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Preparation of silicon carbide ceramic slurry for stereolithography-based additive manufacturing

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Abstract

Stereolithography-based additive manufacturing of ceramics has received tremendous attention in academic and industrial communities. In order to fabricate silicon carbide (SiC) components with complex shapes by stereolithography, a high SiC loaded ceramic slurry with low viscosity and excellent curing ability is required. However, it is challenging to meet these slurry requirements. In this work, the effects of resin formulation, dispersant, particle size, solid content and ball milling time on the properties of SiC ceramic slurry were systematically studied. The SiC slurries were prepared by using four precursor SiC powders having different particle sizes and modified by high temperature oxidation to reduce its UV absorbance. Additionally, the suitable SiC slurries for stereolithography were prepared by ball milling under appropriate processing parameters, and the geometrically complex SiC green bodies were subsequently fabricated.

Keywords: *stereolithography, silicon carbide, additive manufacturing, properties*

I. Introduction

SiC ceramics is an important structural material owing to its excellent chemical stability, high strength, wear resistance and thermal conductivity [1,2]. It has been widely used in the field of aerospace [3,4], nuclear [5,6], chemical industries [7,8], etc. The traditional manufacturing methods of SiC ceramics include dry pressing, gel casting, slip casting, etc. [9,10]. However, SiC parts with highly complex geometries are impossible to produce through those methods as moulds are usually involved in traditional techniques. Moreover, post machining such as milling and drilling is almost always required, resulting in an high cost of operations and a damage to the sample. Hence, it is highly needed to find a novel ceramic forming method.

Additive manufacturing, or 3D printing, is a process of making objects by adding materials layer by layer, instead of removing materials. It allows the creation of ceramic parts with complex and customized shapes, whilst also minimizing the amount of expensive machining required [11,12]. There are several ceramic 3D printing methods, such as selective laser sintering (SLS) [13,14], direct ink writing (DIW) [15,16], laminated object manufacturing (LOM) [17,18], fused deposition modeling (FDM) [19,20] and stereolithography (SLA) [21,22]. Among them, stereolithography is considered as one of the most popular ceramic 3D printing technique due to its high printing accuracy and surface quality [23,24]. Stereolithography can be divided into SLA and digital

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light processing (DLP), the latter stands out due to its relatively fast build speed compared to traditional SLA process [25]. The ceramic slurry used in DLP process must have high solid loading, low viscosity and sufficient curing thickness to achieve dense parts [26,27]. To date, the photosensitive slurries with required properties for oxide ceramics such as ZrO_2 and Al_2O_3 have been widely studied. However, for grey or dark non-oxide ceramics such as SiC, it is difficult to process it due to its high optical absorption and high refractive index, which reduce the curing ability of ceramic slurry. To the best of our knowledge, the systematic investigation of SiC has been limited.

This paper studies the effects of different factors on SiC ceramic slurry. The purpose of this study is to prepare high solid content SiC ceramic slurry with good dispersibility and excellent curing ability. Based on the DLP, the SiC ceramic powder is mixed with the photocurable resin, and the suitable process and method are used to prepare the SiC ceramic slurry. The effects of prepolymer, diluent, dispersant, particle size, powder modification, solid content and ball milling time on the properties of SiC ceramic slurry were studied in detail. Finally, SiC ceramic green body with complex shape was successfully prepared by DLP process.

