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Effect of Nicalon SiC fibre heat treatment on short fibre reinforced β-SiAlON ceramics

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Abstract

SiC Nicalon fibre yarn was heat-treated at elevated temperature in a gas pressure furnace under CO atmosphere. Weak surface coating is essential for ceramic matrix composite (CMC) reinforcement. Therefore Nicalon SiC fibres were coated after CO heat treatment and then used for β-SiAlON ceramic reinforcement. The heat treated fibres were chopped about 1–2 mm, and β-SiAlON $z = 1$ starting powders were prepared with conventional ball milling. The SiAlON starting composition and the short fibres were mixed with the certain amount of water to obtain a plastically formable mud. This mud was unaxially cold-pressed to form green bodies and to decrease water content. The green bodies were hot pressed at elevated temperatures for half an hour to produce CMC samples. Vickers hardness test showed that heat-treated fibre reinforcement of β-SiAlON composites provided higher fracture toughness. Uniform fibre distribution, fibre coating, matrix densification and phase transformation were examined by SEM and XRD analysis.

Keywords: β-SiAlON ceramics; Nicalon SiC fibres; CO heat-treatment; Hot-pressing

1. Introduction

Ceramic matrix composites (CMCs) have potential for being structural materials for high-temperature applications because of their higher refractoriness properties compared with metal alloys, and higher fracture toughness compared with monolithic ceramics.[1–9] Carbon fibres and silicon carbide fibres are both promising fibres to reinforce SiAlON composites for structural applications instead of metallic parts.[10–12] Toughening of CMCs depends upon properties of reinforcing fibres which have ability of arresting crack propagation within the ceramic matrices. This depends upon the 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.[11–13] Therefore, the mechanical performance of CMCs is very sensitive to the properties of the fibre–matrix interface. For ceramic matrix composite production, β-SiAlON ceramics is one of the best materials compared with other ceramic matrices because of elongated grains which come from solution-precipitation sintering mechanism.[12]

SiC-based fibres have a better resistance to oxidation than other ceramic fibres. However, the tensile strength of Nicalon SiC fibres decreases dramatically associated significant with weight loss as the temperature is raised beyond 1200 °C. Coustumer[14] has reported that the polycarbosilane based NL-200 fibres consist of three microstructural constituents: β-SiC grains, turbostratic carbon stacks, and amorphous silicon oxycarbide $SiO_{1.15}C_{0.85}$. Mah et al.[15] investigated the degradation of Nicalon SiC fibres in different atmospheres under atmospheric pressure at 1200 °C. At high temperatures, changes in the fibre chemistry occurred resulting in weight loss (CO evolution) and SiC grain growth. They concluded that the strength reduction observed in the heat-treated fibres was due to evolution of CO gas from the fibre. The oxidation of excess carbon from the fibre leaves surface pits, resulting in drastic loss of strength. Morimoto and Ogasawara[16] have developed a method to measure tensile strength of Nicalon (TM), Hi Nicalon (TM), and Hi Nicalon type S (TM) monofilaments of variable diameters.

Jaskowiak and DiCarlo[17] examined the effect of pressure on the thermal stability of Nicalon fibres. They heat treated Nicalon fibres under three different pressures, vacuum (10^{-9} MPa), ambient pressure (0.1 MPa) and an over pressure (138 MPa), at temperatures ranging from 1000 to 1500 °C. In the over pressure conditions, they found that the fibres were able to withstand 1500 °C for one hour without strength or weight change. It

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is believed that this observed retention of strength was due to the stabilisation of the Si–C–O containing regions of the fibre. At high temperatures the over pressure inhibited the evolution of CO and SiO gas from the fibre surface. The thermal degradation mechanism follows a two step procedure which can be summarised as an endogenous (i.e. self-induced) oxidation mechanism, providing gaseous SiO and CO. They observed that the fibres underwent the endogenous oxidation mechanism as described by reactions (1.1 and 1.2),

In carbon furnace conditions (10 Pa partial pressure of CO), following reaction (1.1) is normally observed.

$$SiC + CO \rightarrow SiO + 2C \quad (1.1)$$

Another possible reaction in a higher pressure CO atmosphere at high temperatures is:

$$2CO \rightarrow CO_2 + C \quad (1.2)$$

Heat treatment in a CO atmosphere offers an attractive route for obtaining carbon coatings on the fibres if reactions (1.1) or (1.2) take place. This is often necessary for specific purposes to assist fibre/matrix compatibility or achieve improved toughness.

In this study, mechanical and surface properties of SiC Nicalon fibres were changed by high pressure CO heat-treatment. As a result of heat treatment, fibres strength increased and a thin carbon coating was formed on the fibres. The aim of this study ability to reinforce β-SiAlON matrices with the chopped heat treated fibres. A care must be taken for powder processing and hot-pressing conditions to end up best SiC fibre reinforced β-SiAlON composites.

