

Kinetics of joint formation during diffusion induced solid-state bonding of titanium and magnesium alloys

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Abstract

This study explores the diffusion bonding of titanium to magnesium. The microstructural evolution and intermetallic compounds formed within the bond region are studied. The microstructure and composition of the bonds were characterized using a Scanning Electron Microscope (SEM) equipped with Energy Dispersive Spectroscopy (EDS). The mechanical properties of the bonds were evaluated using a micro-hardness test to determine the hardness variation across the joint region, while shear strength measurements were used to assess the joint strength. The results indicated that solid-state diffusion bonding of Ti6Al-4V and Mg (Az31) lead to the formation of Ti₃Al and Ti₂Mg₃Al₁₈ reaction layers within the joint region. Evaluation of the mechanical properties of the bonds revealed that the bond strength increased with increase bonding time to 116 MPa at a bond time of 1 hr. The joint formation and strength were attributed to dispersion strengthening due to the nucleation of dispersed intermetallic compounds at the joint interface.

Keywords; Diffusion Bonding, Titanium, Magnesium, Intermetallic, Solid-State

I. INTRODUCTION

The growing concerns regarding fuel consumption within the aerospace and transportation industries make the development of fuel-efficient systems a significant engineering challenge [1]. Currently, materials are selected because of their abilities to satisfy engineering demands for good thermal conductivity, strength to weight ratio, tensile strength, and corrosion resistance. Titanium is attractive for use in aerospace and transportation because, in addition to satisfying the mechanical and thermal requirements, its passivation behaviour allows for its application in industries requiring excellent corrosion resistance [2]. At room temperature titanium has a hexagonal close-packed (hcp) crystal structure which is referred to as the alpha phase, however, at temperatures above 883 °C Ti undergoes a polymorphic transformation which changes the alpha phase to a beta phase having a body-centred cubic (bcc) structure. Magnesium, on the other hand, maintains its hcp crystal structure throughout

temperature fluctuations. Additionally, Mg is the lightest structural metal available and like Ti has excellent thermal conductivity and strength-to-weight ratio. However, there are several limitations to using magnesium, for example, the chemical reactivity of the material. Additionally, chemical reactions between Mg and other elements can lead to the formation of intermetallic compounds, such as Mg₁₇Al₁₂ which forms from a eutectic reaction between Mg-Al alloys or Ti₂Mg₃Al₁₈ which forms in ternary systems of Ti, Mg and Al [3][4]. The application of the Hume-Rothery rules to these two alloys shows that while both Mg and Ti have hcp crystal structure at room temperature the differences in the atomic radii, electronegativity and the number of valence electrons significantly limits the solubility of Mg in Ti and restricts the types of compounds that can form at the interface [5].

Multi-material structures provides the most efficient design solution to lightweighting engineering challenges. The utilization of several metals in the construction of hybrid (multi-material) structures are constrained by the ability of the available welding/joining technologies to join dissimilar materials together, even within a single class of material (e.g. metals) [6][7][8]. Fusion welding process is the method used for joining similar metals. However, the application of any fusion welding technique to join dissimilar metals presents additional difficulties, the first one being; the reaction of the two metals at the joint interface can create compounds that may have unfavourable properties [9][10]. Another limitation to the use of these alloys includes galvanic corrosion due to differences in electrode potential values between the materials. Because of these challenges, dissimilar welding of advance alloys such as titanium and magnesium is limited, even though these materials hold significant potential for product development.

