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Effect of heat treatment in air on the thermal properties of SiC fibre-reinforced composite. Part 1: a barium osumilite (BMAS) matrix glass ceramic composite

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Abstract The thermal properties have been studied on a glass ceramic composite comprised of a barium osumilite (BMAS) matrix reinforced with SiC (Tyranno) fibres which has been subjected to a heat treatment in air in the range of 700–1,200 °C. Microstructural studies were carried out especially on of the interface between fibre and matrix. The presence of a carbon thin layer in the interface is a typical observation in SiC fibre-reinforced glass ceramic matrix composite systems. The microstructural evaluation and thermal properties showed a degradation of interfacial layer occurred at low heat treatment temperatures, (700–800 °C) this was attributed to the fact that, at those heat treatment temperatures the carbon rich layer formed during processing was oxidised away leaving voids between fibre and matrix, which were linked by isolated silicon-rich bridges. After heat treatment at higher temperatures of 1,000–1,200 °C, the thermal properties were retained or even enhanced by leaving a thick interfacial layer.

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Introduction

SiC fibres have been developed and used in a variety of reinforced glass ceramic composites over the last two decades. They are intended to provide reinforcement and to improve the performance of structural ceramics for high temperature applications [1].

Ceramics are brittle, have low fracture toughness and they fail in a catastrophic manner. The brittleness of the material results from sudden propagation of a crack under the applied stress. Thus, for the future application of ceramics at high temperatures it is necessary to develop ceramic fibres as reinforcement in suitable matrices. It is also necessary to understand the fracture behaviour of these composites and behaviour of the interface for design matters in order to optimise their mechanical properties.

One of the most promising glass ceramic matrix systems is barium osumilite (BMAS), first developed by Brennan et al. [2]. Thermal properties of BMAS glass ceramic matrix composites have been studied by Johnson et al. [3]. Although the use of such materials will be governed by the development of the suitable mechanical properties, the accurate thermo-physical properties are also needed such as thermal expansion, thermal diffusivity and conductivity.

During manufacturing stage, due to fibre and matrix reaction, a carbon layer is formed [4, 5] in SiC fibre-reinforced glass ceramic composites. This carbon rich layer has been observed in MAS, calcium aluminium silicate (CAS), barium aluminium silicate (BAS), barium magnesium aluminium silicate (BMAS) glass ceramic matrix composites [6–12].

The in-service performance of these composites depends on the environmental conditions. The thermal, microstructural and mechanical properties of the composites can be affected by the environment with time. Some studies carried out on glass ceramic matrix composites [13–16] show that the interface between fibre and matrix in the composites can be affected by the temperature of the environment. Oxidation occurs at the interface during the heat treatment with temperature and this results in a degradation of the mechanical properties of the composites. This was confirmed using mechanical tests such as tensile, three- or four-point bending, fibre pullout, creep, etc. These studies suggest that this behaviour can also be studied using thermal property tests for composites exposed to a heat treatment at various temperatures.

Limited amount of work has been carried out on the thermal properties of such composites. Hasselmann and co-workers have carried out the most comprehensive series of thermal diffusivity measurement on a wide range of composite systems [3, 17–24]. Thermal diffusivity or conductivity can be affected by the relative volume fraction of the constituents (fibre/matrix and porosity), the orientation of the fibres, the particular processing route chosen, and the structure of the fibre/matrix interface.

Heating to high temperatures can affect the thermal diffusivity of SiC fibre-reinforced composites [19], primarily because of the change in fibre/matrix interface in thermal exposure. A number of studies have indicated that thermal conductivity and diffusivity of composites can be affected by a thermal barrier resistance of the interface [18–21, 24]. The direction of heat flow also plays an important role in determining the effective diffusivity of composites in which there is fibre/matrix interface resistance. The greatest effect will be observed when heat flow is perpendicular to the fibre/matrix interface.

Oxidation resulting in the removal of carbon at interface behaved as a thermal barrier. When the carbon layer is oxidised, the thermal conductivity at the fibre/matrix interface occurs by gaseous conduction and resulted in lower thermal diffusivity in composites [18]. However, there have been relatively few reported observations of the effect of thermal exposure on the thermal properties. Certainly there is not any systematic investigation undertaken on this. It is possible that the measurement of the thermal diffusivity can be used as a qualitative non-destructive tool to determine the integrity of the fibre/matrix interfaces and to monitor microstructural changes occurring in the fibres or matrix.

In this work, a detailed microstructural characterization of the BMAS/SiC system has been presented along with subsequent changes in microstructure brought about by heat treatment. Changes in the microstructure of the specimen were monitored using standard microstructural characterisation techniques and physical properties such as thermal diffusivity of the specimen were measured before and after heat treatment.

