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Electrophoretic deposition in the production of SiC/SiC composites for fusion reactor applications

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Abstract

This paper presents the results of a study aimed at developing a technique for the infiltration of a SiC-fibre fabric with a low-activation SiC-based matrix material by aid of electrophoretic deposition from aqueous suspensions. To achieve the best possible particle packing in the infiltrated matrix and hence to minimise the shrinkage during drying, the effect of the suspension's composition was analysed. Besides the pH change, different types of surfactants – PEI, CTAB, citric acid and Dolapix – were included in the investigation. As a result, during the deposition onto a metallic electrode, the best deposits were obtained with the addition of CTAB or by increasing the pH, while the infiltration of the SiC-fibre fabric was more effective with negatively charged particles in the suspension.

Keywords: Silicon carbide; Aqueous suspensions; Surfactants; Electrophoretic deposition; Electrophoretic infiltration; SiC/SiC composites; Fusion

1. Introduction

Continuous SiC-fibre-reinforced SiC-matrix composites (SiC/SiC) are recognised as promising materials for many demanding applications due to their ability to resist extreme conditions, for example, in heat-engine components in aerospace propulsion and in the structural parts of future fusion reactors.[1,2] The aim of this investigation is to develop a SiC-based composite that will effectively substitute the currently favoured ferrous materials, which are proposed to be used in future fusion reactors mainly due to the high degree of present technological development of relevant alloys. On the contrary, the fusion-relevant SiC-based composites are the least well developed among the candidate materials for a reactor; however, using SiC/SiC composites for the blanket structural component could lead to a significant increase in the maximum operating temperature and, moreover, the material would not decay under neutron irradiation to produce long-lived radioactive waste.[3] There are, however, some critical issues, such as insufficient hermeticity, swelling and various technological obstacles, that limit the potential for SiC-based composites to be seriously considered as suitable materials for the next generation of fusion reactors.[4–6] For this reason, efforts are being made worldwide to solve the demanding issues related to processing of SiC/SiC composites. One key issue is related to the feasibility of achieving full infiltration of the SiC-fibre fabric with a low-activation matrix material.

The infiltration of SiC-fibre woven fabric with a SiC-matrix material has been undertaken by various techniques, notably chemical vapour infiltration (CVI) and polymer infiltration and pyrolysis (PIP). Unfortunately, these are very slow and costly processes and/or result in an incomplete filling of the gaps between the fibres in the tows. In addition to CVI and PIP, which result in incomplete filling and the formation of highly amorphous SiC, further attempts have been based on using ceramic routes. Among them, the recently introduced NITE process, based on a transient eutectic-phase route,[7,8] seems to be the best suited to meet the requirements for a material to be used in a fusion reactor. The infiltration in this process is performed by slip infiltration, followed by pressure-assisted liquid-phase sintering of the SiC matrix using Al_2O_3 and Y_2O_3 as the sintering aids. Most recently, there have been attempts to avoid or at least minimise these additives with the aim to produce a material with the lowest pos-

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sible neutron activation, and to develop dense, high-purity SiC.

According to Nannetti et al.,[9] hybrid techniques offer new possibilities for the production of high-purity gas-impermeable SiC/SiC composites. By filling approximately one third of the inter-bundle voids with SiC powder before PIP, they achieved less than 10% porosity; however, this is still unacceptably high. In our investigation we have looked at the feasibility of electrophoretic deposition (EPD) as a potential technique to fully infiltrate SiC-fibre fabric with SiC powder. The aim of the study was to achieve the highest possible packing density of the powder-infiltrated preform that will be subsequently densified either by PIP or another appropriate technique.

EPD is a commonly used process for producing coatings and self-standing ceramic parts; it is generally recognised as a fast, simple and efficient ceramic processing technique.[10–14] Several successful attempts have been reported on the use of EPD for the production of ceramic fibre-matrix composites.[14–16] It has been shown that, depending on the electrical conductivity of the fibres, the fibre-preform can either be used directly as the deposition electrode or it can be positioned in front of the electrode.