Dissimilar joining of titanium and magnesium has been previously attempted using transient liquid diffusion bonding as a way of limiting intermetallic formation at the bond interface [11]. The process utilizes an interlayer which forms a liquid either by direct melting due to heating beyond the melting point of the interlayer or the formation of a eutectic reaction between the interlayer and the base metal [12]. The method has been shown to promote joining by removing surface oxide and ensuring metal-to-metal contact. Sheng et al. [9] created a Ti/Mg lap joint using tungsten inert gas

Digital Object Identifier (DOI):
<http://dx.doi.org/10.18687/LACCEI2020.1.1.287>
ISBN: 978-958-52071-4-1 ISSN: 2414-6390

welding [13][14]. Joint formation and the strength developed in diffusion bonds have been shown to relate to the width of the reaction layer formed at the interface during joining. The thickness of the reaction layer formed during diffusion bonding can be estimated by the parabolic power law shown in Equation 1

$$d = kt^n \quad (1)$$

Where d is the width of the reaction layer, k is the reaction rate factor, t the diffusion time, and n the time exponent. The use of the parabolic law suggests that the growth kinetics of the intermetallic layer is controlled by inter-diffusion (volume diffusion); therefore, diffusion time is estimated to be $t^{1/2}$ where $n=0.5$. Alternatively, if the growth kinetics were controlled by interfacial diffusion, the time exponent would be $n=1$ [15]. If grain growth takes place in the reaction layer, then n becomes smaller than 0.5 for a boundary diffusion rate-controlling process. If n becomes equivalent to 0.25, the grain growth obeys the power law which means that the mean grain size of the intermetallic layer increases in proportion to the square root of the bonding time [16].

This study investigates the dissimilar solid-state joining of titanium to magnesium and studies the microstructural evolution and intermetallic compounds formed within the bond region that promotes a metallurgical bonding between Ti and Mg. The nature of the layers formed at the interface was also discussed in terms of the thermodynamic and kinetic factors influencing the reaction layer formation and the impact on the bond strength.

II. MATERIALS AND METHODS

A. Materials

In this study, commercially available Titanium (Ti-6Al-4V) and wrought magnesium (AZ31) were used to prepare the Ti-Mg couples by solid-state diffusion bonding. The compositions of the materials studied are listed in Table 1. The properties of Ti and Mg are listed in

Table 2 and shows the significant differences between the properties of the two alloys studied.

Table 1: Chemical composition (wt. %) of the materials used in this study

	Ti	Si	Mn	Mg	Zn	Ag	V	Al
Ti-6Al-4V	Bal.	0.15	0.23	2.7	0	0.86	4	6
Mg (AZ31)		0.03	0.37	Bal.	1.04	0	0	2.66

Table 2: Properties of the materials used in this study [12].

Properties	Ti	Mg
Crystal Structure	HCP to 885 °C BCC above the beta transus	HCP
Thermal Conductivity W/m-K	22	160
Thermal Expansion Coefficient K ⁻¹	8.6 x 10 ⁻⁶	2.48 x 10 ⁻⁵
Melting Point °C	1670	650
Specific Heat Capacity J/g-°C	0.5263	0.1080

B. Sample preparation and bonding process

The samples were prepared for bonding by cutting the Ti and Mg alloys to a dimension of 10 × 10 × 5 mm. A hole was drilled in the Ti sample to a depth of 3 mm at 1 mm from the bonding interface. The bonding surfaces were prepared to 2500 grit SiC finish and subsequently polished to 1 μm using a particle impregnated carrier paste and then cleaned in an acetone bath. To study the effect of interlayer composition, the titanium samples were electrodeposited with Ni/Al₂O₃ coating. The deposition process was described in an earlier study [17],[18].

The specimens were assembled at room temperature and placed on the lower platen within the induction coil and an ungrounded k-type thermocouple inserted into the hole located approximately 1 mm from the joint interface. When 2 × 10⁻³ Torr vacuum was achieved, the assembly was heated to the bonding temperature (500 °C).

The specimens were brought to the joining temperature at a heating rate of 65 °C/min and then held at that temperature for bonding times ranging from 10-60 minutes. At the end of the bonding process, the power was switched off, and the specimens were cooled to room temperature in vacuum. The bonded samples were sectioned transversely to the bond-line by an abrasive saw and mounted in Bakelite. The mounted specimens were prepared according to ASTM standard B253. Each sample was ground progressively on silicon carbide papers from 240–800 grit, followed by a final polish to 1 μm finish. Kroll's reagent was used as the etchant to reveal the grain structure.

