JMEPEG (2010) 19:657–665
DOI: 10.1007/s11665-009-9531-6

1059-9495/$19.00

Analyzing the Effect of Diffusion Bonding Process Parameters on Bond Characteristics of Mg-Al Dissimilar Joints

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(Submitted July 10, 2008; in revised form June 30, 2009)

The principle difficulty when joining magnesium (Mg)-aluminium (Al) lies in the existence of formation of oxide films and brittle intermetallic in the bond region. However, diffusion bonding can be used to join these alloys without much difficulty. Temperature, pressure, and holding time are the three main variables which govern the integrity of a diffusion bond. This paper focuses on the effect of these parameters on diffusion layer thickness, hardness and strength of AZ31B magnesium-AA2024 aluminium dissimilar joints. The experiments were conducted based on three factors, five-level, and central composite rotatable design with full replications technique. Empirical relationships were developed to predict diffusion layer thickness, hardness and strength using response surface methodology. From this investigation, it is found that bonding temperature has predominant effect on bond characteristics.

Keywords aluminium alloy, bonding strength, diffusion bonding, diffusion layer thickness, interface hardness, magnesium alloy, shear strength

1. Introduction

Joining of materials by conventional welding techniques becomes difficult if the physical properties, such as melting temperature and thermal expansion coefficients, of the two materials differ a lot, as it is necessary to have controlled melting on both sides of weld joints simultaneously. Even if this criterion is met, it may not be possible to have an appropriate joint when the two materials are metallurgically incompatible. This is because metallurgical incompatibility may lead to a weld zone and heat affected zone microstructure without adequate mechanical strength (Ref 1). By means of diffusion bonding, it is possible to bond all the materials whose chemical and metallurgical properties are appropriate. In particular, the bonding of advanced materials is not possible by classical welding methods because of unexpected phase propagation at the bond interface (Ref 2). Hence, diffusion bonding introduces convenience to the bonding of materials, which are not possible to bond by conventional welding methods. Furthermore, diffusion bonding is preferred for the materials in which brittle phase formation is unavoidable.

To obtain the desired strength, it is essential to have a complete control over the relevant process parameters to maximize strength on which the quality of a joint is based (Ref 3). Therefore, it is very important to select and control the welding process parameters for obtaining maximum strength. In order to achieve this, various prediction methods can be applied to define the desired output variables through developing mathematical models to specify the relationship between the input parameters and output variables. It has been proved by several researchers (Ref 4, 5) that efficient use of statistical design of experimental techniques allows development of an empirical methodology to incorporate a scientific approach in the fusion welding procedure. Even though sufficient literature is available on diffusion bonding of Mg-Al alloys, no systematic study has been reported so far to correlate the process parameters and mechanical properties of diffusion bonded of Mg-Al dissimilar joints. Hence, an investigation was carried out to understand the effect of diffusion bonding process variables on bond characteristics and this paper reveals the results.

2. Experimental

Square-shaped specimens (50 × 50 mm) were machined from rolled plates of 5 mm thickness AZ31B magnesium alloy and AA2024 aluminium alloys. The chemical composition of the above base metals is presented in Table 1. The polished and chemically treated specimens were stacked in a die made up of 316L stainless steel and the entire diffusion bonding setup, shown in Fig. 1, was inserted into a vacuum chamber (constant pressure of 10^{-3} mmHg was maintained). From the literature (Ref 6) and the previous work done (Ref 7) in our laboratory, the independently controllable primary process parameters affecting the quality of diffusion bonded joint were identified. They are bonding temperature, bonding pressure and holding

