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Densification of a SiC-matrix by electrophoretic deposition and polymer infiltration and pyrolysis process

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Abstract

With the aim to improve the properties of the SiC-matrix in a SiC_f/SiC composite for fusion applications, a new fabrication technique combining electrophoretic deposition (EPD) and polymer infiltration and pyrolysis (PIP) was introduced. By using EPD from a well-dispersed, aqueous suspension we were able to produce SiC green bodies with closely packed particles (>62%TD). This deposition was followed by vacuum infiltration of the green body with pre-ceramic polymer precursor and then pyrolysis and crystallization at 1600 °C. Due to the high initial packing density of the deposits, only a few polymer-infiltration and pyrolysis steps were needed to achieve a relatively high density of the material. The SiC samples fabricated by the combined EPD-PIP process reached the matrix density of ∼86.5% TD and average pore size of ∼90 nm after six consecutive PIP cycles, while high thermal conductivity values (>30 W/mK) were obtained already after one PIP cycle.

Keywords: Precursors-organic; Porosity; Thermal conductivity; SiC; Fusion; Structural application

1. Introduction

The intrinsic properties of silicon carbide (SiC) make it a promising material for high-temperature structural applications. Furthermore, its low neutron activation, when combined with its high mechanical and thermal stability, makes SiC-based composites good candidate materials for structural applications in future fusion reactors. To ensure a sufficient fracture toughness and reliability of the material, SiC can be used in the form of a continuous SiC-fibre-reinforced SiC composite (SiC_f/SiC).[1,2]

The currently developed techniques for the production of SiC_f/SiC composites involve filling the voids in a SiC-fibre fabric preform by chemical vapour infiltration (CVI) or pre-ceramic polymer infiltration and pyrolysis (PIP), both of which are very slow and costly processes and result in an incomplete filling of the gaps between the fibres in the tows.[3,4] However, ceramic routes have been suggested as a possible way of producing a relatively dense SiC_f/SiC composite.[5,6] Unfortunately, the densification of the SiC-matrix for a fusion-relevant material is a very challenging task, because the densification of SiC with the aid of an alumina–yttria transient eutectoid or boron and carbon requires high sintering temperatures, i.e., >1800 °C, which might lead to degradation of the SiC fibres.[7,8] In addition, the sintering aids increase the activation of the material,[9] which negates one of the main advantages of SiC_f/SiC composites in comparison to steels. Therefore, attempts have focused on the development of so-called hybrid routes, which combine different fabrication techniques in order to achieve a dense material without jeopardizing the advantageous properties of the pure SiC.

As one of the main drawbacks of the above-mentioned PIP process in the production of SiC_f/SiC is the large amount of residual porosity and the presence of relatively large voids between the fibres (even after several cycles), a possible method of improvement is offered by filling the voids in the fibre preform with SiC powder before the polymer infiltration. However, one paper in the literature describing attempts to use vacuum or high-pressure-assisted infiltration[10] did not report a successful outcome. In another variant of the PIP process, the green parts produced by the slip casting or pressing of powder and polymer mixtures were impregnated with polymer to fabricate a SiC_p/SiC-PIP matrix.[11] This combined process was presented as a promising technique for fabricating a dense SiC-matrix; however, the impregnation with the polymer was retarded after

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a certain number of PIP cycles due to the formation of a dense layer near the surface of the sample.

As an alternative technique for filling the voids in the fibre preform with SiC powder we proposed electric-field-assisted infiltration. Namely, electrophoretic deposition (EPD) has been confirmed in numerous investigations to be a simple, fast and low-cost technique for the production of complex-shaped ceramics,[12,13] and it was also shown that by careful selection of the suspensions' properties and processing parameters, densely packed SiC green bodies can be prepared.[6] Moreover, two- and three-dimensional SiC-fibre preforms were efficiently impregnated by SiC particles using the electrophoretic deposition process.[14] A densely packed matrix within the fibre preform was proposed to enable a more effective further densification of the composite.

In this work the effectiveness of a combination of electrophoretic deposition (EPD) and polymer infiltration and pyrolysis (PIP) for the production of a SiC-matrix was investigated. The main focus was on the preparation of a dense SiC-matrix, although in this stage the fibres were not included in the composite. The resulting materials were characterised for microstructure, porosity and thermal conductivity and compared with the properties of related fusion-relevant materials.

