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Effects of HF treatments on tensile strength of Hi-Nicalon fibres

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Tensile strengths of as-received Hi-Nicalon fibres and those having a dual BN–SiC surface coating, deposited by chemical vapour deposition, have been measured at room temperature. These fibres were also treated with HF for 24 h followed by tensile strength measurements. Strengths of uncoated and BN–SiC coated Hi-Nicalon fibres extracted from celsian matrix composites, by dissolving away the matrix in HF for 24 h, were also determined. The average tensile strength of uncoated Hi-Nicalon was 3.19 ± 0.73 GPa with a Weibull modulus of 5.41. The Hi-Nicalon–BN–SiC fibres showed an average strength of 3.04 ± 0.53 GPa and Weibull modulus of 6.66. After HF treatment, the average strengths of the uncoated and BN–SiC coated Hi-Nicalon fibres were 2.69 ± 0.67 and 2.80 ± 0.53 GPa and the Weibull moduli were 4.93 and 5.96, respectively. The BN–SiC coated fibres extracted from the celsian matrix composite exhibited a strength of 2.38 ± 0.40 GPa and a Weibull modulus of 7.15. The strength of the uncoated Hi-Nicalon fibres in the composite was so severely degraded that they disintegrated into small fragments during extraction with HF. The uncoated fibres probably undergo mechanical surface damage during hot pressing of the composites. Also, the BN layer on the coated fibres acts as a compliant layer, which protects the fibres from mechanical damage during composite processing. The elemental composition and thickness of the fibre coatings were determined using scanning Auger analysis. Microstructural analyses of the fibres and the coatings were done by scanning electron microscopy and transmission electron microscopy. Stengths of fibres calculated using average and measured fibre diameters were in good agreement. Thus, the strengths of fibres can be evaluated using an average fibre diameter instead of the measured diameter of each filament.

1. Introduction

Hi-Nicalon fibre [1] has a desirable combination of tensile strength, elastic modulus, density and retention of these properties at temperatures up to approximately 1200 °C. Thus it is suitable for a variety of aerospace applications as a reinforcement in high performance structural composites with ceramic, metal and polymer matrices. The Hi-Nicalon fibre surface needs to be protected with appropriate ceramic coating(s) in order to alleviate chemical reactions with ceramic and metal matrices during composite processing and use, and also, to provide a weak fibre–matrix interface for toughened ceramic composites.

In a recent study [2] it was observed that the $0.75BaO–0.25SrO–Al_2O_3–2SiO_2$ (BSAS) celsian matrix composite reinforced with Hi-Nicalon (uncoated) fibres showed low flexure strength along with catastrophic failure. In contrast, the composite reinforced with BN–SiC coated Hi-Nicalon fibres was strong, tough and showed graceful failure. Fibre push-through tests and microscopic examinations indicated a weak fibre–matrix interface in both composites, and no chemical reaction was observed between the fibres and the matrix. It was postulated that the low strength of the Hi-Nicalon (uncoated)–celsian composite was due to degradation of fibre strength during composite processing.

The primary objective of the present research was to verify the postulate of this earlier study. Another objective was to carry out microstructural and chemical analyses of the fibres and the coatings. Room temperature tensile strengths of the as-received Hi-Nicalon fibres and those coated with a dual BN–SiC surface layer were measured. In order to investigate fibre strength degradation during composite processing, the strengths of fibres extracted from celsian matrix composites, by leaching away the oxide matrix in 40% HF for 24 h at room temperature, were also measured. The strengths of fibres that had been treated with 40% HF for 24 h at room temperature were also determined. Weibull statistical parameters were determined for each type of fibre. Elemental compositions and thicknesses of the fibre coatings were determined by scanning Auger analysis. Microstructural analyses of the fibres and the coatings were done by scanning

electron microscopy (SEM) and transmission electron microscopy (TEM).

2. Experimental procedure

2.1. Materials

Hi-Nicalon fibre from Nippon Carbon Co. was used in the present study. The properties of the as-received fibre are shown in Table I. These fibres contain nanocrystalline (10–20 nm) β-SiC along with 30–40 at % excess carbon, which is also reflected in its low elastic modulus. A large variation (10–18 μm) in the fibre diameter was observed, although the manufacturer reports an average value of approximately 14 μm.

The polyvinyl alcohol (PVA) sizing on the as-received Hi-Nicalon fibres was burned off in a Bunsen burner flame. Single filaments were carefully separated from the fibre tow for testing. All the coatings on the fibres were applied by 3M Co. using a continuous chemical vapour deposition (CVD) reactor. The BN coating was deposited at approximately 1000 °C utilizing a proprietary precursor and was amorphous to partly turbostratic in nature. A thin overcoating of SiC was also applied to the BN-coated fibres. The nominal coating thickness was 0.4 μm of BN and 0.3 μm of SiC. Uncoated and BN/SiC coated fibres were extracted from the celsian matrix composites by dissolving away the oxide matrix in 40% HF for 24 h. The BN–SiC coated and uncoated Hi-Nicalon fibres were also treated with 40% HF for 24 h to determine if HF leaching affected fibre strength.

2.2. Electron microscopy

Surfaces of the fibres were examined using SEM and TEM. For cross-sectional analysis, fibres were mounted in a high temperature epoxy and polished before examination. Fibre cross-sectional thin foils for TEM were prepared using a procedure developed for ceramic fibres, which involves epoxy potting, slicing, polishing, dimple grinding and Ar ion-beam milling. A thin carbon coating was evaporated onto the TEM thin foils and SEM specimens for electrical conductivity prior to analysis. The thin foils were examined in a Philips EM400T operating at 120 keV. SEM was performed using a Jeol JSM-840A operating at 15 keV.

