Effect of Sn interlayer on mechanical properties and microstructure in Al/Mg friction stir lap welding with different rotational speeds

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Abstract

The effect of Sn foil layers on the mechanical properties and microstructure of friction stir lap welded 5052 aluminium alloy and AZ31B magnesium alloy was explored in this study. Models of numerous joints were set by different rotational (700, 900, 1100, 1300 and 1500 rpm) and welding speeds (50 mm min\textsuperscript{-1}). Mg/Al dissimilar lap joints with and without Sn interlayer were produced by friction stir lap welding. The results suggest that Mg\textsubscript{2}Sn intermetallic compounds formed instead of Mg\textsubscript{17}Al\textsubscript{12} and Mg\textsubscript{2}Al\textsubscript{3} intermetallic compounds. In direct welding, the joint are only connected by metallurgical bonding between atoms (Mg/Al). In solder joints with Sn interlayer, the joint is connected by the combined effect of metallurgical bonding between atoms (Mg/Sn, Al/Sn) and interface mechanical coupling. For the joint with Sn interlayer, the maximum fracture load of the joint with the Sn interlayer reached 3.72 kN at a rotational speed of 900 rpm. As the rotational speed is increased from 1300 rpm to 1500 rpm, the Sn content on the the advancing side and the retreating side is more, resulting in more Mg\textsubscript{2}Sn crystal content, increased crack content. The joint performance gradually decreases. For the joint without a Sn interlayer, the microscopic morphology was a river-like pattern, which was characterized by brittle fracture. For a joint with a Sn interlayer, the microscopic morphology contained micro-dimples and a small quantity of inclusions, which were characterized by mixed fracture.

1. Introduction

Aluminium is widely used in various fields, including the aerospace, high-speed train, and high-speed ship industries, because it is lightweight, exhibits corrosion resistance, and is easily formed [1, 2]. Magnesium, with a great specific strength, low density, decent heat transfer properties, and respectable electronic shielding performances, is promising for the formation of lightweight materials [3–5]. As high-quality lightweight materials, Mg and Al alloys are increasingly important for the development of lightweight vehicles. Therefore, the successful welding of dissimilar Al and Mg can successfully take advantage of the properties of both metals and has important practical significance [6–8].

At present, many works on the joining of Mg to Al have been performed. However, the combined strength is usually not high. The main reason is that massive brittle Mg-Al intermetallic compounds (IMCs) form at the weld [9–13]. Chen et al [11] found that an intermetallic compound transition layer consisting of Mg\textsubscript{2}Al\textsubscript{12} and Mg\textsubscript{2}Al\textsubscript{3} was formed during friction stir welding (FSW) of AZ31 Mg and AC4C Al. These IMCs occurs because the peak temperature in the welding process exceeds the eutectic temperature of Mg and Al, leading to the eutectic reaction. Mohammadia et al [13] joined the AZ31B Mg and 6061 Al by changing the process parameters. The results instructed that the change of process parameters changed the morphology and distribution of IMCs. However, the formation of Mg-Al IMCs cannot be avoided. Azizieh et al [14] studied the FSW of Al100 and Mg AZ31. The experimental results showed that Mg-Al IMCs generated in the stir zone. The greater the heat input,
Table 1. The chemical composition of material (wt.%).

| Materials | Al   | Mg   | Si   | Fe  | Cu  | Mn  | Cr  | Ni  | Zn  |
|-----------|------|------|------|-----|-----|-----|-----|-----|-----|
| 5052H32  | Bal. | 2.20 | 0.25 | 0.40| 0.10| 0.10| 0.15| —   | 0.10|
| AZ31B    | 2.50 | Bal. | 0.10 | 0.005| 0.05| 0.2 | —   | 0.005| 1.00|

the greater the IMCs content and the hardness of the joint. Mg-Al IMCs become the source of cracks. Changing the welding process and adding other metal layers to control the development of brittle Mg-Al IMCs have become two important areas of research. To date, a number of researchers have studied these by adding an intermediate layer at the interface of laser welding [11], resistance spot welding [15, 16], tungsten inert gas welding [17], or compound welding methods [18, 19] to overpower the formation of Mg-Al IMCs, thereby improving the joint performance.

