Microstructural development of woven mullite fibre-reinforced mullite ceramic matrix composites by infiltration processing

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Abstract

Mullite fibre (Nextel 720\textsuperscript{TM})-reinforced mullite ceramic matrix composites (CMCs) with zirconia weak interface were fabricated from heterococagulated nano-size boehmite/amorphous silica powder particles dispersed in water, using electrophoretic deposition (EPD) and pressure filtration (PF). The nano-size mullite precursor was first prepared and characterised in terms of short-range particle–particle interactions and particle size distribution. Woven Nextel 720 mullite fibres were first desised and then coated with hydrothermally derived zirconia using dip-coating. EPD was performed under constant voltage conditions with varying deposition times, to infiltrate the dispersed powder suspensions into mullite fibre preforms, enabling the parameters necessary for good deposition of stoichiometric mullite to be established. EPD formed bodies were further consolidated using PF. The EPD/PF prepared green body specimens were dried under controlled atmosphere conditions before being sintered at 1200°C for 2 h in air. Mullite fibre mats were fully infiltrated using EPD parameters of 12 V DC applied voltage with 4 min deposition time, then eight EPD infiltrated fibre mats were further consolidated together using PF. The resulting CMC produced contained 35 vol\% fibre loading and showed 81\% theoretical density after sintering at 1200°C for 2 h. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Mullite fibre; Boehmite sol; Silica; Mullite precursor; Electrophoretic deposition; Zirconia interface

1. Introduction

Mullite (3Al\textsubscript{2}O\textsubscript{3}-2SiO\textsubscript{2}) is a very promising material for high temperature applications up to 1400°C due to its high refactoriness, low creep rate, low thermal expansion and thermal conductivity, good chemical and thermal stability as well as good thermal shock resistance [1,2]. Colloidal processing of mullite reduces the sintering temperature as a result of increased chemical homogeneity, the absence of particle agglomerates, and the development of a uniform green microstructure, resulting in the formation of stoichiometric and glassy phase free orthorombic mullite in the temperature range of 1150–1350°C via direct reaction of alumina-rich and silica-rich components of the gel.

Oxide fibre-reinforced mullite matrix composites have attracted great interest for high temperature applications due to their inherent oxidation resistance at high temperature, good thermal shock resistance and damage tolerance properties [3]. The main problem during the processing and high temperature application of ceramic matrix composites (CMCs) is the reaction between the fibre and the matrix, resulting in a strong bonding between them and as a result of this, a sharp decrease in thermomechanical properties of the composite is inevitable. Therefore, the fabrication of fibre reinforced CMCs require the fibre-matrix interface to be weak. In this way, when the composite is deformed, cracks propagate through the matrix until they meet a fibre. Because the interface between the two phases is weak, the separating matrix causes the fibres to pull from either side of the advancing crack front, rather than allowing the crack to continue through the fibre. The energy dissipating mechanisms of friction and crack deflection, which results from this pullout serve to toughen the materials. Fibre coatings, which are essentially an interphase between the fibre and matrix may also be required in some cases to act as reaction barriers or as a protection to fibre oxidation [4]. Zirconia, which is known to be chemically inert in combination with certain other oxide materials, is an important ceramic coating materials for high-temperature structural applications [5].

The electrophoretic deposition (EPD) technique relies on the presence of small charged particles within a colloidal suspension, which, on the application of an electric field,
will move and deposit onto an oppositely charged electrode. Electrophoresis is the movement of the small particles caused by a charge on the surface of the particle with respect to the suspending medium. The fabrication of fibre reinforced mullite composites using EPD requires the matrix precursor ceramic particles to be nano-size and in suspension. The properties of the sintered matrix will then be influenced significantly by the compositional homogeneity and stability of the suspension, and the fabrication techniques. Suspension stability can only be achieved by controlling the short-range particle–particle interactions, which will in turn affect the rheological behaviour of the suspensions [6]. To obtain a uniformly infiltrated green microstructure, EPD requires a kinetically stable, well-dispersed suspension having the possible highest solids-loading but a relatively low viscosity, which affects the particle electrophoretic mobility, and hence, deposition efficiency [7]. Furthermore, the number of charged particles deposited per unit area of the electrode/fibre is influenced strongly by the process time, electrode separation and applied potential [8]. This technique requires only low-cost equipment and offers new possibilities for the design of ceramic monoliths or fibre-reinforced composites with more uniform microstructures.

