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Article:

Xiao, Y., Song, Z., Li, S. et al. (4 more authors) (2020) Bonding and strengthening mechanism of ultrasonically soldered 7075 Al joint using Ni-foam/Sn composite solder foil. *Materials Science and Engineering: A*, 791. 139691. ISSN 0921-5093

<https://doi.org/10.1016/j.msea.2020.139691>

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Bonding and strengthening mechanism of ultrasonically soldered 7075 Al joint using Ni-foam/Sn composite solder foil

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Abstract: Ni-foam reinforced Sn-based composite solder was employed to join ultrafine grain 7075 Al alloy with the assistance of ultrasound. The solder seam was mainly composed of Ni skeletons, Sn-based solder, α -Al phase and very fine (hundreds of nanometers to several microns diameter) Ni_3Sn_4 particles. Discontinuous networks surrounded by the fine Ni_3Sn_4 and α -Al particles were in-situ formed in the solder seam, the diameter of which was decreased with increasing soldering time. Smooth transition of the lattice from Al substrate to Sn-based solder was achieved by the formation of an amorphous Al_2O_3 interlayer at the interface. The microstructure evolution mechanism and the amorphous Al_2O_3 interlayer formation mechanism are discussed in detail. The shear strength of Al/Ni-Sn/Al joints increased with prolonging soldering time. The Al/Ni-Sn/Al joint ultrasonically soldered for 60 s exhibits a shear strength of 71.4 MPa, which was approximately 37 % higher than that soldered with pure Sn solder.

Keywords: ultrafine grained Al alloy, Ni-foam/Sn composite solder, ultrasonic soldering, microstructure, bonding mechanism

1. Introduction

Ultrafine grained (UFG) Al alloys are the subject of extensive attention from aerospace, electronic products and high precision instrument industries for their high strength, high hardness, and good electrical and thermal conductivity [1]. Severe plastic deformation technology is used to fabricate UFG Al alloys, leading to them having an extremely high density of grain boundaries and high strain energy storage in the lattice [2,3]. This makes the UFG Al alloy structure highly sensitive to heat [4], and common joining methods with a significant thermal component, such as friction stir welding, fusion welding and high-temperature brazing, are not applicable to them. Thus, the heat input must be strictly controlled to produce high-quality connections with UFG Al alloys. Among the alloys used to produce UFG Al, 7000 series alloys feature. Such alloys, being heavily alloyed to achieve high strength, also present some challenges for welding and higher temperature process such as brazing. Developments towards joining of such alloys would also be of interest.

Ultrasonic-assisted low temperature soldering of Al alloys using Sn-based solders has germinated tremendous research enthusiasm for meeting the growing application requirement of Al alloys [5-8]. Acoustic cavitations induced by ultrasonic waves in a liquid medium can create extremely high temperature and pressure locally on a solid substrate surface [9,10]. The oxide films on Al substrates can be removed effectively under the action of these acoustic cavitations, and thus the ultrasonic-assisted low temperature soldering of Al alloys has been achieved even without the use of flux [7,11]. However, despite the potential of ultrasonic-assisted low temperature soldering of Al alloys shown in previous studies [6,8], little attention was paid to the underlying bonding mechanism between the Sn based solder and the Al substrate. In fact, the Sn and Al couple has low mutual solid solubility and no interfacial reaction compounds (IMCs) form on the Al-Sn phase diagram [12]. Crystallographic analysis has shown that the lattice mismatch of Al and Sn is large. Thus, achieving a metallurgical bond between Sn solder and an Al substrate is challenging. Guo et al. [13] inferred the formation of a crystalline Al_2O_3 interlayer on the Al substrate surface in an ultrasonically soldered Al/Sn-Zn/Al joint according to scanning electron microscope (SEM)

observation, though this result raises a further question of how the Sn solder connects with the crystalline Al_2O_3 , since large thermo-physical differences exist between their crystals. It seems that more sophisticated techniques such as high resolution transmission electron microscope (HRTEM) are necessary to successfully detect and characterize the interfacial layer.

