800 °C

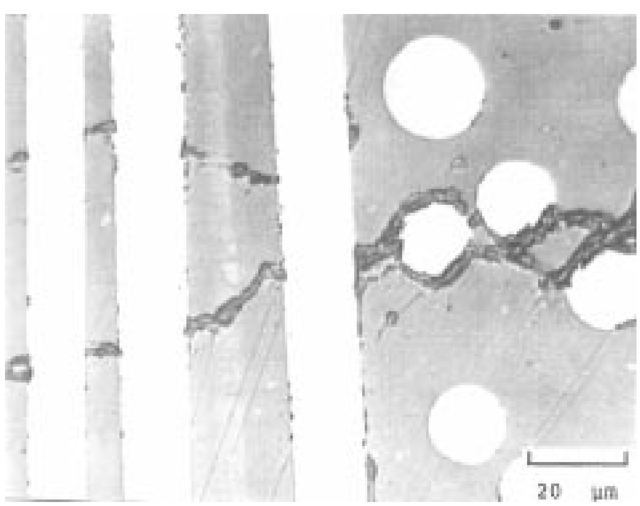

Fig. 3 A crack in a cross-ply Nicalon/CAS-II tensile specimen tested at room temperature. Cracking was perpendicular to the direction of loading.

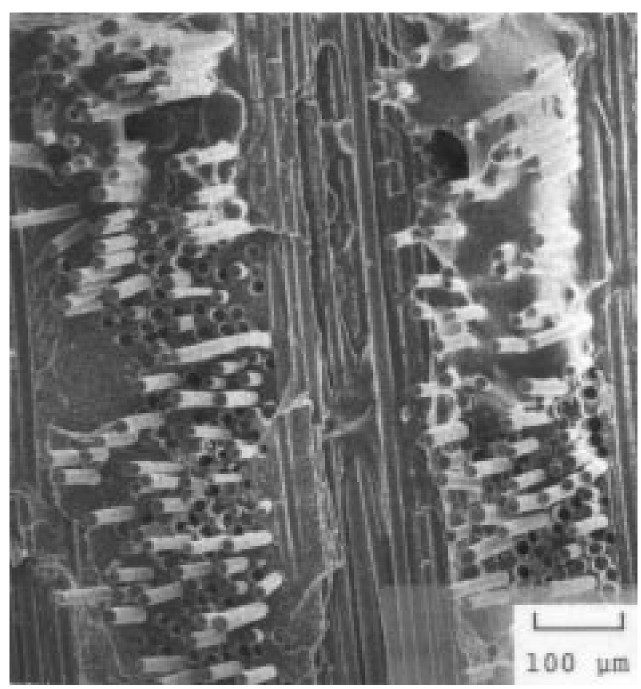

Fig. 4 Fracture surface of a notched cross-ply Nicalon/CAS-II tensile specimen tested at room temperature

Table 1 Microdebonding test results for a cross-ply [0/90] Nicalon/CAS-II sample, as-received

Test	P, mN	d_f, mm	t_m, mm	τ_P, MPa	τ_T, MPa	$\tau_d = \tau_P + \tau_T$, MPa
1	285	14.2	1.0	508	–24.9	483
2	264	14.5	0.9	450	–24.9	425
3	254	13.7	1.9	476	–25.7	450
4	268	14.8	1.5	435	–25.2	410
5	287	15.0	2.6	444	–26.1	418
6	289	15.2	1.8	443	–25.4	418
7	276	14.5	1.4	468	–25.1	443
8	297	15.5	0.9	444	–24.9	419
9	272	15.3	0.5	416	–24.9	391
10	287	14.9	0.7	464	–24.9	439
Average magnitude				455	–25.2	430
Standard deviation				24	0.4	24

P, force at debond initiation (debonding force); d_f, fiber diameter; t_m, matrix thickness (the distance from the fiber tested to the nearest fiber); τ_P, maximum interfacial shear stress created by P; τ_T, residual interfacial shear stress due to ΔT (the difference between the solidification temperature, (1256 °C), and 20 °C at the position of τ_P); τ_d, debonding shear stress (fiber-matrix bond strength). Source: Ref 12

Table 2 Microdebonding tests on fibers within a fiber diameter distance to the 90° ply cracks away from the exposed surfaces of the notched composite tested at 800 °C

Test	P, mN	d_f, mm	t_m, mm	τ_P, MPa	τ_T, MPa	$\tau_d = \tau_P + \tau_T$, MPa
1	392	17.3	5.8	430	–28	402
2	392	16.2	2.2	526	–26	500
3	265	15.4	1.5	398	–25	373
4	333	14.5	3.0	545	–26	519
5	441	16.9	3.7	529	–27	502
6	363	16.5	5.5	437	–28	409
7	367	15.1	2.8	557	–26	531
8	377	15.8	2.1	532	–26	506
9	435	16.7	3.4	537	–26	511
10	279	15.9	1.2	597	–25	372
Average magnitude				489	–26	462
Standard deviation				61	1	61

P, force at debond initiation (debonding force); d_f, fiber diameter; t_m, matrix thickness (the distance from the fiber tested to the nearest fiber); τ_P, maximum interfacial shear stress created by P; τ_T, residual interfacial shear stress due to ΔT (the difference between the solidification temperature, (1256 °C), and 20 °C at the position of τ_P); τ_d, debonding shear stress (fiber-matrix bond strength).

When the unnotched Nicalon/CAS-II composite was tested at 800 °C, reductions in strength and ultimate strain were observed (Fig. 1, curve b) due to interphase oxidation and subsequent silica bond formation (Ref 7-9), which raised the fiber-matrix bond strength to a level (over 800 MPa) where matrix cracks in the 0° plies propagated through the fibers. Noticeable deviation from linearity observed just before fracture was an indication of some matrix cracking in the 0° plies, as confirmed by microscopic examination of the specimen edges after the test. When the fracture surface of the specimen was examined, two distinct regions were observed, planar embrittlement zones at the periphery and a fibrous center (Ref 12).

