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Damage assessment of oxide fibre reinforced oxide ceramic matrix composites using acoustic emission

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Abstract

The damage mechanisms of woven mullite fibre (Nextel 720^{TM}) reinforced alumina ceramic matrix composites (CMCs) were monitored during tensile tests using acoustic emission (AE) technique. The effects of microstructural characteristics, such as porosity and pore size as well as the nature of interfacial zone between fibre and matrix on the type of damage were also examined and correlated with the microstructural observations. It is shown that based on the energy level of acoustic events, during the tensile tests, delamination of the fibres from the matrix takes place first then multiple matrix cracks form and finally reinforcement fibres break before the composite's final failure. It is also shown that porosity content of the composite samples influences the acoustic emission response during the tensile loading, as both amplitude and the energy values are higher for the composites with low porosity level and smaller pore diameter.

Keywords: B. Porosity; Acoustic emission; Ceramic composites; Tensile test

1. Introduction

Continuous fibre reinforced ceramic matrix composites (CMCs) are prime candidate materials for use in advanced structural and high temperature applications due to their excellent thermal stability, good thermal shock resistance and high fracture toughness [1–4].

The fracture toughness and flaw resistance of monolithic ceramics can be increased by the introduction of high strength continuous ceramic fibres resulting in toughening mechanisms, such as fibre-debonding, pull-out, fibre bridging and crack deflection which all contribute to a non-linear stress–strain response and thus high energy dissipation before fracture [5,6]. In service, CMC components, such as gas turbine airfoils, heat exchanger and combustors are subjected to tensile loading and therefore under these conditions the damage mechanisms within the composite should be analysed non-destructively for long-term structural reliability as well as structural design [7].

Acoustic emission (AE) is the class of phenomena whereby an elastic wave, in the range of ultrasound usually between 20 KHz and 1 MHz, is generated by the rapid release of energy from the source within a material [8]. The elastic wave propagates through the solid to the surface, where it can be recorded by one or more sensors. The sensor is a transducer that converts the mechanical wave into an electrical signal. In this way information about the existence and location of possible sources is obtained. AE analysis can be used for the investigation of local damage in fibre-reinforced composite materials. When a fibre-reinforced ceramic composites is subjected to tensile loading, a variety of damage mechanism including fibre debonding, matrix cracking and fibre fracture can take place. As the damage progresses within the composite energy is released in different forms such as acoustic waves. The energy and or the amplitude of the acoustic waves depends on type of fracture event which can be detected by monitoring the AE. Each event can be separated from each other by analysing AE parameters, such as amplitude, duration, energy and time. Several work had been carried out by researchers to investigate the damage accumulation in the ceramic matrix composites [9–11].

The main objective of the present work is to determine the main damage mechanisms in woven mullite fibre-reinforced alumina ceramic matrix composites with weak interface between the fibre and matrix under tensile loading at room

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Table 1
Some physical properties of the composite plates tested.

Sample no	Average pore diameter (nm)	Bulk density (g/cm^3)	Porosity (%)	Tensile strength (MPa)
1	200	2.4	30	102
2	90	2.6	20	155

temperature to establish the damage initiation and progression in real time. First time to date, the effects of porosity and pore size of the composite on the AE parameters are also examined.

2. Experimental work

In the present work the 8HS satin woven mullite (Nextel™ 720, 3M, USA) fibre reinforced alumina matrix composites were tested as some of the physical properties of the samples are shown in Table 1. Composite plates tested here contain different amount of porosity and a constant volume fraction of 40% reinforcement fibres [9].

Tensile tests were performed on an Instron 5584 tension machine using a cross-head speed of 0.1 mm/s and tensile elongation was measured with an extensometer with a gauge length of 10 mm. During the loading acoustic activities of the composite specimens were monitored simultaneously using two nano-30 acoustic sensors with resonance frequency of 300 kHz. Location of the acoustic sensors on the tensile specimens is also given in Fig. 1.

A 40 dB preamplifier was used individually for each channel and the activities were captured and analysed using the Mistras software system loaded into a PC.