C. Mechanical testing

The shear strength of the Ti/Mg bonds was determined using a single lap-joint which was loaded into a shear test fixture and pulled in tension by a GUNT (WP 300 Universal materials tester, Germany) tensile testing machine. The samples were loaded to ensure that each specimen experienced pure shear stress across the bond interface. The bond strength was calculated by dividing the maximum load by the bond area. For each bonding condition, three specimens

were tested, and the average value used to determine the shear strength (bond strength). Micro-hardness testing was performed on the cross-section of the joints using a Leitz micro-hardness tester. Indentations were made at 100 μm spacing using a diamond tip indenter loaded with 0.1 kg and applied for 30 s, after which the length of the diagonals was measured, and the hardness number calculated using Equation 1.

D. Microstructural evaluation

Microscopic examination of the bonded joints was performed using a Leitz optical microscope and an Oxford (FEI Quanta 400 Oxfordshire U.K.) scanning electron microscopy (SEM) equipped with INCA x-sight x-ray. Quantitative compositional analyses were carried out using energy dispersive spectroscopy (EDS). X-ray diffractometry (XRD) measurements were done using a Bruker XRD machine adjusted to the following settings: 40 kV, 40 mA, step-size 0.05 $^\circ$ for 2-Theta ranging from 10 $^\circ$ to 100 $^\circ$ and measuring 1 second per step.

III. RESULTS AND DISCUSSION

E. Microstructural characterization

Fig. 1 (A) shows the SEM micrograph of the sample bonded for 10 minutes. The joint appears to present good bonding between the Ti and Mg sample due to the absence of void, cracks or porosities. When the bonding time was increased to 30 minutes, it was observed that a reaction layer formed on the Mg-side of the joint interface, as shown in Fig. 1. Further increase of the bonding time to 60 minutes resulted in an increase of the width of the reaction layer, as shown in Fig. 2(B). Magnified sections of the joint interface for the sample bonded for 60 minutes is presented in Fig. 2(C) and (D). The compounds formed at the interface appears to be in the form of dispersed particles approximately 20 μm in length. EDS analysis of joint region as a function of bonding time using compositional maps confirmed the inter-diffusion of Ti, Mg and Al during the bonding process. The maps show that as the bonding time increase the depth to which Mg diffused into the Ti-side increased (see Fig. 3). However, given the differences in the diffusion coefficient of Ti and Mg as presented in Table 2, it appears that rate of diffusion of Ti into Mg is prolonged due to the difference in the interdiffusion depth of Mg and Ti as indicated in the elemental maps. Additionally, the results also showed that Al diffuses from both the Mg and the Ti alloy to the interface.

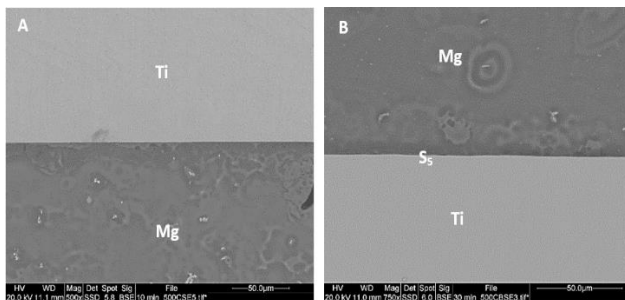


Fig. 1: Solid-state diffusion bonding of Ti and Mg for; (A) 10 minutes and (B) 30 minutes.