2. Experimental

2.1. Materials and sample preparation

Aqueous suspensions with a solids content of up to 60 wt.% were prepared from β-SiC BF12 powder (H. Starck, Germany) with an average particle size of 0.5 μm. Tetramethylammonium hydroxide (TMAH, Sigma–Aldrich, Germany) was used as a dispersant. The suspensions were homogenised for 5 min using an ultrasonic finger processor with an acoustic power density of 109 W/m^2 (Hielscher Ultrasonics, Germany). The electrokinetic behaviour of the suspension was analysed with a ZetaProbe device (Colloidal Dynamics, USA). The suspension at pH ∼10 was chosen for the deposition, where the ζ-potential and the conductivity of the suspension reached their optimal values.[15]

The electrophoretic deposition experiments were performed in a Teflon cell with graphite electrodes (4 cm × 4 cm) separated at a distance of 2 cm using a power source (0–64 V, Thrulby Thandar Instruments, United Kingdom) with an applied voltage of 30 V for 10 min. A cellulose membrane was placed in front of the anode to prevent the porosity caused by bubble formation at the electrode due to the electrolysis of water. The suspension was also used for samples fabricated by slip casting in plaster. For comparison, additional samples were prepared to assess the effect of particle packing on the final sample density by uniaxial pressing the same SiC powder with an applied pressure of 20 MPa and by isostatic pressing of EPD-prepared deposits with an applied pressure of 740 MPa.

The SiC samples formed by EPD or dry pressing were vacuum infiltrated using allylhydrido polycarbosilane (AHPCS) with the commercial name SMP-10 (Starfire Systems Inc., USA). Prior to infiltration, the polymer was agitated with a magnetic stirrer in order to degas the polymer and heated to 60–80 °C to decrease its viscosity from ∼0.1 Pa s at room temperature to 0.03–0.02 Pa s (Fig. 1). The samples were evacuated to 1000–500 Pa and then infiltrated with the polymer using a pressure difference between the chambers with the polymer and the sample. The infiltrated samples were pyrolysed at 850 °C in argon and crystallized at 1600 °C in vacuum according to the procedures suggested by the manufacturer[16] in a high-temperature Astro furnace (Thermal Technologies Inc., USA).

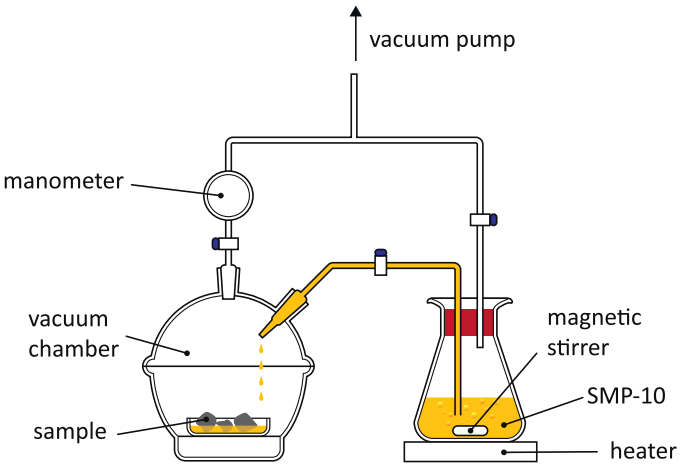

Fig. 1. Schematic representation of the polymer infiltration system.

For comparison, two additional samples were prepared by liquid-phase sintering (LPS) and by solid-state sintering of the green parts prepared by EPD. The samples for LPS were vacuum infiltrated with an $Al(H_2PO_4)_3$ aqueous solution, dried in air and sintered in Ar at 1400 °C, in accordance with the procedure described in Ref. 6. A set of samples is sintered without additives at 2000 °C in vacuum. A list of the samples is given in Table 1.

2.2. Sample characterization

The microstructures of the polished cross-sections of the samples were examined using an optical microscope (Zeiss Discovery V8), a scanning electron microscopes (JEOL JSM-

Table 1
Sample list (SC: slip-casting; UP: uni-axial pressing; IP: isostatic pressing; EPD: electrophoretic deposition; SS: solid-state sintered; SITE-A: slip-infiltration and transient eutectoid; PIP: polymer infiltration and pyrolysis).

ID	Shaping technique	Densification
SC	Slip casting	One PIP cycle[a]
UP	Uniaxial pressing	One PIP cycle
IP	Isostatic pressing	One PIP cycle
EPD + IP	EPD + isostatic pressing	One PIP cycle
EP1	EPD	One PIP cycle
EP3	EPD	Three PIP cycles
EP6	EPD	Six PIP cycles
SS	EPD	Sintering without additives (2000 °C)
SITE-A	EPD	Liquid phase sintering (1300 °C)

[a] PIP cycle refers to samples that were cured, pyrolysed and crystallized at 1600 °C unless stated otherwise.

