Microstructural characterization and mechanical properties of diffusion-brazed AZ91C magnesium alloy to 316L stainless steel

M. Ataei, A. Shamsipur*, S. E. Mirsalehi

Department of Mining and Metallurgical Engineering, Amirkabir University of Technology (Tehran Polytechnic), 424 Hafez Ave, Tehran 15875-4413, Iran

Received 22 April 2019, received in revised form 8 May 2019, accepted 15 May 2019

Abstract

Dissimilar transient liquid phase (TLP) bonding of AZ91C magnesium alloy to 316L stainless steel was carried out using a pure copper filler metal. The bonding process was conducted in two stages where TLP bonding of 316L to Cu interlayer was first performed and then followed by bonding AZ91C to the other side of the copper interlayer. It was indicated that the temperature rise enhanced the inter-diffusion of elements at the AZ91C/Cu interface and encouraged the formation of hard intermetallic Mg$_2$Cu compound. Refinement of Cu grains was observed and attributed to the induced strain from the fixture. This phenomenon assisted the enhancement of mechanical properties of the joints. By increasing the temperature from 495 to 530°C, the shear strength revealed an upsurge of more than 20%. The presence of Mg$_2$Cu was detected as the main contributor to the increased shear strength and microhardness at elevated temperatures.

Key words: diffusion brazing, AZ91C/316L joints, mechanical characteristics, elevated temperatures, microhardness, shear strength

1. Introduction

Energy consumption is viewed to be largely dependent upon the weight of components and, hence, a solution is to utilize lightweight metals instead of heavier ones [1]. In many constructions, using hybrid elements is expected to be one of the best ways for weight reduction and as a consequence, environmental protection and energy preserving [2, 3]. For this purpose, bimetal joints with a lightweight component and steel are taking attention because of particular features of steel metals and lightweight material [4]. Because of the following explanations, steel/Mg joints come to be increasingly attractive compared to other dissimilar joints. Magnesium alloy as the lightest metal with high specific strength has attracted many structural applications, particularly in automotive and aerospace industries [5–7]. The use of magnesium alloys additionally decreases weight in comparison with aluminum alloys. Presently, the most applicable alloy in the industry is steel. Therefore, to the production of hybrid lightweight structures, joining of steel to magnesium is favorable, it consecutively extends the use of magnesium alloys in different areas [8]. In this sense, fusion welding of magnesium to steels is not favorable due to considerable differences between their melting points [9]. Solid-state welding such as friction stir welding or ultrasonic welding has also been investigated [10–13]. However, their application is limited due to poor deformation ability of magnesium alloy.

Transient liquid phase (TLP) bonding is a promising method to join dissimilar metals with different melting points [1, 14]. TLP bonding is a combination of both brazing and diffusion bonding eliminating the detrimental influence of conventional joining methods [15, 16]. In this process, an interlayer is preplaced between the base metals so that the inter-diffusion between the two sides results in the elimination of the interface and, thus, bonding of two base metals [17–19]. Also, considering the difference in metallurgical features of Fe and Mg, they are immiscible and based on the Fe-Mg binary diagram, no intermediate phases can be formed. Consequently, it is important to insert intermediate elements that can have substantial solid

*Corresponding author: tel.: (+98) 2164542978; e-mail address: Shamsipur@aut.ac.ir
Table 1. Chemical composition of AZ91C and 316L base materials (wt.%)

<table>
<thead>
<tr>
<th>Material</th>
<th>Fe</th>
<th>Mg</th>
<th>Al</th>
<th>Cu</th>
<th>Zn</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Mn</th>
<th>Si</th>
<th>Ce</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>Bal.</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>17.3</td>
<td>10.1</td>
<td>2.1</td>
<td>1.9</td>
<td>0.75</td>
<td>0.2</td>
</tr>
<tr>
<td>AZ91C</td>
<td>0.0008</td>
<td>Bal.</td>
<td>8.7</td>
<td>–</td>
<td>0.7</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>0.28</td>
<td>0.019</td>
</tr>
</tbody>
</table>

Table 2. Chemical compositions of etchants used for the base materials and filler metal

