

Microstructure of $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites^①

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Abstract: $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites were prepared and their microstructure was investigated by transmission electron microscope (TEM). The results show that intergranular nano-composites are achieved. The bonding between Al_2O_3 and SiO_2 particles is well and the interface is even. Amorphous phases and nano crystals appear in the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites, which both come into being during the cooling process after sintering. Glass phase does not appear between the Al_2O_3 and SiO_2 particles and only appears among the Al_2O_3 particles, which can be explained with stress model. The quantity of the glass phase is not much and its influence on the high-temperature deformation of the ceramic core nano-composites is little.

Key words: ceramic core nano-composite; microstructure; glass phase; stress model

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1 INTRODUCTION

The development of new generation of high performance aeroengine needs to improve the combustible gas temperature greatly^[1-5], and the adoption of hollow gas cooling blade is an important method^[6-8]. But the key of producing gas cooling blade with high cooling efficiency is the preparation of ceramic core with high properties. With the increase of the cooling efficiency of the gas blade, the property demand of the ceramic cores becomes higher step by step.

The properties of the ceramic core are determined by its materials. Ceramic cores mainly include silicon dioxide series and alumina series. The properties of the alumina-base ceramic core are higher than those of the silicon dioxide, which is mainly used in casting single crystal blades with higher casting temperature. But the disadvantages of the alumina-base ceramic core are that its strength at room temperature and high temperature are still not high enough, and the decorating is very difficult^[9]. It is necessary to develop a new kind of ceramic core materials with higher properties. So by adding SiO_2 nanoparticles into Al_2O_3 matrix and adopting some special methods, the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites are developed. The results show that the bending strength of the ceramic core nano-composites is improved greatly and the other properties are also better than those of the traditional alumina based ceramic core^[10, 11].

In this paper, the microstructure of the $\text{Al}_2\text{O}_3/$

SiO_2 ceramic core nano-composites and the $\text{Al}_2\text{O}_3/\text{SiO}_2$ interface were investigated by TEM.

2 EXPERIMENTAL

Two $\alpha\text{-Al}_2\text{O}_3$ powders with the purity more than 99.7% were used in this study: fine Al_2O_3 powder and coarse Al_2O_3 powder, with mean particle sizes of 3 μm and 30 μm , respectively. The mean particle sizes of SiO_2 nano-particles were 30 nm and the purity was more than 99.9%. Plasticizer was the mixture of 70% paraffin and 30% beeswax.

The SiO_2 nano-particles, fine Al_2O_3 powder and coarse Al_2O_3 powder were mixed. Then the mixture was dispersed with a special way, added into plasticizer and lubricant, heated and stirred for a certain time, and finally pressed with a pressure injection machine. The ceramic cores were put into a casket filled with Al_2O_3 powder, and sintered in a electric resistance furnace at 1250 °C for 4h. If the temperature was too high, the density of the ceramic core nano-composites would increase, which would make the following decorating very difficult. So in a general way, the porosity of ceramic cores was above 30%^[11, 12].

Samples with size of 4 mm × 4 mm × 3 mm were cut from the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites and ground to a thickness of 30 μm , then were twin jet electropolished in a solution of perchloric acid and ethanol. Finally the samples were gold-coated

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and the microstructure was investigated using transmission electron microscope (JEM-2000FX).

3 RESULTS AND DISCUSSION

3.1 Intergranular ceramic core nano-composites

TEM observation of the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites (Fig. 1) shows that intergranular nano-composites are achieved, with the SiO_2 nano-particles present inside the Al_2O_3 grains. The SiO_2 nano-particles are equiaxed after sintering and the sizes are between 300 and 400 nm.

As proper porosity was necessary for decorating later, the sintering temperature could not be too high and there would have certain pores in the ceramic cores. So all the SiO_2 nano-particles distributed inside the Al_2O_3 grains and could not form intragranular nano-composites.

3.2 Interfacial structure of $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites

The TEM micrograph of $\text{Al}_2\text{O}_3/\text{SiO}_2$ bonding is shown in Fig. 2(a), Fig. 2(b) is the zoom of $\text{Al}_2\text{O}_3/\text{SiO}_2$ interface. The interface is even and glass phase does not appear in the $\text{Al}_2\text{O}_3/\text{SiO}_2$ interface, which shows that the bonding between the Al_2O_3 and the SiO_2 nano-particles is strong.

3.3 Amorphous phase and nano crystal in ceramic core nano-composites

Interface between the Al_2O_3 particles is shown in Fig. 3(a) and Fig. 3(b), and Fig. 3(c) is the TEM selected-area electron diffraction pattern of the interface. The diffraction pattern in Fig. 3(c) shows that the amorphous phase appears in $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites, and glass phase exists between the Al_2O_3 particles.