Fig. 2. As-deposited SiC green part.

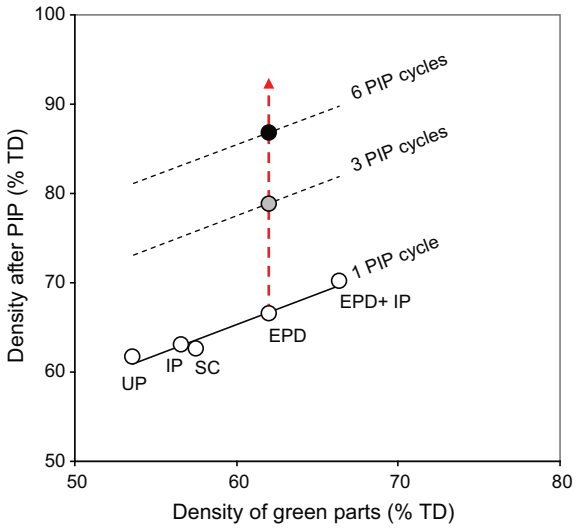

Fig. 3. Green densities and densities after PIP processing.

5800, FE-SEM Zeiss SUPRA 35VP) and a transmission electron microscope (JEOL JEM-2100).

The composition and the crystallinity of the samples were evaluated by X-ray diffraction (XRD, D4 Endeavor, Bruker AXS) using Cu Kα radiation in the range from 10 to 70° 2θ.

The bulk density of the samples prepared by EPD was measured in water by the Archimedes method using lacquer (Ilirija, Slovenia) encapsulation, while the densities of the pressed samples were calculated from their mass and volume. The porosity of the samples was measured with mercury-intrusion porosimetry (Micrometrics-AutoPore IV 9500) in the pressure range from 1.6 kPa to 414 MPa.

The thermal transport properties were measured on a Quantum Design Physical Property Measurement System (PPMS) with a 9 T magnet using the standard four-probe lead configuration. Measurements were preformed in the temperature range between 300 and 400 K. The samples were in the form of small bars with the maximum size 4 mm × 4 mm × 15 mm.

3. Results and discussion

3.1. Shape forming

Electrophoretic deposition was chosen as the shape-forming technique because of its well-known ability to form bulk deposits with high packing densities and, secondly, because it is possible to use a related process, i.e., electrophoretic infiltration (EPI), for the infiltration of the 3D ceramic fabric preform to produce a SiC_f/SiC composite.[14,15]

As we already demonstrated in our previous work,[14] the properties of the SiC green parts formed by EPD strongly depend on the properties of the starting suspensions. Accordingly, we prepared well-dispersed aqueous suspensions with a 60 wt.% solids loading. The ζ-potential of the suspension was −60 mV (±3 mV) and the conductivity was 0.4 mS/cm (±0.1 mS/cm). By using a voltage of 30 V a 5-mm-thick deposit (45 mm × 45 mm) was obtained after 15 min (Fig. 2).

Fig. 3 illustrates the dependency of the density of SiC samples after PIP on density of the green parts. It is also evident that the density of the green parts formed by EPD is significantly higher (62% T.D.) than that formed by slip-casting (SC, 57.5% T.D.) or uniaxially or isostatic pressing (UP, 53.6% T.D. and IP, 56,5% T.D.), and this density can be further increased by subsequent isostatic pressing (IP), to reach 66.4% T.D. It has to be stressed, however, that the latter technique (EPD + IP) for forming the SiC-matrix was only used for a comparison, since due to a possible degradation of the fibres, it cannot be used for the fabrication of a SiC_f/SiC composite. Hence, the results indicate that EPD has a great advantage over other shaping processes because it is capable of producing high packing densities in addition to its ability to infiltrate the specimen.

The porosity measurements (Fig. 4) of the SiC green parts prepared by EPD revealed that the pores are as small as 60 nm, representing quite a challenge for infiltration with a polymer. However, as we will show below, due to the excellent wetting ability of the SMP-10 polymer precursor, the infiltration of the green deposits was not problematic.