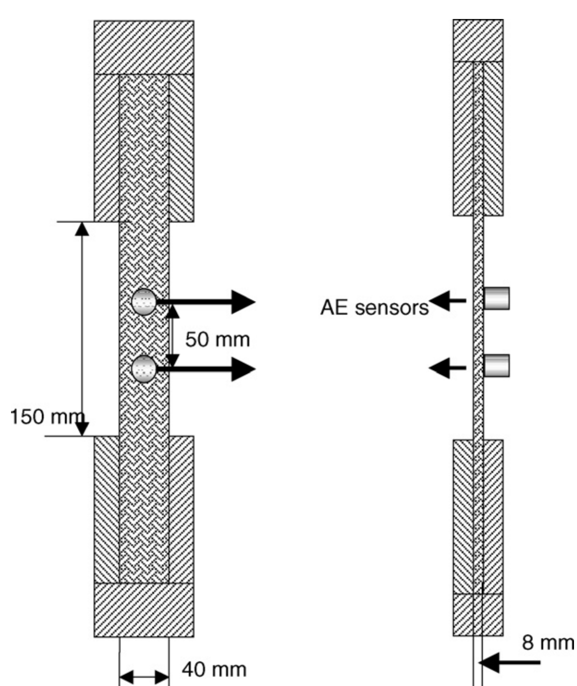

Fig. 1. Schematic diagram of the tensile specimen showing the dimensions of the tensile specimen and location of the acoustic sensors.

3. Results and discussion

In the experiments, two different composite plates with different porosity level were tested, named sample 1 which contains 30% porosity and sample 2 with a porosity level of 20% as shown in Table 1. A typical test sample prepared from the oxide/oxide composite panels is shown in Fig. 2.

The AE results obtained from the sample 1 are shown in Fig. 3 indicating that up to tensile stress value of 30 MPa, no acoustic event has taken place, which may indicate that specimen deforms elastically up to that point of loading. Fig. 3 also shows that the maximum stress level of the sample 1 is just over 100 MPa. During elastic deformation each fibre lay up within the composite moves in shear due to tensile loading as the schematic illustration of the shear movement of the ceramic fibre mats is shown in Fig. 4.

A detailed examination of acoustic parameters such as energy and amplitude values given in Fig. 3 has shown that acoustic events could be categorised into two distinct sub-groups, as shown in Fig. 5. When the energy values of acoustic events are plotted against the amplitude values it could be observed that acoustic events fit in a band, where amplitude values ranges between 50 and 78 dB, while the energy values vary between 1^3 and 1^6 (Joule $\times 10^{-18}$). However, there is a distinct tail at higher energy values of 10×10^3 (Joule $\times 10^{-18}$), which could be identified as fibre fractures as shown in Fig. 5.

Results shown in Figs. 3 and 5 clearly explain that amplitude levels indicate how loud the acoustic hit occurres while the energy values indicate how much energy is released due to fracture of the component. Both of these parameters could be used to identify high stiffness materials such as reinforcement fibres. In ceramic matrix composites both ceramic matrix and the ceramic fibres posses high stiffness, therefore the amplitude values for the fracture of ceramic matrix and fibres could be expected to be comparable in their values. However, both strength (tensile strength of a mullite fibre is approximately 1200 MPa) and the thickness of a ceramic fibre mat (here

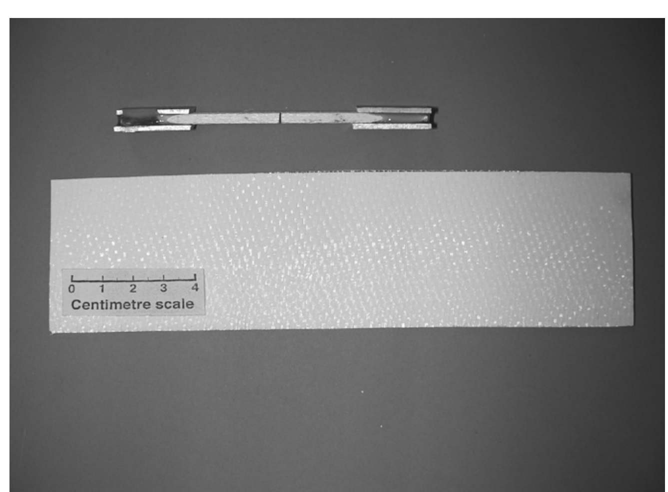

Fig. 2. Woven mullite fibre-reinforced alumina ceramic matrix composite panel tested.