In spite of the clear potential and effectiveness of the EPD process, the mechanisms and kinetics of the formation of the deposit are still not fully understood. Most papers report on results achieved by a trial-and-error approach, which frequently leads to uncertain reproducibility. However, in our study we used the approach of a thorough analysis of the effect of the aqueous suspensions' composition and their electrokinetic behaviour within a wide pH and zeta-potential range. This experimental approach should enable the reproducible deposition of a coating or formation of a matrix material with high green density as well as the infiltration of various fibre fabrics. The use of a water-based system rather than the previously studied ethanol-based systems[17,18] was expected to result in some advantages due to the higher dielectric constant of water and the wider range of suitable surfactants. Moreover, aqueous suspensions are also more attractive because unlike ethanol-based suspensions they are not sensitive to humid environments, which should result in better reproducibility.[19]

The main objective of this investigation was therefore to tailor SiC suspension's properties with positive and negative particle net-surface charges in order to be able to adapt the suspension's properties for the infiltration of a particular type of SiC-fibre fabric. Our work was primarily focused on a comprehensive study of the EPD process; the investigation of the subsequent densification of the SiC matrix produced was not part of this work.

2. Experimental

The substrate material used in this investigation was Tyrano SA SiC-fibre fabric (Ube Industries, Ltd., Japan). For the infiltration we used β-SiC powder BF12 (H. Starck, Germany) with an average grain size of 0.5 μm. Aqueous suspensions containing 25 wt.% of powder were prepared by homogenisation in a multidirectional mixer for 30 min. The zeta-potential (ZP) of the powders was measured in undiluted suspensions using a ZetaProbe analyser (Colloidal Dynamics, USA), while a ZetaPals instrument (Brookhaven, USA) was employed to analyse the ZP of the fibres. Surface charge was modified with hydrochloric acid, citric acid (CA, Johnson Matthey GmbH, Germany), tetramethyl-ammoniumhydroxide (TMAH), polyethylene-imine (PEI 10.000, Alfa Aesar, Germany), cetyltrimethylammonium bromide (CTAB, Sigma–Aldrich) or Dolapix CE64 (Zschimmer & Schwarz, Germany).

The EPD experiments were performed with suspensions containing 25 wt.% of solids at a constant dc voltage of 60 V for 5 min using steel or copper electrodes. The electrodes (20 mm × 20 mm × 0.5 mm) were positioned vertically in the EPD cell at a distance of 2 cm. In the experiments, the SiC-fibre woven fabric was used as the deposition electrode or placed in front of a steel electrode. The porosity of the deposits due to bubble formation was minimised by placing a cellulose membrane in front of the steel cathode or by using copper as the anode. Before deposition the SiC-fabrics were rinsed with acetone and distilled water or pre-treated with a sodium dioctyl-sulfosuccinate (SDOSS) solution.[20] The progress of the deposition was monitored by the change in the current. The bulk deposits were evaluated in terms of the final weight and the solids content, while the degree of particle packing was assessed with a scanning electron microscope. The microstructures of the infiltrated samples were observed in the green state using optical and electron microscopy.

3. Results and discussion

3.1. Characteristics of the suspensions

The properties of the aqueous SiC suspensions for electrophoretic deposition were analysed in undiluted form, i.e., containing 25 wt.% solids. As shown in Fig. 1, the natural pH (pH ~ 4) of the SiC suspension nearly matches the IEP, which explains why the suspension is very unstable. Titration with HCl resulted in a significant increase in the conductivity and only slight increase in the ZP up to a maximum value of 11 mV at pH 2.8, where the stability of the suspension was still

