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Improving strength and microstructure of SiC reticulated porous ceramic through in-situ generation of SiC whiskers within hollow voids

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ABSTRACT

SiC reticulated porous ceramic with excellent strength and high-density ceramic struts was successfully prepared using the polymer replica method, followed by pressureless sintering under a buried charcoal atmosphere. First, a polyurethane (PU) template was coated with a Si slurry and then a SiC-containing slurry, and subsequently heated under the buried charcoal atmosphere. To ensure excellent coating ability of the slurries, the viscosity, thixotropy, and yield stresses of the Si slurry were optimized by adjusting the content of the thickening agent. During heating, Si in the coating layer reacted with the residual C and CO gas from the PU template and buried charcoal, forming SiC whiskers that filled hollow voids within the SiC struts. Additionally, catalyst ferric nitrate was added to the Si slurry to promote the generation and growth of SiC whiskers. As a result, when compared to the untreated SiC reticulated porous ceramic, the SiC reticulated porous ceramic pre-coated with Si layers exhibited significant improvements in mechanical strength and thermal shock resistance, despite minor differences in porosity. Furthermore, an industrial test conducted in the copper smelting industry showed that the structure of SiC reticulated porous ceramic, prepared in this study and used as filters, remained intact even after 7 days of continuous use. Meanwhile, a significant number of inclusions was adhered to the surfaces of the filters. Therefore, the processes combined with in-situ generation of SiC whiskers is an ideal and low-cost method for fabricating SiC filters with excellent properties.

1. Introduction

Fabrication of high-purity metal casting products plays a crucial role in ensuring the advancement of high-performance manufacturing industries. SiC reticulated porous ceramic has been applied as filters in the metal smelting industry to reduce gas and inclusions in castings. This is due to its high mechanical strength, chemical stability, and resistance to thermal and liquid metal flow shocks, even in high-temperature environments [1,2]. Currently, the most popular method for preparing reticulated porous ceramics is the polymer replica method, which is favored for its ease of operation, low cost, and versatility [3]. However, the polymer template completely burns out during the heating process, resulting in concave pore channels with triangular cross-sections inside the ceramic struts of the reticulated porous ceramic (Fig. 1). This leads to stress concentration at the corners of the struts, thereby degrading their mechanical strength and high-temperature stability [4]. To address this issue, various techniques such as recoating [5] and vacuum infiltration [6] have been developed. Liang et al. enhanced the mechanical strength of SiC reticulated porous ceramic by using the vacuum infiltration method with an aluminosilicate slurry [7]. Chen et al. successfully optimized the microstructure of the voids within the ceramic struts and improved the mechanical strength and thermal shock resistance of reticulated porous ceramics using the recoating method [8]. Furthermore, slurries with different viscosity have been recoated on pre-coated polymer templates to improve the structure of the ceramic struts and the strength of reticulated porous ceramics [9]. In terms of industrial production, compared to the vacuum infiltration technique, the precoating process shows great promise in enhancing the strength and properties of reticulated porous ceramics due to its advantages of simplicity and low cost. However, it is challenging to completely fill the hollow voids

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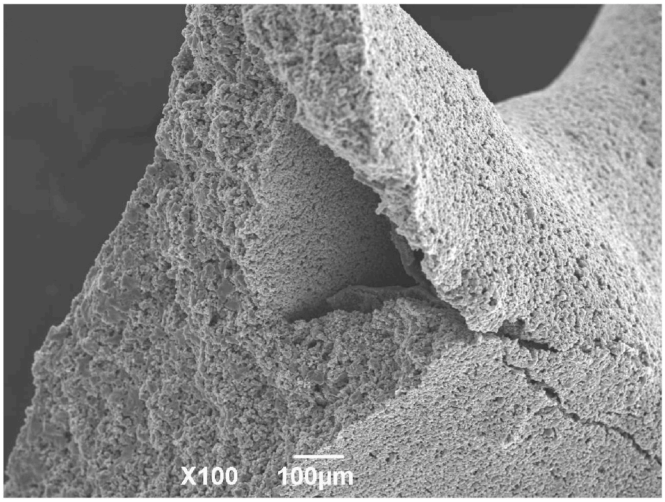

Fig. 1. The morphology of struts in reticulated porous ceramics.

within the ceramic struts through the precoating process, which ultimately leads to inadequate mechanical properties of the reticulated porous ceramics.