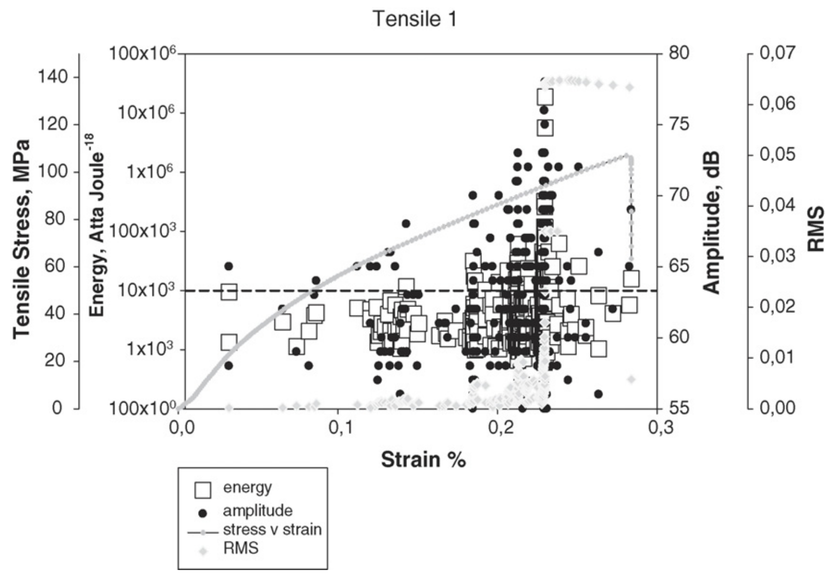

Fig. 3. Tensile test and AE results of the sample 1 which contains 30% porosity.

approximately 1200 individual ceramic fibres with diameters of 10 μm form bundles with an average overall thickness of 1 mm) are much higher than tensile strength of un-reinforced mullite matrix,which is about 250–300 MPa and the thickness of the un-reinforced ceramic matrix layer is about 100–200 μm. Hence, high energy value acoustic events could be interpreted to be generated by the fracture of ceramic fibre bundles. Furthermore, high energy value ($>10^{3+}$ Joule $\times 10^{-8}$) acoustic events are observed to be occurring towards to the end of the tensile loading, when energy values are plotted against the time of the events, as shown in Fig. 6. These events could be associated with the fracture of the mullite fibre bundles. As both the thickness and the elastic modulus of the fibre mats are high, acoustic energy generated by the fracture of fibre mats are expected to be higher [8].

Fig. 7 shows the acoustic emission response of the composite labelled as sample 2 in Table 1. Compared to the

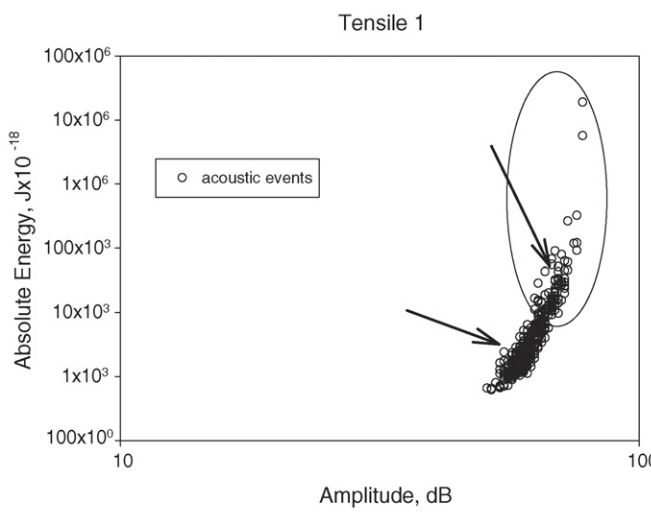

Fig. 5. Acoustic emission response of sample 1 indicating that acoustic events could be divided into two distinct categories which could be identified as, ‘‘matrix related’’ and ‘‘fibre fracture related’’ events, if absolute energy values are plotted against amplitude values.