2. Experimental

Nicalon SiC fibres, Type NL-207, were supplied from Nippon Carbon, Japan, 500 fibre filaments per yarn with 14 μm average diameters. After burning out organic binder from the fibre surfaces at 500 °C, the fibre yarn was rolled up onto a graphite reel and put into a graphite crucible. The crucible containing the reel was put in a home-made gas pressure furnace (GPS) with maximum limit 50 bar pressure for heat treatment. The fibres were heat-treated at temperature range 1000–1700 °C for 30 min under CO pressure. The aim of heat treatment was to improve as received fibre properties. At beginning, one atmosphere argon, nitrogen and CO were used to perform heat-treatment in GPS, then higher CO gas pressures were used for SiC fibre heat-treatment. The CO gas pressure was 15 bar at ambient temperature and reached aprrox. 50 bar at the heat-treatment temperatures.

Hot-pressing conditions of β-SiAlON $z = 1$ starting powders were developed to obtain highest density before fibre addition as described in previous study.[12] Sintering additives, 2% MgO, 7% Y_2O_3 and 8% Sm_2O_3 were added to β-SiAlON $z = 1$ starting powder composition which contains Si_3N_4, Al_2O_3 and AlN. The powder mix including sintering additives was ball milled within isoproponal alcohol for 24 h and dried. The dried powder mix was hot-pressed at elevated temperatures within a graphite die to examine densification behaviour of β-SiAlON ceramics. The best sintering conditions of monolithic β-SiAlON $z = 1$ ceramic was determined elsewhere.[12] Therefore in this study, it was focused on densification behaviour of short fibre reinforced β-SiAlON ceramics rather than monolithic β-SiAlON ceramics. The heat-treated fibres were chopped and mixed with β-SiAlON $z = 1$ starting powders and isoproponal alcohol to prepare plastic formable mixture. The mixture was mixed until plastic consistency mud was obtained. This thick paste mud was cold pressed to form green bodies and to expel excess liquid. The short fibre containing green bodies were unaxially hot-pressed within a graphite die at temperatures between 1550 and 1700 °C.

After hot-pressing, composite samples were polished and examined by optical Nikon Eclipse L 150A. Scanning electron microscopy (SEM), JEOL JSM-6700F was only used for SiC fibre surface examination. Mercury density measurements were carried out according to Archimedean Principle. Micro hardness test, Future Tech-Corp. FM-700, was used for hardness and fracture toughness test. The Vickers hardness, Hv and fracture toughness K_C were calculated using expression of Marshall and Lawn.[18] Tensile strength of monofilament fibre was applied to both as received and heat treated fibres because of bundle gripping difficulty. At least 10 filaments were tested to determine tensile strength of the each heat-treatment temperature. An image analysis system was used to measure fibres diameter and coating thickness. XRD pattern of the existence phases within the resulting CMCs were obtained by using RIGAKU D/MAX/2200/PC.

3. Results and discussion

3.1. Fibre heat treatment

Nicalon SiC fibres contain silconoxcarbide and free carbon in addition to main SiC phase. Therefore with the effect of heat and carbon monoxide partial pressure, internal and external reaction was probably occurred during heat treatment described in Eq. (1.2). Since the fibres were degraded under one atmosphere of argon and nitrogen heat-treatment, it was focused CO heat-treatment since the significant strength retention were obtained with one atmosphere CO heat treatment. As well as one atmosphere pressure, high pressure carbon monoxide heat treatments were carried out in a carbon element gas pressure furnace. As a result of pressurised CO heat-treatment, surprisingly a thin carbon layer (∼1 μm) was formed on the fibre surfaces and strength increase was observed. Fig. 1 shows this surface change at 1600 °C and 1700 °C heat treatment. As temperature increased no surface reaction observed up to 1300 °C after this temperature a beneficial shell started to form and thicken on the fibre surface up to 1600 °C. After 1600 °C, the fibres started to degrade sharply and over heat-treatment took place at 1700 °C as shown Fig. 1b. Although a thickest shell was formed on the fibre surfaces no significant fibre strength left at 1700 °C heat treatment.