In-situ generation of whiskers is an ideal way to improve the properties of porous ceramics, which has received extensive attention from researchers [10]. Lao et al. used a recoating process combined with in-situ generation of mullite whiskers and liquid phase to repair the hollow voids within struts and improve the strength of reticulated porous ceramics [11]. However, one issue is that it is difficult for the in-situ generated mullite whiskers and liquid phases to completely fill the voids. Additionally, the liquid phase affects the mechanical strength of the reticulated porous ceramics at high temperatures. Zhou et al. improved the density and strength of mullite foamed ceramics by in-situ generating SiC whiskers through the reaction of metal Si with carbon [12]. Furthermore, they successfully fabricated bauxite-carbon composite materials with high-density and excellent mechanical strength by formation of SiC whiskers in the matrix [13]. To the best of the authors' knowledge, no studies have been conducted on the complete filling of hollow voids and the improvement of properties in reticulated porous ceramics through a combination of recoating processes and in-situ generation of SiC whiskers. Furthermore, very few researchers have focused on the utilization of residual carbon in polymer templates. This work was designed to address these knowledge gaps. Metal Si precoating slurries were prepared, and their rheology (viscosity, thixotropy, and yield stress) was optimized by adjusting the amount of thickening agent. Furthermore, the effects of the precoating process and sintering temperature on the mechanical properties and microstructure of SiC reticulated porous ceramic were investigated. Finally, the service performance and impact on the quality of copper rods was studied by utilizing SiC reticulated porous ceramic as filters in the copper smelting industry.

2. Materials and methods

Commercial metal Si powder (D50 = 5.0 μm, purity ≥99 wt%, Sinosteel Luoyang Institute of Refractories Research Co., Ltd), SiC powder (D50 = 3.5 μm, purity ≥98 wt%), Al_2O_3 powder (D50 = 2.5 μm, purity ≥99 wt%, Wuxi Chenguang Refractories Co., Ltd), and clay powder (D50 = 5 μm, Wuxi Chenguang Refractories Co., Ltd) were utilized as raw materials. Ferric nitrate ($Fe(NO_3)_3$), polycarboxylate (FS20) and carboxymethyl cellulose (CMC) were used as catalyst, dispersant and thickening agent, respectively. The formulation ratios of the powder mixture for the precoating and recoating slurries are presented in Table 1. Precoating aqueous (water) slurries consisting of 60 wt% metal Si with a thickening agent content ranging from 0 to 0.6 wt%

Table 1
The formulation ratios of the powder mixtures (wt.%).

	Metal Si	SiC	Al_2O_3	Clay	FS20	CMC	Fe $(NO_3)_3$
Precoating slurry	100	/	/	/	/	0~0.6	1
Recoating slurry	5	75	10	10	0.2	0.2	/

were prepared by ball milling for 1 h at 200 r/min. SiC-containing recoating aqueous (water) slurry with a solid content of 75 wt% was prepared through ball milling for 1 h at 300 r/min.

Commercial polymeric sponges were immersed in Si precoating slurries and passed through rotating rollers. After that, the sponges were coated with SiC-containing recoating slurry and any excess slurries were removed using a blower. The resulting coated sponges were then dried at 80 °C for 24 h and subsequently heated at different temperatures (1400 °C, 1450 °C, 1500 °C, and 1550 °C) for 3 h, with a heating rate of 1 °C/min, in carbon (charcoal) embedded condition.

2.1. Characterization

The rheological properties of the Si slurries were measured using a rheometer (Anton Paar MCR 102). The thixotropy of the slurries was assessed using the hysteresis area [14]. The porosity and density of the SiC reticulated porous ceramic samples were determined by utilizing a modified Archimedes (vacuum) method with DI water as the suspension medium. Compressive strength of the SiC reticulated porous ceramic was tested using a universal testing machine (Wance TSE254C). Thermal shock tests were performed via water quenching method [15]. The reticulated porous ceramic samples were heated to 1100 °C and rapidly submerged into flowing water at room temperature. After three thermal shock cycles, the residual compressive strength of the reticulated porous ceramic samples was tested at ambient temperature. The residual strength ratio was calculated to evaluate the change in compressive strength before and after water quenching. The phase compositions of the SiC reticulated porous ceramic samples were characterized using X-ray diffraction (XRD, Phillips X'Pert MRD). The microstructure was analyzed using scanning electron microscopy (SEM, FEI Quanta 200).

