MICG Midlands Industrial Ceramics Group

Midlands Advanced Ceramics for Industry 4.0

CASE STUDY

REF No: 2506001

WS7 - Scale up

COMPARISON OF IMMERSION AND INFUSION TECHNIQUES FOR CERAMIC POWDER INFILTRATION INTO FIBRE PREFORMS

MODELLING & DESIGN | RAW MATERIALS PROCESSING | **FORMING** | DRYING & SINTERING | ANALYTICAL

THE CHALLENGE

Long fibre reinforced ceramic matrix composites (CMCs) often require the infiltration of ceramic powders into the fibre preform prior to densification, either for sintering or as reinforcing agents. One common method involves immersing the fibre preform in a ceramic slurry under negative pressure (i.e., vacuum), which promotes the infiltration of ceramic particles. However, air bubbles trapped within the fibre preform can result in pores that may become closed porosity after densification. This air entrapment poses a particular challenge when producing large CMC components, as it is often difficult to eliminate all air bubbles.

Infusion is an alternative to immersion that involves infiltrating a liquid into an evacuated fibre preform. The resin infusion technique is widely used to produce defect-free, large components of carbon fibre reinforced polymer matrix composites for the automative, submarine and aerospace industries. The infusion technique can also be applied to CMCs as a pre-processing step, enabling the infiltration of ceramic slurries into fibre preforms in an air-free environment. However, efficient infiltration depends on several factors such as suspension viscosity (and thus its solid loading), the porosity of the substrate and its pore size distribution.

In this study, we compared the infusion and immersion techniques by infiltrating silicon nitride (Si_3N_4) and silicon carbide (SiC) particles into 3 mm thick carbon preform substrates. We evaluated the infiltration efficiency of both methods based on the weight gain of the substrates and penetration depth of the particles.

Initially, we produced 2.5D porous carbon fibre substrates using a manual wet-lay-up technique. 2D carbon fibres were immersed in a preceramic polymer, and 10 layers of wetted fibres were stacked with 0° orientation. The resulting preform was pyrolyzed at 700 °C, forming an amorphous ceramic network that bonded the fibre layers. The preform was then cut into smaller substrates with the dimensions of 20x20x3 mm, as displayed in Figure 1.