<table>
<thead>
<tr>
<th>Material</th>
<th>Etchant composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L stainless</td>
<td>15 ml HCL + 5 ml Nitric acid + 70 ml Water</td>
</tr>
<tr>
<td>steel</td>
<td>6 ml Nitric acid + 122 ml HCL + 8.5 g Ferric chloride + 2.4 g Cupric chloride + 122 ml Ethanol</td>
</tr>
<tr>
<td>AZ91C</td>
<td>5 gr Picric acid + 10 ml Acetic acid + 80 ml Ethanol</td>
</tr>
<tr>
<td>Pure Cu foil</td>
<td>NH₄OH + H₂O₂</td>
</tr>
</tbody>
</table>

Fig. 1. (a) SEM micrographs of diffusion bonded Cu/316L at 495 °C, (b) magnified graph of areas pointed in Fig. 1a.

Fig. 2. Optical micrographs of TLP bonded AZ91C/Cu/316L at 495 °C (a) and 530 °C (b).

In the present work, double-stage TLP bonding of AZ91 magnesium alloy to 316L stainless steel was conducted by using pure copper as the filler metal, so this
study aims to investigate bonding mechanism and mechanical properties of AZ91C/Cu/316L joints.

2. Materials and methods

AZ91C magnesium alloy and 316L stainless steel with the chemical compositions shown in Table 1 were considered as the base materials. They were sectioned into 2 mm × 20 mm × 120 mm specimens. The faying surfaces were then ground by 60–2000 grade silicon carbide papers. Diamond suspension with 1 μm particle size was used as the polishing solvent. To fully eliminate surface contaminant, all the specimens were ultrasonically cleaned in an acetone bath for 15 min. A pure copper foil of 100 μm thickness was used as the interlayer.

Double-stage diffusion brazing was carried out in a tube furnace with a vacuum of 10⁻⁴ torr. In the first stage, the diffusion bonding of 316L stainless steel to pure copper foil was performed at 930 °C for 15 min. The free surface of copper foil was then polished and assembled to AZ91C part; afterward, the whole assembly was fixed with a fixture of ~ 5 MPa pressure. TLP bonding as the second stage was conducted at various bonding temperatures of 495 and 530 °C for 8 min and then followed by furnace cooling.

Metallographic and scanning electron microscopes

Fig. 3. (a) SEM image of diffusion brazed joint AZ91C/Cu side and (b) is magnified morphology of (a).

Fig. 4. Metallographic images of AZ91C/Cu bonded at 495 °C (a) and 530 °C (b), (c) is 500 × magnification of (b).
were utilized for microstructural investigation. To observe each type of metals in the bonding area, various etchants, as shown in Table 2, were applied. The further microstructural observation was accomplished by a Mira 3-XMU scanning electron microscope equipped with EDS elemental analysis detection of the chemical composition of different zones in the bonding area. Microhardness and tensile strength testing across the bonding area were carried out in accordance with ASTM-E92 and ASTM D1002, respectively [21, 22].

3. Results and discussion

SEM micrograph of the bonding area on the 316L/Cu side is illustrated in Fig. 1. The highlighted arrows indicate the interface between two sides, where it is usually referred to as the inter-diffusion region. 316L base metal was fully bonded to Cu foil without any voids, discontinuities and unbonded areas. The Fe-Cu inter-diffusion region was measured to be as small as 1 µm. Indeed, the bonding parameters were sufficient for achieving a sound joint between the 316L base material and pure Cu interlayer, with a negligible distinct interface. The soundness of Cu/304L under identical bonding conditions was also confirmed by Yuan et al. [23].

Figure 2 illustrates the optical micrographs of specimens bonded at 495 and 530 °C. As can be seen, the interface zone is free from voids and unbonded area. A continuous phase was formed at the interface zone at both temperatures. According to Mg-Cu phase diagram, the eutectic transformation occurred at 487 °C resulted in the formation of Mg₂Cu compound [24]. The thickness of Mg₂Cu increased considerably from 8 to 30 µm by rising temperature from 495 to 530°C. This can be related to the ease of diffusion at elevated temperatures. At temperatures higher than that of Mg-Cu eutectic transformation (487°C), the interdiffusion of Mg and Cu and their subsequent reaction formed the transient liquid phase which then solidified to Mg₂Cu intermetallic compound and Mg(Al, Cu) solid solution at the interface zone.