Achieving adequate bonding strength is another important concern for the practical application of soldered Al alloy joints. Unfortunately, Al alloy joints ultrasonically soldered with Sn-based solder invariably display poor mechanical properties because of the strength limitation of the Sn-based alloy [5,8,14]. The addition of second phase strengthening particles or fibers [15-17] into Sn-based solders may improve their strength, though the development of effective methods to avoid the agglomeration of particles and to improve the wetting of the reinforcement phase and the Sn matrix remain significant challenges. An alternative way to improve the strength (and also the stability against remelting) of low melting metals is replacing the particles or fiber reinforced phases with high melting point alloy foams [18,19]. The 3D continuous network structure of alloy foam can provide the low melting point metal matrix with an interpenetrating strengthening structure and superior mechanical properties [18,19]. Considering the structural and functional advantages (an open 3D network structure and excellent metallurgical reactions with Sn-based solder), open-cell Ni-foam would be a good choice for this purpose. Our previous studies [20,21] found that when Ni-foam was used as the reinforcement phase of Sn-based solder, the strength of the joints can be largely improved by the combined effects of the presence within the joint of the Ni-foam skeletons and Ni/Sn interfacial intermetallic compounds (IMCs) which are generated during the joining process. The strengthening effects of the Ni-foam correlate closely with the morphology and distribution of Ni/Sn interfacial reactants; for example, formation of a continuous Ni_3Sn_4 IMC layer distributed between the strips of Ni skeletons could deteriorate the joint strength [21]. Thus, it is important to tailor the morphology and distribution of Ni/Sn interfacial reactants during the ultrasonic soldering process.

In this study, Ni-foam reinforced Sn-based composite solder was used to join UFG

7075Al alloy with the assistance of ultrasound, with an emphasis on correlating the application of ultrasonic waves with the microstructure evolution and interfacial bonding mechanisms. A special fixture was designed to avoid the compression of Ni skeletons, and to realize optimization of joint microstructure by ultrasound. In-situ formed interfacial reactants surrounding the 3D network strengthening structures were found in the solder seam. Moreover, an amorphous Al_2O_3 interlayer was identified at the Sn-based composite solder/ Al substrate interface. The effect of ultrasonic soldering time on the microstructure and mechanical properties of joints was investigated. The findings of this study would be helpful in fabricating highly reliable Al alloy joints by ultrasonic soldering at low temperature using a novel Sn-based composite solder.

2. Experimental materials and procedure

UFG 7075Al alloy sheets with dimensions of $20 \times 10 \times 3 \text{ mm}^3$ were used as substrates. Pure Sn ingots were used as the solder matrix, and Ni-foam slices (Wuxi Guiyan Co., Ltd) with a thickness of 0.5 mm, purity of 99.99 wt. % and porosity of 98 % were used as the strengthening phase. The composite solder alloy was fabricated by first immersing the Ni-foam into molten Sn solder kept at $300 \text{ }^\circ\text{C}$. After the Ni-foam was completely infiltrated with pure Sn solder (which happens spontaneously, as molten Sn wets Ni), the composite solder slice was pulled out and rolled in a series of passes to a thickness of 0.25 mm in an electric rolling machine (Kejin Co., Ltd, MSK-2150). Prior to the soldering process, the surfaces of Al substrates and composite solder foils were polished and cleaned in absolute ethanol.

The ultrasonic-assisted soldering process conducted in this study is shown schematically in Fig. 1. The Al substrates were assembled in a lap-joint configuration with the Ni-foam/Sn composite solder foil inserted between them. The faying area of joint was $10 \times 10 \text{ mm}^2$. A bespoke stainless steel platform was used to control a gap of $150 \text{ }\mu\text{m}$ between upper and lower Al substrates during the soldering process. A vertical pressure was exerted on one side of the upper Al substrate (i.e. the load did not pass across the joint) with a weight of 5 kg. Ultrasonic vibration was imposed on the lower Al substrate surface through a titanium horn.