2.3. Scanning Auger analysis

The elemental compositions of the fibre near the surface and of the fibre surface coatings were analysed [3] with a scanning Auger microprobe (Fisons Instruments Microlab Model 310-F). The fibres for this analysis were mounted on a stainless steel sample mount by tacking the ends with colloidal graphite. Depth profiling was performed by sequential ion-beam sputtering and Auger analysis. The ion etching was done with 3 keV Argon ions rastered over an approximately 1 mm^2 area on the specimen. The etch rate in Ta_2O_5 under these conditions was 0.05 $nm\,s^{-1}$.

Auger electron spectroscopy (AES) analysis was performed using an electron beam current of approximately 1.5 nA. The beam was rastered over a 2×20 μm^2 area of the fibre with the long axis of the area aligned with the long axis of the fibre. Spectra were acquired in integral (as opposed to derivative) form at a beam energy of 2 keV and depth profiles were created by plotting peak areas against ion etch time. The atomic concentrations were calculated by dividing the peak areas by the spectrometer transmission function and the sensitivity factors for each peak, then scaling the results to total 100%. The sensitivity factors were derived from spectra of ion etched Ag, Si, B, Ti, SiC, BN and TiO_2 standards. The sensitivity factors used for each element should not be trusted to better than $\pm$ 20%. The depth scale is from the Ta_2O_5 calibration and no attempt has been made to adjust for the actual etch rate for each material. Only the fibres with a smooth surface coating, rather than those having thick and rough coating morphologies, were used for Auger analysis.

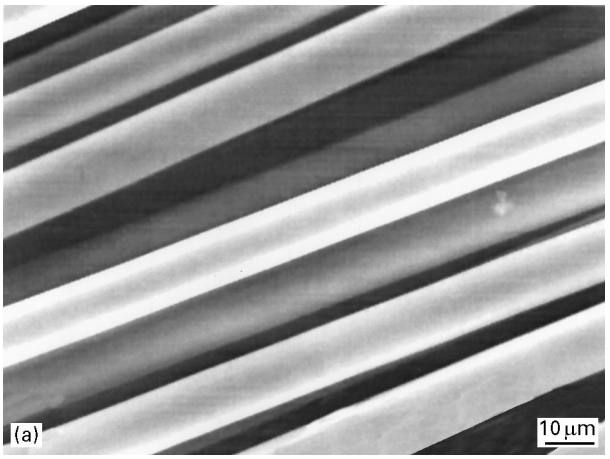
2.4. Tensile strength measurement

Room temperature tensile strengths of the individual filaments were measured in ambient atmosphere with a screw driven Micropull fibre test frame equipped with pneumatic grips. A 1000 g load cell, Sensotec model 34, was used. Measurements were carried out at a constant crosshead speed of 1.261 $mm\,min^{-1}$. A single filament was mounted on a 20 lb paper tab using Hardman extra fast setting epoxy. The side portions of the tab were cut with a hot wire before application of the load, producing a fibre gauge length of 2.54 cm. Twenty filaments of each type of fibre were tested. For the as-received fibres, the actual diameter of each filament tested was measured using a Zeiss Axioskop microscope with a Sony DXC-960MD video camera attached to a Boeckeler VIA-150 video measurement system. Magnification range from X100 to X800, depending upon the size of the fibre. Weibull parameters for tensile strength of as-received fibres were calculated using the measured diameters and also an average diameter of 13.5 μm. For all other fibres tested, tensile strengths were calculated based only on an average filament diameter of 13.5 μm.

3. Results and discussion

3.1. Microstructure

SEM micrographs showing the surface of as-received, flame desized Hi-Nicalon fibres are presented in Fig. 1. The fibre surface appears to be fairly smooth and featureless. Energy dispersive spectroscopy (EDS) compositional spectra (Fig. 2) taken from the polished cross-section of the fibre indicate the presence of only Si and C along with a small amount of oxygen, which is in qualitative agreement with the manufacturer's data (Table I). The surface of HF-treated Hi-Nicalon fibres is also fairly smooth and featureless as seen from the SEM micrograph in Fig. 3. SEM micrographs from the surface and polished cross-section of the BN–SiC coated Hi-Nicalon fibres are given in



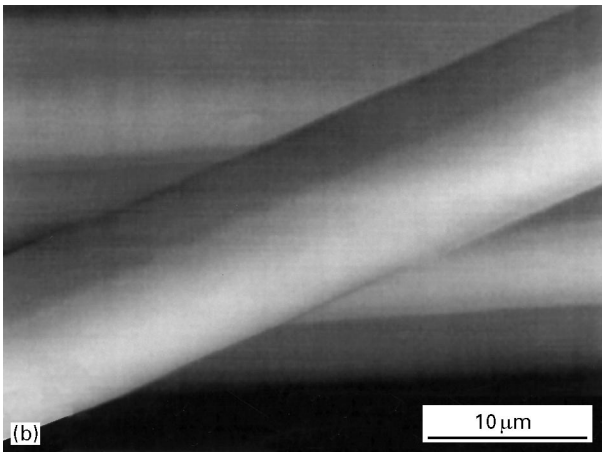


Figure 1 SEM micrographs showing surface of desized Hi-Nicalon fibres.