The stress-strain curve for the notched specimen tested at room temperature is shown in Fig. 2, curve c. A tough composite fracture was achieved with extensive fiber pull-out (Fig. 4), similar to that of the unnotched specimen tested at room temperature. Although the matrix cracking was extensive, the cracks were not as open as in the case of the unnotched specimen, as shown in Fig. 5, especially in regions away from the notch. The ultimate strain of the composite was not different either. However, the strength of the composite was of course significantly lower than in the unnotched case, due to stress concentration effects at the notch tip.

Testing the notched Nicalon/CAS-II specimen at 800 °C reduced both the strength and the ultimate strain of the composite significantly (Fig. 2, curve d). When the specimen edges were examined, no matrix cracking in the 0° plies was observed. Matrix cracks of 90° were observed; however, they were tightly closed. The bond strength of the fibers next to those 90° ply cracks (Table 2) was similar to that of the control material (as supplied at room temperature), indicating that oxidation of the interphase occurs only when the cracks open sufficiently for the oxidative environment to penetrate.

The fracture surface was planar without any noticeable fiber pull-out, including the central region of the fracture surface (Fig. 6). This might be attributed to the penetration of oxidation into the interior of the composite due to the gradual growth and larger opening of the crack at the notch tip. This would give sufficient time for oxidation to spread across the cross section of the composite before the failure of the unexposed interior, contrary to the case of the unnotched specimen.

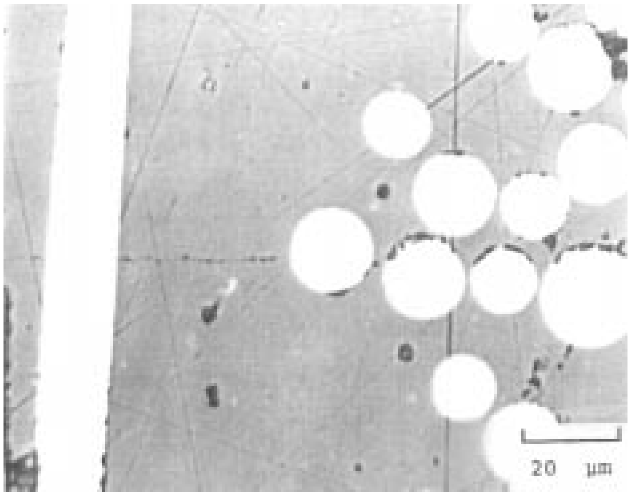

Fig. 5 A crack in a notched cross-ply Nicalon/CAS-II tensile specimen tested at room temperature

The gradual failure of the notched specimen at 800 °C (Fig. 2, curve d), in contrast to the unnotched specimen tested at the same temperature, is believed to be a result of bending effects at the notch tip. Local strain was observed to be nonuniform across the specimen, being lower at the unnotched edge of the specimen than at the notched edge. However, as mentioned above, a crack opening large enough to cause embrittlement of the composite was achieved at a much lower average tensile stress than for the unnotched composite.

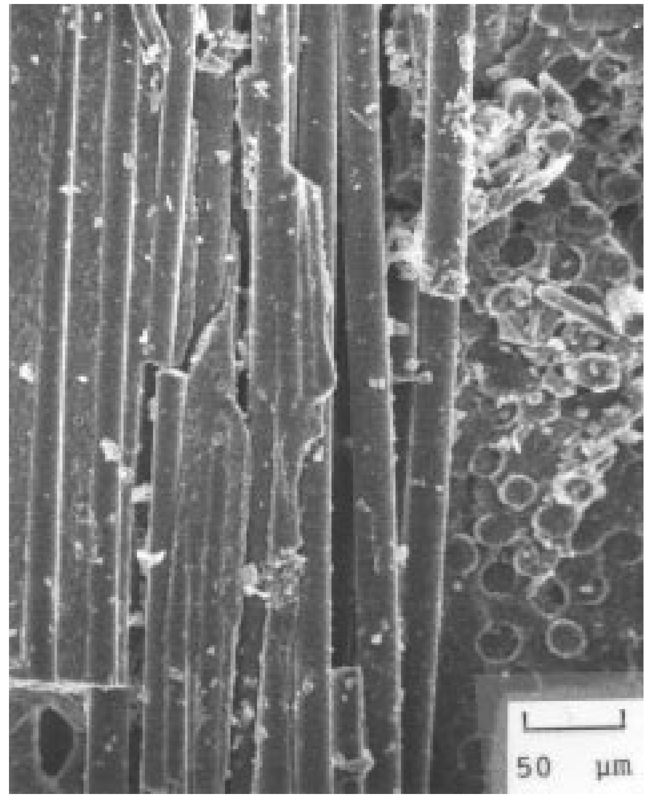

Fig. 6 Fracture surface of a notched cross-ply Nicalon/CAS-II tensile specimen tested in air at 800 °C

4. Conclusions

Although the toughness of the Nicalon/CAS-II composite is notch insensitive at room temperature, it becomes notch sensitive at 800 °C. The embrittlement process is a result of the interphase oxidation, followed by formation of a stronger silica bond between the fiber and the matrix as the crack that initiates at the notch tip opens and encounters fibers. The notch grows through the fibers in planar fashion, covering the entire fracture surface. Stress concentration effects at the notch tip appear to increase the embrittlement effect on the notched composite relative to the unnotched material. Bending effects, on the other hand, are believed to result in the gradual growth of the notch across the composite.

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