The frequency of the ultrasonic vibration was 20 kHz, and the maximum output power was 500 W. The temperature of the joint was monitored with a K-type thermocouple by inserting it into the soldering seam. All samples were heated to 280 °C in air and kept stable for several seconds, then the ultrasonic vibration was applied for 5 s, 30 s, 60 s and 120 s, respectively. When the ultrasonic vibration stopped, the as-soldered joint was cooled in air and moved from the heating platform. For comparison, Al alloy joints ultrasonically soldered with pure Sn solder with the same soldering parameters were also fabricated.

The cross-sections of as-soldered joints were prepared for metallographic observation. Microstructure and elemental compositions of joints were examined by scanning electron microscope (SEM, Hitachi S-4700) equipped with an energy dispersive X-ray spectrometer (EDS). Phase constitutions of joints were identified by X-ray diffraction (XRD, Rigaku D/max-2500PC, Cu K α). The interface between the composite solder and the Al substrate was characterized with high-resolution scanning transmission electron microscopy (HRSTEM, Titan G2 60-300 Probe Cs Corrector). A TEM-lamella was cut using a dual focused ion beam (FIB) system in nova NanoSEM 450 scanning electron microscope. A Pt capping layer was deposited on the specimen surface prior to the FIB cutting procedure. Tensile test of solder foil was performed with an electronic universal mechanical testing machine (MTS-E44.104) at a constant cross-head speed of 0.5 mm/min. The solder foil was cut into a dumbbell shape, with a length, width and transition radius of 10 mm, 2.5 mm and 15 mm, respectively. The shear strength of joint was tested on a universal testing machine (WCW-100) at a constant cross-head speed of 0.5 mm/min. **The test employed is a modification of the standard ASTM: D1002-10, which is used to test the shear strength of metal-to-metal lap joints.** Five samples were tested to obtain an average value for each parameter.

3. Results and discussion

3.1 Microstructure and properties of composite solder

Fig. 2a shows the microstructure of the Ni-foam before the infiltration treatment. The open-cell Ni-foam exhibits a continuous network structure. The pore diameter is in the range of 200 to 400 μm , which is much larger than the critical size for the formation of acoustic

cavities [22,23]. Fig. 2b shows the cross-section image of Ni-foam/Sn composite solder foil. The space in the Ni-foam is completely filled with pure Sn solder, and gray Ni skeletons are distributed randomly in the Sn matrix. The cross section also shows how the Ni-foam is a hollow strut structure, which results from the manufacturing process, where a removable polymer template is coated with nickel; it is notable that the interior spaces, although narrow (10 μ m or less in some cases), appear to have been infiltrated by the Sn. As Ni is wetted by liquid Sn, this infiltration will have been aided by capillary forces. Fig. 3 shows the average tensile strengths of pure Sn and Ni-foam/Sn composite solders. The measured tensile strength of pure Sn solder is only 15.05 MPa, while the measured tensile strength of Ni-foam/Sn composite solder foil is 49.77 MPa, which is approximately 34.72 MPa higher than that of pure Sn solder. This demonstrates that the Ni-foam has the potential to serve as an excellent strengthening phase for Sn-based solder.

3.2 Microstructure of joints

Fig. 4 shows the SEM images of Al alloy joints ultrasonically soldered with pure Sn solder at 280 °C for different times. No gaps or cavities are found in the joint, even after soldering for only 5 s (Fig. 4a and 4b). The Sn/Al interfaces are curved with some dissolution pits formed on the Al substrate surface. This indicates that ultrasound-induced acoustic cavitation effects have formed in the soldering seam, which result in the removal of oxide films from the Al substrate surfaces and the erosion of the Al substrate into the Sn matrix [6,7]. Some α -Al particles are found in the solder seam, the larger of which have a faceted shape. These are formed by precipitation of the dissolved Al element leading to growth of the initially formed α -Al phases during the soldering process [5]. By prolonging the ultrasonic soldering time to 120 s (Fig. 4c and 4d), the dissolution of material from the Al substrate into the molten Sn solder is increased, resulting in a greatly increased quantity of the bulkier α -Al particles in the solder seam. These larger α -Al forms tend to accumulate near the upper Al substrate because of the buoyancy forces caused by the large density difference between Sn (7.31 g/cm³) and Al (2.69 g/cm³). Interestingly, some of these larger Al-containing regions near to the Al substrate surface are identified to be an O-rich phase (Table 1), which exhibit a

gray color compared with the darker α -Al precipitates. This phenomenon is associated with the segregation of elemental O near the Al substrate, and will be discussed in more detail subsequently.