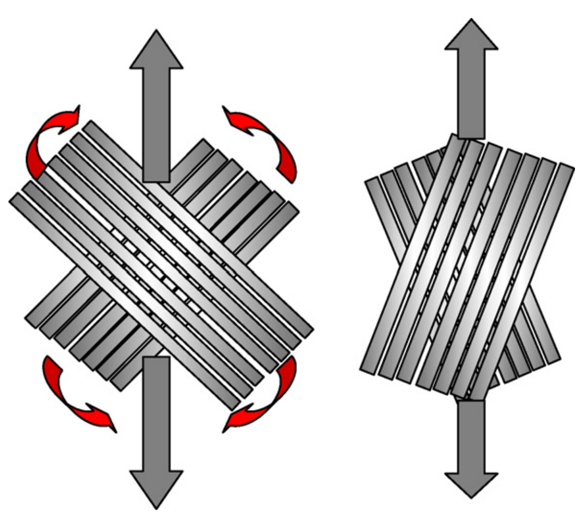

Fig. 4. Schematic illustration of shear movement of the fibre mats within the composite specimen.

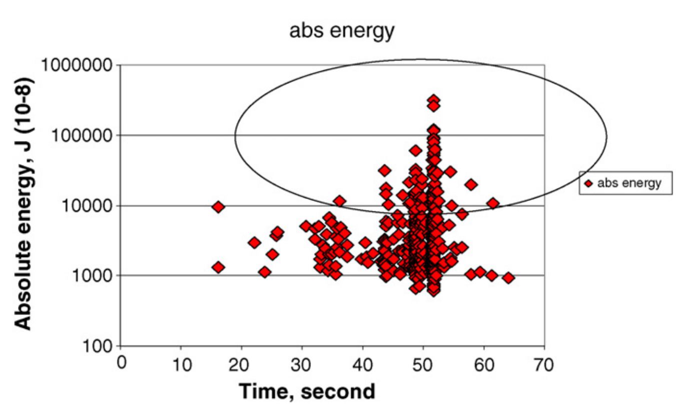

Fig. 6. Acoustic events plotted against their time of event for sample 1 indicating that high-energy value hits occur towards the end of the tensile loading.

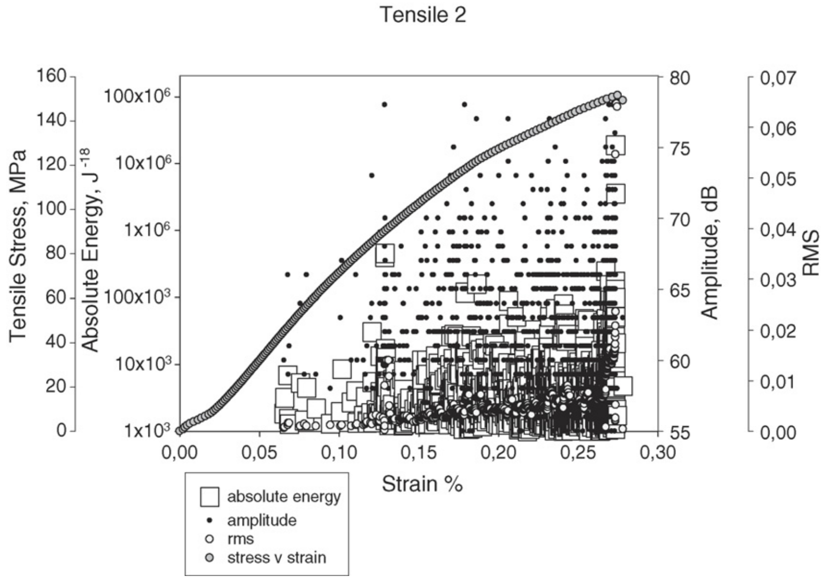

Fig. 7. Tensile test and AE results of sample 2 showing that maximum tensile strength of this sample (155 MPa) is 50% higher than that of sample 1.

acoustic response of sample 1 shown in Fig. 3, both energy and the amplitude values of this particular specimen are observed to be higher (see Figs. 3 and 6). Minimum and maximum amplitude values recorded from sample 1 are lower than those recorded during the tensile testing of sample 2, because the porosity values of these specimens are different as given in Table 1. Pores in sample 1 are approximately 200 nm in diameters, whereas the size of the pores that observed in sample 2 is about 90 nm. The porosity levels of sample 1 and sample 2 are calculated to be 30 and 20%, respectively. Correspondingly, bulk densities of two samples are measured to be 2.4 and 2.6 g/ cm^3, respectively.