Sn is a low-melting metal element. As seen from binary diagrams, it can be found that Sn could interact with Mg and Sn-Al is infinitely solid solution. In addition, the selection of Sn in the current study was also based on the findings that Sn improved the wettability of Mg and Al alloys during the welding process [20]. Sn has been extensively used as an additive element in Mg/Al joining to expand the joint strength [20–24]. For example, Patel et al [22] investigated improving the strength of Mg-Al dissimilar joint by Sn interlayer throughout ultrasonic spot welding. The results show that the brittle Mg17Al12 IMCs was successfully eliminated. The joint strength gets a significant improvement. Dai et al [25] considered the influence of Sn interlayer in arc-assisted ultrasonics seam welded Mg/Al joint. The results showed that Sn-based solid solution and Mg2Sn was formed in the joint, instead of Mg-Al IMCs. The maximum peak load of the joints increases 30% over the joint without Sn interlayer, which up to 1.3 KN. However, few works has been reported in the joining of Al to Mg with a Sn interlayer by FSW. In the process of FSW, determining whether the addition of Sn foil can optimize the weld structure and build up the joint mechanical properties is worth examining. Thus, the main purpose of the current work was to examine the effects of a Sn interlayer on the mechanical properties and microstructural evolution of friction stir lap welding (FSLW) joints of the Mg to Al alloys.

2. Materials and experimental procedure

The base materials hired in this work were 5052 Al and AZ31 Mg sheets with sizes of 300 mm × 75 mm × 3 mm. The Sn foil used in the experiment was high-purity Sn with size of 300 mm × 40 mm × 0.1 mm. Table 1 is the chemical compositions of two materials. Before the welding process, the surfaces of the Mg alloy, Al alloy, and Sn foil were cleaned with sandpaper and an alcohol solution. The Sn foil was placed between the Al and Mg plate, and the two plates were fixed by a clamp. The welding configuration was a lap joint. The Al sheet attended as the superior sheet, and the lap joint area was 30 mm in length according to our previous works [26]. All welds were made by FSW-TS-S08 machine (As shown in figure 1). H13 tool steel was made for a threaded pin [27]. The diameters of the shoulder and the pin were 10 and 3.5 mm, respectively. The length of the pin was 2.8 mm. The drop depth of the revolving tool shoulder and the slope angle with respect to the z-axis were 0.2 mm and 2°, respectively. According to the report by Mishra et al [28], there is the following equation (1) between the welding temperature and the rotation speed during the FSW.

\[ \frac{T}{T_m} = K \left( \frac{w}{v \times 10^4} \right)^\alpha \]

Where T is the supreme welding temperature; \( T_m \) is the melting point of the material; K and \( \alpha \) are the constant; w is the rotational speed; v is the welding speed. With the increase of the rotational speed, the maximum temperature in the weld nugget (WN) gradually increases, and the welding heat input slightly increases. This article mainly studies the effect of Sn interlayer on the properties and microstructure of joints under different heat input. On the basis of pervious works [26], the welding process parameters were set as follows: the welding speed was 50 mm min \(^{-1}\), and the rotational speed was 700–1500 rpm.

After welding, the welds were cut into metallographic samples by the wire cutting technology. After grinding and polishing, the tasters were etched with a picric acid solution (10 ml of acetic acid, 6 g of picric acid, 100 ml of ethanol, and 10 ml of demonized water) for 30 s. Scanning electron microscopy (SEM) with energy-dispersive x-ray spectroscopy (EDS) was used to observe and analyse the microstructures of the FSW joints and fracture surfaces after the tensile tests. The joint strength was determined by a CMT5105 tensile tester. The tensile specimen size was 120 mm × 20 mm. The tensile testing was performed at a loading speed of 2 mm min \(^{-1}\). Each process prepared three tensile samples. X-ray analyses were done on the fracture surfaces of the Al and Mg sides.
of joints with and without Sn interlayers after the tensile shear tests. In addition, a Vickers hardness tester was used to amount the hardness of the weld. The load was 0.1 N, and the loading time was 10 s.

