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High-performance SiC-fibre reinforced β-sialon CMCs prepared from heat-treated Nicalon fibres

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Abstract

In a previous paper (Demir, A. and Thompson, D. P., Heat-treatment of Nicalon SiC fibres in various atmospheres and pressures. In *Proc. 3rd Int. Turkish Ceramic Congress, Vol. 2, Engineering Ceramics*, ed. V. Gunay, H. Mandal and S. Ozgen. Turkish Ceramic Society, Istanbul, 1996, pp. 180–185) it was reported that a high-pressure, high-temperature carbon monoxide treatment of Nicalon SiC fibres resulted in significantly improved strength and refractoriness, and at the same time deposited a thin carbon coating on the surface of the fibres. In the present study, fibres heat-treated in this way have been used to reinforce a range of β-sialon ceramic matrices, and the resulting mechanical properties (bend strength, fracture toughness) have been compared with results for similar materials prepared from non-heat-treated Nicalon fibres. All samples showed significant increases in strength and density, with in general, not much change in fracture toughness. The presence of the coating on the fibres assisted the densification of the composites by decreasing friction during hot-pressing, thereby increasing density and matrix strength. SEM micrographs indicated that the carbon coating is still present after fabrication and is sacrificed during fracture, assisting in easy pull-out of fibres.

Keywords: C interphase; Composites; Mechanical properties; Sialons; SiC fibres

1. Introduction

A common problem with SiC fibres prepared via polycarbosilane or other pre-ceramic polymers relates to the residual oxygen left in the fibre after processing, which normally resides in the boundaries between SiC grains. The excess oxygen generates significant problems during processing and causes instability of the fibre under certain conditions. The majority of oxygen found in the final ceramic fibre comes from either oxygen introduced during the synthesis of the pre-ceramic polymers, or from oxygen interjected into the polymer in order to cross-link the fibre to make it infusible.[1–10] Yajima and co-workers described the Nicalon fibre structure as a fine-grained microcrystalline silicon carbide with excess carbon and oxygen also present.[3] Further studies suggested a fibrous structure consisting of 1–3 nm silicon carbide microcrystals mixed in with clusters of polyaromatic carbon and silica, and an intergranular oxycarbide phase. Many researchers have investigated the effects of high temperature heat-treatment on fibre strength in a variety of environments[1,9–13] and it has been shown that fibres heat-treated in inert environments (Ar, N_2, vacuum) show outgassing of volatile products whereas fibres treated in an oxidising environment (O_2, air) showed slightly less degradation. On the other hand partial pressures of carbon monoxide not only retain, but also increase strength.[9] In fact, Demir and Thompson [1] not only achieved a 50% strength increase, but also deposited a thin carbon coating on the fibres after high pressure CO heat-treatment.[2] These two points, and also the improved refractoriness after this heat-treatment, offer significant advantages for the use of Nicalon fibres to reinforce ceramic matrices at high temperatures.

The achievement of high toughness in CMCs is the major aim in fabricating these materials, and whether this is successful or not depends on the ability of rein-

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forcing fibres to inhibit crack propagation. This, in turn, depends upon mechanisms of energy absorption such as fibre debonding and frictional pullout, and a balance must be achieved between load transfer across the fibre/matrix interface and fibre debonding and slip in the region of the propagating crack.[14] Therefore, the successful achievement of high toughness in CMCs is very sensitive to the properties of the fibre/matrix interface. It might also be felt that the presence of high-strength fibres, which are well bonded to the matrix, might also improve strength. However, this depends on a number of microstructural parameters, and also, very importantly, on whether full density has been achieved during firing.

Nicalon fibres have been the subject of much research in recent years stemming from their potential as a high temperature reinforcement material.[1–20] These fibres can impart high toughness to certain glass-ceramic composites due to the in-situ formation of a carbon interface.[2] This carbon phase dramatically increases the work of fracture by promoting crack deflection, frictional debonding and fibre pull-out.[21] However, the mechanism of carbon formation is not well understood. While Nicalon fibre reinforced glasses or glass ceramics have been extensively researched, the available literature on Nicalon fibre reinforced sialon ceramics is very limited.[22] Although β-sialon has found many applications in engineering, the range of applications would be enlarged by increasing its toughness by fibre reinforcement. Because of both chemical and thermal compatibility, SiC fibres are the most suitable reinforcement for Si_3N_4 and sialon ceramics. Whilst high-purity SiC fibres can withstand high temperatures, the refractoriness of the oxygen-containing Nicalon fibres is limited and this is why most previous work has focused on their use as a reinforcement for glass matrix composites. However, in the present study, the use of CO heat-treated Nicalon fibres has enabled the higher temperatures required for the densification of β-sialon ceramics to be reached without any associated degradation of the fibres, or without fibre-matrix sticking which inhibits pull-out and decreases toughness.