In theory, the amorphous phase may come into being during the grinding or sintering process. But the grinding during the preparation of the ceramic core nano-composites was only adopted to make the powders mix uniformly, and the size of the Al_2O_3 particles was only several microns, which could not be made finer by grinding, so the amorphous phase could not appear in the grinding and it comes into being during the sintering process. The inclusions in Al_2O_3 powder lead to liquid phase during the sintering, and the liquid phase became glass phase in the cooling process after sintering. Fig. 3(d) shows that the nano crystals are separated out from the amorphous phase, so both the amorphous phase and the nano crystals appear during the cooling process after sintering.

Although the glass phase appears, the quantity of the glass phase is not much, so its influence

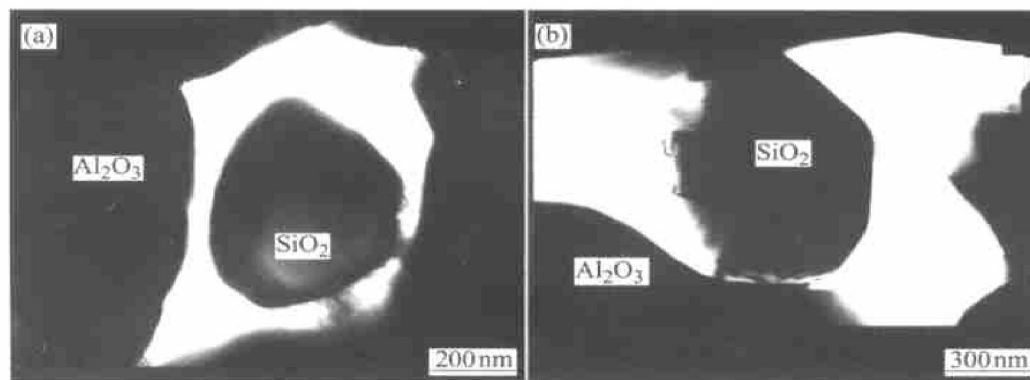


Fig. 1 TEM micrographs of $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites

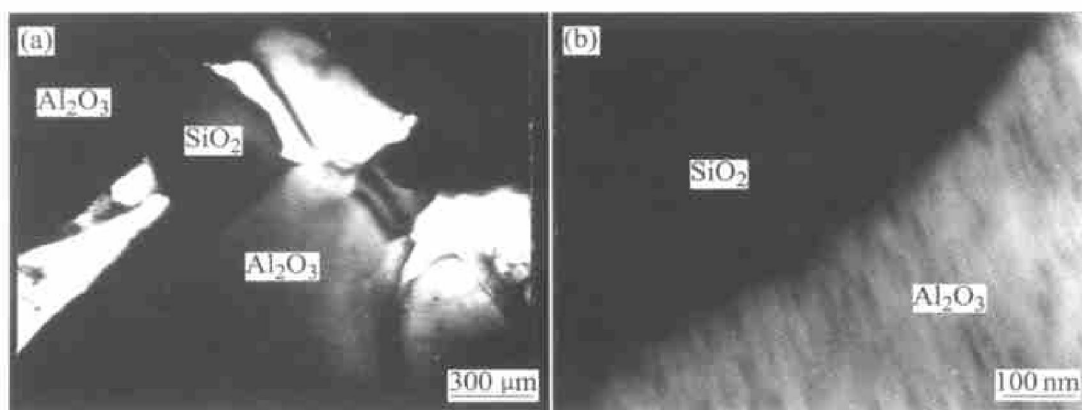


Fig. 2 Morphologies of interfacial structure of $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites