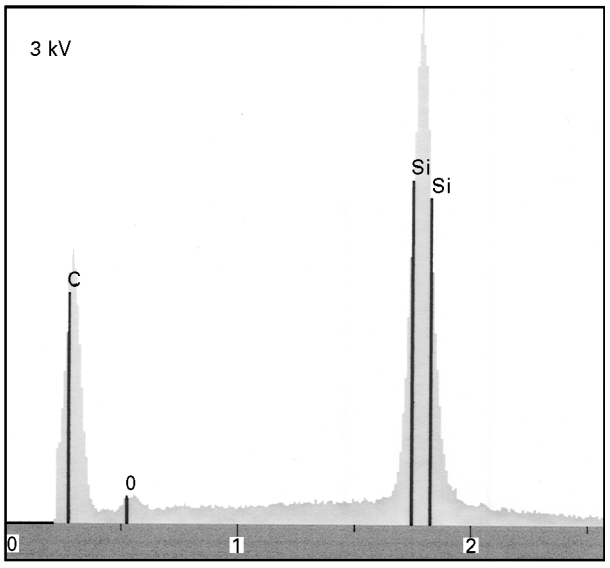
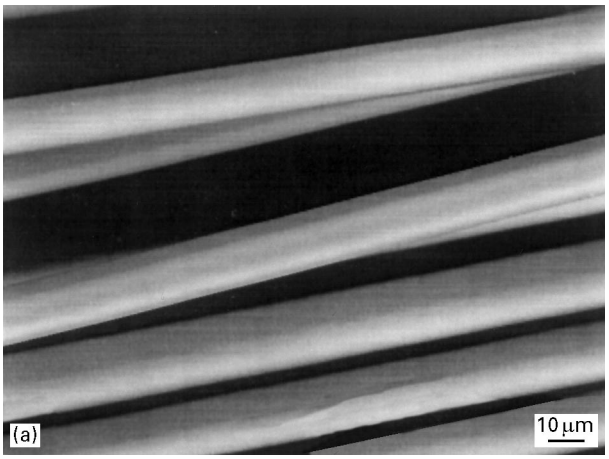


Figure 2 EDS spectra from the cross-section of desized Hi-Nicalon fibre at 3 kV.

TABLE I Properties of Hi-Nicalon fibres[a]

Property	Value
Density, g cm^{-3}	2.74
Diameter, μm	14
Filaments per tow	500
Denier, g (9000 m)$^{-1}$	1800
Tensile strength, GPa	2.8
Elastic modulus, GPa	269
Thermal expansion coefficient, 10^{-6} K^{-1}	3.5 (25–500) [b]
Specific heat, J g^{-1} K^{-1}	0.67 (25) [b] 1.17 (500) [b]
Thermal conductivity, W m^{-1} K^{-1}	7.77 (25) [b] 10.1 (500) [b]
Electrical resistivity, Ω cm	1.4
Chemical composition, wt %	63.7 Si, 35.8 C, 0.5 O
C/Si, atomic ratio	1.3–1.4
Temperature capability, °C	1200

[a]Data from Nippon Carbon Co.
[b]Values in parentheses are in degrees celcius.



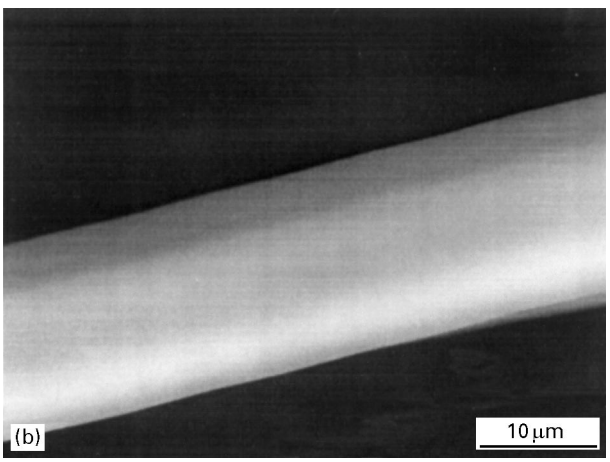
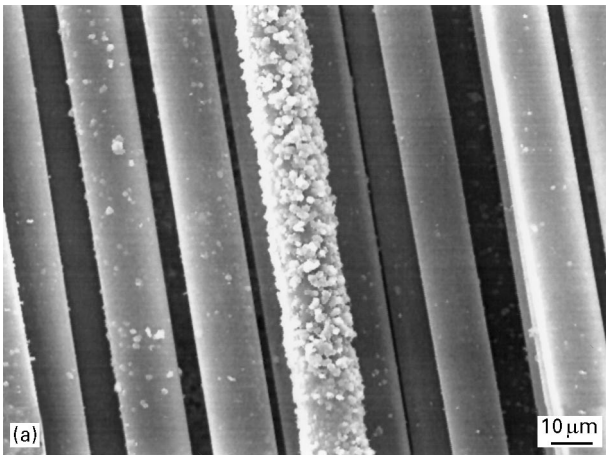


Figure 3 SEM micrographs showing surface of Hi-Nicalon fibre after treatment with HF.

Fig. 4a, b. The coating on most of the fibres is smooth and uniform, but some fibres have a quite granular coating. The BN coating is often nodular as can be seen in Fig. 5. The nodules in the BN layer remain or, often, are enhanced by the subsequent overcoating of SiC. The number density of the nodules varies considerably from filament to filament, but is more consistent along an individual fibre. Within a tow, the fibres on the outside had thicker and more granular coatings than those on the inside. Overall, the coating thickness was reasonably uniform. Generally, the BN coating thickness was 0.2–0.4 μm, but could be much



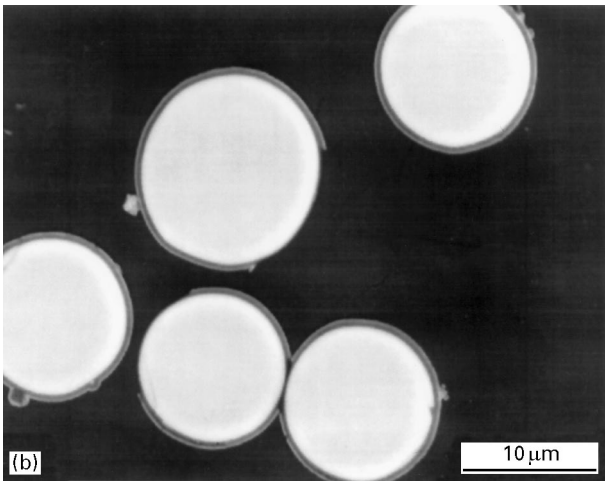


Figure 4 SEM micrographs from (a) surface and (b) polished cross-section of BN–SiC coated Hi-Nicalon fibres.