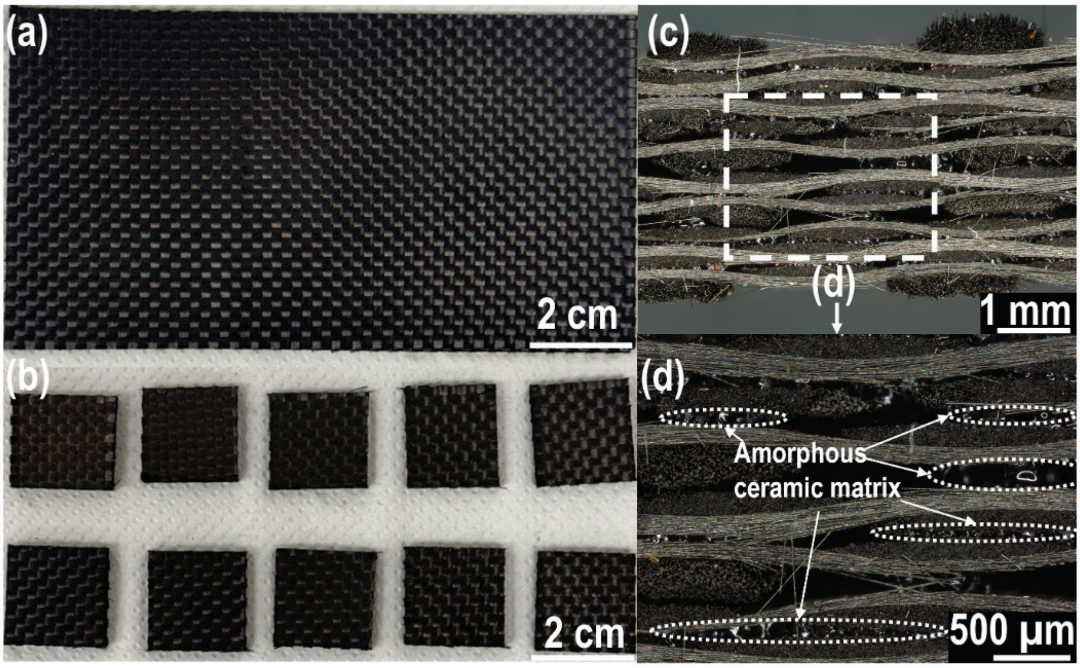

Figure 1
(a) As-pyrolyzed preform and (b) 2x2 cm cut substrates for the infusion trials. (c) Optical micrograph of the substrate cross-section. (d) Magnified view of the cross-section showing the amorphous ceramic matrix bonding the fibre layers.

Water-based Si_3N_4 and SiC suspensions were infused using the following procedure: (i) the substrates were evacuated for 30 minutes to remove air; (ii) the suspensions were then introduced into the chamber, and (iii) the substrates were maintained under a vacuum pressure of 0.9 bar for anadditional 30 minutes. The infiltrated substrates were dried at 150 °C for 30 minutes, and the entire process was repeated for 3 cycles. The weight gain of the substrates was recorded after each cycle, and their cross-sections were examined to assess the depth of infiltration. For the immersion process, the non-evacuated substrates were directly placed into the suspensions and the same procedure was repeated.

The weight gain of the substrates after infiltration with Si_3N_4 slurries containing 40, 50, 60 and 70 wt.% solid loadings is shown in Figure 2. The infused substrates exhibited weight gains of 32.4, 28.4, 53.1 and 50.55 wt.% for the respective solid loadings, while corresponding weight gain values for the immersed substrates are found as 33.95, 35.95, 55.27 and 50.1 wt.%.

Although a few percentages of weight gain difference are observed between immersion and infusion techniques, the variations are not statistically significant. It is worth noting that residual slurry accumulated on the substrate surfaces was manually cleaned after each infiltration cycle, which may introduce a source of experimental error.