(a) —Nano SiO_2 among Al_2O_3 particles; (b) —Zoom of interface

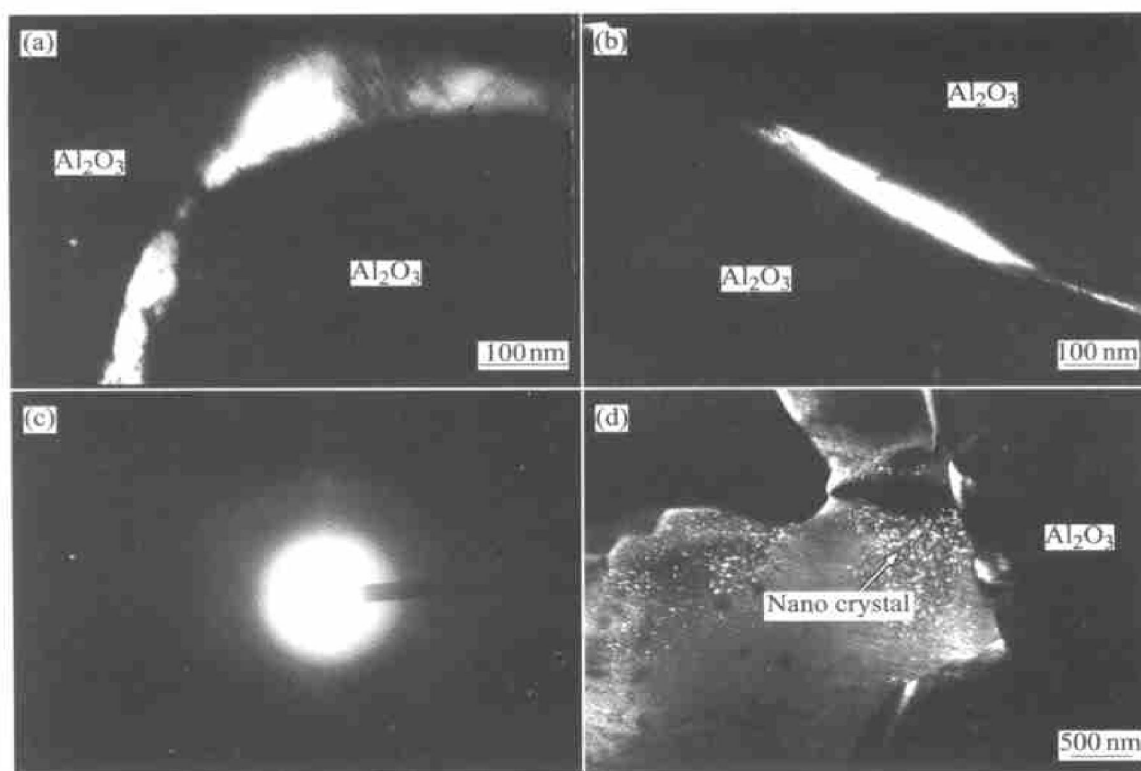


Fig. 3 Amorphous phase and nano crystal between Al_2O_3 particles

(a) and (b) — Morphologies between Al_2O_3 particles;
(c) — TEM selected-area electron diffraction pattern of amorphous phase; (d) — Morphology of nano crystals

ence on the high-temperature deflection of the ceramic cores is little. The strengthening of the SiO_2 nano-particles is primary. Furthermore, strengthening treatment after sintering could also descend the high temperature deflection of the ceramic core nano-composites produced by the glass phase. By adopting proper strengthening agent and technology, the high temperature deflection of the ceramic core nano-composite at 1550°C for 1 h was less than $1\text{ mm}^{[13]}$. Moreover, a little glass phase was also necessary for ceramic cores to sinter at lower temperature and to obtain certain strength^[14]. That is because the glass phase could wet the surface of solid phases, and the ceramic core was densified by dissolving-depositing and viscous flowing.

The glass phase only appears among the Al_2O_3 particles (Fig. 3) and does not appear between the Al_2O_3 particles and SiO_2 particles, which is accordant with the Ohji's results^[15] about the grain boundary of Al_2O_3 -17% SiC nanocomposites: the liquid phase exists among the Al_2O_3 particles and there is no liquid phase between the Al_2O_3 particles and SiC particles. Ohji explained this with stress model. The stress model was based on the hypothesis of most SiC particles existing at the boundary of the Al_2O_3 particles and tensile stress appearing there. That hypothesis was fit with the intergranular structure of $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites. So the phenomena that the glass phase in $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-com-

posites only appeared among the Al_2O_3 particles could be explained by Ohji's stress model. The thermal expansion coefficient of Al_2O_3 is $9.2 \times 10^{-6}/^\circ\text{C}$, and that for SiO_2 is $(0.51 - 0.62) \times 10^{-6}/^\circ\text{C}$. Stress would come into being during the cooling process after the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites were sintered. Tensile stress appeared in the $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ interface and compressive stress appeared in the $\text{Al}_2\text{O}_3/\text{SiO}_2$ interface, which made the liquid phase flow from the $\text{Al}_2\text{O}_3/\text{SiO}_2$ interface to the $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ interface. So there is no glass phase between the Al_2O_3 and SiO_2 particles. The stress model is shown in Fig. 4.

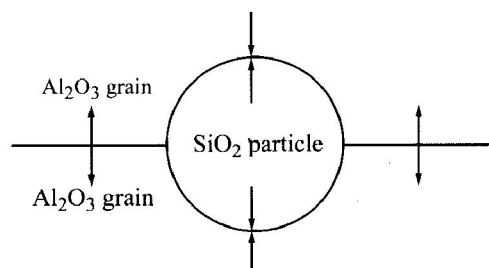


Fig. 4 Schematic illustration of internal stresses in $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites

4 CONCLUSIONS

- 1) $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nano-composites are

intergranular nanocomposites. The bonding between the Al_2O_3 and SiO_2 particles is well and the bonding face is even.

2) Amorphous phases and nano crystals appear in the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ceramic core nanocomposites, which both come into being during the cooling process after sintering. The quantity of the glass phase is not much and its influence on the high-temperature deformation of the ceramic core is little. The high temperature deflection can be dropped by the strengthening treatment after sintering.

3) Glass phase only appears among the Al_2O_3 particles and does not appear between the Al_2O_3 and SiO_2 particles. That can be explained with stress model.

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