Experimental

Materials

A 0/90° laminated SiC/BMAS composite was supplied by the National Physical Laboratory in UK. The composite was manufactured by Harwell Technology in England. The preparation route was for the Tyranno fibre tow to be desized in a furnace, taken through a slurry of glass frit, removed and wound on a wheel unit, allowed to dry for 20 min, cut and laid up manually in layers for hot pressing in a graphite die at ~1,200 °C for 10 min. It was then crystallised via a proprietary heat treatment, which involved in heating to a temperature not exceeding 1,300 °C [AEA Harwell Technology, private communication].

Thermal diffusivity and thermal expansion measurements were carried on the as-received material and after heat treatments in air at temperatures of 700 °C, 800 °C, 900 °C, 1,000 °C, 1,100°C, 1,200°C for times of ranging from 1 to 30 h.

Microstructural examination

X-ray diffraction analysis

X-ray diffraction studies were carried out to identify the phases present in the composites. These were performed using a PHILIPS E'XPERT diffractometer PW 3710 by using nickel-filtered copper K radiation with a graphite secondary monochromator. Scans at a step width of 0.005° for 2θ values from 20 to 70° were used on samples that were solid bulk plates 10 mm square by 2 mm thick. The diffraction traces obtained were compared against standard XRD patterns for a range of materials.

Optical and scanning electron microscopy

Sample preparations were taken in three stages; the specimens were ground on a Buehler DATAMET-microprocessor grinding/polishing system. METLAP 4 wheel with 9 µm METADI diamond slurry, with the wheel contra-rotating at 25 r.p.m, then on a Beuhler Metlap 2 wheel with 6 µm diamond slurry, the wheel contra-rotating at 120 r.p.m. After that polishing was done using a TEXMET platen with a wheel using 1 μm diamond slurry. Final polishing was carried out with colloidal silica. Each section takes 10 min and the polishing pressure was set to 1 N per sample and maintained at that level during preparation. The samples were then finished by washing with water for 1 min and dried. After preparation of the specimens, they were mounted on to an aluminium stub and initially coated with carbon or gold in order to prevent charging in the microscope. An Edwards coating system E 306A was used for coating. A conducting silver paste was used with the carbon-coated samples painted on the edge of the sample connecting it with the stub to improve electrical contact. The surface of the heat-treated samples were examined using Philips 525 scanning electron microscopes (SEM) operating at 20 kV with scanning facility operating with a computer programme-connected microscope.

Transmission electron microscopy

Discs of the same diameter as the electron microscope specimen holder (3 mm in diameter and 2 mm thick) were cut from the heat-treated specimens. The specimens were prepared for transmission electron microscopy (TEM) by a combination of mechanical polishing and ion beam thinning techniques. The specimens were initially ground on 1200 grit wet SiC abrasive until the thickness of the specimen was reduced to ~150 m. Further grinding was carried out until the specimen thickness was 70 μm. The foils were then transferred to a Gatan ion beam thinner. Several milling conditions were considered but the most suitable condition was found to be as follows; 5 kV ion beam energy 0.4 A current at 30, 25 and 16 impingement angle for 30 h.

After milling, the foils were then taken from the ion beam thinner and placed directly into the electron microscope for examination. The analytical electron microscopy was carried out using a Philips EM 400 and CM 20 operated at 120 kV and 200 kV, respectively. Both were equipped with an Energy dispersive spectroscopy (EDS) system, and the investigation was conducted using bright field, lattice imaging diffraction and micro diffraction techniques.

Thermal diffusivity measurement

Thermal diffusivity measurements were carried out using the laser flash method originally described by Parker et al. [25]. The thermal diffusivity equipment used at UMIST has been previously described by Taylor [26].

The specimens used in the measurements were in the form of 10 mm^2 plates with a thickness of approximately 2 mm. The specimens were cut from the composite plate by using a slow speed diamond saw. In order to ensure optimum absorption of the laser flash at the sample front surface and maximum emissivity for monitoring the transient temperature at the opposite face of the sample, the both faces of the sample were coated with a colloidal graphite film. Measurements were performed from 100 °C up to 1,000 °C with the 100 °C interval. At least three measurements were taken at each measured temperature and averaged value was taken for plotting of the graphs.

