Investigation on Preparation and Mechanical Properties of Al_2O_3/Epoxy Resin Composites Epoxy

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Abstract: In this paper, the organic phase is penetrated into the ceramic skeleton using a vacuum impregnation process to prepare a biomimetic composite with shell pearl layer structure,which the Al_2O_3 porous ceramics preparing by freeze-drying method are inorganic phase skeleton, epoxy resin curing system as weak interface layer organic phase. The mechanical properties of the composites are improved by adjusting the ratio of the curing system. The results showed that the strength and toughness of the composites are the best when the ratio of curing is 1: 1.

1. Introduction

After millions of years of evolution, it has evolved a lot of natural biological materials which structure and function almost reached the perfect level[1-4]. Compared to two component materials, nature forms a natural biological material with ductility and higher intension, which combines natural brittle ceramics of low macroscopic properties with a small amount of natural macromolecules[5,6]. This natural composite materials with composite structure of complex self - assembly classification and Organic - inorganic hybrid,which the main feature is composition material is simple but having excellent overall performance , mainly including bones, shell pearls and dentin.The structure and performance characteristics of above materials provide a fresh idea for people to design materials and promote the development of human materials to the direction of new composite materials which blend the characters of evironmentalization、itelligent and cmpound[7-9]. Sarikaya and Akasay et al [10]first try to prepare imitation shell-like Al-B_4C layered material and tenacity than the single component B_4C increased by 4 times. Clegg et al. [11] deposited SiC thin layer with a thickness of about 200 μm on a graphite substrate and then sintered it. The resulting material has a fracture toughness of 15MPa $m^{1/2}$, which increased by 4 times compared with the fracture toughness of one - component SiC and whose breaking work improved by about 100 times. Li and Wang et al [12] preparated the Si_3N_4/BN layered composites using the casting method to study he effect of interfacial layer strength on the fracture properties of the composites. They found that the incorporation of 5% Si_3N_4 composites cracked more stable(Crack T-type expansion, the longest length) and with the increase of Si_3N_4 content, the crack propagation tends to be unstable(Crack perpendicular to the slice extension, the length decreases). Therefore, choosing the appropriate weak interface content can effectively improve the fracture toughness of layered materials. Munch et al[13] and Ritchie et al[14] changed the viscosity of the slurry and the solvent composition by adding sucrose to the alumina slurry thus affecting the ice crystal forming process to preparate a ceramic skeleton with a certain bridge density. After the low-intensity second-phase pressure is infiltrated into the ceramic skeleton, it is sintered, eventually this continuous layered structure transformed into a "brick-mud" structure. The thickness of the inorganic layer is about 10 to 20μm, the thickness of the organic resin is only 1μm, the volume fraction of the ceramic phase is 80%. The composite material prepared by this method has a strength of about 210 MPa, and the fracture toughness is twice the simple mechanical composition of the composition.

In conclution, in this paper, the organic phase is penetrated into the ceramic skeleton using a vacuum impregnation process and adjusted the ratio of the curing system to prepare a biomimetic composite with shell pearl layer structure, which the Al_2O_3 porous ceramics preparing by freeze-drying method are inorganic phase skeleton, epoxy resin curing system as weak interface layer organic phase.

2 Preparation of test materials

2.1 Preparation of ceramic matrix

Figure 1 shows the macroscopic and microscopic SEM images of alumina porous ceramics prepared by freeze-drying. It can be seen from the SEM photograph that its microstructure exhibits a lamellar structure , the thickness of which is about 25 μm, and the distance between the layers is about 12 μm. A single layer of surface are rough, rugged, and the interior of the layer has a large number of submicron holes. Therefore, the alumina porous ceramic prepared by freeze-drying method is a multi-scale layered structure. This is very similar to the aragonite in the structure of the pearl layer

