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Thermal diffusivity measurement of SiC fibre reinforced BMAS glass ceramic composites exposed mechanical damage

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Abstract

Gas turbine blades in aerospace engines are one of the possible application areas for glass ceramic composites which may be subjected to various mechanical loads and can be damaged during operation. Therefore, various types of ceramic matrix composite are being evaluated for high temperature applications. Barium osumilite is a glass ceramic matrix formulation and believed to be suitable using for those applications. In this study, SiC fibre reinforced barium magnesium aluminium silicate (BMAS) glass ceramic matrix composites exposed to mechanical damage by the tensile fatigue test at the temperature of 700, 900, 1000 °C and then the effect of mechanical damage on the thermal diffusivity of the composites were investigated. The thermal diffusivity measurements were performed from 100 to 1000 °C. The obtained results indicated that the thermal diffusivity values of the composites that exposed to mechanical damage by thermal fatigue at various temperatures resulted in 32% lowering in the thermal diffusivity values of the composites fatigued at 700 °C compared with the values of as received.

Keywords: Composites; Glass ceramics; Thermal properties; Fatigue; BMAS

1. Introduction

Ceramic matrix composites offer a lot of advantages over single phase materials in view of their enhanced fracture toughness, non-catastrophic failure mode and increased thermal shock resistance. The increasing need of high performance materials for use in high temperature applications, glass ceramic matrix composites with ceramic fibre has generated a great deal of attention due to their high strength, high stiffness, excellent toughness and low density.[1–3] Glass ceramics reinforced with the continuous SiC fibres also offer superior properties. The high strength and modulus of the SiC fibres provide superior mechanical properties and can prevent catastrophic brittle failure in composites by preventing crack advance.[2–4]

Barium magnesium aluminium silicate (BMAS), that is also called as a barium osumilite in short and a glass ceramic matrix developed firstly by Brennan et al.[5] and believed to be suitable for high temperature applications. A number of researches have focused on the mechanical behaviour[6–12] and thermal properties.[13–15]

It is required from those materials to sustain under different conditions such as tensile, or compression loads and subjected to fatigue and creep conditions during the application. Therefore, it would be necessary to know their behaviour under those circumstances and their subsequent properties before using. The fatigue behaviour of composites under cyclic loading has been investigated by some researchers.[8,16–19] Thermal properties such as thermal diffusivity and expansion can give some valuable information on the performance of these materials. The effects of mechanical damage on the thermal properties of the composites need to be investigated. Such mechanical tests can cause some damage in the microstructure such as matrix cracking and debonding at the fibre matrix interface.[17] The environmental conditions during the test also affect on microstructure and subsequent the properties on the composites.[13–16] Thus, working conditions might cause an affect on the thermal properties of the composites as well as on the mechanical properties.

The laser flash diffusivity method has been used for determining diffusivity of relatively homogeneous solid materials such as composites.[20] The aim of this research was to determine the thermal diffusivities of the composites that exposed to mechanical damage by tensile fatigue test at various temperatures of 700, 900 and 1000 °C in air using laser flash method. The obtained results were discussed with the knowledge available in the related literature.

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2. Experimental procedure

2.1. Material

The material used in this study is the barium osumilite glass ceramic reinforced with SiC Tyranno fibre with density of 2.58 g cm^{-3}. Optical micrograph of a cross section of the composite is presented in Fig. 1. The micrograph clearly shows the 0°/90° lay-up with layer with thickness of approximately 0.2 mm and fibre distribution is fairly uniform and fibre volume fraction is approximately 48%. The total thickness of the composites is 3 mm. The composite has been fabricated by AEA Harwell technology using slurry impregnation and hot pressing methods. Later, it was crystallised by heat treatment and glass ceramic matrix is composed of three main phases including barium osumilite ($BaMg_2Al_3(Si_9Al_3O_{30})$); celsian ($BaAl_2(Si_2O_8)$); cordierite ($Mg_2Al_4Si_5O_8$).

