

Reaction-bonded SiC derived from resin precursors by Stereolithography

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Abstract

A photo-curable resin with a high carbon yield after pyrolysis was developed in the present research. It consisted of phenolic epoxy acrylate resin, phenolic resin, triethylene glycol as pore forming agent and benzoin dimethyl ether as photoinitiator. The well-prepared mixed resin was used by Stereolithography to form resin prototypes. The influence of mixed resin composition on the process parameters was studied to meet the requirement for the cured thickness. Carbon preforms with open porosity of 27% and bending strength of 4.48 MPa were obtained after pyrolyzing the resin prototypes. After molten silicon infiltration at the temperature 2300°C, the carbon preform converted to reaction-bonded SiC. The maximum bending strength of the produced SiC samples was 127.8 ± 0.5 MPa as the pore forming agent content was 40 wt.%. Neither residual carbon nor silicon remained in the reaction-bonded SiC sample according to the XRD analysis.

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1. Introduction

Silicon carbide ceramic has attracted many researchers' attention as an advanced ceramic because of its superior mechanical properties, wear resistance, and corrosion resistance in the high temperature conditions. However, shaping of silicon carbide components is very difficult and expensive because of the mentioned properties. Lack of net-shaping capability and high process temperature are the bottleneck of the low-cost engineering applications of silicon carbide. Reaction-bonded SiC (RBSC) by using molten silicon infiltration technique is a lower temperature fabrication process, which has been extensively studied by many researchers. The mixture of furfural resin and furfural alcohol was used to produce porous carbon preforms after pyrolysis and then be infiltrated with molten silicon [1,2]. RBSC components had also been fabricated via infiltrating the molten silicon into the machined porous oak charcoal [3–7]. The reactive kinetics of molten silicon infiltration into the porous medium was studied in some literatures [8–10]. In these conventional fabrication processes, the porous carbon preforms have been produced by machining natural wood or casting the

liquid mixtures into a mould previously machined. But the moulds or the preforms with complex outer and inner fine structures could not be machined or could be machined only with high cost and low efficiency. So these RFSC processes still have a bottleneck-mould or preform fabrication.

Rapid prototyping is a new fabrication method which can build three-dimensional parts with complex outer and inner structures directly from a computer aided design (CAD) model by adding materials layer by layer instead of removing materials like the conventional fabrication process. Ceramic slurries have been employed in the Stereolithography (SL) [11] and Selective Laser Sintering (SLS) process [12,13] to produce ceramic components. Friedel and Travitzky [14] firstly combined the SLS process with the molten silicon infiltration process to fabricate the ceramic parts. The components manufactured by this process have a rough surface because of the low fabrication resolution of the SLS process. Stereolithography (SL) has higher fabrication accuracy than SLS and specially fit for the fabrication of components with complex structures.

In the present work, a high carbon yield and photo-curable resin was developed for the SL process to fabricate resin precursors which can be pyrolyzed and converted to porous carbon preforms. RBSC components were derived from the carbon preforms after molten silicon infiltration. In order to

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achieve proper carbon yield after pyrolysis and suitable viscosity for the Stereolithography process, the composition of the resin mixture was investigated. Pore forming agent was added into the resin mixture to produce porous microstructure of the carbon preform which is positive to the molten silicon infiltration process. Process parameters were optimized according to the thickness of cured single layer. The influence of the composition of the resin mixture on the microstructure and mechanical properties of the produced RBSC components was also studied in the present research. By comparison with the conventional preparation process of carbon preforms, the process in the present research possessed the advantages of rapid, near-net shaping characteristics in the preparation of the carbon preforms. Some RBSC samples were fabricated to demonstrate the feasibility of the fabrication process in this study.

2. Experimental procedure and characterization

The phenolic epoxy acrylate resin (SWANCOR (Shanghai) fine chemical CO., LTD., China) and the phenolic resin (Xi'an resins factory, Xi'an, China) were mixed to prepare the high carbon yield system. Pore forming agent-triethylene glycol (Shanghai Experiment Reagent Co., Ltd., China) was used to form the porous microstructure of the carbon preform. Finally, the photoinitiator-benzoin dimethyl ether was provided by Hongtai Chemical Engineering Co., Ltd, Jingjiang, China. The viscosity of the mixture was detected by using the viscometer (NDJ-79, Shanghai Precision & Scientific Instrument Co, Ltd, China). The well-prepared resin mixture was used on the Stereolithography rapid prototyping machine (SPS-600, Shannxi Hengtong Intelligent Machine Co., Ltd, China) by using UV laser with wavelength of 350 nm, power of 200 μ W, hatch spacing of 0.1 mm, and the scan speed from 50 mm/s to 8000 mm/s. The fabricated resin prototypes were pyrolyzed at 850 $^{\circ}$ C for 1 h to obtain the carbon preforms in the homemade furnace with Ar gas protection. The carbon preform was infiltrated by the molten silicon (Latitude Co., Ltd., Dongguan, China) in the self-made high temperature infiltration furnace with maximum temperature of over 1410 $^{\circ}$ C for 30 min.

Standard testing samples of 3 mm, 4 mm, and 40 mm in height, width, and length, respectively were fabricated for the characterization. Weight loss of the resin samples in the

pyrolysis process was conducted on the thermal gravimetric analyzer (NETZSCH STA449C, Germany). The density of the carbon preform was measured by using Archimedes's method. The microstructure of carbon preform and RBSC samples were observed by using SEM (DJM-840, JEOL, Japan). The phase composition was analyzed by XRD (D/max2400, Japan). The mechanical strength of the carbon preforms and the RBSC samples were measured by using three-point fracture method on MicoTester1195, Instron.

