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Aqueous tape casting process for SiC

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Abstract

The condition for the preparation of stable SiC slurries by aqueous tape casting was identified. To acquire stable uniform slurries, the influences of dispersant, solid loading, sintering additives, binder and plasticizer on the rheological properties and viscosity were investigated. The conditions for preparing stable SiC slurries were studied and optimized. After tape casting and drying, the green SiC sheets showed smooth surface and homogeneous microstructure. The SiC ceramic can be densified to 98.89% after hot-pressing at 1850 °C (at 25 MPa in Ar for 30 min). The flexural strength, hardness, and toughness are 779.5 ± 39.2 MPa, 21.51 ± 0.70 GPa and 5.54 ± 0.26 MPa $m^{1/2}$, respectively. SEM shows a fine microstructure with few pores in the sintered samples. The fracture surface exhibited predominantly intergranular fracture type.

Keywords: A. Suspensions; A. Tape casting; C. Mechanical properties; D. SiC

1. Introduction

Tape casting is a widespread and cost effective method to produce flat, thin and large area ceramic substrates and multilayer structures mainly for the electronic industry [1–4]. Basically, it consists in the preparation of a suspension of the ceramic powder in a solvent, with addition of a dispersant, binder and plasticizer [5]. Organic-solvent-based tape casting systems are widely used mainly because one can obtain improved quality of tape and because of easy and fast evaporation of solvents during drying stage. However, in recent years, much effort has been made to replace the highly toxic organic systems by aqueous medium [6–14] because water as a solvent has the advantage of being non-toxic, non-flammable, easy available and cheap [15].

The combination of high strength, hardness, creep resistance and oxidation resistance makes silicon carbide a candidate material especially for high temperature structural applications and heat exchangers. Silicon green tapes are required for Si/SiC and other composites, functionally graded materials and applications. In the literature, several studies have been carried out concerning non-aqueous tape casting of SiC powders [16,17]. Aqueous tape casting of SiC with PEI and carboxylic acid as the dispersant has also been described in literature [18,19]. However, a systematic study of the dispersion and aqueous tape casting of SiC is needed for this technologically important material.

A well-deagglomerated suspension is necessary to get a high green density. In the present work, aqueous tape casting of submicron SiC powder was studied. Concentrated SiC slurry with sintering aids suitable for aqueous tape casting was obtained. High quality SiC green sheets were prepared. The mechanical and microstructural properties of as sintered SiC samples were also studied.

2. Experimental procedure

2.1. Starting materials

The powder used was silicon carbide (FCP-15, Norton Co., MA) with average particle size of 0.50 μm and a BET surface area of 14.13 m^2/g. The sintering aids were Al_2O_3 (Shanghai Wusong Chemical Plant, China) and Y_2O_3 (Shanghai Yuelong New Materials Co., Ltd., China).

Tetramethylammonium hydroxide (TMAH, supplied as a 25 wt.% water solution, Shanghai Chemical Reagents Co., China) was selected as dispersant. PVA and 1,3-propylene

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glycol (Shanghai Chemical Reagents Co., China) were used as binder and plasticizer.

2.2. Zeta potential tests

Zeta potential values of the SiC powder suspensions were measured with Zetaplus (Brookhaven Instruments Corp., U.S.A.) at various pH values. SiC suspensions (0.01 vol.%) were prepared in the absence and presence of TMAH at various pH values, which were adjusted using HCl or NaOH.

2.3. Adsorption measurement

The adsorption of TMAH on the SiC surface was evaluated using the solution depletion method. Dilute SiC suspensions were prepared with various amounts of TMAH at pH = 10.5. After being prepared, the suspensions were centrifuged for 30 min at 6000 rpm. A portion of the supernatant was analyzed by using a total organic carbon (TOC) analyzer DC-190 (Rosemount Analytical Co.) to determine the TMAH concentration in it. The initial TMAH concentration was calculated from the added amount of TMAH. The amount that was adsorbed was taken as the difference between the initial and the residual concentration.