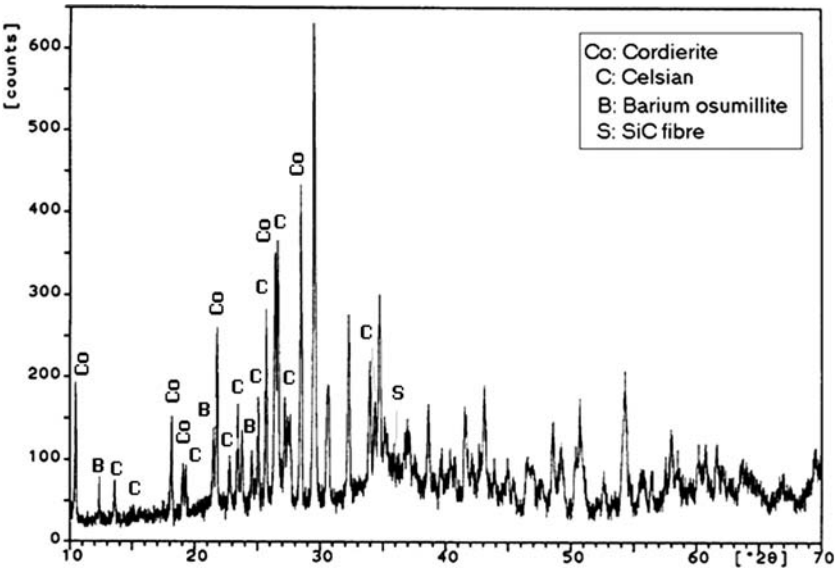


Fig. 1 X-ray diffraction spectrum of as-received material of BMAS/SiC CMC

Results

Characterisation of as-received material

To identify the phases present in the as-received material, the X-ray diffraction studies were carried out on the samples. Figure 1 shows a typical scan over the range $10 < 2\theta < 70$ from which three crystalline phases have been identified: Barium osumilite ($BaMg_2Al_3(Si_9Al_3O_{30})$), hexacelsian ($BaAl_3Si_2O_8$) and cordierite ($Mg_2Al_4Si_5O_{18}$).

Thermal properties

Thermal diffusivity measurements

Thermal diffusivity was measured over the temperature range 100–1,000 °C. There was no change noted between heating and cooling measurement runs. To assess material variability, measurements were taken on four samples cut from different regions of the as-received plate. The results are plotted in Fig. 2 and show a scatter of ±3%. The median value obtained from this curve will be included, for comparison purposes in all future diffusivity/temperature plots.

Changes in thermal diffusivity were noted for the samples heat treated for times as short as 1 h at temperatures of 700 and 900 °C. However for higher temperature anneals at 1,000–1,200 °C, a negligible change was noted. The thermal diffusivity data after 1 h heat treatment in the range 700–1,200 °C are presented in Fig. 3. It can be seen that the greatest degradation in thermal diffusivity is noted for the sample heat treated at 700 °C with the value of 4.4×10^{-3} cm^2 s^{-1} was measured at 100 °C and fairly constant value 3.44×10^{-3} cm^2 s^{-1} of being recorded above 350 °C.

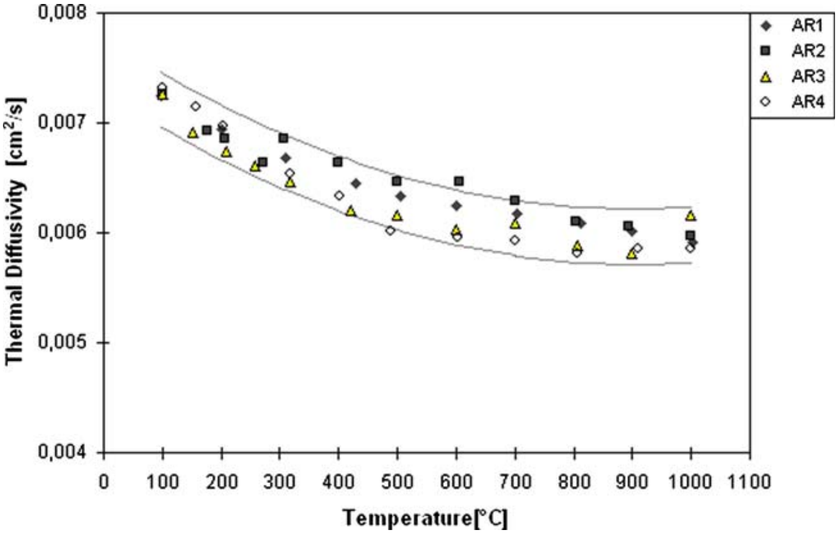


Fig. 2 Measurement of thermal diffusivity of BMAS/SiC as-received materials. AR: Samples cut from different regions of the as-received composite