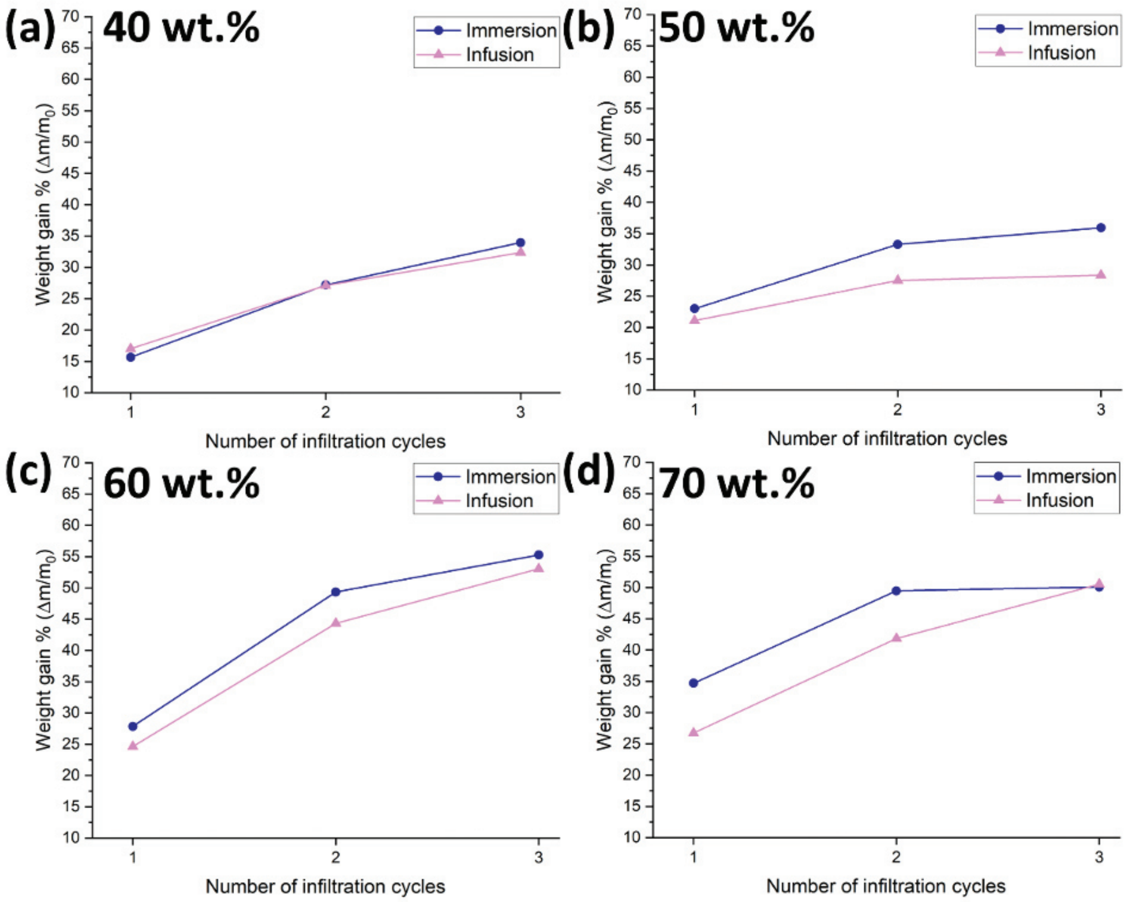

Figure 2
Weight gain (Δm) as a percentage of the initial substrate weight (m 0) for Si_3N_4 slurries with solid loadings of (a) 40, (b) 50, (c) 60 and (d) 70 wt.%, infiltrated using infusion and immersion techniques.

Figure 3 shows the polished cross-sections of carbon fibre preforms infused (upper image) and immersed (lower image) by Si_3N_4 slurry after 3 infusion cycles. The interbundle regions are fully infiltrated by Si_3N_4 particles, and infiltration appears consistent throughout the thickness of the fibre preform, although some porosity remains. Notably, infiltrated Si_3N_4 particles were observed within microcracks (2-3 µm wide) around the amorphous ceramic matrix. However, infiltration into the fibre-rich regions was limited, likely due to the electrical shielding effect of fibres. This phenomenon may arise from electrostatic repulsion between the fibres and particles in the polar solvent (i.e., water), which hinders particle mobility and penetration in densely packed fibre areas.

