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Processing and Characterisation of 2-D Woven Metal Fibre-reinforced Multilayer Silica Matrix Composites Using Electrophoretic Deposition and Pressure Filtration

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Abstract

A novel, cost-effective and rapid processing route including electrophoretic deposition (EPD) and pressure filtration (PF) has been developed for the fabrication of 2-D woven metallic fibre mat reinforced multilayer silica matrix composites. Commercially available silica sol containing ultrafine ceramic particles (15 nm) was used as the matrix whilst 2-D woven metal stainless steel 316L fibre mat was used as the metal reinforcement to produce a composite having 2-D isotropic properties. The colloidal silica sol was modified with boria and boehmite in order to produce a silica matrix which could be sintered at 900°C (the maximum use temperature for the particular fibre architecture employed), and with densification taking place before crystallisation. An in-situ *electrophoretic deposition (EPD) cell capable of measuring the weight gain in real time during deposition was designed. This technique enabled the woven fibre inter/intra tow regions to be infiltrated with the ultrafine silica particles in a very short time (2 min). Green bodies made from electrophoretically deposited fibre mats were further consolidated using pressure filtration. The EPD parameters were optimised in terms of time, voltage and deposition thickness as well as deposit formation rate. Microstructural observation indicated that the composites produced were dense and of high microstructural homogeneity.*

Keywords: electrophoretic deposition, composites, SiO_2, metal fibres, pressure filtration.

1 Introduction

Considerable research effort is being undertaken on the optimisation of fibre reinforced ceramic composite systems, with particular emphasis being placed on the development of reliable and cost-effective fabrication procedures.[1] These composite systems afford promise as a means of producing lightweight, structural materials combining high-temperature strength with improved fracture toughness and damage tolerance. Ceramic composites incorporating 2 or 3-D fibre reinforcements are, however, particularly prone to exhibiting uncontrolled microstructures and residual porosity. This is because it is extremely difficult to achieve complete infiltration of the matrix material into the fibre tows, where the intra-tow openings may be down to the order of <100 nm.

A novel, simple and inexpensive method for achieving complete infiltration of tightly woven fibre preforms has been developed recently.[2] It is based on the electrophoretic deposition (EPD) of colloidal ceramic sols into the fibre preforms. Using nanoscale ceramic particles in a stable non-agglomerated form and exploiting their net surface electrostatic charge characteristics whilst in colloidal suspension provides an appropriate means of effectively infiltrating the densely packed fibre bundles. To do this, the deposition electrode in an

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EPD cell is replaced by the conducting fibre preform, thus, the suspended particles will be attracted into and deposited within it. In a recent review article by Sarkar and Nicholson,[3] a complete description of the EPD technique and its applications in ceramic technology is presented. The feasibility of the EPD process to infiltrate ceramic woven fibre preforms has been demonstrated for single and mixed component sols in earlier studies.[2,4–8] Mainly SiC-based,[2,6–7] mullite[5–7] and alumina [6,8] woven fibre mats have been employed.

Metallic fabrics are commercially available also and are made from a variety of metals including stainless steel and special alloys (e.g. Hastelloy X). Besides a few reports on continuous ductile reinforcement of glass matrices,[9,10] however, no significant research has been devoted in recent years to metallic continuous fibre reinforcement of ceramics, despite the advantages they may offer over their ceramic-ceramic counterparts. These include an increased resistance to damage during composite processing due to the intrinsic ductility of metallic fibres and the possibility of exploiting their plastic deformation for composite toughness enhancement.[9] One reason for the lack of studies on these systems may be the general complexity of some of the processing technologies used for the fabrication of metal-ceramic composites with 2 or 3D interpenetrating microstructures (e.g. chemical reaction processes or the metal infiltration of porous ceramic preforms).[11,12]

The recent commercial availability of woven metal fabrics has, however, allowed them to be used in conjunction with the relatively simple EPD processing route. Preliminary studies have demonstrated the feasibility of infiltrating the fibre mats with boehmite sols and silica sols and using a dry pressing route to consolidate infiltrated mats to form composite preforms.[13,14] In this contribution, the use of the EPD technique *in situ,* in a more controlled manner, to prepare silica sol infiltrated 2-D metallic fabric preforms is considered. The preforms are further consolidated into multilayer composite preforms by a pressure filtration process rather than dry pressing prior to sintering at high temperatures.