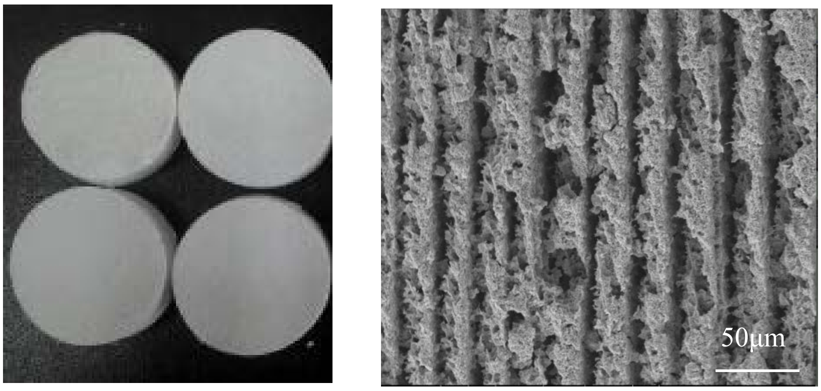

Fig.1 Macro and SEM photographs of Al_2O_3 porous ceramics

2.2 Second phase curing system

In this experiment, epoxy resin is selected as the impregnating resin. Low molecular weight polyamide resin as a curing agent for epoxy resin is non-toxic and nonvolatile and lower irritating to human skin. The curing products of low molecular weight polyamide resin with epoxy resin, and the adhesion and impact resistance between curing agent and basis have the excellent mechanical properties. Moreover, the use of low molecular weight polyamide as an epoxy resin curing agent added to the curing system to get the toughening effect to curing products to a certain extent to improve the shortcomings of epoxy resin brittle. Therefore, in view of the above reasons, in this study, low molecular weight polyamide 650 curing agent is used. The diluent is the active diluent propylene oxide butyl ether 660A. It can enhance the activity of epoxy resin, reduce viscosity, easy to operate and not easy to volatile.

1) Pretreatment

The porous ceramic is ultrasonically cleaned in alcohol for 15 minutes and then dried at 120℃ to remove the porous ceramic hole wall of dust, impurities and so on and improve the resin and ceramic wetting

2) Dipping treatment

A certain amount of epoxy resin is weighed, water bath heated it to pre-curing temperature of 30℃ for 30min. 60% diluent is added to the epoxy resinand and repeated heating and stirring evenly, so that epoxy resin has a certain mobility. The curing agent is further added to the epoxy resin-diluent mixture in five proportions（1：0.7、1:0.8、1:0.9、1:1.0、1:1.2）and the mixture is continuously stirred at 30℃. After mixing, the pre-treated Al2O3 porous ceramic is placed in a plastic container containing a resin curing systemAfter mixing, the pre-treated Al_2O_3 porous ceramic is placed in a plastic container containing a resin curing system and transfer it to a vacuum heating furnace for vacuum impregnation

3) After curing treatment

After immersing at 30℃ for 6h, the Al_2O_3/epoxy composite material is heated to 115℃ for

10℃/min. Heat preservation for 2 hours to make the resin curing system is fully cured. Finally, cooling with the furnace to reduce resin curing shrinkage.

3 Microstructure Characterization of Composites

In order to prepare the composite material of the pearl layer structure, the epoxy resin curing system is penetrated into the pores of Al_2O_3 porous ceramics using vacuum impregnation method. The impregnation process has been described in detail in the previous section. Figure 2 shows SEM images of Al_2O_3/epoxy composites with different solid content and additives. It can be seen from the figure that the epoxy curing system has been completely impregnated into the ceramic micropores of Al_2O_3. And due to the fine control of the resin curing process. The layered and the mesh-like skeleton of ceramic itself is well preserved, and the interface between the ceramic and the resin is very close. Table 1 compares the resin content of the Al_2O_3/epoxy resin composites and the content of organic matter in the pearl layer. It can be concluded that as the solid content increases, the resin content decreases gradually (shown in Table 1) and gradually approaching the content of organic matter in the nacre layer.