Many researchers have observed that 75% fibre strength can be retained in one atmosphere CO heat-treatment at temperatures up to 1300 °C, but no one has reported a strength increase

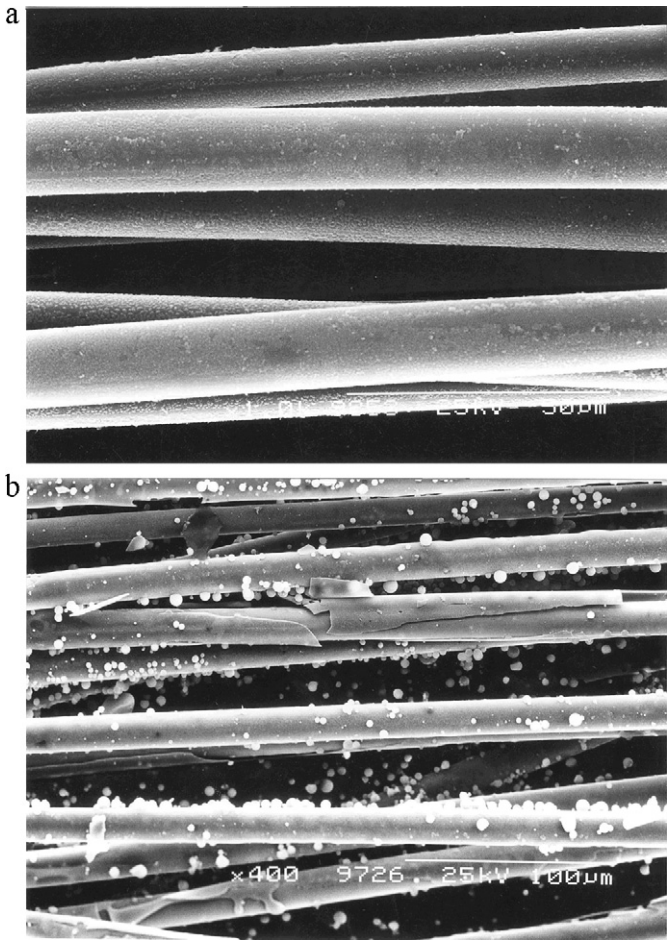

Fig. 1. Surface coating after Nicalon SiC fibre heat treatment at pressurised CO atmosphere (a) 1600 °C, (b) 1700 °C (over heat-treated).

neither in one atmosphere CO heat treatment nor in pressurised CO heat treatment.[13–17] In this study therefore, high pressure CO heat treatment has applied to the polymer precursor Nicalon SiC fibres. For high pressure heat treatment, at least it was expected that the as-received fibre strength might be retained, but surprisingly significant amount of strength increase was observed for all temperatures except 1700 °C heat treatment. In fact, at each temperature up to 1600 °C, an increase in strength was measured. As shown in Table 1 the as-received fibre strength was 2.8 GPa. The strength of the fibres after heat treatment at 1000 °C fibre was 3.7 GPa which was 32% higher than the as-received value. At 1100 °C the heat-treated fibres reached a maximum strength of 4.2 GPa which represented a 50% overall strength increase. At this point the strength started to decrease as weight gains were observed associated with shell formation. However, between 1300 and 1400 °C the strength of the fibres started to increase again, with weight gain and shell formation, reaching another peak at 1500 °C representing an increase of 42% compared with the strength of the as-received fibres. Above the 1500 °C, weight gain as result of carbon deposition was also increased sharply in spite of fibre degradation at 1700 °C.

Table 1
Strength increase as a result of heat treatment of SiC fibres in 45 bar CO pressure for 30 min.

Temperature (°C)	CO pressure (bar)	Weight gain (%)	Fibre strength (GPa)
As supplied	–	–	2.8
1000	41	0	3.8 ± 0.14
1100	43	0	4.2 ± 0.16
1200	44	0	4.1 ± 0.15
1300	45	0	3.1 ± 0.12
1400	45	0.6	3.8 ± 0.15
1500	46	1.5	4.1 ± 0.20
1600	46	6.5	3.9 ± 0.18
1700	47	18.5	NA

Above 1600 °C, fibre strength decreased sharply because of chemical degradation and the increasing fibre shell thickness causing decrease in fibre diameter. The microstructure of the fibre becomes porous under the shell at 1700 °C resulting in strength lost. The use of high pressure CO suppressed degradation up to 1600 °C but after this point, the fibre degraded suddenly and measurement of fibre strength at 1700 °C became impossible.

3.2. Short Nicalon fibre reinforced β-SiAlON ceramics

In previous works[12] different combination of additive compositions were used to densify CO heat-treated continuous fibre reinforced β-SiAlON $z = 1$ ceramics. Hot-pressing was the sintering method to densify fibre reinforced β-SiAlON composites. The study revealed that heat-treated fibre within the β-SiAlON matrices eased the densification and increased composite strength compared with as received continuous fibre reinforcement. The most successful oxide additive combination was 2% MgO, 7% Y_2O_3, 8% Sm_2O_3 to densify β-SiAlON composite. This composition provided higher densification at low temperature (1550 °C) and resulted in higher strength and fracture toughness values 670 MPa, 13 MPa $m^{1/2}$ respectively compared with other composition for the continuous fibre reinforcement.