2. Experimental

Nicalon SiC fibres, Type NL-207, supplied by Nippon Carbon Japan, were used as reinforcement. The properties of these fibres was improved by a 45 bar CO heat-treatment at 1600°C for 30 min in a gas pressure sintering furnace, as described previously.[1] β-sialon matrices were reinforced with both the as-supplied and the CO heat-treated fibres in order to identify the advantages imparted by the heat-treatment process. Incorporation of continuous fibres into the matrix was done using slurry infiltration The fibre yarn was fed through a slurry tank and the slurry impregnated fibre wound on to a drum, which was then partially dried. This was then cut into short segments, stacked together and cold-pressed into a green body of dimensions 20×11×45 mm. Consolidation of the green bodies was carried out by hot-pressing at 20 MPa pressure for 30 min in a specially machined graphite die, at temperatures appropriate for the particular densification additive(s) used. A protective layer of BN was used to avoid direct reaction between the carbon of the die and the sialon matrix and also to protect the sample from attack by the CO atmosphere generated by the surrounding graphite. Temperatures for composite fabrication were 50°C higher than those required for full matrix densification to take account of the negative effect of fibres on densification. After hot-pressing, samples changed dimensions to 25×5×46 mm, being not only thinner but also wider after hot-pressing. All experiments showed that widening is essential to achieve a good densification rate. The thickness of green bodies also had an effect on densification, with density decreasing as thickness increased. This behaviour could be related to fibre movement during densification. In the pressing direction, fibre friction prevents fibre movement, and unless the sample is simultaneously allowed to increase in width, fibre movement is prevented and poor densification is obtained.

Bending strength and fracture toughness of the composites were determined by three-point bending. Bar shaped specimens were cut from hot-pressed samples, and ground and polished into 3×4×45 mm bars for bend strength measurement, and notched 2×4×40 mm bars for fracture toughness measurement. All the tests were done using an Instron testing machine operating in the range 2–20 kN. The load was applied at a rate of 0.1 mm/min and the bending spans were 40 mm. At least three specimens were measured to obtain the mean value for each sample. The fracture surfaces and polished sections of the composites samples were examined using a Hitachi S-2400 Scanning Electron Microscope.

3. Effect of fibre heat-treatment on the densification of β-sialon composites

In the previous study, it was shown that 45 bar CO heat-treatment of Nicalon fibres for 30 minutes at 1600°C gave the most improved fibre properties for use in CMCs,[1] and these and the untreated fibres were incorporated into a range of β-sialon matrices as shown in Table 1. The latter have been discussed previously,[19] and correspond to either $z=1$ (Samples 1–5) or $z=3$ (Samples 6–9) compositions. The prefixes A and H have been used to distinguish as-received fibres, and those which received the 1600°C CO treatment. The main difference between the samples is in the use of different sintering aids which allowed densification to be achieved

Table 1
Starting compositions used as matrices for CO heat-treated Nicalon fibres

As-received samples	Powders	Heat-treated samples
A1	7% Y_2O_3 + 77.86% Si_3N_4 + 7.24% AlN + 7.90% Al_2O_3	H1
A2	7% Y_2O_3 + 3% MgO + 75.35% Si_3N_4 + 7.01% AlN + 7.64% Al_2O_3	H2
A3	2% MgO + 8% Sm_2O_3 + 75.35% Si_3N_4 + 7.01% AlN + 7.64% Al_2O_3	H3
A4	2% MgO + 7% Y_2O_3 + 8% Sm_2O_3 + 64.49% Si_3N_4 + 6.47% AlN + 7.05% Al_2O_3	H4
A5	3.38% Li_2O + 2.65% MgO + 18.97% SiO_2 + 75% in-house β-sialon powder ($z = 1$)	H5
A6	7% Y_2O_3 + 3% MgO + 44.93% Si_3N_4 + 14.93% AlN + 30.1% Al_2O_3	H6
A7	7% Y_2O_3 + 3% MgO + 90% PP[a] β-sialon powder ($z = 3$)	H7
A8	25% LAS Nitrogen glass + 75% PP β-sialon powder ($z = 3$)	H8
A9	3.38% Li_2O + 2.65% MgO + 18.97% SiO_2 + 75% PP β-sialon powder ($z = 3$)	H9

[a] PP, pre-prepared. LAS, lithium aluminosilicate.

at different temperatures. For the first sample (A1) 7% Y_2O_3 was used as the only sintering aid and this required 1750°C for complete densification. To lower the densification temperature to 1600°C either 3% MgO or 8% Sm_2O_3 was used (Samples 2–3). Sample 4, with all three additives present, allowed a further reduction of the hot-pressing temperature to 1550°C, and this was the lowest densification temperature which could be obtained without the use of Li_2O additions. In Sample 5, in-house $z = 1$ preprepared β-sialon powder was used rather than a mix of binary oxide/nitride starting powders. Of the $z = 3$ samples, Sample 6 used a β-sialon mix of binary oxides/nitrides, whereas Samples 7–9 used a commercial preprepared β-sialon powder [supplied by International Syalons (Newcastle) Ltd] but with different additives present to vary the densification temperature.