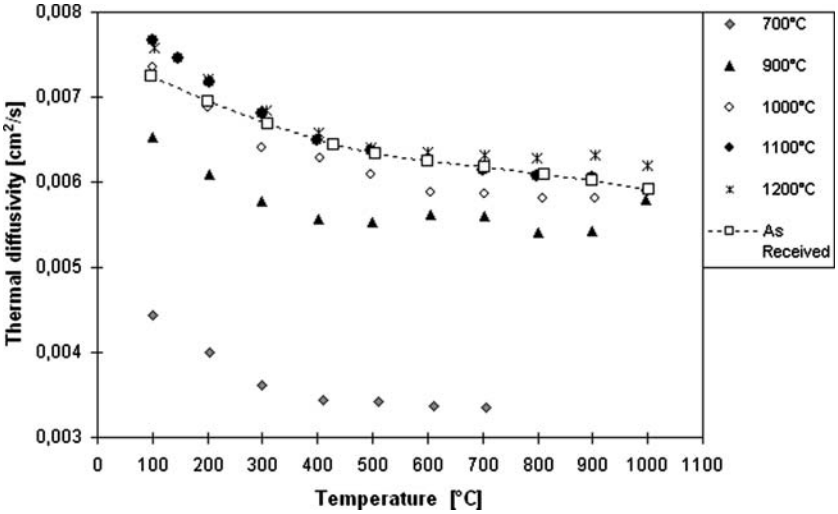


Fig. 3 Measurement of thermal diffusivity of BMAS/SiC after heat treatment for 1 h in air

This value is 39% lower than the value measured for the as-received material.

A more detailed set of thermal diffusivity results after 10 h heat treatments are shown in Fig. 4. The 1,200 °C treatment shows a slight enhancement of thermal diffusivity, as does the 1,100 °C, whereas heat treatment at 1,000 °C shows similar values to the values of the as-received material. All the other heat treatments at temperatures lower than 1,000 °C show lower thermal diffusivities than the as-received material. However, the 700 °C heat-treated sample again shows the lowest thermal diffusivity values.

These trends are also maintained for the 30-h heat treatments for which thermal diffusivity results are shown in Fig. 5. Again the heat treatment at 1,200 °C shows higher thermal diffusivity values than those for the as-received material, whereas data for samples heated at 1,000 °C and 1,100 °C are very close to that of the as-received composite. On the other hand, the thermal diffusivity values after 700–900 °C heat treatments all show lower values than the as-received materials with the ranking order of greatest change shown after annealing at 700 °C, 800 °C and 900 °C.

Microstructural studies

SEM studies

Limited SEM studies were carried out on samples heat treated at temperatures between 700 °C and 1,200 °C. The most noteworthy observation was that a gap between fibre and matrix was noted at low temperature. This is illustrated in Fig. 6 for a sample heat treated at 700 °C for 30 h. These may indicate that residual stresses exist in the samples heated at lower temperatures. Residual glassy phases would tend to flow through any gaps in the matrix but the extent to which this occurs depends on temperature of the heat

Fig. 4 Measurement of thermal diffusivity of BMAS/SiC CMC after heat treatment in air for 10 h

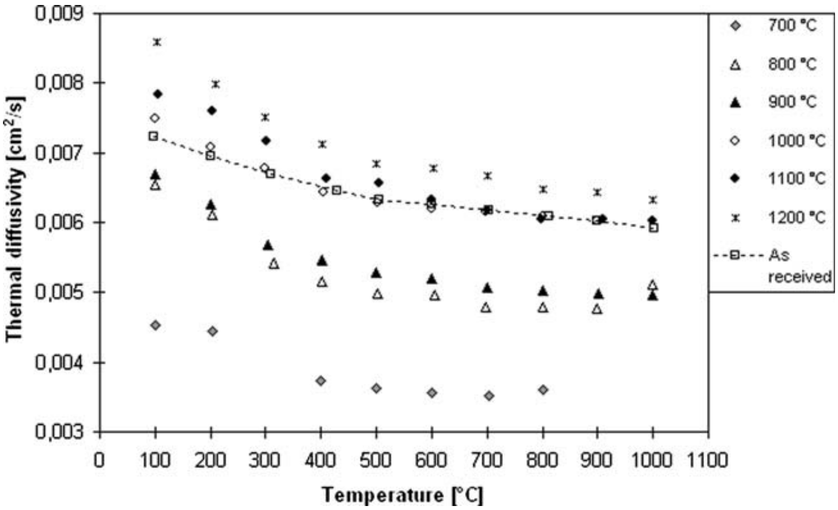
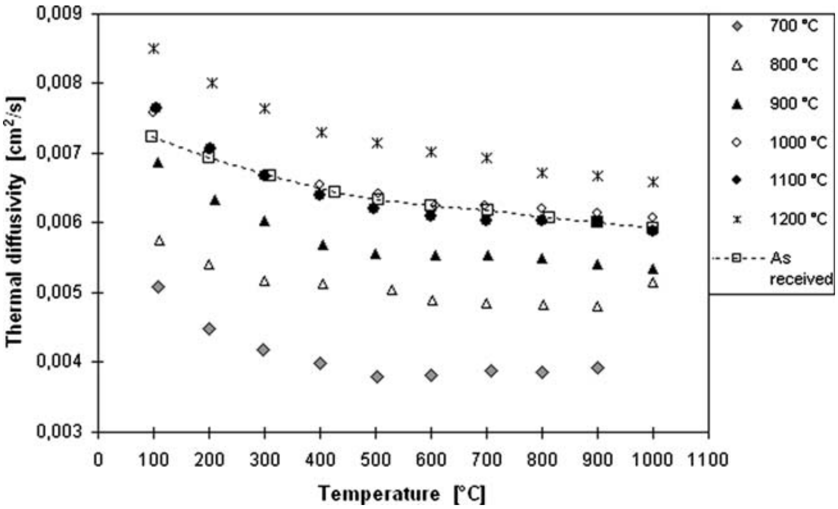