2 Experimental Work

2.1 Materials

A commercially available colloidal silica sol (Bindzil PIC-P, Eka Nobel, Nobel Industries, Sweden) was used for the electrophoretic filtration and pressure filtration of the woven fibre mats. It is stable in the pH range 9–10, has a solids content of 30 wt% and the colloidal silica particles are hexagonal with an average particle size of 15 nm. At 20°C, the sol has a viscosity value of < 10 mPas and a density value of 1·2 g/cm^3. This colloidal silica sol was modified with additions of 2·5 wt% B_2O_3 (boric oxide) and 2 wt% alumina in the form of a boehmite (AlOOH) sol (Remal A20, Remet Corp). These additions were necessary in order to reduce the viscosity of the silica matrix, and delay the early crystallisation of the silica to cristobalite prior to densification during sintering,[15] respectively. The average particle size of the boehmite sol was 50 nm and the particles exhibited a lath shape. The final modified suspensions were first stirred magnetically for 15 h and then ultrasonic agitation was employed at 15 kHz for 20 min for further dispersion of the particle agglomerates present. It was envisaged that this final silica sol composition could be densified at temperatures below 1000°C before the onset of crystallisation. The particle sizes and the viscosity of the sols were measured using a BI-XDC (X-ray disc centrifuge) system and a computer-controlled rotary cone-plate rheometer (Carri-Med CSL-500 Viscometer), respectively.

The reinforcement elements were woven satin 316 L stainless steel continuous fibre mats laid-up in a 2D labyrinth architecture and sintered to form a durable, pleatable material ('Bekipor ST', Bekiterm FA, Belgium). The individual fibre diameter is in the range 10–25 μm and the weaves have a nominal thickness of 0·4 mm. This grade of stainless steel fibre mat can be used up to 900°C in an oxidising atmosphere and has a thermal expansion coefficient of $16{\cdot}8\times10^{-6}$ 1/°C (20–700°C).[16] It has been considered as reinforcing element in other glass and ceramic matrix composite systems fabricated recently.[13,14]

The surface charge properties of the silica particles and the stainless steel fibres were measured using a DELSA 440 surface charge analyser. In order to characterise the fibres it was necessary to produce a metal powder from them. This was achieved by sectioning a fibre mat to form fibre splinters, which were then milled further using a ceramic mortar and pestle. Suspensions having 0·05 wt% solids-loading were prepared in aqueous solutions of varying pH values by additions 0·025 N HCl and 0·015 N NH_4OH solutions and then particle electrophoretic mobility was measured as function of the pH of the suspensions.

2.2 *In-situ* electrophoretic deposition

The EPD technique relies on the presence of (nanosize) charged particles within a colloidal suspension, which, on the application of an electric field, will migrate toward and deposit on an oppositely charged electrode.[2,3] A schematic diagram of an *in-situ* electrophoretic deposition cell incorporating a

woven stainless steel fibre mat as the deposition electrode, which was designed for the present investigation, is shown in Fig. 1. The fibre mat acting as electrode was connected to a balance linked to a computer. The apparatus is able to record the weight gain per millisecond during the deposition process, i.e. in real time. 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 d.c. power supply. EPD was performed subsequently under constant voltage conditions (1, 2, 4 and 6 V) using varying deposition times (0·5, 1, 1.5, 2 and 3 min) with a constant electrode separation distance of 15 mm. This enabled the EPD process parameters of most importance in the formation of fully infiltrated dense green body compacts to be determined in terms of deposition voltage, deposition time, deposit thickness and the deposition rate. Under the applied electric field, ultrafine colloidal silica particles, possessing a net negative surface charge, migrated towards the positive electrode, i.e. the metallic fibre mat, infiltrating the fibre mat and being deposited until a sufficient matrix thickness, which enveloped the fibre mat, was achieved. The variation of the pore distribution within deposits formed under different EPD conditions was studied also.

2.3 Pressure filtration of deposited fibre mats

The pressure filtration (PF) technique, which involves mechanical application of pressure to a slurry in order to force the suspension through a filter assembly, was employed for the incorporation of the silica matrix between 5 electrophoretically deposited fibre mats, thus forming the composite green bodies. This colloidal processing method does not damage the fibres. The schematic diagram of the die assembly used is shown in Fig. 2. In these experiments, a constant load of 20 kN was applied using a constant ram displacement rate of 0·05 mm/min. After reaching the maximum load, the ram displacement was held for 5 min and then the load was removed using the same rate. The consolidated green composite samples (20×20 mm^2) containing 35–40 vol% metallic fibre were first kept in a controlled humidity atmosphere (80% relative humidity) for 2 days and then in a 55% relative humidity atmosphere for one day, followed by 2 days drying in normal air. The two-step-controlled humidity drying process prevented the formation of any cracks due to the slow removal of water from the green body. Dried samples were pressureless sintered at 900°C for 2 h in air. The green densities of fully dried samples were obtained from dimensions and weight measurements, whilst the Archimedes' technique was used to measure the sintered density of the composites. A series of composites, produced using only pressure filtration (without using EPD), are reported also, in terms of their green and sintered density as well as microstructural features.