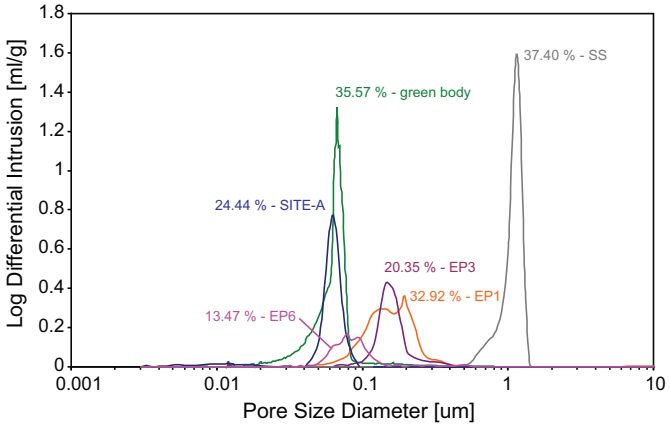

Fig. 4. Pore size distribution of green body, SITE-A, SS and different EP samples.

3.2. Polymer infiltration and pyrolysis

The polymer infiltration and pyrolysis (PIP) process involves several stages. The pyrolysis can be done in a single step (direct pyrolysis) or in a two-step process (two-step pyrolysis), involving the curing of the pre-ceramic polymer precursor and the pyrolysis.[16–18] To obtain a crystalline material the amorphous residue after pyrolysis has to be given a further thermal treatment ("crystallization step"). Ly et al.[17] claimed that curing the pure SMP-1 as a separate process before pyrolysis increases the yield of the polymer-to-ceramic transformation. The importance of the curing process for the production of the crystalline material was also confirmed in our experiments, where the pure SMP-10 polymer was pyrolysed in a single or a two-step pyrolysis and crystallized at 1600 °C. From the XRD spectra, presented in Fig. 5, we can see that the SMP-10 polymer pyrolysed in a single step (direct pyrolysis) remained amorphous even after the heat treatment at 1600 °C, whereas the one pyrolysed in a two-step process resulted in crystalline SiC after the heat treatment at 1600 °C. This confirmed that curing is a necessary step to achieve crystalline SiC at 1600 °C.

The SMP-10 polymer pyrolysed in a single step and pyrolysed in a two-step process were also examined with the transmission electron microscope. The sample pyrolysed in a single step remained mainly amorphous with small (1–2 nm) nanocrystalline regions of SiC (Fig. 6a and b), whereas the sample pyrolysed in a two-step process exhibited a more pronounced crystallinity with larger regions of predominantly cubic β-SiC (>5 nm) (Fig. 6c and d).

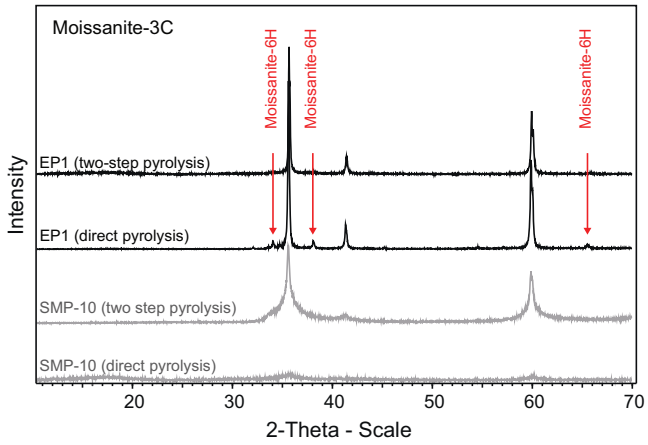

Fig. 5. XRD spectra of crystallized pure SMP-10 with or without curing (gray) and XRD spectra of EPD samples densified with one PIP cycle with or without curing prior to pyrolysis (black).

As an alternative, SiC powder incorporated into the SMP-10 may serve as a major crystalline phase in the SiC-matrix. The samples produced by EPD and densified with one PIP cycle (EP1), with or without curing, both exhibit a crystalline XRD