3. Results

3.1. Macrostructure

Figure 2 shows the cross-sections of the joints for dissimilar process parameters. A sound weld without evident welding defects was attained. For the FSLW joint without an Sn interlayer, with the growth in the welding heat input, there was almost no difference in the interface morphologies of the lap joint for 900–1300 rpm rotation speeds. However, when the rotation speed reached 1500 rpm, the appearance changed significantly and exhibited a bowl-like shape containing hook-shaped defects [29]. This was a consequence of overlarge weld heat input. When the welding heat input was too tall, the yield stress of the plate material was small on the tool shoulder region, resulting in rapid flow of the upper plater material. The material of the lower plate moved vertically upward, resulting in evident hook defects [30].

Figures 2(b), (d), (f), (h) shows the cross-sections of joints with Sn interlayers, where the shapes of the joints did not change significantly with the growth in the rotational speed. All of them exhibited an ‘arc’ shape [31]. The height of the weld nugget (WNH) means the vertical distance between the bottom of the weld nugget (WN) and the bottom plate, as shown by the arrow in figure 2. With the rotation speed increasing from 900 rpm to 1500 rpm, the WNH value of the direct welded joint decreases as shown in figure 3 black line. This result is due to the fact that, with the increase of welding rotation speed, the heat input increases continuously, the metal flow speed speeds up, and the area of the WN area increases, resulting in the decrease of WNH value. This implies that WNH can be used to describe the fluidity of WN metal. The smaller WNH value is, the better the fluidity of the WN will be.

For the joints with Sn interlayer, the change of process and WNH value conforms to the change law of direct welding. Comparing the WNH value of the joint with and without a Sn interlayer under the same parameter, it can be found that the WNH value of the joint with Sn interlayer is smaller. This difference was even more pronounced at the welding rotation speed of 900 rpm. The WNH value decreasing from 2.81 mm for direct welding to 2.47 mm after adding Sn interlayer. This indicates that under the same process, the fluidity of WN metal becomes better after adding Sn interlayer. This phenomenon is attributed to the addition of Sn foil. The highest temperature in the WN is higher than the melting point of the Sn foil (232 °C) during FSLW [32]. Because of its low melting point, the Sn melts under the action of FSW heat, acting as a lubricant and reducing the flow stress of the metal in the WN area. Ji et al [33] found similar phenomenon when they studied the improvement of friction stir welding of Mg and Al by adding zinc foil.
3.2. Microstructure

Figure 4 displays the microstructure of Mg/Al dissimilar joint without a Sn interlayer at 900, 1300, and 1500 rpm. There was a transition region that was different from the base material. As the rotational speed enlarged, the width of the transition zone continued to expand. It can be found that the mutual presence of Al and Mg across the reaction layer from the elemental analysis of line 1 in figure 4(b) as shown in figure 4(d), signifying that the IMC interlayer formed at the Al/Mg boundary during FSW. During the FSW process, the formation of IMCs is related to the heat generation \[13, 34\]. According to equation (1), it can be found that when the welding speed is 50 mm min \(^{-1}\), with the rotation speed gradually increasing from 900 rpm to 1300 rpm and
1500 rpm, the heat input of the welded joint is increasing, which intensifies the eutectic reaction of Mg and Al, leading to the formation of more IMCs \([34, 35]\).

Figure 5 shows the interface microstructures on the advancing side (AS) of the joints with Sn interlayers from the rectangular regions shown in figures 2(b)–(f). As exposed in figure 6, a eutectic microstructure was present at the interface between the Al and Mg. In addition, the IMC layer thickness increased with increasing rotation speed.