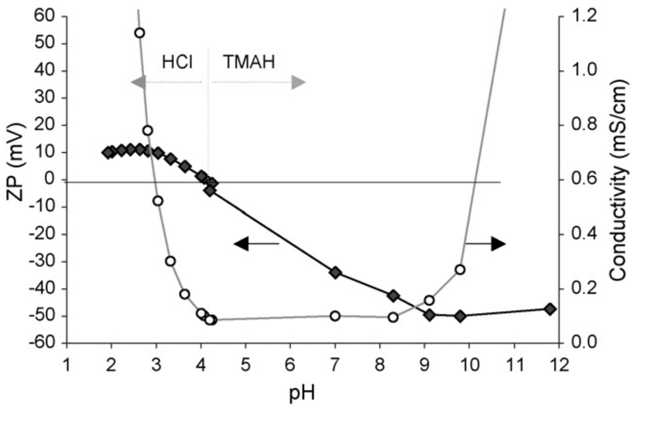

Fig. 1. The influence of pH change on the zeta-potential (ZP) and the conductivity of an aqueous suspension of SiC powder (solids content: 25 wt.%; pH adjusted by HCl and TMAH).

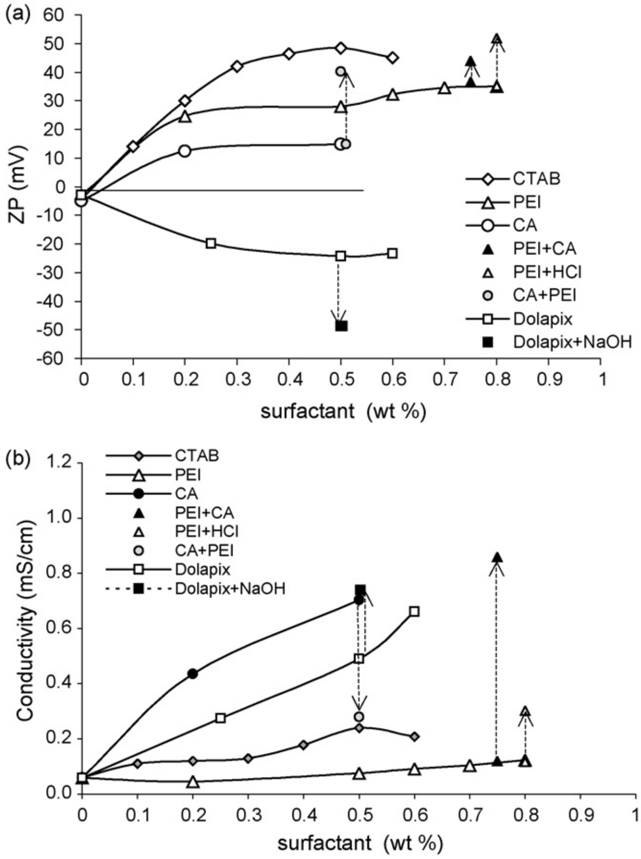

Fig. 2. The ZP (a) and conductivity change (b) due to the addition of different surfactants to SiC suspensions (solids content: 25 wt.%). The arrows show the effect of an additional pH change for a particular surfactant addition.

very low. This implies that a pH decrease will probably not be sufficient for EPD and hence, to increase the positive net-surface charge, the addition of a cationic surfactant is required. In contrast, the pH increase with TMAH or NaOH resulted in a much more pronounced effect on the ZP, giving a value of −50 mV at pH 9. However, a further increase in the pH only increased the conductivity, whereas the ZP began to decrease slightly.

Fig. 2a and b illustrates the effect of the different surfactants on the ZP and the conductivity of the SiC suspension. It is clear that the addition of the cationic surfactant CTAB increased the ZP up to 48 mV without a significant change in the conductivity, while the addition of PEI resulted in a smaller increase in the ZP, reaching a maximum value of 35 mV with 0.8% PEI. As reported by Zhang et al.,[21,22] due to the low dissociation of PEI in the alkaline region, PEI does not stabilise the alkaline suspension, while any further addition of citric acid should lead to, according to their reports, a significant increase in the ZP.[21,22] In our measurements the ZP reached its highest value (44 mV) only after the subsequent addition of 2 wt.% of citric acid (at pH 2.8), whereas the conductivity was already very high. A better result was obtained when the pH of the PEI-containing suspension was adjusted with HCl: at a pH of 8 the ZP reached 52 mV (the effect of the pH changes are indicated with the dashed arrows in Fig. 2a and b).

