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Fabrication technologies for oxide–oxide ceramic matrix composites based on electrophoretic deposition

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Abstract

Electrophoretic deposition (EPD) was used to fabricate alumina matrix composites with high volume fraction of woven fibre mat (Nextel™ 720) reinforcement in a multilayer structure. Colloidal suspensions of Al_2O_3 nanoparticles in ethanol medium with addition of 4-hydrobezoic acid were used for EPD. Two different techniques were developed for fabrication of Al_2O_3 matrix/Nextel™ 720 fibre composites. The first method is a combination of standard EPD of single fibre mats with a subsequent lamination procedure to fabricate the multilayered composite. The second method involves the simultaneous infiltration of several (three or more) Nextel™ 720 fibre mats by EPD in a tailor-made cell. The composites exhibit a homogeneous matrix microstructure, characterised by a very high particle packing density and relatively low porosity after sintering at 1300 °C. The EPD cell allows production of relatively large bodies (10 cm diameter). By combination of the multilayer EPD infiltration and lamination processes developed here, thick ceramic matrix composite components (>10 mm thickness) can be fabricated, which opens the possibility of greater industrial application of the materials.

Keywords: Suspensions; Composites; Al_2O_3; Electrophoretic deposition

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1. Introduction

Continuous fibre-reinforced oxide ceramic matrix composites (CMCs) have attracted significant scientific and technological interest for high temperature structural applications in gas turbines, rocket engines, heat exchangers and hot filters, due to their low specific weight, damage-tolerant behaviour, oxidation resistance and high resistance to creep and thermal shock.[1–6] Significant research effort is being expended in the optimisation of CMCs, with particular emphasis on the establishment of reliable and cost effective fabrication procedures.[1–14]

Electrophoretic deposition (EPD) is the process by which charged particles in a liquid medium move under an applied potential towards an oppositely charged electrode and coagulate there to form a stable deposit.[15–17] EPD has been used for the production of CMCs with a variety of ceramic matrices and fibres.[15,16,18–25] The main advantages of EPD over a conventional slurry route are the reduced processing times and improved control over green body microstructure.[15,16] Aqueous or non-aqueous suspensions of ceramic (nano)particles are usually considered for forming the matrix, and both conductive (e.g. SiC Nicalon®, carbon) and non-conductive (e.g. Nextel™) fibres have been used as reinforcement.[18–25] In the case of non-conductive fibres, the fibre weave is placed in front of the deposition electrode and the ceramic deposit forms on the electrode and grow around and through the fibre mat.[15,25] A schema of a typical EPD cell, commonly used for the infiltration of single non-conductive fibre mats, is shown in Fig. 1.

This paper presents EPD based methods for the fabrication of multilayer Nextel™ 720 fibre-reinforced alumina matrix composites. Two techniques are described, based on:

(i) EPD of single Nextel™ 720 fibre mats and subsequent lamination using Al_2O_3 paste to form the matrix and

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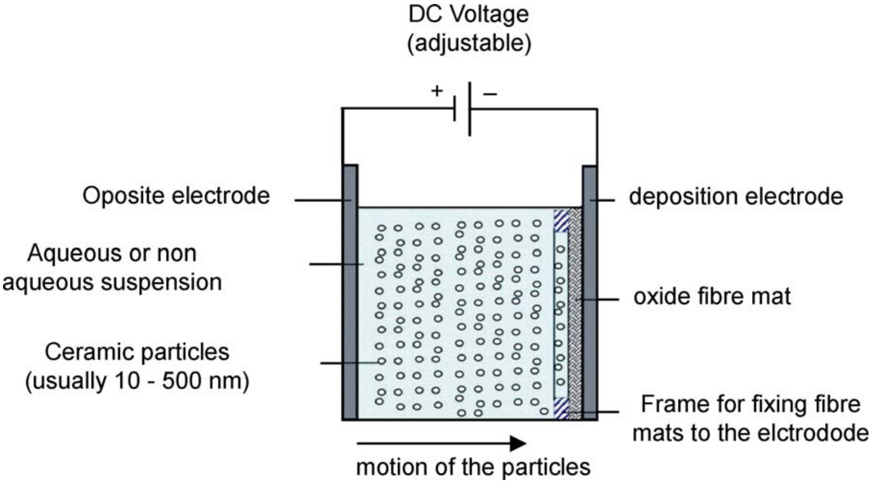

Fig. 1. Schematic representation of the typical EPD cell suitable for the infiltration of non-conductive fibre mats with ceramic particles (positively charged in this schema), for the fabrication of oxide–oxide ceramic matrix composites.

(ii) simultaneous infiltration of multiple Nextel^{TM} 720 fibre mats with Al_2O_3 particles by means of a single EPD step in a newly developed EPD cell.

2. Experimental procedures

2.1. Materials

The fibres used in this work are Nextel^{TM} 720 (3M Corporation, St. Paul, MN, USA) woven into eight-harness satin fabrics.[26,27] The woven fabrics contain ~400 individual filaments with diameters between 10 and 12 μm. Before using the fibres for composite fabrication, they were desized by a heat treatment at 700 °C in air for 10 min. The 100% α-alumina powder used for the matrix was the commercial powder AKP-50 (Sumitomo, Chemicals, Tokyo, Japan), which has a particle size distribution between 100 and 300 nm and a BET surface area of 10.6 m^2/g (manufacturer data).