3. Results and discussion

3.1. Rheological behavior of Si slurries

The rheology of the slurry plays a crucial role in the coating processes. The coating slurry should be adjusted to meet various rheological requirements, such as viscosity, thixotropy, and yield stress. Fig. 2a shows the viscosity of Si precoating slurries with different contents of CMC. As the content of CMC increased, the viscosity of the Si slurries gradually increased. The reticular molecular chain of CMC continuously hydrolyzed into polyanions and absorbed onto the surface of Si powder particles in the slurries. Consequently, association networks formed between strands of Si in the slurry through the addition of CMC, leading to the inhibition of Si powder particle movement [16]. Pseudoplastic properties of the slurries were evaluated using the Herschel-Bulkley model (Equation (1)) [17]:

$$\tau = \tau_0 + \kappa\gamma^n \tau = \tau_0 + \kappa\gamma^n \quad (1)$$

where τ is shear stress, τ_0 is yield stress, γ is applied shear rate, κ is the consistency index, and n is the shear-thinning index. The calculated shear thinning indices of the slurries (0.87, 0.92, 0.81, and 0.75) were all lower than 1. As a result, all of the Si slurries demonstrated non-Newtonian fluid properties and exhibited shear-thinning behavior. An enhancement of shear-thinning behavior in the slurries was observed with an increase in CMC content, which was advantageous for the

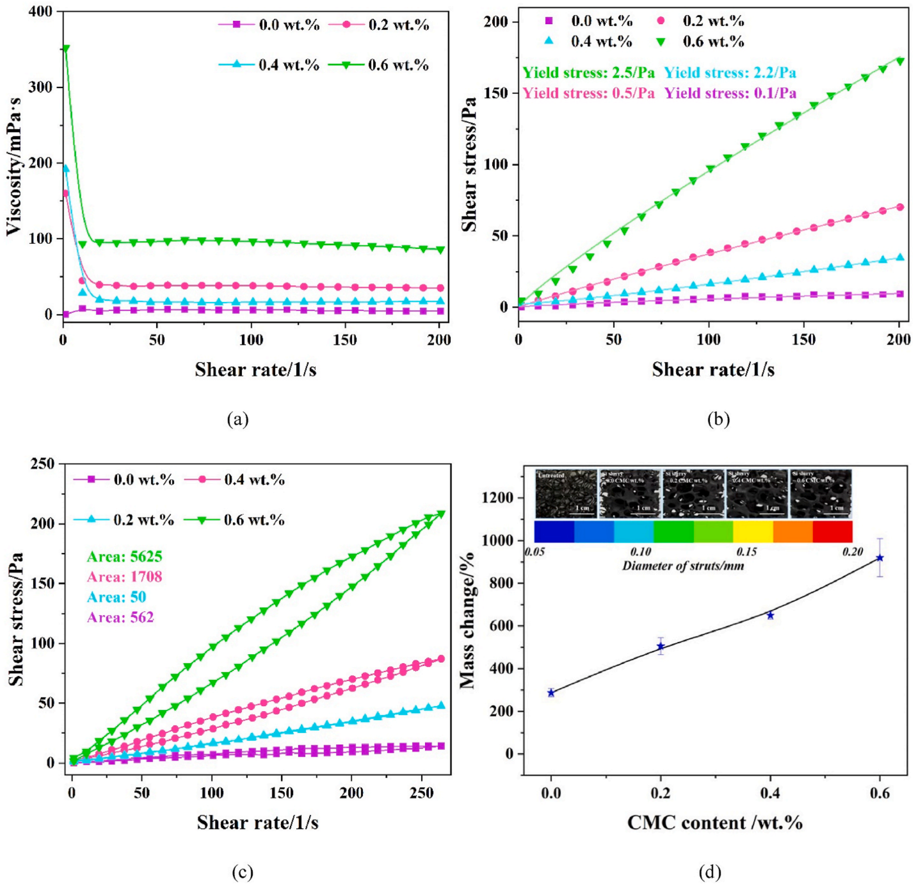

Fig. 2. Effects of the content of CMC on the rheological behavior of metal Si slurries and mass gain of green bodies (a) viscosity, (b) the relationship between shear rate and shear stress, (c) thixotropy loop, (d) mass gain and diameter of struts. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