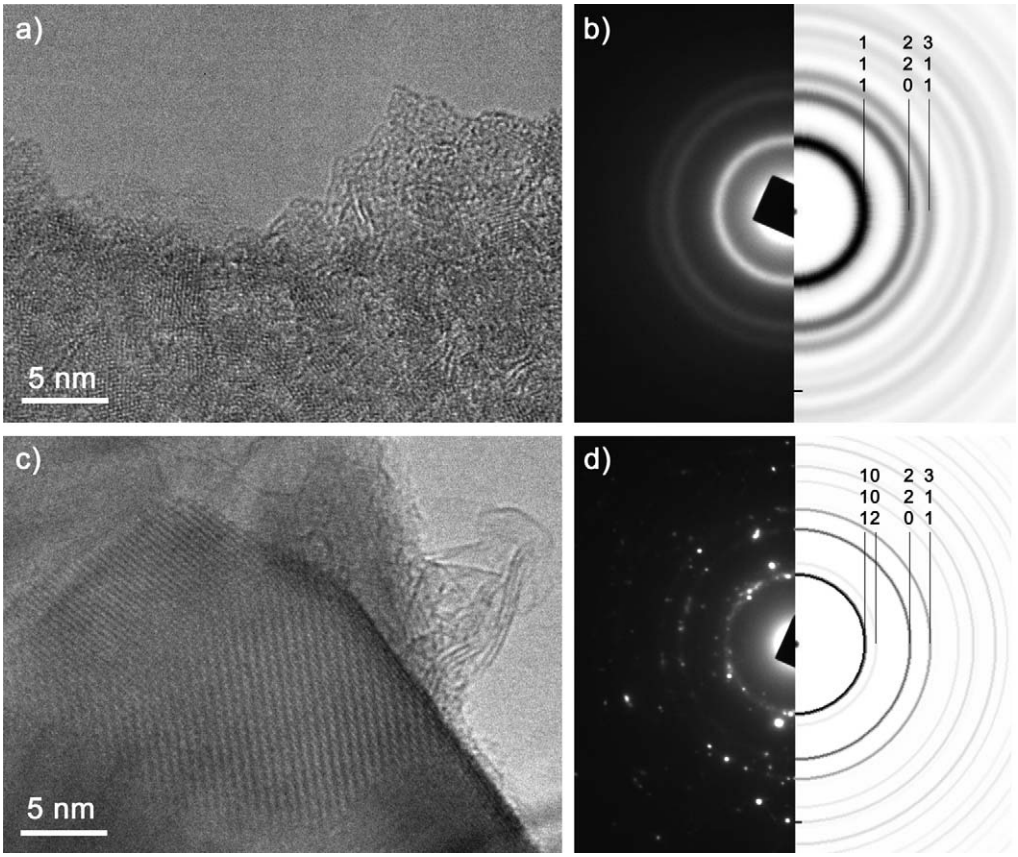

Fig. 6. Transmission electron micrograph of a crystallized SMP-10 without curing (a) with experimental diffraction pattern of the sample and calculated diffraction pattern for nanocrystalline (2 nm) cubic SiC (b), and crystallized SMP-10 with curing (c) with experimental diffraction pattern of the sample and calculated diffraction pattern for cubic SiC (d).

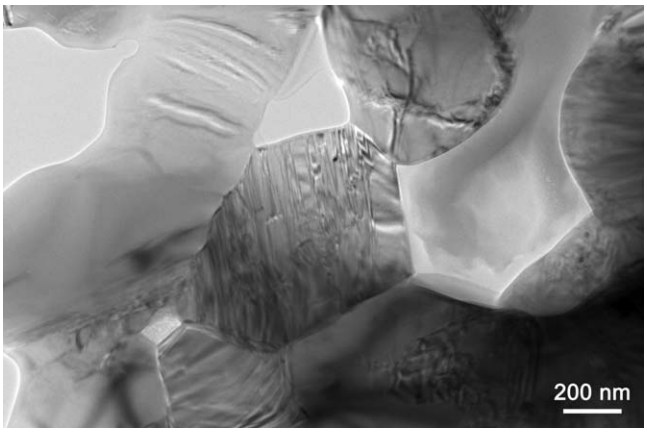

Fig. 7. Transmission electron micrograph of a crystallized EP1.

spectrum (Fig. 5) due to the large amount of crystalline β-SiC from the used powder.[19] The EP1 sample without curing also contains small amounts of α-SiC (Moissanite 6H), probably present as an intermediate phase of the β-SiC crystallization. The transmission electron micrograph of the sample after first PIP cycle, EP1 (Fig. 7), indicates that the original SiC particles with an irregular shape grew epitaxially on account of the SMP-10 to form a microstructure of connected grains and well-defined grain boundaries. The specific contrast within the grains confirms the presence of the different SiC polytypes.

3.3. Density and microstructure

The densities of the samples after PIP are illustrated in Fig. 3 as a function of the green densities achieved by different shaping techniques and of the number of PIP cycles. As expected, a linear relationship can be observed between the green and the final density after PIP. It is also obvious that the density of the samples formed by EPD and subsequently infiltrated with the pre-ceramic polymer, pyrolysed and crystallized (PIP) increases progressively with the number of PIP cycles, achieving 86.5%TD after six PIP cycles. This is a significant improvement in comparison with the literature data,[11] which reports the need for twelve PIP cycles using high-pressure impregnation to achieve the same level of density. Hence, our results clearly indicate that for full densification after PIP, a higher green density and a few additional PIP cycles would be needed.

