



Effect of organic additives on mechanical properties of SiC ceramics prepared by a modified gelcasting method

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Abstract

A novel and simple gel system of isobutylene and maleic anhydride (PIBM) was used to prepare SiC ceramics. The rheological behaviour of the SiC slurries was investigated as function of organic additives. The SiC slurries with 0.2 wt.% PIBM and 0.2 wt.% tetramethylammonium hydroxide (TMAH) showed low viscosity, which was favourable for casting SiC green bodies. In order to obtain homogeneous green bodies, polyvinyl alcohol (PVA) was used to assist the dispersion of carbon black in the slurries, and polyethylene glycol (PEG) was added to inhibit the surface exfoliation of green bodies. The content of PVA was controlled carefully to avoid the warpage of green bodies during the drying process. Finally, homogeneous defect-free SiC green bodies were successfully fabricated via aqueous gelcasting. The SiC ceramics sintered at 2100 °C (prepared from slurries with solid content of 60 wt.%) showed an average flexural strength of 305.7 MPa with porosity of 19.92%.

Keywords: gelcasting, PIBM, SiC slurries, SiC green bodies, rheological behaviour

I. Introduction

Silicon carbide (SiC) ceramics possess a unique combination of desirable properties, such as excellent high temperature strength, high chemical stability, high heat shock stability, and outstanding wear resistance [1–4]. SiC ceramics is a potential candidate for high-temperature structural applications such as in advanced heat engine parts, heat exchangers, friction and wear-resistant components, etc. [5–7]. Many processing technologies, such as dry-pressing, isostatic pressing and injection moulding, have been used to fabricate SiC ceramics [8–10]. However, these traditional forming methods are not suitable for preparing complicated shaped ceramic parts. Gelcasting technique has exhibited many advantages for fabricating complicated shaped and large sized ceramic parts with homogeneous microstructure since the first development in 1991 by Omatete *et al.* [11,12].

The gelcasting process is generally based on polymerization of organic monomers. However, the commonly used monomer like acrylamide exhibits neural toxicity [13], which limits its application. Recently,

Yang *et al.* [14] reported a novel and simple gelcasting method by the use of a water-soluble nontoxic copolymer of isobutylene and maleic anhydride (PIBM), the commercial name of PIBM is Isobam. The copolymer is a promising candidate that acts as both dispersing and gelling agent for gelcasting of ceramics at room temperature. PIBM has been successfully applied in fabrication of Y₂O₃ ceramics [15], YAG ceramics [16], AlON ceramics [17], and Si₃N₄ ceramics [18]. However, SiC ceramics fabricated through the gel system of PIBM have rarely been reported until now. In addition, the comprehensive research on the SiC slurries and SiC green bodies of this gel system is necessary for preparing SiC ceramics with good performance.

The aim of the present work is to prepare homogeneous SiC slurries and SiC green bodies with the novel PIBM gel system and investigate their mechanical properties. The influence of PIBM and other organic additives on the rheological behaviour of SiC slurries was investigated. To obtain homogeneous SiC slurries and SiC green bodies, PVA was added into the slurries to promote the dispersion of sintering additives, and PEG was used to inhibit the surface exfoliation of SiC green bodies. The effects of organic additives and solid contents on the densities of SiC green bodies were researched. The porosity and mechanical property were evaluated

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after the green bodies were sintered.

II. Experimental

The starting powder was commercially available SiC powder ($d_{50} = 0.5 \mu\text{m}$, α -phase, FCP15C, Sika Tech., Lillesand, Norway). B_4C powder ($d_{50} = 1 \pm 0.2 \mu\text{m}$, Mudanjiang Jingangzuan Boron Carbide Co., Ltd, China) and carbon black (CB) powder ($d_{50} = 0.5 \mu\text{m}$, Shanghai Coking & Chemical Development, Shanghai, China) were used as the sintering additives in the solid-state-sintering process. The weight percentages of CB and B_4C powder based on the SiC powder were 2.5 wt.% and 0.5 wt.%, respectively. PIBM 104# (Kuraray Co., Ltd., Osaka, Japan) was used as gelling agent. Tetramethylammonium hydroxide (TMAH, TaixingHaoshen Chemical Trading CO., Ltd, China) was used as dispersant. Polyvinyl alcohol (PVA, Sinopharm Chemical Reagent Co., Ltd, China) was used to improve the dispersibility of CB [19]. PVA was dissolved in deionized water to make a solution with concentration of 5 wt.% at first. Polyethylene glycol (PEG, Sinopharm Chemical Reagent Co., Ltd, China) was used to inhibit the surface exfoliation of silicon carbide green bodies. The weight percentages of PIBM, TMAH, PVA and PEG based on the SiC powder were 0.2–0.6 wt.%, 0.2 wt.%, 0.1–0.3 wt.% and 2.0 wt.%, respectively. The above mixtures with different solid loadings (55–65 wt.%) and organic amounts were milled with SiC balls for 3 h at 300 rpm. The obtained homogeneous slurries were casted into plastic moulds and then consolidated. After demoulding, the green bodies were dried at 60 °C for 12 h. Finally, the samples were sintered at 2100 °C for 2 h in Ar atmosphere to obtain the SiC ceramics.