The EPD technique has been used to fabricate SiC–TZP ceramic composites [9], and also very successful applications of the technique to monolithic and fibre reinforced composites have been reported [10–15]. Much work has also been performed on the fabrication of woven ceramic fibre reinforced CMCs using alumina and silica slurry; both separately and in conjunction via a process which we have been developing for CMC fabrication since 1991 employing EPD, electrophoretic filtration deposition, and pressure filtration (PF) [16–28]. Previous studies using mixed sols have shown that preferential species migration during EPD leads to a loss of stoichiometry, and is influenced by the size ratio of the sol species involved [7]. Recently, a comprehensive study on the kinetic mechanism of EPD was published [24].

The present study examines the feasibility of fabricating mullite fibre-reinforced mullite CMCs from heterocoagulated alumina/silica precursors via a single-infiltration EPD, followed by pressureless sintering. A dip coating technique was used to coat the mullite fibre with zirconia in order to create a weak interface. Zirconia coating of fibres using such methods allows the nano-sized sol particles, produced by hydrothermal processing, to penetrate between the substrate fibre tows and adhere to the fibres by wetting. The relationships between the mullite precursors employed — the EPD process parameters and the microstructure of the final sintered product were correlated.

2. Experimental work

2.1. Fibre preparation

8 HS satin woven mullite (Nextel™ 720, 3M, USA) fibre mats were used in this work. It is very important in any coating procedure to ensure that the substrate material is properly prepared before the application of the coating. The fibre mats were pre-treated before the coating by desizing to remove the organic protection layer from the fibre, followed by preparing a negative surface by soaking in an ammonium salt of polymethacrylic acid. This process maximises the electrostatic attraction between the fibre surface and the positively charged zirconia particles in the coating sol.

2.2. ZrO₂ sol coating procedure

In this work, the ZrO₂ sol used to coat the fibres was synthesised by a hydrothermal process, as described previously [5]. Briefly, zirconium acetate precursor material (ZrO(CH₃COO)₂, Mel Chemicals, UK) was processed in an autoclave at 220°C under autogenous pressure for 2 h. The resultant product, which remains after autoclaving is a zirconia sol suspended in an acetic acid solution. The sol was then dried at 110°C for 1 h, calcined at 600°C for 4 h, to remove excess acetics and dispersed in water. The calcined sol solution had a pH of 2, indicating that the zirconia sol particles retained an adsorbed layer of acid throughout the calcining procedure. This adsorbed acidic layer produced a positive surface charge on the zirconia particles when redispersed in water.

During zirconia coating, the fibre mats were immersed in the zirconia sol under sonication causing the positively charged zirconia particles to adhere to the negatively charged fibre surface. The immersed fibre mats were extracted to air through an octanol layer, which is a less dense immiscible liquid floating above the zirconia sol, to prevent intra-tow filament adherence. Afterward, the fibre mats were dried in air and then fired to strengthen the coating layer on fibre surface. A schematic diagram of zirconia coating process is shown in Fig. 1.