2.2. Preparation of suspensions for EPD

Ethanol was chosen as the suspension medium to avoid any formation of porosity in the deposits during EPD due to gas evolution, which usually occurs in EPD from aqueous suspensions.[28] According to previous investigations on the electrochemical behaviour of Al_2O_3 particles in ethanol,[29] suspensions containing an optimised concentration of 25 wt.% Al_2O_3 powder were prepared. 4-Hydroxybenzoic acid (4-HBS) was used as dispersant agent in a concentration of 4 wt.% of solid content. This was the optimised concentration of dispersant found by electronic sonic amplitude (ESA) measurements, which showed that Al_2O_3 particles in ethanol exhibit a positive charge. The suspension was dispersed and homogenized in high frequency ultrasound equipment (Sonifier 450 Branson Ultrasonic SA, Caronge-Geneve, Switzerland). Subsequently, the suspension was left to rest for 24 h at room temperature before being used for EPD.

The electrochemical behaviour of the Nextel^{TM} 720 fibre immersed in ethanol with addition of 4-HBS was characterised using ESA signal measurements. For this experiment, desized Nextel^{TM} 720 fibres were ground using a ball mill (Type P6, Fritsch Company, Germany) to a fine powder of mean particle size 1.35 μm, with particle sizes varying between 0.5 and 2.5 μm, as determined by laser scattering particle analysis (Zetasizer, Malvern Instruments GmbH). The objective of the ESA signal measurements on the powered Nextel^{TM} fibres was to characterise the surface charge acquired by the fibres when immersed in ethanol and the effect of the 4-HBS dispersant on the surface charge of the fibre.

2.3. Composite processing

2.3.1. General description of the process

Two different techniques were developed for the fabrication of the Al_2O_3 matrix/Nextel^{TM} 720 fibre composites. The first method is a combination of EPD of single fibre mats (individual layers) with a subsequent lamination procedure to fabricate the multilayered composite. The second method involves the simultaneous infiltration of several (three or more) Nextel^{TM} 720 fibre mats by a single EPD step in an advanced tailor-made EPD cell. Fig. 2 shows the flow chart describing the basic steps involved. The following main processing parameters were used after a preliminary trial-and-error optimisation approach similar to the one discussed in the literature[30]: constant electric voltage = 100 V and distance between electrodes = 2 cm. In all cases, the CMCs green bodies after EPD were dried in air at room temperature and subsequently