Zeta potential of SiC powders with organic additives was examined by Zetaplus (Brookhaven Instruments Corp., USA). Rheological measurements were performed on a stress controlled rheometry (SR-5 Rheometric Scientific Instrument Company, USA), the measurement was carried out at 25 °C. The densities of the green bodies were calculated from the mass and

the volume. The porosities of sintered SiC ceramics were measured by the Archimedes method. Specimens were machined to a dimension of $3.0 \times 4.0 \times 36.0 \text{ mm}^3$ to test the flexural strength at room temperature via the three-point bending test (Model AUTOGRAPHAG-I, Shimadzu Co. Ltd., Japan) with a support distance of 30.0 mm and a cross-head speed of 0.5 mm/min; 4 bars of each kind of sample were tested. The linear shrinkage was calculated by measuring the width of the sample before and after sintering. The fracture surface of the fabricated SiC green bodies and sintered specimen were observed by scanning electron microscopy (SEM, JSM-6700F, JEOL, Akishima, Japan).

III. Results and discussion

3.1. Effect of PIBM content on rheology of SiC slurries

Yang et al. [14] have reported the molecular structure of PIBM where the copolymer has the active $-\text{COO}^-\text{NH}_4^+$ and $-\text{CONH}_2$ groups. The copolymer molecule containing multi-functional groups can act as both dispersant and gelling agent. The gel mechanism of the slurry with PIBM might be the cross-linking through ionic interaction and/or hydrogen bonding among different functional groups of PIBM molecules absorbed on the surface of SiC particles [20].

The rheological properties of 55 wt.% SiC slurries with different PIBM content (without other organic additives) are shown in Fig. 1. All slurries exhibited a shear-thinning behaviour. The viscosity of the slurry increased apparently with changing the PIBM contents from 0.2 wt.% to 0.6 wt.% due to the hydrodynamic coupling between free PIBM molecules and SiC particles [21]. In addition, without the addition of PIBM, the 55 wt.% SiC slurries showed no fluidity and could not be operated. Thus, 0.2 wt.% PIBM was more suitable in preparing slurries with better casting performance.

3.2. Effect of TMAH on rheology of SiC slurries

The PIBM has been proved to be a promising dispersant in preparing alumina slurries with solid content as high as 50 vol.% [20]. The Si_3N_4 slurries prepared through this gel system were suitable for casting with solid content of 35 vol.% [18]. It is obvious that dispersion impact of PIBM varies in different slurries. In this work, although PIBM can act as dispersant in the SiC slurries, the slurries with solid content of 55 wt.% (nearly 28 vol.%) still exhibited relatively high viscosity as shown in Fig. 1, which led to inhomogeneous dispersion of the slurry. To decrease the viscosity of the slurries, another commonly used dispersant TMAH was added into the slurries. Figure 2 shows the effect of TMAH on the viscosity of 55 wt.% SiC slurries with 0.2 wt.% PIBM. By adding 0.2 wt.% TMAH into the slurry, the viscosity decreased greatly, indicating that TMAH acts as dispersant and can improve the performance of the SiC slurries.

