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Fabrication of Fiber-Reinforced Celsian Matrix Composites

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FABRICATION OF FIBER-REINFORCED CELSIAN MATRIX COMPOSITES

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SUMMARY

A method has been developed for the fabrication of small diameter, multifilament tow fiber reinforced ceramic matrix composites. Its application has been successfully demonstrated for the Hi-Nicalon/celsian system. Strong and tough celsian matrix composites, reinforced with BN/SiC-coated Hi-Nicalon fibers, have been fabricated by infiltrating the fiber tows with the matrix slurry, winding the tows on a drum, cutting and stacking of the prepreg tapes in the desired orientation, and hot pressing. The monoclinic celsian phase in the matrix was produced *in situ*, during hot pressing, from the $0.75BaO\text{-}0.25SrO\text{-}Al_2O_3\text{-}2SiO_2$ mixed precursor synthesized by solid state reaction from metal oxides. Hot pressing resulted in almost fully dense fiber-reinforced composites. The unidirectional composites having ~42 vol % of fibers exhibited graceful failure with extensive fiber pullout in three-point bend tests at room temperature. Values of yield stress and strain were 435±35 MPa and 0.27±0.01 percent, respectively, and ultimate strengths of 900±60 MPa were observed. The Young's modulus of the composites was measured to be 165±5 GPa.

INTRODUCTION

Monoclinic celsian $BaAl_2Si_2O_8$ (BAS) and $SrAl_2Si_2O_8$ (SAS) are refractory materials having melting points higher than 1700 °C. These materials are resistant to oxidation and reduction and also show reasonably good resistance to alkali attack. These materials are phase stable up to ~1600 °C and chemically compatible with alumina, mullite and silicon nitride (in inert or nitrogen atmospheres) at elevated temperatures. They show low values of dielectric constant and loss tangent which make them promising materials for electromagnetic windows or radome applications (ref. 1) at high temperatures, packaging for microelectronics, high voltage condensers and other electric insulating products. Celsian is also useful as an environmental barrier coating for SiC_f/SiC composites and protects against the loss of silica as volatile silicon hydroxide species in the combustion products in turbine engines at elevated temperatures (ref. 2). Celsian is also being investigated as a matrix material for fiber-reinforced composites (refs. 3 to 6) for high temperature structural applications in hot sections of turbine engines.

BAS exists in three different polymorphs, the monoclinic, hexagonal, and orthorhombic phases. The monoclinic phase, commonly known as celsian, is the naturally occurring phase. The hexagonal phase, also known as hexacelsian, and the orthorhombic phase are found only in synthetic products. The hexacelsian phase is thermodynamically stable at temperatures between 1590 °C and the melting point whereas the celsian phase is stable at temperatures below 1590 °C. However, hexacelsian can exist as a metastable phase at all temperatures from 1590 °C to room temperature. At ~300 °C, hexacelsian undergoes a rapid, reversible structural transformation (ref. 7) into the orthorhombic form, accompanied by a large volume change of ~3 percent. Thus, hexacelsian is an undesirable phase.

In both the BAS and SAS systems, hexacelsian is always the first phase to form. However, on heat treatment at ~1200 °C or higher temperatures, its transformation into the monoclinic phase is very sluggish in BAS (ref. 8) and very rapid in SAS (ref. 9). It is known that doping of BAS with SAS accelerates the hexacelsian to monoclinic celsian transformation (ref. 10). Fortunately, BAS and SAS form solid solutions in the entire composition range (refs. 1 and 10). The starting composition of $0.75BaO\text{-}0.25SrO\text{-}Al_2O_3\text{-}2SiO_2$ (BSAS) was used for the synthesis of monoclinic celsian in the present study.

Processing and properties of celsian glass-ceramic matrix composites reinforced with large diameter CVD SiC SCS-6 monofilaments (refs. 3 to 6) and the multifilament small diameter Nicalon (ref. 11) and HPZ (ref. 12) fibers have been described earlier. The objective of this study was to develop the fabrication of small diameter, multifilament tow fiber-reinforced celsian matrix composites. Microstructures of the resulting FRCs were characterized and room temperature mechanical properties were measured. Strong, tough, and almost fully dense composites reinforced with BN/SiC-coated, Hi-Nicalon fibers have been obtained.

MATERIALS AND EXPERIMENTAL PROCEDURE

The matrix of $0.75BaO\text{-}0.25SrO\text{-}Al_2O_3\text{-}2SiO_2$ (BSAS) composition was synthesized by a solid-state reaction method (ref. 13). The starting materials used were $BaCO_3$ (Alfa Products), $SrCO_3$ (Alfa Products), Al_2O_3 (Baikowski International Corp., high purity CR 30), and SiO_2 (Cerac Inc., 99.9 percent purity, –325 mesh) powders. Appropriate quantities of various powders were slurry mixed in acetone and ball milled for ~24 hr using alumina milling media. Acetone was then evaporated and a part of the mixture was subjected to thermogravimetric analysis (TGA) in air. The oxide mixed powder was calcined at ~900 to 920 °C for decomposition of the carbonates into oxides, followed by cooling to room temperature and grinding. A small part of the calcined powder was loaded into a graphite die and hot pressed at 1300 °C for 2 to 3 hr under 27.5 MPa (4 ksi) pressure.

Polymer derived Hi-Nicalon fiber tows (1800 denier, 500 filaments/tow) with low oxygen content produced by Nippon Carbon Co. were used as the reinforcement. These fibers have an average diameter of ~14 mm; a reported (refs. 14 and 15) chemical composition (wt %) of 62.4%Si, 37.1%C, and 0.5%O; and C/Si atomic ratio of ~1.39. The Hi-Nicalon fibers mainly consist of SiC microcrystals with an average grain size of 4 nm and amorphous carbon. These fibers have a density of 2.7 g/cm^3, room temperature tensile strength of ~2.8 GPa, elastic modulus of 270 GPa, and an average coefficient of thermal expansion of 3.5×10^{-6}/°C from room temperature to 500 °C. Hi-Nicalon fibers having a dual surface layer of BN overcoated with SiC were used in the present study. The fiber coatings were applied by a commercial vendor using a continuous chemical vapor deposition (CVD) reactor. The BN coating was deposited at ~1000 °C utilizing a proprietary precursor and was amorphous to partly turbostratic in nature. A thin overcoating of SiC was also deposited by CVD onto the BN-coated fibers. The SiC layer was crystalline. The nominal coating thicknesses were 0.4 μm for BN and 0.3 μm for SiC. The BN interfacial layer acts as a weak, crack-deflecting phase, while the SiC overcoat acts as a barrier to diffusion of boron from BN into the oxide matrix and also prevents diffusion of matrix elements into the fiber.