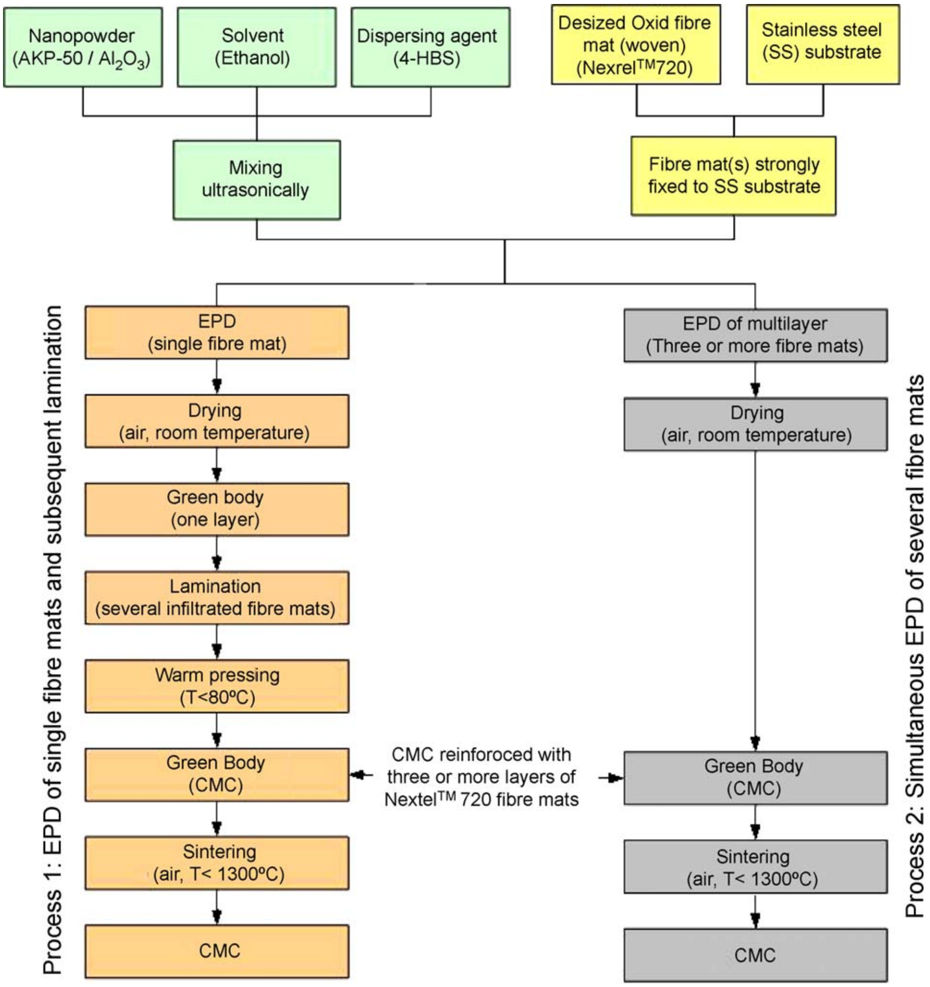

Fig. 2. Flow chart describing the two methods developed to produce ceramic matrix composites (CMCs) with alumina matrix and three or more layers of Nextel$^{\mathrm{TM}}$ 720 fibre mats as reinforcement.

a pressureless sintering process was used to densify the composites.

2.3.2. *Process I: EPD of single fibre mats and subsequent lamination*

Fibre mats of square shape with an effective area for infiltration of 45 mm × 45 mm were used. The stable suspension was incorporated in a typical (standard) electrophoretic deposition cell, as the one shown in Fig. 1. Under the application of the external electrical field, the positively charged Al_2O_3 particles in the suspension moved towards the oppositely charged electrode, before which the non-conductive Nextel$^{\mathrm{TM}}$ 720 fibre mat was placed. The electric field of 50 V/cm was kept constant for all experiments and the deposition time was 3 min. Electrophoretically infiltrated single fibre mats were dried in air at normal pressure and stored at room temperature in dissicators for later use.

For the lamination process, the procedure shown schematically in Fig. 3 was used. A paste was made by mixing polyvinylalcohol (PVA) with α-Al_2O_3 particles and deionised water according to the composition described in Table 1. The paste coating on wet surfaces of fibre mats previously infiltrated by EPD was applied using a Doctor Blade type method (see Fig. 3). The thickness of the coating was between 0.5 and 1 mm. The procedure was repeated for three layers and subsequently the composite (green body) was consolidated, forming a three-layer "sandwich" packet, by warm-pressing. The application of heat during pressing was required to reduce the viscosity of the paste, and thus to generate better conditions for impregnation and

Table 1
Composition of the paste used in the lamination procedure for CMCs fabrication (see Fig. 3)

Composition of the paste	Concentration
Non-ionized water	50 wt.%
α-Al_2O_3 (AKP-50) powder	50 wt.%
Polyvinilalcohol binder (Polyviol LL 6035) (20% solution)	10–20 wt.% of solid content

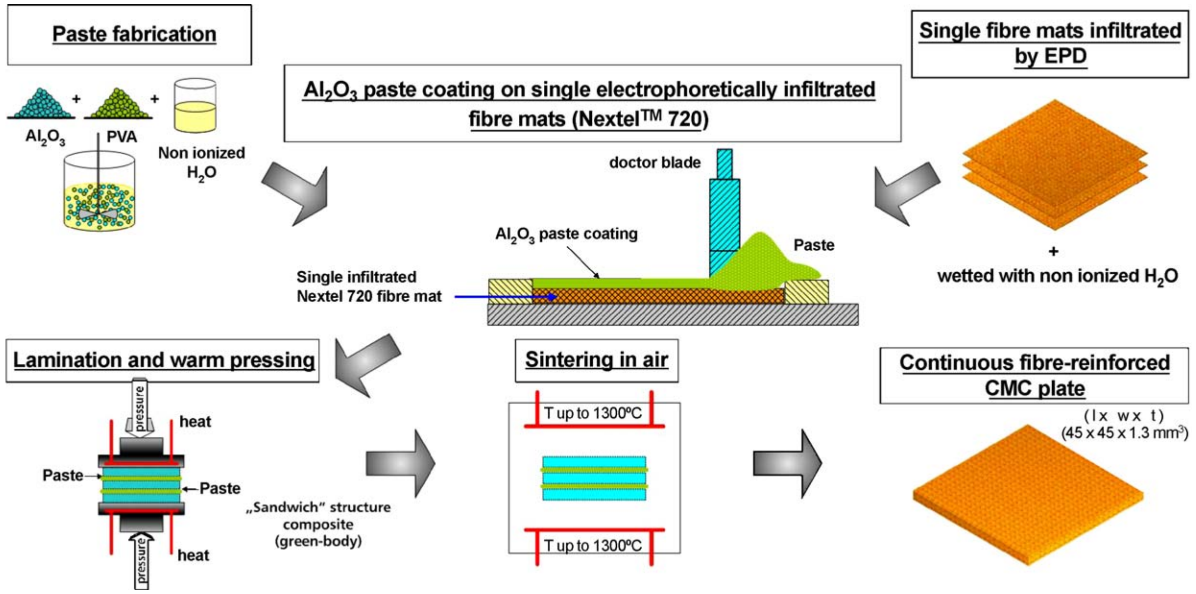

Fig. 3. Schematic diagram showing the different processing steps for fabrication of alumina matrix composites with Nextel^{TM} 720 fibre mats reinforcement. Fibre mats that had been previously electrophoretically infiltrated with Al_2O_3 particles were laminated by the doctor blade method and subsequently warm pressed and pressureless sintered.

interlocking between the single Al_2O_3 layers. The pressure was kept at <5 MPa in order to avoid damage of the fibres and the temperature was ~80 °C to avoid excessive evaporation of volatiles from the green body. Composite fabrication was completed with a sintering stage at 1300 °C in air using an electric furnace. The holding time was 1 h, heating rate was 10 °C/min and the cooling rate was 6 °C/min.