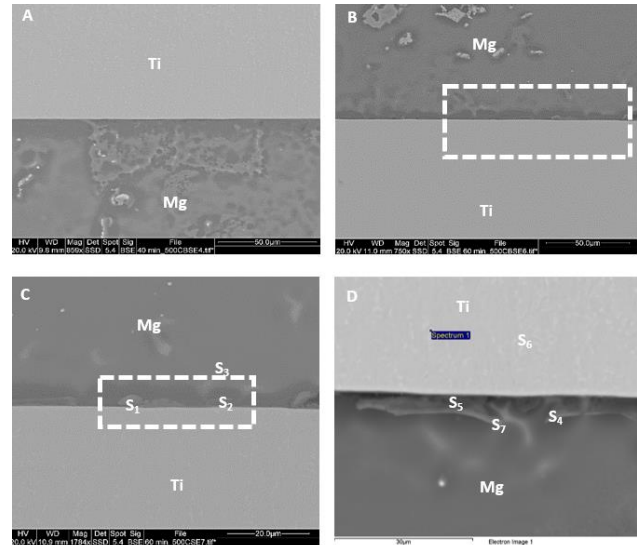


Fig. 2: Solid-state diffusion bonding of Ti and Mg for; (A) 40 minutes; (B) 60 minutes; (C) Detailed view of the reaction layer at the bond interface; (D) imperfection at the bond interface showing compositional variation.

Further EDS point analysis of the samples bonded for 30-60 minutes, revealed the formation of several compounds at the bond interface. The composition of each compound identified and the likely molecular formula is shown in Table 3. The results show that when the sample is held at the bonding temperature; Al reacts with Ti and Mg to form TiAl_3 and $\text{Ti}_2\text{Mg}_3\text{Al}_{18}$ intermetallic compounds at the Ti interface. The $\text{Mg}_{17}\text{Al}_{12}$ intermetallic compounds appear to have formed at the Mg grain boundaries. The $\text{Mg}_{17}\text{Al}_{12}$ compound is believed to have been produced by a eutectic reaction between Al and Mg.

The joint formation was attributed to metallurgical bonding driven, leading to the formation of TiAl_3 and $\text{Ti}_2\text{Mg}_3\text{Al}_{18}$. Additionally, the differences in the properties of Ti and Mg are believed to contribute to achieving good contact during the bonding process. It is expected that the differences in the melting temperature of the two alloys that at the bonding temperature of 500 $^\circ\text{C}$ the Mg sample with plastically deform to ensure complete contact with the Ti sample. An analysis of the surface roughness of the polished surfaces of the Mg and Ti samples bonded revealed Ra values of 41 μm and 5.3 μm for the Ti and Mg samples, respectively. The differences in the surface roughness of the alloys would ensure the as Mg is deformed at the bonding temperature the Ti asperities would protrude into the surface of the Mg-sample.

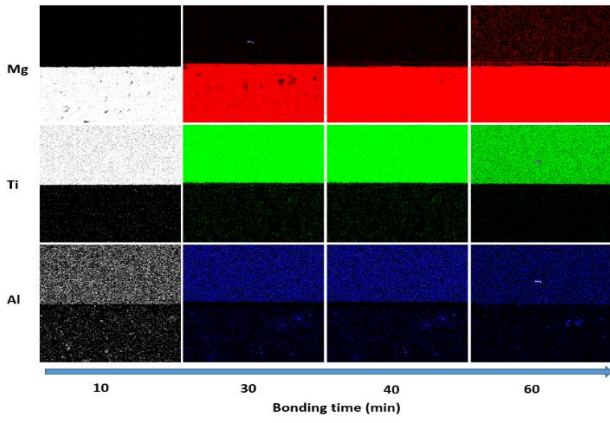


Fig. 3: EDS compositional maps of the diffusion bonds as a function of bonding time.