Table 2 and Fig. 1 show densification behaviour for both as-received and CO heat-treated SiC fibre reinforced β-sialon composites. Broadly speaking, a higher densification was obtained with the CO heat-treated fibres. Surprisingly heat-treatment of the fibres affected densification rate, although the compositions and hot-pressing conditions were the same. This was most probably because of the carbon coating on the heat-treated fibres. The coating eases fibre movement during hot-pressing by decreasing frictional forces between the fibres and the matrix or by deformation due to the weak and brittle nature of the coating.

The best densification (99% of theoretical density) was achieved in Samples H1 and H6, and the worst densification (85% of theoretical), was achieved in sample A5. While for Samples H3, H4 and H9 significant density differences were observed between as-received and heat-treated fibre composites, for other samples only small density differences were observed. The biggest difference occurred between Samples A3 and H3 with densities of 91 and 98% of theoretical density respectively. Although Sample A5 showed 99% of theoretical density for small samples hot-pressed at 30 MPa pressure, for large samples hot-pressed at 20 MPa pressure only 85% of theoretical density was obtained. Heat treatment of the fibre did not change the extent of

Table 2
Hot-pressing of as-received and heat-treated SiC fibre/β-sialon composites

Sample	Temperature (°C)	Time (min)	Pressure (MPa)	Density (gcm^{-3})	T.D. (%)
A1	1750	30	20	2.95	97
H1	1750	30	20	3.00	99
A2	1600	30	20	2.84	98
H2	1600	30	20	2.72	95
A3	1600	30	20	2.62	91
H3	1600	30	20	2.85	98
A4	1550	30	20	2.67	90
H4	1550	30	20	2.82	95
A5	1200	30	20	2.41	85
H5	1200	30	20	2.50	89
A6	1550	30	20	2.91	98
H6	1550	30	20	2.93	99
A7	1550	30	20	2.90	97
H7	1550	30	20	2.91	98
A8	1500	30	20	2.74	95
H8	1500	30	20	2.79	97
A9	1450	30	20	2.55	94
H9	1450	30	20	2.69	98

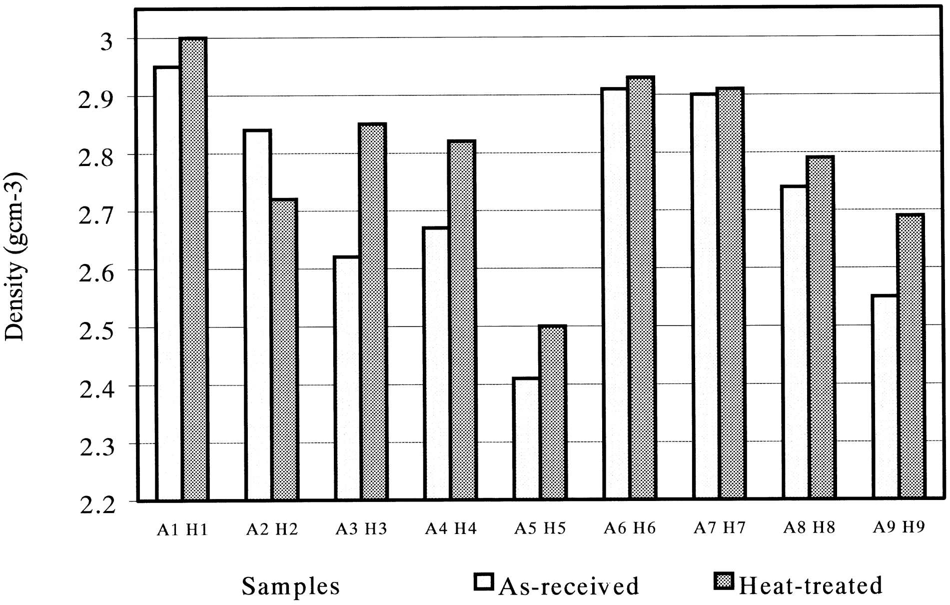


Fig. 1. Densification behaviour of as-received and heat-treated fibre composites.

densification either. Both A6 and H6 showed good densification, probably because of the large amount of Al_2O_3 present. Samples A2 and H2 were unusual in showing an inverse density behaviour. Most probably the thickness of the green body of the H2 sample was larger than that of A2 and this led to worse densification, Therefore heat-treatment of the fibre did not have as large an effect as in other samples. The matrix in all samples was essentially β-sialon, with the exception of samples A4/H4 which retained some 50% of α-Si_3N_4 and Samples A2/H2/A3/H3 which retained approximately 5% of α-Si_3N_4.

4. Effect of using CO heat-treated fibres

The fracture behaviour and strength of the nine sets of samples prepared in the last section were measured by three point bend testing as described previously. After hot-pressing, three bars were cut from each hot-pressed sample for three-point bend testing and three others for fracture toughness measurement. The strength test samples were ground and polished into 3×4×46 mm bars, and the fracture toughness samples to a final size of 2×4×46 mm with a 2 mm deep notch. However, the polishing of these samples was difficult because of the residual porosity. Therefore the results show considerable variation, due to varying relative sizes of critical flaws. The load/displacement curve was drawn and a mean strength calculated. Table 3 shows the bending strength and fracture toughness results for both as-received and heat-treated samples. For bending strength, most samples showed an enormous strength increase between the as-received and heat-treated fibre state whereas for fracture toughness less significant changes were observed. The strength increase can also be clearly seen in Fig. 2. Without exception, all samples showed a strength increase when the heat-treated fibre was used.