2.4 Microstructural characterisation

Transmission electron microscopy (TEM) (Philips CM 20) was used to observe and characterise the silica sol particle shape, and size. Green specimens produced using different deposition voltages were mounted using vacuum impregnation and then polished down to 1 μm. The various microstructural features of the infiltrated and sintered silica precursor powder bodies were characterised using high resolution SEM, Hitachi S-4000 Field Emission Gun and Philips 5410 SEM equipped with a digital image analyser. Features investigated were: the degree of deposition and infiltration, porosity distribution and location, and grain boundary morphology. TEM chemical analysis of the sintered specimen was conducted also, using a JEOL 4000 FX TEM equipped with EDX analysis.

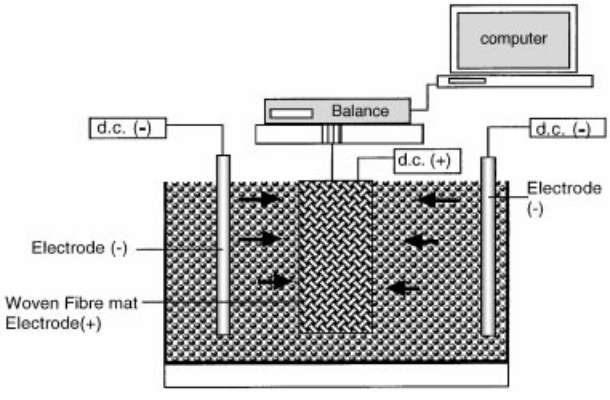


Fig. 1. Schematic diagram of an electrophoretic deposition cell incorporating a woven stainless steel fibre mat as the desposition electrode for producing metal fibre-reinforced silica matrix composites.

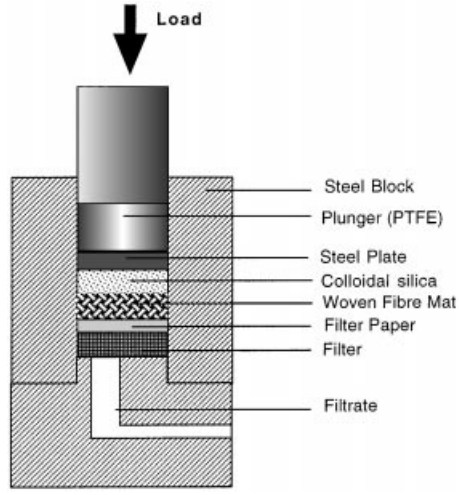


Fig. 2. Schematic diagram of the pressure filtration assembly for further consolidation of the woven stainless steel fibre mat reinforced silica matrix composites.

Powder samples of the material deposited in between the layers of stainless steel fibres 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.

3 Results and Discussion

Figure 3 shows bright-field TEM micrograph of the silica particles in the suspension used for the EPD experiments, illustrating that the suspension was very well dispersed and that there were no significant particle agglomerates. It is evident also that the silica particles are hexagonally shaped with a narrow particle size distribution and an average particle size of ~15 nm.

Figure 4 shows the particle electrophoretic mobility data for the nano-sized colloidal silica powders and crushed stainless steel fibres in aqueous suspensions as a function of the pH. From these data, it is evident that the silica particles and the metallic fibre mats are negatively-charged at the working pH value of 9·5. Thus, there is a very strong repulsive interaction between them at high pH values and this suggests that the use of simple dry pressing or colloidal dipping techniques may not be effective to produce dense fibre-reinforced composites with these materials. An external force, i.e. an electric field on the application of voltage, should be therefore, employed in order to force the negatively charged silica particles to move and deposit onto the fibre mat acting as the positive electrode. One can, therefore, conclude that EPD is a most convenient candidate processing technique, because it is possible to control the suspension medium-electrode interactions. The data show also that the most stable silica suspension (having the highest particle electrophoretic mobility) can only be achieved at pH values of 9 or 9·5