Table 3: EDS analyses showing the compositional variation across the interface of a sample bonded for 60 minutes

Spectrum	Ti	Mg	Al	O	Phase
S ₁	9.06	46.81	3.08	31.2	Mg + Ti ₂ Mg ₃ Al ₁₈
S ₂	25.84	47.10	2.87	33.28	MgO + Mg ₁₇ Al ₁₂
S ₃	0.22	89.08	1.80	4.15	Mg
S ₄	54.49	8.80	0.37	21.87	Ti + MgO
S ₅	89.03	1.71	5.88	0	Ti ₃ Al
S ₆	89.97	0.55	5.83	0	Ti ₃ Al
S ₇	56.33	12.39	0.47	21.23	Ti + MgO

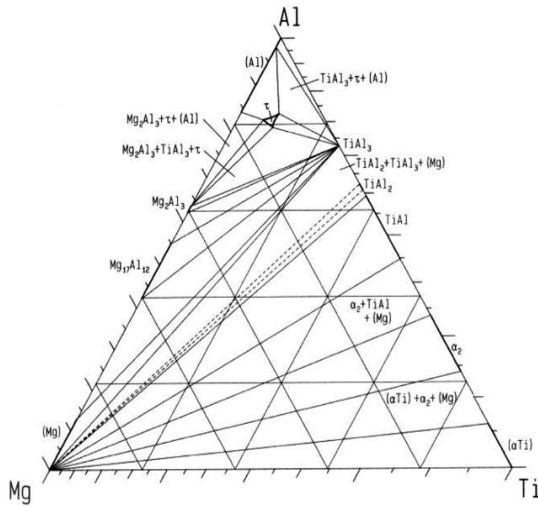


Fig. 4: Ti-Mg-Al ternary phase diagram [19]

F. Kinetics of joint formation

According to the Ti-Mg-Al ternary phase diagram (see Fig. 4), the following phases are likely to form at the bonding temperature and pressure used in this study. These phases include Mg₂Al₃, Mg₁₇Al₁₂, TiAl₃ and TiAl₂ [3]. According to the literature, the Gibbs energy for the formation of TiAl₃ is approximately 234 kJ/mol, TiAl₂ is 237 kJ/mol [4]. The diffusion of Al to the bond interface from Ti-side to the Mg-side led to the formation of a compound having a

stoichiometric composition of Mg₁₇Al₁₂, where 43.95 wt% of the compound is Al [16].

The Gibbs free energy of formation for the compound Mg₁₇Al₁₂ is -6 kJ/mol. Within a temperature range of 700 K - 1000 K, the Gibbs free energy of formation increases to -3.9 kJ/mol. From the Gibbs free energy data, the Mg₁₇Al₁₂ intermetallic compound is expected to form first at the Mg-interface. The Gibbs energy for the formation of the ternary compound Ti₂Mg₃Al₁₈ was found to be approximate -15kJ/mol. [19]. The width of the reaction layer that forms at the interface is believed to be time-dependent. As such, the layer thickness bears direct relation with growth kinetics. The average thicknesses of the TiAl₃ and Ti₂Mg₃Al₁₈ layers and the total intermetallic layer was used to determine the kinetic parameters involved in the diffusion process.

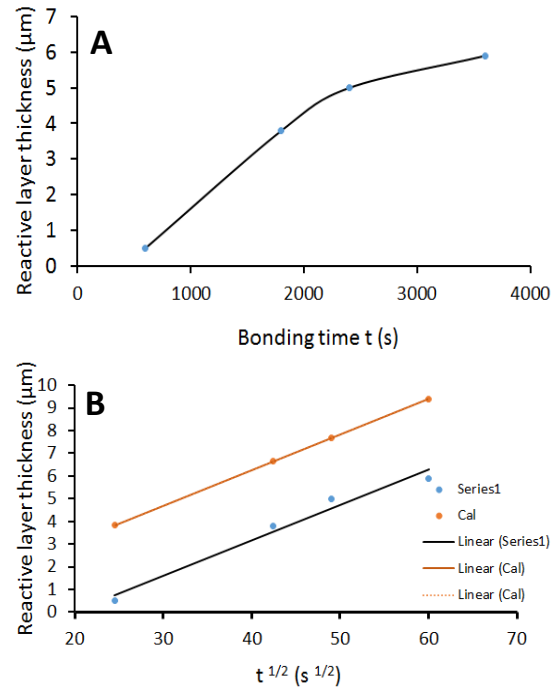


Fig. 5: (A) shows the relationship between the thickness of the reaction layer and the bonding time and (B) the predicted relationship between the width of the reaction layer and the bonding time according to the parabolic rate law.