A sketch of the set-up used for infiltration of the matrix slurry into the fiber tows is shown in figure 1. This is similar to the set-up reported earlier by Prewo (ref. 16). BSAS powder that had been calcined at 900 to 920 °C for 20 to 24 hr was made into a slurry by dispersing it in methyl ethyl ketone along with organic additives as binder, surfactant, deflocculant and plasticizer followed by ball milling. Tows of BN/SiC-coated Hi-Nicalon fibers were spread using rollers and coated with the matrix precursor by passing through the slurry. Excess slurry was squeezed out of the fiber tow before winding at 0.977 mm tow spacing (26 fiber tows/in.) on a rotating drum. After drying, the prepreg tape was cut to size. Unidirectional fiber-reinforced composites were prepared by tape lay up (12 plies) followed by warm pressing at ~150 °C to form a "green" composite. The fugitive organics were slowly burned out of the sample in air at ~500 °C, followed by hot pressing under vacuum in a graphite die. Mêe hot pressed fiber-reinforced composite panel was surface polished and sliced into test bars (~50.4×6.4×1.9 mm) for mechanical testing.

Thermogravimetric analysis (TGA) of the calcination process was carried out at a heating rate of 5 °C /min under flowing air (~60 ml/min) from room temperature to 1500 °C using a Perkin-Elmer TGA-7 system which was interfaced with a computerized data acquisition and analysis system. X-ray diffraction (XRD) patterns were recorded at room temperature using a step scan procedure (0.02°/2θ step, time per step 0.5 or 1 sec) on a Philips ADP-3600 automated diffractometer equipped with a crystal monochromator employing Cu K_α radiation. Density was measured from dimensions and mass as well as by the Archimedes method. Microstructures of the polished cross-sections and fracture surfaces were observed in an optical microscope as well as in a JEOL JSM-840A scanning electron microscope (SEM). For transmission electron microscopy (TEM), thin foils of the composite samples were prepared by slicing, polishing, dimple grinding, and argon ion beam milling. A thin carbon coating was evaporated onto the TEM thin foils and the SEM specimens for electrical conductivity prior to analysis. The thin foils were examined in a Philips EM400T operating at 120keV. X-ray elemental analyses on the TEM were acquired using a Kevex thin window energy dispersive spectrometer (EDS) and analyzer. Mechanical properties were determined from stress-strain curves recorded in three-point flexure using an Instron 4505 universal testing instrument at a crosshead speed of 1.27 mm/min (0.05 in./min) and support span

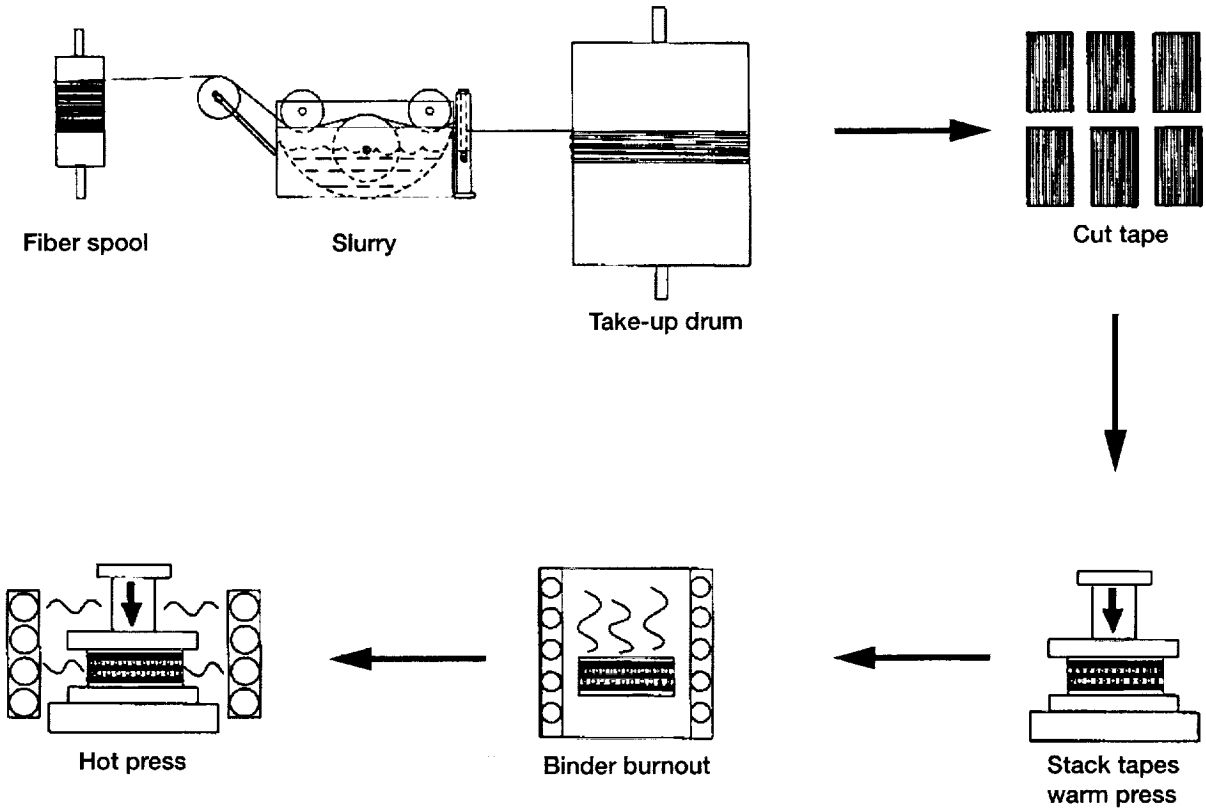

Figure 1.—Schematic of the set-up used for fabrication of small diameter multifilament fiber tow reinforced ceramic composites by matrix slurry infiltration and hot pressing.