The maximum measured strength of 687 MPa was achieved for sample H3 which is an $MgO + Sm_2O_3$ densified β-sialon composite, and when compared with Sample A2 has almost twice the strength. This difference can also be observed in Fig. 3(a) and (b). Crack initiation started at about 0.18 kN and the load sharply

Table 3
Mechanical properties of as-received and heat-treated SiC fibre/β-sialon composites

Sample	Temperature (°C)	T.D. (%)	Bending strength[a] (MPa)	Fracture toughness* (MPa $m^{1/2}$)
A1	1750	97	–	–
H1	1750	99	–	–
A2	1600	98	381±21	635±0.8
H2	1600	85	585±35	7.28±0.6
A3	1600	91	349±31	11.71±1.8
H3	**1600**	**98**	**687±52**	**11.04±1.5**
A4	1550	90	375±112	10.66±2.1
H4	**1550**	**95**	**623±53**	**13.36±1.8**
A5	1200	85	251±20	4.58±0.1
H5	1200	89	254±11	4.52±0.1
A6	1550	98	419±55	11.93±1.9
H6	**1550**	**99**	**627±25**	**11.87±0.7**
A7	1550	97	399±44	6.14±1.5
H7	1550	98	467±31	10.18±1.2
A8	1500	95	268±12	5.37±1.9
H8	1500	97	361±8	6.29±1.2
A9	1450	94	408±32	11.03±2.5
H9	**1450**	**98**	**512.±24**	**10.50±0.4**

[a] Average value of three samples measured.

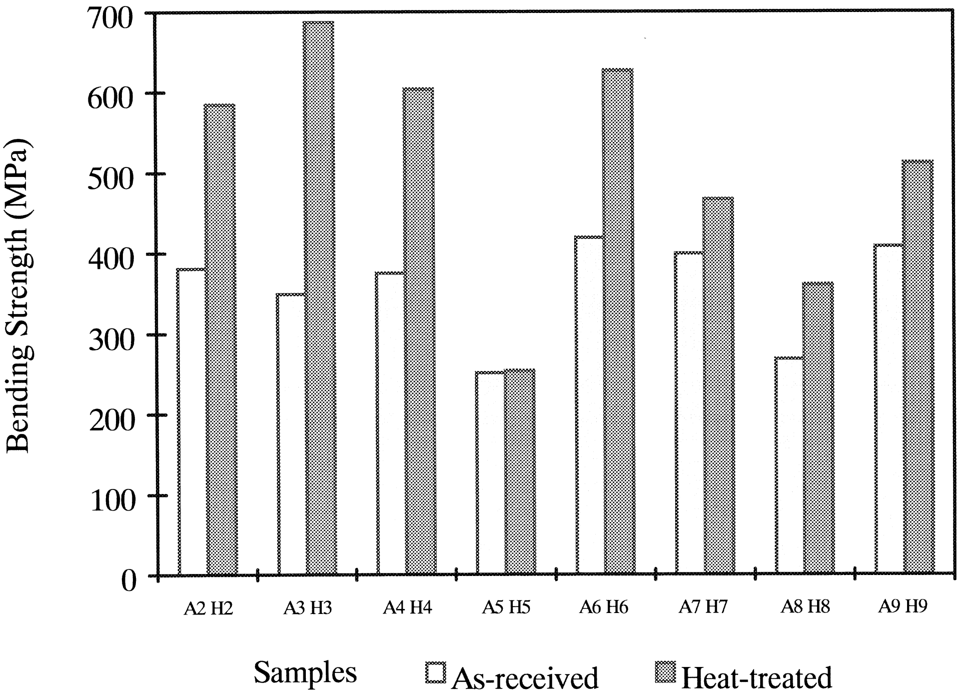


Fig. 2. Strength values for β-sialon composites before and after CO heat-treatment of the fibres.