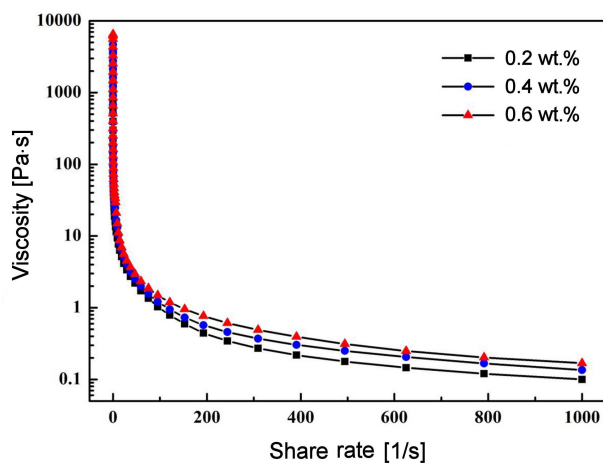


Figure 1. Effect of PIBM content on the viscosity of 55 wt.% SiC slurries

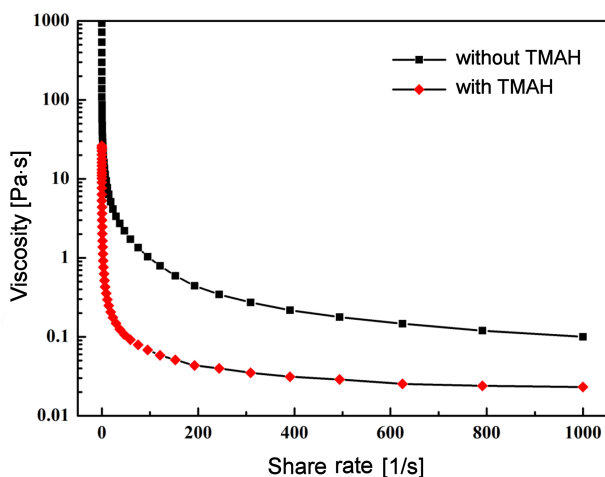


Figure 2. Effect of 0.2 wt.% TMAH on the viscosity of 55 wt.% SiC slurries with 0.2 wt.% PIBM

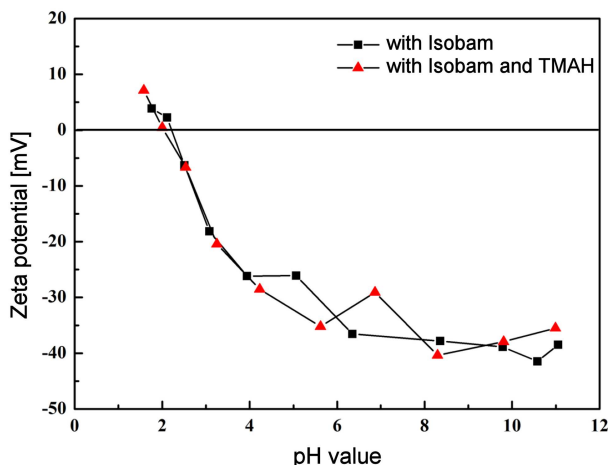


Figure 3. Effect of TMAH on the Zeta potential of SiC powder with PIBM

The zeta potentials of SiC particles in the absence and presence of TMAH are shown in Fig. 3. The addition of TMAH did not show obvious effect on the pH_{IEP} of SiC particles (with PIBM), suggesting no specific adsorption of TMAH in this pH range. It is proposed that the adsorption of TMAH is driven by a non-electro-static force (around pH_{IEP}), similar to that reported by Paik *et al.* [22].

3.3. Effect of PVA on properties of SiC slurries and green bodies

Figure 4 shows the dispersibility of carbon black (CB) in the 55 wt.% SiC slurries, the width of the square plastic moulds was 50 mm. In Fig. 4a, poor dispersion of CB was clearly observed in the initial slurry because of the bad wettability of CB in water. The addition of TMAH showed limited effect on the dispersion of CB in the slurry. However, with the addition of 0.1 wt.% PVA, the CB was dispersed homogeneously in the slurry, and uniform slurry was obtained in Fig. 4c. This demonstrated that small addition of PVA can assist the dispersion of CB in SiC slurries.

CB particles typically contain 90–99% element carbon, with oxygen and hydrogen as the other major constituents. Thus, CB particles are strongly hydrophobic and tend to aggregate when dispersed in water [23]. The surface modification has great effects on the dispersion of CB. One of the strategies is physical adsorption modification by encapsulating CB particles with surfactants and polymers. The experimental result has proved that highly hydrolysed PVA is suitable to prepare hydrophilic modified CB particles [24]. Molecular dynamics simulation has also showed that the PVA molecules can form incomplete single-layer, complete single-layer, defective double-layer, double-layer and triple-layer film through physical adsorption [25]. The interaction energy of CB particles changes greatly with the adsorption of PVA molecules onto the CB particles, which makes the CB/water interface more stable and significantly improves the dispersion stability of CB particles.