2.4. Sedimentation tests

5 vol.% SiC suspensions in the absence and presence of dispersant were made for sedimentation measurements. Reading of the sediment volume was recorded for 7 days. The pH value was adjusted by HCl and NaOH.

2.5. Rheological analysis

SiC suspensions were prepared at different solids loading with different amounts of TMAH. The apparent viscosity was measured at steady shear rate (175 s^{-1}) using a viscosity meter (NDJ-7, Shanghai, China). The optimal dosage was evaluated and kept constant. The rheological behavior of concentrated SiC slurry was characterized using parallel-plate system on Universal Stress Rheometer SR5 (Rheometric Scientific, U.S.A.).

2.6. Preparation of SiC slurry

Aqueous SiC slurry was prepared by mixing SiC powder and dispersant into distilled water. Slurries were ball milled with SiC balls for 24 h, followed by adding pre-prepared PVA solution and propylene glycol. The slurries were further ball milled for another 24 h.

2.7. Tape casting and sintering

After homogenizing, the slurries were degassed under vacuum to remove air bubbles. Finally, tape casting was performed on Procast Precision Tape Casting Equipment (Division of the International, Inc., Ringoes, NJ) with the gap height of 400 μm at a speed of 100 mm/min. After drying freely in open air at ambient temperature for some time, green tapes were obtained.

After laminating and binder removal at 600 °C for 1 h, the tapes were hot-pressed at 1850 °C for 30 min under a flowing argon atmosphere, with an applied pressure of 25 MPa.

2.8. Microstructure, mechanical and other properties

The density and apparent porosity of the sintered samples were measured using Archimedean method. Microstructure of both the binder-removed film and the sintered body were observed using a scanning electron microscopy (SEM, JSM-6700F, Japan). The tensile test of green tapes were performed at a constant load speed of 10 mm/min at a span length of 20 mm by using a computer-controlled INSTRON universal testing machine (Model 5566, Instron Corp., High Wycombe, U.K.). The three point flexural strengths of sintered rectangular specimens (3.0 mm × 4.0 mm × 36.0 mm) were measured, using a span width of 30 mm and a crosshead speed of 0.5 mm/min (universal testing machine, Model 5566, Instron Corp.). Hardness and toughness were measured by indentation test on Wilson-wolpert Tukon 2100B (Instron), and the load and holding time were 50 N and 10 s.

3. Results and discussion

3.1. Zeta potential of SiC powder

The surface of SiC particles is usually covered by a thin film of silica [20]. TEM observations have demonstrated that SiC powder contains 0.3–0.7 nm coating of native amorphous SiO_2 layer [21,22]. It is generally accepted that there are 4–5 Si–OH groups/nm^2 [23,24] on the smooth, amorphous silica surface. The ionization of the acid Si–OH groups on SiC surface can be described by the following equation [25]:

$$\equiv\mathrm{Si{-}OH} + \mathrm{H_2O} \rightarrow \equiv\mathrm{Si{-}OH_2^+} + \mathrm{OH^-} \quad K_1 \tag{1}$$

$$\equiv\mathrm{Si{-}OH} + \mathrm{H_2O} \rightarrow \equiv\mathrm{Si{-}O^-} + \mathrm{H_3O^+} \quad K_2 \tag{2}$$

where H^+ and OH^- are potential determining ions. The isoelectric point (IEP) can be expressed as follows:

$$\mathrm{pH_{IEP}} = 7 + \frac{\log K_1 - \log K_2}{2} \tag{3}$$

The dissociation of surface Si–OH groups of silica has been studied by Iler [23]. Results showed that the pK_a of the Si–OH groups on the surface of amorphous silica was 6.5. So in the acidic pH range, the density of surface negative charge sites should be very low, corresponding to a low zeta potential value in this region. In addition, with the increase of pH, the surface charge density of SiC particles will increase [see Eq. (2)] too, corresponding to a downward trend in zeta potential curve; see Fig. 1.