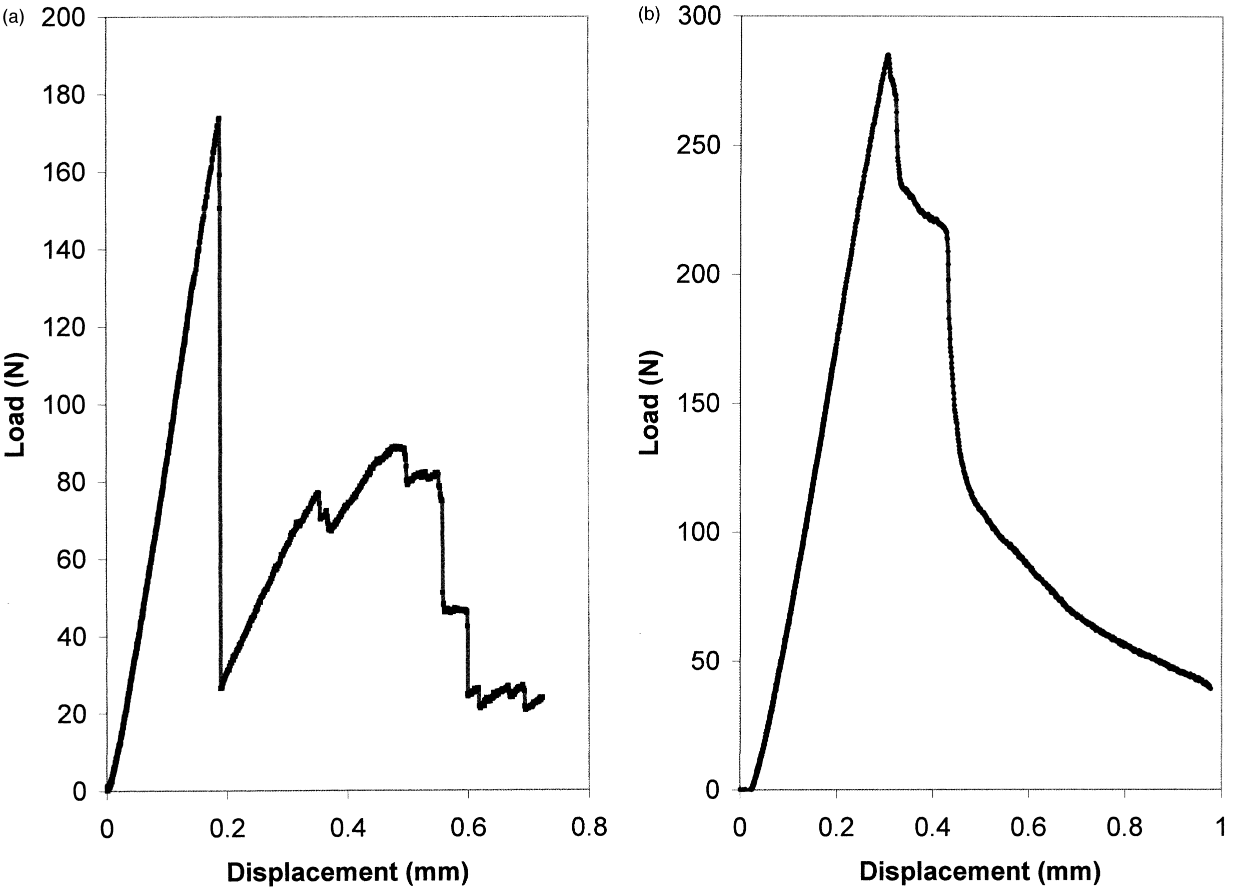


Fig. 3. Comparison of fracture behaviour of as-received and heat-treated fibre composites, (a) Sample A3 and (b) Sample H3.

dropped to 0.03 kN for Sample A3 whereas for sample H3, crack initiation started at about 0.3 kN and the load was transferred to the fibres at 0.25 kN, indicating successful fibre reinforcement. The second highest value of bending strength was obtained for Sample H6 (627 MPa), which is a densified $z = 3$ β-sialon composite. Even though the sintering additives are the same as Sample H2, Sample H6 densified at a lower temperature and gave a higher density possibly because of the easier densification at the higher z-value and this has given the higher strength value. When the z-value is increased along the β-sialon line, more Al_2O_3 is needed in the starting mix, and since Al_2O_3 also plays the role of a sintering additive, it lowers the densification temperature and also increases the densification rate. Although sample H6 had the higher strength, the fracture toughness of both H3 and H6 are slightly lower than the as-received fibre samples but a good strength increment is still obtained for the heat-treated fibre sample. However, it is impossible to obtain a reliable fracture toughness value based on three samples. Therefore when comparing the results for any two samples, only large differences should be noted.

For sample H4, both bending strength and fracture toughness increased, changing from 375 to 604 MPa and 10.66 to 13.36 MPa $m^{1/2}$, respectively. Both samples H4 and A4 were prepared using a $MgO + Y_2O_3 + Sm_2O_3$ densified β-sialon matrix but the improvement in the Nicalon fibres by CO pressure heat-treatment provided a significant strength and toughness increase in the final composites. Their load/displacement curves look similar, but sample H4 failed at a much higher loading [Fig. 4(a) and (b)]. The highest bending strength measured for all the pre-prepared β-sialon composites (512 MPa) was achieved for sample H9, a mixed $Li_2O + MgO + Sm_2O_3$ densified industrial $z = 3$ pre-prepared β-sialon powder. Sample H7 with 467 MPa strength and 10.18 MPa $m^{1/2}$ fracture toughness was the second best sample in this group; this sample was densified with the $MgO + Y_2O_3$ mix.

In Table 3, strength and fracture toughness samples H3, H4, H6 and H9 are highlighted. All four samples showed that toughening had been successfully achieved. After the first crack initiation none of the samples broke instantly; on the contrary, crack deflection occurred and load was transferred to the fibres (Fig. 4). Fibre bridging then occurred and fibres pulled out as the displacement increased. Eventually the fibres snapped and the samples failed. It is very clear that β-sialon matrices were successfully reinforced by the CO pressure heat treated fibres.