Continuous fibre reinforcement provides higher strength along the fibre direction but across the fibre direction gives poor mechanical properties. In order to avoid disadvantage of the continuous fibre reinforcement, short fibre reinforcement was needed to provide isotropic mechanical properties. Therefore in this study, heat treated fibres were cut into couple of mm and mixed with the most successful β-SiAlON composition. 5, 10, 15 vol.% of heat treated short fibres were added the β-SiAlON starting powders and mixed in a mortar with isopropanal alcohol so that fibre containing ceramic mud was able to be obtained. Thereby, short fibres within the ceramic paste were isotropically oriented. After cold-pressing of the ceramic mud, the dried green bodies were embedded in BN powder inside the graphite die and unaxailly hot-pressed at elevated temperatures to provide matrix densification. During hot-pressing, liquid phase started to form at about 1350 °C and increased as the temperature was increased. During liquid phase sintering of the β-SiAlON matrices, alpha silicon nitride particles were dissolved in liquid phase and long grain β-SiAlON grains were precipitated as shown in Fig. 2.

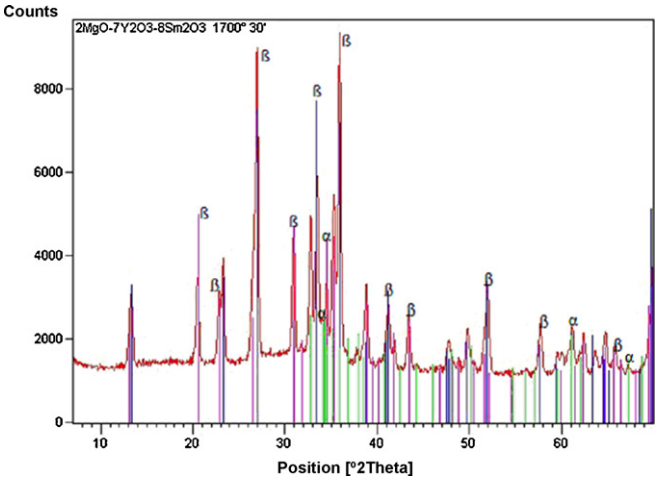

Fig. 2. X-ray analysis of β-SiAlON composite hot-pressed at 1700 °C.

Density measurement, hardness and fracture toughness test have been applied to the hot-pressed ceramic matrix composite samples. Initially, density measurements of the samples were carried out to examine densification behaviour. Densities of short non-heat-treated fibre reinforced composites dropped slightly as the fibre content increased. However in the case of fibre heat-treatment, the surface coating as a result of CO heat treatment behaved as solid lubricant within ceramic matrix and helped densification during rearrangement after liquid phase formed. Because of that, heat-treated fibre addition did not dropped sample densities as much as non-heat-treated fibres as determined in previous study.[12]

Optical micrographs of the hot-pressed short fibre reinforced β-SiAlON ceramic composites are shown in Figs. 3–6. The pictures shown in Fig. 3a–b have been obtained from 5% short fibre reinforced β-SiAlON composites after hot-pressing at 1550 °C. The homogeneous fibre distribution is shown in Fig. 3a but some pores are present at the ceramic matrix which means that densification has not been completed and fibre/matrix interface are visible in Fig. 3b that is to say carbon coatings stay there. Fig. 4a shows uniform fibre distribution of 10% short fibre reinforced β-SiAlON composite hot-pressed at 1600 °C and Fig. 4b shows high magnification of this composite for interface examination. From that picture, fibre diameter (17.81 mm) and interface thickness (about 1 μm) resulting from carbon coatings are clearly shown. The fibre coating gives thick interface which will be sacrificed during fibre pull-out after fracture.

10% CO heat-traded short fibre reinforced β-SiAlON composites hot-pressed at 1650 °C are shown In Fig. 5a–b. As shown in Fig. 5a, a uniform fibre distribution and dense matrix have been achieved at this temperature. Although this is the most desirable structure for ceramic matrix composites it is not enough to give high mechanical properties unless weak interface layer is present as shown in Fig. 5b. Otherwise, strong interfaces in CMCs cause catastrophic fracture like monolithic ceramic. At this temperature, fibres have not been degraded and the strengths of the fibres have been kept during hot-pressing. Thus, all desired properties; maximum densification, uniform distribution, weak interface and non-degraded fibres have been