2.3.3. Process II: simultaneous EPD of several fibre mats

The main advantage of this direct procedure is to dramatically reduce the number of processing steps required for the fabrication of multilayer CMC components which are unavoidable in the conventional fabrication methods.[14] The technique involves the simultaneous electrophoretic deposition of the matrix material onto three or more fibre mats to manufacture multilayer CMCs in just one step (see Fig. 2). This method should also reduce manipulation of the infiltrated fibre mats before the material has been densified by sintering, thus reducing the possibility of microstructural damage. A new EPD cell was designed and built for this purpose, as shown in Fig. 4. The cell provides a relatively large effective deposition area allowing the fabrication of components of 100 mm in diameter. For the present experiments, the space between the stainless steel electrodes was fixed at 2 cm. The deposition time was 8 min. The EPD process was always carried out in vertical position, i.e. the movement of the particles was in direction opposite to the gravity force, in order to reduce agglomeration effects of the suspension and also to promote the infiltration of the smallest and lightest particles into the fibre mats avoiding preferential deposition of the heaviest (and largest) agglomerates. After EPD, the composite green bodies were dried for 24 h in air at room temperature and densified by sintering under the same conditions given above.

2.4. Microstructural characterisation

All composites were characterised by scanning electron microscopy (SEM) (XL 30, FA Philips/FEI Eindhoven and LEO 1525, Gemini). For SEM observation, composite samples in "green" state and after sintering were infiltrated with epoxy resin under vacuum, then sectioned using a diamond saw and polished using a diamond suspension to 1 μm finish.

3. Results

3.1. Characterisation of the suspension for EPD

A suitable suspension for electrophoretic deposition should contain ceramic particles with high surface charge well dispersed in a liquid of high dielectric constant.[17] ESA signal measurements allowed us to assess the effect of 4-hydroxybenzoic acid as additive on the stability of suspensions of Al_2O_3 particles in ethanol (25 wt.%) and on particle mobility. It was found that the surface charge of the Al_2O_3 particles in ethanol was always positive with a maximum value of 450 μPa m/V at concentrations of 4-HBS >3 wt.% of the solids content.[29] From these results, the concentration of 4-hydroxybenzoic acid for the present investigation was chosen as 4 wt.%.

Fig. 5 shows the results of the ESA signal measurements on the milled Nextel^{TM} 720 fibre in ethanol suspension

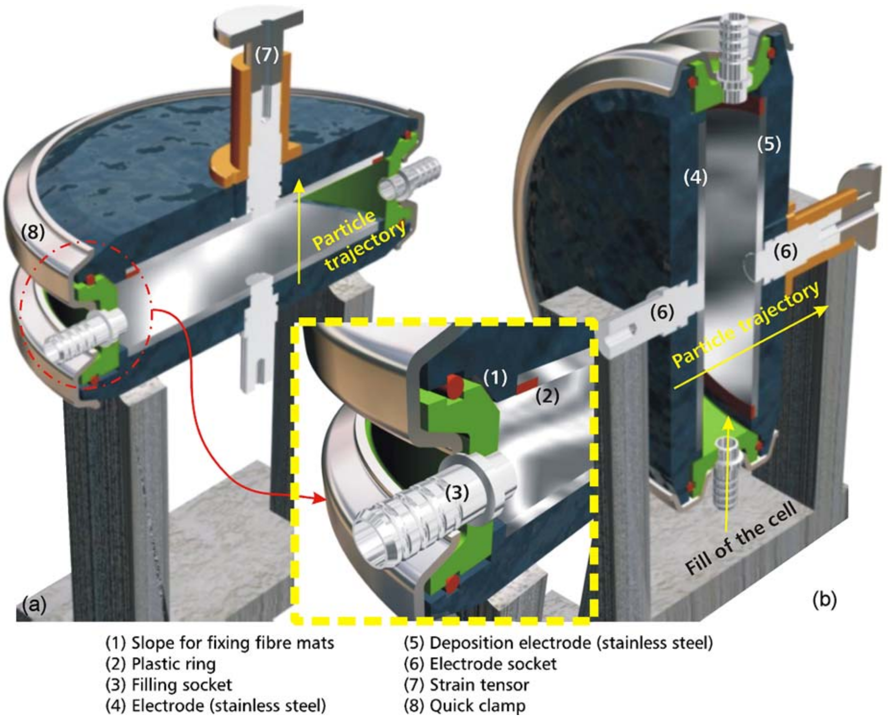

Fig. 4. Design of the new electrophoretic deposition cell for the simultaneous infiltration of several Nextel^{TM} 720 fibre mats: (a) schematic diagram showing the position of the cell for vertical deposition, with direction of particles movement opposite to the gravitation force; and (b) schematic diagram showing the cell for horizontal deposition direction.