Table 1
Compositions and properties of the characteristic suspensions used in the EPD experiments and corresponding properties of wet deposits

No.	Composition		Suspension		Deposit	
	Additive	wt.%	pH	Conductivity	ZP	wt.% solids
1	0		4	0.08	−3	/
2	HCl		2.8	0.78	14	/
3	CTAB	0.4	3.9	0.24	48	67
4	PEI	0.8	8	0.12	35	/
5	(PEI) + CA	0.75 + 2	2.8	0.86	44	54
6	(PEI) + HCl	0.8	8	0.3	52	64
7	CA	0.5	2.6	0.7	15	/
8	(CA) + PEI	0.5 + 0.3	3.3	0.3	40	59
9	NaOH		9	0.15	−54	66
10	TMAH		9	0.15	−50	67
11	DCE64	0.5	6.3	0.5	−24	/
12	(DCE64) + NaOH	0.5	9	0.74	−49	62

Fig. 2a also shows that the addition of citric acid to the SiC suspension has a minor effect on the ZP, but it significantly increases its conductivity. However, the conductivity decreased considerably with further additions of PEI, while the ZP increased from 15 to 40 mV. This observation agrees with previous reports[22,23] which showed that the surfactant-addition sequence has a large effect on the stability of the suspensions.

The addition of the anionic deflocculant Dolapix CE64 increased the absolute value of the ZP up to −24 mV, which was further increased to −49 mV by adjusting the pH to 9; however, the subsequent conductivity increase was relatively high (see Fig. 1a and b).

The above results are summarised in Table 1 and indicate that high, positive ZP values can be obtained with the addition of 0.4% CTAB, 0.8% of PEI at pH 8 or through combined addition of 0.5% CA and 0.3% PEI, while high, negative values can be achieved by a pH increase to 9 or with the addition of Dolapix CE64 at pH 9. However, some of these compositions show a strong increase in the conductivity too. Based on these results, in a further step, we performed EPD experiments using a selected composition with characteristic ZP values.

3.2. EPD experiments

The EPD experiments were performed by using suspensions with 25 wt.% of solids at selected characteristic points in the ZP vs. pH diagrams. In order to evaluate the quality of the deposits, steel electrodes were used in the first trials. The suspensions' compositions and properties, related to the final result of the EPD experiments, are listed in Table 1.

First, an EPD experiment was performed using a suspension without any additive. As expected, due to the absence of charged particles in the SiC suspension at its natural pH of 4.1, no deposit was observed after 5 min at an applied voltage up to 60 V. When the pH of the suspension was adjusted to 2.8, where the ZP was 11 mV, a very loose deposit was formed on the cathode, but it slid from the electrode during removal from the suspension.