As shown in Fig. 1, the IEP of SiC powder is at pH 3.2, close to that of SiO_2 [23]. From Eq. (3), it can be konwn that $K_2 \gg K_1$. Therefore, Eq. (2) is the dominate reaction. The IEP shifts slightly to pH 4 after adding TMAH. The absolute value

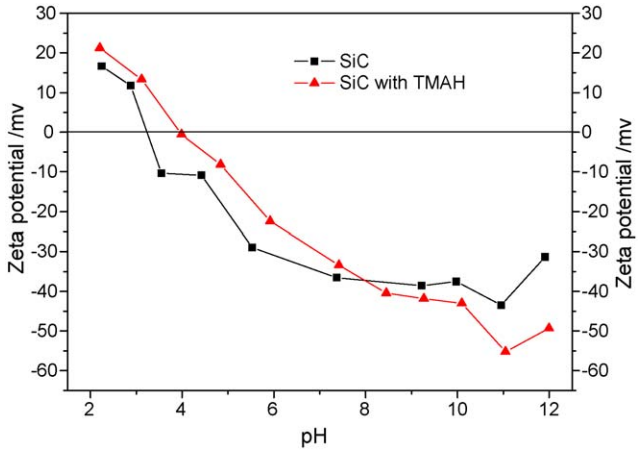

Fig. 1. Zeta potential of SiC powder with and without TMAH.

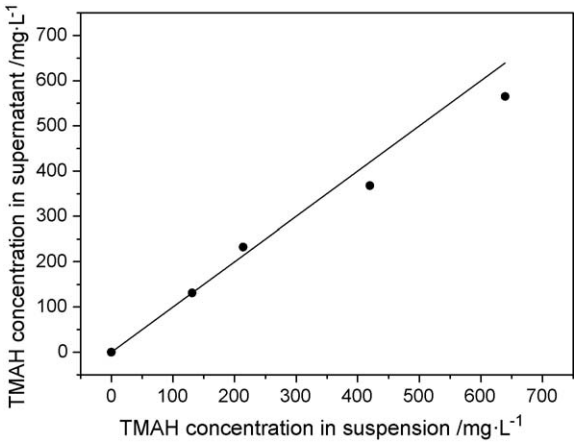

Fig. 2. Adsorption amount of TMAH on the surface of SiC with different concentrations of TMAH (pH = 10.5).

of zeta potential of SiC powder increases a little from pH 7 to pH 12 with the addition of TMAH, indicating that TMAH works mainly by adjusting the pH value of suspension, not the adsorption like other polymer or polyelectrolyte dispersants [26,27].

3.2. Adsorption of TMAH

Fig. 2 shows the residual TMAH concentration in supernatant at different initial concentration of TMAH. The straight line in Fig. 2 represents no adsorption state, i.e. the residual TMAH concentration in supernatant is equal to the initial TMAH concentration. The dots represent the test values in the measurement. It can be seen that all the dots are very close to the beeline, which means that there is no adsorption of TMAH on the surface of SiC. Therefore, the stabilization mechanism of TMAH to SiC suspensions is mainly electrostatic repulsion. The result is in consistent with the zeta potential of SiC (Fig. 1).

3.3. Sedimentation study

The stability of suspensions in the absence and presence of dispersant is evaluated from the change of sediment volume as a function of pH and time. As shown in Fig. 3(a), in the absence of TMAH, the lowest sediment for the SiC suspension occurs in the pH range of 8–11, and the sediment increases as time prolonged. The suspensions display a very high settling volume at pH < 7, which is due to the low surface charge of SiC particles (see Fig. 1). The highest sediment of SiC suspension occurs at pH ≈ 3, which is in agreement with the IEP. From Fig. 3(b), in the presence of TMAH, the stability of SiC suspensions can be achieved in the pH = 10–12 range.