with additions of the same additive (4-HBS). This curve demonstrates that milled Nextel^{TM} 720 fibres have a positive polarity in ethanol suspension. It is also observed that the original value of ESA signal increases from ∼107 to 170 μPa m/V for concentrations of 4-HBS of up to 0.8 wt.%. For higher concentrations of 4-HBS, the signal value stabilized at 170 μPa m/V. Thus, at a 4-HBS concentration of 4 wt.%, chosen for the present study, the fibres and the Al_2O_3 particles posses the same positive polarity, which has advantages regarding the mechanism of electrophoretic deposition, as discussed below (Section 4.1).

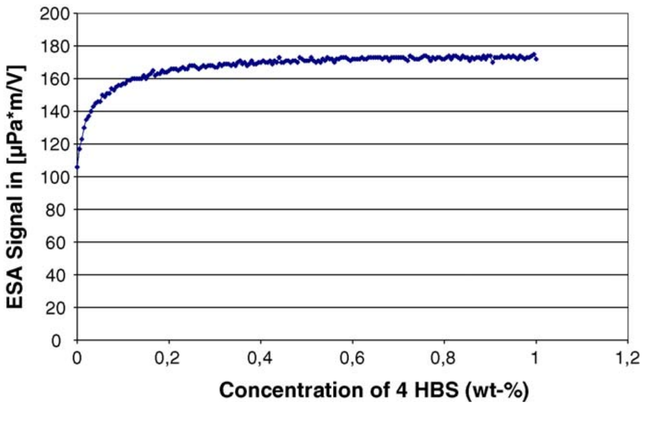

Fig. 5. ESA signal in ethanol suspension of milled Nextel^{TM} 720 fibres as function of 4-hydroxybenzoic acid concentration.

3.2. Fabrication of Al_2O_3 matrix/Nextel^{TM} 720 fibre composites by EPD

3.2.1. Process I: EPD of single fibre mat and subsequent lamination

A fibre mat showing high-quality infiltration of Al_2O_3 particles by EPD is shown in Fig. 6a. The mean particle size of the Al_2O_3 starting powder, in the range 100–300 nm, is confirmed in Fig. 6b, which demonstrates that this particle size is appropriate to effectively infiltrate the Nextel^{TM} 720 fibre mats. This image qualitatively indicates also the high particle packing density in the deposited matrix. The high-quality of the infiltration and the homogeneous matrix microstructure confirm thus the suitability of the suspension composition and the selected concentration of 4-hydroxidbezoic acid. This behaviour is in broad agreement with extensive evidence in the literature on the high versatility of the EPD technique to infiltrate 2D and 3D fibre performs with ceramic (nano)particles.[15,16,18–25]

The SEM micrographs in Fig. 7 compare the microstructures of the EPD-infiltrated Nextel^{TM} 720 fibre mat in “green” and sintered state. It is possible to observe isolated pores in the sintered matrix. This is an indication that the organic additive used in the EPD suspension has been burned out during the sintering process, leaving some residual porosity. Inspection of Fig. 7b indicates also that the sintering temperature used (1300 °C) might have been too high because some chemical reaction at the fibre/matrix interface is apparent. This effect is detrimental for the mechanical properties of the composite,

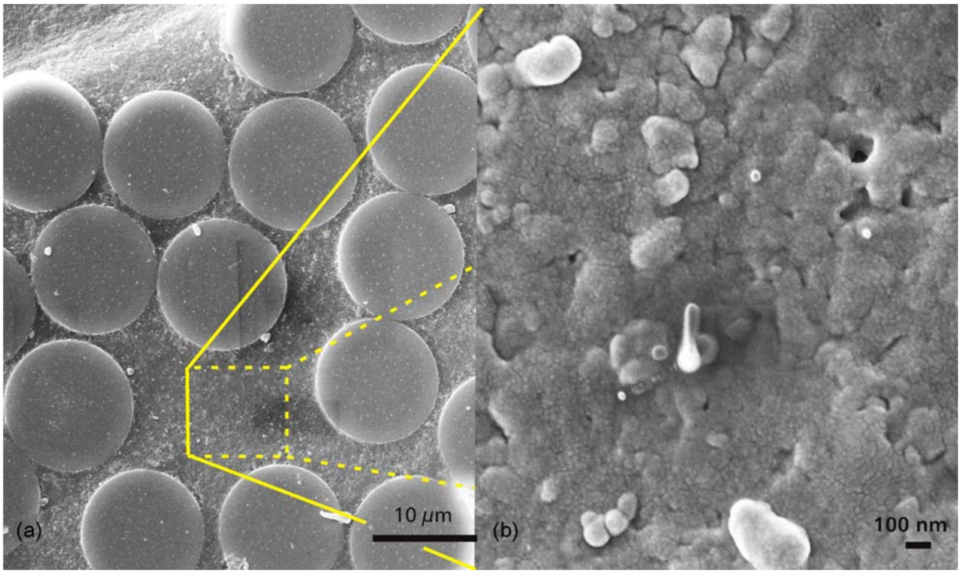

Fig. 6. SEM micrographs of a Nextel^{TM} 720 fibre mat infiltrated with alumina particles by EPD showing: (a) complete and homogeneous infiltration of the Nextel^{TM} 720 fibre mat; and (b) high "green" density of the matrix.

since it would limit fibre "pull-out" during fracture and the material would fail in a brittle manner.[1] The possibility of obtaining an efficient densification of the matrix using shorter sintering time at lower temperatures must be therefore explored.