It is also observed from the Fig. 7 that the tensile strength of the sample 2 is approximately 50% higher than that of the sample 1 (maximum tensile strength of sample 1 is measured to be 102 MPa, whereas the tensile strength of the sample 2 is measured to be 155 MPa). Therefore, it could be decisively said that high porosity content decreases the tensile strength of composites. Porosity content of the ceramic samples is also found to be influencing the acoustic emission response during the tensile loading, as both amplitude and the energy values of acoustic events recorded during the tensile loading of sample 2 are observed to be higher than those recorded during the tensile loading of the sample 1 (see Figs. 3 and 7). This result also explains that each pore is actually a discontinuity in the material therefore under tensile load, higher stresses than that of the rest of the cross-section are concentrated around the pores, which may then increase the possibility of crack initiation. Therefore, as the loading increases matrix ligaments located between the pores fracture as this process continues pores merge in to a bigger discontinuity, decreasing the cross-section area, rapidly. As a result the net stress on the entire specimen increases leading to a rapid fracture. Acoustic emission results are affected by the loss of the tensile strength, because as explained above if the energy generated by the fracture is high, relevant acoustic event possesses high-level energy and amplitude values. As a result there is a direct relationship between the tensile strength and the acoustic emission response of the materials as high strength compact material emits high level of acoustic activities.

Compared to sample 1 (see Fig. 6), high-energy value acoustic events which are identified as fibre fractures, occur earlier in sample 2, as shown in Fig. 8. An acoustic event with absolute energy level higher than 10^{3+} Joule $\times 10^{-8}$ is recorded in 20th second of the monotonic loading, which may indicate that the damage tolerant behaviour observed in low interfacial strength composites is in action in sample 2. This may indicate that stress transfer from the relatively weak matrix ligaments to the fibre bundles is occurring more effectively when the porosity content is low. From the initial fibre fracture, the events of the fibre fractures are also observed to be regular, i.e. couple of fibre bundles fracture every 10 s, as shown in Fig. 8.

Detail microstructural observations were also carried out to find out the effects of the porosity level and pore size on the fracture behaviour of the composite plates. Fig. 9a shows the cross-sectional SEM micrograph of the sample with 20% porosity with an average pore size of 90 nm whilst the SEM image of the fracture surface of the composite plate having 30% porosity with an average pore size of 300 nm is shown in Fig. 9b.

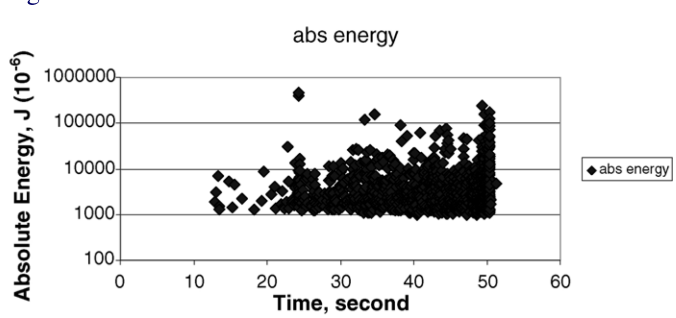

Fig. 8. Absolute energy versus time of occurrence plot for sample 2 indicating that damage tolerant behaviour occurs in this particular sample.

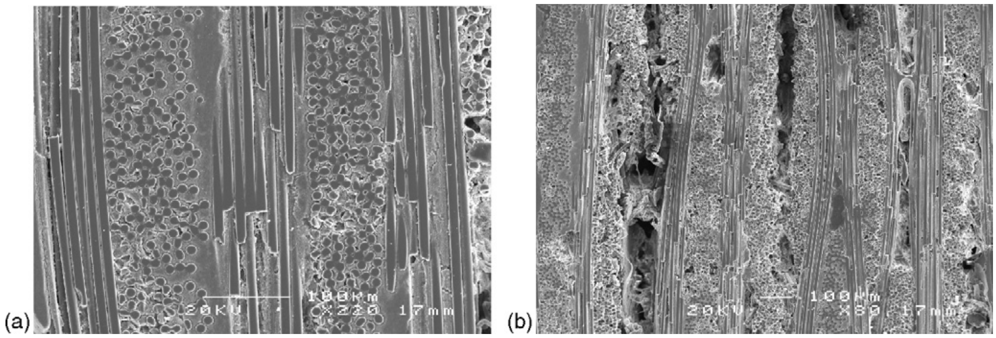

Fig. 9. SEM micrographs of the composite samples with different porosity level and pore diameter: (a) porosity level of 20% with the average pore size of 90 nm and (b) porosity level of 30% with the average pore diameter of 200 nm.