3.4. Effect of TMAH content

It was difficult to disperse SiC powder in aqueous media without adding TMAH, and the maximum solid content is less than 35 vol.%. After adding TMAH, SiC slurry with the solid content higher than 50 vol.% can be achieved. Fig. 4 shows the effect of TMAH content (based on SiC powder) on the viscosity of 50 vol.% SiC slurries. The pH values of the slurries are in the range of 10–11.7. As shown in Fig. 4, the slurry containing 0.3 wt.% TMAH solution shows a shear thickening behavior at high shear rate (about 500 s^{-1}). With the increase in TMAH, the shear thickening behavior disappears, which indicates that TMAH is effective to improve the fluidity of concentrated SiC slurry. It can be seen that the slurry with relative lower viscosity

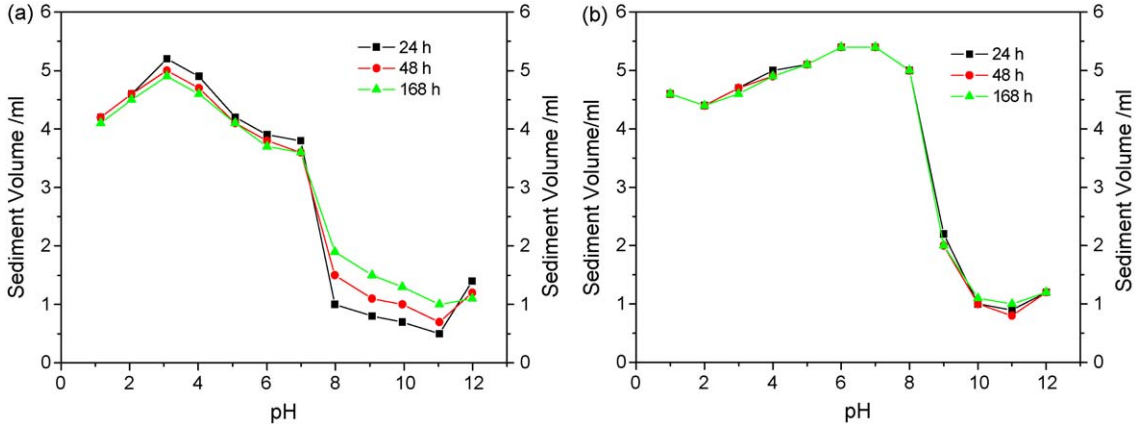

Fig. 3. Sediment volume versus pH value: (a) with TMAH and (b) without TMAH.

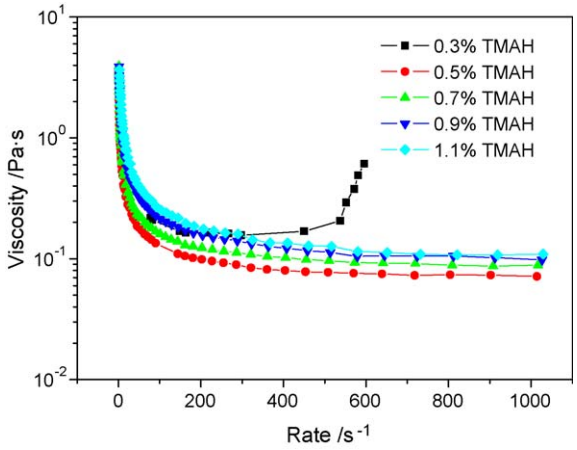

Fig. 4. Effect of TMAH content on the viscosity of 50 vol.% SiC slurry.