II. Experimental

2.1. Raw materials

Four precursor SiC powders with average particle sizes of 1, 5, 10 and 15 μm were selected from Shandong Weifang Kaihua Silicon carbide Micro Powder Co. Ltd. Three kinds of prepolymers were used for the preparation of photosensitive resin, including: bis trimethylol propane acrylate (Di-TMPTA; Refractive index: 1.474; Jiangsu Changshu Hengyao New Materials Co. Ltd.), polyester acrylate oligomers (7210; Guangdong Jiangmen Xinghan Trading Co. Ltd.) and polyurethane acrylate (U600; Shanghai Guangyi Chemical Co. Ltd.). Two kinds of diluents, 1,6-hexadiol diacrylate (HDDA; Refractive index: 1.457; Shanghai Guangyi Chemical Co. Ltd.) and tripropylene glycol diacrylate (TPGDA; Jiangsu Kailin Ruiyang Chemical Co. Ltd.) were used to prepare the photosensitive resin. Diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide (TPO; Shanghai Guangyi Chemical Co. Ltd.) was used as a photoinitiator. KOS110 (Guangzhou Kang'ou Shuang Trade Co. Ltd.), TX-100 (Sinopharma Chemical Reagents Co. Ltd.), PVP (K16-18, China Shanghai Alighting Biochemical Technology Co. Ltd.) and BYK-103 (Guangzhou Guangyuan New Materials Co. Ltd.) were selected as dispersants.

2.2. Preparation of SiC ceramic slurry

Figure 1 shows the flow chart of SiC ceramic slurry preparation. Firstly, the corresponding prepolymer, diluent and photoinitiator (TPO) with weight ratio of 70:30:2 were mixed under continuous stirring to prepare the photosensitive resin. At the same time, the precursor SiC powder was oxidized slightly at 900 °C, forming silicon dioxide layer on the surface of particles. The modified SiC powder and 3 wt.% dispersant were incrementally blended into the photosensitive resin to produce a slurry with solid content of 70 wt.%. Finally, the SiC ceramic slurry was obtained by ball milling for 3 h in the planetary ball mill (YXQM-2L, Changsha Mitr Instrument Equipment Co. Ltd). The ball milling used zirconia balls at a speed of 150 r/min.

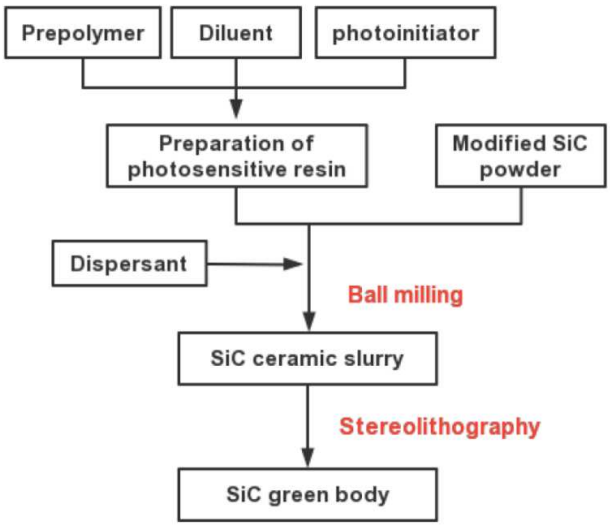

Figure 1. Flow chart of SiC ceramic slurry preparation for stereolithography

2.3. Stereolithography process

A commercial DLP 3D printer (405 nm, Shenzhen Dazu Laser Technology Co. Ltd, China) was used to fabricate SiC parts. For all builds, the layer thickness was set at 30 μm and exposure parameters were optimized to 50 mW/cm^2 and 15 s. The SiC ceramic slurry was poured into the slurry tank and evenly coated on the bottom surface of transparent film by a scraper. The slurry was solidified to form a single layer. Then the printing platform was moved upwards, and the tank rotated to lay a new layer of slurry. This process is repeated until the entire SiC green body is completely obtained.

2.4. Characterization

Viscosities of the resin and ceramic slurries were measured by digital display rotational viscometer (Shanghai Fongrui Instrument Co. Ltd, China) at room temperature. The rotor was immersed in the resin and slurries, and the rotor speed was adjusted to 6, 12, 30 or 60 r/min. Unless otherwise specified, viscosity was measured at a rotor speed of 60 r/min and the viscosity test results were recorded by rotating the rotor for 30 s. Sedimentation tests were used to estimate the stability of the ceramic slurry. The SiC ceramic slurries were put into a 50 ml centrifugal tube, and the settlement ratio was calculated by the static settlement method [28]. After a certain period, the initial height H and the height