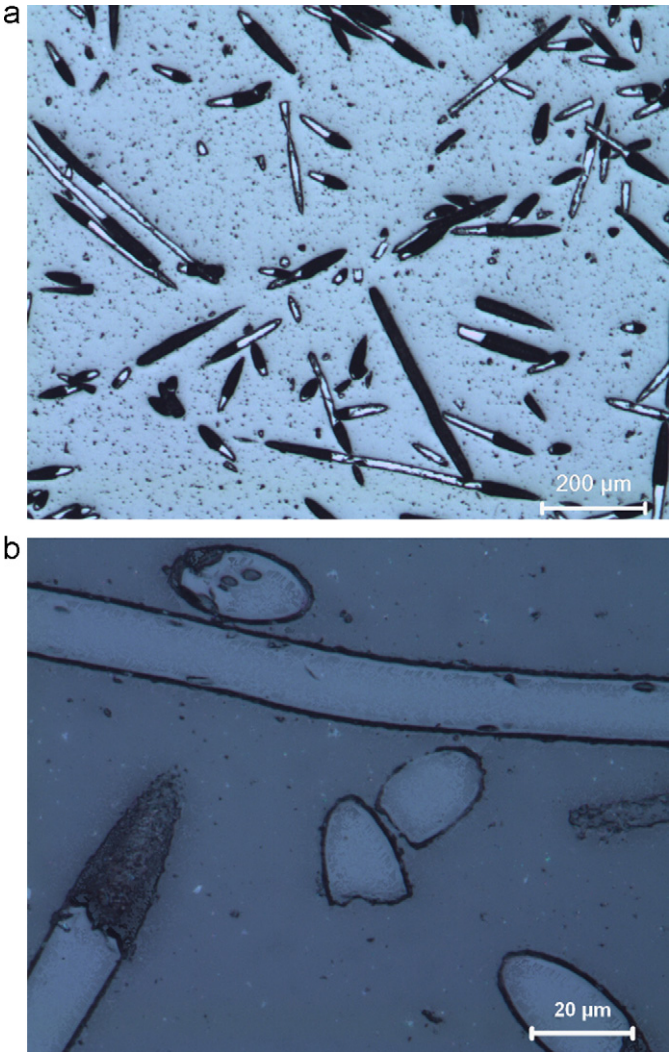

Fig. 3. 5% short fibre reinforced β-SiAlON composites hot-pressed at 1550 °C.

obtained at this temperature. As a result of high pressure CO heat treatment, weaker C coating was occurred compared with SiC fibre. When load is applied, a crack initiates and propagates up to fibre/matrix interface. If the interface is weak and the fibre is strong, the crack is deflected at interface and the load transfer to the fibres and toughening is achieved as in this study. With a strong fibre grip within matrix, the propagating crack moves across the fibres and toughening does not occurs.

In Fig. 6a–b, microstructures of 15% CO heat-treated short fibre reinforced β-SiAlON composites hot-pressed at 1700 °C are shown. Homogeneous fibre texture has been achieved as a shown in Fig. 6a for the 15% fibre addition but microstructure of the fibres has been chemically degraded as a shown in Fig. 6b. In spite of BN encapsulation, hot-pressing pressure and CO environment coming from graphite, the fibres have degraded at 1700 °C and lost their strength according to degradation mechanism as explained previously.[13–17] These hot-pressing conditions suppress fibre degradation and delays degradation temperatures from 1200 °C to 1650 °C but beyond this temperature endogenous reaction starts within the fibres and fibre loose their strength.

Table 2
Mechanical property changes depending on fibre rate and hot-pressing temperatures.

Temperature (°C)	Short heat-treat fibre reinforced β-SiAlON $z = 1$ ceramics				
	Properties	Monolithic	5%	10%	15%
1550 °C	Density, g cm^{-3}%	3.22	3.15	3.08	3.03
	Relative density, %	94	93	92	92
	Hardness, GPa	18.50	16.20	14.00	12.80
	Fract. tough., MPa $m^{1/2}$	5.6	6.6	7.4	7.8
1600 °C	Density, g cm^{-3}	3.35	3.28	3.23	3.18
	Relative density, %	98	97	97	96
	Hardness, GPa	19.20	17.20	14.90	13.75
	Fract. tough., MPa $m^{1/2}$	5.6	7.5	8.6	9.5
1650 °C	Density, g cm^{-3}	3.39	3.35	3.27	3.20
	Relative density, %	99	99	98	97
	Hardness, GPa	19.70	17.00	15.40	14.20
	Fract. tough., MPa $m^{1/2}$	6.0	7.6	8.4	9.8
1700 °C	Density, g cm^{-3}	3.41	3.36	3.28	3.24
	Relative density, %	100	99	98	98
	Hardness, GPa	20.60	16.80	14.20	13.25
	Fract. tough., MPa $m^{1/2}$	6.2	6.4	6.3	6.2