2.2. Thermal diffusivity measurements

The thermal diffusivity was measured by flash method. This method has the advantage of using simple shaped samples with the small size and good accuracy. Therefore, there is increasing use of this technique to measure heterogeneous materials such as composites.[20] The detailed information about the thermal diffusivity apparatus used in this study has been given earlier.[21] The specimens for those measurements were cut in rectangular platelets (8 mm × 8 mm × 2 mm) from the place near to the tensile fatigue damage. The heat pulse is supplied by Nd glass laser. The specimens lightly coated with a thin layer of colloidal carbon to promote absorption and prevent direct transmission of the laser pulse. The specimen is heated up to the measurement temperature and temperature rise at the rear face of the specimen is monitored by collecting radiation from the rear face of the sample which is collected using a CaF_2 lens system. An indium antimonide (InSb) infrared radiation photo voltaic detector was used to monitor the transient temperature rise of the specimen. The signal from the detector has to be balanced and amplified before it is processed by micro computer.

Heat flow can be assumed to be one-dimensional because the thickness of the specimen is quite small. The thermal diffusivity

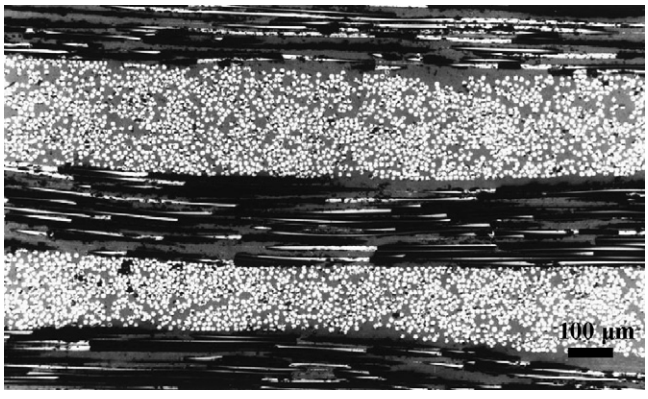

Fig. 1. Optical micrograph of a cross section of the BMAS glass ceramic composite reinforced with SiC fibre.

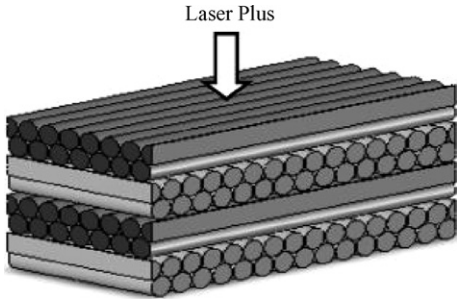

Fig. 2. Schematic diagram of the sample arrangement for thermal diffusivity measurements.

is obtained by the following equation:

$$\alpha = \frac{\omega/\pi^2}{t_{1/2}} \times L^2 \quad (1)$$

where L is the thickness of the sample and $t_{1/2}$ is the time it takes for the rare face to reach half of its maximum temperature rise. ω is dimensionless parameter for the half rise at the rear face, becomes 1.37.

The thermal diffusivity measurements were performed from 100 up to 1000 °C with intervals of about 100 °C in vacuum. Several measurements were taken at each temperature and averaged to give the final value of thermal diffusivity. The measurements values very close each other. Schematic diagram of the sample arrangement for thermal diffusivity measurement is given in Fig. 2. Heat flow during measurements in all samples is perpendicular to fibre axis. In this case, the fibre direction in the composite is not taken in to consideration for comparison obtained thermal diffusivity values.

2.3. Tensile fatigue test

The tensile fatigue test pieces were supplied by National Physical Laboratory (NPL). The tensile fatigue test pieces were stressed at three temperatures with similar exposure times. The fatigue cycle was saw-tooth 0.5 ± 0.5 kN on the test piece for 20,001 cycles at 10 s per cycle with the frequency of 0.1 Hz giving total exposure test time of 55 h. Tensile fatigue tests were carried out at the temperatures of 700, 900 and 1000 °C.

