



# Microstructure and mechanical performance of composite joints of sapphire by ultrasonic-assisted brazing



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## ABSTRACT

A method of lowering the coefficient of thermal expansion (CTE) of a bond metal of sapphire joints was examined for inhibiting the interfacial cracking of joints. An interlayer with the sandwich structure of Zn-Al/(SiCp/A356)/Zn-Al was fabricated and the surface of sapphire blocks was coated with a Zn-Al alloy by assistance of ultrasound. The assembly of sapphire and interlayer was brazed under the action of ultrasound. A metallurgical bond is performed between the sapphire and interlayer, and the composite material of SiCp/A356 is dissolved by Zn-Al alloys during brazing, resulting in a joint consisting of a new composite material of SiCp/Zn-Al-Si. It was composed of Zn-Al-Si alloy matrix and SiC particles with  $\sim 40$  vol.%, and has a CTE of  $\sim 7.4 \times 10^{-6} / ^\circ\text{C}$  estimated by using Turner's model, approaching that of sapphire ( $\sim 5 \times 10^{-6} / ^\circ\text{C}$ ). The maximum shear strength of the composite joints reached  $\sim 155 \pm 10$  MPa, an increase of  $\sim 250\%$  in comparison with that joined using the Zn-Al alloys.

## 1. Introduction

Brazed joints of  $\text{Al}_2\text{O}_3$ , including both alumina (multi-crystalline  $\text{Al}_2\text{O}_3$ ) and sapphire (monocrystalline  $\alpha\text{-Al}_2\text{O}_3$ ), are widely used in various applications.  $\text{Al}_2\text{O}_3$  is a typical oxide ceramic with a low coefficient of thermal expansion. During joining of  $\text{Al}_2\text{O}_3$  ceramic, the poor wettability of the liquid brazing alloy on the  $\text{Al}_2\text{O}_3$  ceramic surface and the large thermal residual stress in the joints are usually taken into account. The active brazing by means of a chemical reaction between an active element and  $\text{Al}_2\text{O}_3$  ceramic is often employed. The large thermal residual stress is ascribed to the notable mismatch of the coefficients of thermal expansion (CTE) between the sapphire and brazing alloy. For examples, Ag-based or Cu-based active brazing alloys are adopted during active brazing of  $\text{Al}_2\text{O}_3$  ceramics. These alloys have high solidus temperatures (more than  $750^\circ\text{C}$ ) and high CTE values (CTE of  $\sim 19.5 \times 10^{-6} / ^\circ\text{C}$  for Ag and  $\sim 17 \times 10^{-6} / ^\circ\text{C}$  for Cu), and high thermal residual stress is thus generated in the joint. The thermal residual stress is likely to lower the mechanical properties and reliability of the joint and is the focus of the present study.

Using the filler metals with a lower solidus temperature is an effective way of reducing the residual stress in a joint. To date, many efforts have been made to join  $\text{Al}_2\text{O}_3$  ceramics with the Sn-based alloys. Kolenak et al. (2011) soldered  $\text{Al}_2\text{O}_3$  ceramic using a Sn-Ag-Ti alloy. In

their experiments, a Ti-O interlayer formed at the ceramic/metal interface. Both RE and Ti acted as active elements, forming the oxide interlayers and promoting bonding at the alloy/ceramic interface. However, as an intrinsic disadvantage, the strength of the  $\text{Al}_2\text{O}_3$  ceramic joints with the Sn-based alloys is relatively low. It thus remains a great challenge to reduce thermal residual stress and to obtain a strong joint by lowering the solidus temperature of the brazing alloy. Decreasing the CTE of the filler materials is another way of reducing thermal stress. In the previous studies, the particles with a low CTE, such as  $\text{Al}_2\text{O}_3$ ,  $\text{Si}_3\text{N}_4$  and W, have been added into the filler metals to be used as the filler materials for joining of ceramics. However, the volume fraction of particles (VFP) obtained in this process is usually very low. For example, Yang et al. (2003) brazed  $\text{Al}_2\text{O}_3$  using a Ag-Cu-Ti alloy with  $\text{Al}_2\text{O}_3$  particles. As the VFP of  $\text{Al}_2\text{O}_3$  exceeded 10%, the strength of the joints decreased dramatically because of very low fluidity of the liquid filler metal. Chuang et al. (2011) used  $\text{Al}_2\text{O}_3$  nano particles to reinforce a Sn-Ag-Cu alloy. The mechanical properties began to degrade as the content of  $\text{Al}_2\text{O}_3$  was merely over 1.5 wt%, which is far from sufficient to decrease CTE of the filler metal.