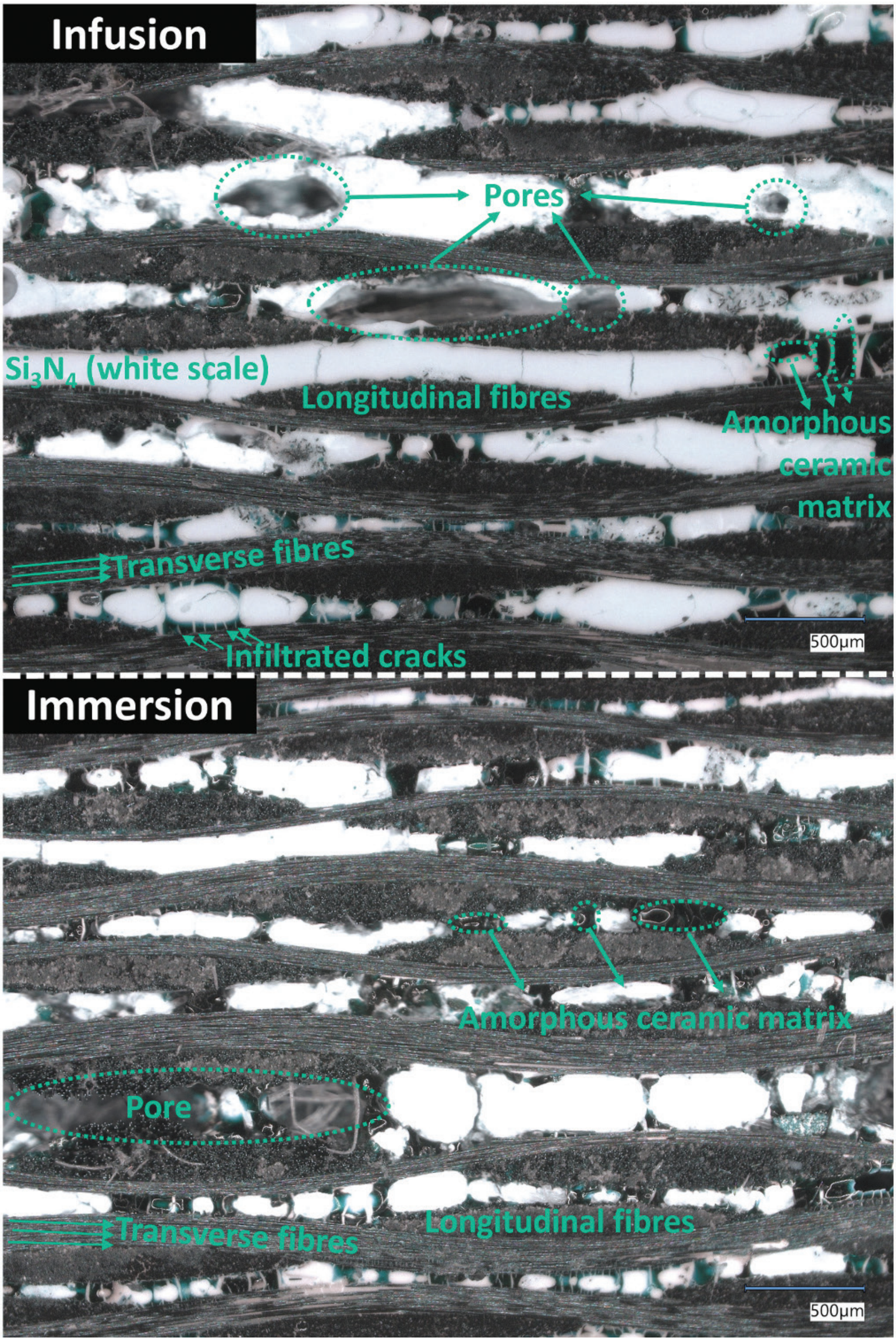

Figure 3

Cross-sectional micrographs of carbon fibre preforms infiltrated with Si_3N_4 slurry after 3 infusion cycles using (a) the infusion technique and (b) the immersion technique.

IMPACT

This project demonstrates the feasibility of both immersion and infusion techniques as pre- processing methods for CMC production. No significant differences in infiltration efficiency were observed between the two techniques when applied to small substrates with dimensions of 20x20x3 mm. Immersion is a well-established technique and widely adopted across various industries. On the other hand, the infusion technique has been specifically designed to provide an air-free infiltration method that can support reliable CMC fabrication, particularly at larger scales. The technique is also compatible with the vacuum bagging processes for producing green CMCs, enabling both infiltration and curing of preceramic polymers, which can subsequently be densified via polymer impregnation and pyrolysis.

While this project demonstrated the capability of both infusion and immersion techniques to infiltrate the interbundle regions of the fibre preform, it also highlighted the challenges in infiltrating fibre-rich regions, particularly when using woven preforms. Further development is needed to improve the infiltration of water-based ceramic slurries into the areas of high fibre density, which is essential for achieving optimum material properties.

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