Figure 5(a) shows the electrophoretic deposit weight, measured *in situ* as explained earlier, as a function of the deposition time for different applied voltages. The increase in weight is almost linear with increasing deposition time (up to 4 min) and voltage. An applied voltage of 5 V seems ideal based on the amount of deposited material. However, the

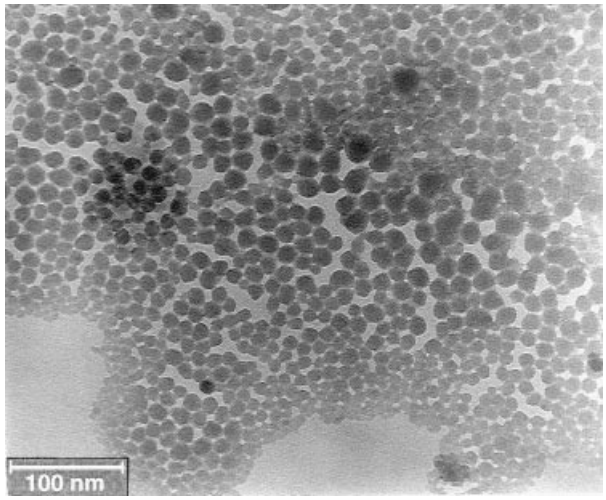


Fig. 3. Bright field TEM micrograph showing the shape and size of the colloidal silica particles and the absence of any agglomeration/flocculation.

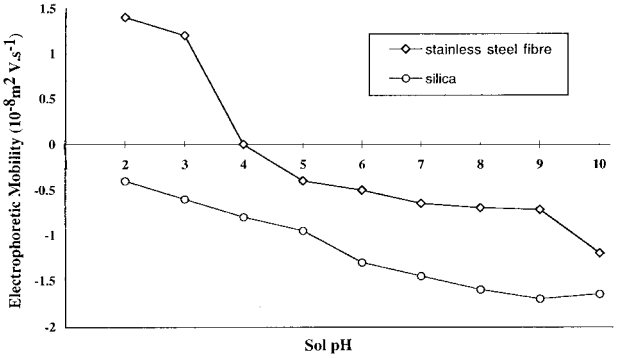
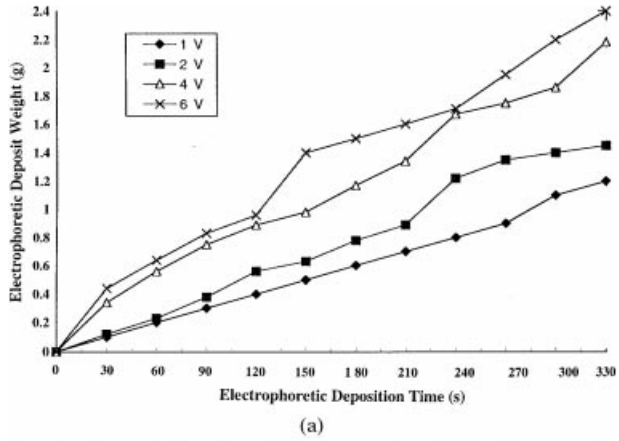
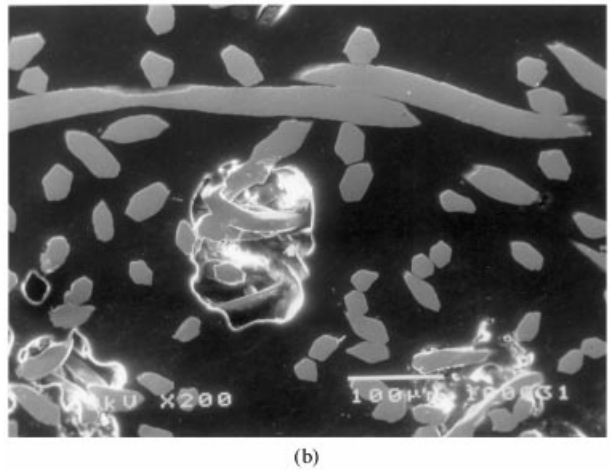


Fig. 4. Particle electrophoretic mobility data for colloidal silica powders and crushed stainless steel fibres in aqueous suspensions as a function of the suspension pH (solids-loading is 0·05 wt%).