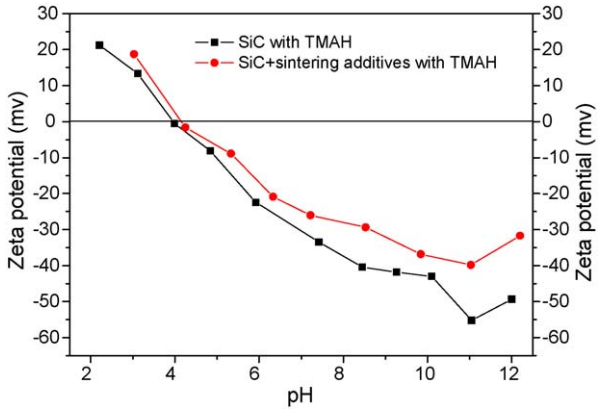

Fig. 6. Zeta potential of SiC with and without sintering additives in the presence of TMAH.

is obtained with 0.5 wt.% of TMAH solution. It can be explained that with the adding of TMAH, the pH value of slurry increases and the electrostatic repulsion between SiC particles increases. When the TMAH content is higher than 0.5 wt.%, though the slurries still show shear thinning behavior, the viscosity and shear stress showed an increase trend with the increase of TMAH amount. At strong basic pH range, the silica on the surface of SiC particles and the impurities will dissolve [23,28], so the concentration of electrolyte ions will increase, which might lead to the decrease in electrostatic force and the increase in slurry viscosity.

3.5. Influence of solid loading

In tape casting, high solid loading is required so that green tapes have high green density. However, a suspension with enough fluidity is required to ensure that green tapes have uniform structure and few defects such as bubbles and cracks. Therefore, it is important to obtain high solid loading slurry with good fluidity. The influence of solid loading on the flow behavior of SiC slurry after adding 0.5 wt.% TMAH solution is shown in Fig. 5. It can be seen that the slurries containing 48 and 50 vol.% of SiC powder show shear thinning behavior, which is suitable for tape casting. The shear stress increases approximately linearly with the increasing of shear rate. With the solid loading increasing to 52 or 54 vol.%, the slurry immediately shows a shear thickening behavior and the shear stress increases sharply at a shear rate of about 1 s^{-1}, indicating that SiC particles in these slurries are in agglomerated state. When the solid loading is above 54 vol.%, the slurry is too viscous to measure its rheology behavior. In our experiments, 50 vol.% SiC slurries are used for further tape casting process.

3.6. Effect of sintering additives

To obtain dense SiC ceramics, alumina and yttria were selected as sintering additives [29,30] for liquid phase sintering. The amount of the sintering additives is 9 wt.%, based on the total ceramic powders. The IEP of SiC, Al_2O_3 and Y_2O_3 is at pH 3.2, 7.8 and 8.1, respectively. This difference in pH_{IEP} can be presumably ascribable to the difference in surface charge properties. Zhang et al. [31] improved the co-dispersion of SiC and sintering additives with PEI and citric acid as dispersants. Fig. 6 shows the zeta potential of SiC with and without sintering additives in the presence of TMAH. The addition of sintering additives did not show obvious effect on the pH_{IEP} of SiC