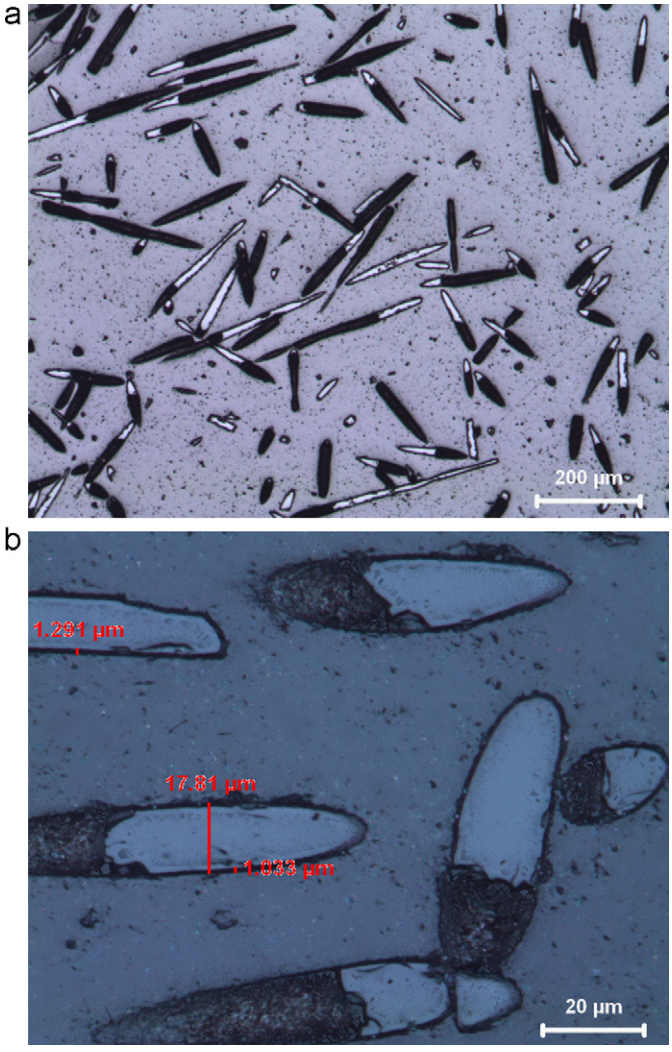

Fig. 4. 10% short fibre reinforced β-SiAlON composites hot-pressed at 1600 °C.

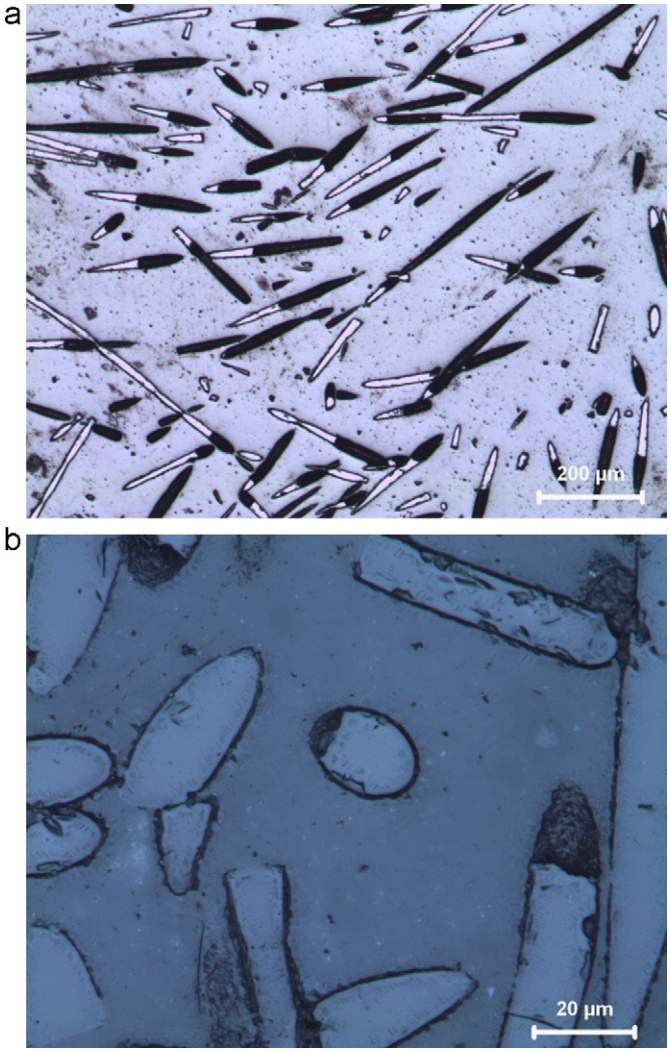

Fig. 5. 10% short fibre reinforced β-SiAlON composites hot-pressed at 1650 °C.