h after settling were recorded to characterize the stability of the slurry. Note that the higher the value of h/H, the higher the stability of slurry. To investigate the curing thickness, a series of single layer parts were fabricated using a DLP printer with 405 nm UV light. The spiral micrometer (Dongguan Sanliang Precision Measuring Instrument Co. Ltd, China) was used to measure the single layer curing thickness. The slurry was coated on the release film and illuminated with a single exposure. The measured thickness of the solidified layer was the curing thickness of the slurry. Ultraviolet spectrometer (Shimadzu Company, Japan) was used to investigate the absorbance value of SiC powder under the wavelength of 250 to 550 nm.

III. Results and discussion

3.1. Effects of prepolymer and diluent

To investigate the effects of the different resin constituents (i.e. prepolymers Di-TMPTA, 7210 and U600) on rheological behaviour and curing ability, the same diluent was added in each experiment. The viscosity measurement results are shown in Table 1. The slurry with Di-TMPTA was found to have the lowest viscosity and better rheological properties than the slurries with 7210 and U600. The resin polymers are Newtonian fluids, so viscosity measurements vary little at different rotor speeds. For investigating the effects of the prepolymers on curing ability of SiC ceramic slurry, three slurries with the same composition except prepolymer were prepared. The prepolymer and diluent were evenly mixed with a magnetic stirrer, and the monolayer curing thickness of the polymer was measured using a DLP printer, as shown in Table 2. U600 was found to have the best curing ability. However, it offered the highest viscosity and it was not beneficial to prepare high SiC loaded slurry. Di-TMPTA with low viscosity and excellent curing ability was therefore selected as the prepolymer.

In the photocuring resin system, diluents acted as a monomer that could participate in the photocuring crosslinking reaction, and its low viscosity characteristics could significantly reduce the viscosity of photocured resin. In this paper, two reagents with strong diluent ability were selected, i.e. HDDA and TPGDA. The designed experiment is shown in Table 3. It is noted that HDDA's dilution ability is stronger than TPGDA and thus the former is selected as the diluent in this work. Based on the above studies, the resin was formulated by mixing the prepolymer and diluent at a weight ratio of 70:30 with addition of 2 wt.% TPO.

3.2. Optimization of dispersant

Dispersant plays an important role in the preparation of SiC slurry with high solid content and low viscosity. Four dispersants (KOS110, TX-100, PVP and BYK-103) were used in this work. The amount of each dispersant was designed to be 1, 3, 5 and 7 wt.% with respect to SiC powder mass. The photocured resin as described in Section 3.1 and the same SiC powder with average particle size of 10 μm were used for each set of experiments. The solid content of the slurry in each experimental group was 50 wt.%.

Figure 2 shows the effects of dispersants on viscosity of the SiC ceramic slurries. It can be seen that the slurries with KOS110 and BYK-103 show the shear thinning behaviour and the lowest viscosity, and dispersant concentrations have little effect on the viscosity. BYK-103 is somewhat better than KOS110 in reducing viscosity. In contrast, PVP and TX-100 increase the viscosity with increasing concentrations. In general, when the amount of the dispersant added in slurry is too small,

Table 1. Viscosity of resins with different prepolymers

Group	Viscosity [mPa·s]			
	6 r/min	12 r/min	30 r/min	60 r/min
70% Di-TMPTA + 30% HDDA	69.4	68.2	73.7	72.5
70% 7210 + 30% HDDA	124	121	136	129
70% U600 + 30% HDDA	968	957	960	967

Table 2. Curing thickness of the SiC slurries with different prepolymers

Group	Dispersant (PVP) [wt.%]*	Solid content [wt.%]	Ball milling [h]	Curing thickness [μm]
70% Di-TMPTA + 30% HDDA	2	30	4	133
70% 7210 + 30% HDDA	2	30	4	106
70% U600 + 30% HDDA	2	30	4	235