(L) of 40 mm. Strain gauges were glued to the tensile surfaces of the flexure test bars. Stress was calculated using beam theory. The yield stress was calculated from the stress-strain curves from the point where the curve deviates from linearity. Elastic modulus of the composite was determined from the linear portion of the stress-strain curve up to the yield point using linear interpolation.

RESULTS AND DISCUSSION

The TGA curve of the mixed BSAS powder consisting of metal carbonates and oxides is shown in figure 2. Minor weight loss near room temperature is due to evaporation of the residual moisture and acetone. A major event showing a large weight loss, due to the decomposition of barium and strontium carbonates into oxides, is observed between ~750 to 1000 °C. A calcination temperature of 900 to 920 °C was chosen for decomposition of the carbonates. The mixed powder was calcined at this temperature for 20 to 24 hr in air. TGA analysis of this calcined powder showed no further weight loss indicating complete decomposition of the metal carbonates during the calcination step.

The XRD pattern of the mixed powder, calcined at ~915 °C for 20 hr in air, is presented in figure 3. SiO_2 (α-quartz) and $BaAl_2O_4$ were the major phases present. Small amounts of Ba_2SiO_4, α-Al_2O_3, and $Ba_2Sr_2Al_2O_7$ were also identified. Figure 4 shows the XRD pattern taken from the surface of a monolithic sample made by hot pressing the precalcined powder at 1300 °C for 2 hr under 27.5 MPa (4 ksi). All the XRD peaks correspond to the monoclinic celsian phase with complete absence of the undesirable hexacelsian phase.

SEM micrographs showing the surface and the cross-sections of the BN/SiC coated Hi-Nicalon fibers are given in figure 5. The coatings on some of the fibers appear to be smooth and of uniform thickness whereas on other fibers the coating is very thick and granular. SEM micrographs taken from the polished cross-sections of the fibers are shown in figure 6. The double coating on the fiber surface is clearly visible. The dark layer is BN and the bright layer is SiC.

Some of the composite physical characteristics are given in table I. The fiber volume fraction in the composites was calculated to be ~42 percent. From their densities, the composites appear to be almost fully dense. The XRD pattern taken

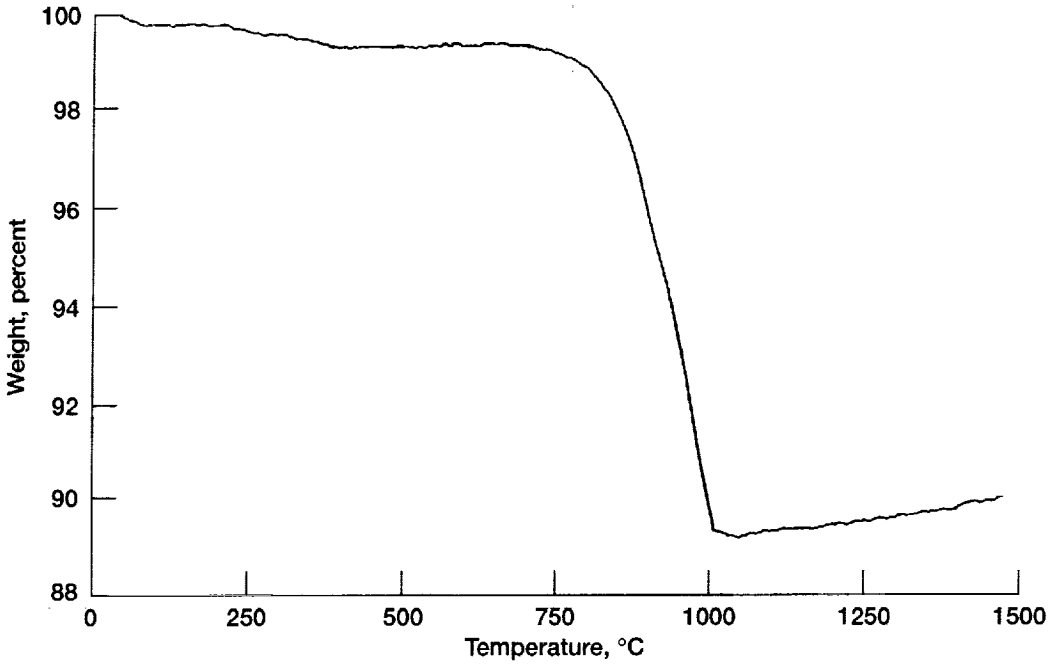

Figure 2.—TGA curve of $0.75BaCO_3$–$0.25SrCO_3$–Al_2O_3–$2SiO_2$ mixed powder at a heating rate of 5 °C/min in air.

