



# TLP bonding of Ti-6Al-4V and Mg-AZ31 alloys using pure Ni electro-deposited coats



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## ABSTRACT

Transient liquid phase (TLP) bonding of Mg-AZ31 and Ti-6Al-4V alloys was performed using pure thin Ni electro-deposited coat interlayer (12  $\mu\text{m}$ ). The effect of bonding temperature, time and pressure on microstructural developments and subsequent mechanical properties across joint interface was studied at a temperature range from 500 to 540 °C, bonding time from 1 to 60 min and bonding pressure from 0 to 0.8 MPa. The mechanisms of bond formation varied across the joint region, with solid-state diffusion dominant at the Ti-6Al-4V interface and eutectic diffusion at the Mg-AZ31 interface. Joint microstructure was examined by scanning electron microscopy (SEM), and energy dispersive spectroscopy (EDS). X-ray diffraction (XRD) was used to detect the formation of intermetallic phases at the fracture surface. The maximum joint shear strength of 61 MPa was obtained at a temperature of 520 °C, 20 min and at a bonding pressure of 0.2 MPa. This joint strength was three times the bond strength reported for joints made using adhesives and represents 50% of the Mg-AZ31 alloy shear strength.

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## 1. Introduction

Transient liquid phase (TLP) bonding is a process which involves the use of an interlayer placed between the two bonding surfaces; the assembly is held at a temperature above the eutectic temperature of the interlayer and base metal for a sufficient time until the joint zone solidifies isothermally. Ideally, when autogeneous bonds are made with the base alloys, a sufficient hold time can result in a homogenised interface with a continuous grain microstructure across the joint region. Yan et al. (2004) outlined TLP bonding is highly desirable especially when traditional fusion welding techniques fail to achieve a joint.

One of the most challenging aspects of TLP bonding is optimising the process parameters, such as bonding time (Kenevisi and Mousavi Khoie, 2012), bonding temperature (Bakhtiari et al., 2012), and bonding pressure (Saha and Khan, 2007). Although Saha and Khan (2007) showed that bonding pressure is necessary to hold the joint assembly and is essential for joint formation, there has been little work done on the effect of bonding pressure on joint

formation. TLP bonding has been successfully applied to different alloys, for example: Al-7075 and Ti-6Al-4V alloys (AlHazza et al., 2010), titanium alloys (Cam et al., 2008), and magnesium alloys (Jin and Khan, 2012). MacDonald and Eagar (1992) indicated that the optimisation of TLP bonding parameters is essential to achieve an ideal joint. In this study, TLP bonding was utilised to fabricate joint between Ti-6Al-4V and Mg-AZ31 alloys. The effect of bonding temperature, bonding time and bonding pressure on microstructural developments and the corresponding changes in mechanical properties were investigated.

## 2. Materials and experimental procedure

Mg-AZ31 and Ti-6Al-4V were commercially available in the form of 10 mm diameter rods from Goodfellow (Cambridge, UK) and William Gregor (London, UK), respectively. For TLP bonding, 5 mm specimens were cut, ground and polished down to 1000 grit. For Ni electro-deposition on Ti-6Al-4V, the Ti alloy surface was soak cleaned with E-Kleen 102-E for 10 min at a temperature of 60 °C and stirring of 200 RPM. Followed by cleaning with E-Kleen 129-L for 2 min at a temperature of 75 °C and stirring of 200 RPM, and finally rinsing with tap water for 30 s and then acid pickling with 31% HCl for 2 min at room. The electro-deposition of Ni coating was carried out using Watt's plating solution prepared by dissolving: 250 g