removal of slurry from the template during rolling. Furthermore, the yield stress of the slurries increased as the CMC content increased (Fig. 2b). When the CMC content was below 0.2 wt%, the yield stress of the slurry was less than 0.5 Pa. Consequently, this made the coating layer structure susceptible to damage, which had a negative impact on the coating process. The weakened intermolecular interaction between the CMC molecules was considered to be the main reason for this [18]. Additionally, thixotropy is a time-dependent characteristic that describes the time needed for the slurry structure to recover from damage. The content of CMC had a significant influence on the thixotropy of the Si slurry (Fig. 2c). As the CMC content increased, the thixotropy of the slurry initially increased and then decreased. When the CMC content was 0.2 wt%, the slurry exhibited poor thixotropic behavior, which was unfavorable for the coating processes. However, at CMC contents of 0.4 wt% and 0.6 wt%, both slurries demonstrated the ability to rapidly regain viscosity after rolling, aiding in the coating of the slurry on the template surface. This phenomenon was primarily attributed to the higher rate of CMC macromolecule disentanglement compared to re-entanglement [18,19]. Fig. 2d presents the impact of CMC content on the mass change (gain after pre-coating) and morphology of precoated green bodies. With an increase in CMC content from 0 to 0.6 wt%, the mass change rate of the green bodies increased from 280% to 910%. Additionally, the diameter of coated struts increased from 0.1 mm to 0.2 mm, as the thixotropy and shear thinning behavior of the slurry improved with increasing CMC content. This resulted in an increase in coating layer thickness. However, when the CMC content reached 0.6 wt %, a considerable amount of residual slurry remained in the template after rolling, leading to a reduction in the porosity and permeability of the final product. Therefore, the Si slurry containing 0.4 wt% CMC was chosen to precoat polymer templates.

3.2. Phase composition and microstructure of SiC reticulated porous ceramic

Fig. 3 presents phase compositions of SiC reticulated porous ceramic samples that were heated at different temperatures. It was observed that as the temperature increased, there was an increase in the intensity of the diffraction peaks of SiC. This was because the Si in the precoating layer reacted with the residual carbon from the polymer template and the CO gas produced by the reaction between carbon bed and O_2, resulting in the generation of SiC whiskers. The relationship between the ΔG and temperature for the reactions (Equations (2)–(5)) is presented in Fig. 4. During the heating process, as the temperature ranged from 1400 °C to 1550 °C, the ΔG values for all reactions were negative. This indicates that the reactions would take place under these temperature conditions. Consequently, various reaction routes would be responsible for the generation of SiC whiskers. Additionally, a small amount of mullite phase were found in the matrix when the temperature reached 1400 °C. The intensity of diffraction peaks of mullite phase increased with the heating temperature. Due to the strong impact of liquid formation (caused by clay in the recoating layer) at elevated temperatures, mullite formation was significantly slower at 1400 °C but remarkably quicker at 1550 °C.

$$C_{(S)} + 0.5O_2 \rightarrow CO_{(g)} \Delta G = -833621 + 72.4T \quad (2)$$

$$2Si_{(S)} + CO_{(g)} \rightarrow 2SiO_{(g)} + C_{(S)} \Delta G = -439281 + 150.88T \quad (3)$$

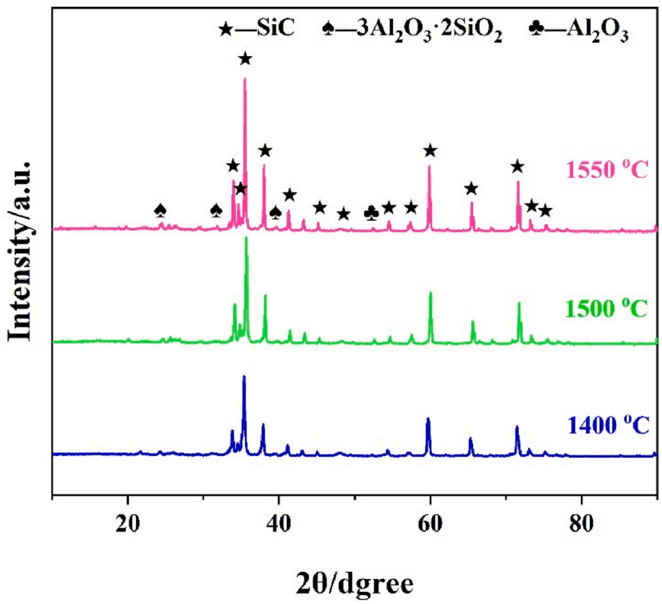

Fig. 3. Phase compositions of the SiC reticulated porous ceramic sintered at different temperatures.