The gradual density increase with the number of PIP cycles was also confirmed by mercury intrusion porosimetry. As is clear from Fig. 4, after one PIP cycle the porosity increased from an initial 35.57% (with an average pore size 60 nm) to 32.92% (with an average pore size of ∼180 nm). After each further PIP cycle the porosity was reduced, achieving 13.47% after six PIP cycles, with an average pore size of 90 nm. The increase in porosity in the first cycle suggests that a rearrangement of the particles took place during the thermal treatment in the PIP process. For comparison, the porosity of the samples formed by EPD and solid-state sintered (SS) or liquid-phase sintered according to the procedure described in Ref. 6 (SITE-A) are also presented. It is evident that a thermal treatment of SiC without sintering aids at 2000 °C results in the highest porosity and pores with an average pore size of ∼1 μm. The porosity of the low-temperature sintered material approximately equals that of the porosity after six PIP cycles, while the pore size remains unchanged.

The significant reduction in porosity during PIP processing is also illustrated in Fig 8a–d, which show optical and scanning electron micrographs of the polished cross-sections of the EP (EPD + PIP) samples after one and six PIP cycles, respectively. Fig. 8a and b indicates that the green samples formed by EPD were fully impregnated with the pre-ceramic polymer throughout the thickness, while the residual porosity was homogeneously distributed throughout the volume of the samples. This represents a significant improvement over the recent literature report.[11] Here, the authors used the same type of polymer to impregnate slip-cast and dry-pressed green parts, but they observed highly porous regions in the central parts of the samples and dense regions near the surface, which was suggested as the main processing issue for further polymer infiltration.

In addition, the comparison of the appearance of the fracture surfaces of the green EPD-formed part (Fig. 9b) and the body after the first PIP cycle confirms that the pre-ceramic polymer deposits onto the SiC particles, resulting in SiC grain growth to form well-faceted SiC grains (Fig. 9d). The transmission electron micrographs of the EP1 sample (Fig. 9c) in comparison to the SiC-BF12 powder (Fig. 9a) also indicate a minor increase in grain size, which appears to be the result of the epitaxial crystallization of the polymer precursor on the surface of the SiC particles.

3.4. Thermal conductivity

A sufficiently high thermal conductivity, i.e., >25 W/mK at RT,[20] is one of the main target properties of the structural material to be used in a future fusion reactor, since the heat has to be effectively transmitted through the wall. Literature values for thermal conductivity of bulk SiC material at RT vary significantly, depending on the fabrication process and consequently on the grain size, nature of grain boundaries, impurities and porosity. Yamada et al.[21] reported on thermal conductivity of ∼25 W/mK for SiC matrix in SiC/SiC composites prepared by polymer infiltration and pyrolysis (PIP) process. SiC matrix prepared by chemical vapour deposition (CVD) method exhibits higher thermal conductivity values from 60 to 350 W/mK depending on the grain size,[22] however the related process of chemical vapour infiltration (CVI) used for infiltration of SiC fabric preform, is unable to produce a sufficiently dense SiC_f/SiC composite. SiC matrix densified by liquid phase sintering (LPS) exhibits thermal conductivity values between ∼30 and ∼100 W/mK, depending on the amount of secondary phase,[23] which reduces the low-activation property of the material and is therefore not desired in fusion relevant materials. Literature values for bulk SiC produced by pressureless sintering with the addition of boron and carbon are in the range between 100 and 180 W/mK,[24–26] however the temperatures used in this process (>2000 °C) are detrimental to the properties of SiC fibres and thus cannot be used for fabrication of SiC_f/SiC composite.