2.4. Microstructural examinations

Microstructural characterizations were carried out to the develop understanding of the mechanism of damage evaluation after the fatigue test. The surface of the heat treated samples were examined using Philips 525 scanning electron microscopes (SEM) operating at 20 kV.

3. Results and discussion

The thermal diffusivity results for the composites exposed to tensile fatigue test at various temperatures are given in Fig. 3. As clearly seen from the figure that all thermal diffusivity measurements show lower thermal diffusivity values than that of as-received materials. This indicates that mechan-

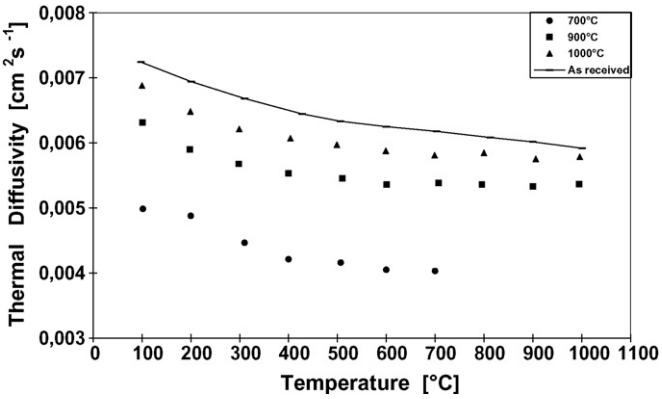

Fig. 3. Measurement of thermal diffusivity of BMAS/SiC after mechanical damaged by tensile fatigue test.

ical damage has great influence on the thermal diffusivity values. The highest thermal diffusivity values were obtained with the composite sample fatigued at 1000 °C. Trace of the thermal diffusivity values as a function of temperature gradually decreases with increasing temperature from a value of 6.9×10^3 cm^2 s^{-1} at 100 °C to 5.8×10^3 cm^2 s^{-1} at 1000 °C. A similar trace was obtained for the sample fatigued at 900 °C but the thermal diffusivity values were slightly lower with values of 6.3×10^3 cm^2 s^{-1} at 100 °C. These values remain almost constant through from 400 to the 1000 °C with the values of 5.5×10^3 cm^2 s^{-1}. The samples fatigued at 700 °C showed the lowest value of 5.9×10^3 cm^2 s^{-1} at 100 °C, decreasing to 4.0×10^3 cm^2 s^{-1} at 700 °C. Thermal diffusivity measurements of the sample fatigued at 700 °C were not carried out at temperatures above 700 °C. This is because higher temperatures affect interface structure of the sample as seen on the author's earlier works.[15] The results obtained in this study indicated that tensile fatigue test temperatures have a great influence on the thermal diffusivity values. Differences in the thermal diffusivity values of the composites fatigued at 1000 °C is 5% lower then that of the composite of as received materials while the sample fatigued at 700 °C shows 32% lower thermal diffusivity values than that of the composite of as received materials.

Fatigue damage analyses were carried out by microstructural examination of the test specimens. The fracture surface of the composite damaged by the fatigue test at 700 °C shows no fibre pull out behaviour during the fatigue test (Fig. 4). Quantitative analysis of debonded and broken fibres was conducted and correlated with the change in thermal diffusivity values and tensile fatigue test temperatures. There is significant fibre damage within the ply regions of the fractured surface of the fatigued test at 700 °C as can be seen in Fig. 5.

Fig. 6(a) shows that some debonding of the fibres was observed on the fractured surface after the tensile fatigue test at 900 °C. The sample exhibits normal fibre pull-out. However, fracture surface of the fibre is not smooth. Longitudinal matrix cracking along the fibre is also seen in Fig. 6(b). The testing at 1000 °C resulted in slightly increasing in fibre pull out length over that of the sample tensile fatigued at 900 °C (Fig. 7). Tensile fatigue test of SiC fibre reinforced BMAS glass ceramic matrix composites at elevated temperatures higher than 900 °C demon-