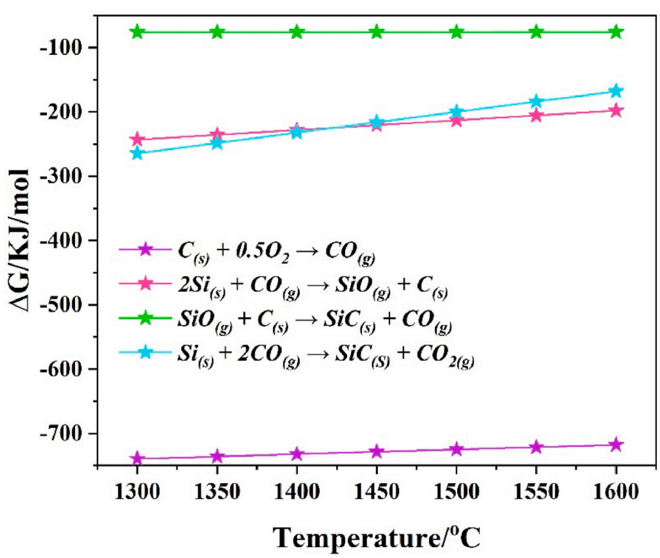

Fig. 4. Gibbs' free energy change for reactions as a function of temperature.

$$SiO_{(g)} + C_{(S)} \rightarrow SiC_{(s)} + CO_{(g)} \Delta G = -76759 + 0.6059T \quad (4)$$

$$Si_{(S)} + 2CO_{(g)} \rightarrow SiC_{(s)} + CO_{2(g)} \Delta G = -682834 + 321.85T \quad (5)$$

Additionally, the catalyst $Fe(NO_3)_3$ accelerated the generation and growth of SiC whiskers during the heating process. The effect of the heating temperature on the microstructure of SiC reticulated porous ceramic is shown in Fig. 5. It was observed that the SiC whiskers were generated and filled the hollow voids within the SiC struts. The CO gas, which was formed by the reaction between the carbon powder bed and oxygen, covered the reticulated porous ceramic. Due to its high flowability at high temperatures, the CO gas was able to easily fill the voids within the struts. Furthermore, the CO atmosphere prevented the volatilization of residual carbon in the burned-out polymer templates. Therefore, during the heating process, the Si in the precoating layer reacted with the CO gas and residual carbon, resulting in the formation of SiC whiskers in the hollow voids, as depicted in Fig. 6. Nevertheless, at a temperature of 1400 °C, the voids were not completely filled with generated SiC whiskers, leading to the presence of pores in the SiC struts. This resulted in a degradation of the strength and thermal shock resistance of the reticulated porous ceramic. As the temperature increased, the porosity of the struts decreased gradually. Meanwhile, the SiC whiskers grew gradually, leading to their increased aspect ratio. The relationship between the reaction rates and temperature was determined by using the Arrhenius equation (6) [20]:

$$k = Ae^{\frac{-Ea}{RT}} \quad (6)$$

where k is the rate constant (frequency of collisions resulting in a reaction), T is the absolute temperature, A is the pre-exponential factor, Ea is the activation energy for the reaction, and R is the universal gas constant. The reaction rate between Si and CO rose as the temperature increased. This was due to the enhanced chemical reactivity of CO gas and Si at higher temperatures. Furthermore, at a temperature of 1414 °C, the solid phase of Si transformed into a liquid phase. As the temperature increased, the viscosity of the liquid phase of Si decreased considerably. According to the VLS theory [21], the presence of a low-viscosity liquid phase had a positive impact on the growth of SiC whiskers. Consequently, increasing the temperature was beneficial to promote the process of filling pores with the whiskers.