The cross-section images of Al alloy joints ultrasonically soldered with Ni-foam/Sn composite solder at 280 °C for 5 s, 30 s, 60 s and 120 s are shown in Fig. 5. Sound soldering joints are obtained and no cracks or cavities can be found in the solder seam. The microstructure of the solder seam is largely different from that of the initial Ni-foam/Sn composite solder shown in Fig. 2b. Fig. 5a and 5b show the cross-section images of Al/Ni-Sn/Al joint ultrasonically soldered for 5 s. In these the Ni-foam maintains the continuous, reticulated structure and random distribution in the Sn-based solder. Some black contrast structures, a few microns in size, can be found in the solder seam, which were identified to be α -Al phase according to the EDS analysis results shown in Table 2. A gray reaction layer is formed on the Ni skeleton surface, which is most clearly visible on the inner side of Ni skeleton and was identified to be Ni_3Sn_4 phase with some Al dissolved in it. A large number of very fine gray particles with a diameter ranging from hundreds of nanometers to several microns are distributed in the Sn matrix and exhibit a network structure. According to the EDS analysis results, the fine particles are inferred to be a mixture of Ni_3Sn_4 and α -Al phases. With increasing ultrasonic soldering time to 30 s and 60 s, as shown in Fig. 5c-5f, the number of tiny gray particles increases sharply, accompanied by a decrease in the characteristic size of the networks these particles form. There are no apparent interfacial reaction layers on the Al substrate surface (Fig. 5d and 5f). This result is different from what was found in our previous studies [21], in which an Al_3Ni reaction layer was found on the Al substrate surface when the Ni skeletons came into close contact with the Al substrate in an Al/Ni-Sn/Al ultrasonically soldered joint. The XRD profiles of Al/Ni-Sn/Al joints ultrasonically soldered at 280 °C for 5 s and 30 s are shown in Fig. 6. Only peaks corresponding to the Ni_3Sn_4 phase, along with those of elemental Ni, Sn and Al, are found in the in soldering seam. This convincingly demonstrates that the tiny particles in the solder seam are the mixture of Ni_3Sn_4 and α -Al phases, and Al-Ni interfacial reactants are not

formed on the Al substrate surface.

Further prolonging the soldering time to 120 s, as shown in Fig. 5g and 5h, the network structure of the fine particles becomes indistinguishable as the quantity of particles increases and the feature size of the network diminishes. The Ni skeletons are fragmented into strip-like forms, and the reticulated structure of Ni-foam can no longer be identified. Deep dissolution cavities are formed on the Al substrate surface, indicating that significant dissolution of the Al substrate is taking place during the soldering process. Moreover, larger-sized α -Al particles are not found to be accumulating near the Al substrate, indicating that the dissolved Al element is uniformly distributed in the soldering seam.

3.3 Microstructure evolution mechanism of joints

As it has been demonstrated that Ni_3Sn_4 IMC layers are formed on Ni skeleton surfaces, the fine Ni_3Sn_4 particles should originate from the reaction layer formed at the Sn/Ni interface. In fact, an Ni/Sn IMCs layer is known to be a good element diffusion barrier layer, which can retard the mutual diffusion of Ni and Sn and thus decrease the further growth rate of the IMC layer at the interface [24,25]. However, this situation may be changed when the Sn/Ni liquid/solid interfacial reaction couple is exposed to ultrasonic waves. Previous studies [26,27] reported that acoustic cavitations tended to be created at the liquid/ solid interface, since the pits or cracks located on the solid substrate could decrease the nucleation energy of acoustic cavities. This indicates that acoustic cavitations are likely to form at the Sn/Ni interface during the ultrasonic soldering process. The micro-jets and shock waves induced by acoustic cavitations can create extremely high pressure and shearing force on the Ni_3Sn_4 IMC layer [28,29]. Combined with the shearing force created by the ultrasonic wave-induced streaming effects [10], the Ni_3Sn_4 IMC layer could easily be stripped from the Ni skeleton surface and broken into tiny particles. Thus, the Ni/Sn interfacial reaction rate can be effectively enhanced by the continuous removal of the IMC layer, and the stirring action of the molten Sn solution with the help of ultrasonic waves. As a result, fine particles of Ni_3Sn_4 form in large numbers and are homogeneously distributed in the solder seam.