Fig. 5 Measurement of thermal diffusivity of BMAS/SiC CMC after heat treatment in air for 30 h



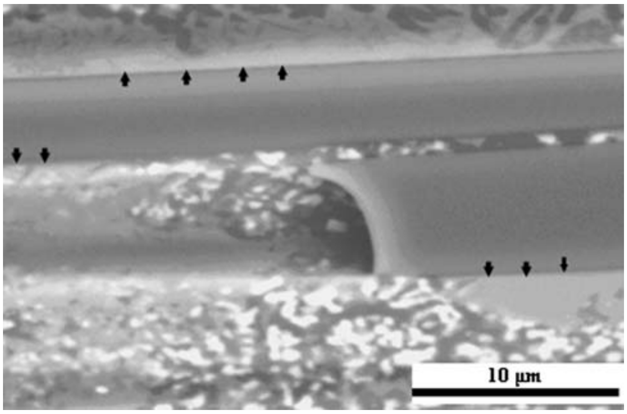


Fig. 6 Back-scattered electron SEM images showing by arrows glassy phases around the fibre after heat treatment at 700 °C for 30 h

treatment. However, it was not easy for all the glass to flow. Therefore, local concentrations of the glassy phase occurred. After a higher temperature anneal at higher than 900 °C (Fig. 7) and reduced glassy phase content was seen in the matrix. This reduction suggests that some recrystallisation of the residual glassy phases had occurred.

TEM studies

TEM studies were carried out on the selected samples. Whereas, SEM studies were intended primarily to identify the changes in phase structure and glassy phase content in the TEM studies. A special emphasis was paid to studying the fibre/matrix interface where microstructural changes had been previously reported [13–15]. Because of difficulties in preparing of suitable TEM samples, it was decided that the heat-treated samples to be studied were those heat treated for 30 h in air since heat treatment for this period had resulted in significant diffusivity changes at the lower heat treatment temperatures (700–800 °C). There was a marked change in the microstructures of samples heat treated in this temperature range where voids and gaps can be seen at the interfaces. In Fig. 8, there is a clearly identifiable gap of ~8 nm at the interface between fibre and matrix but also linkages between fibre and matrix were also seen as noted by Plucknett et al. [14].

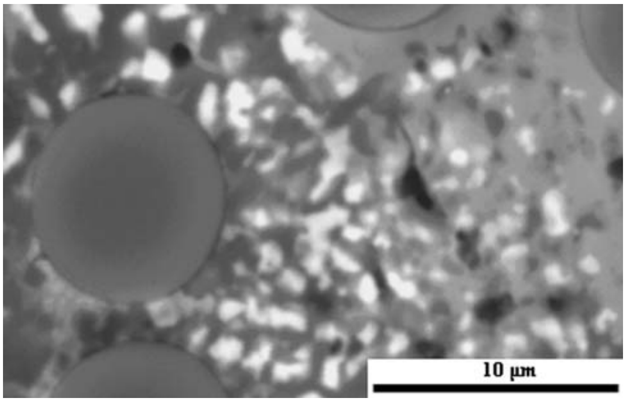


Fig. 7 SEM micrograph after heat treatment at 1,000 °C

After heat treatment to higher temperature no evidence of voids was detected. Examples of these are the micrographs in Fig. 8(a) and (b), which show interfaces in samples heated at 1,100 °C and 1,200 °C. It is noticeable that the interface has now thickened to ~20–25 nm. For an even higher heat treatment temperature the interface is even thicker (~40–50 nm), as evident with sample heat treated at 1,200 °C (Fig. 9b).