* Relative to SiC powder mass

Table 3. Viscosity of resins with different diluents

Group	Viscosity [mPa·s]			
	6 r/min	12 r/min	30 r/min	60 r/min
70% Di-TMPTA + 30% HDDA +	69.4	68.2	73.7	72.5
70% Di-TMPTA + 30% TPGDA +	144	142	145	144
100% Di-TMPTA	833	825	814	775

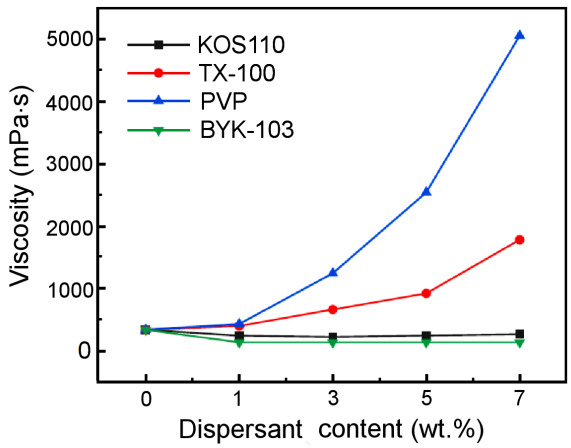

Figure 2. Viscosity of the SiC slurries with different dispersants

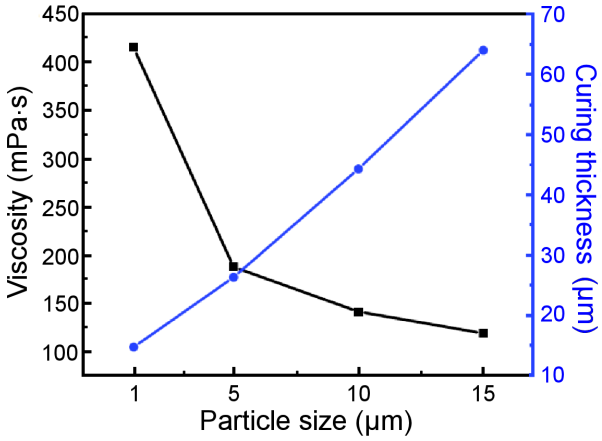

Figure 3. Viscosity and curing thickness of slurries with different size of SiC particle

dispersant cannot be fully absorbed on the surface of all SiC particles. Non-modified particles tend to agglomerate, resulting in uneven dispersion and high viscosity of slurry. When the additive amount increases, it should be beneficial for more SiC particles to form an organic protective film on the surface to overcome the attractive force. In this case, the SiC particles are homogeneously dispersed and reduce viscosity of the slurry. When the concentration of dispersant is further increased, excessive dispersant will cause the flocculation of the SiC particles and increase viscosity of the slurry. For preparing SiC ceramic slurry with high solid content, it is necessary to select optimal dispersant which can effectively reduce the viscosity. In this work, 3 wt.% BYK-103 was selected for further research.

3.3. Effects of particle size

The SiC powders with average particle sizes of 1, 5, 10 and 15 μm were selected to study the effects of particle size on slurry properties. The SiC ceramic slurry was prepared under the same conditions and its viscosity, stability and curing thickness were studied. The amounts of TPO photoinitiator and BYK-103 dispersant were 2 and 3 wt.% (with respect to SiC powder mass), respectively, and the solid content of the slurry in each experiment was 50 wt.%. Figure 3 shows the measurement results of viscosity and curing thickness. As the size of SiC powder increases, the viscosity of the slurry decreases and the curing thickness increases. Viscosities of the slurries prepared from the SiC powder with average particle size of 1, 5, 10 and 15 μm are 415, 188, 141 and 119 mPa·s, respectively. The reason might be that the smaller the size of SiC powder, the larger the specific surface area of ceramic particles and the larger the surface atomic number and surface energy are. This will cause adsorption capacity of SiC particles to increase, and agglomeration between particles is more likely to occur. These reasons lead to the increase of the viscosity of SiC ceramic slurry with small particle size. In addition, the ceramic slurry with high viscosity has low curing ability and small curing thickness. The curing thicknesses of the slurries prepared by the SiC powder with average particle sizes of 1, 5, 10 and 15 μm are 15, 26, 44 and 64 μm, respectively. The high specific surface area will increase the absorbance value of the SiC powder, so that the UV light radiated by the DLP equipment cannot be fully absorbed by the resin. Therefore, the curing thickness of SiC ceramic slurry with small particle size will be lower.