At the same time, the large quantity of fine Ni_3Sn_4 particles are helpful to the refine the

α -Al phase and distribute the α -Al particles homogeneously in the solder seam. Our previous studies [21] found that the solubility of Al in the Ni_3Sn_4 phase could reach 5.8 at. %, indicating that the α -Al phase has good bonding activity with the Ni_3Sn_4 phase. During the ultrasonic soldering process, the initially formed Ni_3Sn_4 particles can act as heterogeneous nucleation sites for the precipitated α -Al phase, and induce the refining of α -Al particles. Fig. 7 shows the EDS mapping results of the solder seam in the Al/Ni-Sn/Al joint ultrasonically soldered at 280 °C for 60 s. Corresponding to the cross-section image of soldering seam (Fig. 7a), the distributions of Al, Ni and Sn elements are presented in Fig. 7b-7d. The large particles can be identified to be α -Al phase, while the long strip-like phase is part of the broken Ni skeleton. Large amounts of Al and Ni elements come from the Al substrates and Ni skeleton, and are distributed uniformly in the Sn matrix under the combined effects of acoustic cavitation and streaming. This further demonstrates that the formation of significant amounts of the Ni_3Sn_4 phase may be helpful to promote the homogeneous distribution of the α -Al phase in the Sn matrix. Thus, a homogenous microstructure is obtained by inducing the formation of numerous fine Ni_3Sn_4 particles under the action of ultrasonic waves.

The Ni_3Sn_4 phase has the same density as the Sn solder, and, although an open structure, the reticulated Ni skeleton can inhibit the floating of particles. Moreover, fine particles would be expected to float at a lower velocity than large size particles [30]. Thus, the amount of particles formed can be stable in their homogenous distribution in the soldering seam when the ultrasonic vibration stops. During solidification of the solder seam, the Ni_3Sn_4 and α -Al particles are pushed to the growth front of the β -Sn phase, and thus, when the soldering seam is solidified, the particles are mainly aggregated at the grain boundaries of the β -Sn phase, resulting in the 3D discontinuous network structure seen. With increasing soldering time, the largely formed Ni_3Sn_4 and α -Al particles may induce refinement of the β -Sn phase. One effect comes from the initially formed Ni_3Sn_4 and α -Al particles acting as heterogeneous nucleation sites for the β -Sn phase, increasing the nucleation rate and refining the β -Sn phase grain size [31], and as another effect, the initially formed particles accumulated at the solidification front of the β -Sn phase can inhibit the growth of β -Sn phase [32]. As a result, the measured

diameter of the structures in the networks that fine particles surround gradually decreases with prolonging of the soldering time.