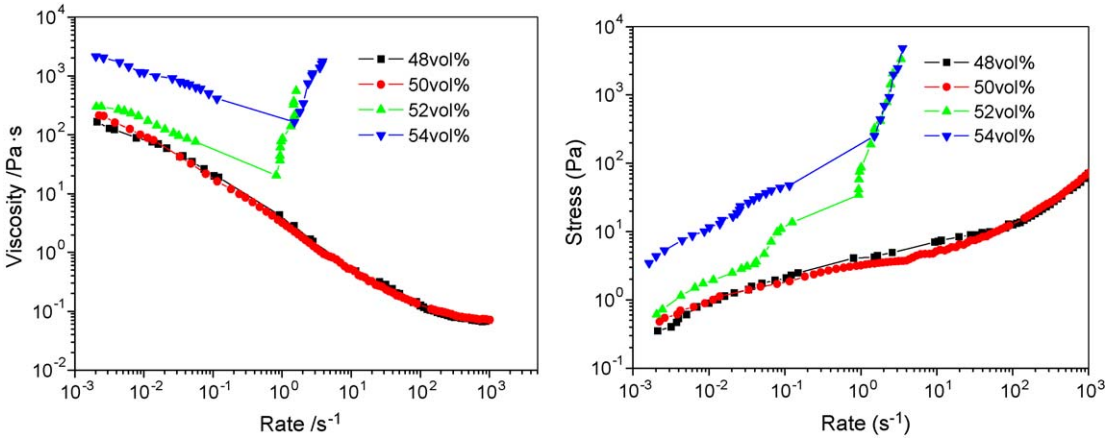

Fig. 5. Rheological behavior of SiC slurries with different solid loadings.

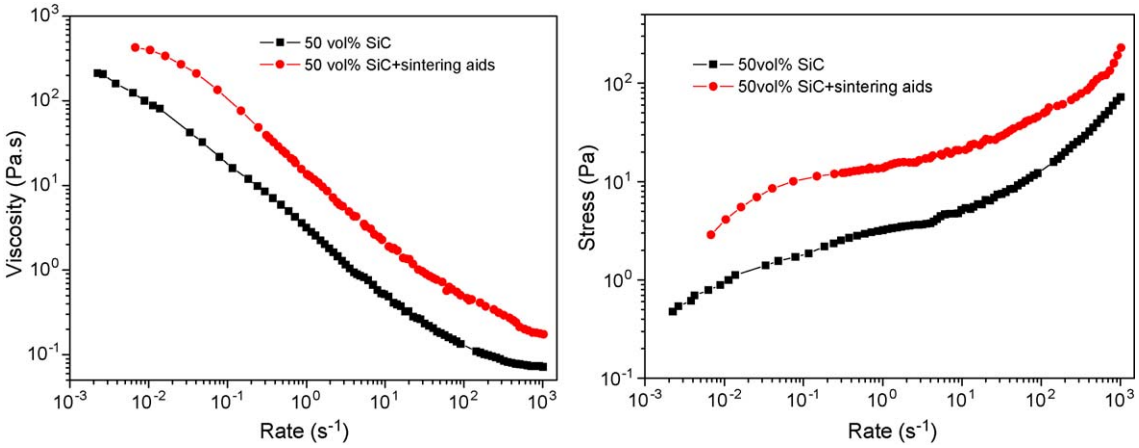

Fig. 7. Rheological behavior of 50 vol.% SiC slurries with and without sintering additives.

particles, due to their relatively small amount. But a decrease in the absolute value of zeta potential of SiC at pH > pH_{IEP} was observed, owing to their different surface properties. However, the zeta potential of mixture powders is still high enough (−40 mV at pH 11) for electrostatic force to take effect.

After the addition of sintering additives, the stability of SiC slurry was also characterized through rheological measurement (see Fig. 7). It can be seen that after adding sintering additives, the SiC slurry still shows shear thinning behavior, and the viscosity and shear stress is just a little higher than that of the SiC slurry without sintering additives. At the shear rate of about 100 s^{-1}, after adding sintering additives, the viscosity of slurry increases from 0.13 to 0.47 Pa s, which is still suitable for tape casting. Based on the zeta potential and rheological results, it can be concluded that TMAH is beneficial to the co-dispersion of SiC powder and the sintering additives.

3.7. Influence of binder/powder ratio (X)

Binders are added to slurries in order to enhance the strength of the green tape for easy handling and storage. The binder remains in the tape, forming organic interparticle bridge, resulting in a strong adhesion after solvent evaporation. PVA with the hydrolysation degree of 87–89% (PVA 1788) was a commonly used binder in aqueous tape casting for its good compatibility with the aqueous suspensions and effective at low concentration [13,14]. However, the tape casting slurries with PVA 1788 cannot spread on the hydrophobic mylar. In our study, PVA with the hydrolysation degree of 50% (PVA 1750) was selected as the binder. The slurry can spread well on the mylar after casting. The influence of binder/powder ratio (X) was studied (Table 1). Fig. 8 shows strength curve of green tapes with increasing binder content. It can be seen that the slurry viscosity and tensile strength increase with increasing X ratio ranging from 0.07 to 0.10. On the premise of forming good tape, the amount of binder in the slurry is required as little as possible. So $X = 0.08$ is selected in this study.