Figure 6 shows a higher enlargement SEM image from the rectangular regions marked in figure 5(c). The transition region was mainly composed of a large number of blocky grey and acicular substances, as shown in figures 6(b) and (c). The compositions of the blocky grey matter and acicular substances, indicated by point...
labelled A, B, and C in figures 6(a)–(c), are shown in figures 6(d)–(f). The blocky grey matter phase was a Sn solid solution. The acicular substances were Mg$_2$Sn and a Sn-based solid solution. Furthermore, it can be found that the needle-like Mg$_2$Sn was connected by cracks to form a dense crack network from figure 8(c). This phenomenon was attributed to the reaction of Mg and Sn. During the synthesis of Mg$_2$Sn, a large amount of heat was released, and thermal stress caused cracks. Li et al. [23] studied the addition of Sn through transient liquid phase bonding of Mg/Al. The result showed a transition zone composed of Mg$_2$Sn at the joint, but there were no micro-cracks in the excessive zone. The reason for this difference was the strong shear and thermal stress effects of FSW. The specific causes of cracks will be further elaborated in the discussion section.

Figure 7 shows the elemental line scan results for the transition zone of the joints with and without Sn foil at 900 rpm (Square in figure 2). The transition zone consisted of a dark band and a light band, and the two were separated by a white transition layer that varied in thickness. The chemical compositions of the top and bottom layers were analysed by EDS. The results show that the top and bottom layers were composed of Mg$_5$Al$_2$ (56.93% Mg and 43.07% Al (at.%)) and the Mg$_2$Al$_3$ (40.83% Mg and 59.17% Al (at.%)), respectively. The interface appeared as a distinctive white colour. In addition, the transition layer of the Mg-Al IMCs was relatively thin. Figures 7(c) and (d) shows that there was a clear platform for the joint with a Sn interlayer. The results confirmed that Mg-Sn IMCs formed with a thickness of 2–4 μm.

In addition, there was a small crack band happening the Al side of the joint (figure 7(a)). The source of this micro-crack was mainly the variance in thermal expansion coefficients of Mg and Al [36]. It may have also been due to the principle solidification in the welding process, where the stress concentration caused by the rapid cooling caused micro-cracks to form [11, 37].

3.3. Mechanical properties and fracture morphology

Figure 8 shows the tensile shear fracture load outcomes of joints with and without Sn interlayers under different process parameters. As shown in figure 8, for two different processes, the tensile shear fracture load of the joint first improved and then reduced as the rotational speed increased. For joints without a Sn interlayer, the
maximum load was only about 3.24 kN, which occurred when the rotation speed was 1300 rpm. For the joints with Sn interlayers, the maximum load of the welded joint was achieved: 3.72 kN at 900 rpm.

Figure 9 is the fracture morphologies of the Mg/Al FSLW with and without Sn interlayers at a rotational speed of 900 rpm. For both weld processes, the break happened at the interface transition layer, not the crack (figures 9(a) and (b)). Figures 9(a) and (c) shows that when the Sn foil was not added, the microscopic morphology exhibited a river-like pattern, which was characterized by brittle fracture. The surface of the Al/Mg joint with a Sn interlayer contained micro-dimples and a small quantity of inclusions, which were characterized by mixed fracture. After the tensile lap shear tests for the joint obtained at 900 rpm, XRD investigation was carried out on the breakage surfaces of the Al and Mg sides, as shown in figures 9(g) and (h). For the joint with a
Sn interlayer, Mg$_2$Sn was noticed, which established the accuracy of the above EDS line-scan analysis. However, it is not detect any Mg$_7$Al$_{12}$ IMC at the fracture surface on the Mg side (figure 9). Even though a reaction layer was detected at figure 9(a), the quantity of the phase was beyond the resolution of XRD [10, 38, 39]. Table 2

**Figure 9.** Fracture path of direct welding (a); Fracture path of FSW joint with Sn interlayer (b); SEM image of fracture surface of (c) Al side of direct joint, (e) Mg side of direct joint, (d) Al side of FSW joint with a Sn interlayer, and (f) Mg side of FSW joint with Sn interlayer. XRD pattern of the (g) direct joint and (h) FSW joint with a Sn interlayer.
shows the results of the EDS point examination corresponding to figure 9. The Mg-Al IMCs can be detected on Mg side of direct joint. On Mg side of direct joint, Mg-Sn IMCs attached with some pure Mg can be found. The results are in line with the break locations discussed before.