3.4 Interfacial bonding mechanism of joints

Since there are no apparent reaction layers or diffusion layers found at the Sn-based solder/Al substrate interface under SEM examination, the bonding mechanism of the joint is unclear. To observe the interfacial microstructure between Al substrate and Ni-foam/Sn composite solder in more detail, TEM characterization was performed. A FIB sample was cut from the composite solder/Al substrate interface in the joint ultrasonically soldered at 280 °C for 120 s (Fig. 8a). A bright field image of the interface is shown in Fig. 8b. The gray region marked as “I” was identified to be the Al substrate according to the electron diffraction pattern shown in Fig. 8c, while the dark region marked as “II” was identified to be Sn-based solder, according to the electron diffraction pattern shown in Fig. 8d. A transition layer with a thickness of approximately 150 nm can be found at the interface (Fig. 8b). The EDS analysis results reveal that this layer is composed of 38.50 at.% Al and 61.49 at.% O, which is consistent with the stoichiometry of Al₂O₃. Interestingly, no Ni or other elements are discovered in this layer, although Ni and Al elements have strong reaction activities as reported in our previous studies [21]. The high-resolution transmission electron micrographs (HRTEM) image of the “III” region marked in the transition layer is shown in Fig. 8e; this region exhibits an amorphous structure. The HRTEM image of the “IV” region, as shown in Fig. 8f, indicates that there is a nano-crystalline γ -Al₂O₃ grain surrounded by the amorphous Al₂O₃ in the transition layer. Thus, it can be identified that the transition layer located between the composite solder and the Al substrate is mainly composed of amorphous Al₂O₃ with some crystallized γ -Al₂O₃ mixed in it. **As a result, a metallurgic bonding between Sn-based solder and Al substrate is achieved by the amorphous Al₂O₃ interlayer. The disordered arrangement of atoms in the amorphous Al₂O₃ interlayer can realize a smooth transition of the lattice from Al substrate to Sn-based solder. The same results can also be found in recent studies that an amorphous interlayer realized the metallurgic bonding between Sn-based solder and ZrO₂ ceramics [33], as well as Sn-based solder and graphene [34].**

It is known that, during the ultrasonic-assisted soldering process, the ultrasonic waves inducted in the molten solder can lead to sonochemistry effects between the molten solder and the metal substrate [11,33]. Some amorphous layers were reported to have been created at the liquid Sn-based solder/solid substrate interface during ultrasonic soldering or brazing [33,34]. However, it seems that the formation of an amorphous Al_2O_3 interlayer in this study does not correlate with the interfacial non-equilibrium reaction environment induced by ultrasonic cavitation. Since the oxide films on the Al substrate surfaces are stripped under the combined effects of acoustic cavitation and streaming effects, this means that the amorphous Al_2O_3 interlayer should be formed after the ultrasonic vibration process. Previous studies [35-37] reported that up to a certain critical oxide-film thickness, a thin amorphous Al_2O_3 film on Al substrate was found to be more stable than the corresponding crystalline Al_2O_3 film due to the relatively low formation energy of amorphous Al_2O_3 film at temperatures lower than 300 °C. Thus, the acoustic cavitation effects initially help to remove the oxide film on the Al substrate surface during the ultrasonic vibration process, providing an intimate metal-to-metal contact for the molten Sn-based solder and the Al substrate. Then, an amorphous (as opposed to crystalline) Al_2O_3 layer forms on the Al substrate surface after the ultrasonic vibration stops as the bare Al substrate oxidises. However, the amorphous oxide-film exhibits a slow growth rate and limited thickness at low temperatures, because the chemical diffusion of elements across an amorphous Al_2O_3 layer is negligibly small [35]. The reported thickness of amorphous Al_2O_3 films attached to fresh Al substrates is always less than 10 nm at temperatures lower than 300 °C [35,38], which is much thinner than that found in this study. The reason why an amorphous Al_2O_3 layer can grow to such a thickness as seen here is ascribed to the deposition reaction of Al and O on the Al substrate.

Since Al substrates are dissolved in the soldering seam, an ample supply of Al for the formation of the amorphous Al_2O_3 layer is available by diffusion from α -Al particles to the Al substrate surface. However, there may be insufficient oxygen for the formation of a thick amorphous Al_2O_3 layer, since the solubility of elemental O in molten Sn solder is extremely low at 280 °C [39]. Previous studies [33,40,41] reported that oxygen tended to segregate at