2.3. Mullite precursor preparation

To produce mullite fibre-reinforced-mullite (3Al₂O₃·2SiO₂) multilayer CMCs, commercially available woven type mullite (Nextel 720) fibre mats were used as the reinforcement material. The powders and sols which were used in this study are shown in Table 1. To prepare the alumina–silica mixed sol for the synthesis of stoichiometric 3:2 mullite (i.e. after sintering, 71.82 wt% alumina and 28.18 wt% silica), the base boehmite sol was stirred vigorously whilst the fumed silica powder was added to boehmite sol at the rate of 0.5 g/min; this low rate of addition was used in order to prevent the formation of large heterofocculated clusters [7]. Simultaneous ultrasonic agitation was used to enhance the powder dispersion, while the solids-loading was kept at 30 wt% of the dispersion liquid. The pH of the suspension was adjusted to 4.5 using aqueous ammonia additions; because, according to the manufacturers, the dispersion stability of the alumina sol is
maximised at this pH value. Our experimental observations confirmed this fact, in that the boehmite–silica mixed suspension gelled at pH values much higher or lower than 4 [6]. Kinetically stable, well-dispersed suspensions of boehmite and silica particles were thus obtained at a pH of 4.5. At this pH, the alumina particles are positively charged and surrounded by the negatively-charged silica particles, and this heterocoagulated particle cluster behaves effectively as a single negatively-charged sol particle [7]. The particle size of the mullite precursor was measured using a X-ray Disc Centrifuge (BI-XDC) system.

2.4. Electrophoretic deposition

An EPD cell which allows the electrode separation to be varied was designed as shown in Fig. 2. A filter membrane was placed adjacent to the positive electrode before applying the voltage in order to prevent gas bubbles being incorporated within the deposited ceramic matrix. A foldable holding-cell was used to hold the electrode, filter and fibre mat together. After the fibre mat was placed in the sol, the system was vacuum degassed to remove any entrapped air, and then the cell electrodes were connected to a 0–60 V DC power supply. EPD was performed subsequently under constant voltage conditions (12 V) using varying deposition times (2, 4, 6 and 8 min) and electrode separation distances at (1, 1.5, 2 and 2.5 cm). This enabled the EPD process parameters necessary for good infiltration to be established. Under the applied electric field, heterocoagulated composite particles possessing a net negative surface charge migrated towards the positive electrode, infiltrating the fibre mat and being deposited out of suspension until a sufficient matrix thickness enveloping the fibre mat, was achieved. Different fibre mat thicknesses were infiltrated to determine the deposition thickness and the pore distribution within the

![Fig. 1. Schematic diagram of zirconia coating on fibre mat using dip-coating technique [5].](image)

![Fig. 2. Schematic diagram of the custom-built EPD cell for producing fibre-reinforced CMCs.](image)

| Material  | Form            | Average particle size (nm) | Particle shape | pH stability range | Solids-loading (wt%) |
|-----------|-----------------|-----------------------------|----------------|--------------------|----------------------|
| Remal A20<sup>a,b</sup> | Boehmite sol     | 50                          | Lath           | 4.5                | 20                   |
| Aerosil 200<sup>c</sup> | Silica powder   | 14                          | Spherical      |                    |                      |

<sup>a</sup> Particle size confirmed using both TEM and particle size analysis.
<sup>b</sup> Remet Corp., USA.
<sup>c</sup> Degussa Ltd., Germany.
fibre mat. Following EPD infiltration, eight electrophoretically deposited fibre mats were compacted (in green state) in order to obtain 30–35 vol% fibre loading within the final CMC using PF. The PF technique, which involves the mechanical application of pressure to a slurry in order to force the suspension through a filter assembly, was employed for the formation of mullite green bodies. This colloidal wet forming method eliminates the use of some intermediate processing steps, such as drying and milling of the sol derived materials [23]. In these experiments, a constant load of 20 kN was applied cyclicly using a constant ram displacement rate of 0.5 mm/min. After reaching the maximum load, the ram displacement was held for 10 min and then the load was removed using the same rate. The consolidated green samples contained were first kept in a humidity (from 80 to 55% relative humidity) controlled chamber for 2 days to allow the residual water to remove slowly from the green body, thus preventing the formation of any cracks, followed by 1 day drying in normal air. The resulting deposited/pressure filtered multilayer specimens were then dried using a two-step humidity drying process. For the drying, specimens were first kept in a humidity-controlled chamber (85% relative humidity) for one day then in another chamber (65% relative humidity) for another day, and finally left to normal air for one day. The fibre-reinforced CMC samples were sintered at 1200°C for 2 h in air using a 3°C/min heating and cooling rate. EPD deposited matrix materials were also extracted between the deposited fibre mats in the green state and then sintered at 1400°C for 3 h in order to determine the mullite formation temperature.