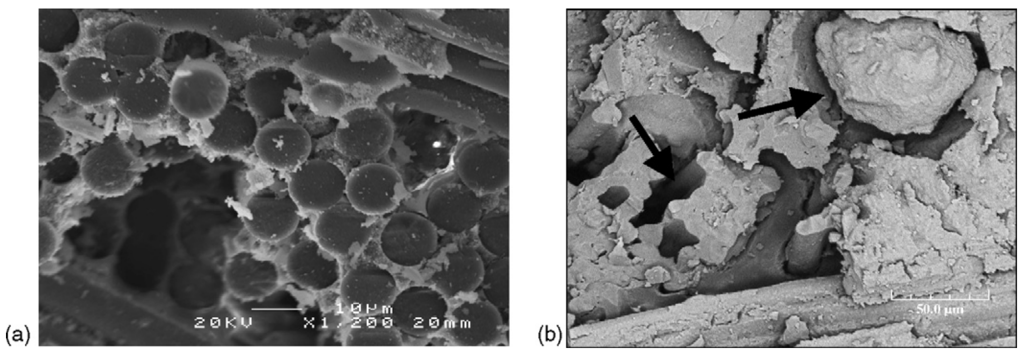

Fig. 10. SEM micrographs of the fractured composite samples after tensile tests: (a) porosity level of 20% with the average pore size of 90 nm and (b) porosity level of 30% with the average pore diameter of 200 nm.

The apparent difference in microstructure of the two composite plates is clearly seen from the SEM micrograph shown in Fig. 9. The microstructure shown in Fig. 9a represents the structure of the composite with a pore size of 90 nm and lower porosity level of 20% whilst the microstructure shown in Fig. 9b indicates the presence of large pores of about 200 nm and higher level of porosity up to 30%. The fracture behaviour of the composite plates shown in Fig. 9 is also different from each other as shown in Fig. 10.

As shown in Fig. 10a, the results obtained from the tensile tests confirm that composite plate with lower porosity level and smaller pore size exhibit homogeneous fracture behaviour as most of the fibres failed at the similar loads. However, the microstructure of the sample with higher porosity level (30%) and larger pore size (200 nm) shows very different fracture behaviour as shown in Fig. 10b which indicates that large pores are acting as crack initiation sites (as arrowed on the micrograph shown in Fig. 10b) and therefore the cracks that are formed in these regions propagate within the brittle ceramic matrix resulting in final failure of the composite sample in a short time. The apparent difference in fracture behaviour of the two different composite samples after tensile tests is also clearly seen in Fig. 11.

As shown in Fig. 11a, lower porosity level and pore size cause some degree of fibre pull-out which indicates the presence of damage-tolerant behaviour with in the composite sample. However, when the pore size and porosity level are quite high, 200 nm and 30%, respectively in this case, brittle fracture behaviour is seen within the composite plate as shown in Fig. 11b. The microstructures shown in Figs. 10b and 11b prove that large pores are responsible for the sudden failure of the sample as these cracks are acting as crack initiation sites or large flaws.

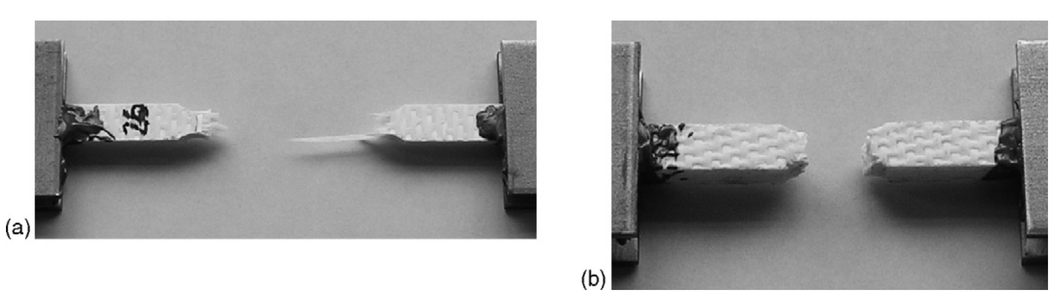

Fig. 11. Micrographs of the fractured composite samples after tensile tests: (a) porosity level of 20% with the average pore size of 90 nm and (b) porosity level of 30% with the average pore diameter of 200 nm.