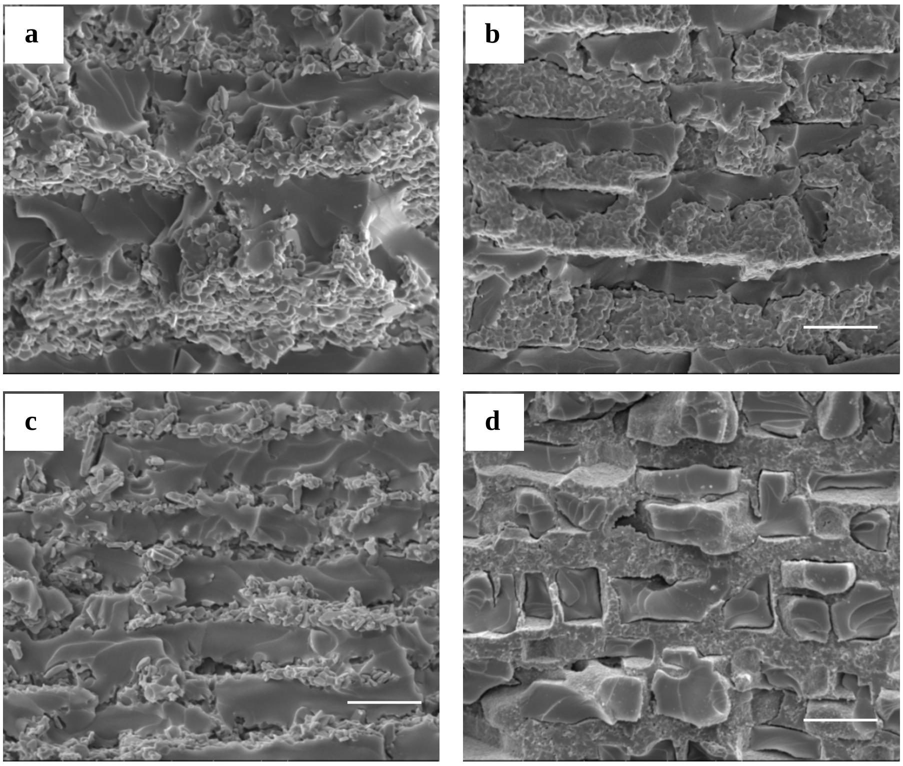

Fig.2 SEM images of Al_2O_3/epoxy composites with different solid content and additives
(a) Solid content 15% without glycerol; (b) Solid content of 20% without glycerol
(c) Solid content 15% glycerol; (d) Solid content 20% glycerol

Table 1 Comparison of the resin content in the composite material with the organic matter in the pearl layer

Solid content	With or without additives	Resin content	The content of organic matter in the pearl layer
15%	without	50.1%	
15%	with		5%
20%	without	40.2%	
20%	with		

4 Discussion

The Al_2O_3/epoxy resin composites with the curing ratio (epoxy resin and curing agent weight ratio) of 1:0.7、1:0.8、1:0.9、1:1and 1:1.2 are prepared respectively. The ceramic skeleton uses a solid content (the weight content of the ceramic paste) of 15% two additives including without additives and glycerol containing additives. The flexural strength and fracture toughness of the composites under different curing ratios are shown in Figure 3 and Figure 4. As the curing ratio changes, the basic change trend of composite material mechanical properties is that, when the curing ratio is less than 1: 1, with the increase in the amount of curing agent, the bending strength and fracture toughness of the material are increasing and when the curing ratio is 1: 1, the bending strength and fracture toughness of the material reached the maximum. With the content of curing agent are continued to increase, the bending strength and fracture toughness of the material are reduced. Indicating that there is an optimal value for the cure ratio. The main reason for this change is that, when the curing agent content is low, the resin curing system curing is not complete, did not form a three-dimensional network, highly cross-linked structure, therefore, the strength and toughness of the cured product is low. While the curing agent content is too much, there will be too much branching in the structure of the cured product or a large amount of unreacted curing agent remains in the crosslinked structure of the cured product, thus the mechanical properties of the material are affected. At the same time, it is found that the mechanical properties of the composites are not the same at the same curing ratio. Composites containing glycerol additives have higher flexural strength and fracture toughness than composites that do not contain glycerol additives. There are two reasons for this change. First, the addition of glycerol changed the microstructure of the material a ceramic bridge structure are introduced which connects the upper and lower surfaces of the ceramic pieces. When the material is subjected to an external load, the resistance between the layers is increased, and the strength of the material is increased. At the same time, the ceramic bridge also increases the resistance of the crack propagation, increasing the material fracture work, so that the toughness of the material increased. This is the same as the mechanism of mineral bridge in the pearl layer. Second, the addition of glycerol to the material d/λ ratio closer to the proportion of the layer of pearls, which is to say the introduction of glycerol to make the material layer structure is more natural selection and evolution of the natural pearl layer to improve, so the mechanical properties of the material has improved.