Fig. 8a shows a schematic diagram describing the interlocking mechanism of Al_2O_3 particles which takes place during the warm lamination process. In Fig. 8b, a SEM micrograph of the microstructure of a composite fabricated by lamination is shown. During the lamination process at ~80 °C, Al_2O_3 particles from the electrophoretically infiltrated fibre mats and from the paste used for lamination come into close contact. Pores in the "green" matrix are filled by Al_2O_3 particles being pressed together due to the relatively low viscosity of the PVA in the paste at ~80 °C. Consequently, a compact of high "green" density and relatively homogeneous microstructure can be obtained, as observed in Fig. 8b.

3.2.2. Process II: simultaneous EPD of several fibre mats (Nextel^{TM} 720)

Fig. 9 shows a representative SEM micrograph of a sintered composite fabricated by simultaneous electrophoretic deposition of four fibre mats, using the cell designed in this study (Fig. 4). The micrograph reveals that the fibre tows were well infiltrated by the Al_2O_3 particles. In previous investigations, the potential of EPD to infiltrate several oxide fibre mats simultaneously has been suggested.[25] However, this is the first time that a high-quality infiltration of several non-conductive fibre mats by ceramic particles using a single EPD cycle has been demonstrated, which represents a substantial improvement in terms of saving processing time and

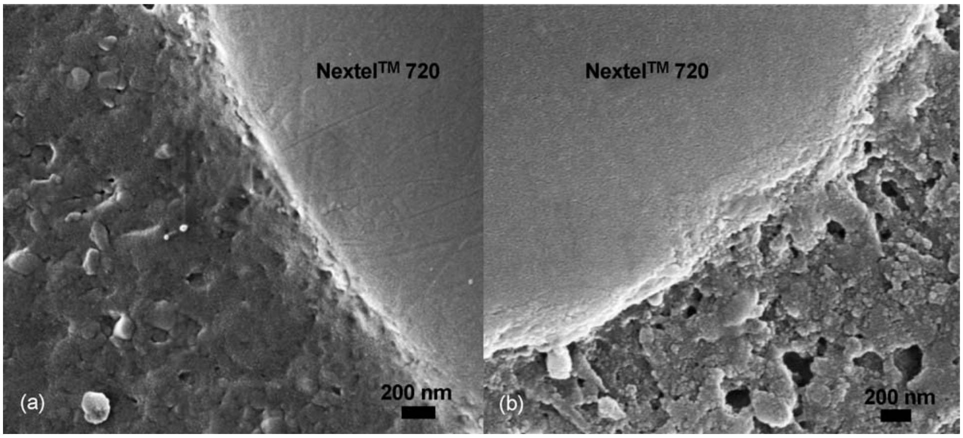

Fig. 7. SEM micrographs showing the interface region between a Nextel^{TM} 720 fibre and the alumina matrix: (a) "green body"; and (b) sintered composite (1300 °C for 1 h).

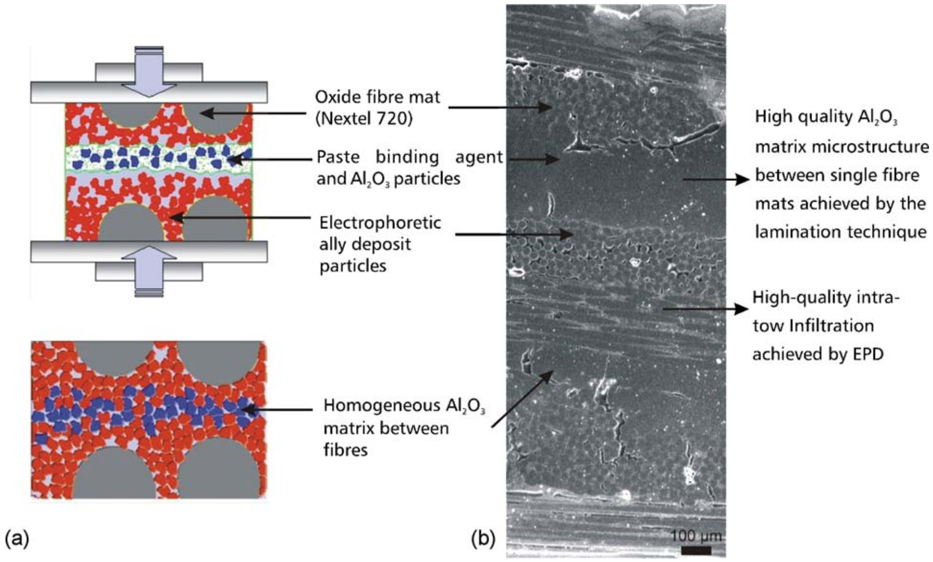

Fig. 8. (a) Schematic diagram depicting the mechanism of interlocking of Al_2O_3 particles during the lamination process between two electrophoretically infiltrated fibre mats; and (b) SEM micrograph of a sintered multilayer composite sample (1300 °C for 1 h) consisting of three fibre mats layers.