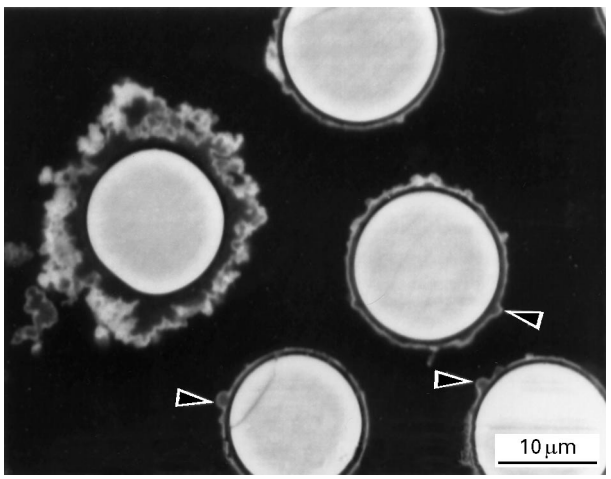


Figure 5 SEM micrograph from polished cross-section of BN–SiC coated Hi-Nicalon fibres showing nodular coating. Some of the nodules are indicated by arrows.

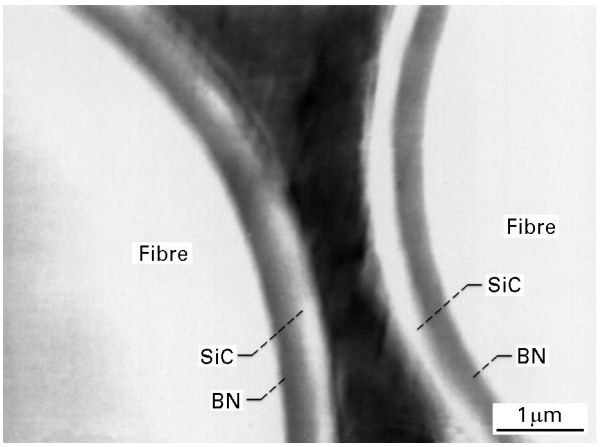


Figure 6 SEM micrograph at high magnification from polished cross-section of BN–SiC coated Hi-Nicalon fibres showing sharp contrast between the fibre and the coatings.

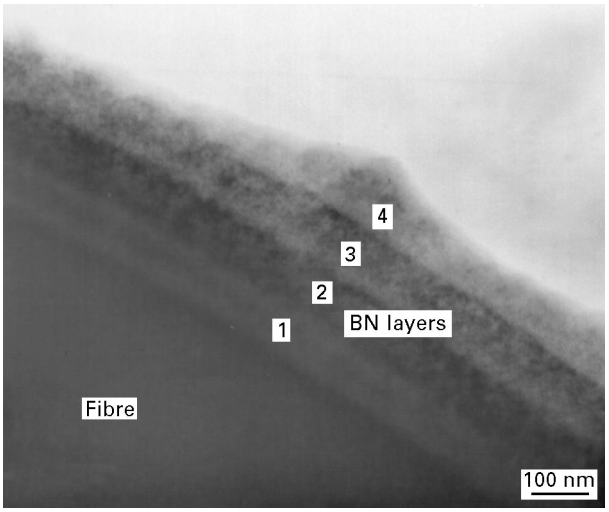


Figure 7 TEM bright field image showing cross-section of the as-received BN–SiC coated Hi-Nicalon fibre; four distinct sub-layers are visible within the BN coating.

higher at the nodules. The total coating thickness on a fibre could be as high as 7 μm when a granular cluster is attached (Fig. 5). Typically, the SiC coating thickness varied between 0.2–0.3 μm. Sharp contrast is observed between the fibre, BN coating and SiC coating in polished cross-sections (Fig. 6). The microstructure of these coatings is quite similar to those of BN–SiC coatings on HPZ ceramic fibres [3].

TEM analysis of the loose, BN–SiC coated Hi-Nicalon fibres in cross-section revealed four distinct layers in the BN coating (Fig. 7). While there was no detectable variation in composition between the layers, there was a subtle variation in crystallographic structure. All four layers were turbostratic in structure, but layers 1 and 3 (counting from the fibre outward) were more directional. The basic structural units of the BN are more aligned parallel to the fibre surface, giving sharper cusps in microelectron diffraction patterns (Fig. 8). The SiC layer on the as-coated fibres typically ion milled away before electron transparent thin regions were obtained, but appeared to be adherent to the BN layers.

After treatment with HF, the BN–SiC surface coating was cracked, and spalled off at some places on the fibres, as seen in the SEM micrographs in Fig. 9. SEM micrographs showing surfaces of the uncoated BN–SiC coated fibres extracted from the celsian composite are given in Figs. 10 and 11, respectively.

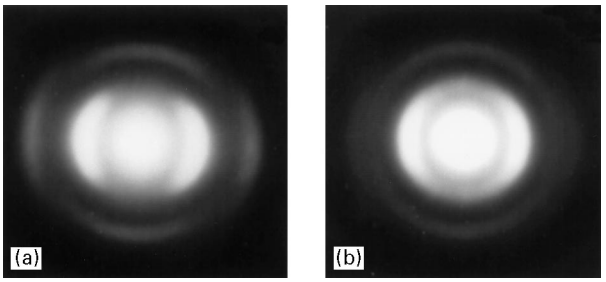


Figure 8 Microelectron diffraction patterns typical for BN layers in BN–SiC coated Hi-Nicalon fibres: (a) layers 1 and 3 and (b) layers 2 and 4.