2.5. Microstructural characterisation

All coated fibre mats were analysed by scanning electron microscopy (SEM). For the preparation of coated fibre mats for cross-sectional SEM, the fibre mat was placed in a vacuum chamber for 20 min, vacuum-impregnated with Epofix resin (Struers Tech A/S, Denmark), and then left overnight to harden followed by sectioning using a diamond saw at very low speed. Afterward, the sample was remounted using cold mounting powder with liquid hardener (MetPrep Ltd, UK). The mounted samples were ground and polished to 1 μm for examination using SEM (JEOL 5410, Japan). For SEM work, all samples were additionally gold coated.

A Philips CM 20 TEM was used to observe and characterise the sol particle shape, size and degree of agglomeration, as well as the nano-scale particle–particle interactions. TEM chemical analysis of the sintered specimen was then conducted, using a JEOL 4000 FX TEM equipped with EDX analysis. SEM (Hitachi S-4000 Field Emission Gun SEM) was employed to characterise the various microstructural features of the infiltrated and sintered mullite precursor powder bodies, such as grain shape and size and porosity distribution and location. Powder samples of the material deposited in between the layers of mullite fibre in each of the EPD-infiltrated green compacts were extracted. These samples were then subjected to differential thermal analysis (DTA) in order to determine the phase transformation temperatures. Other samples of this powder were calcined at 1400°C for 3 h and then analysed using X-ray (Cu Kα radiation) powder diffraction to identify the phases present.

3. Results and discussion

It has been recognised that the key factors in the dip-coating process are the wetting of the fibres by the ZrO2 sol and the electrostatic attraction between the fibre surface and ZrO2 sol particles. Previous work by the authors has shown that the type of zirconia sol used in the coating process is of paramount importance to the success of the technique. Several commercial zirconia sols have been investigated during the development of this technique, but it has been found that only the ZrO2 sols synthesised hydrothermally from zirconium acetate solution have the desired properties for the successful dip-coating of oxide woven fibre mats [5].

Fig. 3 shows an SEM backscattered electron image (BEI) of a hydrothermally synthesised ZrO2 sol particle coating on a fibre mat. The light regions around the fibres correspond to the presence of ZrO2, indicating a coating thickness of approximately 1–2 μm with minimal inter-fibre bridging. The average zirconia particle size is small enough to infiltrate in between the fibre mat intra-tow regions. Since the hydrothermally produced zirconia particles retain a layer of adsorbed H⁺ ions during and after the 600°C, 4 h calcination stage, each discrete zirconia particle exhibits a net positive surface charge on redispersion. Given that this charge is dissimilar enough to the negatively charged fibre surfaces, it promotes particle–fibre mutual electrostatic attraction. It is suggested that this novel behaviour of the hydrothermally
synthesised zirconia sol is responsible for its uniquely successful coating of woven fibre mats using this dip-coating method.

Fig. 4 shows the particle electrophoretic mobility data for the nano-size boehmite, fumed silica (Aerosil 200), and boehmite–silica aqueous sol suspensions as a function of sol pH. From these data, it is evident that the silica particles are negatively-charged over the whole pH range, whereas the boehmite particles are positively-charged below pH 8.0 and negatively-charged above pH 8.0. Hence, under the conditions used to obtain a stable mixed sol (pH 4.5), there is an electrostatic attraction between the positively-charged, boehmite particles and the negatively-charged silica particles, enabling the resultant heterocoagulated sol particle clusters to move as single, negatively-charged composite particles.

