



# Microstructural evolution of SiC joints soldered using Zn–Al filler metals with the assistance of ultrasound

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## ABSTRACT

SiC ceramics were successfully soldered with the assistance of ultrasound. Two kinds of filler metals, namely non-eutectic Zn–5Al–3Cu and eutectic Zn–5Al alloys, were used. The effects of ultrasonic action on the microstructure and mechanical properties of the soldered joints were investigated. The results showed that ultrasound could promote the wetting and bonding between the SiC ceramic and filler metals within tens of seconds. For the Zn–5Al–3Cu solder, a fully grain-refined structure in the bond layer was obtained as the ultrasonic action time increased. This may lead to a substantial enhancement in the strength of the soldered joints. For the Zn–5Al solder, the shear strength of the soldered joints was only ~102 MPa when the ultrasonic action time was shorter, and fractures occurred in the brittle lamellar eutectic phases in the center of the bond layer. With increasing ultrasonic action time, the lamellar eutectic phase in the bond layer of SiC joints could be completely transformed to a fine non-lamellar eutectic structure. Meanwhile, the grains in the bond layer were obviously refined. Those results led to the remarkable enhancement of the shear strength of the joints (~138 MPa) using the Zn–5Al solder, which had approached that enhancement using the Zn–5Al–3Cu solder. The enhanced mechanical properties of the joints were attributed to the significant refinement of the grains and the change in the eutectic structure in the bond layer. Prolonged enhanced heterogeneous nucleation triggered by ultrasonic cavitation is the predominant refinement mechanism of the bond metals of the SiC joints.

## 1. Introduction

Currently, as one of the most promising ceramic materials, SiC ceramics are becoming increasingly attractive for applications in several industries such as the aerospace, automotive, energy, electronics and optical devices [1–3] and have been especially attractive for high-performance microelectronic packaging due to their remarkable mechanical properties, excellent thermal conductivity and low thermal expansion coefficient. In practical applications, SiC ceramics must be firmly joined to themselves or to metals. Therefore, reliable joining of SiC ceramics is absolutely essential for broadening their applications.

Brazing is the most commonly used method for the joining of ceramics. Most work on brazing of SiC ceramics has been performed by using mainly the filler metals of Ag–Cu–Ti [4,5], Co-based [6,7], Fe–Si [8] and Ni–Cr [9] alloys. Nevertheless, the brazing process cannot be applied in electronic engineering due to their high brazing temperature ranging from 800 to 1200 °C, which can give rise to thermal damage to electronic components. Therefore, the joining at relatively low temperature is in demand for use in applications.

The Sn-based lead-free solders possess very low melting points and

are currently commonly used as joining materials in the electronic packaging industry [10,11]. However, due to the low melting point of Sn-based solders, which is lower than 250 °C, these alloys are unable to meet the requirements for application in the power electronics devices at elevated temperatures, which makes further investigation of the soldering processes highly necessary.

High temperature solders are used to produce the reliable electronics to which a high-density packaging technology and high-operating temperature are applied. The solidus temperature for the high temperature solder is above 370 °C and the liquidus is below 400 °C. The Zn–Al alloys possess a moderate melting temperature above 350 °C, higher corrosion resistance, excellent thermal and electrical conductivities and superior mechanical properties, making them reasonable candidates as high-temperature solder alloys used extensively in die-attach, power semiconductor, and optical device packaging, as well as flip-chip packaging [12–15]. Recently, Zn–Al based filler metals have been used for joining various materials including ceramics and metals with high service temperature. For examples, Ji et al. [16] soldered alumina to copper using Zn–14Al solders with the assistance of ultrasonic irradiation. Xiao et al. [17,18] soldered copper to copper or

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copper to aluminum using Zn–3Al filler metals by ultrasonic-assisted soldering. The eutectic Zn–5Al solder is a better choice for joining SiC ceramics because they possess a low melting point of 381 °C. However, a large amount of brittle Zn–Al eutectics will inevitably deteriorate the mechanical properties of the joints. Hence, the microstructure should be optimized through the design of alloy composition or the soldering process optimization. In our previous research [19], we found that the addition of Cu to Zn–Al alloys can lead to a change in the microstructure by eliminating the brittle Zn–Al eutectic phase and leading to the improvement in the mechanical properties of the joints. In addition, it is also known that the application of an ultrasonic field in melt treatment has positive effects on the refinement of the crystal microstructure of the alloy, thereby improving the mechanical properties [20]. An ultrasonic-assisted soldering process has been developed in recent years. Several studies have revealed that solder joint strengthening can also be achieved by integrating ultrasonic vibrations into the soldering process [21–23].

In our present study, Zn–5Al and Zn–5Al–3Cu alloys were selected as the filler metals to join SiC ceramics by ultrasonic-assisted air soldering. The objective of this work was to identify the microstructural evolution of the joints and its relationship with the corresponding mechanical properties and fracture modes. This study is expected to be helpful for the development of high-reliability SiC ceramic joints for high-density electronic packaging application.

## 2. Experimental procedures

The pressureless sintering SiC ceramic used in this study was provided by Shanghai Unite Technology Co., Ltd. Shanghai, China. The SiC ceramic was sectioned with a diamond blade to obtain samples with the dimensions of 40 mm × 10 mm × 3 mm. Prior to the soldering, all joining surfaces were polished by 1 μm diamond paste, and then were ultrasonically cleaned in acetone for 10 min. The Zn–5Al (wt%) eutectic alloy and Zn–5Al–3Cu (wt%) alloy were used as filler metals. High purity metals of Al (99.99%), Zn (99.99%), and Cu (99.999%) were used for alloy preparation. The alloys were prepared in a glove box filled with high purity Ar (99.9999%). A pre-measured quantity of Al–5Ti–1B grain-refining master alloy rod was preheated and added to the molten alloy at 1% level (upper limit used in industry). The chemical compositions and solidus ( $T_S$ ) and liquidus ( $T_L$ ) temperatures of the filler metals are shown in Table 1. The  $T_S$  and  $T_L$  values of alloys were determined using a differential scanning calorimeter (DSC, Netzsch-STA449F3) at the heating rate of 10 K/min in a purified Ar atmosphere.