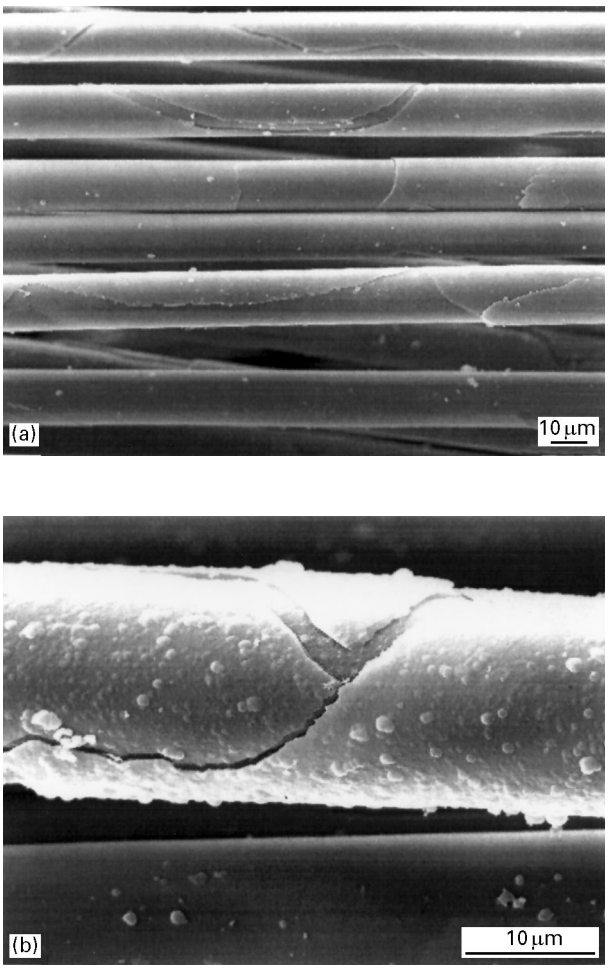
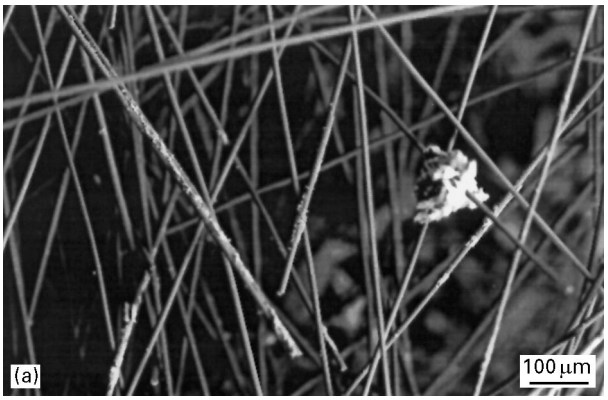


Figure 9 SEM micrographs showing surface of BN–SiC coated Hi-Nicalon after treatment with HF.

Matrix particles are still sticking to many of the fibres and cracks in the coating are observed on those fibres having a thick granular coating.

3.2. Scanning Auger analysis

Elemental composition depth profiles, as obtained from scanning Auger analysis of as-received, but desized, Hi-Nicalon fibres and those having BN–SiC surface coatings are shown in Fig. 12a, b. Depth profiles of the uncoated and coated fibres, after treatment with HF, are also presented in Fig. 12c, d. The elemental composition (atomic percent) of the as-received fibre is approximately 42% Si and 56% C with C/Si



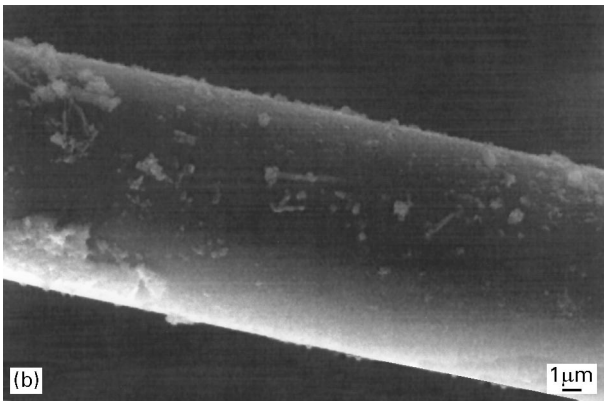


Figure 10 SEM micrographs showing surface of uncoated Hi-Nicalon fibres extracted from celsian matrix composite by dissolving away the matrix in HF.

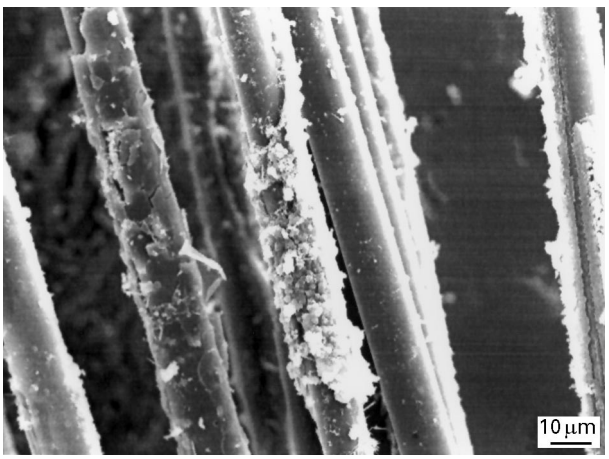


Figure 11 SEM micrograph showing surface of BN–SiC coated Hi-Nicalon fibres extracted from celsian matrix composite by dissolving away the matrix in HF.

atomic ratio of 1.33. A trace amount of oxygen is also detected. These results are in good agreement with the fibre composition supplied by the manufacturer (Table I). The composition of the HF treated Hi-Nicalon fibre is found to be approximately 43% Si, 54% C and a trace of oxygen. The Hi-Nicalon–BN–SiC fibre has approximately 130 nm thick layer of Si-rich SiC followed by approximately 500 nm thick layer of B-rich BN. However, the coating thickness was non-uniform from filament to filament as seen from the SEM micrographs in Figs. 4 and 5. The fibres