Fig. 5 shows the particle size distribution (% cumulative mass distribution, smaller than) of a mullite precursor prepared from Remal A20 boehmite sol and Aerosil 200 fumed silica powders. From the particle size distribution data shown in Fig. 5, it is clear that heterocoagulation of boehmite/silica takes place, resulting in homogeneous suspension microstructure, as there are no big agglomerates having particle sizes bigger than 300 nm which shows the absence of heteroflocculation within the suspension. Under these conditions, i.e. lath shape positively-charged boehmite particles are fully covered by negatively charged silica particles and these heterocoagulated clusters are expected to behave as a single powder having negative surface charge (also see Fig. 6) which will move and deposit onto a positive electrode under the application of DC voltage.

The TEM observations confirmed that heterocoagulated...
clusters of boehmite/silica particles formed as a result of mutual electrostatic attraction under the experimental conditions used, as shown in Fig. 6. It can be seen from a bright-field TEM micrograph that heterocoagulated clusters of silica/boehmite particles formed via electrostatic attraction as lath shape boehmite particles are fully covered by negatively-charged silica particles, resulting in formation of heterocoagulated cluster.

The results of the EPD trials showed that the optimum electrode separation distance was 15 mm with an optimum constant applied voltage of 12 V D.C. Under these conditions, an optimised deposition time of 4 min was found to be sufficient for the full infiltration of woven mullite fibre mat. The relationships between the deposition time and deposit thickness formed onto fibre mat are shown in Table 2. A deposition time of 2 min did not provide an acceptable deposit thickness to provide reasonable green strength. However, a deposition time of 4 min was found to be very effective in obtaining a deposit thickness of 650 μm. Deposition times higher than 4 min did not improve the deposit thickness significantly due to the increase in deposit resistance.

Fig. 7(a) shows the green microstructure of one of the tightest fibre tow regions, which was fully deposited/infiltrated with the mullite matrix via EPD. The use of EPD enabled the heterocoagulated boehmite/silica matrix to be infiltrated between two closely packed mullite fibres separated only by 200–300 nm, as shown in Fig. 7(b). In comparison, samples produced using only PF resulted in low green and sintered densities, as shown in Fig. 8(a) and detailed in Table 3. This confirms that a single PF step is not enough to infiltrate/deposit the inter/intra-tow regions of the fibre mat with matrix material. Using just PF provided a sintered density of 72% TD while the use of EPD and PF together resulted in a theoretical density of

| Deposition time (min) | Deposit thickness (μm) |
|-----------------------|------------------------|
| 2                     | 200                    |
| 4                     | 650                    |
| 6                     | 680                    |
| 8                     | 710                    |
processing of EPD coupled with PF can be successfully employed.

Fig. 9(a) shows a SEM image of a sintered mullite fibre (Nextel 720) reinforced mullite CMC specimen produced using optimised EPD parameters, PF, and dried in humid atmosphere, followed by sintering at 1200°C for 2 h (in this study, a relatively low sintering temperature (1200°C) was used in order to prevent the degradation (and loss in mechanical performance) of the mullite fibres during composite consolidation. It is seen that full and complete infiltration was achieved without the formation of any small internal or external cracks within the sintered matrix. Fig. 9(b) shows a dense matrix microstructure after sintering at 1400°C for 3 h. Quantitative EDX analysis of the matrix material showed in Fig. 9(b) indicates the presence of the stoichiometric 3:2 mullite composition (72.4 wt% alumina and 27.6 silica). Note that the Au peak is due to gold coating of the sample.

X-ray diffraction pattern (XRD) for the calcined powders extracted from the regions in between each layer of the mullite fibre mat at the post-EPD green-body stage, is shown in Fig. 10. The precursor containing boehmite and fumed silica produced mullite peaks, together with additional peaks corresponding to either excess cristobalite or excess alumina after sintering at 1200°C for 2 h as shown in Table 4. Only when using a sintering temperature of 1400°C for 3 h did the XRD patterns show the peaks for the desired crystalline orthorhombic 3:2 type mullite with small amount of residual alumina, as shown in Fig. 10.