However, the content of PVA needs to be controlled carefully. By increasing the PVA content from 0.1 wt.% to 0.3 wt.%, warpage occurred in the SiC green body as shown in Fig. 5. Therefore, the PVA content should be controlled below 0.3 wt.% to avoid warpage of the SiC green bodies. During the drying process of green bodies, warpage can be caused by inner stresses due to non-symmetrical shrinkage, and warpage can arise even in a wet cast material with a high homogeneity [26]. If the shrinkage throughout the process is uniform, the green body will not deform or warp. The liquid water in the wet green body is transported to the surface of the part

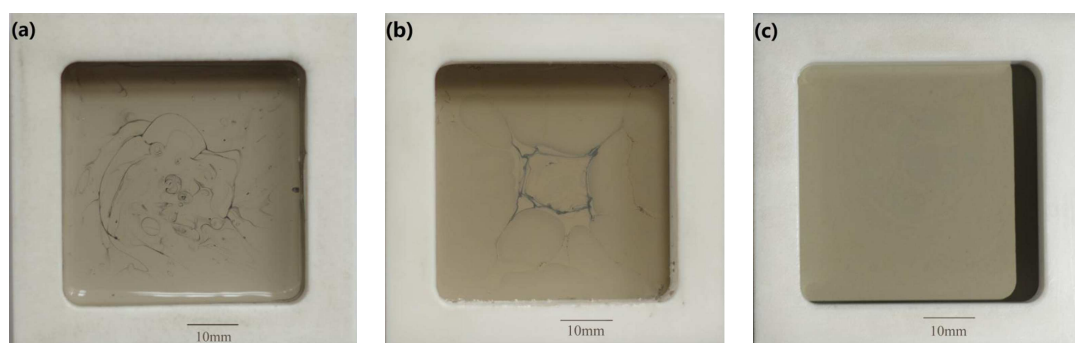


Figure 4. Dispersibility of C in the 55 wt.% SiC slurries: a) with 0.2 wt.% PIBM; b) with 0.2 wt.% PIBM and 0.2 wt.% TMAH and c) with 0.2 wt.% PIBM, 0.2 wt.% TMAH and 0.1 wt.% PVA

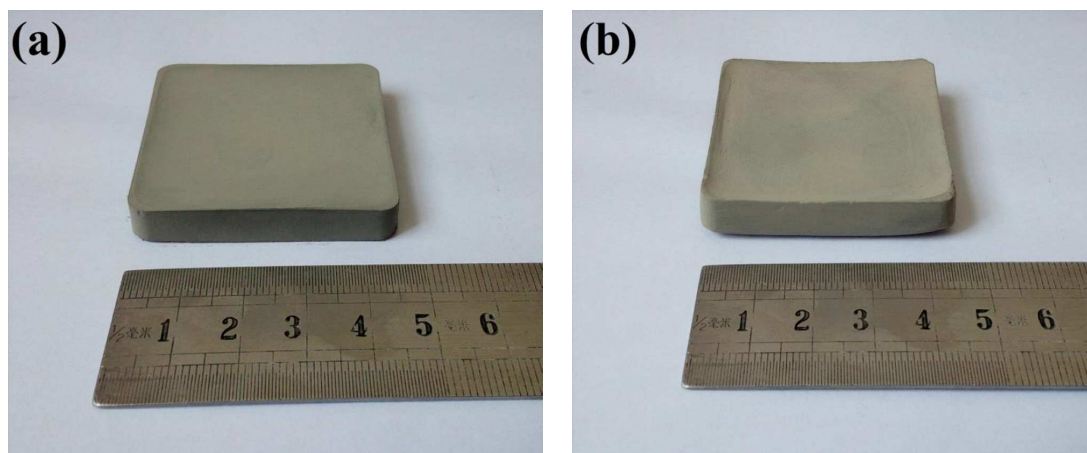


Figure 5. Effect of PVA content on the warpage of SiC green bodies prepared from the 55 wt.% slurries: a) with 0.1 wt.% PVA and b) with 0.3 wt.% PVA

primarily by capillary forces, and the large compressive stresses caused by the capillary forces lead to the bend of the gel matrix [27]. The SiC particles that firmly attach to the gel network move towards each other when the network collapses, which in turn causes bulk shrinkage of the green body. The added PVA molecules adsorb on the particles and partly dissolve in the water, which will change the capillary forces between the SiC particles. Thus, the warpage might be caused during the drying process if the content of PVA cannot be appropriately controlled.