The stability of slurry prepared from the SiC powders with different particle sizes is shown in Fig. 4. It can be seen from the settlement ratio that large SiC particles will decrease the stability of slurry. The slurry with small size is not easy to settle due to its poor fluidity. However, the slurry particles with large size have a greater tendency of downward settlement, resulting in poor slurry stability.

Generally speaking, the larger the particle size of SiC powder, the poorer the stability of the slurry prepared under the same solid content, leading to a low viscosity and an enhancement in curing thickness. Therefore, the larger the particle size of SiC powder is more beneficial to the preparation of high solid content of SiC ceramic slurry for stereolithography. However, the strength and density of SiC formed with large particles tend to decrease after sintering. This section studies the viscosity, curing thickness and stability of SiC slurries with different particle sizes, providing reference for researchers to select particle sizes. In summary, 10 μm SiC powder was used in this work.

3.4. Oxidation modification of SiC powder

In the process of stereolithography, each layer of a slurry is cured and bonded together. The principle of slurry curing is that the photoinitiator in the layer of slurry is activated by the photons in the irradiated UV light to produce free radicals. Then, these free radicals initiate polymerization of the resin monomer. As light intensity increases, the suspension reaches a gel point where solidification occurs. When light hits the slurry surface the intensity decreases with depth, meaning that the slurry receives different light energy at each depth.

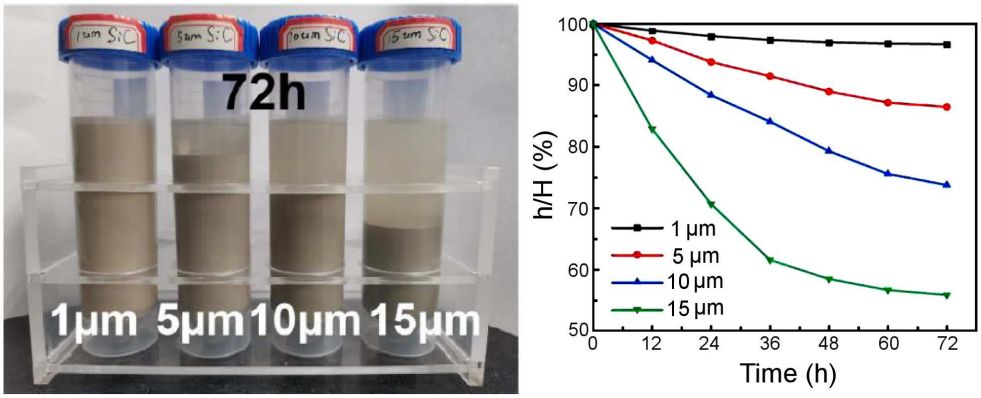

Figure 4. Effects of SiC particle size on slurry stability

The curing thickness is the depth at which light causes the degree of polymerization to reach the gel point. In other words, the depth at which the gel point occurs is the thickness of cure. The amount of energy to reach this depth is the critical energy. The curing thickness is a key parameter to determine whether the ceramic slurry can be formed by stereolithography. The curing thickness is affected by resin sensitivity, light energy and critical energy, as shown by the Jacob's Beer-Lambert law [29]:

$$C_d = D_p \cdot \ln \frac{E}{E_c} \tag{1}$$

where C_d is absolute curing thickness, D_p is resin sensitivity, E and E_c are light energy and critical energy, respectively.