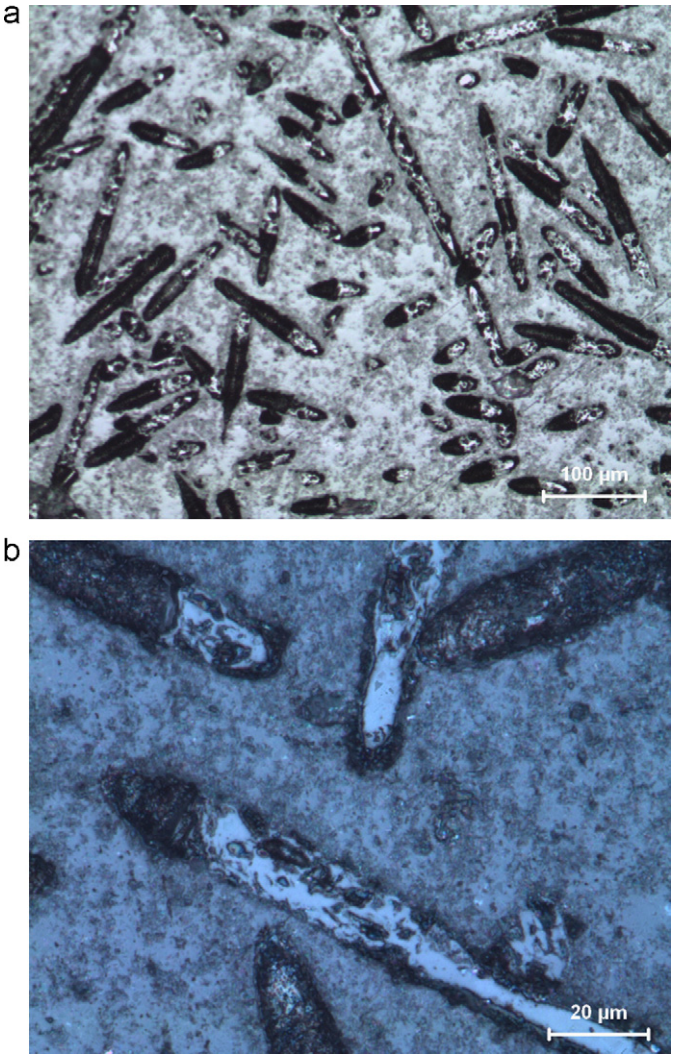

Fig. 6. 15% short fibre reinforced β-SiAlON composites hot-pressed at 1700 °C.

Micro Vickers indentation hardness test and associated with fracture toughness test were carried out for both monolithic β-SiAlONs and their heat treated fibre reinforced composites The results were tabulated in Table 2 depending on hot-pressing temperatures and density values. In general, fibre addition causes slight decrease in density but dramatic decrease in hardness and moderate increase in fracture toughness. The fibre density is 2.55 g cm^{-3} and theoretical density (TD) of this composition is about 3.42 g cm^{-3}. Therefore, fibre addition to the fully dense matrix drops total theoretical density slightly and gives 99–97% relative density for the 1650 °C hot-pressed sample. When liquid phase forms at the grain boundaries during hot-pressing, the fibres hinder particle re-arrangement and lead to low densification. When these heat-treated fibres were used as reinforcing agent the weak fibre coating eased particle movement during hot-pressing. Since hardness is very sensitive to the density changes in ceramics or CMCs, even though significant density changes have not been observed depending on fibre ratio increase, the hardness values have dramatically dropped for the composites as the reinforcement phase increase. Hence, fracture toughness values should be examined in CMCs rather than hardness.

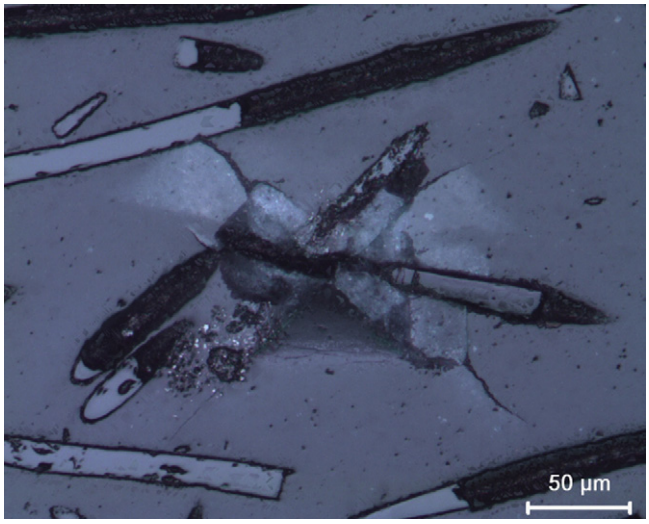

Fig. 7. Crack hindering mechanism occurring in the β-SiAlON composites after hardness test.