3. Results and discussion

3.1. High-carbon yield resin prototypes

3.1.1. High-carbon yield photo-curable resin

The fabrication process proposed in the present research required a special photo-curable resin which possesses a high-carbon yield after pyrolysis. The shape and inner structure of the carbon preforms could be inherited from the resin prototypes due to the high-carbon yield. However, the carbon yield of the common photo-curable resin was normally less than 18%. It is not high enough to realize the fabrication process of the reaction-bonded SiC components. It is found that most photo-curable resins have a low carbon yield, but high-carbon yield resin normally cannot be photo-polymerized. In the present research, different resin mixtures were studied in order to obtain these two kinds of properties simultaneously. Finally, phenolic resin with a high carbon yield of about 60% was chosen to be added into the photo-curable phenolic epoxy acrylate (EA) resin to produce a resin system with both high-carbon yield and photo-curable properties. Phenolic epoxy EA has a relative higher carbon yield of 18% than that of normal photo-curable resins (1–5%). Their structural formulas are shown in Fig. 1. The phenolic structure can be found in the phenolic EA in addition to the high-carbon yield double bond structure, as shown in Fig. 1(b). Due to the structural similarity of phenolic and phenolic EA resins, the mixed resin system was stable even after 48 h deposition. There was no phase separation or delamination being observed in the mixture, as shown in Fig. 2.

The viscosity of the mixed resin is important in the rapid prototyping process. The temperature and holding time are two critical factors which affect the viscosities of the mixed resin. In

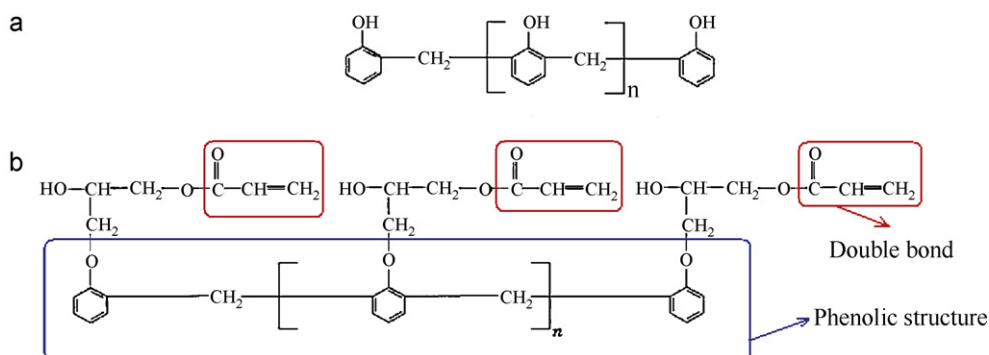


Fig. 1. Structural formulas of the phenolic resin and phenolic epoxy acrylate resin.

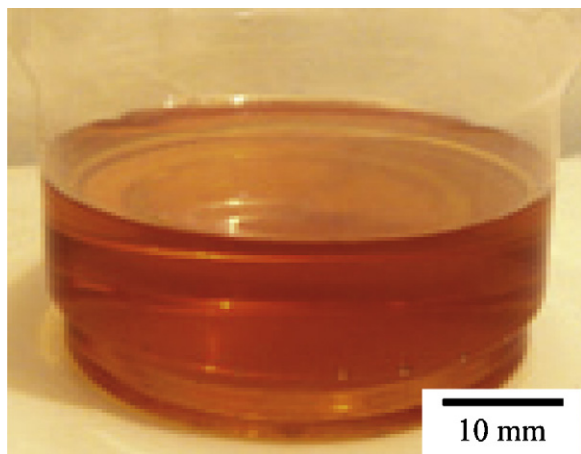


Fig. 2. Stable resin mixture of phenolic and phenolic EA resins after 48 h.

order to investigate their influence on the viscosity, a mixed resin with 58.2 wt.% thermosetting phenolic resin, 38.2 wt.% phenolic epoxy acrylate resin, and 3 wt.% photo initiator (benzoin dimethyl ether) were prepared in the present research. The relationship between the temperature of mixed resin and its viscosity are shown in Fig. 3. The viscosity decreased with an increasing temperature from 27 °C to 90 °C. In the rapid prototyping process, the viscosity can be controlled according to changing the temperature of the resin.

However, if the temperature was too high, the resin would be more viscous with a longer holding time. The resin mixtures were held in the thermostatic water bath for 45 h at 30 °C, 60 °C, 90 °C, respectively. The viscosities of the mixed resin were measured by using the viscometer at different interval, as show in Fig. 4. The mixture resin held in the 30 °C water bath has a relative stable viscosity during the holding period. But at the temperature of 60 °C and 90 °C, viscosity of the mixed resin increased dramatically after holding in the water bath more than 35 h. It is because that the mixed resin used in the present research included thermosetting phenolic resin. After long time holding at the temperature higher than 60 °C, the phenolic resin was thermo-set partially and the viscosity increased. So, the

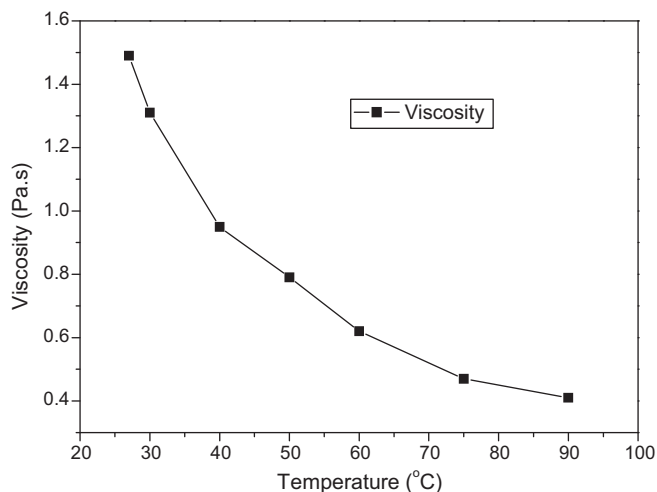


Fig. 3. Influence of temperature on the viscosity of the mixed resin.