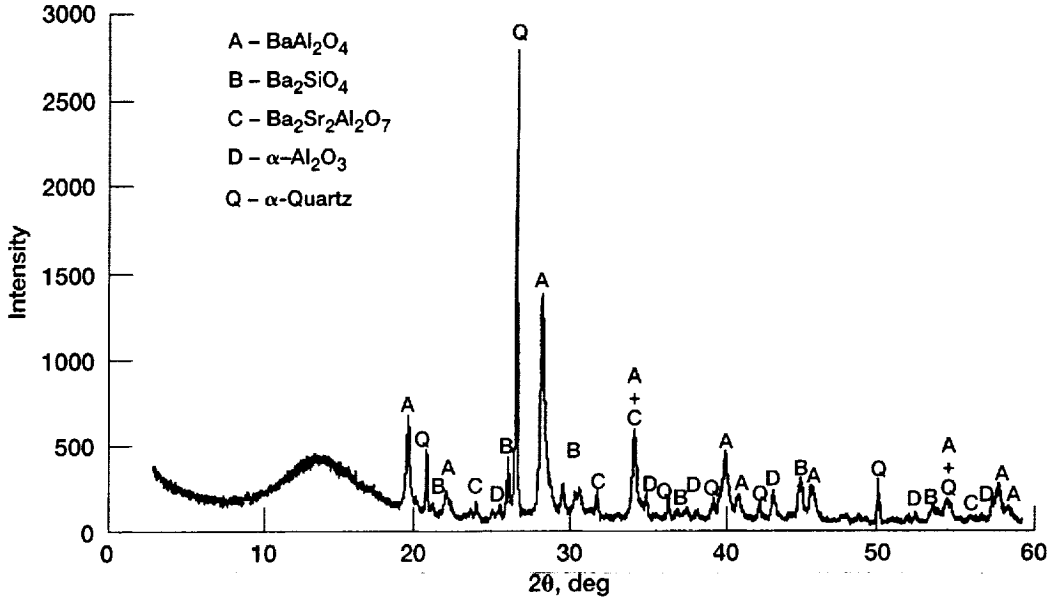

Figure 3.—Powder x-ray diffraction pattern of the mixed $0.75BaCO_3$–$0.25SrCO_3$–Al_2O_3–$2SiO_2$ powder after calcination at 915 °C for 20 h in air.

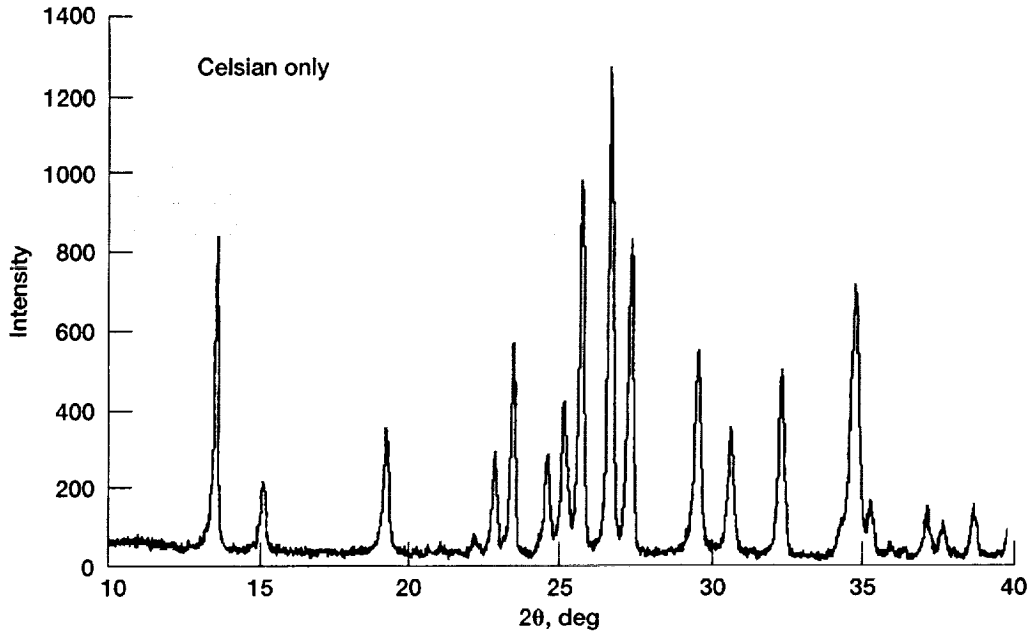

Figure 4.—X-ray diffraction spectra from the surface of a $Ba_{0.75}Sr_{0.25}Al_2Si_2O_8$ plate hot pressed at 1300 °C for 2 hr at 27.6 MPa. All the diffraction peaks match with the monoclinic celsian phase.