Overall, by analysing the AE parameters shown in Figs. 3–8, combined with the microstructural observations presented in Figs. 9–11, it can be concluded that porosity level and pore size affect the strength and acoustic events of the composite. As the composite plates tested here have weak interfaces [5] between fibres and ceramic matrix, acoustic activities with the lowest energy level can be classified as fibre debonding, activities with moderate energy level are considered heavily cracks formation within the ceramic matrix and activities with the highest energy level characterise the fibre fracture. Fibre fracture takes place earlier in a composite sample with higher porosity level as the large pores act as flaw sites. EA monitoring is shown to be an effective and rapid technique to evaluate the damage mechanisms in fibre-reinforced ceramic composites in real time. Using this technique, relationships between microstructure and mechanical properties and their effects on damage behaviour of composite materials can easily be correlated for design purposes.

4. Conclusion

The damage mechanisms of mullite fibre (Nextel 720^{TM}) reinforced alumina ceramic matrix composites with two different porosity contents are investigated under tensile tests using acoustic emission technique. Samples with high porosity level (30%) and bigger average pore size (200 nm) show lower tensile strength compared to the composite sample with lower porosity and pore size of 20% and 90 nm, respectively. It is also found that porosity level influences the acoustic emission response during the tensile loading, as both amplitude and the energy values of acoustic events of the sample with lower porosity level are observed to be higher than those with higher porosity contents. Acoustic emission results supported by the SEM observations indicate that during the tensile tests of the sample with higher level of porosity, large pores within the composite act as crack initiation sites and therefore many fibres fracture as soon as the tests start resulting in lower mechanical properties. Based on the energy level of acoustic events, it is also found that during the tensile tests, delamination of the fibres from the matrix takes place with low energy level then multiple matrix cracks form with moderate energy level and finally reinforcement fibres break with high energy level before the composite's final failure.

References

[1] K.K. Chawla, Ceramic Matrix Composites, second ed, Kluwer, Academic Press, Norvell (MA), Dordrecht, The Netherlands, 2003.

[2] C. Kaya, E.G. Butler, A. Selcuk, A.R. Boccaccini, M.H. Lewis, Mullite (Nextel 720^{TM}) fibre-reinforced mullite matrix composites exhibiting favourable thermomechanical properties, J. Eur. Ceram. Soc. 22 (2002) 2333–2342.

[3] K.K. Chawla, Z.R. Zu, J-S. Ha, Processing, structure and properties of mullite fiber mullite matrix composites, J. Eur. Ceram. Soc. 16 (1996) 293–299.

[4] C. Kaya, X. Gu, I. Dawery, E.G. Butler, Microstructural development of woven mullite fibre-reinforced mullite ceramic matrix composites by infiltration processing, Sci. Technol. Adv. Mater. 3 (2002) 35–44.

[5] C. Kaya, F. Kaya, H. Mori, Damage assessment of alumina fibre-reinforced mullite ceramic matrix composites subjected to cyclic fatigue at ambient and elevated temperatures, J. Eur. Ceram. Soc. 22 (2002) 447–452.

[6] C. Kaya, E. G. Butler, A. R. Boccaccini, M. H. Lewis, Processing and Characterisation of mullite (Nextel^{TM} 720) fibre-reinforced mullite matrix composites from hydrothermally processed mullite precursor, in: W., Krenkel, R., Naslain, H., Schneider, (Eds.), in: Proceedings of High temperature ceramic matrix composites, Wiley-VCH, Germany, 2001, pp. 639–644.

[7] A.G. Beattie, Acoustic emission, principals and instrumentation, J. Acoust. Emission 2 (1983) 95–128.

[8] F. Kaya, Ph.D. thesis, The University of Birmingham, UK, 2003.

[9] G.N. Morscher, Modal acoustic emission of damage accumulation in a woven SiC/SiC composite, Comp. Sci. Technol. 59 (1999) 687–697.

[10] G.N. Morscher, A.L. Gyekenyesi1, The velocity and attenuation of acoustic emission waves in SiC/SiC composites loaded in tension, Comp. Sci. Technol. 62 (2002) 1171–1180.

[11] G.N. Morscher, Stress-dependent matrix cracking in 2D woven SiC-fiber reinforced melt-infiltrated SiC matrix composites, Comp. Sci. Technol. 64 (2004) 1311–1319.