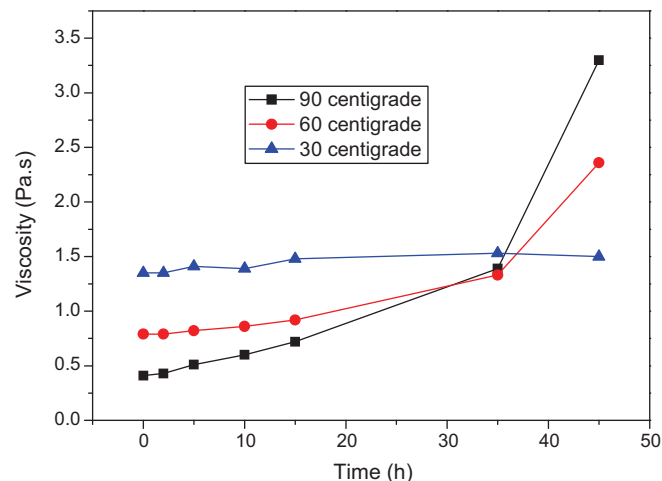


Fig. 4. Influence of holding time on the viscosity of the mixed resin at different temperature levels.

mixed resin should be kept at the temperature lower than 30 °C to be sure that the viscosity is stable in the whole fabrication process.

3.1.2. Pore forming agent

Besides the carbon yield, the porous microstructure is also required for the carbon preforms to realize the melt silicon infiltration. However, the microstructure of carbon preforms from pure resins is always dense instead of porous, such as those of phenolic and phenolic EA resins as shown in Fig. 5. In order to achieve the porous microstructure, some pore forming agent should be added into the mixed resin.

In the present research, a pore forming agent, triethylene glycol, was added into the resin mixture, which consisted of 60 wt.% phenolic EA and 40 wt.% phenolic resin. The patterns of mixed resin with different triethylene glycol content (20 wt.%, 40 wt.% and 50 wt.%) are shown in Fig. 6. The colors of the mixed resins with varying triethylene glycol content were different. The inconsistent refraction indexes of the mixed resins and the pore forming agent probably caused the opaque mixture system. However, it was proved by experiments that these mixed systems can be cured by the UV laser in the rapid prototyping process. The influence of the content of the pore forming agent on the photo-cured layer thickness and the microstructure of the carbon preforms will be discussed in the following sections.

3.1.3. Single layer thickness of the cured resin by UV laser

The thickness of the cured single resin layer is very important in the laser scanning process. Enough thickness is required to be sure that there is no delamination appeared in cured resin prototypes. The contents of phenolic resin, pore forming agent as well as photoinitiator were investigated in the present research to obtain proper composition of the photo-curable resin with high carbon yield.

The influence of phenolic resin content on the thickness of cured layer is shown in Fig. 7. There was no pore forming agent in the mixed resin. With increasing phenolic resin content, the

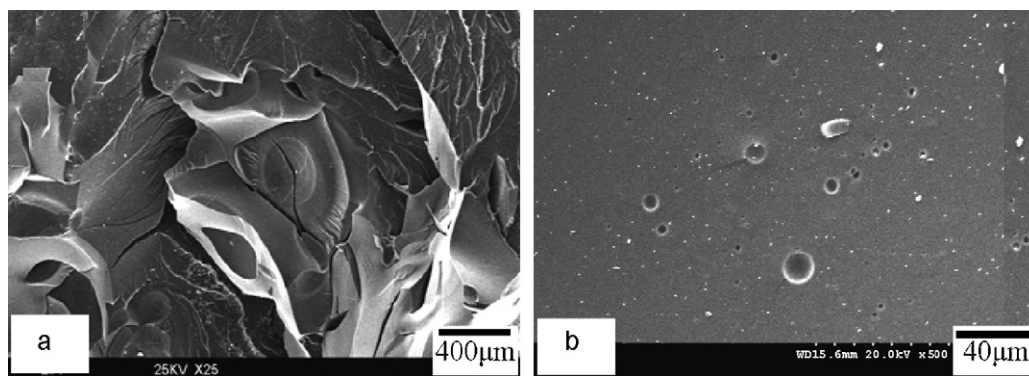


Fig. 5. SEM microstructures of the carbon preforms after the pyrolysis of (a) phenolic resin, (b) phenolic EA resin.

cured layer thickness decreased. Low scan speed can improve the thickness of cured layer. The thickness with scan speed of 50 mm/s was about twice larger than that of the layer with scan speed of 200 mm/s. When phenolic resin included in the mixed resin exceeded 65 wt.%, the cured samples were not hard enough to keep the designed shape during the post handling processes. It was because that the phenolic resin could not be polymerized in the laser scanning process. The produced samples comprised phenolic EA skeleton and the liquid phenolic resin. Too much phenolic resin would destroy the strength of the phenolic EA skeleton and caused deformation of the produced samples. So, the content of the phenolic resin should be limited to less than 65 wt.% in the mixed system. The minimal cured thickness with scan speed of 200 mm/s was

280 μm, which was still larger than the required layer thickness of 100 μm.