The data collected shows that as the bonding time increases the width of the reaction layer also increased as predicted by the parabolic law shown in Equation 1. Fig. 5 shows the relationship between the bonding time and the thickness of the reaction layer formed at the interface and shows that as the bonding time increased, the width of the reaction layer also increased. When the parabolic rate law is applied to the results, the rate coefficient k was calculated to be 15.7×10^{-7} m/s. The value for n was assumed to be 0.5 since the growth of the reaction layer was assumed to be controlled by inter-diffusion. When the calculated rate coefficient is substituted into the power law shown in Equation 1, the results show that the model overestimated the width of the intermetallic layer was 1.

G. Effect of interlayer composition on joint microstructure

When a $\text{Ni}/\text{Al}_2\text{O}_3$ electrodeposited coating was used as the interlayer, the interface was observed to contain three distinct regions as identified by the difference in the shade (Fig. 6). A light grey blocky structure surrounded by dark grey plate like phases throughout the centre of reaction layer. Fig. 6 shows that diffusion took place predominantly on the magnesium side of the interface. When the bonds form was compared to the solid-state joint formed, it was observed that the width of the reaction layer was approximately twenty times thicker than the reaction layer formed during solid-state bonding of the same alloys. Additionally, several intermetallic compounds are at the interface during the bonding process.

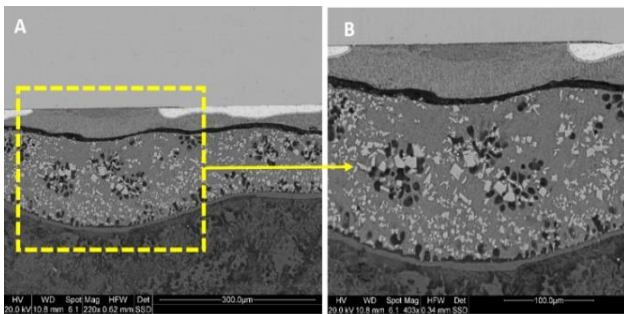


Fig. 6: Ti and Mg joint bonded using $\text{Ni}/\text{Al}_2\text{O}_3$ interlayer for 60 minutes at 500 °C.

H. Hardness Measurements

The effect of bonding time on the micro-hardness across the joint region is shown in Fig. 7. The microhardness values were measured across the joint starting at 500 μm from the joint centre. The figure shows that the hardness of the Ti sample fluctuated from 390–420 VHN up to 100 μm from the joint centre as the bonding time was increased from 10 minutes to 60 minutes. The hardness within the joint centre was observed to decrease to 190VHN after 10 minutes bonding time and 250 VHN after 60 minutes bonding time. The hardness of the Mg sample was found to be significantly lower than that of the Ti sample with a hardness ranging from 60 VHN after 10 minutes bonding time to 65 VHN. The variation of the hardness across the interface is attributed to the differences between the mechanical properties of Ti and Mg. The hardness at the centre of the bond is believed to have been caused by the formation of the reaction layer at the joint interface [20]. The reaction layer was shown to be made-up of TiAl_3 and $\text{Ti}_2\text{Mg}_3\text{Al}_{18}$ intermetallic compounds dispersed within the joint region.