4. Discussion

In order to better explain the reason of adding Sn interlayer to improve the property of joint, the atomic diffusion diagram of welded with and without Sn interlayer at 900 rpm is given here. Figure 10(f) shows an abridged general view of atomic diffusion at the interface. The line 1 was the atomic evolution diagram of direct joint. With the progress of welding, the Mg and Al atoms were mutually diffused by the dual effects of heat and downforce. Finally, a Mg-Al IMCs interlayer was formed and the interface of Mg/Al behaves as a straight line (figure 7(a)).

The line 2 was the atomic evolution diagram of FSW joint with a Sn interlayer. At the beginning of soldering, the Sn foil melted and became liquid due to the low melting point. Under the complex action of the axial force and vertical force of the pin, the liquid Sn undergoes 'wave motion' (figure 10(e)). At the same time, a portion of the Sn was squeezed out of the weld zone, and another Sn entered the Mg matrix. The upper Al atoms is no longer in contact with Mg due to the obstruction of the interlayer. Due to the special motion state of liquid Sn, the flow of Al is affected, resulting in a complex z-shaped state of the Al-Sn bonding interface. In combination with Al-Sn binary alloy phase diagram [40], it is known that Al-Sn IMCs is difficult to form. Only a simple

Table 2. EDS results of points 1–5 marked in figure 9.

| Point | Composition (at.%) | Possible phase |
|-------|--------------------|----------------|
|       | Mg  | Al  | Sn   |                  |
| 1     | 57.44 | 42.56 | —    | Mg17Al12         |
| 2     | 65.05 | 34.95 | —    | Mg17Al12         |
| 3     | 56.74 | 43.26 | —    | Mg17Al12         |
| 4     | 65.80 | 7.06  | 27.15 | Mg-Mg2Sn         |
| 5     | 82.3  | 7.28  | 10.42 | Mg               |

Figure 10. Interface of FSW joint with Sn interlayer at 900rpm (a); EDS results (b)–(e); Schematic diagram of atomic diffusion principle for weld formation (f).
mechanical occlusion appears at the interface junction. The difference in the electronegativity values of Mg and Sn is the largest (the electronegativity values of Sn, Al, Mg and are 1.96, 1.61, and 1.3, respectively.). Sn and Mg had high affinities. This is evident from the scan results of the surface of figure 7(d). During the process, As the Sn and Mg had high affinities, the Mg atoms in the lower layer will preferentially diffuse to the Sn-dense locations. Due to the wave motion of Sn, the Mg atoms in the lower layer also tend to wave motion. Meanwhile, the eutectic reaction between Mg and Sn occurred readily to form Mg2Sn because the temperature of the eutectic reaction (L → Mg2Sn + Mg) was very low (203 °C) [40], which enriched the transitional area structure.

Comparing the two lines, it can be found that the reasons for the improvement of the joint performance were as follows. First, the formation of Mg-Al brittle IMCs was inhibited. Second, in direct welding, the joint properties are only connected by metallurgical bonding between atoms (Mg/Al). In solder joints with Sn interlayer, the joint performance is connected by the combined effect of metallurgical bonding between atoms (Mg/Sn, Al/Sn) and interface mechanical coupling.

Figure 11 shows an abridged general view of atomic diffusion at the interface with and without Sn interlayer. For Mg/Al FSW without a Sn interlayer, as the rotation speed increased, the welding heat input increased, and the eutectic reaction (L → Mg2Al12 + Mg 437 °C/L → Mg3Al5 + Al 450 °C) occurred more intensely. As the welding heat input continual to rise, the quantity of IMCs continued to increase (figures 4, 11(a)), and the excess layer continued to widen. Oikawa et al.[41] found that the joint performance of dissimilar welds is affected by the thickness of the IMC layer formed at the interface. The joints are connected by metallurgical bonding between the atoms. The IMC transition layer is a factor that provides strength and also causes deterioration of joint performance. At 1300 rpm, the joint performance is maximum. Too small and too thick IMC layers result in poor joint performance.