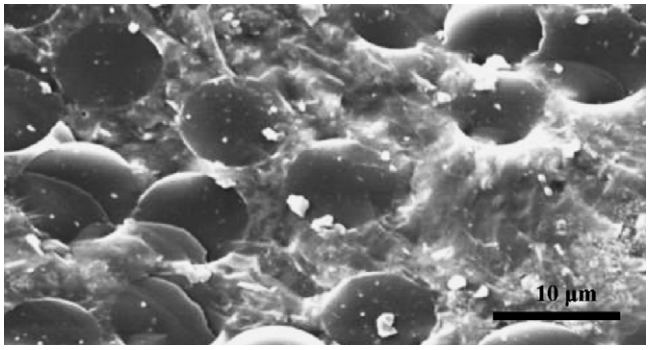

Fig. 4. SEM micrograph of a sample fatigued at 700 °C for 55 h showing no fibre debonding.

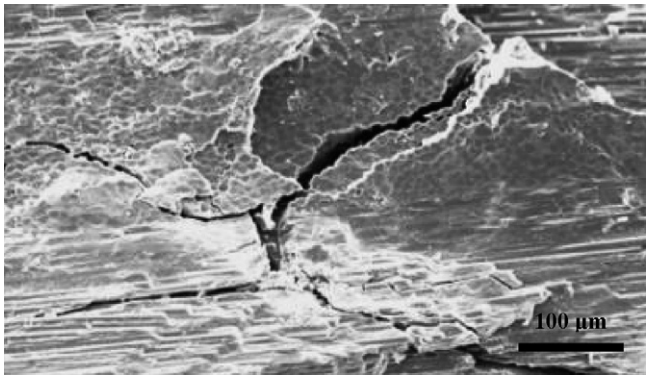

Fig. 5. Surface characteristic of the sample after cycling to 700 °C for 55 h.

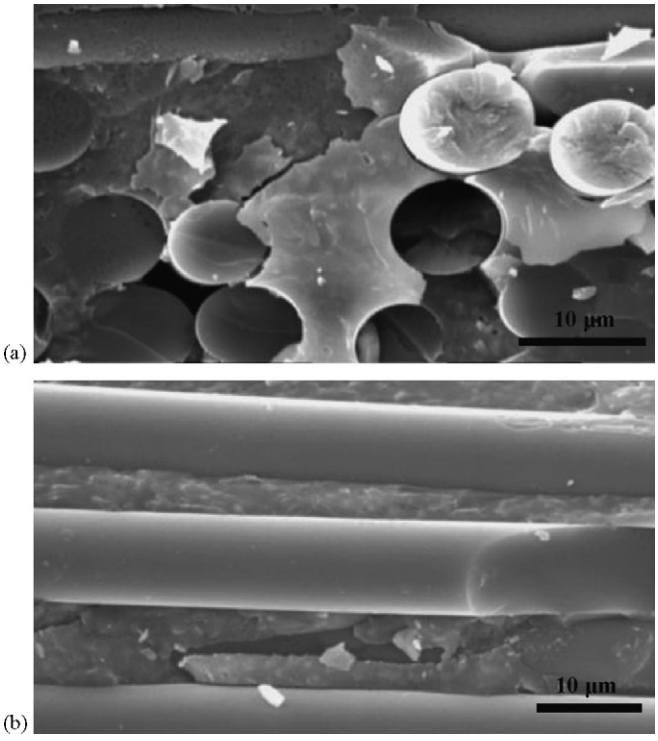

Fig. 6. SEM micrographs of the fracture surface of a sample (a) fatigued at 900 °C showing significant fibre debonding (b) cracking in the matrix.