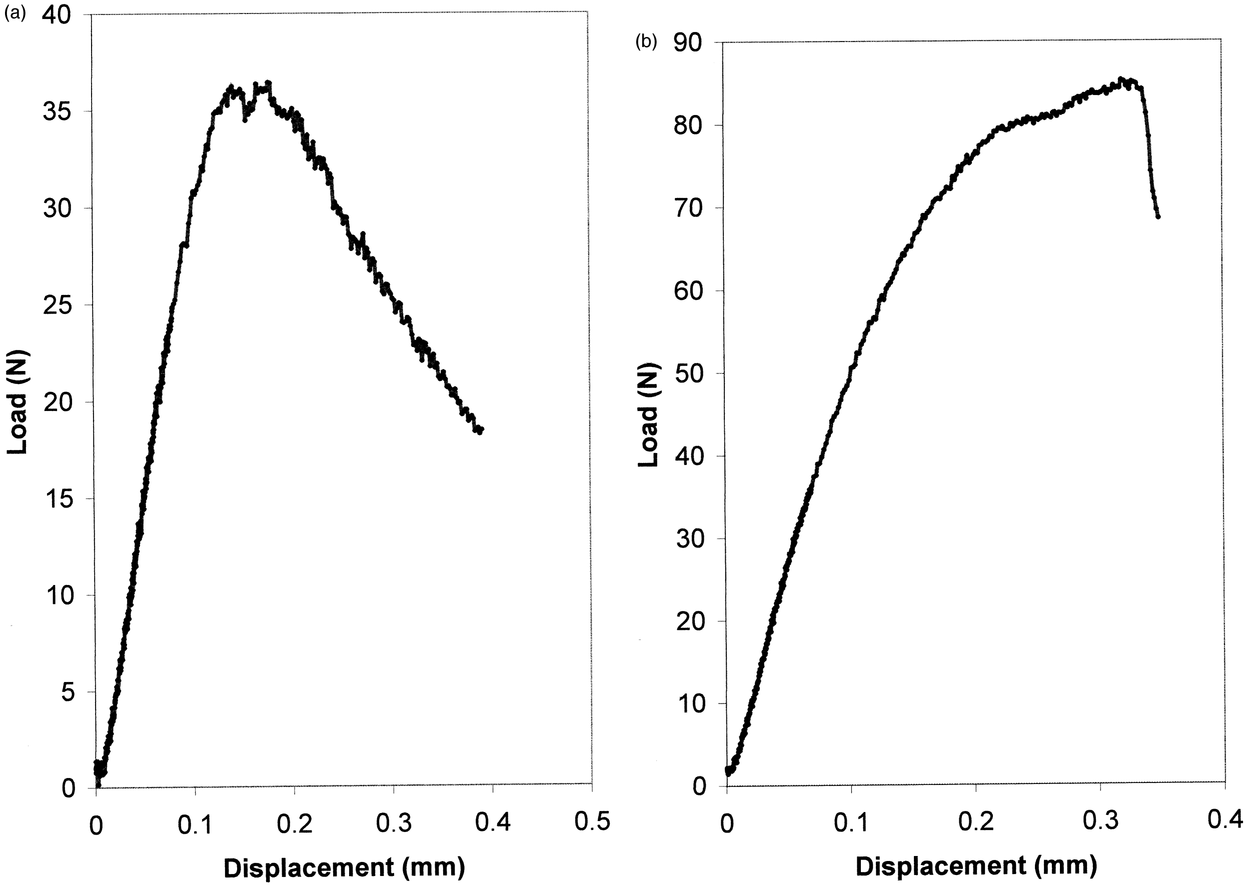
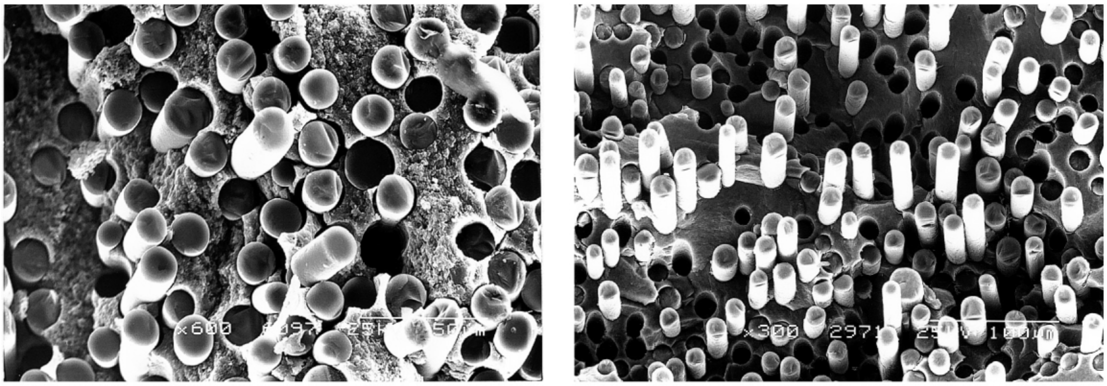
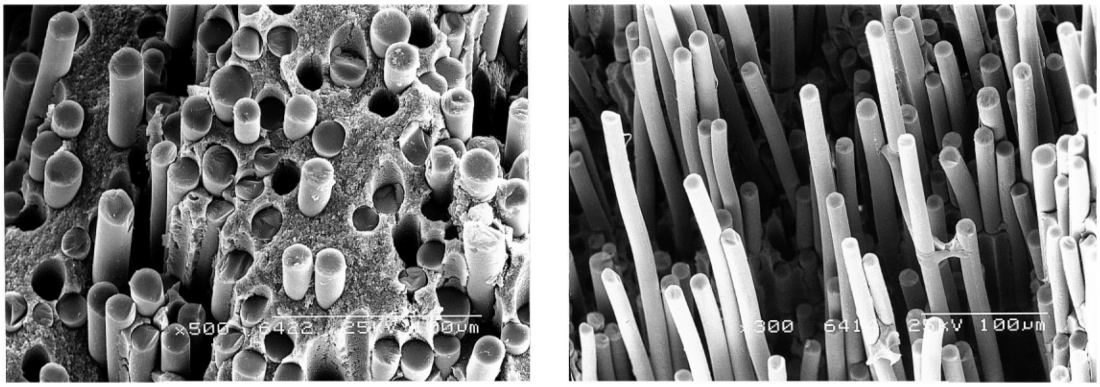