3.4. Effect of PEG on the surface exfoliation of SiC green bodies

Figure 6 shows the photos of the green bodies prepared by gelcasting of 55 wt.% SiC slurries in the absence and presence of PEG. The green body without PEG had an obvious exfoliation layer, and the surface exfoliation would restrict the application of this gel system. The exfoliation of SiC green bodies has also been found in other paper [28]. No exfoliation layer was observed in the green body from SiC slurry with 2 wt.% PEG, which meant that PEG could inhibit the surface exfoliation of SiC green bodies prepared through the

gelcasting with PIBM. Li *et al.* [28] have systematically studied the effect of PEG on the surface exfoliation of SiC green body prepared by gelcasting. The green body with rigid surface without exfoliation layer in their gel system was obtained at PEG content of 3.0 wt.%. In this work, we can obtain the fine green body with PEG content of 2.0 wt.%. More PEG addition should be avoided because the PEG will deteriorate the mechanical properties of green bodies at the same time [29].

3.5. The properties of SiC green bodies

The densities of the SiC green bodies were calculated through the mass and volume. Table 1 summarizes the densities of the SiC green bodies with different organic additives. The SiC green bodies were obtained from 55 wt.% slurries, the content of PIBM, TMAH, PEG and PVA were 0.2 wt.%, 0.2 wt.%, 2.0 wt.% and 0.1 wt.%, respectively. The density of the green bodies after the addition of PVA changed a little in Table 1, which meant that the 0.1 wt.% PVA did not affect the package of the SiC powders. In addition, the density of the green bodies changed negligibly with the addition of PEG, even though the PEG might partly inhibit the contact of the gelling agent [28]. Taking the error values in the mea-

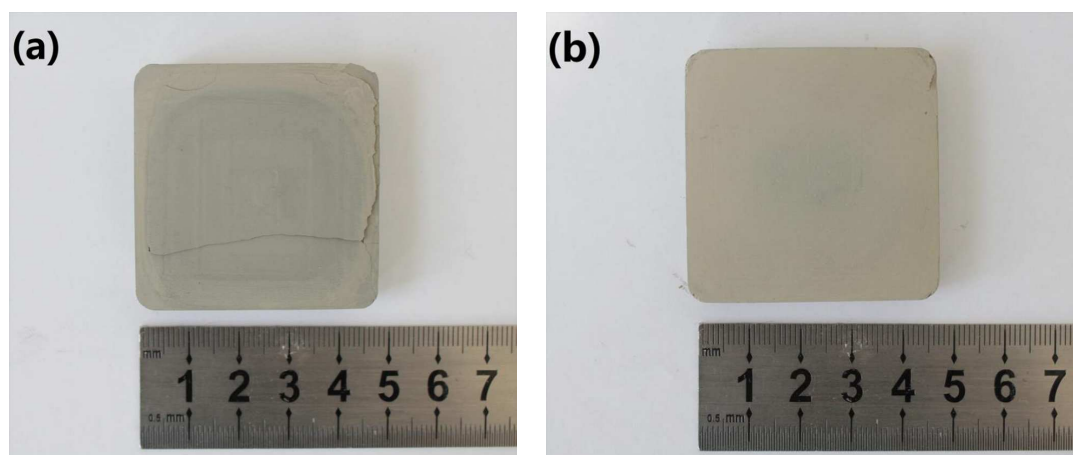
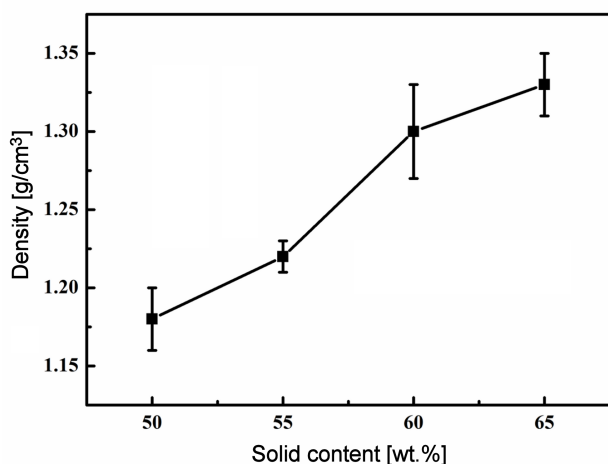
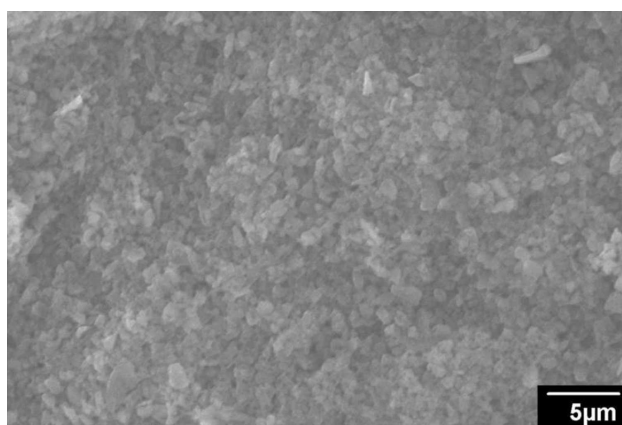