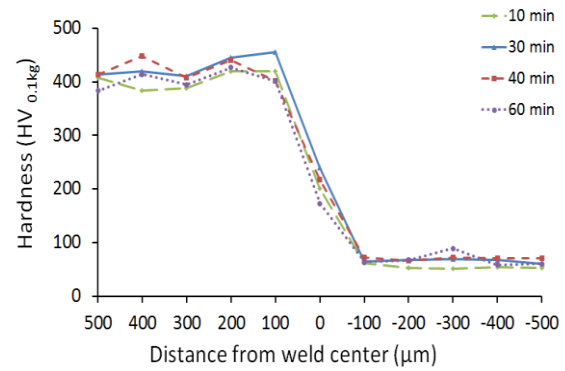


Fig. 7: Micro-hardness measurements across the joint region as a function of bonding time

I. Shear strength measurements and fractography

The effect of bonding time on the shear strength of Ti/Mg solid-state bonds are presented in Fig. 8. The results show that as the bonding time increased, the shear strength of the bonds also increased from 15.6 MPa after 10 minutes bonding time to 116 MPa after 60 minutes bonding time. The increase of the bond strength with bonding time was attributed to inter-diffusion of Ti, and Mg resulted in the formation of the compounds TiAl_3 and $\text{Ti}_2\text{Mg}_3\text{Al}_{18}$ as predicted by EDS analyses. When a $\text{Ni}/\text{Al}_2\text{O}_3$ interlayer was used, the joint strength increased by more than 30% (see Fig. 9).

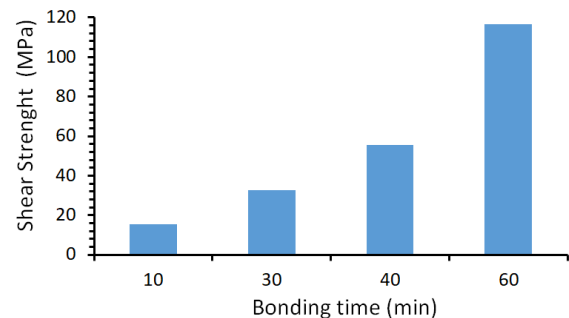


Fig. 8: Shear strength measurements as a function of bonding time

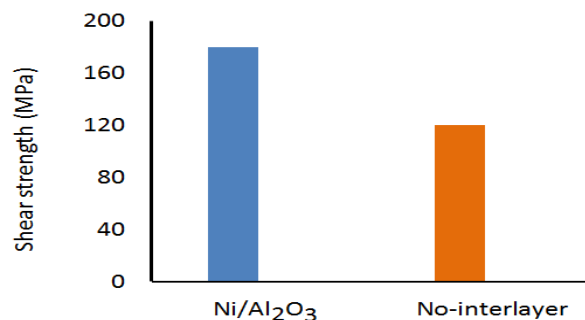


Fig. 9: Shear strength measurements as a function of interlayer composition

The precipitation of dispersed intermetallic compound served to reinforce the joint region causing an increase of the joint strength. The SEM micrograph presented in Fig. 10 shows the

fractured surface for samples bonded for 60 minutes. The surface of the fractured sample showed evidence of a mixed-mode of fracture due to the presence of both cleavage planes and micro-voids. Fig. 11 shows the XRD spectra for a sample bonded for 60 minutes. The spectra confirm the presence of several intermetallic compounds which are believed to be responsible for the type of failure observed. A schematic of the joint interface is presented in Fig. 12(A) and shows that fracture propagated through the TiAl_3 and $\text{Ti}_2\text{Mg}_3\text{Al}_{18}$ intermetallic phase present at the interface.