(a)



(b)

Fig. 5. Graph showing the variation of the electrophoretic deposit weight measured *in situ* as a function of deposition time for different voltages (a), and high resolution SEM micrograph showing incomplete infiltration with an applied voltage of 2 V (b).

deposited matrix microstructure is very porous as a result of the gases evolved at the electrode surface (due to the electrolytic decomposition of the aqueous medium at these relatively high voltages), becoming trapped in the deposit. The amount of porosity in the deposits was obtained using digital image analysis by taking into account the total pore surface on a certain area (100 mm^2). The results are shown in Table 1. An applied voltage of 4 V was chosen as the optimum voltage to prevent the significant evolution of gases. In previous investigations, voltages of 4 V were also used to deposit ceramic nano-particles onto both metallic[13,14] and SiC-based[2,6–7] fibre preforms using EPD, and a high level of solid infiltration was achieved in agreement with the present results. Under applied voltages of 1 and 2 V, the deposition rate is very slow due to the low particle mobility in the suspension, and the quality of the deposits is poor, as shown in Fig. 5(b). The thickness of selected electrophoretic deposits, measured from the fibre surface, is plotted against deposition time for different voltages in Fig. 6. The formation of the deposit is very rapid in the first 30 s and then increases very steadily, as explained in the following paragraph (see also Fig. 7).

In situ (real time) deposit weight measurements provide very reliable data to determine the accurate deposition rate. Figure 7 shows, for example, the rate of deposit formation as a function of time for a constant optimised applied voltage of 4 V. The deposition rate was normalised for the weight of the woven stainless steel fibre mat tested. The rate of electrophoretic deposition is given in grams of electrophoretic deposit formed per gram of fibre per second. From the graph, it is concluded that the rate of deposition is very high at the beginning (up to 30 s) and then it starts to decrease with increasing deposition time. The decrease in deposition rate is attributed to the increase in the resistance of the deposit, as the current diminishes due to the increase in deposit thickness and the removal of charged silica particles from the sol. As a result of the decrease in the potential drop across the suspension, the velocity and deposition rate of the charged colloidal silica particles decreased also.

Electrophoretic deposition parameters were optimised in order to achieve a fully infiltrated metallic fibre preform with the minimum amount of excess material being present in the outer regions of the preform. In the present study, the optimum deposition voltage and time were determined as 4 V and 2 min, respectively. Using these parameters it was possible to produce fully infiltrated mats with only thin (350–450 μm) excess silica layers. This is beneficial as the mats then have a reduced propensity for the formation of large cracks during the drying stage. This cracking is due to the differential shrinkage of the gel network which generates tensile stresses at the surface of the deposits. These stresses may reach values higher than the mechanical resistance of the gel, especially with thicker sections, leading to the initiation of cracks.[17]

The key issue in incorporating a colloidal matrix into a reinforcing fibre mat using EPD is the tightness of the fibre architecture. As shown in Fig. 8(a) and (b), the woven metallic fibres used in this study have a very tight inter/intra fibre preform structure with inter fibre distances of ca 5 μm. The final green and sintered densities and, hence, the mechanical properties of composites made from these fabrics, will be poor if these regions are not fully infiltrated

Table 1. Porosity in the electrophoretically obtained deposits on metallic fibre fabrics as a function of applied voltage (n.d. = non determined)

Applied voltage (V)	*Porosity (from density measurements) (Vol%)*	*Porosity (from image analysis) (Vol%)*
2	n. d.	n. d.
4	3·7	3·2
6	14·3	13·7

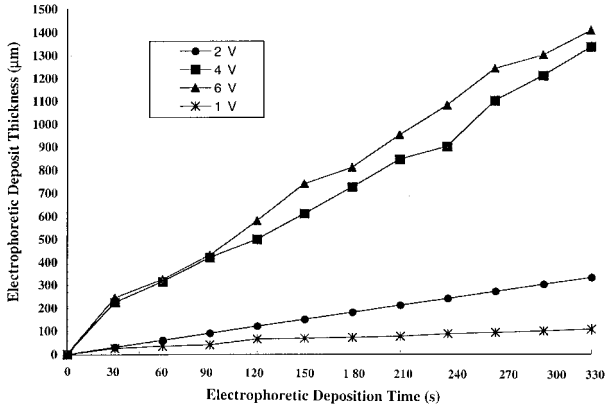


Fig. 6. Variation of the electrophoretic deposit thickness (measured from the fibre surface) as a function of the deposition time for different voltages.