the liquid/solid interface during the ultrasonic soldering process, which induced the formation of O rich phases on the surfaces of Al alloys and ceramic substrates. This phenomenon can also be found in Fig. 4d, where that some large O-rich α -Al particles are found near the Al substrate surface. It has been suggested that oxygen enters the Sn-based solder by diffusing from the atmosphere/liquid solder interface during the soldering process [42]. However, the non-equilibrium segregation mechanism of oxygen at the liquid/solid interface under ultrasonic action is still unclear. It seems that the oxygen accumulation at the liquid/solid interface may be attributed to gas absorbing effects induced by acoustic cavitation [43]. During the expansion stage of the acoustic cavities, the gas dissolved in the molten Sn solder may diffuse rapidly into the cavities under negative pressure. When the cavities collapse, the oxygen that has this precipitated out cannot diffuse completely back into the molten Sn solder, because of the low contact area. Since acoustic cavities tend to form near the liquid/solid interface [26,27], segregation of oxygen towards the Sn/Al interface occurs. Thus, when the ultrasonic vibration stops, Al and O elements in molten Sn-based solder can precipitate on the bare Al substrate surface at high enough levels to result in the rapid growth of an amorphous Al_2O_3 layer. As a result, a smooth transition of the lattice from Al substrate to Sn-based solder is formed through the amorphous Al_2O_3 interlayer, which achieves the metallurgic bonding between Al substrate and Sn-based solder.

The thermodynamic stability of an amorphous Al_2O_3 film on an Al substrate surface is correlated with the environment temperature and oxide-film thickness [35]. When the thickness of the amorphous Al_2O_3 layer is small, the bulk Gibbs free energy of the amorphous oxide film can be compensated for by the lower sum of the surface and interfacial energies of the Al substrate/ amorphous Al_2O_3 film systems, and the amorphous Al_2O_3 structure is relatively stable [35,38]; otherwise, the amorphous Al_2O_3 layer would be unstable and may be partially transformed into crystalline phase because of the increase of bulk energy. The critical thickness of amorphous Al_2O_3 on Al alloy surface is several nanometers at temperatures lower than 300 °C [35]. Since the thickness of amorphous Al_2O_3 interlayer found in this study is about 150 nm (Fig. 8b), the amorphous phase has a tendency to crystallize even if the

crystallization temperature is not reached. This may be the reason why nano-crystalline γ - Al_2O_3 phase is found in the amorphous Al_2O_3 transition layer. It should be noted that the detailed nucleation and growth kinetics of the γ - Al_2O_3 in the thick amorphous Al_2O_3 layer at a temperature lower than 280 °C is still unclear. This will be further studied and published in our future work.

3.4 Mechanical properties of the joints

The microstructure achieved will affect the bonding strength of the joints formed. To evaluate the effect of the morphology of the solder seam and the formation of the amorphous Al_2O_3 interlayer on the mechanical properties of Al alloy joint, shear tests were performed. Fig. 9 shows the shear strength of Al/Ni-Sn/Al and Al/Sn/Al joints ultrasonically soldered for different times. It can be seen that the Al/Ni-Sn/Al joints exhibit much higher shear strength than that of Al/Sn/Al joints. The shear strength of Al/Ni-Sn/Al joints increases sharply from 42.37 MPa at 5 s to 71.05 MPa at 60 s, then increases more steadily to 72.19 MPa at 120 s. Over the same range, the shear strength of Al/Sn/Al joints is 37.44 MPa at 5 s, then increases to 53.01 MPa at 60 s, and goes up to 56.59 MPa at 120 s. The shear strength of an Al/Ni-Sn/Al joint is only 4.93 MPa larger than that of an Al/Sn/Al joint when soldered for 5 s, but this difference increases to 18.04 MPa when the soldering time is increased to 60 s. These results demonstrate that the shear strength of Al alloy joint can be improved to an appreciable degree by using Ni-foam/Sn composite solder.

Fig. 10 shows the cross-section images after failure in shear of Al/Sn/Al and Al/Ni-Sn/Al joints ultrasonically soldered for 5 s and 60 s, respectively. For the Al/Sn/Al joint soldered for 5 s (Fig. 10a-10c), cracking mainly occurs along the Al/Sn interface (Fig. 10a) then partially turns into the solder layer (Fig. 10b), and finally reaches the Al/Sn interface on the other side of the joint (see Fig. 10c). The same fracture mode can also be found in the Al/Ni-Sn/Al joint soldered for 5 s (Fig. 10d-10f). These indicate that the Sn-based solder/Al substrate interfaces are the weakest areas in these joints. It may be difficult to completely remove the initial oxide films on the Al substrate surface with the help of acoustic cavitations when the ultrasonic soldering time is short. This can be confirmed by the observation in Fig. 10a and Fig. 10d that

the Al substrates are only partially coated with Sn based solder. The weak interfacial bonding between Sn-based solder and the Al substrate is the main reason for the low strength of joints soldered for 5 s.