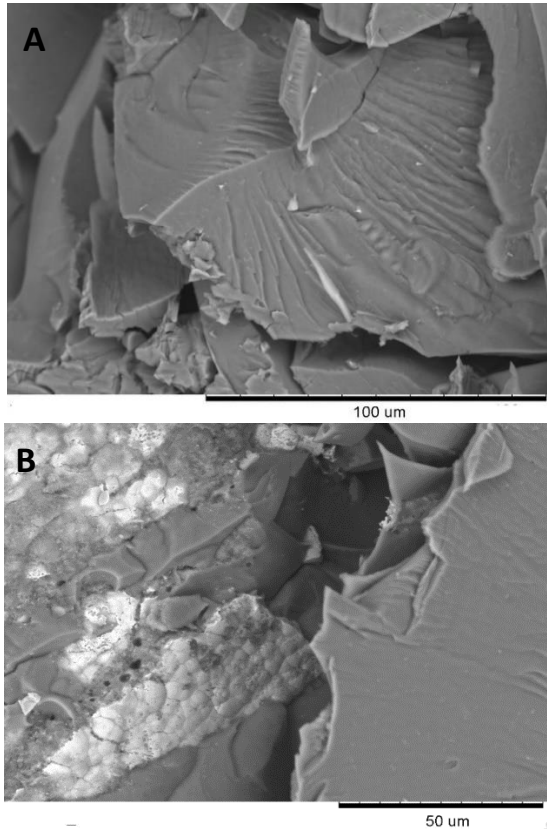


Fig. 10: (A) SEM micrograph of the Ti-side of the fractured surface for samples bonded for 60 minutes (B) SEM micrograph of the Mg-side of the fractured surface for samples bonded for 60 minutes.

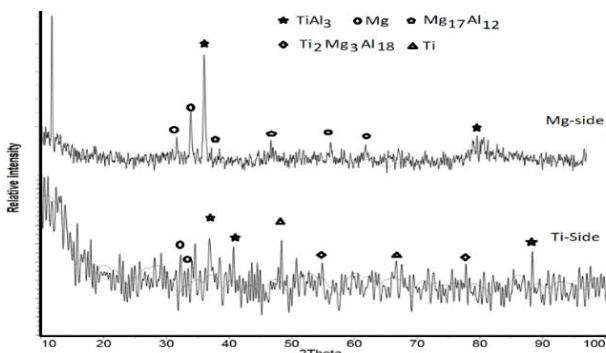


Fig. 11: XRD spectrum of the fractured surfaces of a sample bonded for 60 minutes

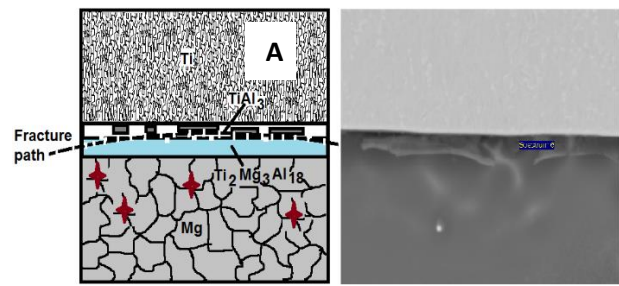


Fig. 12: (A) Schematic of the Ti/Mg bond showing the fracture path (B) SEM micrograph of Ti/Mg interface showing the reaction layer formed during bonding.

IV. CONCLUSION

The objective of the study was to evaluate the potential of diffusion bonding Ti6Al-4V and Mg (Az31) and to study the microstructural evolution and intermetallic compounds formed within the bond region. The results of the study confirmed that Ti and Mg alloys containing Al could be joined successfully using solid-state diffusion bonding with or without an interlayer. The presence of Al leads to the precipitation of TiAl_3 and $\text{Ti}_2\text{Mg}_3\text{Al}_{18}$ intermetallic compounds at the interface which promoted strong a metallurgical bond between the Ti and Mg sample.

As the bonding time increase the strength of the bonds also increase. Variation of the bonding time from 10 minutes to 60 minutes, results in an increase of the bond strength from 15.6 MPa at 10 minutes to 116 MPa at 60 minutes bond time. When an interlayer was used, the bond strength increased to 180 MPa. The increase in joint strength was attributed to the formation of dispersed intermetallic compounds within the joint region. The presence of these compounds is believed to reinforce the interface by dispersion strengthening. Additionally, microstructural analysis of the bonds formed showed the absence of oxide layers which confirmed that quality joint could be made by solid-state joining.

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