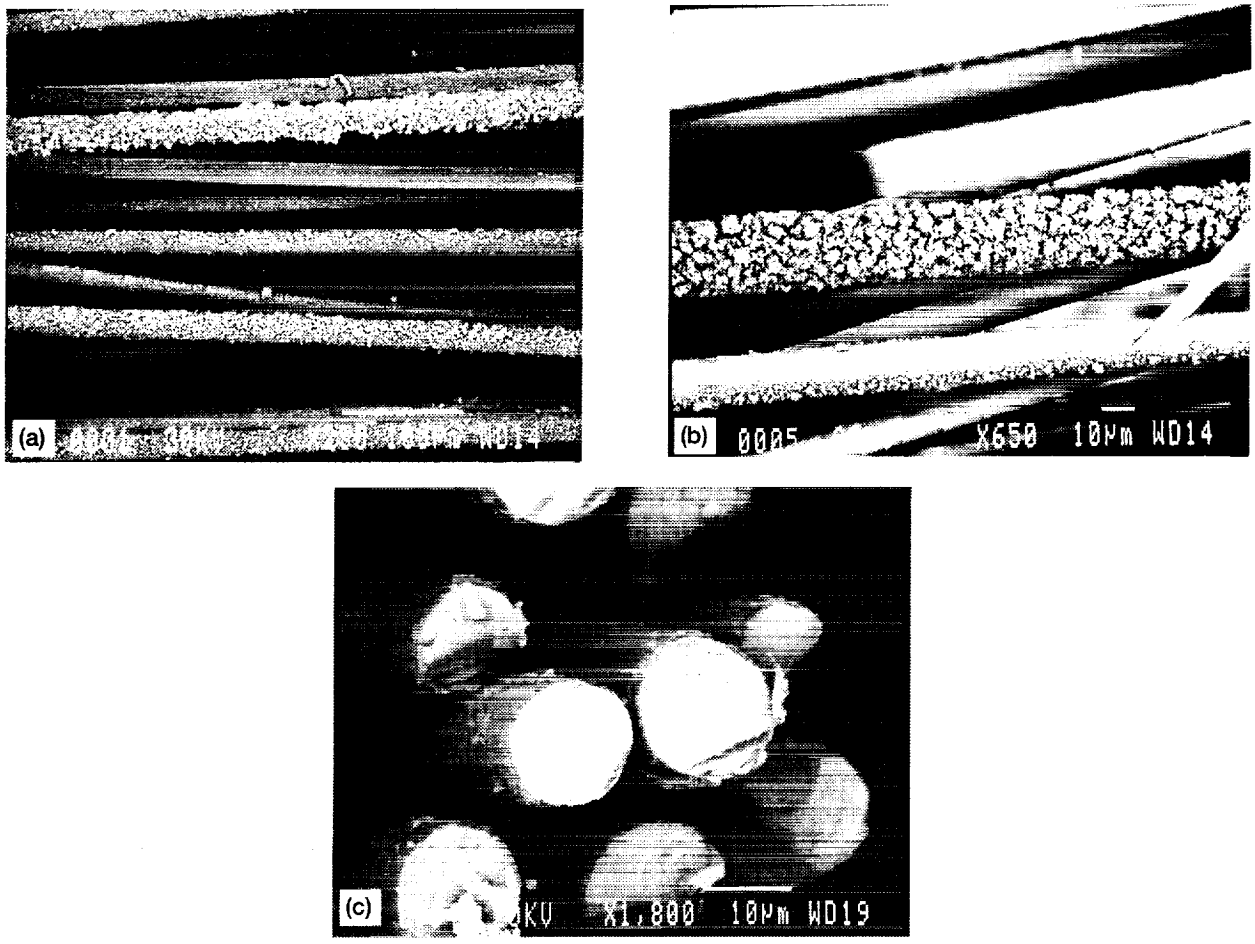

Figure 5.—SEM micrographs showing surface (a and b) and cross-section (c) of BN/SiC coated Hi-Nicalon fibers.

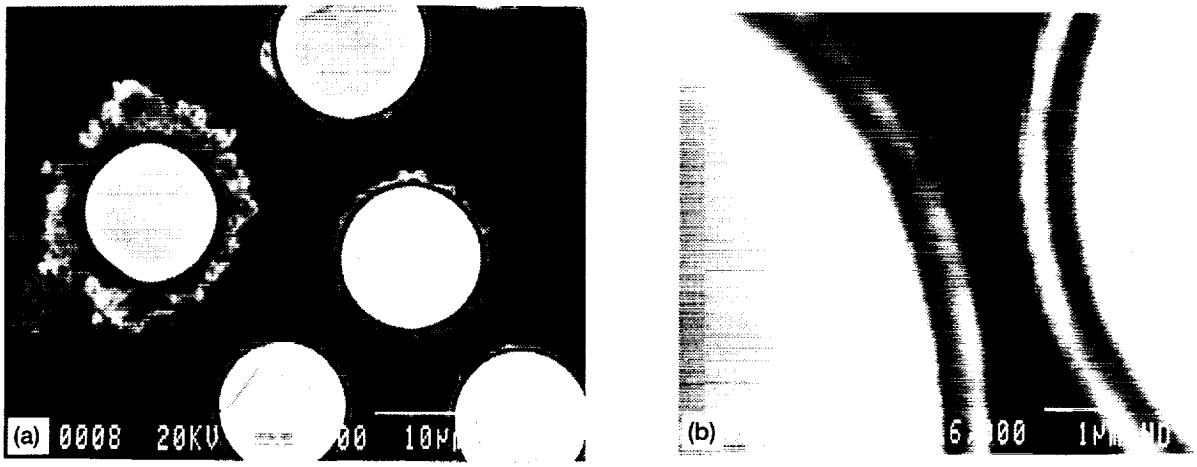

Figure 6.—SEM micrographs showing polished cross-section of Hi-Nicalon fibers with a duplex CVD BN/SiC coating. (a) Lower magnification. (b) Higher magnification.

TABLE I.—HI-NICALON FIBER-REINFORCED $Ba_{0.75}Sr_{0.25}Al_2Si_2O_8$ CELSIAN COMPOSITES (UNIDIRECTIONAL; 12 PLIES)

Sample number	Hot pressing conditions	Fiber coating	Fiber content, V_f	Density, (g/cm^3)	Phase from XRD
HI-NIC-BSAS-1-29-96	Less aggressive	BN/SiC	0.42	3.05	Monoclinic celsian
HI-NIC-BSAS-1-31-96	More aggressive	BN/SiC	0.42	3.09	Monoclinic celsian

from the polished surface of the hot pressed composite was similar to that shown in figure 3. Monoclinic celsian was the only crystalline phase present and the undesired hexacelsian phase was not detected from XRD. This implies that the desired, thermodynamically stable, monoclinic celsian phase is formed *in situ*, from the mixed oxides precursor, during hot pressing of the composite. Doping with SrO facilitates (refs. 9 and 10) the formation of monoclinic celsian in the matrix. In a recent study (ref. 17), a minor amount of hexacelsian phase has been detected in the matrix using Raman microspectroscopy.

SEM micrographs taken from the polished cross-section of the unidirectional hot pressed composite are shown in figure 7. Uniform fiber distribution and good matrix infiltration within the fiber tows is evident. Some of the filaments are of irregular shape rather than having circular cross-section. The manufacturer reports an average fiber diameter of ~14 μm, but a large variation in the diameter of the filaments within a fiber tow can be seen. The BN/SiC surface coating has been detached from some of the fibers during metallography or composite processing. Debonding or loss of the fiber coating may lead to adverse reactions between the fibers and the oxide matrix at high temperature resulting in strong fiber-matrix bonding.