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$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ , 45 g  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ , 35 g  $\text{H}_3\text{BO}_3$ , and 1 g Saccharin in 1 L of distilled water. The current density was set at  $5 \text{ A/dm}^2$ , with the pH level maintained at 3.5, the coating temperature kept at room temperature, agitation of the bath was sustained with a magnetic stirrer at 200 RPM, and the coating time was set for 15 min. TLP bonding was carried out in a vacuum chamber with a pressure of  $4 \times 10^{-4}$  Torr (0.053 Pa). Induction heating was used with heating rate of  $50^\circ\text{C/min}$ , and a K-type thermocouple was inserted in a hole made in the Mg-AZ31 sample at a distance of 2 mm from the bonding surfaces. The joint interface was examined by scanning electron microscopy (SEM) (JEOL JXA 8200) equipped with energy dispersive X-ray spectroscopy (EDS) for composition analysis. X-ray diffraction (XRD) was also used to analyse intermetallic phases at the fracture surfaces. Vickers micro-hardness tests were carried out for all specimens using a 50 g load within  $600 \mu\text{m}$  on each side. The shear strength measurements were carried out using a 25 kN load-cell tensile testing machine (Tinius Olsen H25KS) at a cross head-speed of  $0.5 \text{ mm/min}$ , three samples were tested for each parameter. The shear testing has been included as a more critical test for assessing joints with interlayers or coatings. This is because the coating at a joint under tensile loading will show a “plastic constraint” effect which may result in an inadequate strength evaluation, and can give a UTS value close to 90% of the parent alloy although the contact area may only be 60%, and in particular the potential deleterious effect of oxides and/or intermetallic layers may be underestimated.

### 3. Results and discussion

#### 3.1. Effect of temperature

The SEM micrograph in Fig. 1 illustrates the joint interface for bonds made at 20 min, 0.2 MPa using a  $12 \mu\text{m}$  coat at different bonding temperatures from 500 to  $540^\circ\text{C}$ . At a bonding temperature of  $500^\circ\text{C}$ , the joint width was  $17 \mu\text{m}$  and the joint interface intermetallics were difficult to observe. Increasing the bonding temperature from  $510^\circ\text{C}$  to  $520^\circ\text{C}$  resulted in an increase in joint width to  $73 \mu\text{m}$ . Furthermore, increasing the bonding temperature to  $530^\circ\text{C}$  and  $540^\circ\text{C}$  resulted in a joint width of  $78 \mu\text{m}$  and  $91 \mu\text{m}$ , respectively. This increase was attributed to the formation of more eutectic liquid which results in the dissolution of Mg-AZ31 interface as the bonding temperature increased. It was observed that the size of phases and intermetallics formed inside the joint region varies with bonding temperature. The size of intermetallics decreases as the bonding temperature increases from  $510$  to  $520^\circ\text{C}$ . No change was seen for a temperature of  $520$  to  $530^\circ\text{C}$ , and the size of these intermetallics increased as the bonding temperature increased to  $540^\circ\text{C}$  (see Fig. 1). These results show that the joint region was heterogeneous, with the formation of various intermetallic phases due to the presence of a concentration gradient for Mg diffusion during the bonding process. Raghavan (2009) reported the Al–Mg–Ni isotherm at  $427^\circ\text{C}$  and this coincides with these results, where different phases such as  $\tau = \text{Ni}_2\text{Mg}_3\text{Al}$ ;  $\text{Mg}_2\text{Ni}$ ; (Mg) may form simultaneously in the Al–Mg–Ni system. Fig. 2 shows the SEM micrograph and EDS point analysis for bonds made at  $530^\circ\text{C}$ , 0.2 MPa,  $12 \mu\text{m}$  and 20 min. The EDS analysis of Mg-AZ31 interface showed strong peaks for Mg with 97% Mg, 2% O and 1% Al. The white phases that developed inside the joint zone dispersed at both interfaces of Ti-6Al-4V and after the high concentration Mg layer showed the same phase composition with 66% Mg, 7% Al, and 27% Ni. The dark phases showed 80% Mg and 20% O, this high concentration of oxygen is expected to affect the joint strength. AlHazzaa et al. (2010) reported that the oxide formation deteriorated the joint strength in TLP bonding. The differences in the coefficient of thermal expansion between the oxide and the non-oxidised Mg phases caused

the dark region to develop micro-cracks which may lead to further crack propagation and joint failure (see Fig. 2).