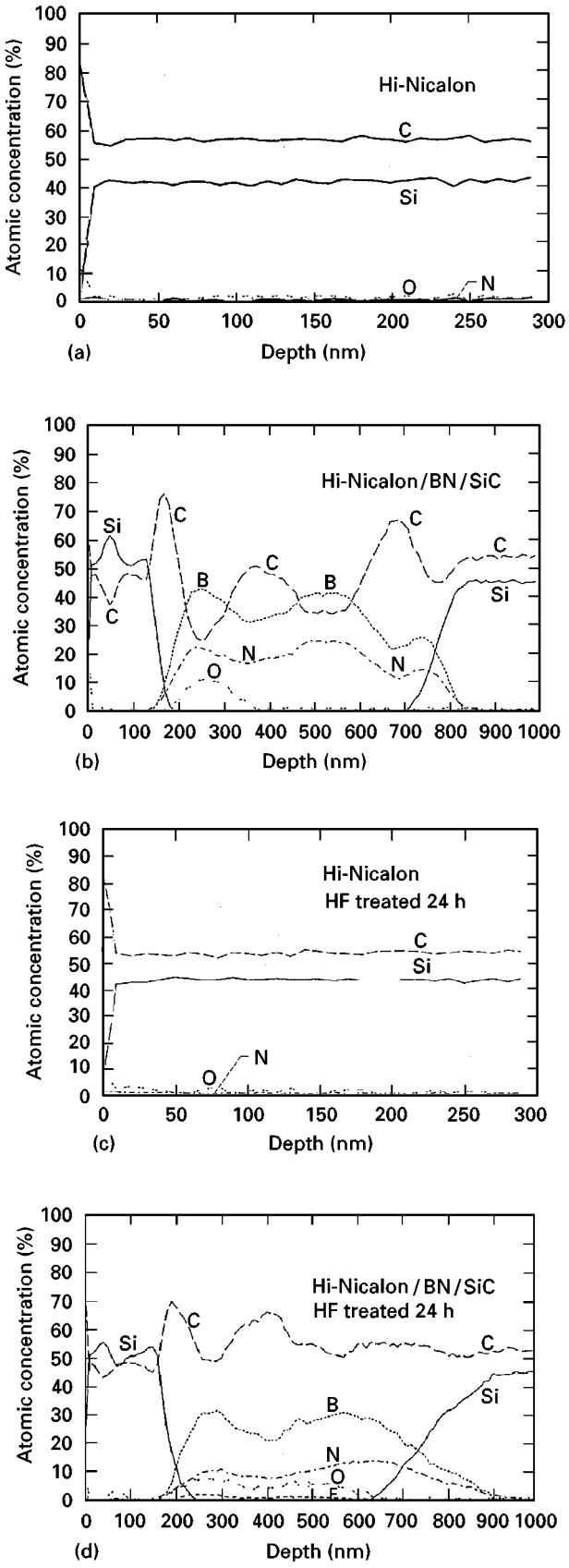


Figure 12 Surface depth profiles of various elements obtained using scanning Auger microprobe for (a) Hi-Nicalon, (b) Hi-Nicalon–BN–SiC, (c) HF treated Hi-Nicalon and (d) HF treated Hi-Nicalon–BN–SiC fibres.

on the outside of a tow had thicker and more granular coatings than those on the inside. The BN coating is contaminated with oxygen and contains a high concentration of carbon. In fact, the concentration of carbon is larger than those of boron and nitrogen. The results for the HF treated Hi-Nicalon–BN–SiC fibre are quite similar to those for the untreated fibre. However, a trace amount of F is also detected in the BN layer of the treated fibre.

3.3. Tensile strength

The strength of ceramic fibres is determined by the statistical distribution of flaws in the material. The tensile strength of ceramic fibres is generally analysed on the basis of the well known Weibull statistics [4]. An excellent review of the subject was provided by Van der Zwaag [5]. The Weibull modulus describes the distribution of strength in materials that fail at defects according to weakest link statistics. The probability of survival of a material is given by the empirical equation

$$P_s = 1 - P(\sigma) = \exp\{-V[(\sigma - \sigma_u)/\sigma_o]^m\} \quad (1)$$

where P_s is the survival probability ($P(\sigma) = 1 - P_s$ is the failure probability) of an individual fibre at an applied stress of σ, V is the fibre volume, σ_u is the stress below which failure never occurs, σ_o is the scale parameter, and m is the shape or flaw dispersion parameter. For a given material, the Weibull modulus m and σ_o are constant. Assuming $\sigma_u = 0$ and uniform fibre diameter along the length, L, corresponding to the volume, V, Equation 1 can be rearranged as

$$\ln\ln(1/P_s) = \ln\ln\{1/[1 - P(\sigma)]\} = m\ln\sigma + \text{constant} \quad (2)$$

The experimental data are ranked in ascending order of strength values and the cumulative probability $P(\sigma_i)$, is assigned as

$$P(\sigma_i) = i/(1 + N) \quad (3)$$

where i is the rank of the tested fibre in the ranked strength tabulation and N is the total number of fibres tested. A least-squares linear regression analysis is then applied to a plot of $[\ln\ln 1/P_s]$ versus $\ln(\sigma)$, whose slope is the Weibull modulus m.