3.3. Mechanical properties of SiC reticulated porous ceramic

The temperature range of 1400 °C–1550 °C was utilized for sintering in order to investigate the impact on the mechanical properties of the SiC reticulated porous ceramic (Fig. 7). In comparison to the properties of SiC reticulated porous ceramic previously reported by others [22,23], the bulk density of the SiC reticulated porous ceramic precoated with Si layer did not show any significant change. Additionally, the porosity of the precoated SiC reticulated porous ceramic remained at a high level (≥76%), which played a crucial role in ensuring their permeability. Furthermore, the precoated reticulated porous ceramic exhibited a higher compressive strength. The generated SiC whiskers filled the voids within the SiC struts, resulting in an improvement of the density of the struts. Moreover, the generated SiC whiskers strengthened and toughened the SiC matrix. As the temperature increased, the linear shrinkage, bulk density, compressive strength, and thermal shock resistance of the precoated SiC reticulated porous ceramic gradually increased; however, its porosity decreased. These phenomena were mainly attributed to the formation of a liquid phase at higher temperatures, which accelerated the sintering process of the ceramic coating layer. Additionally, the generation and growth of SiC whiskers were promoted at high temperatures, facilitating filling voids and reducing the number of flaws (pores and cracks) in the ceramic struts. This led to an increase in thermal conductivity of the ceramic struts, reducing the generation of thermal stresses caused by temperature differences. Furthermore, the hollow voids were filled with SiC whiskers, optimizing the structure of the voids and inhibiting stress concentration. At a temperature of 1550 °C, the porosity, bulk density, compressive strength, and residual strength ratio of the pre-coated SiC reticulated porous ceramic reached 76%, 0.55 g/cm^3, 1.85 MPa, and 83%, respectively.

3.4. Industrial application test

Large-sized SiC reticulated porous ceramic parts prepared and sintered at 1550 °C in this study (Fig. 8) were used as filters in the copper smelting industry. The SiC filters had a pentagon shape with dimensions of 540 mm in width, 300 mm in height, and 50 mm in thickness. The SiC filters with smaller-sized pores (30 ppi) were chosen in order to purify the molten copper due to their higher blocking efficiency and larger adsorption area. It was found that the hollow voids within the SiC struts were filled with generated SiC whiskers, which had a large aspect ratio. This endowed the filter with excellent properties. To assess the service

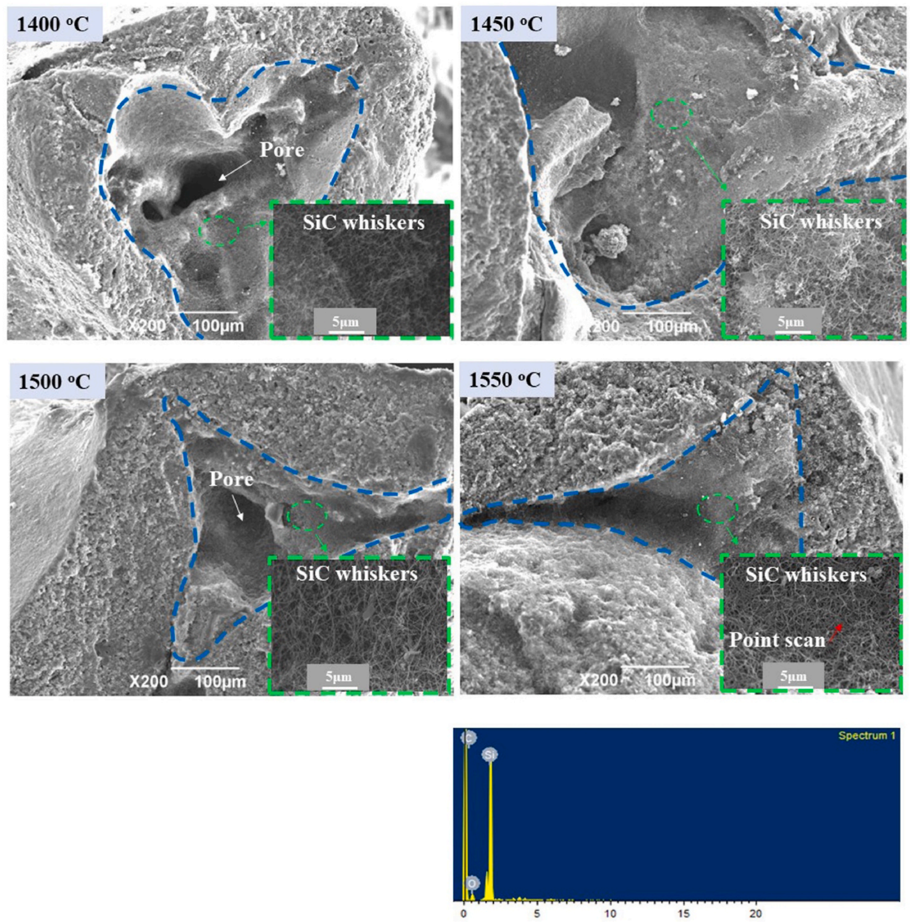

Fig. 5. SEM images showing the microstructure of the SiC reticulated porous ceramic sintered at different temperatures. The bottom is an energy-dispersive X-ray spectroscopy (EDS) spectrum.