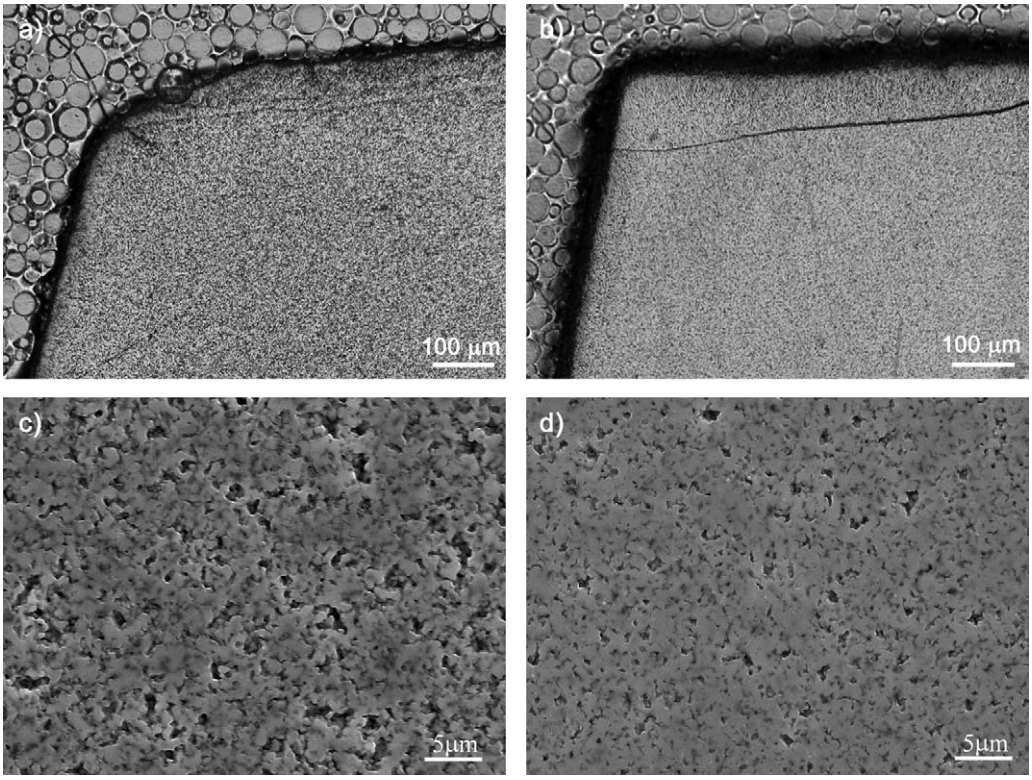

Fig. 8. Optical and scanning electron micrographs of the polished cross-section of a sample after one PIP cycle (a), (c) and after six PIP cycles (b), (d).

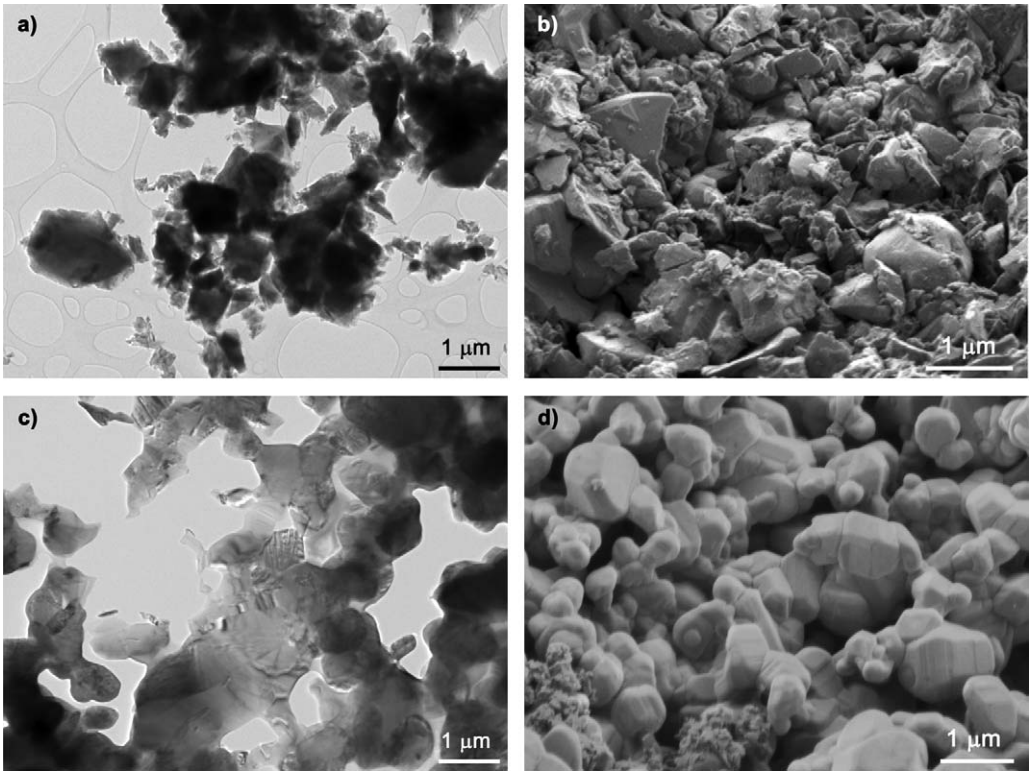

Fig. 9. Transmission electron micrograph of BF12 powder (a) and scanning electron micrograph of a fracture surface of green deposit (b) and transmission and scanning electron micrographs of EP1 sample fracture surface after crystallization (c), (d), respectively.