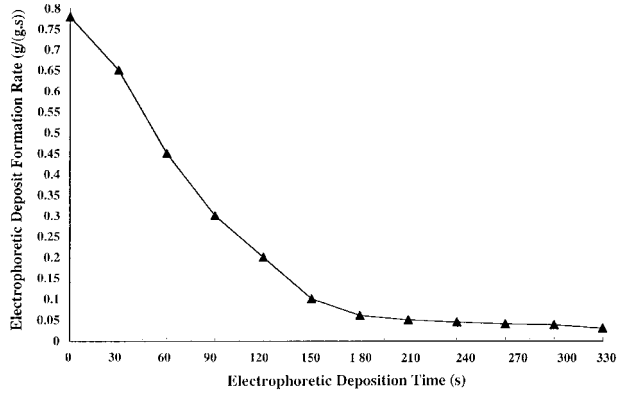


Fig. 7. Variation of the rate of electrophoretic deposition as a function of the deposition time for a constant optimised applied voltage of 4 V.

with matrix material during EPD. SEM-EDX analysis of the metallic fibres is given in Fig. 8(c), confirming the stainless steel chemical composition.

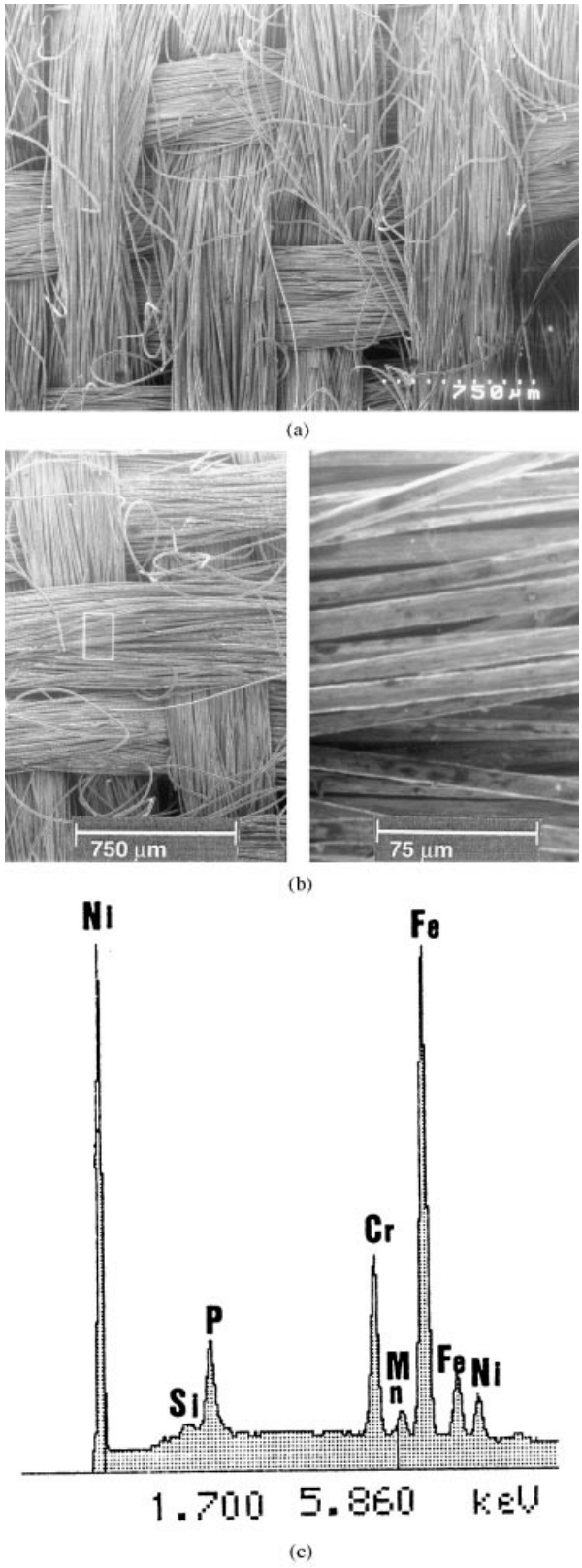


Fig. 8. High resolution scanning electron micrograph (HRSEM) of the woven stainless steel fibre preforms, showing the general fibre architecture (a), the detailed fibre arrangement (b), and the SEM-EDX chemical analysis of the stainless steel fibre (c). Note the very tight intra/inter regions. The average fibre diameter is in the range 15–25 μm and the distance between each individual fibre is in the range 50–150 nm.