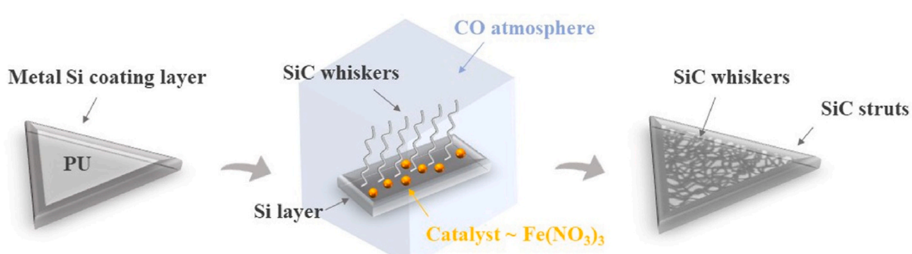

Fig. 6. Schematic diagram of structure evolution of reticulated porous ceramic with Si coating layers.

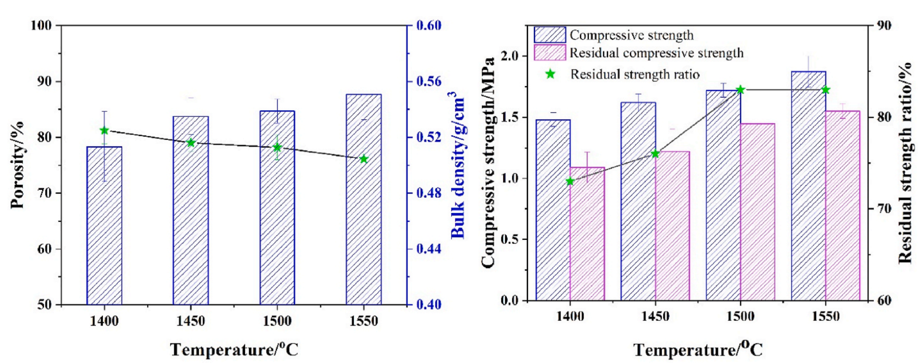

Fig. 7. Mechanical properties and thermal shock resistance of SiC reticulated porous ceramic sintered at different temperatures (a) porosity and bulk density and (b) compressive strength and residual strength ratio.

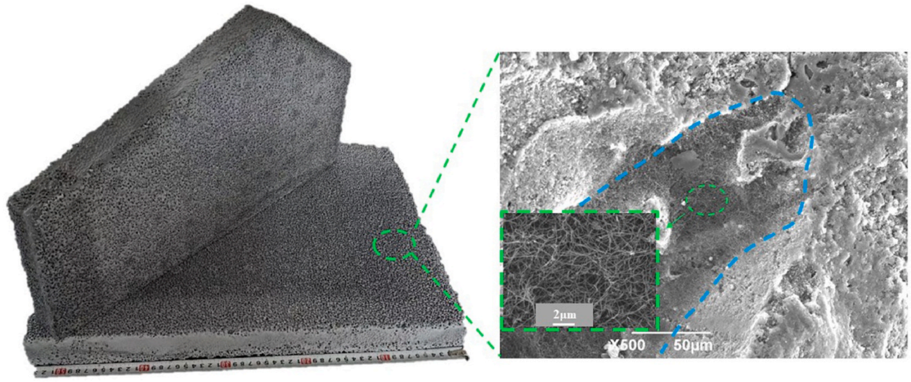

Fig. 8. Macrostructure and microstructure of SiC filter sintered at 1550 °C.

performance of the fabricated SiC filters, they were implemented in the SCR4500 copper smelting system by Southwire company in the USA. Even after a work cycle of approximately a week, the structure of the SiC filters remained intact. Meanwhile, a significant amount of inclusions adhered to the surface of the filters (Fig. 9 a and b). These inclusions primarily consisted of Al_2O_3 and SiO_2, which might be caused by the spalling of refractories within the furnace lining. Additionally, traces of metal copper and copper oxides were present on the filter's surface. Notably, the resulted copper matrix did not contain impurities such as refractory and copper oxides, which led to a clear improvement in the quality of the copper rod (Fig. 9c). Accordingly, the combination of the precoating processes and the in-situ SiC whisker generation proved to be an effective method for fabrication of high-performance SiC filters for industrial applications.