Among them, the sensitivity D_p of photosensitive resin depends on the solid content of slurry and ceramic particles size. At the same time, it is related to the refractive index of photosensitive resin and ceramic powder. The ceramic particles in the slurry scatter some of the light. When the refractive index difference between resin and ceramic particles is large, the scattering of resin particles also increases significantly. Griffith [30] gave the formula for their relationship:

$$D_p = \frac{2}{3}\frac{d}{Q}\frac{1}{\Phi}\left(\frac{n_0}{\Delta n}\right)^2 \tag{2}$$

where d is ceramic powder particle size, Q is scattering efficiency value, Φ is volume fraction of ceramics, n_0 is photosensitive resin refractive index, Δn is refractive index difference ($\Delta n = n_{ceramic} - n_0$).

It can be seen from Eq. 2 that the effect of scattering on resin sensitivity is dominant. For the slurry system of ceramic material with high refractive index, the resin sensitivity D_p will depend on the refractive index of photosensitive resin n_0 and the difference Δn between it and the refractive index of ceramic powder. On the contrary, in the case of small refractive index difference between ceramic material and resin, D_p will be mainly affected by particle size and solid content [31]. As a kind of powder with high optical absorbance and refractive index, the refractive index difference between SiC and resin is large (SiC: 2.6, resin: <1.5) [32]. According to the Griffith formula, D_p will be greatly reduced, resulting in the reduction of curing thickness C_d. In addition, SiC has a high absorption rate of 405 nm UV light used in the stereolithography moulding experiment, resulting in less light absorbed by the photoinitiator, which also affects the curing thickness of the SiC ceramic slurry.

In order to reduce the absorption of the precursor SiC powders at 405 nm UV light, high temperature oxidation modification of powder was adopted. The powders were slightly oxidized at a certain temperature by placing them in a high temperature furnace. SiO_2 layer is formed on the surface of the SiC particles, which reduces the absorption of UV light at 405 nm. The UV absorption value of the SiC powders at different oxidation temperatures is shown in Fig. 5. It can be seen that as the temperature increases from 800 to 1100 °C, the UV absorption value of the SiC powder decreases. For reducing the absorbance of SiC powder without generating too much SiO_2, in this work we used oxidizing process at 900 °C for 30 min at a heating rate of 5 °C/min.

3.5. *Effects of solid content*

High solid loading is beneficial to fabricate highly dense and crack-free parts, therefore it is necessary to improve the solid content of slurry. The effects of dif-

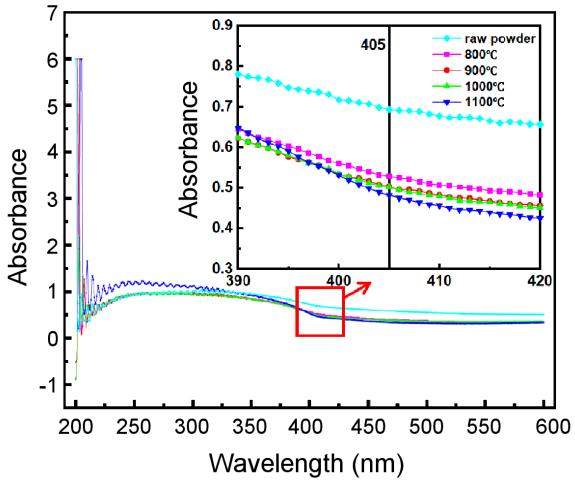

Figure 5. Effect of different oxidation temperature on the absorbance value of SiC powder

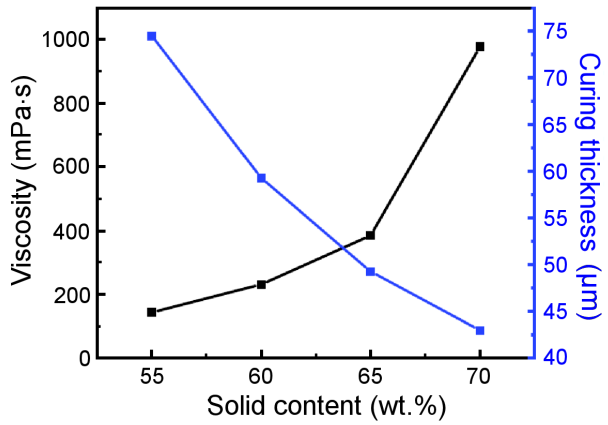

Figure 6. Viscosity and curing thickness of SiC slurries with different solid content