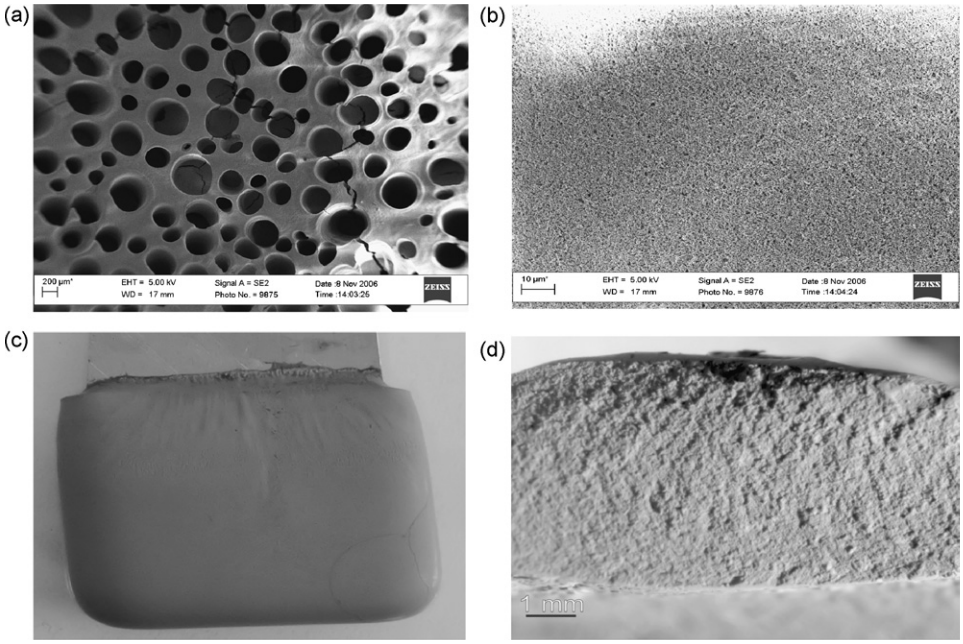

Fig. 3. SEM micrographs of deposits formed from a CTAB-stabilised suspension on a steel electrode (a and b), macroscopic view of the deposit formed on the copper electrode (c and d).

As expected, a firm deposit formed on the cathode from the suspension with the addition of 0.5 wt.% CTAB, characterised by a high ZP. As illustrated in Fig. 3a, the deposit contained large channel-like pores due to water electrolysis, while the particles in the bulk were densely and homogeneously packed (Fig. 3b). In further EPD experiments the presence of bubbles in the deposits was prevented by placing a porous membrane in front of the deposition electrode, which led to a firm and non-porous deposit (Fig. 3c and d). By measuring the weight change during the drying of the deposit, it was determined that the solids content in the deposit made by EPD increased to 67 wt.% comparing to the initial 25% solids in the starting suspension.

The EPD experiments using suspensions with the addition of PEI were less successful; no deposit was retained on the electrode after its removal from the suspension. In spite of the ZP increase, the further addition of 2 wt.% of citric acid did not significantly improve the deposition: the deposit was loose and weak and, accordingly, the weight change during drying revealed low solids content in the fresh deposit (54 wt.%). A firm and dense deposit (64 wt.%) was obtained from the suspension containing the 0.8% PEI and the pH adjusted to 8 by HCl. No deposit was obtained from the suspension containing citric acid, which contrasts with the successful deposition in ethanol reported previously.[17,18]

In further experiments the deposit was formed on the anode using negatively charged powder suspensions. In this case the presence of bubbles was prevented by using a Cu electrode, which consumes the oxygen formed during the electrolysis. The alkaline suspension with the addition of TMAH resulted in a firm and dense deposit, containing 67 wt.% solids. A similar result was obtained if the pH was adjusted to 9 by adding NaOH. In contrast, no deposit was formed from the suspension with Dolapix until the pH was adjusted to 9, where the Dolapix is well dissociated and helps to produce dense and firm deposits.

The above results suggest that for electrophoretic deposition the most appropriate suspensions for cathodic deposition were those containing CTAB, while for deposition on an anode, the addition of Dolapix and an increase in the pH seem to give the best results, since they lead to a high particle-packing density in the deposits.

3.3. Infiltration of SiC-fibre woven fabrics

In the next set of experiments we explored the electrophoretic infiltration of SiC-fibre woven fabrics by SiC particles. Based on the above results we selected the two compositions with the highest ZP: the suspension of positively charged particles with a 0.5% CTAB addition (48 mV) and the suspension with negatively charged particles and Dolapix addition (−49 mV). For better understanding of the infiltration, the ZP of the SiC fibres was also analysed, as shown in Fig. 4. The as-received fibres used