As to Al/Sn/Al and Al/Ni-Sn/Al joints ultrasonically soldered for 60 s, as shown in Fig 10g-10i and 10j-10l, the Al substrates were found to be completely coated with the Sn-based solder, and the failure mainly occurred in the solder layer. The improved interfacial bonding that results from extending ultrasonic soldering time is helpful to increase the strength of joint. Once reliable bonding between the Sn-based solder and the Al substrate has been obtained, the shear strength of the joint is mainly affected by the microstructure of the solder seam. For the Al/Sn/Al joint, the dissolution of Al and the precipitation of Al particles in the Sn-based solder are main reasons for the strength improvement of the joint that is found when prolonging soldering time from 5 s to 60 s. However, the strengthening mechanism of the Al/Ni-Sn/Al joint may be more complicated than that of the Al/Sn/Al joint. The 3D continuous interpenetrating structure of Ni-foam accompanied by the 3D discontinuous network distribution of very fine particles are most probably behind the rapid strength improvement of the Al/Ni-Sn/Al joint. Moreover, the dissolution of Al and Ni in the Sn-based solder, coupled with the refinement of the β -Sn phase are also beneficial to the mechanical properties of joint. Thus, a higher shear strength difference was obtained between the Al/Sn/Al and Al/Ni-Sn/Al joints because of the multiple strengthening factors operating in the Al/Ni-Sn/Al joints.

On further increasing the ultrasonic soldering time from 60 s to 120 s, the solubility of Al in Sn would be saturated and the precipitated Al tends to form larger accumulated α -Al particles, and thus the strengthening effects caused by the dissolution of Al are decreased for the Al/Sn/Al joint. For the Al/Ni-Sn/Al joint, the Ni skeletons begin to fracture and become fragmented under the acoustic cavitation-induced erosion effects. As a result, the rate of shear strength increase for Al/Sn/Al and Al/Ni-Sn/Al are both reduced.

4. Conclusion

An open cell Ni-foam reinforced Sn-based composite solder was used to ultrasonically

solder UFG 7075 Al alloy. Reliable bonding of UFG 7075 Al alloy was realized by ultrasonically soldering using this composite solder. The soldering seam of the Al/Ni-Sn/Al joint was mainly composed of Sn-based solder, Ni skeletons, α -Al particles and Ni₃Sn₄ IMC. Ni₃Sn₄ and α -Al particles formed in discontinuous network structures in the soldering seam, the diameter of which was decreased with increasing ultrasonic soldering time. This was mainly ascribed to the combined effects of acoustic cavitation and streaming, such as accelerating the Ni/Sn interfacial reaction, refining the Ni₃Sn₄ and α -Al particles, and homogenizing the solder seam. An amorphous Al₂O₃ interlayer was found at the composite solder/Al substrate interface, which allowed a smooth lattice transition from the Al substrate to the Sn-based solder. The formation of an amorphous Al₂O₃ layer was attributed to the low formation energy of the amorphous phase and segregation of oxygen to the liquid solder/Al substrate interface induced by acoustic cavitation. The shear strength of Al/Ni-Sn/Al joint was increased with prolonging ultrasonic soldering time. An average shear strength of 71.05 MPa was obtained for Al/Ni-Sn/Al joints soldered at 280 °C for 60 s, which was approximately 18.04 MPa higher than that soldered with the same parameters but using pure Sn solder. The strength improvement of Al/Ni-Sn/Al joint was mainly ascribed to the strengthening effects of continuous Ni skeletons, as well as very fine Ni₃Sn₄ and α -Al particles formed around discontinuous networks.

Acknowledgments

This work was supported by National Nature Science Foundation of China (No. 51605357). The authors would like to thank Dr. Zhi Zhang from Huazhong University of Science and Technology for FIB sample preparation and TEM characterization. **Dr. Yong Xiao is grateful to the funding of Sir SY Chung Fellowship in The University of Sheffield.**

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