The DTA traces (heating rate: 10°C/min) for the powder samples obtained from the regions between deposited fibre mats (some samples were also prepared from dried mullite precursor sols) exhibited a major exothermic peak at 970°C and a minor peak at 1250°C, as shown in Fig. 11. The former exotherm corresponds to the formation of an Al-Si spinel (2Al2O3·SiO2) which transforms to orthorhombic mullite at temperatures above 1200°C. This transformation is evident from the minor exothermic peak at 1250°C. The TG curve shows a total weight loss of about 25%.

4. Conclusions

It has been demonstrated that mullite fibre-reinforced mullite multilayer CMCs can be fabricated using a single-stage EPD process followed by PF and pressureless sintering and that this technique provides a means for producing cost-effective CMCs owing to the very short processing time. Furthermore, a zirconia weak interface was successfully deposited onto the mullite fibre using a dip coating technique. The sintering temperature was found to be a critical parameter in generating stoichiometric sintered mullite via EPD of a stable aqueous suspension of mullite composition heterocoagulated nano-size alumina/silica sol particle clusters. The maximum use temperature for the reinforcement fibre used in this work is 1200°C, thus higher

![Fig. 8. FEG SEM picture of the sintered (1200°C for 2 h) mullite fibre-reinforced mullite CMC showing partial deposition and presence of some large un-deposit regions when just PF is used as a processing technique (a) and fully deposited sintered CMC microstructure produced by EPD/PF and dried in humid atmosphere, followed by sintering at 1200°C for 2 h, (b) note the absence of any drying internal or external cracks within the matrix.]
sintering temperatures were not used for EPD formed CMC samples, however sintering studies were carried out on matrix material extracted between the deposited layers. A sintering temperature as low as 1400°C was found to be sufficient for the formation of glassy phase free stoichiometric 3:2 mullite. Overall, the technique was shown to be very effective in CMC processing using EPD for mixed suspensions comprising oppositely charged particles. It has been also shown that dense, homogeneous green mullite composition and sintered mullite microstructures can be prepared successfully only by controlling the short-range particle–particle interactions of the dissimilar particle species. The final mullite fibre-reinforced alumina + mullite CMCs having 35 vol% fibre loading were successfully produced using optimised EPD parameters of 12 V applied voltage and deposition time of 4 min. The sintered sample exhibited a sintered density of 81% theoretical density.

Table 4
Effect of the sintering temperature on the phase composition

| Sintering temperature (°C) | Phase composition     |
|----------------------------|-----------------------|
| 1200 (2 h)                 | Mullite, α-alumina, cristobalite |
| 1300 (2 h)                 | Mullite, α-alumina     |
| 1400 (3 h)                 | Mullite, α-alumina     |

Fig. 9. SEM images of (a) sintered mullite fibre (Nextel 720) reinforced mullite CMC specimen produced using optimised EPD parameters, PF, and dried in humid atmosphere, followed by sintering at 1200°C for 2 h. It is seen that full and complete infiltration was achieved without the formation of any small internal or external cracks within the sintered matrix and (b) dense matrix microstructure after sintering at 1400°C for 3 h. Quantitative EDX analysis of the matrix material showed indicates the presence of the stoichiometric 3:2 mullite composition (72.4 wt% alumina and 27.6 silica). Note that the Au peak is due to gold coating of the sample.
Fig. 10. XRD (Cu Kα) patterns for sintered samples produced from Remal A20 boehmite sol–Aerosil 200 amorphous silica mixed sols showing the presence of stoichiometric 3:2 mullite with some minor peaks of alumina after sintering at 1400°C for 3 h (α-alumina).

Fig. 11. DTA and TGA traces for the stoichiometric mullite precursor sol-derived powder produced from a mixed Remal A20 boehmite sol and Aerosil 200 amorphous fumed silica sol.

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