Fig. 4. Comparison of the fracture behaviour of as-received and heat-treated fibre composites, (a) Sample A4 and (b) Sample H4.

SEM images of fracture surfaces are shown in Fig. 5. It can be clearly seen that there is a correlation between the amount of fibre pullout and composite strength. Samples H3 and H4 showed massive pullout and therefore their strength ranges between 512 and 687 MPa. Samples such as A3 and A4, reinforced with as-received fibres, failed at lower loads and showed less pullout because of the higher shear strength of the fibre/matrix interface. The fibre/matrix ratio in all samples was about 40% and the fibre distribution was not entirely uniform (Fig. 5). On the other hand, the fibre/matrix ratio in Sample H8 was about 30% and the fibre/matrix distribution was uniform. The only difference in this sample was that a pre-prepared $Li_2O/Al_2O_3/SiO_2/Si_3N_4$ glass sintering additive[18] had been used rather than mixed oxide additives. Most probably the glass powder

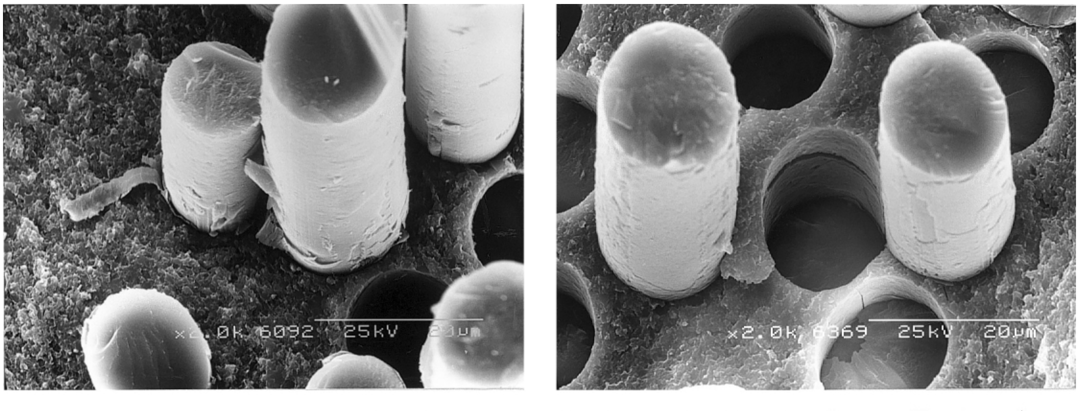


a) Sample A3 b) Sample H3



c) Sample A4 d) Sample H4

Fig. 5. Pullout in fracture surfaces of as-received and heat-treated fibre samples.



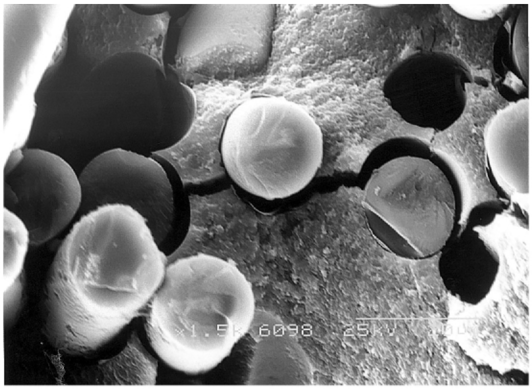
a) Sample H2, showing the sacrificed coating on the fibre surface.

b) SampleH3, showing the sacrificed coating on a "pushed out" fibre surface.