#### 3.2. Effect of time

Fig. 3 shows the SEM micrograph of the microstructure at the joint interface for bonds made at  $520^\circ\text{C}$ ,  $12 \mu\text{m}$  coat thickness, 0.2 MPa bonding pressure for 1–60 min. For a 1 min hold, the joint zone was not uniform and part of the Ni coat had not melted within the joint region. An average joint width of  $46 \mu\text{m}$  was produced and new phases and intermetallics were observed at the bonded zone. Increasing the bonding time to 5 min resulted in a uniform joint zone and two reaction layers were observed with a total width of  $60 \mu\text{m}$ . This increase was attributed to the increase in the amount of eutectic formed for a longer bonding time. An increase in bonding time from 5 to 20 min resulted in an increase in joint width. The joint width was measured to be 63 and  $73 \mu\text{m}$  for a bonding time of 10 and 20 min respectively. The intermetallics formed were finer, smaller in size and agglomerated at the joint centerline when compared with a bond made at 5 min (see Fig. 3c and d). The joint width decreased as the bonding time increased from 20 to 60 min. The joint width was 69 and  $70 \mu\text{m}$  for bonding time of 30 and 60 min respectively. This observation suggested the onset of isothermal solidification at a bonding time of 20 min. Fig. 4 shows the SEM micrograph and EDS point analysis for bonds made at  $520^\circ\text{C}$ , 0.2 MPa,  $12 \mu\text{m}$  for 5 min. The EDS analysis of the reaction layer at Mg-AZ31 interface showed a composition of 96% Mg, 2% Al and 2% O for 5 min, and three distinctive areas were formed inside the joint zone; first a rich Mg zone was seen inside the bond region with 98% Mg and 2% O. The second region was a large grey “island” with 68% Mg, 25% Ni and 7% Al. The third region was the small agglomerated “islands” with 44% Mg, 33% Ni, 18% Al, and 5% O. The formation of a metallurgical bond at the Ni/Mg-AZ31 interface is expected to occur due to the formation of this intermetallic zone at the bond interface.

#### 3.3. Effect of pressure

Fig. 5 illustrates the SEM micrograph for bonds made at  $520^\circ\text{C}$ ,  $12 \mu\text{m}$ , 20 min and bonding pressure from 0 to 0.8 MPa. Bonding specimens without external pressure resulted in a joint width of  $88 \mu\text{m}$ . In Fig. 5(a) it was noticed that the intermetallics formed were larger in size compared to intermetallics formed when a higher bonding pressure was used during the bonding process. Upon increasing the bonding pressure to 0.2 MPa the joint width was measured at  $73 \mu\text{m}$ . The decrease in joint width on increasing the pressure was anticipated because of the greater “squeezing action” on the eutectic liquid. A joint width of  $102 \mu\text{m}$  was noticed at 0.4 MPa bonding pressure and the size of the intermetallics formed appeared to be finer than those observed at a 0.2 MPa bonding pressure. This increase in bonding pressure from 0.2 to 0.4 MPa was expected to increase the surface to surface contact and enhanced the diffusion of Ni into the Mg-AZ31 and Ti-6Al-4V alloys, hence resulting in joint zone width increase. However, upon increasing the bonding pressure to 0.6 MPa and 0.8 MPa, the average joint width was 69 and  $66 \mu\text{m}$  respectively. This decrease in joint width was attributed to the outward pushing effect of the eutectic liquid from the joint interface during the bonding process. Hence the amount of eutectic liquid remained at the joint was minimal. The width of the reaction layer formed towards Mg-AZ31 was noticed to increase as the bonding time increased. The EDS analysis of the reaction layer at the Mg-AZ31 interface showed a higher concentration of Ni and Al at the grain boundaries, suggesting that the diffusion of Ni and Al towards the Mg-AZ31 alloy was dominated by grain boundary diffusion (see Fig. 6). The point analysis showed that at the middle of the grains, the chemical composition

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