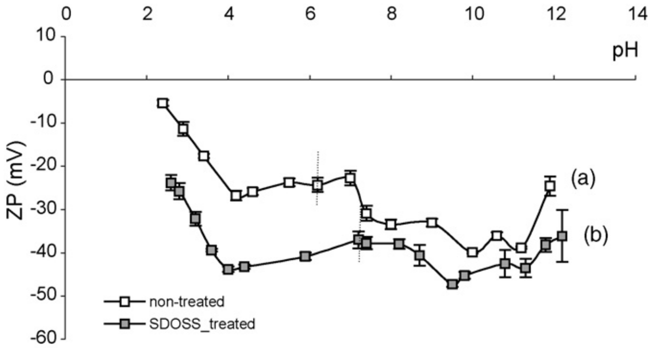

Fig. 4. Zeta-potential of non-treated (a) and (b) SDOSS-treated SiC fibres as a function of pH (the fibres were cut and crushed for the analysis).

in this study were seen to be negatively charged (Fig. 4, open square symbols) exhibiting similar behaviour to the SiC powder (see Fig. 1). The fibres were then treated with the anionic surfactant SDOSS in order to improve the wetting with aqueous slurries, which also resulted in a slight increase in the negative ZP value (Fig. 4, close square symbols).

The SiC-fibre fabric was first used as a deposition electrode, but no deposit was formed within 5 min. The current during the deposition was very low, probably due to the conductivity of the fibres being too low. Then, one or several layers of the fabric were placed in front of the steel electrode, so that the particles were forced to travel through the fabric towards the electrode. In order to prevent the formation of bubbles in the deposit a membrane was placed in front of the fabric at the cathode or, in the case of negatively charged particles, a copper electrode was used as the anode. The infiltrated green samples were dried slowly and examined with an optical microscope. Further they were vacuum infiltrated with a resin and examined by SEM to estimate the success of the infiltration.

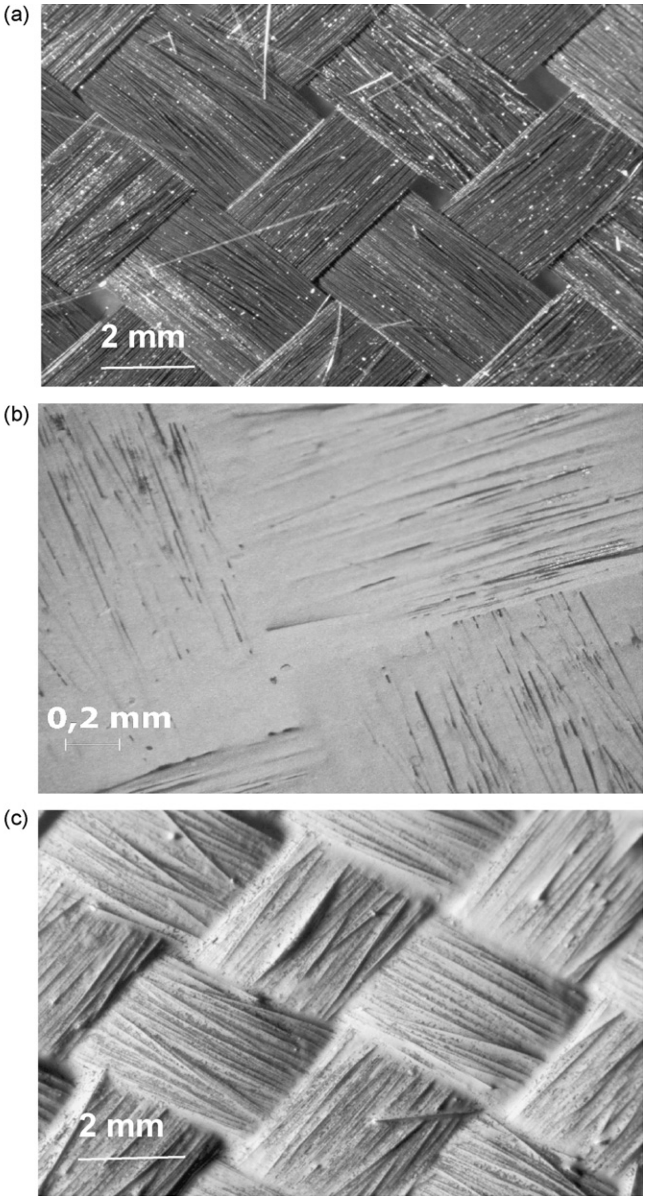

Fig. 5. Optical micrographs of the starting fibre fabric (a), the fabric infiltrated with a TMAH-dispersed SiC suspension (b) and the fabric infiltrated with the CTAB-dispersed suspension (c).