TEM micrographs showing the fiber/matrix interface region of the Hi-Nicalon/BN/SiC/BSAS composite are presented in figure 8. The BN coating consists of four distinct layers as seen in figure 8(a). From EDS analysis, the fiber/BN interface was found to be rich in C along with the presence of Si and O. The SiC overcoating was often found to be missing from the BN/matrix interface, but was intact in this particular case. A crack running along the fiber/BN interface may also be seen. The BN coating, when viewed via the TEM, did reveal an internal reaction. Some areas show no evidence of coarsening (fig. 8(a)) while others show varying amounts of reaction. Figure 8(b) shows coarsening of three BN layers, from outside in towards the fiber, during hot pressing of the composite. However, the fiber/BN interface and the inner BN layer remain unchanged. EDS spectra obtained from the coarsened region (fig. 9(a)) show no obvious

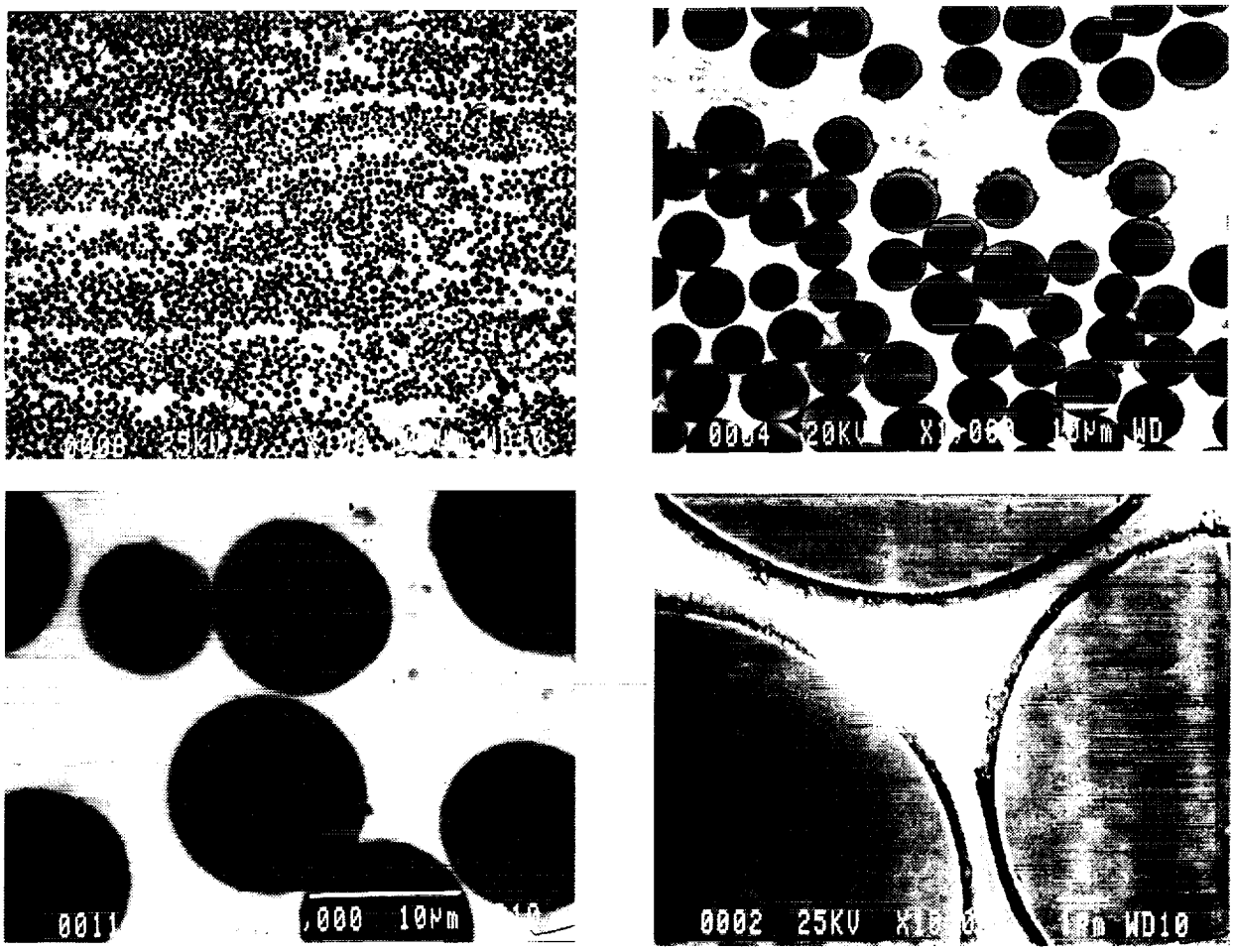

Figure 7.—SEM micrographs showing polished cross-section at different magnifications of a unidirectional Hi-Nicalon/BN/SiC/BSAS composite.

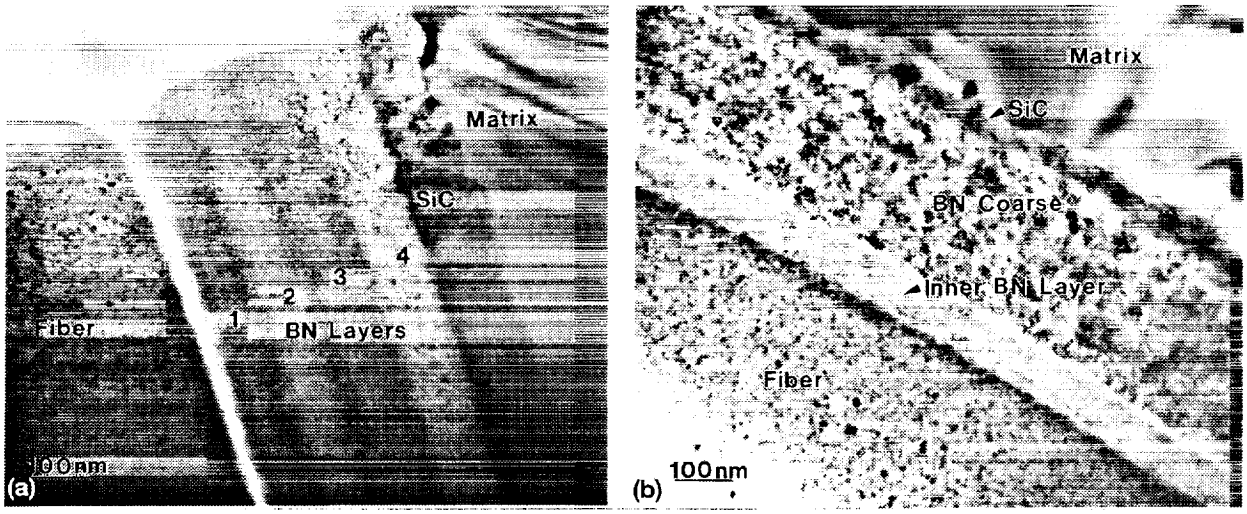

Figure 8.—TEM bright field image from cross-section of Hi-Nicalon/BN/SiC/BSAS composite showing (a) multiple BN layers and (b) coarsening of the outer three BN layers. The inner BN and the SiC interface layers are unaffected.