The content of pore forming agent, triethylene glycol, also had a significant influence on the thickness of the single cured resin layer, as shown in Fig. 8. Phenolic EA of 58.2 wt.%, phenolic resin of 38.8 wt.% as well as photo initiator 3 wt.% constituted the original mixture for this experiment. Different percentages of the pore forming agent were added into this mixture to observe their influence on the thickness of the cured resin layer. With an increasing triethylene glycol, the thickness decreased remarkably to 480 μm from the maximum value of 1100 μm. The laser scan speed used in this experiment is 50 mm/s. However, if the triethylene glycol content exceeded 50 wt.% in this mixture with phenolic EA of 58.2 wt.%, the

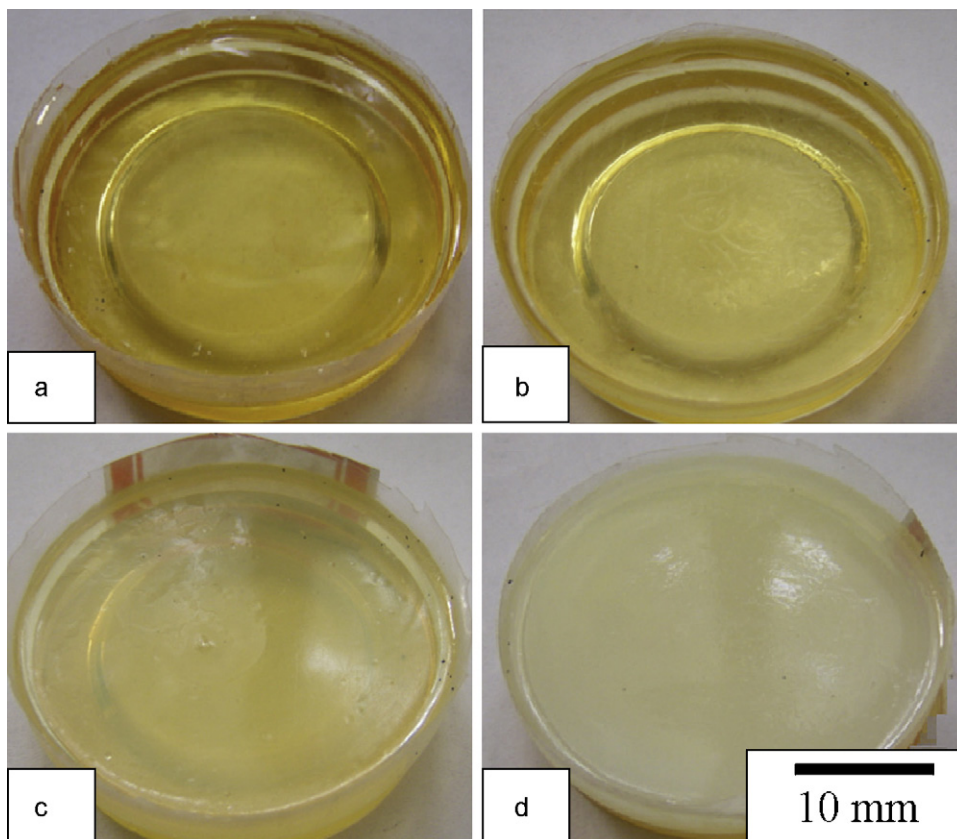


Fig. 6. Pictures of the mixed resin with (a) 0 wt.%; (b) 20 wt.%; (c) 40 wt.%; (d) 50 wt.% triethylene glycol, respectively.

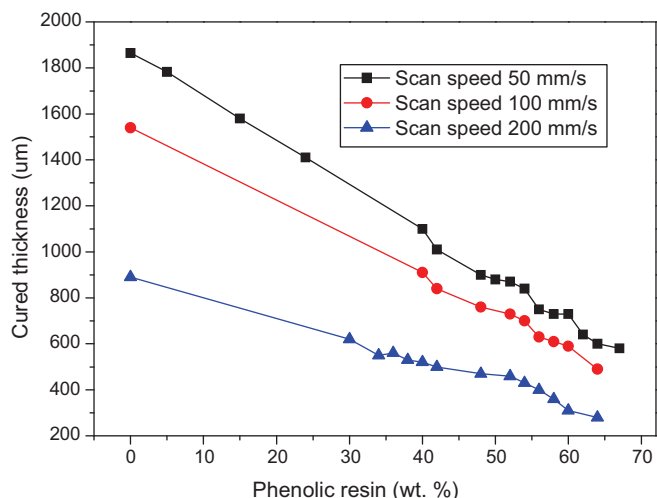


Fig. 7. Influence of phenolic resin content on the thickness of photo-cured resin layer with different scan speeds.

cured samples lost their mechanical strength. So, for this resin composition, the pore forming agent should be less than 50%. Its influence on the microstructure of the produced carbon preforms will be discussed later.

Photoinitiator is a prerequisite composition of the photo-curable resin, which can absorb laser irradiation energy and decompose into reactive intermediates. The reactive intermediates will react with the monomer and prepolymer in the mixed resin. And then, the polymerization of resin happens. The photoinitiator used in the present research was benzoin dimethyl ether. The content of photoinitiator had a significant influence on the thickness of one single cured layer, as shown in Fig. 9. There was no pore forming agent added in the mixed resin in the present experiment. When the photoinitiator was less than 1 wt.%, the increasing content improved the cured thickness. However, the thickness of the cured layer kept a relative constant with an increasing photoinitiator larger than 1 wt.%. So, the photoinitiator of 1 wt.% was used in the present research. At the same photoinitiator level, the phenolic resin was disadvantageous to the cured thickness, but the corners of

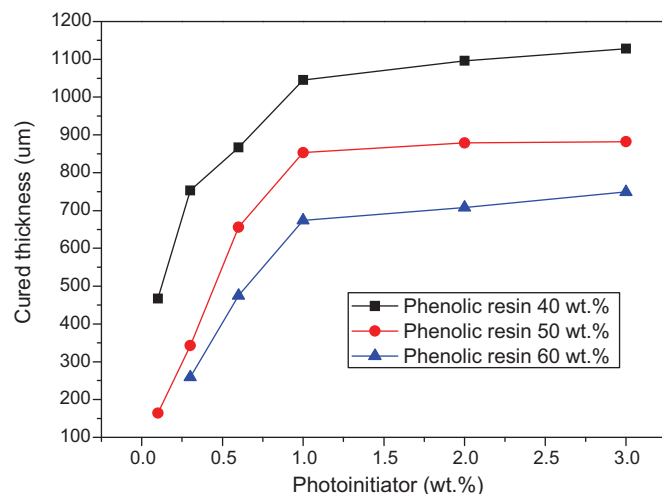


Fig. 9. Influence of photoinitiator content on the thickness of photo-cured resin layer with different phenolic resin content.

different curves in Fig. 9 appeared at the same photoinitiator content of 1 wt.%. It means that the phenolic resin content has no significant influence on the dosage of the photoinitiator, which is just related with the phenolic EA. A similar result can be derived that the content of the pore forming agent will not affect the dosage of the photoinitiator.