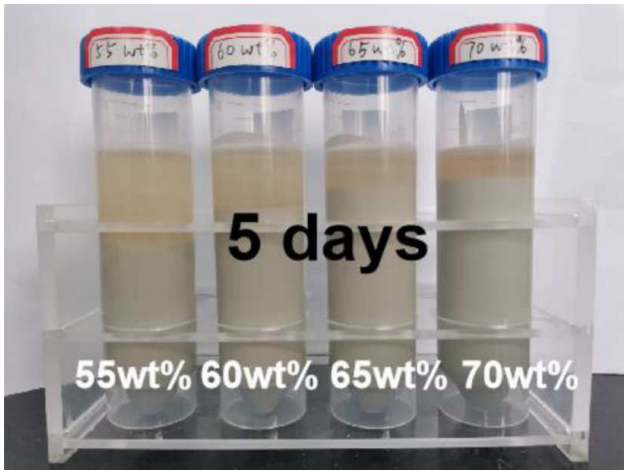

Figure 7. Effect of solid content on the stability of SiC slurries

ferent solid content on the SiC ceramic slurry properties were studied, and relationships between viscosity/curing thickness and solid content were presented in Fig. 6. With the increase of solid content, the viscosity of the SiC ceramic slurry increases and the curing thickness decreases accordingly. The viscosities of the SiC ceramic slurries with solid content of 55, 60, 65 and 70 wt.% were 144, 232, 385 and 977 mPa·s, respectively. The curing thicknesses were 74.5, 59.3, 49.3 and 43 μm, respectively. When the number of SiC powder particles increases, the fluidity of SiC ceramic slurry decreases and the viscosity increases. UV light in the slurry will be difficult to transfer, resulting in reduced curing thickness. Figure 7 shows the stability of SiC ceramic slurry with different solid content. With the increase of solid content, the slurry tends to be more stable. The slurry with large solid content has poor fluidity and the particles are not easy to settle.

In order to ensure strong bonding between interlayers, the cured thickness must be greater than layer thickness of 30 μm as set by DLP equipment. The SiC ceramic slurry with different solid content above all meets the layer thickness requirements. The slurry with viscosity less than 3000 mPa·s is suitable for stereolithography [33]. The preparation of SiC ceramic slurry with high solid content also improves the stability of the slurry. Based on the discussion of experimental results, the optimal solid content of the SiC ceramic slurry was determined to be 70 wt.%.

3.6. Effects of ball milling time

The SiC ceramic slurry was mixed after the composition and dosage of various substances are fixed. The following ball milling process is the key step to make the SiC ceramic slurry evenly dispersed and reduce the viscosity. It has been reported that the viscosity of slurry cannot be reduced indefinitely, and the photoinitiator and dispersant in ceramic slurry may also be affected by ball milling time. Therefore, it is necessary to study the effects of ball milling time on the viscosity and curing thickness of SiC ceramic slurry to determine the best ball milling time. For the SiC ceramic slurry with solid content of 70 wt.% prepared in Section 3.5, the experimental study was designed for different ball milling time of 1 to 6 h.