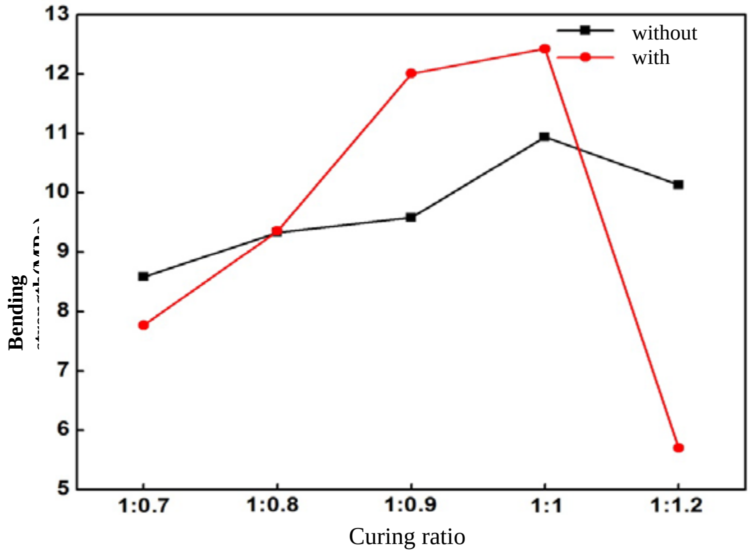

Fig.3 Bending strength of resin composites with solid content 15%/different penetration cure ratio

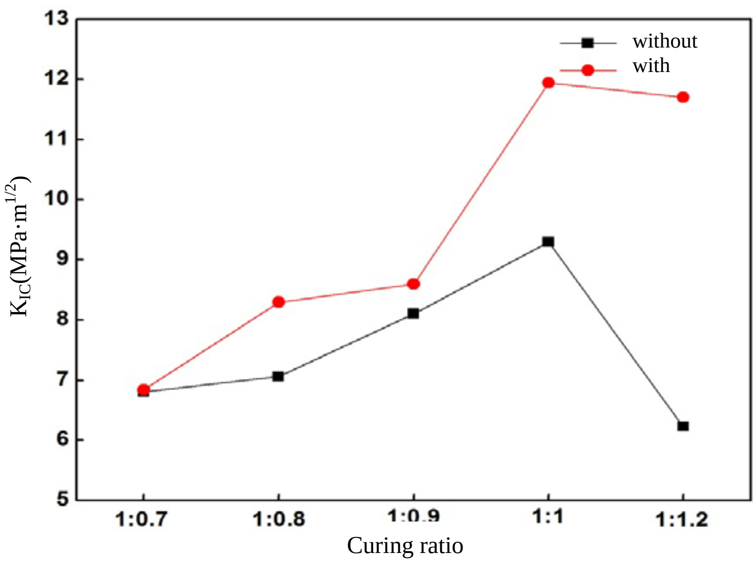

Fig.4 Fracture toughness of resin composites with solid content 15%/ different penetration cure ratio

5 Conclusion

In this paper, the microstructure and mechanical properties of imitation shell pearl layer Al_2O_3/epoxy resin composites prepared by vacuum impregnation process are studied. By analyzing the influence of different curing ratios on the mechanical behavior of the composites, the imitation shell pearl layer Al_2O_3/epoxy resin composites with excellent comprehensive performance are prepared. Mechanical properties of composite materials, which composed of 15% Solid content of ceramic skeleton and epoxy resin, with the increase in the amount of curing agent increas first and then lower. When the curing ratio is 1: 1(The weight ratio of epoxy resin and curing agent is 1: 1), the flexural strength and fracture toughness of the material reache the maximum.

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