4. Conclusions

SiC reticulated porous ceramic with excellent permeability and high mechanical strength were prepared using the polymer replica method combined with the precoating technique. The viscosity and thixotropy of the metal Si precoating slurries, with a solid content of 60 wt%, were adjusted by adding a thickening agent. The optimal amount of thickening agent was determined to be 0.4 wt%. After being heated at high temperatures in a carbon embedded condition, the reticulated porous ceramic with the double-coated layer structure exhibited outstanding properties. The SiC whiskers were produced through an in-situ reaction between Si and CO/C and filled the hollow voids in the SiC struts created by burning out the polymer template. Furthermore, as the temperature increased, the SiC whiskers visibly grew and completely filled the voids. Industrial tests were also conducted to evaluate the service performance of SiC filters. After a week of use in the SCR4500 copper smelting system, the structure of the SiC filter with a porosity of ~30 ppi remained undamaged. Additionally, a significant amount of inclusions, such as Al_2O_3 and SiO_2, in the molten copper were adhered to the surface of the SiC filter. The combination of the precoating and recoating processes and the in-situ generation of SiC whiskers allowed for low-cost and high-

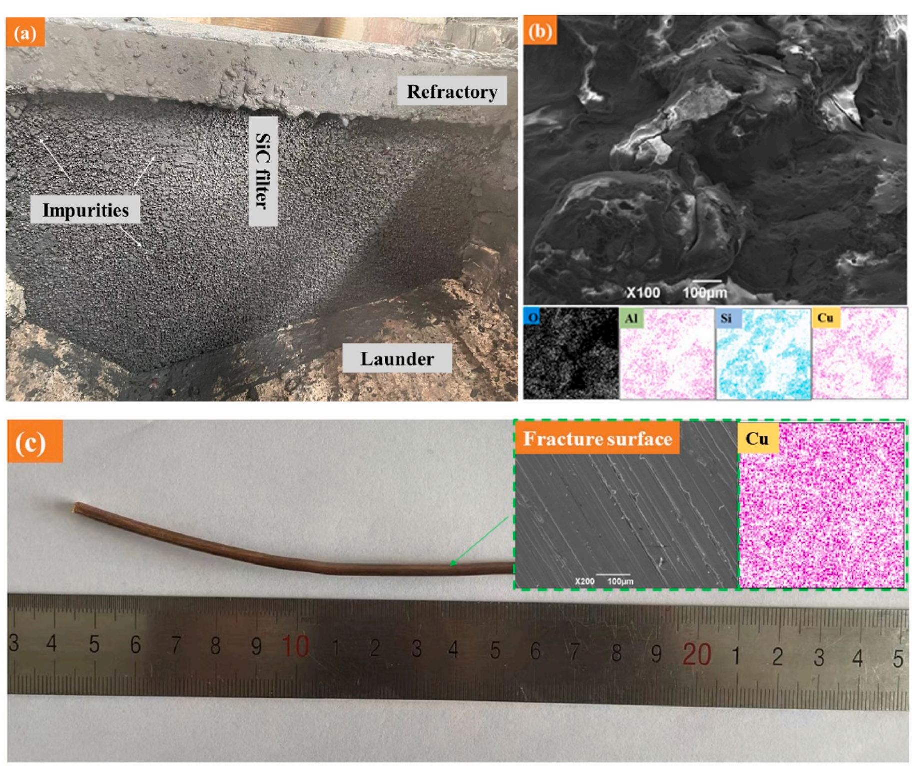

Fig. 9. Industrial application test result on SiC filters with pentagon shape (30 ppi): (a) macrostructure of the filter after use for a week, (b) SEM image and EDS elemental maps of inclusions adhered to the filter, (c) macrostructure, microstructure and EDS elemental map of copper rods produced with the use of the SiC filter.

efficiency fabrication of high-performance SiC filters for industrial applications.

Declaration of interest statement

We declare that we have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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