The $\ln\ln(1/P_s)$ versus $\ln\sigma$ Weibull probability plots for room temperature tensile strength of uncoated Hi-Nicalon fibres using measured and average diameters of the filaments are presented in Fig. 13 and the values of Weibull parameters obtained from linear regression analysis are summarized in Table II. The tensile strength of as-received Hi-Nicalon fibre is 3.30 ± 0.57 GPa and the Weibull modulus, m, is 7.03 using measured diameter for each filament tested. The average value of fibre diameter was found to be 13.5 μm. Using this average diameter, values of fibre tensile strength and m were calculated to be 3.19 ± 0.73 GPa and 5.41, respectively. The manufacturer's information data sheet [1] reports a value of 2.8 GPa for the room temperature tensile strength of these fibres. The strengths of fibres obtained from measured and average fibre diameters are in good agreement. Thus, an average fibre diameter, instead of the measured diameter of each fibre, can be used in determining fibre strength. Weibull moduli calculated

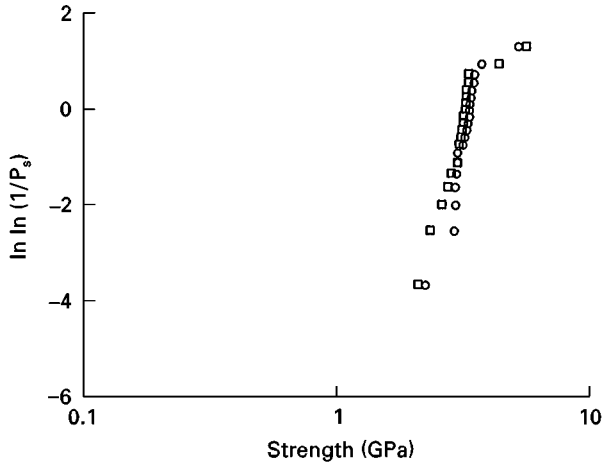


Figure 13 Weibull probability plots for room temperature tensile strengths of Hi-Nicalon fibres using (a) measured and (b) average diameters of the filaments: ○, a; □, b.

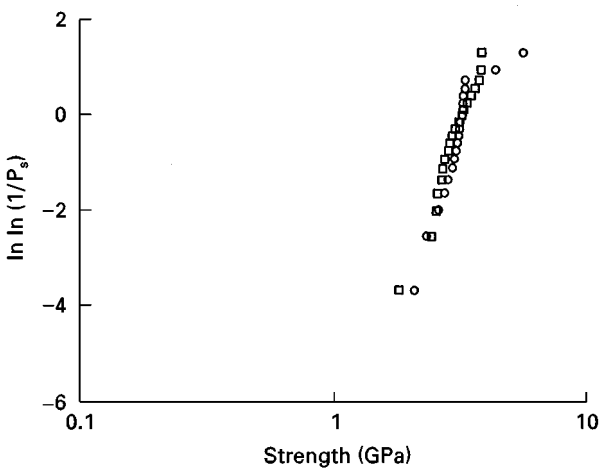


Figure 14 Weibull probability plots for room temperature tensile strengths of fibres: (a) Hi-Nicalon and (b) BN–SiC coated Hi-Nicalon: ○, a; □, b.

from measured and average filament diameters are also in reasonable agreement. Similar results have also been reported very recently by Petry *et al.* [6] from tensile strength measruements of Nicalon and alumina–yttrium aluminum garnet eutectic fibres. The Weibull parameters for the tensile strengths of other fibres tested in the present study were evaluated using an average filament diameter of 13.5 μm.

Weibull plots for the uncoated Hi-Nicalon fibres and those having a dual BN–SiC surface coating are shown in Fig. 14. Similar Weibull plots for the uncoated and BN–SiC coated Hi-Nicalon fibres, which have been treated with 40% HF for 24 h, and those extracted from the celsian matrix composite are given in Fig. 15. Values of Weibull parameters obtained from linear regression analysis for various fibres are summarized in Table II. The BN–SiC coated Hi-Nicalon fibres show an average tensile strength of 3.04 ± 0.53 GPa with an m value of 6.66 indicating no strength degradation during fibre coating by CVD. After HF treatment, both the as-received and the BN–SiC coated fibres show strength degradation of approximately 8–15%. The tensile strength of the BN–SiC coated fibres extracted from the celsian matrix composite by dissolving away the matrix in HF has degraded to 2.38 ± 0.40 GPa with an m value of 7.15. Exposure of the fibres to high temperature during hot pressing of the composites could probably account for this strength degradation. In contrast, the fibres extracted from the Hi-Nicalon (uncoated) celsian composite were so weak that they disintegrated into small pieces during extraction with HF. The strengths of these uncoated fibres probably degrade due to mechanical surface damage during composite hot pressing. The BN layer in the BN–SiC coated fibres acts as a compliant layer protecting the fibres from mechanical damage during composite processing. SEM micrographs showing typical fracture surfaces of uncoated and BN–SiC coated Hi-Nicalon fibres in the celsian matrix composite are shown in Fig. 16. Fracture surfaces of the two fibres are seen to be quite different. The fracture of the uncoated fibre is featureless, whereas fracture mirror and heckle are present in the fracture surface of the coated fibre. Uncoated Hi-Nicalon fibres extracted from lithium aluminosilicate (LAS) glass-ceramic matrix composites [7], which

TABLE II Weibull parameters for room temperature tensile strength of Hi-Nicalon fibres (Gauge length = 2.54 cm; crosshead speed = 1.261 mm min^{-1})

Fibre treatment	Fibre diameter (μm)	Average strength (GPa)	Standard deviation[c] (GPa)	Weibull modulus (m)
As-received, flame desized Hi-Nicalon	Actual[a]	3.30	0.57	7.03
As-received, flame desized Hi-Nicalon	13.5 (average)	3.19	0.73	5.41
As-received, flame desized Hi-Nicalon; treated with 40% HF, 24 h	13.5 (average)	2.69	0.67	4.93
BN–SiC coated Hi-Nicalon	13.5 (average)	3.04	0.53	6.66
BN–SiC coated Hi-Nicalon; treated with 40% HF, 24 h	13.5 (average)	2.80	0.53	5.96
Uncoated, extracted from composite	—	b	—	b
BN–SiC coated Hi-Nicalon, extracted from celsian composite by dissolving away matrix in 40% HF, 24 h	13.5 (average)	2.38	0.40	7.15

[a]Actual fibre diameter measured for each filament tested.
[b]Fragmented into small pieces during extraction with HF (could not measure strength).
[c]20 filaments tested for each fibre type.