3.1.4. Post treatment

After the Stereolithography, the phenolic EA resin was polymerized by UV laser scanning. But the phenolic resin in the mixed resin remained as liquid in the prototypes. The liquid phenolic resin decreased the mechanical strength and cause deformation of the produced prototypes. So, the post treatment process was necessary to solidify the liquid resin and then improve the hardness and mechanical strength of the prototypes. The phenolic resin used in the present research could be solidified by thermosetting reaction. There were two stages in the solidifying reactions for the phenolic resin. At the beginning of the heating process during the temperature from 90 °C to 170 °C, the length of the molecular chains increased. When the temperature was higher than 170 °C, cross linking reaction happened between the molecular chains. At the temperature of 200 °C the cross linking reaction completed and the resultant of the reaction was insoluble and infusible. So in the present research the resin prototypes were firstly heated to 200 °C with a heating rate of 2 °C/h and then held for 3 h to finish the reactions. Two resin prototypes were made by Stereolithography on the rapid prototyping machine using the mixed resin discussed previously, as shown in Fig. 10.

3.2. Pyrolysis process and properties of carbon preforms

3.2.1. Pyrolysis process

Thermogravimetric (TG) analysis is very helpful to establish the pyrolysis process of the resin prototypes in order to avoid the cracks and deformation in the carbon preforms. As shown in Fig. 11(a), the weight loss curve was measured by using the resin sample which was made from the mixed resin consisting

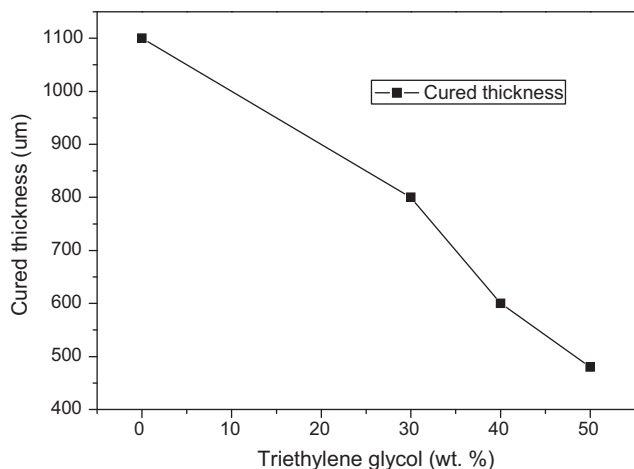


Fig. 8. Influence of triethylene glycol content on the thickness of photo-cured resin layer.

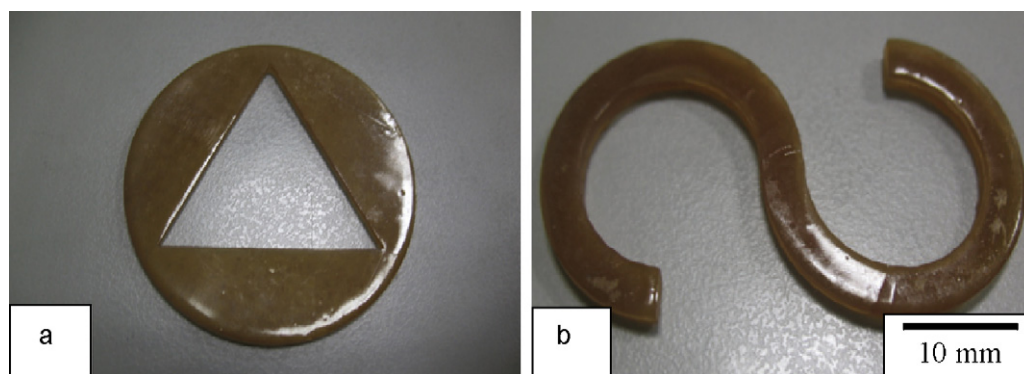


Fig. 10. Two resin prototypes made by Stereolithography on rapid prototyping machine.

of 47.5 wt.% phenolic resin, 31.7 wt.% phenolic EA resin, 19.8 wt.% pore forming agent, and 1% photoinitiator. There were three stages in the pyrolysis process of the resin sample. The preheating stage was from the room temperature to 300 °C. No burning happened to the resin. Almost nothing was lost in the resin body. As the temperature was increasing up to 600 °C, the weight loss of the resin sample increased dramatically due to the release of small molecule compounds, such as H₂O, CO, CO₂, CH₄, and H₂. When the temperature was higher than 600 °C, the weight loss slowed down because there were just some molecules with strong chemical bonds decomposing, for instance, the dehydrogenation reaction of the benzene rings.

According to the TGA result, the pyrolysis process was established, as shown in Fig. 11(b). The inset in Fig. 11(b) is the carbon preform of the resin prototype in Fig. 10(b) after pyrolysis. The preform had a good shape preserving.