Figure 11(b) shows an abridged general view of atomic diffusion at the interface with Sn interlayer from the AS shown in figures 2(f)–(h) at different positions at 900, 1300, and 1500 rpm. For the joint with a Sn interlayer, as the rotation speed was sequentially increased from 900 to 1300 rpm and then to 1500 rpm, although the heat of the joint continuously increased, the Mg-Al IMCs was not formed at the joint due to the hindrance of the Sn foil.

With the increase of the rotational speed, a large amount of liquid Sn in the WN was stirred to AS and RS (figure 5), resulting in the actual content of Sn on both sides of the WN gradually increasing. On the other hand,
the reaction between Mg and Sn intensified on both sides of WN as the increment of heat input, and the content of Mg–Sn increased significantly.

The welding and solidification process has gone through two stages. First, the liquid–solid stage, due to the extremely high stability of Mg–Sn (melting point reach at 770 °C), the Mg2Sn content is already high, and a small amount of contact occurs between adjacent Mg2Sn crystals. As the temperature decreases, Sn crystals increase and grow. After entering the solid–liquid phase (Second stage), most of the liquid Sn metal has solidified into crystals. At this time, the basic characteristic of plastic deformation is the reciprocal movement of crystals, and the crystals themselves will also undergo some deformation. When Sn crystals and Mg2Sn crystals alternately grow to form a dendritic skeleton, a small amount of liquid Sn remains in the form of a thin film between the crystals, and is therefore difficult to flow. Because the liquid film has low resistance to deformation, the deformation will be focused on the intergranular region where the liquid film is located, making it a weak link. Due to the large stirring force peculiar to friction stir welding and the strong wave motion of liquid Sn caused by this, there is a sufficient welding stress at the joint, and between the plastic deformation of the crystal, the grain boundary where the liquid film is located will have priority cracking, and finally solidification cracks, needle-shaped distribution (figure 6(b)). As the rotation speed is increased from 1300 rpm to 1500 rpm, the Sn content on both sides of the WN is more, resulting in more Mg2Sn crystal content, more possibility of contact between neighbouring crystals, and increased crack content (figures 5(f), (i)).

In summary, the addition of Sn foil could produce Mg2Sn IMCs, and the formation of Mg–Al brittle IMCs was inhibited. However, there were a large number of micro-cracks in the transition zone, and the performances of the joints were not significantly improved compared to the joints without Sn interlayers under the same process parameters. In this article, the main role of Sn was to increase the ultrasonic spot welding of Mg–Al. Welding energy at optimal joint performance reduced from 1250 J to 1000 J.

5. Conclusion

(1) Sound welding joints were produced among the 5052 Al and AZ31B Mg alloys through FSW with and without Sn interlayers. The interfacial microstructures of the joints with Sn interlayers contained the Mg–Sn IMCs, rather than of the brittle Mg–Al IMCs in the joints without Sn interlayer. For the joint without a Sn interlayer, the microscopic morphology was a river-like pattern, which was characterized by brittle fracture. For a joint with a Sn interlayer, the microscopic morphology contained micro-dimples and a small quantity of inclusions, which were characterized by mixed fracture.

(2) The maximum lap shear failure load of the joints without Sn interlayers was achieved at a rotation speed of 1300 rpm. However, the maximum lap shear failure load with a Sn interlayer was achieved at 900 rpm. The addition of Sn interlayer further led to energy saving since the rotational speed required to achieve the tensile shear fracture load decreased from 1300 rpm to 900 rpm in the Mg–Al dissimilar joint. It is worth mentioning that Patel et al [42] compared the effects of adding Sn foil to the joint on the performance of the joint during the dissimilar ultrasonic spot welding of Mg–Al. Welding energy at optimal joint performance reduced from 1250 J to 1000 J. Conclusions are similar.

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