chemical changes in the various BN layers. Corresponding microdiffraction patterns (fig. 9(b)) show coarsening to the point where individual diffraction spots are seen. It may be pointed out that reaction through all four BN layers or visible degradation of the fiber was never observed. The SiC layer remains apparently unaffected.

Typical stress-strain curves recorded in three-point flexure for the unidirectional BSAS matrix composites reinforced with BN/SiC-coated Hi-Nicalon fibers hot pressed at two different temperatures are shown in figure 10. The fiber volume in the composites was ~42 percent. Both the composites show graceful failure. The values of yield stress, σ_y, yield strain, ε_y, elastic modulus, E, ultimate strength, σ_u, and ultimate strain, ε_u, of the composites hot pressed at two different temperatures are given in table II. Values of yield stress of 400 to 470 MPa, ultimate strength of 850 to 960 MPa and elastic modulus of 160 to 170 GPa were observed. The measured elastic modulus is in good agreement with a value of 169 GPa, calculated from the rule-of-mixtures ($E_c = V_m E_m + V_f E_f$ where V is the volume fraction and the subscripts c, m, and f refer to the composite, matrix, and fiber, respectively) using $E_m = 96$ GPa (ref. 13) and $E_f = 270$ GPa (refs. 14 and 15). Values of yield strain and the ultimate strain are ~0.26 to 0.28 percent and ~0.6 to 0.8 percent, respectively.

SEM micrographs of fracture surfaces of the two composites, after the three-point flexure tests, are shown in figure 11. Extensive long lengths of fiber pullout are observed indicating toughening behavior. An SEM micrograph of the polished cross-section of a Hi-Nicalon/BN/SiC/BSAS composite, formed under more aggressive processing conditions and flexure tested at room temperature under ambient conditions, is presented in figure 12. Debonding at the fiber-matrix interface and crack deflection around the reinforcing fibers is observed indicating a tough composite. The results of this study clearly indicate that reinforcement of BSAS matrix with Hi-Nicalon fibers having a duplex BN/SiC coating results in a strong and tough composite.

Figure 9.—EDS spectra from the coarsened BN region (a) and the corresponding microdiffraction pattern (b).

TABLE II.—ROOM TEMPERATURE MECHANICAL PROPERTIES OF HI-NICALON FIBER-REINFORCED $Ba_{0.75}Sr_{0.25}Al_2Si_2O_8$ CELSIAN COMPOSITES UNIDIRECTIONAL; 12 PLIES)

Sample number	Fiber coating	Fiber content, V_f	Elastic modulus, E (GPa)	Yield stress, $_y$ (MPa)	Yield strain, $_y$ (%)	Ultimate stress, $_u$ (MPa)	Ultimate strain, $_u$ (%)
HI-NIC-BSAS-1-29-96	BN/SiC	0.42	168 159	468 405	0.283 0.258	958 850	0.661 0.801
HI-NIC-BSAS-1-31-96	BN/SiC	0.42	170 165	436 464	0.264 0.283	862 960	0.583 0.771

*Measured in 3-point flexure.

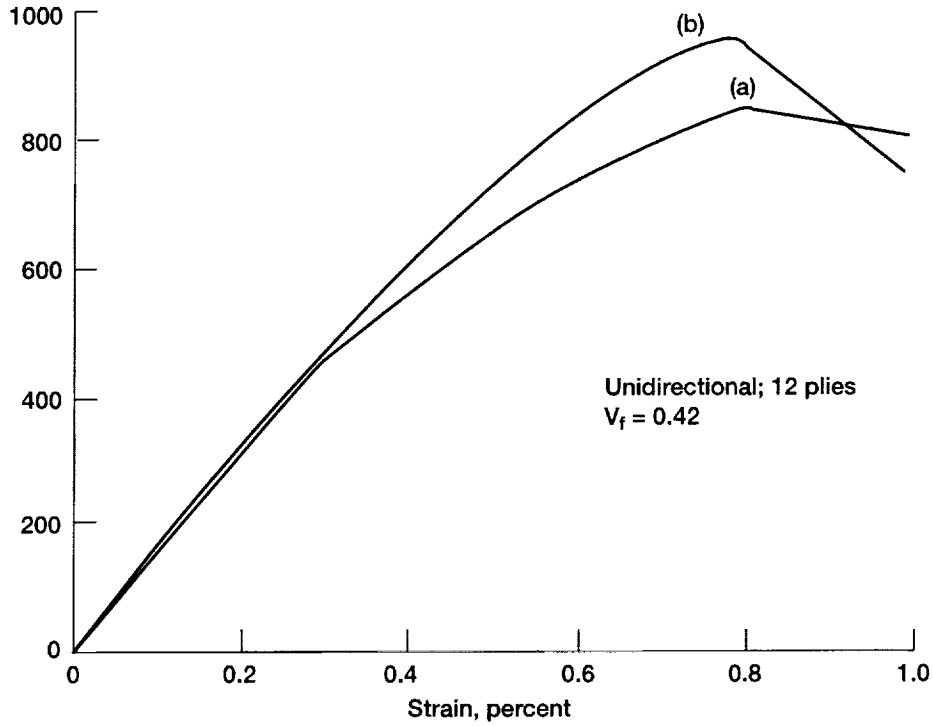

Figure 10.—Stress-strain curves recorded in 3-point flexture for unidirectional Hi-Nicalon/BN/SiC/BSAS composites hot pressed under (a) less and (b) more aggressive conditions.