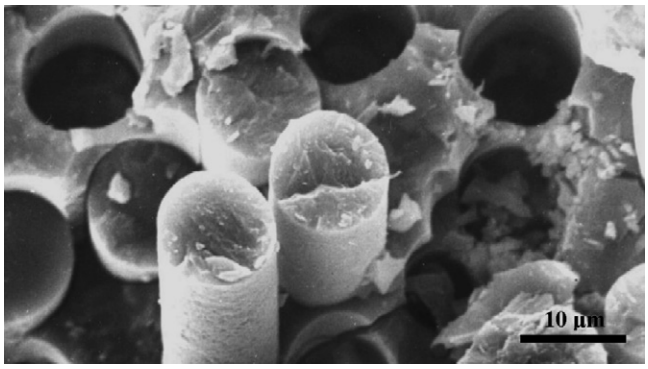

Fig. 7. SEM micrographs of the fracture surface of a sample fatigued at 1000 °C.

strated a similar fracture surface. The observations indicate that those samples with similar interface structures have better interfacial properties. However, the sample fatigued at 1000 °C seems to have much weaker interface that provides longer fibre pull out. This situation reflected that higher thermal diffusivity values were obtained, which were slightly lower than that of as received material.

As earlier study[16] indicated that there is a good correlation between the percentage of broken fibres and the observed stiffness. These observations of the damage evaluation during fatigue are shown in a schematic diagram. Fig. 8 reveals the formation of crack perpendicular to the surface and parallel to the fibre direction. During the tensile fatigue test, due to thermal expansion coefficient mismatch between fibre and matrix, internal stresses were generated across the interface resulted in a debonding between the fibre and matrix and then fibre pull out is observed at higher test temperatures such as at 900 and 1000 °C while not observed at a test temperature of 700 °C. This situation indicates that a weaker bond due to presence of a carbon layer occurred at processing temperature during fabrication of the composites provides debonding at the fibre matrix interface while a strong bond at the fibre matrix interface does not allow any debonding at all as seen in Fig. 4.

Density of matrix cracking in the composites depends on various parameters such as applied load and cycles speed.[17] According to the earlier study,[18] it was suggested that the crack density increases with the number of the fatigue cycles and the crack generally starts in the 0° ply and later on separates in the 90° ply. The crack density in the 0° ply is greater than the crack density in the 90° ply.

As mentioned in earlier studies,[11,12,22] carbon rich interface is formed between fibre and matrix during the processing of glass ceramic matrix composites reinforced with continuous SiC fibre. Degradation of the carbon layer at the interface due to lower temperature heat treatment was confirmed by the observations of other researchers.[12,15] The degradation occurs at lower temperatures either

$$C_{(s)} + O_{2(g)} \rightarrow CO_{2(g)} \quad (2)$$

or more probably

$$2C_{(s)} + O_{2(g)} \rightarrow 2CO_{(g)} \quad (3)$$

and other reaction between fibre surface and oxygen proceeds as following:

$$SiC_{(s)} + 2O_{2(g)} \rightarrow SiO_{2(s,l)} + CO_{2(g)} \quad (4)$$

or

$$SiC_{(s)} + 1.5O_{2(g)} \rightarrow SiO_{2(s,l)} + CO_{(g)} \quad (5)$$

The loss in strength is attributed to the degradation of the carbon rich interface following removal of the carbon between the fibre and matrix. Later, oxidation progressing of SiC fibre resulted in formation of a SiO_2 and then caused silica bridges and gap at the interface between fibre and matrix.[13,15] The strong interfacial bonding with the consequent degradation of strength and composite ductility contributes to the brittle behaviour of the composite and results in a decrease in the thermal and mechanical properties.[12,15] In an earlier study,[19] it was indicated that the specimen after 700 °C cyclic fatigue oxidized and showed lower strength but during the higher temperature thermal fatigue