Figure 6. Surface of SiC green bodies prepared from the 55 wt.% slurries: a) without PEG and b) with 2 wt.% PEG

Table 1. The average densities of SiC green bodies prepared from slurries with different organic additives

SiC green body from 55 wt.% slurry with 0.2 wt.% TMAH	With 0.1 wt.% PVA and 2.0 wt.% PEG	With 0.1 wt.% PVA only	With 2.0 wt.% PEG only
Average density [g cm^{-3}]	1.22 ± 0.04	1.26 ± 0.02	1.22 ± 0.03

**Figure 7.** Densities of the SiC green bodies with different solid contents**Figure 8.** SEM micrograph of SiC green body fabricated from slurry with solid content of 55 wt. %

surements into consideration, the addition of 0.1 wt.% PVA and 2.0 wt.% PEG had negligible effect on the density of SiC green bodies. Figure 7 shows the densities of the green bodies with different solid contents. With increasing the solid contents from 50 wt.% to 65 wt.%, the densities increased from 1.18 to 1.34 g cm^{-3} . Higher solid loading could lead to higher green density due to the packing behaviour in the slurries as well as in the gelcasting procedure.

The fracture surface of the SiC green body fabricated from slurry with solid content of 55 wt.% is shown in

Fig. 8. The fracture surfaces showed a rather uniform SiC particles distribution, and no obvious defects or big pores were observed in the well packed body.

3.6. Effect of solid content on properties of SiC ceramics

The homogeneous SiC green bodies were sintered at 2100 °C to obtain SiC ceramics. Table 2 shows the flexural strength, porosity and linear shrinkage of the sintered SiC ceramic samples. With the increasing of solid content from 55 wt.% to 65 wt.%, the porosity of SiC ceramics varied from 26.29% to 19.40%, and the flexural strength was in the range of 131.3–305.7 MPa. For the samples sintered from 60 wt.% solid content slurry, a high average flexural strength of 305.7 MPa was obtained. Figure 9a shows the microstructure of the SiC ceramics fabricated from slurry with solid content of 60 wt.%. The distribution of SiC grains was uniform and no defects existed. However, the ceramics prepared from 65 wt.% solid content slurry exhibited lower flexural strength of 161.7 MPa with a similar porosity. The linear shrinkage of SiC ceramics after sintering at different solid contents was almost the same, which meant the low flexural strength was not caused by the sintering procedure. Comparing with the slurry at 60 wt.% solid content, the slurry with 65 wt.% solid content had relatively high viscosity.

Figure 10 shows viscosity of the SiC slurries with solid contents of 60 wt.% and 65 wt.%. In the slurry with solid content of 65 wt.%, the viscosity reached high value both at higher and lower shear rate. Air bubbles in the high viscosity slurry were hard to eliminate completely, and the residual air bubbles were entrapped into the green bodies in the gelling process, which acted as the defects in the ceramics and deteriorated the mechanical properties. The defects caused by residual air bubbles are shown in Fig. 9b. The results indicate that the sintered SiC ceramics are not fully dense and the further experiment is under investigation to improve density and mechanical properties. To obtain SiC ceramics with densities above 90 %TD, we might need to increase sintering temperature or increase the holding time. In addition, a better combination of different dispersing agents to obtain green bodies with higher densities will also lead to ceramics with higher densities. The SiC ceramics