cost in comparison with the standard procedure of infiltrating single fibre mats in different EPD cycles. As Fig. 9 shows, full intra-tow and inter-tow infiltration has been achieved and a very compact structure with high fibre volume fraction was obtained without the need for subsequent processing steps. Moreover, it is seen (Fig. 9) that the gap between adjacent fibre mats is ∼50 µm, which is much smaller than that obtained in the laminated composites (Fig. 8). Matrix-rich regions, which are prone to microcracking and microstructural defects, are thus minimised. It should be also pointed out that these samples had a diameter of 10 cm, being therefore the largest ceramic composite components fabricated by EPD to date, as far as the authors are aware.

4. Discussion

4.1. Mechanism of EPD of Al_2O_3 particles onto oxide fibre mats

In the following, a phenomenological description is presented of the mechanism of electrophoretic infiltration of oxide fibre mats by ceramic particles exhibiting the same surface charge polarity, as in the present experiments.

As schematised in Fig. 10, it is proposed that the total trajectory of the particles before reaching the electrode can be divided in two regions. The first region, namely the "approaching trajectory", occurs in the suspension at a given

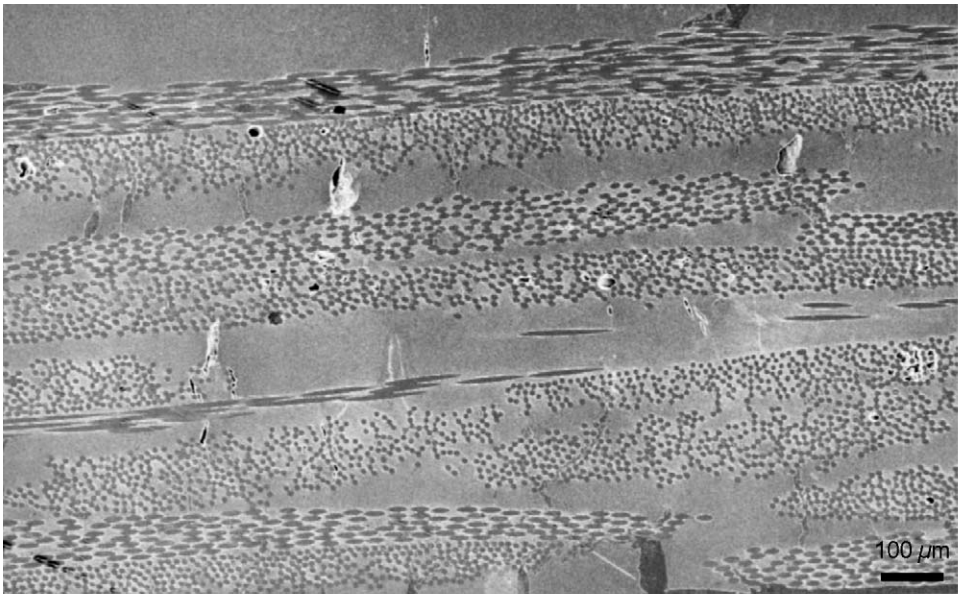

Fig. 9. SEM micrograph of a Nextel^{TM} 720 fibre-reinforced alumina matrix composite containing four fibre layers (fibre mats) fabricated by simultaneous electrophoretic deposition. The sample was sintered at 1300 °C for 1 h.