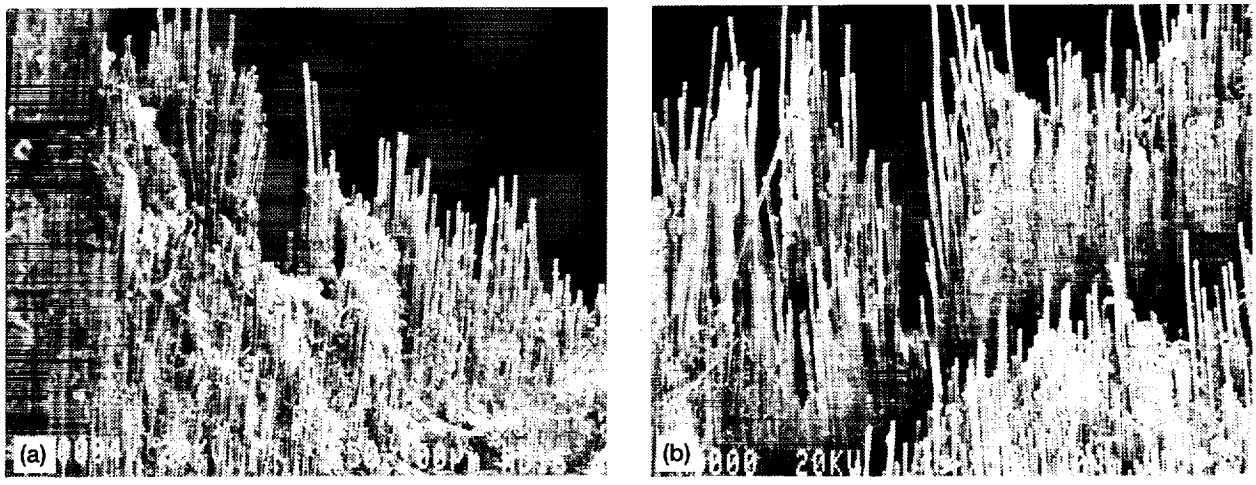

Figure 11.—SEM micrographs from the fracture surfaces of unidirectional Hi-Nicalon/BN/SiC/BSAS composites showing extensive fiber pullout; composites hot pressed under (a) less and (b) more aggressive conditions.

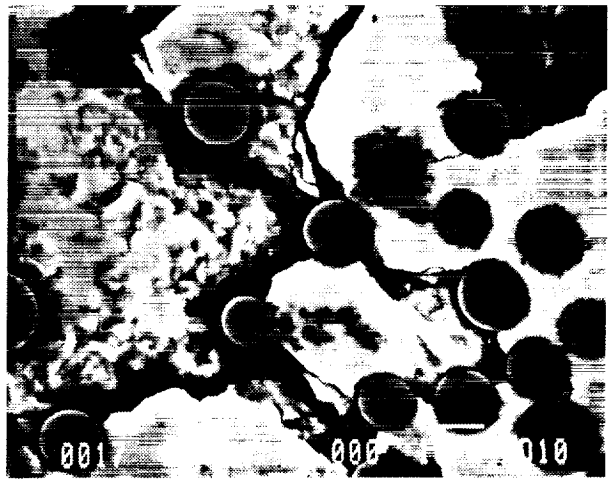

Figure 12.—SEM micrograph from the polished cross-section of a unidirectional Hi-Nicalon/BN/SiC/BSAS composite after the flexure test.

SUMMARY OF RESULTS

Hot pressing of mixed powder of $0.75BaO\text{-}0.25SrO\text{-}Al_2O_3\text{-}2SiO_2$ composition, obtained from calcination of metal carbonates and oxides, results in monoclinic celsian with complete absence of the undesirable hexacelsian phase. A method has been developed for the fabrication of small diameter multifilament tow fiber-reinforced ceramic matrix composites. BN/SiC-coated Hi-Nicalon fiber-reinforced monoclinic celsian composites have been produced by impregnation of the matrix slurry into the fiber tows, winding the tows on a drum, cutting and stacking of the prepreg tapes, and hot pressing. Strong, tough, and almost fully dense unidirectional composites having ~42 vol % of fibers were obtained. These composites exhibited graceful failure with extensive fiber pull out in a three-point flexure test. The yield stress of ~400 to 470 MPa and ultimate strength as high as 960 MPa have been observed. The yield strain was ~0.26 to 0.28 percent. The Young's modulus of the composite was measured to be ~165 GPa.

CONCLUSIONS

It may be concluded that reinforcement of the monoclinic celsian with BN/SiC-coated Hi-Nicalon fiber results in strong, tough, and almost fully dense composites.

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13. ABSTRACT ***(Maximum 200 words)***

A method has been developed for the fabrication of small diameter, multifilament tow fiber reinforced ceramic matrix composites. Its application has been successfully demonstrated for the Hi-Nicalon/celsian system. Strong and tough celsian matrix composites, reinforced with BN/SiC-coated Hi-Nicalon fibers, have been fabricated by infiltrating the fiber tows with the matrix slurry, winding the tows on a drum, cutting and stacking of the prepreg tapes in the desired orientation, and hot pressing. The monoclinic celsian phase in the matrix was produced *in situ*, during hot pressing, from the $0.75BaO\text{-}0.25SrO\text{-}Al_2O_3\text{-}2SiO_2$ mixed precursor synthesized by solid state reaction from metal oxides. Hot pressing resulted in almost fully dense fiber-reinforced composites. The unidirectional composites having ~42 vol % of fibers exhibited graceful failure with extensive fiber pullout in three-point bend tests at room temperature. Values of yield stress and strain were 435±35 MPa and 0.27±0.01 percent, respectively, and ultimate strengths of 900±60 MPa were observed. The Young's modulus of the composites was measured to be 165±5 GPa.

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