As shown in Fig. 7, propagating crack from indented diamond edge is hindered or deflected by the carbon coated fibres. When the crack deflected, extra energy absorbed and catastrophic fracture is prevented. If the extra load is applied after crack deflection, the weak interface coating will be sacrificed causing fibre debonding, modulus will be transferred to the fibres and fibre pull-out will be occurred. These sequential toughening mechanisms can be taken place for chopped CO heat treated Nicalon SiC fibre reinforced β-SiAlON ceramics as shown in Fig. 7.Hardness and fracture toughness behaviour are plotted in Fig. 8a–b. As shown in Fig. 8a, hardness values of all samples produced different hot-pressing temperatures go down as the fibre ratio increased. As explained above fibre addition drops densities slightly and low density causes low hardness. However dramatic hardness value drop is not expected with slight relative density changes. This is most probably because of soft interface coating of the fibres. Otherwise, it has been expected same hardness value drop for the monolithic β-SiAlON ceramics as the hot-pressing temperatures is decreased. 18.5 GPa Hv and 94 relative density have been obtained for the sample hot-pressed at 1550 °C without fibre reinforcement. This hardness value is quite high compared with composites having same relative density. In Fig. 8a, all hardness values drop with same behaviour except for sample hot-pressed at 1700 °C because of fibre degradation. Effect of fibre reinforcement on fracture toughness of β-SiAlON composites has been given in Fig. 8b. As the amount of fibre reinforcement has increased, the fracture toughness values of the composite samples have gradually increased. According to these results, It can be revealed that with the effect of heat treatment, the Nicalon SiC fibres has been self coated with carbon, short fibre reinforcement of the β-SiAlON ceramics has been carried out via hot-pressing route and finally toughening of the β-SiAlON matrices has been achieved by the heat-treated fibres. However, because of fibre degradation at high temperature hot-pressing, the toughness values have remained constant as fibre ratio is increased.

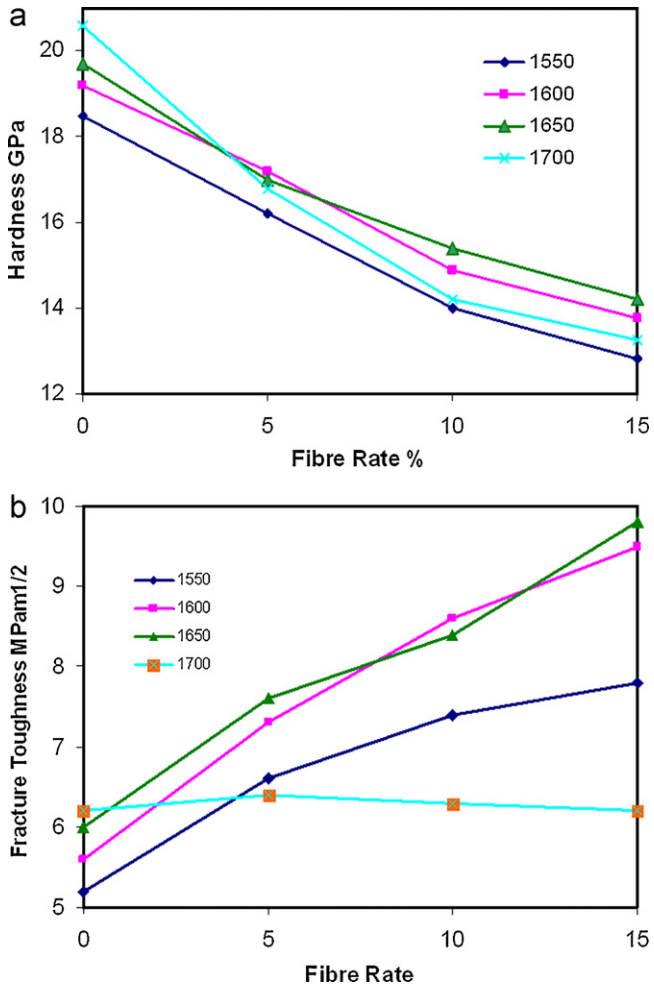

Fig. 8. Mechanical behaviours of β-SiAlON composites as a consequence of short fibre rate (a) hardness, (b) fracture toughness.

4. Conclusion

Nicalon SiC fibres successfully coated with self reaction under pressurised CO environment at elevated temperatures. This coating heat treatment somehow led to about 50% strength increase as well. Short heat-treated fibre reinforcements of β-SiAlON matrices were successfully achieved via hot-pressing route. Increasing hot pressing temperature (up to 1650 °C) and decreasing reinforcement fibres have resulted in density increase in β-SiAlON composite. All toughening mechanisms have been observed for the β-SiAlON matrix composites especially for the hot-pressed at temperature between 1600 and 1650 °C by means of heat-treated short SiC fibre reinforcement. Increasing fibre ratio has increased fracture toughness of the composite. On the other hand, as the fibre content is increased, hardness of the composites has dramatically dropped. Since the heat-treatment of the fibres at 1700 °C caused fibre degradation, these fibres were not used for composite fabrication.

As a result of high pressure CO heat-treatment, isotropic mechanical property improvements were provided for β-SiAlON matrix composites with the short fibre reinforcement. These new isotropic ceramic composite materials can be used as structural part instead of continuous fibre reinforced CMCs especially for high temperature applications.

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