The change on the interface structure as a result of thermal treatment can be summarised as a transition from a carbon-rich interphase in the as-fabricated composite to a carbon-free interphase after heat treatments in the intermediate temperature range (700–800 °C) and finally to retention of a carbon-rich interface at higher ageing temperatures (900–1,200 °C). The interphase formed at the higher heat treatment temperature is also much thicker (45 nm) than that formed in the as-received composite (20 nm) [12].

Figure 10 collates information from the EDS traces of the various regions of the interface and compares the information with that obtained for as-received material after heat treatments at 700 °C and 1,200 °C. Although EDS analysis does not give reliable results due to its limitations for low atomic weight elements such as O and C and the results were affected by the back ground noise, this can provide a useful

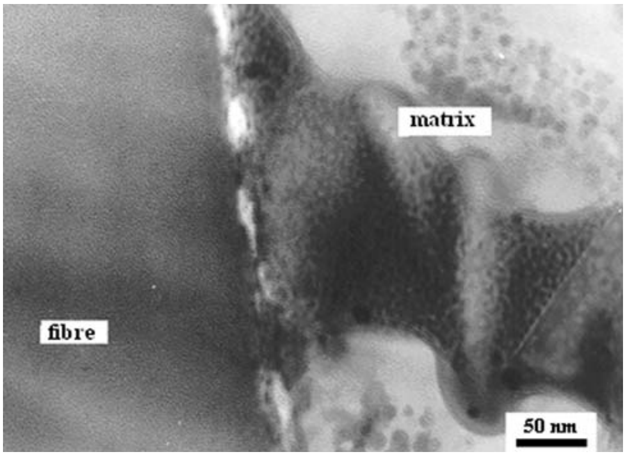


Fig. 8 TEM bright field images of the sample heated at 700 °C for 30 h showing gap and voids at the interface

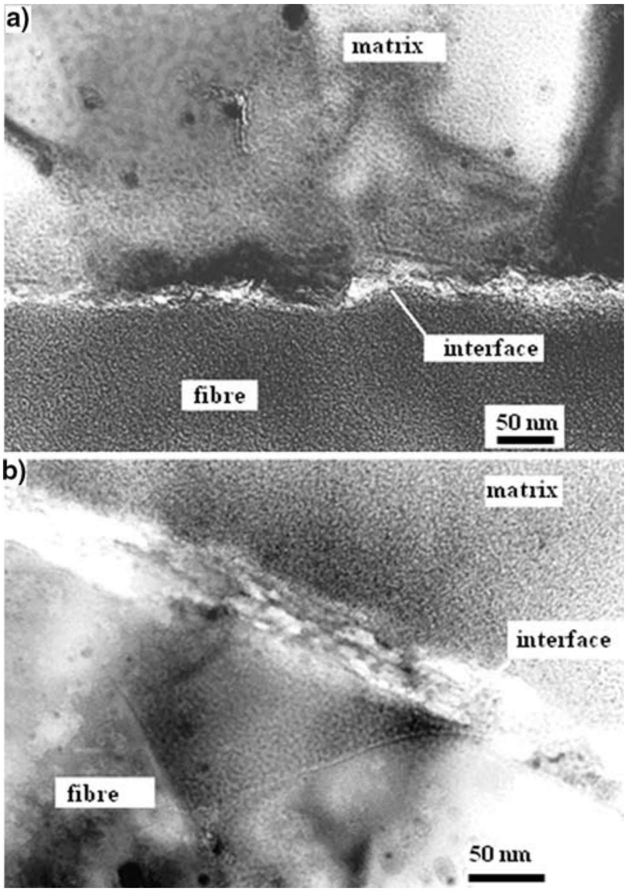


Fig. 9 TEM image of the sample heated (**a**) at 1,100 °C and (**b**) at 1,200 °C