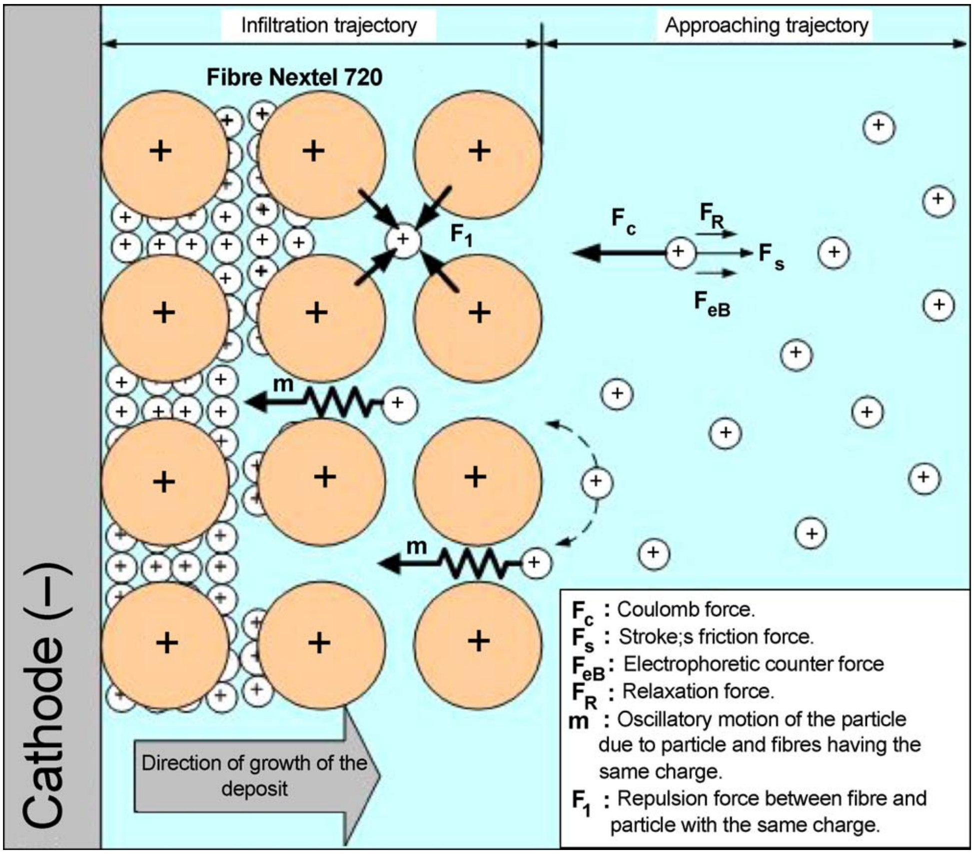

Fig. 10. Schematic diagram describing the motion of positively charged α-Al_2O_3 particles as they infiltrate a positively charged Nextel^{TM} 720 fibre mat during EPD.

distance away from the electrodes, where the particles move towards the deposition electrode only under influence of the externally applied electric field. The second region, the "infiltration trajectory", occurs close to the deposition electrode where the charge of the fibres influences the motion of the charged particles. In the ideal case of a stable suspension, the particles move as separate entities (no agglomeration), and they all move simultaneously in random manner due to the attraction and repulsions forces between them (DVLO-theory).[17] A simple linear motion of the particle under the external voltage can be assumed in the "approaching trajectory". In the "infiltration trajectory", however, additional interaction forces appear between particles and the charged fibres. In the present case, where fibres and particles possess the same charge, there are repulsion forces (F_1) acting between particles and fibres as shown in Fig. 10. For each particle, these forces depend on the relative distance between particles and fibres. Due to the applied external electrical field, each positively charged particle is attracted to the fibre mat because this is fixed to the cathode. However, the particles are repelled before they can reach the fibre surfaces (coagulation point) due to the positive charges on the fibres. It can be hypothesised that under the effect of the repulsive forces due to the surrounding fibres, the particles will follow the path with the fewest possible obstacles until reaching the next interstice between adjacent fibres. Thus, when the particles reach the electrode or the surface of previously deposited particles, they have no further possibility to move and so the electrophoretic ceramic deposit grows with a high particle packing density. For the case where fibre and particle have opposite surface charge, on the contrary, it is expected that coagulation will occur on the first layer of fibres encountered by the travelling particle. Consequently, the formation of a deposit on the outer fibre layer will block or at least make more difficult the movement of the particles towards the interior of the fibre mat, which could result in poor infiltration and low quality microstructure of the green body.

4.2. Comparison of the two processes

Both techniques developed here are convenient routes to the manufacture of multilayer Nextel^{TM} 720 fibre-reinforced alumina matrix composites. The method based on the simultaneous EPD of several fibre mats resulted in higher fibre volume fraction, higher matrix density and more homogeneous matrix microstructure than the standard method of EPD of single fibre mats. As assessed by SEM, less structural damage developed in the matrix during sintering. In addition, damage was minimised due to less extent of manipulation of the green body, as the intermediate steps of forming the composite by lamination were eliminated. This benefit becomes apparent when comparing Figs. 8b and 9. Nevertheless, the advantage of the method based on single layer EPD and lamination is that it offers the possibility to produce thick ceramic composite plates, without any limitation other than the number of extra lamination steps required. Indeed, the lamination of multilayer "green" bodies fabricated by simultaneous EPD of several fibre mats, effectively combining both methods, would lead to even larger and thicker CMC components.

5. Conclusions

Two processes for the manufacture of alumina matrix composites with multilayer Nextel^{TM} 720 fibre reinforcement based on EPD were developed and evaluated. Stable suspensions of Al_2O_3 particles (25 wt.%) in ethanol were used, with controlled addition of 4-hydroxicbenzoic acid as the charging additive. Measurements of the ESA signal of suspensions have confirmed the importance of obtaining the same sign of electric charge on both the fibres and particles. The first processing method, based on infiltration of single fibre mats and subsequent lamination, leads to composites of relatively high density, however, some microstructural damage in form of matrix microcracks developed during sintering. The second method involved the simultaneous infiltration of several fibre mats (up to 4) in a single EPD experiment. These composites exhibit a very homogeneous "green" microstructure, characterised by a very high particle packing density. By combination of the multilayer EPD infiltration and lamination processes, relatively thick components (>10 mm thickness) could be fabricated. The focus of current work is the optimisation of the densification heat treatment and the measurement of the mechanical properties of the composites.

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