Table 2. Flexural strength, porosity and linear shrinkage of SiC ceramics after sintering

Solid content [wt.%]	55	60	65
Porosity [%]	26.29	19.92	19.40
Flexural strength [MPa]	131.3 ± 15.3	305.7 ± 21.6	161.7 ± 18.3
Linear shrinkage [%]	16.52	16.43	16.75

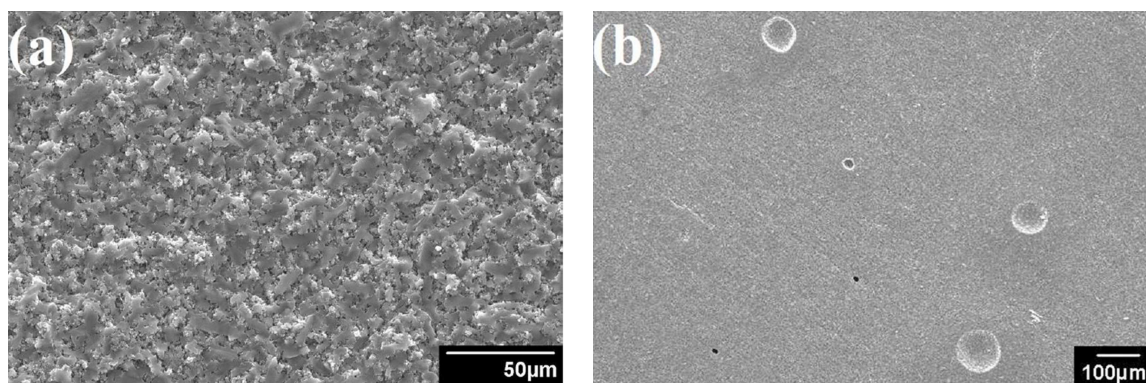


Figure 9. SEM micrographs of the fracture surface of SiC ceramics: (a) high magnification shows the microstructure of ceramic fabricated from 60 wt.% slurry; (b) shows the defects caused by the residual air bubbles in ceramic fabricated from 65 wt.% slurry

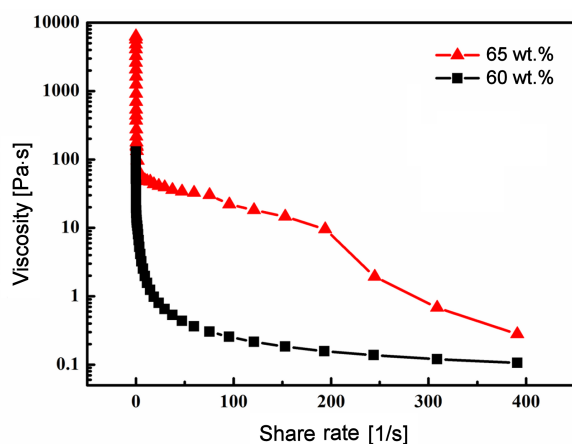


Figure 10. Effect of solid content on the viscosity of SiC slurries

with 19.40% porosity showed a high flexural strength of 305.7 MPa, which was higher than the SiC ceramics sintered at 2100 °C with density above 97 %TD reported by Wei *et al.* [30].

IV. Conclusions

Homogeneous SiC slurries and green bodies were prepared by the gel system of PIBM. The effects of PIBM and organic additives on the SiC slurries and SiC green bodies were studied. The PIBM content of 0.2 wt.% was proved to be suitable for preparing SiC slurries with fine rheological properties. TMAH acting as dispersant can lower the viscosity of the slurries without damaging the stability of SiC particles in the slurries. The experimental results also showed the addition of 0.1 wt.% PVA can promote the dispersion of CB in the slurries, but the content of PVA should be controlled below 0.3 wt.% to avoid the warpage of SiC green bodies. The role of PEG in this system was to eliminate the surface exfoliation of SiC green bodies gelled in air. After gelcasting and drying, green bodies with homogeneous microstructure were obtained. The SiC ceramics fabricated from slurry with solid content of 60 wt.% showed a flexural strength of 305.7 MPa. This study provides the overall modified design of the gel system

of PIBM in fabricating homogeneous SiC slurries and green bodies, which is helpful for preparing SiC ceramics by the PIBM gel system.

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