Table 1
Slurry viscosity and tensile strength of green tapes at various X ratios.

X ratio (binder/powder)	Slurry viscosity (Pa s)	Tensile strength (MPa)
0.07	1.9	Tape cracks
0.08	2.1	2.29 ± 0.26
0.09	2.5	2.67 ± 0.19
0.10	2.75	2.76 ± 0.09

3.8. Influence of plasticizer/binder ratio (Y)

Plasticizers are added to the slurry to confer sufficient flexibility to the green tape for easy handling and storage. The most important effect of the plasticizer is to reduce the binder T_g at room temperatures or less [32]. This allows working at temperatures higher than T_g and, therefore, leads to better conditions of plasticity. Glycerine was the most-commonly used plasticizer for PVA [13]. In our study, we selected 1,3-propylene glycol as plasticizer because it showed good wettability with the surface of mylar. In order to study the influence of plasticizer/binder ratio (Y), powder/binder ratio (X) was fixed to be 0.08. The amounts of binder and plasticizer were adjusted to yield plasticizer/binder ratios ranging from 1.3 to 1.6 (Table 2 and Fig. 9). The results show that in the range of Y tested, the slurry viscosity and tensile strength decrease with increasing Y ratio. This behavior

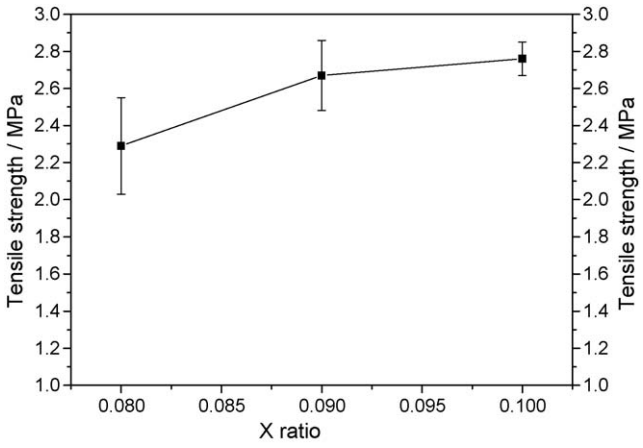

Fig. 8. Tensile strength of green tapes for various X ratios.

Table 2
Slurry viscosity and tensile strength of green tape at various Y ratios.

Y ratio (plasticizer/binder)	Slurry viscosity (Pa s)	Tensile strength (MPa)
1.3	2.3	Brittle
1.4	2.1	2.29 ± 0.26
1.5	2.0	1.94 ± 0.02
1.6	1.8	1.77 ± 0.09

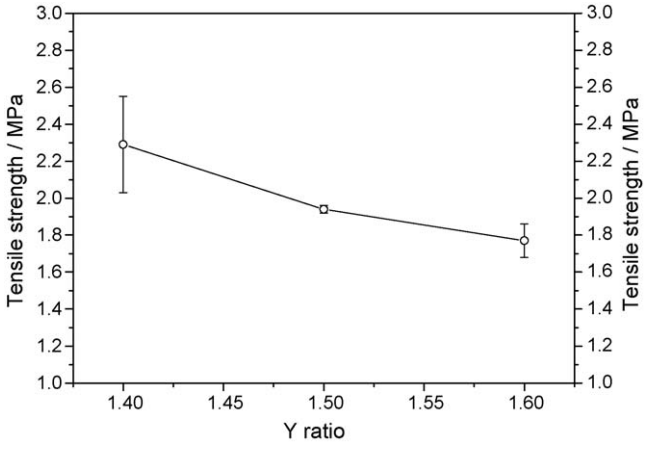

Fig. 9. Tensile strength of green tapes for various Y ratios.