Zn-Al alloys have a eutectic melting temperature of  $382^\circ\text{C}$ , only a little higher than that of Au-Sn alloys, and they usually have relatively high strength. Therefore, Zn-Al alloy is an ideal candidate for the brazing of  $\text{Al}_2\text{O}_3$  ceramic. If a robust bonding between the alloy and

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$\text{Al}_2\text{O}_3$  can be achieved, a large number of low-CTE particles can be considered to be added to this alloy. Bonding between Zn-Al and sapphire could be performed by introducing ultrasound during the brazing process. Brujan et al. (2008) revealed that a collapsing bubble at the liquid-solid interface could induce a high pressure of 1000 MPa. McNamara et al. (1999) studied the temperature in the center of single and multiple bubbles, finding that the temperature could reach 6000 K. To date, ultrasound has been successfully used to promote the wetting and bonding between ceramic particles and metals in the fabrication of metal matrix material composite. Cui et al. (2015) found that ultrasound could induce a nano- $\text{Al}_2\text{O}_3$  particle layer at the interface between Sn-Al alloy and sapphire, which dramatically improved the bonding. Moreover, in our previous studies, Yan et al. (2011) used Zn-based alloy to braze aluminum alloy matrix composites reinforced with SiC ceramic particles. The filler materials were fabricated by mechanical stir casting and ultrasonic treatment. The filler metal treated by ultrasound was free of defects and produced a non-porous bond strengthened with uniform particles.

The phenomenon was used for reference in this study to fabricate a composite structured bond. An interlayer of  $\text{SiC}_p/\text{A356}$  composite materials was used, and dissolved partially or completely with a filler metal of Zn-Al alloy during the ultrasonic-assisted brazing, forming a composite structure joint. Comparing with the widely-used powder mixing method, this work provides a new route for fabricating a composite structured bond with a much higher volume of ceramics particles, which leads to significant reduction in CTE of the joints. The aim of this paper is to provide a new route for fabricating brazed joints with a low CTE for ceramics. Sapphire is used as a representative example. This method can be applied to various ceramics.

## 2. Material and methods

Sapphire blocks with dimensions of  $5 \times 10 \times 10$  mm were provided by Hefei Yuanjing Crystal Co. Ltd. The  $10 \times 10$  mm face was highly polished to a surface roughness of  $\sim 0.3$  nm. Composite bulks comprising 55 vol%  $\text{SiC}_p/\text{A356Al}$  were made by squeeze casting. The size of the SiC particles is 10–50  $\mu\text{m}$ . The A356 Al matrix contains 6.5–7.5 wt% Si and 0.25–0.45 wt% Mg. The composites were cut into  $10 \times 10$  mm slices, with the thicknesses of 400, 600, and 800  $\mu\text{m}$ . The slices were ground and polished by a series of abrasive papers from 200# to 1500#. They were then polished using a polyamide polishing cloth, with a turbid liquid containing 1- $\mu\text{m}$  diamond particles. All sapphire blocks and composite slices were ultrasonically cleaned in acetone before brazing. A Zn-4 wt% Al alloy was made by melting and mixing commercial pure zinc and aluminum. The solidus and liquidus temperatures of the alloy are  $\sim 380$  and  $\sim 400$   $^\circ\text{C}$ , respectively.

Fig. 1a and b shows the ultrasonic-assisted coating procedure. The sapphire blocks or composite slices were placed into the Zn-Al alloy bath heated to  $420$   $^\circ\text{C}$ . They were then sonicated for 50 s and 10 s, respectively. Both sides of the composite slices were coated. After the coating, the blocks and slices were removed from the bath and cooled in air. They were covered with residual Zn-Al alloy having thickness of  $\sim 200$   $\mu\text{m}$ . Fig. 1c–e shows the ultrasonic-assisted brazing procedure. One composite slice was sandwiched between two sapphire blocks. The assembly was then placed in a specially designed holder in an open furnace. As the assembly was heated to  $420$   $^\circ\text{C}$ , an ultrasonic horn was pressed to the top sapphire block with a force of  $\sim 20$  N. Sonication was then applied for 10 s. Finally, the pressure was increased to  $\sim 200$  N with the aim of extruding most of the liquid Zn-Al alloy from the joints. Joints using Zn-Al alloy were made only for comparison.

Cross-sections of the sapphire joints were cut from the brazed joints and then ground and polished. As sapphire and SiC particles are insulating, it is difficult to observe the cross-section of the joints employing scanning electron microscopy (SEM). Therefore, an Au layer was plated on the samples by ion sputtering before SEM. The shear strength of six samples was tested using an Instron-5500R (Norwood,

Ma, USA) electromechanical material testing machine. The strain rate was 1 mm/min. The VFP of the joints and composites was measured using Image-Pro software.