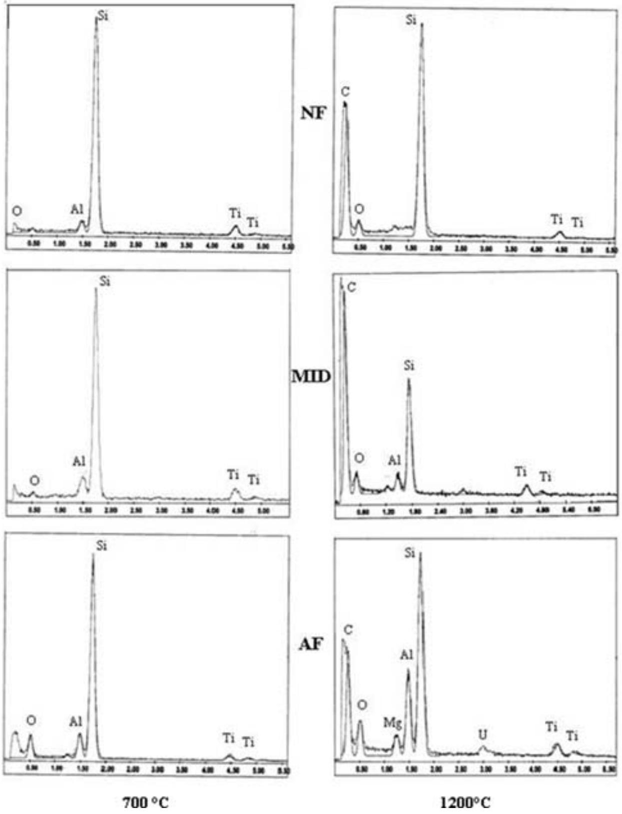


Fig. 10 EDS analysis showing the composition of SiC/BMAS composite interface after heat treatments at 700 °C and 1,200 °C (NF: EDS analysis from near fibre, MID: Middle of interface, AF: Away from fibre, U: Unidentified phase)

semi-quantitative analysis. These results may give a general idea about the composition at the interface. Those clearly show that after the 700 °C heat treatment the carbon present at the interface is removed leaving a silicon-rich layer whereas after the 1,200 °C heat treatment the carbon layer is not only still present but is even enhanced. Near the fibre and near the matrix are two regions which are carbon rich. Interestingly Ti, which can only have originated in the fibre is present in all the EDS spectra even that remote from the fibre interface. The presence of Si, O, Mg, Al and Ti is attributed to diffusion of the matrix elements to the interface. This has good agreement with previous findings [13, 14].

Discussion

Clearly the fibre/matrix interface plays an important role in the thermal properties of the composites. A discontinuity between fibres and matrix can result in a decrease in thermal diffusivity, a feature that has been noted by other workers [3, 20–23] and estimated theoretically [24]. Temperature changes during service of a component can result in structural changes such as development of porosity or the formation of other phases at the interface. The diffusivity sample thickness was 2 mm so since the ply thickness was ~0.2 mm some interfaces were always normal to heat flow so any gap at the interface would be expected to affect the thermal diffusivity of the composite. Significant effects were observed particularly in the temperature range 700–900 °C. The diffusivity measurements were supported by SEM and TEM studies. During the processing of SiC fibre-reinforced composites in a glass ceramic matrix a carbon layer is formed at the fibre/matrix interface either by the reaction first mentioned by Cooper and Chyung [4] (Eq. 1)

$$\mathrm{SiC} + \mathrm{O_2} \rightarrow \mathrm{SiO_2} + \mathrm{C} \tag{1}$$

or that also suggested by Cooper and Chyung [4], Bemson et al. [5] and Le Strat et al. [27] (Eq. 2)

$$\mathrm{SiC} + 2\mathrm{CO} \rightarrow \mathrm{SiO_2} + 3\mathrm{C} \tag{2}$$

The BMAS/SiC (Tyranno) fibre composite as received had a carbon-rich interface typically 20 nm thick, a typical thickness also noted by Plucknett et al. [14]. The thickness of the carbon-rich layer does however depend on hot pressing temperature and time [28, 29] and thicknesses as high as 250 nm have been noted [10]. The carbon exists as an amorphous phase at lower manufacturing temperatures but if manufacturing temperatures are higher than 1,250 °C graphitic carbon formation has been seen [10, 11]. The EDS analyses of the interfacial layer shows it to consist of C (~42%), O (~13%) and Si (23%) which suggests a mixture of C and SiO_2. Interestingly for the BMAS composite the interfacial layer also contained some Ti which can only have come from the Tyranno fibre suggesting that the formation of the interfacial layer had occurred by oxidation of the fibre via one of the two reactions proposed by Eq. (1) or (2).