The principle of ultrasonic-assisted soldering is schematically represented in Fig. 1a. The SiC samples were placed in a single overlap configuration with the ~20 mm overlap length. Then, the samples were heated to 420 °C in air at the rate of 50 °C/min and ultrasonic vibration was applied (Fig. 1b). The amplitude of the ultrasonic vibration was ~3.5 μm, as measured by a laser Doppler vibrometer (Polytec OFV-505/5000, Germany). The ultrasonic frequency, pressure and power were fixed at 20 kHz, 0.4 MPa and 300 W, respectively. The ultrasonic action time is one of the main process parameters, and ranged from 5 s to 20 s. Subsequently, the soldered couples were held for 3 min at the soldering temperature, and then cooled in air to room temperature.

After joining, the soldered joints were mounted in epoxy resin and

**Table 1**  
Chemical compositions, and solidus ( $T_S$ ) and liquidus ( $T_L$ ) temperatures of the filler metals.

Filler metals	Chemical composition (wt%)				Solidus and liquidus temperatures (°C)	
	Zn	Al	Cu	Si	$T_S$	$T_L$
Zn–5Al	Balance	5.8	0.3	0.05	373	381
Zn–5Al–3Cu	Balance	5.2	3.1	0.08	375	385

then were cut perpendicular to the bonding interface for the cross-sectional microstructure analysis. In addition, the soldered joints were cut parallel to the bonding interface until the bond layer was exposed for the electron backscattering diffraction (EBSD) microstructure analysis of the solder matrix. EBSD measurements were obtained using commercially available software (supplied by HKL Technology APS). The joints were then observed using a scanning electron microscope (SEM) (FEI; Quanta 200FEG) equipped with an energy-dispersive X-ray spectroscopy (EDS) BRUKER AXS system. The grain size of the bond layer was evaluated in the EBSD mode. The shear strength tests were measured by shear testing using a specially designed fixture as illustrated in Fig. 1c. The size of shear test samples of joints was 10 mm × 5 mm × 6 mm. The soldered specimens were compressed at the constant speed of 0.5 mm/min at room temperature. The shear strength was calculated by the load at the fracture divided by the nominal area of the joint. To ensure reliable strength results, five samples were examined at the given set of parameters to calculate the average value of the joint as well as the standard deviation of the shear strength. After shear testing, the fracture path and surfaces were investigated by SEM.

## 3. Results and discussion

### 3.1. Microstructural examination of joints

Fig. 2 shows the representative microstructures of the joints using the Zn–5Al filler metal and Zn–5Al–3Cu with ultrasonic action for 5 s. As shown in Fig. 2a and b, the joints are free of obvious defects such as voids and cracks. The observations of the cross-sectional interface at greater magnification show that no obvious reaction products could be observed at the interface (Fig. 2c and d). The elemental compositions from the EDS analysis (at.%) for the different regions in the joints are listed in Table 2. According to the results of Table 2, it can be seen that the bond layer of the joints using the Zn–5Al solder (Fig. 2e) mainly consisted of the η-Zn phase (light) and a large region of the Zn–Al eutectic interdendritic structures (textured pattern). According to the Zn–Al binary phase diagram [15], the primary η-Zn phase precipitates from the molten solder at first during the solidification. When the soldering temperature is below 382 °C, a eutectic transformation occurs, and α + η lamellar eutectics are established from the remaining liquid enriched with solute segregation. For the Zn–5Al solder, the η-Zn phase was in a grain-like morphology, while the eutectic phases are mainly the lamellar structure in the center of the bond layer and only a small amount of the non-lamellar structure (Fig. 2e).

As shown in Fig. 2f, for the Zn–5Al–3Cu solder, the η-Zn phases and eutectic phases were identified as the main phases in the bond layer. In addition to the lamellar Zn–Al eutectic phase, non-lamellar eutectics were also observed in the bond layer. According to the Zn–Al–Cu phase diagram and the EDS results presented in Table 2, the non-lamellar eutectic phases were found in the form of the gray η-Zn matrix embedded with the white rod-like or plate-like ε-CuZn<sub>4</sub> phase and the dark whisker-like α-Al phase. The formation of CuZn<sub>4</sub> phase was due to the strong reaction activity between the Zn and Cu elements [24]. The lamellar structure was composed of the dark α-Al phase and the gray η-Zn phase. The result was similar to the results in Refs. [12–14]. Compared to the joint soldered with the Zn–5Al filler metal shown in Fig. 2a, it can be seen that the volume fractions of the primary η-Zn phases increased, but the amount of the eutectic structure decreased. That is, the addition of Cu to the Zn–5Al alloy could promote the heterogeneous nucleation of the η-Zn phases but suppressed the formation of eutectic structures; therefore, it will be helpful for improving the mechanical properties of the Zn–Al based alloy.

Fig. 3 shows the representative microstructures of the joints using the Zn–5Al filler metal with the ultrasonic action time of 20 s. As the ultrasonic action time increases, the morphology of the eutectic structure varies considerably and the lamellar Zn–Al eutectic structure in the

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