Fig. 5a illustrates the as-received SiC-fibre fabric before infiltration, while Fig. 5b and c show the surfaces of the fabrics electrophoretically infiltrated with negatively or positively charged SiC particles, respectively. In the first case the fibres were pre-treated with SDOSS. In this case most of the large voids between the fibre tows were filled and the particles homogeneously coated the surface of the fabric (Fig. 5b), while the positively charged suspension seems to flocculate on contact with the negatively charged fibres. SEM observations (Fig. 6a and b) suggest that in the case of positively charged particles, they attach themselves to the fibres rather than entering the fibre tows interspaces. This is in agreement with a previously proposed mechanism for particle infiltration of fibrous substrates by EPD, which indicates that a more efficient infiltration can be achieved by using equal-sign charges of the fibres and particles rather than using opposite charges.[24]

A low-angle cross-section of a green part prepared with a negatively charged suspension, infiltrated with a polymer and carefully polished, was also observed by means of SEM, as shown in Fig. 7, taking special care to distinguish the SiC-infiltrated parts (the light phase in Fig. 7a) from the polymer-infiltrated parts (dark areas in Fig. 7a, see the arrows). As illustrated in Fig. 7b and c, which show the light areas, SiC particles have effectively filled the narrow gaps between the

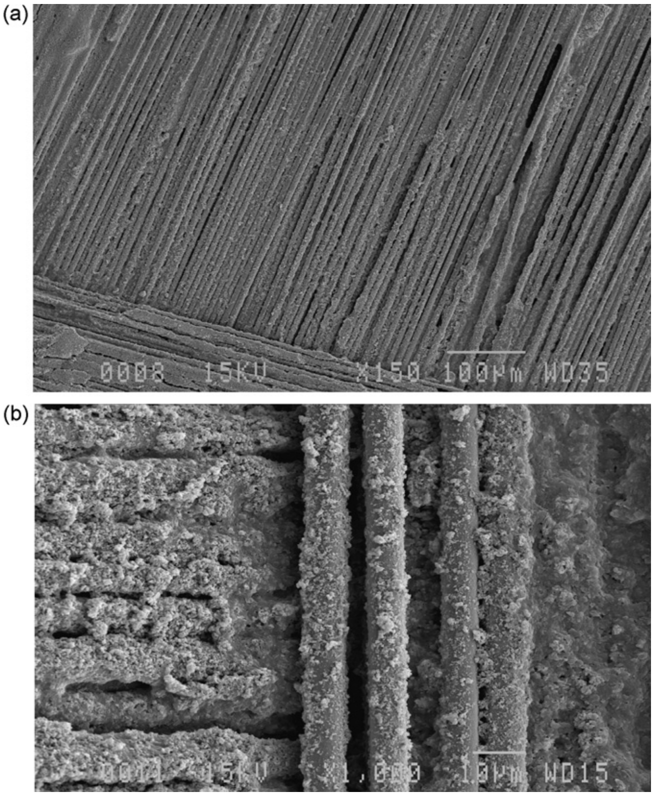

Fig. 6. SEM micrographs of the surface of the fabric infiltrated with an alkaline SiC suspension (a and b: different magnifications).