## 3. Results and discussion

For comparison, sapphire blocks were firstly brazed merely using the Zn-4Al alloy. Fig. 2 shows interfacial microstructures of the sapphire/Zn-Al and Zn-Al/composite after ultrasonic-assisted coating. The parameters were selected based on our previous experiments. As shown in Fig. 2a, a metallurgical bond formed between Zn-Al alloys and the substrate of sapphires, with no obvious cracks or other flaws were found in the bond region. However, owing to the limitation of the SEM resolution, no obvious reaction layer was revealed at the interface. As shown in Fig. 2b, the left side is the residual Zn-Al alloy and the right side is the precursor composite at the Zn-Al/precursor composite interface. In between there is a 100–200- $\mu\text{m}$ -thick  $\text{SiC}_p/\text{Zn-Al}$  composite layer. In our previous work, Zhang et al. (2010) ultrasonically brazed the same  $\text{SiC}_p/\text{A356}$  composites with the Zn-Al alloy, a composite joint was obtained quickly and the aluminum content in the metal matrix of the joint was as high as 11–31 wt%. It is believed that the notable dissolution of aluminum matrix was due to turbulent acoustic streaming and cavitation in the ultrasound field.

For comparison, sapphire blocks were brazed using a filler metal of Zn-4Al alloy. Fig. 3 shows the microstructure of the sapphire joints brazed with the Zn-4Al alloy after ultrasonic-assisted coating. The brazing sonication was 10 s. In the ultrasound field, the oxide films on the liquid Zn-Al alloy were broken, and the bond region between the two blocks was fully filled by the Zn-Al alloy. A seemingly good bond was obtained. On the contrary, the cracking occurred at the alloy/sapphire interface, as shown in Fig. 3b. In the cooling process after ultrasonic coating, the shrinkage of the residual Zn-Al alloy near the interface was restricted by the sapphire substrate, while the surface of Zn-Al alloy could shrink freely on the other side. The residual stress was thus not high enough to cause cracks at the alloy/sapphire interface. However, in the cooling process after brazing, the Zn-Al alloy was sandwiched between two sapphire blocks, and its shrinkage was thus strongly restricted on both sides. This may lead to much higher thermal stress during cooling, which consequently generated cracking at the alloy/sapphire interface. Owing to the existence of cracks, the shear strength of the joints was very low, merely 30–50 MPa.

In the brazing procedure, the Zn-Al alloy melts. Pressing and ultrasonic vibration are applied to the sapphire blocks. Part of the liquid Zn-Al alloy is squeezed out of the bond region. Thus, the final thickness of the bond region is less than the sum of the thickness of the precursor layers and coated layers. Fig. 4 shows the microstructure of the brazed joints with the precursor interlayers. The residual precursor layer in the joints was  $\sim 400$   $\mu\text{m}$  thick.  $\text{SiC}_p/\text{Zn-Al}$  composite layers with a thickness of  $\sim 100$   $\mu\text{m}$  formed on both sides of the precursor after the ultrasonic coating. As the precursor layer was 600  $\mu\text{m}$ , the residual layer in the joints was  $\sim 300$   $\mu\text{m}$  thick, and the thickness of  $\text{SiC}_p/\text{Zn-Al}$  composite layers was still  $\sim 100$   $\mu\text{m}$ , as shown in Fig. 4b. As the precursor layer was 400  $\mu\text{m}$  thick, the residual precursor layer disappeared in the joints. The matrix A356 alloy of the precursor was completely dissolved by the Zn-Al alloy, resulting in an integrated  $\text{SiC}_p/\text{Zn-Al}$  composite joint, as shown in Fig. 4c. Fig. 4d shows the detailed structure of the new composite joint. SiC particles distributed in the Zn-Al alloy evenly. The VFP in the bond region was 37–42 vol%. The analysis by Electron-induced x-ray fluorescence indicated that the content of aluminum in the Zn-Al alloy matrix was 11–13 wt% on average, which is very close to the saturated solubility of Al in Zn at  $420$   $^\circ\text{C}$ . During the ultrasonic-assisted brazing, the aluminum matrix in the precursor layer was mixed with the Zn-Al alloy. The rise of the Al content in the joints resulted from the dissolution procedure. Microstructure of the interface between the  $\text{SiC}_p/\text{Zn-Al}$  composite and sapphire substrate is shown in the Fig. 4e. Cracks at the sapphire/alloy interface were not found,

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