Interestingly, the grain size reduction of Cu side with an average grain size of 3 µm was observed in the vicinity of the interface zone and increasing the joint strength and grain boundary bonding [25], as shown in Fig. 3. Since Cu foil was in a pure state,
such grain refinement could not be achieved via heat treatment; hence, this can be mainly attributed to the strain induced by the fixture.

Further investigation of the bond between AZ91C and Cu indicated the existence of $\alpha$(Mg), $\beta$(Mg$_{17}$Al$_{12}$) and $\alpha + \beta$ phases on AZ91C side and Mg$_2$Cu intermetallic in the interface zone, as shown in Fig. 4, and it is consistent with the literature [26]. SEM morphologies of the joint made at 495 and 530°C are shown in Fig. 5. As seen, in the interface zone highlighted in yellow, the distribution of both Cu and Mg elements is evident, which indicates the inter-diffusion of these elements. However, by rising bonding temperature to 530°C, the diffusion of Cu to AZ91C side increased, forming thicker Mg$_2$Cu intermetallic compound (see Fig. 6).

Various zones of the AZ91C/Cu joints made at 495 and 530°C are illustrated in Fig. 7 and the chemical compositions of these zones are given in Table 3. As can be seen, zone A contains 56.44% Mg, 25.08% Al, 18.48% Cu, and 0% of other elements.
Al and 18.48% Cu; therefore, Mg(Al, Cu) solid solution and Mg$_2$Cu intermetallic compound might be formed via eutectic reaction in this zone [27]. Zone p was found to be rich in Al, indicating the formation of Mg(Al, Cu) solid solution. The analysis of the white zone B in Fig. 7 showed a considerable amount of Ce element. Ce, as a rare earth element can form Mg$_{11}$Ce$_3$ intermetallic and reduce $\beta$-Mg$_{17}$Al$_{12}$ via increasing corrosion resistance of the substrate [28].

In zone C, the presence of oxygen is noticeable so that this zone is susceptible to oxide compounds which are detrimental to mechanical properties of the joint. The existence of oxide layers is mainly due to the primary stage of AZ91C production. Zones D and E consist of a combination of $\alpha$(Mg) and $\beta$(Mg$_{17}$Al$_{12}$) phases. The areas designated by s were found to be enriched in Mg and Al and can be suggested to be $\beta$(Mg$_{17}$Al$_{12}$).

Microhardness gradient across the joint area is shown in Fig. 8. The highest value was obtained in the zone between Cu interlayer and AZ91C, where the Vickers values of 247 and 445 were measured for joints made at 495 and 530°C, respectively. The higher hardness at 530°C as compared to that of 495°C can be attributed to a larger amount of inter-diffusion between Cu and AZ91C sides encouraging the formation of a hard intermetallic compound, Mg$_2$Cu. The hard-
ness for Cu layer increased to 77.1–77.6 HV whereas the hardness of pure Cu foil was 62 HV. On the 316L side, the hardness values became uniform without any fluctuation in both specimens. The hardness values on AZ91C substrate were slightly higher for the sample made at 530°C, which can be related to a larger amount of Cu diffusion to AZ91C side. Ultimate shear strengths of 316L/Cu/AZ91C joints are shown in Fig. 9. The shear strength values of 58 and 47 MPa were measured for samples bonded at 530 and 495°C, respectively. The higher shear strength at elevated temperature is most possibly related to the greater amount of Mg2Cu at the interface of bond made at 530°C.

4. Conclusions

In this study, microstructural and mechanical properties of double-stage diffusion brazed AZ91C to 316L joints were investigated. The most important results are as follows:

1. A sound interface between the 316L substrate and Cu foil was obtained without the presence of any defects or unbonded areas.

2. It was found that the Mg2Cu eutectic compound was formed at the interface of AZ91C/Cu, while its thickness increased by rising temperature from 495 to 530°C, as a result of the ease of diffusion.

3. Grain refinement of Cu was observed and related to the induced strain of the fixture. This grain refinement subsequently improved the mechanical properties of the joint.

4. The mechanical evaluation revealed higher hardness and shear strength at the elevated bonding temperature of 530°C, which can be attributed to the greater amount of hard Mg2Cu intermetallic formed in this condition.

References

[22] International A. Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading (Metal-