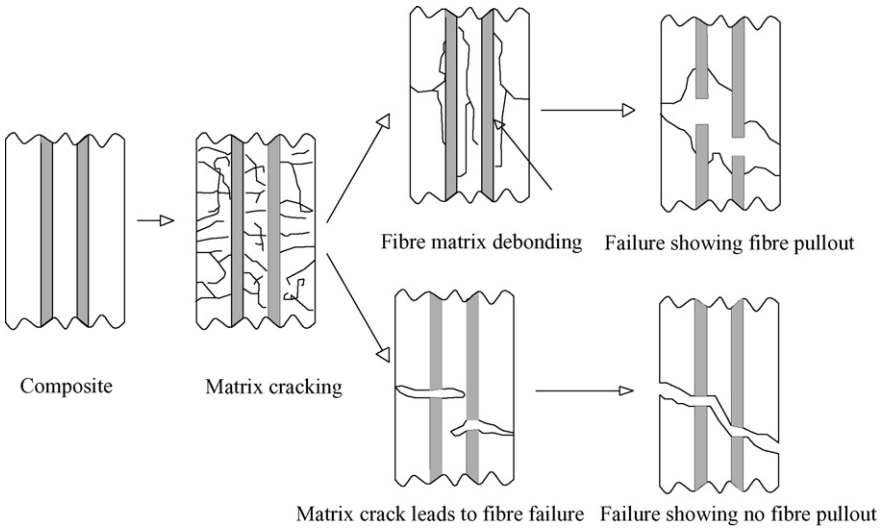

Fig. 8. Schematic diagrams depicting the progression of fatigue damage under different interfacial conditions.

tests these specimens retained most of their strength. The similar observations were obtained in this research. The samples tensile fatigued at temperatures higher than 900 °C resulted in higher thermal diffusivity values due to carbon layer existence at interface provides better bonding between fibre and matrix.

A number of previous studies have indicated that thermal conductivity of the composites was affected by a thermal barrier resistance at the fibre matrix interface.[23–26] The resistance can arise from thermal expansion mismatch between composite constituents that resulted in an interfacial gap that is one of the reasons for low thermal diffusivity values. The coefficient of the SiC fibre is higher than that of the matrix with the values $1.8 \times 10^{-6}\,K^{-1}$ for barium osumilite and $3.30 \times 10^{-6}\,K^{-1}$ for SiC fibre.[14] This implies that under the tensile loading, the stresses generated across the interface. Weakly bonded interface provides perfect adhesion that can lead to lower interfacial contact resistance between fibre and matrix and resulting in a better heat transfer therefore, an increase in thermal diffusivity while the strong bonded interface resulted in to interfacial gap between fibre and matrix hence lower thermal diffusivity values. In this study, thermal diffusivity measurements were carried out in vacuum atmosphere. Thermal diffusivity measurements of the samples having a gap at the interface in vacuum show lower the thermal diffusivity values comparing with other gaseous environments. The conductance occurs by direct fibre to matrix contact must be interfacial heat transfer in vacuum due to the absence of gaseous environment. In this case, direction of heat flow can play important role in determining the thermal diffusivity of the composite in which fibre matrix interface resistance exist. Crack in the matrix or the gap between fibre and matrix has been shown to be very effective in lowering the thermal diffusivity values. The greatest effect in reducing thermal diffusivity can be for heat flow perpendicular to the fibre matrix interface.[26]

The experimental observations in this study strongly indicated that the interface structure plays significant role, and makes a significant contribution to the heat transport behaviour across the composite. Therefore, it has a great influence on the thermal diffusivity values of the SiC fibre reinforced glass ceramic matrix composite. Thermal barrier and matrix cracking are also very effective in lowering thermal diffusivity.

4. Conclusions

The following conclusions can be drawn from this study:

1. The samples damaged by exposing tensile fatigue test at various temperatures have lower thermal diffusivity values than that of the as-received materials.
2. Tensile fatigue test temperature has a great influence on the thermal diffusivity values. After tensile fatigue test at 700 °C, the lowest thermal diffusivity values were obtained due to structural degradation at the interface of the composites cased by the removal of carbon and leaving the gaps between the fibres and matrix.
3. Fibre debonding was observed after tensile fatigue test at 900 and 1000 °C indicating weak bonding while none was observed at 700 °C test due to strong bonding between fibre and matrix as the one was confirmed by the pull outs.
4. In general conclusion of this work, it was shown that the laser flash measurement techniques can be employed in the determination of thermal diffusivity of the composites that exposed mechanical damage.

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