3.2.2. Properties of the carbon preforms

The carbon preforms were obtained after pyrolysis of the resin prototypes at 900 °C according to the heating process shown in Fig. 11(b). Its properties, carbon yield, shrinkage, and bulk density were affected by the composition of the mixed resin. The influence of phenolic resin and pore forming agent (triethylene glycol) on the properties of carbon preforms were investigated. The relationships between phenolic resin content and carbon yield, shrinkage, and bulk density are shown in Fig. 12. The carbon yield and bulk density increased with

increasing phenolic resin content in the mixed resin due to phenolic resin's higher carbon yield than that of phenolic EA resin. Meanwhile, the shrinkage of carbon preform after the pyrolysis decreased as the increasing phenolic resin content. The linear shrinkage varied between 26.2% and 30.8%. The corresponding volume shrinkage was from 60% to 67%. It was a huge obstacle to fabricate SiC components with high accuracy and should be solved in the future research. Considering the shrinkage and bulk density, more phenolic resin in the mixed resin was positive to the properties of the final carbon preform as well as the reaction-bonded SiC components.

The pore forming agent also had a significant influence on the properties of the carbon preform, as shown in Fig. 13. With an increasing content of the triethylene glycol, the carbon yield and bulk density decreased and the linear shrinkage rose. This was consistent with the microstructures of the carbon preforms in Fig. 14. The microstructure of carbon preform without pore forming agent was dense, as shown in Fig. 14(a). By adding triethylene glycol into the mixed resin, pore appeared in the carbon preform (Fig. 12(a–c)). The patterns of the microstructure were affected by the content of the pore forming agent. With 20 wt.% triethylene glycol, pores formed in the carbon preform in Fig. 14(b). More pores appeared in Fig. 14(c) when the content of triethylene glycol increased by comparison with Fig. 14(b). However, the interconnected pores just appeared with a pore form agent of 50 wt.%, as shown in Fig. 14(d). An open porosity of 23.1% was obtained by using

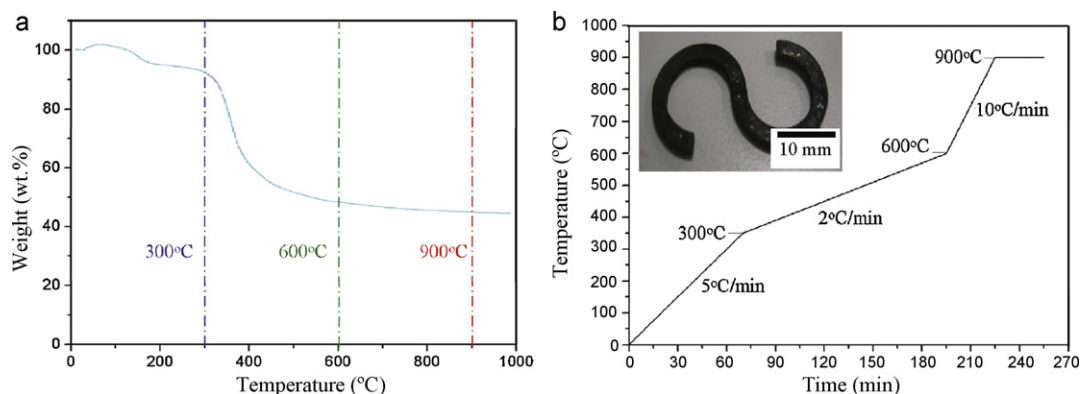


Fig. 11. TG analysis result (a) and established pyrolysis process (b) of the resin prototypes, carbon preform (inset) after pyrolysis of resin prototype shown in Fig. 10(b).

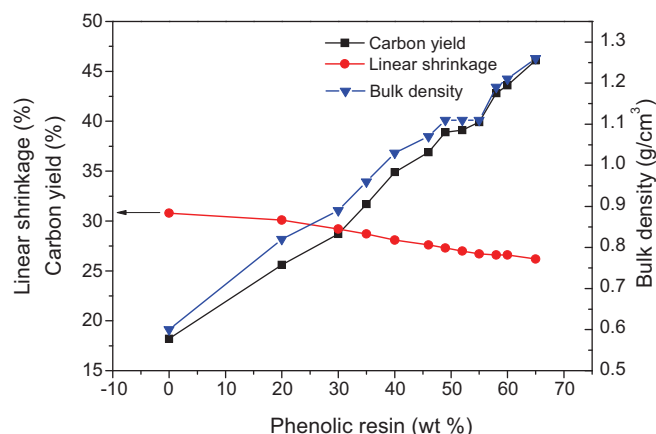


Fig. 12. The relationships between phenolic resin content and carbon yield, shrinkage as well as bulk density.

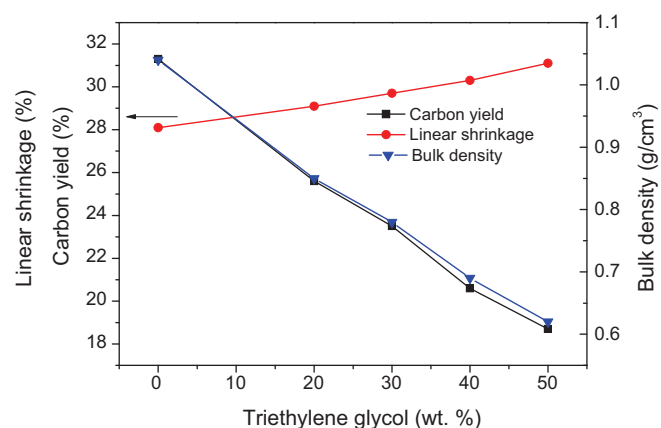


Fig. 13. Influence of pore forming agent (triethylene glycol) content on the properties of the carbon preforms.

Archimedes' method on the sample shown in Fig. 14(d). High open porosity was positive to the following silicon infiltration process. However, the fracture strength of the carbon preform from the mixed resin with 50 wt.% pore forming agent was 4.48 MPa, which was lower than that of the resin mixture without pore forming agent (7.43 MPa).