Figure 9(a) shows a green microstructure of one of the tightest regions, which was fully deposited/infiltrated with the silica matrix via EPD and subsequent PF. The use of EPD enabled the silica

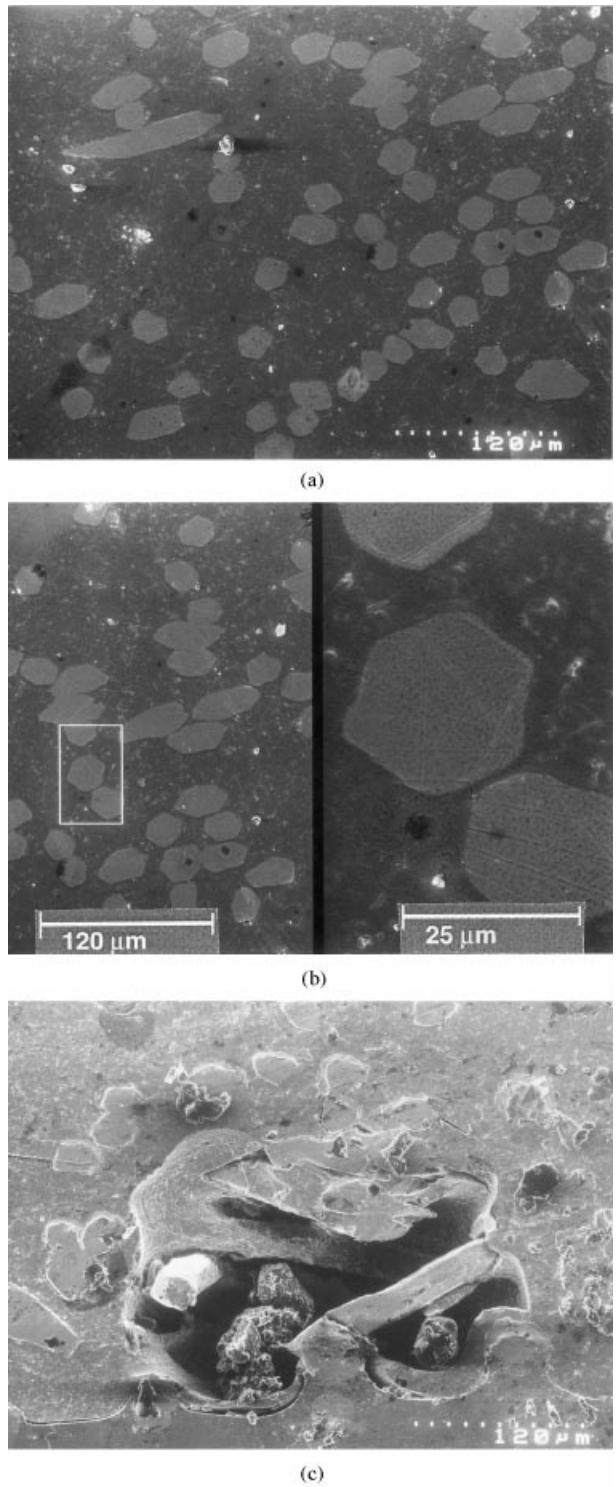


Fig. 9. High resolution SEM micrographs, showing full deposition and infiltration of the silica matrix into the very tight fibre preform (a), the formation of dense electrophoretic deposit between two fibres with a seperation of 250 nm (b), and incomplete deposition, resulting in a very porous microstructure (c). The samples labelled (a) and (b) were obtained by a combination of EPD and PF. The sample in (c) was produced using only PF. Note the infiltrated regions are very dense, however, due to strong repulsive forces between stainless steel fibre and silica, the middle section of the fibre mat can not be deposited via a single pressure filtration technique.

Table 2. Comparison of green and sintered densities of fibre mat reinforced silica matrix composites obtained by different processing techniques (PF: pressure filtration, EPD: electrophoretic deposition)

Processing route	*Green density (%)*	*Sintered density (%)*
PF	42	76
EPD	53	84
EPD + PF	66·5	97·3

matrix to be infiltrated between two closely packed metallic fibres separated only by 100–300 nm, as shown in Fig. 9(b). In comparison, samples produced using only pressure filtration resulted in very low green and sintered densities, as shown in Fig. 9(c) and detailed in Table 2. This confirms that a single pressure filtration step is not enough to infiltrate/deposit the inter/intra tow regions of the fibre mat with matrix material. The present results indicate that both techniques must be used in combination in order to form a dense deposit in the fibre preform, thus reducing the drying shrinkage and removing the requirement of multiple infiltration steps. Using this two-step processing technique results in an excellent deposition/infiltration of the woven metallic fibre fabrics and, hence, a green density as high as 66·5% could be achieved.