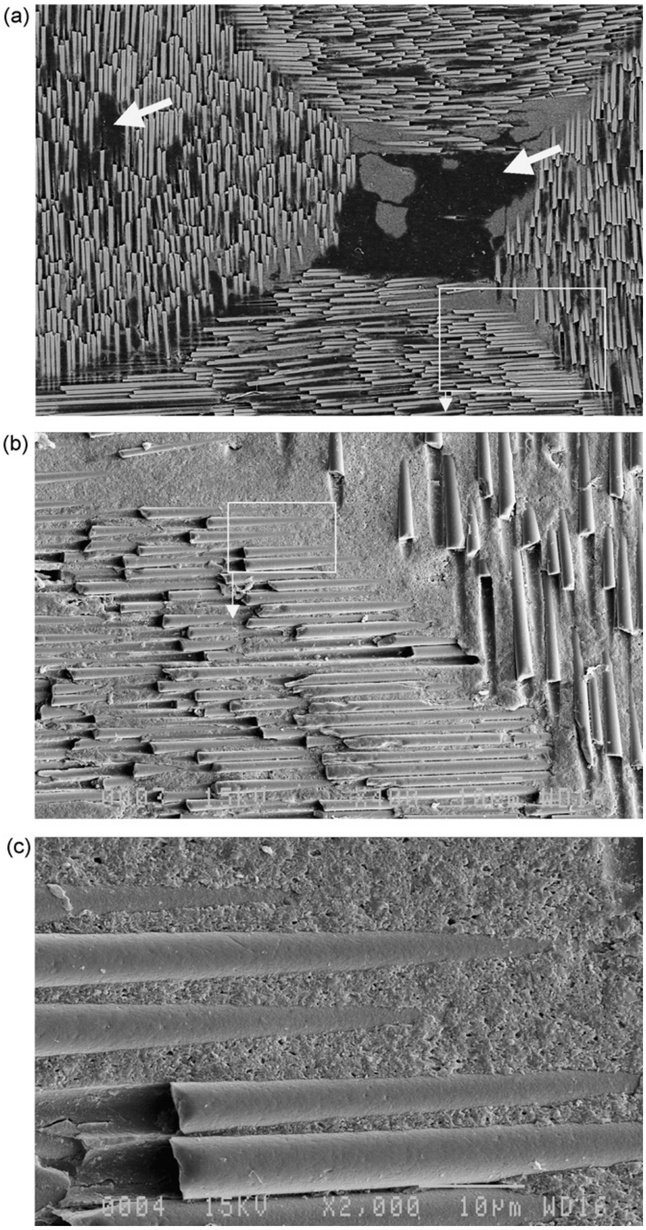

Fig. 7. SEM micrographs of a polished low-angle cross-section of the fabric infiltrated with an alkaline SiC suspension (a–c: different magnifications).

individual fibres, demonstrating the success of the developed EPD technique.

4. Conclusions

This study has investigated the influence of surface charges on the electrophoretic deposition of SiC powder in aqueous suspensions and has demonstrated the usefulness of the electrophoretic infiltration of SiC-fibre fabrics for production of SiC/SiC composites.

Based on the characteristics of the suspensions and of the bulk deposits formed on metallic electrodes, we confirmed that a high zeta-potential is of outmost importance for the EPD process, while the suspension conductivity plays only a secondary role. For the powder used in this investigation the best results, i.e., a high zeta-potential leading to firm, dense deposits, were obtained for the addition of 0.5 wt.% of cationic deflocculant CTAB or by adjusting the pH of the suspension to 9 and adding Dolapix. The formation of bubbles due to electrolysis was prevented by using a membrane, in the case of cathodic deposition, and a Cu electrode, in the case of the anodic deposition.

The infiltration of an SDOSS-pre-treated Tyrano SA SiC-fibre fabric with a negatively charged SiC suspension resulted in dense fibre-based green parts, while the infiltration with a suspension of positively charged particles was less effective.

Powder infiltration prior to the PIP process may considerably shorten the densification time of SiC/SiC composite production by avoiding the several required steps of polymer immersion. Electrophoretic infiltration may also significantly improve the density of SiC/SiC composites, in particular by filling the large voids between the fibre tows. This should enable the fabrication of SiC/SiC composites with a close-to-zero porosity, with any remaining pores being closed pores, thus ensuring a hermetic material.

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