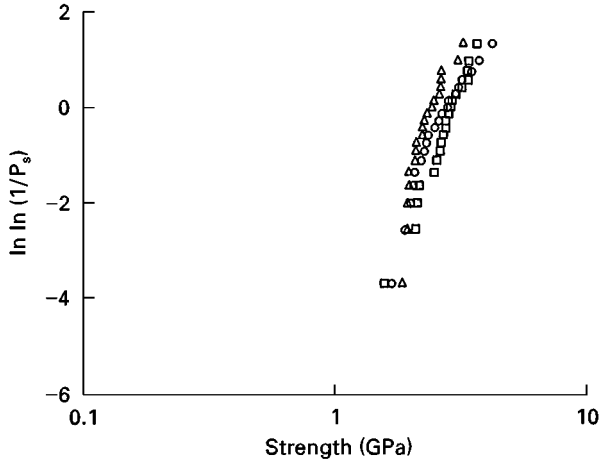
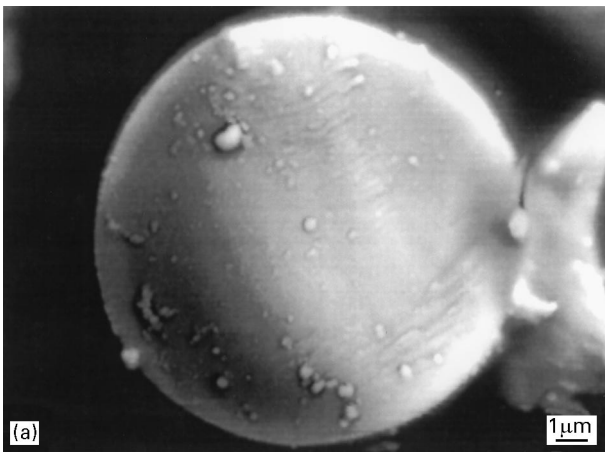


Figure 15 Weibull probability plots for room temperature tensile strengths of chemically treated fibres: (a) HF treated Hi-Nicalon, (b) BN–SiC coated Hi-Nicalon treated with HF and (c) BN–SiC coated Hi-Nicalon extracted from celsian matrix composite by leaching away matrix in HF: ○, a; □, b; △, c.



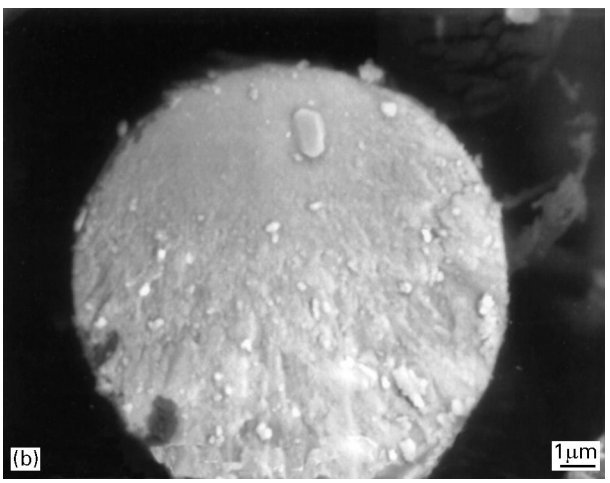


Figure 16 SEM micrographs showing typical fracture surfaces of (a) as-received and (b) BN–SiC coated Hi-Nicalon fibres in celsian matrix composites.

were hot pressed at 1360 °C for 40 min, showed only 20–25% reduction in tensile strength. This is because the LAS composites were densified for a short time just above the matrix melting point, making use of the viscous flow of glass.

3.4. Summary of results

Room temperature tensile strengths of as-received Hi-Nicalon fibres and those having a dual BN–SiC surface coating have been determined. Tensile strengths of these fibres following treatment with HF for 24 h have also been measured. Room temperature tensile strengths of uncoated and BN–SiC coated Hi-Nicalon fibres extracted from celsian matrix composites, by leaching away the matrix in HF for 24 h, were also determined. The average tensile strength of as-received Hi-Nicalon was 3.19 ± 0.73 GPa with a Weibull modulus of 5.41. Also, tensile strengths of uncoated fibres calculated using average and measured fibre diameters were in good agreement. The BN–SiC coated Hi-Nicalon fibres showed an average strength of 3.04 ± 0.53 GPa and Weibull modulus of 6.66. After HF treatments, the average strengths of the uncoated and BN–SiC coated Hi-Nicalon fibres were 2.69 ± 0.67 and 2.80 ± 0.53 GPa and the Weibull moduli were 4.93 and 5.96, respectively. The BN–SiC coated fibres extracted from celsian matrix composite exhibited a strength of 2.38 ± 0.40 GPa and Weibull modulus of 7.15. The strength of the uncoated Hi-Nicalon fibres in the composite had been so severely degraded that they disintegrated into small pieces during extraction with HF. This strength degradation is probably caused by mechanical surface damage to the uncoated fibres during hot pressing of the composites. The elemental composition and thickness of the fibre coatings were determined using scanning Auger analysis. HF leaching had no effect on the composition of the fibres. The BN coating, consisting of four distinct layers, was amorphous to partly turbostratic and contaminated heavily with carbon and somewhat with oxygen. The silicon carbide coating was crystalline, non-stoichiometric, non-uniform and sometimes flaky. Microstructural analyses of the fibres and the coatings gave no evidence that HF treatment damaged the fibres or that surface damage to the fibres (excluding the coatings) occurred.

4. Conclusions

The BN coating in the BN–SiC coated Hi-Nicalon fibres acts as a compliant layer protecting the fibres from mechanical damage during composite hot pressing. Tensile strengths of fibres can be evaluated using an average diameter instead of the measured diameter of each filament. This should circumvent the amount of time and effort spent in the measurement of diameters of individual filaments.

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