3.3. Reaction-bonded SiC samples

Two different carbon preforms were infiltrated by silicon melt at the temperature of 2300 °C. The microstructures of

SiC samples are shown in Fig. 15(b and d) corresponding to the carbon preforms shown in Fig. 15(a and b). There were no pores in the carbon preform made from mixed resin without pore forming agent, as shown in Fig. 15(a). But the melt silicon infiltration reaction still completed at the temperature of 2300 °C. SiC crystals with a diameter of 100–200 μm were found in SiC sample, as shown in Fig. 15(b). XRD pattern of the produced SiC samples proved that all the carbon reacted with silicon and converted to SiC crystals, as shown in Fig. 16. Compared with the dense microstructure in Fig. 15(a), porous carbon preform in Fig. 15(c) produced SiC

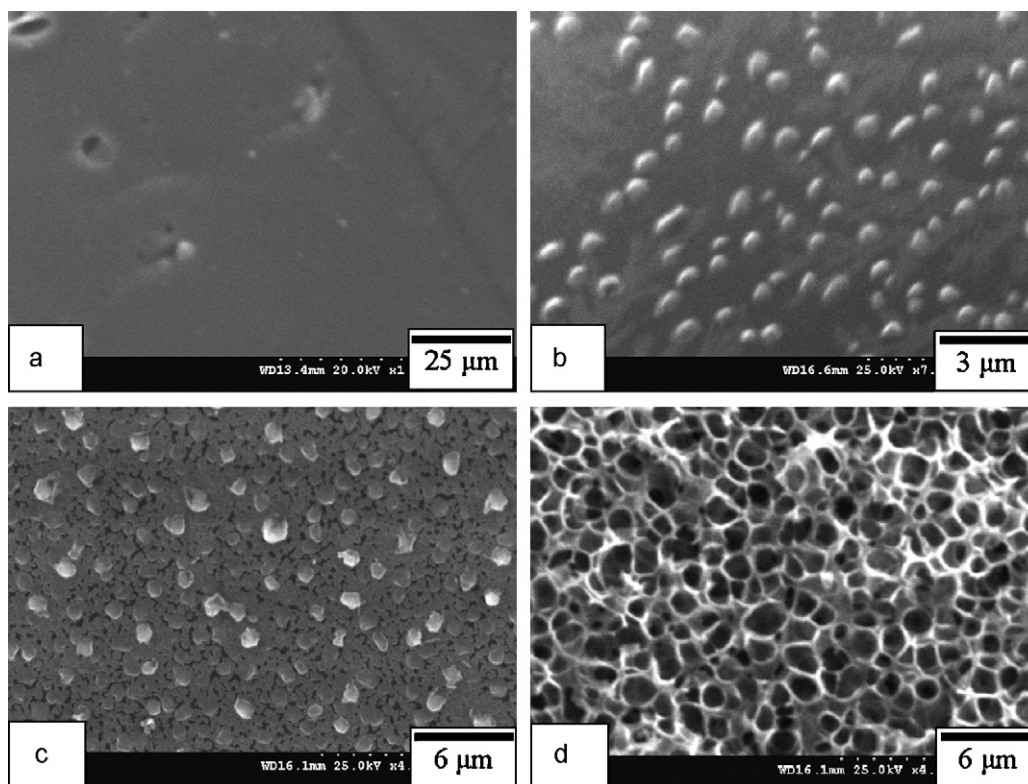


Fig. 14. Microstructures of the carbon preforms with different pore forming agent (triethylene glycol) content: (a) 0 wt.%, (b) 20 wt.%, (c) 40 wt.%, (d) 50 wt.%.

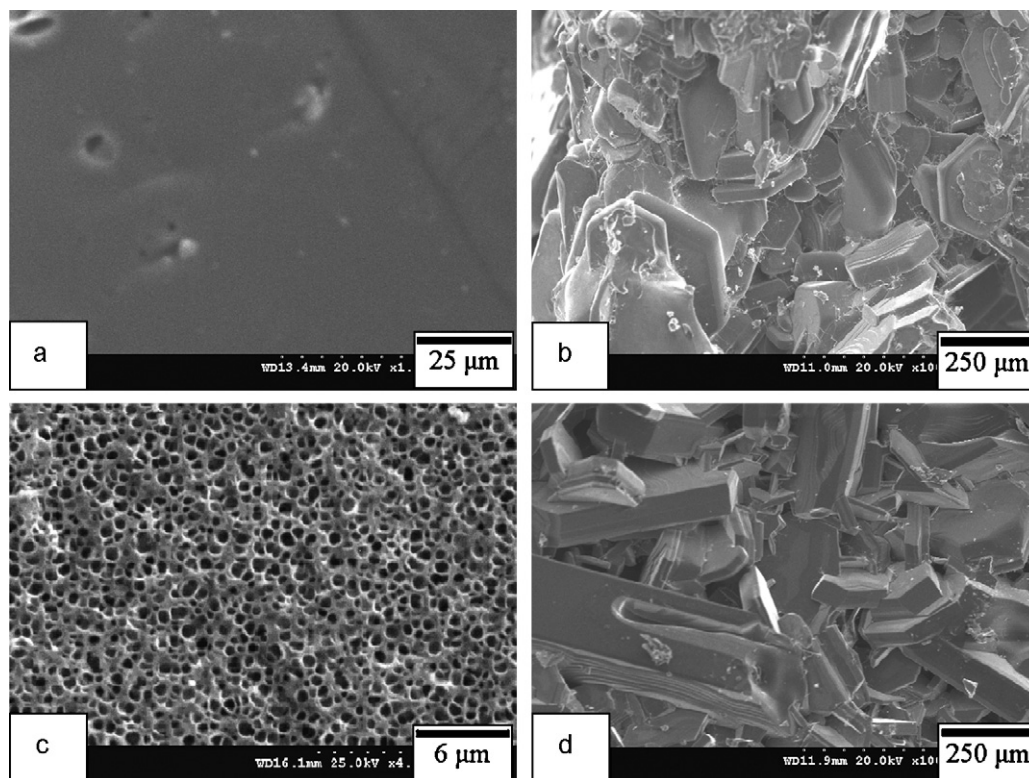


Fig. 15. Microstructures of the carbon preforms and the produced SiC samples: (a) and (b) without pore forming agent; (c) and (d) with 50 wt.% pore forming agent.