Table 3
Properties of SiC ($X = 0.08$ and $Y = 1.4$) sintered ceramic.

Property	Value
Relative density (%)	98.89 ± 0.32
Flexural strength (MPa)	779.5 ± 39.2
Hardness (GPa)	21.51 ± 0.70
Toughness (MPa $m^{1/2}$)	5.54 ± 0.26

indicates that the PVA binder exhibits higher strength when unplasticized and has a brittle behavior. Plasticizer reduces the glass transition temperature of the polymer and the viscosity of the suspension. The low molecular weight species of the plasticizer increases the flexibility of the green tape, but at the expense of the tensile strength. So $Y = 1.4$ is selected in this study.

3.9. Microstructure and mechanical properties of sintered ceramic

The mechanical properties of sintered SiC were shown in Table 3. Fig. 10 displays the SEM micrographs of the sintered SiC samples. A fine microstructure with few pores is found in the sintered samples. The average grain size of SiC is about 0.8 μm. Based on SEM observation, the fracture mode is mainly intergranular type.

4. Conclusions

Stable SiC slurry with 50 vol.% solid loading was prepared using 0.5 wt.% TMAH solution as the dispersant. The SiC powder and sintering additives, Al_2O_3 and Y_2O_3, can be co-dispersed in tape casting slurries. The slurry showed shear thinning behavior, which is suitable for tape casting. The amounts of binder and plasticizer were decided by detailed viscosity analysis and tensile measurement. The binder/powder ratio X and the plasticizer/binder ratio Y was determined to be 0.08 and 1.4, respectively. SiC green sheet with smooth surface and uniform microstructure was prepared after tape casting and drying. After de-binding and hot-pressing, well-densified SiC ceramics were obtained and the flexural strength, hardness, and toughness are 779.5 ± 39.2 MPa, 21.51 ± 0.70 GPa and 5.54 ± 0.26 MPa $m^{1/2}$, respectively. The facture surface of SiC samples showed a fine and uniform microstructure. The sample exhibited predominantly intergranular fracture type.

Acknowledgements

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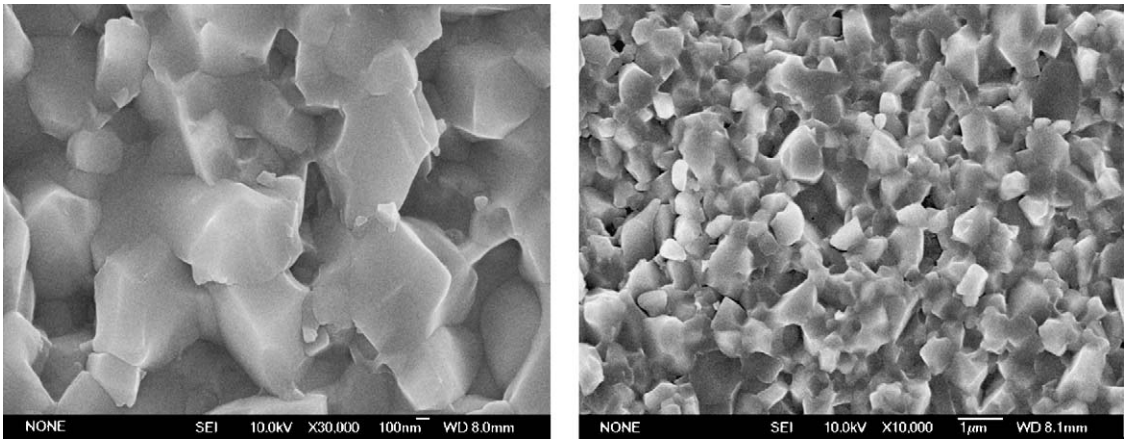

Fig. 10. SEM micrographs of fracture surface of SiC sintered body.

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