The most dramatic results during the heat treatments are for the heat treatments in the temperature range 700–900 °C where significant decreases in diffusivity and thermal expansion coefficient are noted. TEM micrographs clearly show the development of a gap at the interface after heat treatments in this temperature range. This is clearly due to the oxidation of the carbon to leave a silicon oxide-rich region forming the bridges between matrix and fibre. Plucknett et al. [14] reported similar behaviour and stated that the bridges they observed were silica. The degradation of the C layer occurs at a temperature lower than that at which the fibre oxidises. Two reactions are possible

$$C + O_2 \rightarrow CO_2 \quad (3)$$

or more probably

$$2C + O_2 \rightarrow 2CO \quad (4)$$

Irrespective of whether the oxidation of C occurs via the formation of CO or CO_2 the mechanism whereby the reaction occurs is worth investigation. Oxygen diffusion from the BMAS matrix is unlikely at these low temperatures. The interfaces themselves may act as pipe oxidation channels providing a route for gas transport from the exposed ends of the fibres.

The reduction in oxygen content of the interfacial layer suggests that this mechanism may be a contributory factor although not necessarily the only one. The largest change in diffusivity occurs after the 700 °C heat treatment. It is questioned why degradation is most rapid at 700 °C. The rate of oxidation of C increases with temperature and the fact that degradation is less rapid at low temperatures could be due to slower kinetics of oxidation. However as the temperature is increased, two effects could contribute to reducing the effect;

(a) Any residual glassy phases in the matrix could begin to soften. The extent to which these may flow to fill gaps depends on the viscosity. As temperature is increased, viscosity is reduced and viscous flow can occur.
(b) As the temperature is increased further oxidation of the silicon carbide could occur to produce more SiO_2 and further carbon (pipe line diffusion).

The results examined for the anneals at the higher temperatures (>1,000 °C) revealed that the thermal diffusivity is changed by very little as a result of these heat treatments. The thickness of the interface however is much thicker than that of the as-received composites, amounting to some ~45 nm after the 30 h heat treatment at 1,200 °C. Examination by EDS of various regions of the interface after a 30 h heat treatment reveals it to contain Si, O and C possibly as a result of reaction. The interfacial layer contained Ti all the way through thus supporting the view that the reaction products were the result of oxidation of the SiC fibre. Interestingly the presence of Ba, Al, Mg was also noted near the matrix interface suggesting some diffusion from the matrix also. The effect of these heat treatments on the diffusivity is difficult to quantify except that for the higher temperature heat treatments there is a slight increase in thermal diffusivity after the heat treatment at 1,200 °C of some 4% after 30 h. Various reasons may be postulated for this, these are

(a) Interfacial effects,
(b) Crystallisation of residual glassy phases, and
(c) Changes in phase structure of the phases in the matrix.

Changes in the interface will affect thermal diffusivity if any contact resistance is reduced. Although the differences in thickness of the interface were noted, this is difficult to quantify. There is a reduction in the glassy phase content which could increase the diffusivity. According to Winter et al. [30] crystallisation starts at 900–950 °C with the formation of a magnesium aluminosilicate of high-quartz type structure. Osumilite starts to crystallise at around 970 °C. The other phases in the matrix begin to crystallise at around 1,000 °C. This recrystallisation in the residual glasses is possible at around those temperatures. However, crystallisation of those phases is a time-dependent process and with increasing heat treatment, the percentage of the crystallised phases is increased.

Barium osumilite and celsian both start to crystallise at around 970 °C, whereas the cordierite start to crystallise at about 1,020 °C and the highest percentage of crystallisation occurs at 1,100 °C.

Conclusions

The following conclusions can be drawn from this study:

1. The interface is one of the key factors which affect thermal properties such as diffusivity of the composites.
2. Thermal properties were determined before and after heat treatment at various temperatures and times in air. It has been found that heat treatment at the lower temperatures causes a considerable degradation in the thermal diffusivity and the thermal expansion with the greatest affect being shown after 700 °C heat treatment. However temperatures higher than 900 °C heat treatments resulted in retention in the thermal property values and sometimes even higher thermal diffusivity values were obtained.
3. TEM analysis showed interfacial structure degradation after heat treatments that were carried out at lower temperature (700 °C) on glass ceramic matrix composites caused by the removal of carbon and the occurrence of gaps between fibre and matrix and isolated silicon rich bridges linking the two.
4. TEM analysis after higher heat treatment temperatures such as 1,200 °C showed that the interfacial reaction layer was much thicker which sometimes resulted in higher values in the thermal diffusivity.
5. SEM studies show that at lower heat treatment temperature (700 °C) residual glass in the matrix migrated to the voids in particular to interfaces after degradation of carbon layer at interfaces.
6. SEM studies indicated that crystallisation in the residual glass, which resulted in an increase in thermal properties of the composites, occurred after heat treatment at temperature (>1,100 °C)
7. As a general conclusion it may be said that the thermal diffusivity can be used as a qualitative non-destructive technique to determine the integrity of the fibre/matrix interface and to monitor microstructural changes occurring in the fibres, matrix and interface during manufacturing or service.

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