In this study, a relatively low matrix sintering temperature (900°C) was necessary in order to prevent the degradation (and loss in mechanical performance) of the metallic fibres during composite consolidation. The modified silica matrix developed could be sintered at the required temperatures without the early cristobalite formation, which could retard the viscous flow assisted densification.[15] The final glass formed contained mostly amorphous and semi-crystalline phases. The absence of major crystallisation reactions below the sintering temperature was confirmed via DTA and TG measurements.

Figure 10(a) shows a backscattered SEM image of a specimen produced using optimised EPD parameters, pressure filtration, and dried in humid atmosphere, followed by sintering at 900°C. It is seen that full and complete infiltration was achieved without the formation of any small internal or external cracks within the sintered matrix. On the other hand, using an applied voltage of 6 V and deposition time of 3 min, resulted in complete infiltration, as desired, but also extensive crack formation within the sintered matrix appeared, as shown in Fig. 10(b). Hence, in order to fabricate woven metallic fibre reinforced silica matrix composites, this simple two-step processing of EPD coupled with pressure filtration can be successfully

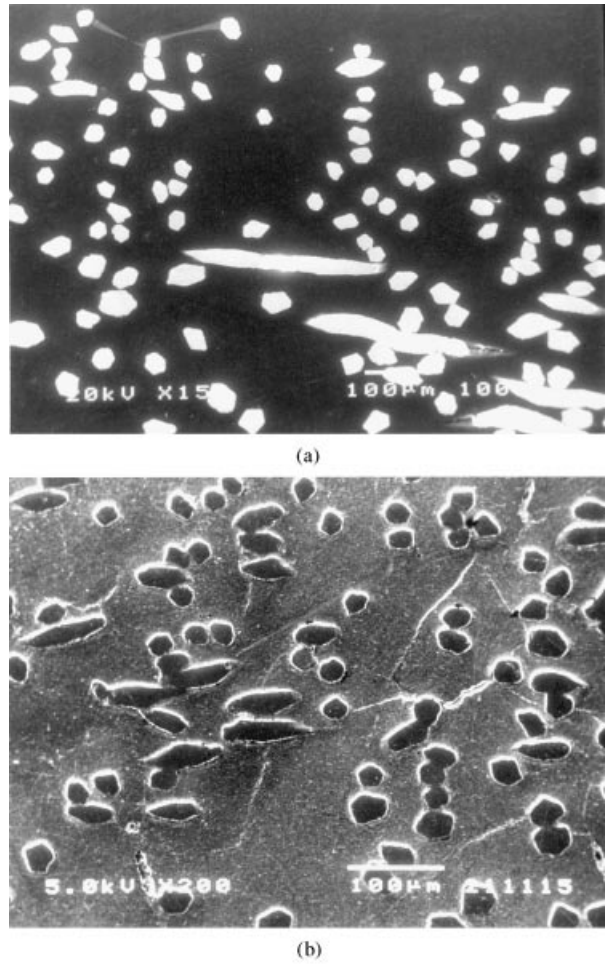


Fig. 10. SEM micrographs (backscattered imaging) of composite samples fabricated by EPD and PF, followed by controlled humidity drying. Absence of cracks under optimised EPD parameters, i.e. 4 V and 2 min (a), and extensive cracking formation due to higher deposition voltage and time, causing a thick excess silica layer (800–900 μm) (b).

employed. The process improves on earlier studies of woven metallic fibre systems[13,14] by an enhanced *in situ* control of the EPD infiltration stage, coupled with the use of pressure filtration to consolidate infiltrated fibre mats rather than the previously employed dry pressing technique.

4 Conclusions

The processing technique developed which combines electrophoretic deposition and pressure filtration, is a powerful technique for the infiltration of multilayer woven stainless steel fibre preforms with nano-size colloidal silica particles. The process involves very short preform fabrication times, supplying compositional homogeneity and the practicality of being simple and cost-effective. The formation of drying cracks was completely eliminated using a two-step-controlled humidity drying process. The high density and homogeneous microstructure of the materials produced here suggest the development of silica matrix composites

with attractive mechanical properties. The simplicity of the processes offers great potential for the industrial application of the technique for the manufacture of high-quality metallic fibre-reinforced ceramic matrix composites.

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