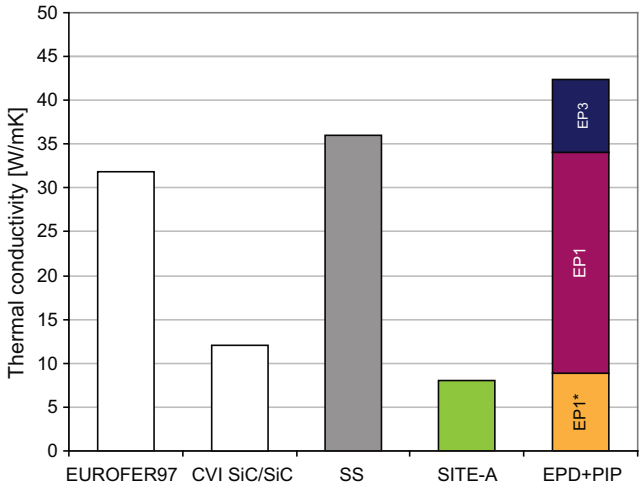

Fig. 10. Thermal conductivity measurements. Reported thermal conductivity values for EUROFER97 and for through thickness thermal conductivity of CVI SiC/SiC is 30 W/mK [28] and 12 W/mK[27] respectively. Sample EP1* refers to sample that was only pyrolysed at 850 °C and not crystallized.

In order to reliably characterise the prepared materials, samples of fusion-relevant materials, i.e., the Eurofer97 steel and the CVI SiC_f/SiC composite (Cerasep N3-1) were measured and compared to literature data.[27,28] The measuring technique gave a good agreement with the reported values.

The results presented in Fig. 10 indicate that the crystallinity of the SiC ceramics is the governing parameter for thermal conductivity. The measured values for the sample sintered at 2000 °C (without sintering aids) was relatively high (36 W/mK), especially when compared to the sample densified according to SITE-A process[6] (<10 W/mK), where the SiC ceramic contains a certain amount of amorphous phase. For the samples densified by PIP the thermal conductivity increases from 8 W/mK, for the pyrolysed-only sample (without crystallization), to 34 W/mK after one PIP cycle. Further densification with consecutive PIP cycles further increased the thermal conductivity, achieving 42 W/mK after three PIP cycles. The observed increase in the thermal conductivity of the samples with the increased number of PIP cycles can be ascribed to the decreased porosity, which strongly supports the need for further optimization of the densification process.

Measured values for EP samples (fabricated by combined process of EPD and PIP) are relatively high in comparison to thermal conductivities of SiC matrix prepared by conventional PIP process.[21] Higher values can be ascribed to higher crystallinity of samples due to large amount of initial crystalline powder in the material. Lee et al.[11] reported on similar thermal conductivity values (50 W/mK) for SiC_p/SiC-PIP matrix, where the green parts produced by slip casting or pressing of powder and polymer mixtures samples densified after twelve PIP cycles with high pressure infiltration of polymer.

The values presented in Fig. 10 are, however, the measured thermal conductivities for the SiC-matrix without incorporated fibres. Nevertheless, based on the achieved >40 W/mK for the matrix and the values reported for the crystalline SiC fibres, i.e., ~60 W/mK,[21] the conductivity of the fibre-reinforced SiC matrix is expected to be above the required value of 25 W/mK at RT.

4. Conclusions

The combination of electrophoretic deposition and polymer infiltration and pyrolysis has proven to be an effective way to produce SiC-matrix material with limited porosity and sufficient thermal conductivity. Using electrophoretic deposition, a relatively high initial packing density of the green samples was achieved and therefore only a few PIP cycles were needed to densify the material and to achieve a high crystallinity. The porosity level after six PIP cycles was ~13.5% and the average pore size was only ~90 nm. Thermal conductivity values above 30 W/mK were achieved after just one PIP cycle, and these values were further increased to 42 W/mK after three PIP cycles. Although the porosity of the samples influences the thermal conductivity to some extent, the crystallinity of the samples was shown to be the governing parameter. This hybrid process should be further optimized and applied to the infiltration of the fabric preform for the production of dense SiC_f/SiC composites.

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