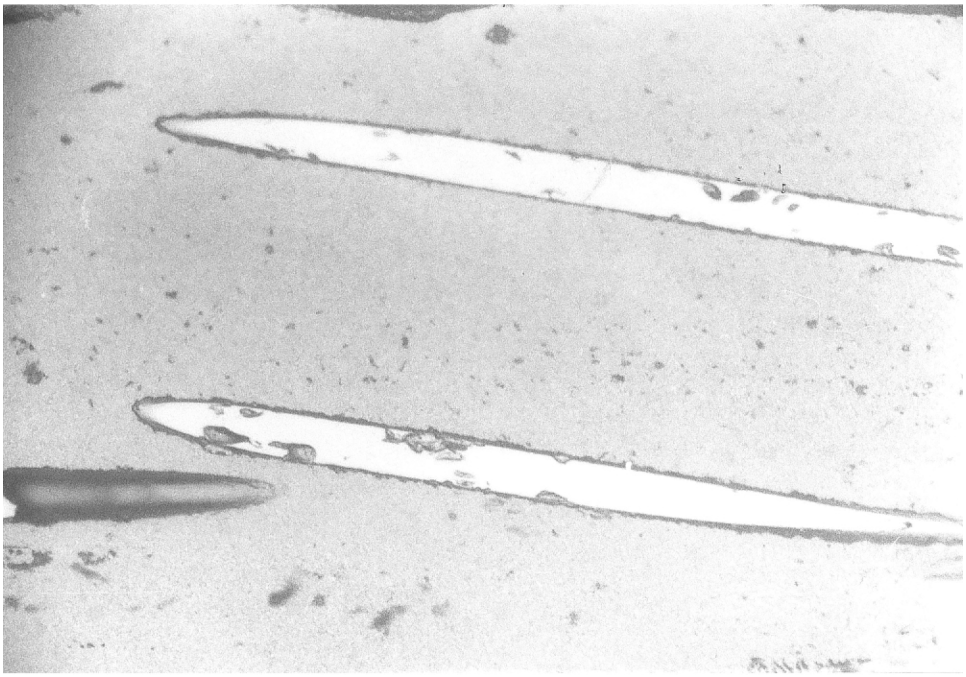
Fig. 6. Pullout at the fracture surfaces of heat-treated fibre containing samples.

played a significant role in achieving improved homogeneity during densification.

As explained above, the formation of fibre coatings during high pressure treatment significantly assisted fibre pullout during fracture. The coating was sacrificed during fracture and this allowed fibres to pull out easily as shown in Fig. 6(a). In Fig. 6(b) this situation can be seen as "pushed out" fibres, i.e. the fibres have been pushed out and the coating left in the fibre hole. The thickness of this coating can be seen from the hole wall. The coating assisted pullout, and provided crack deflection during fracture as seen in Fig. 7(a). In Fig. 7(b) an optical micrograph of a polished surface of a heat-treated fibre-reinforced sample (H3) is shown. The fibre/matrix contrast is clearly visible in this sample, and in particular, the thin black line defining the boundary between the fibre and the matrix clearly shows the carbon coating in a much more convincing way than on the SEM micrographs. Another advantage, which may be the most important benefit provided by the carbon coating, is that it prevents fibre degradation. During densification of β-sialon composites, the carbon coated heat-treated fibres did not lose their strength because the coating became a protective layer against outer and inner reactions. In particular it prevented the endogenous reaction inside the fibre by suppressing SiO and CO formation until densification had taken place. It also protected the fibre from the outer gas or glass reactions. Therefore with the heat-treated Nicalon fibres composite fabrication up to 1600°C could be achieved (Sample H3).



a) Crack deflection at fibre surfaces in Sample H2



b) Optical micrograph of the fibre-matrix interface in Sample H3

Fig. 7. SEM and optical micrographs of heat-treated fibre composite samples.

For all these heat-treated samples, the values of strength and fracture toughness obtained are remarkably good and show that reinforcement has been successfully achieved. If denser samples had been obtained, then these could have been polished more successfully, and it is believed that further improvements in strength and possibly fracture toughness, could have been obtained.

5. Conclusions

High-temperature, high-pressure CO heat-treated Nicalon fibres gave CMCs with improved properties compared with equivalent materials produced using non-heat-treated fibres. The carbon coating on the heat-treated fibres reduced frictional effects and assisted densification during hot-pressing, resulting in higher-density products with increased strength. It also prevented reactions between the liquid phase in the matrix and surface silica on the fibre, retaining a fibre/matrix interface which could still promote good pull-out behaviour. The improved refractoriness of the heat-treated fibres allowed hot-pressing temperature of up to 1600°C to be used, without any apparent degradation of fibre strength. A slightly puzzling feature of the results was that fracture toughness values were not very different for samples prepared using the heat-treated fibres. In general, these were similar to those for samples prepared using un heat-treated fibres, but varied quite significantly and apparently inexplicably from one sample to another. Further work is needed to identify the exact reason for the variation in K_{Ic} behaviour observed.

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