crystals with a bigger diameter of 200–500 μm , as shown in Fig. 15(d). The high infiltration temperature (2300 $^{\circ}\text{C}$) in the present research was much higher than the normal temperature of 1450 $^{\circ}\text{C}$ [15], which was a little bit higher than the melting point of silicon and used in most melt silicon infiltration reactions. This high temperature is close to the boiling point of silicon. So, the infiltration reaction in this temperature is equivalent to silicon vapor infiltration (VI). That could be the reason why relative dense carbon preform can be infiltrated by the silicon. Meanwhile, the porous microstructure provided channels for the silicon melt or vapor to permeate carbon preform conveniently. Enough time can be taken by the silicon to react with carbon and grow quickly. That is the reason why the size of SiC crystals in

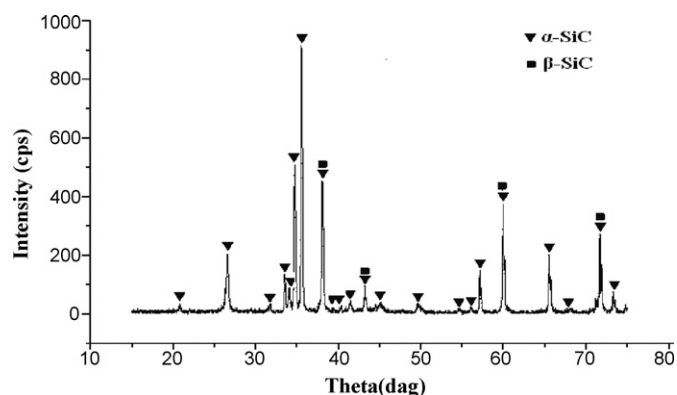


Fig. 16. XRD pattern of the produced reaction-bonded SiC.

Fig. 15(d) is much bigger than that in Fig. 15(b). The content of triethylene glycol affected the microstructures of the carbon preforms (Fig. 14) which also had an influence on the mechanical strength of the produced SiC samples, as shown in Fig. 17. The SiC sample derived from the mixed resin with 40 wt.% triethylene glycol obtained the maximum fracture strength of 127.8 ± 0.5 MPa. This fracture strength is still lower than the commercial reaction-bonded SiC products probably due to the layered manufacturing process. Further

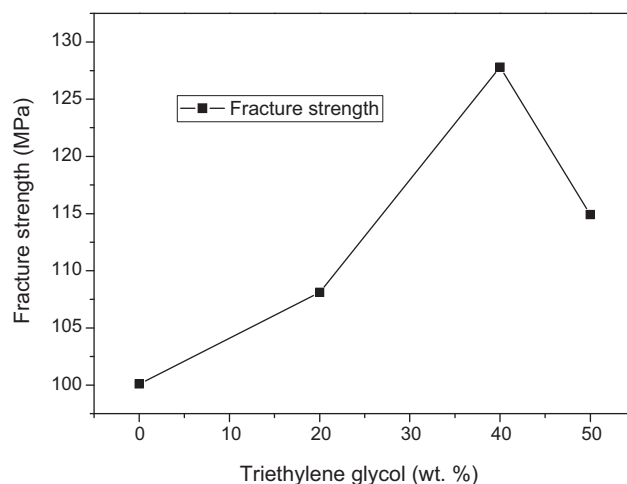


Fig. 17. Effect of pore forming agent (triethylene glycol) on the fracture strength of the reaction-bonded SiC.

investigation should be conducted to improve the mechanical strength of the SiC components.

4. Conclusions

A new fabrication process of SiC components was investigated by combining the reaction infiltration with Stereolithography. The critical factor of this fabrication process was the preparation of photo-curable resin with high carbon yield. A resin mixture of the phenolic epoxy acrylate resin, phenolic resin, pore forming agent, and photoinitiator was put forward and investigated in the present research. Stereolithography parameters were affected by the composition of the mixed resin and the environment conditions, such as temperature, and the holding time. With increasing temperature the viscosity of the mixed resin decreased. However, if the temperature was higher than 30 °C, the viscosity of the mixed resin increased dramatically after long time deposition. More phenolic resin always increased the carbon yield after pyrolysis, but decreased the thickness of cured single resin layer because it cannot be cured by the UV laser. So, the content of phenolic resin should be lower than 65 wt.% to be sure that the resin prototypes could obtain enough strength for the following handling process. Pore forming agent was used to achieve porous microstructure of the porous carbon preform. Like phenolic resin, the pore forming agent should be kept lower than 50 wt.%. The RBSC samples derived from the resin prototypes with 40 wt.% pore forming agent achieved the maximum mechanical strength of 127.8 ± 0.5 MPa. Pure SiC phase was observed in the RBSC samples and no residual carbon or silicon was detected by the XRD analysis. Some resin samples were fabricated using the Stereolithography process. Some carbon preforms and SiC samples were produced to demonstrate the feasibility of the fabrication process.

In the future, the composition of photo-curable resin should be further optimized according to the requirement of the Stereolithography. The properties of the photo-curable resin should be improved to get higher carbon yield and lower shrinkage, finally obtain SiC components with higher accuracy.

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