Viscosity and curing thickness of the SiC ceramic slurries with different ball milling time are shown in Fig. 8. With the increase of ball milling time, viscosity of the SiC ceramic slurry decreases at first and then increases. When the ball mill starts, the slurry can be dispersed evenly, continuously reducing the viscosity of the slurry. As the milling time continues to increase, the dispersant in the slurry will be destroyed, resulting in the increase of slurry viscosity. The curing thickness will decrease with the increase of ball milling time. The reason is that the particle size of the SiC powder will be reduced by ball milling, and the photoinitiator in the slurry may also be destroyed, leading to the reduction of curing thickness. Generally speaking, the curing thickness should be 10 μm larger than the layer thickness set by DLP equipment. The curing thickness of the slurry is 43 μm after 3 h ball milling, while the curing thickness of the slurry decreases to 35.3 μm after 4 h ball milling, which is not conducive to the bonding between layers

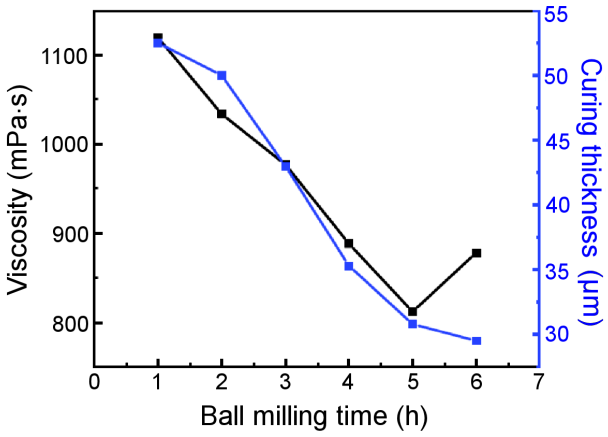

Figure 8. Viscosity and curing thickness of SiC slurries with different ball milling time

during stereolithography. In order to reduce the viscosity of SiC ceramic slurry and disperse the slurry evenly, the ball milling time was set to 3 h in the experiment on the basis that the solidified thickness of the slurry could meet the layer thickness requirements of stereolithography. After the SiC ceramic slurry was obtained by ball milling, the SiC green bodies with complex shapes were prepared by stereolithography, as shown in Fig. 9.

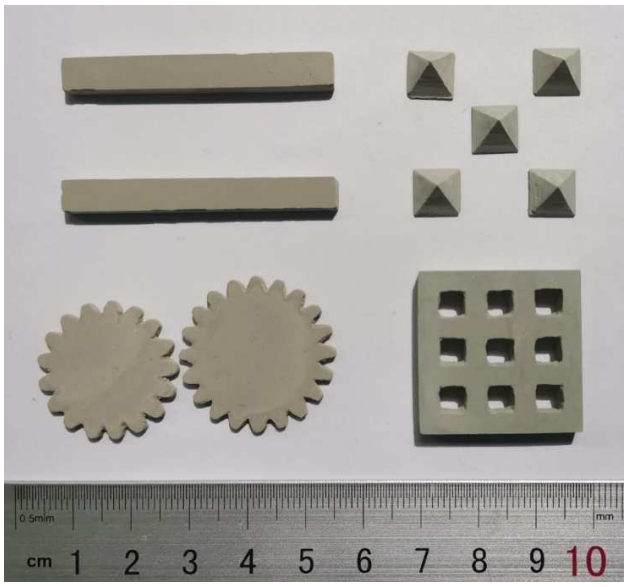

Figure 9. Green SiC ceramics with complex shapes fabricated by stereolithography

IV. Conclusions

The effects of resin formulation, dispersant, particle size, solid content and ball milling time on the properties of SiC ceramic slurries were systematically studied. It was shown that complex SiC parts could be successfully fabricated using the suitable ceramic slurry.

Moreover, the high temperature oxidation method was used to modify SiC powder to reduce its UV absorbance, which is more conducive to stereolithography. Di-TMPTA and HDDA were determined as prepolymer and diluent respectively. 3 wt.% BYK-103 was considered as the best dispersant to prepare SiC ceramic slurry. Furthermore, the SiC ceramic slurry with solid content of 70 wt.% was prepared based on investigating the effects of SiC powder particle size, solid content and ball milling time on the properties of SiC ceramic slurry. The slurry shows shear thinning behaviour and high solid content, which can be used to prepare SiC green bodies with good properties. This study aims to provide a comprehensive study on ceramic stereolithography process, which can be extended to fabricate other ceramics such as silicon nitride.

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