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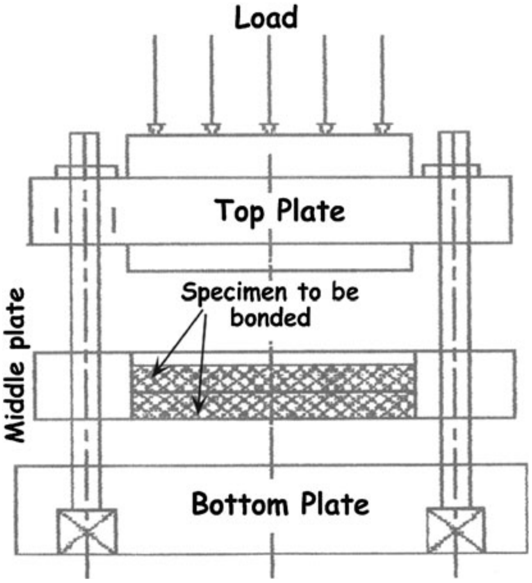

Fig. 1 Configuration of the diffusion bonding setup

Table 1 Chemical composition (wt.%) of (a) AA2024 aluminium alloy and (b) AZ31B magnesium alloy

(a)

Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
0.5	0.5	4.9	0.9	1.8	0.10	0.25	0.15	Bal

(b)

Al	Mn	Zn	Mg
3.0	0.20	1.00	Bal

Table 2 Important diffusion bonding process parameters

		Levels				
Parameter	**Notation**	**−1.682**	**−1**	**0**	**+1**	**+1.682**
Bonding temperature	T, °C	400	420	440	460	480
Bonding pressure	P, MPa	4	8	12	16	20
Holding time	t, min	10	30	50	70	90

time. The working limits of each parameters were identified (Table 2) and they are reported elsewhere (Ref 8). Due to wider range of working limits of process parameters, a three-factor, five levels, central composite rotatable centered design was chosen to conduct the experiments, as presented in Table 3. The specimens were heated up to the bonding temperature using induction furnace with a heating rate of 25 °C/min; simultaneously the required pressure was applied. After the completion of bonding, the samples were cooled to room temperature before removal from the chamber.

As the joints were not large enough for normal lap shear testing, a non-standard test was devised to measure the shear strength of the bonds. The lap shear tensile specimens and ram tensile test specimens, as shown in Fig. 2, were prepared from the Mg/Al diffusion bonded joints by a line cutting machine (electric spark cutting). Test was carried out in 100 kN capacity servo controlled Universal Testing Machine (make: FIE-Bluestar, India; model: UNITEK-94100). In ram tensile test (Fig. 2c), though a compressive load was applied from the top of the specimen, it will make the joint interface to fail by tension, in order to evaluate the bonding strength of the joint. Vicker's microhardness testing machine (make: Shimadzu, Japan; model: HMV-2T) was used to measure the diffusion layer hardness with 0.05 kg load. Hardness was measured at five different locations of interface region and the average is presented in Table 3. Microstructural examination was carried out at interface region using a light optical microscope (make: MEIJI, Japan; model: MIL-7100) incorporated with an image analyzing software (Metal Vision). Diffusion layer thickness was measured at three different locations of interface region using the Metal Vision Image analyzing software and the average is presented in Table 3. The optical micrographs of interface region of diffusion bonded joints are displayed in Fig. 3.

3. Results and Discussion

3.1 Developing Empirical Relationships

The responses, diffusion layer thickness (DL), interface hardness (IH), shear strength (SS) and bonding strength (BS) are the function of bonding temperature (T), °C, bonding pressure (P), MPa, and holding time (t), min, and it can be expressed as

$$\mathrm{DL} = f_1\{T,P,t\};\ \mathrm{IH} = f_2\{T,P,t\};\ \mathrm{SS} = f_3\{T,P,t\}\ \text{and}\ \mathrm{BS} = f_4\{T,P,t\}. \quad \text{(Eq 1)}$$

The second-order polynomial (regression) equation used to represent the response surface 'Y' and the selected polynomial could be expressed as (Ref 9)

$$Y = b_0 + b_1(T) + b_2(P) + b_3(t) + b_{11}(T^2) + b_{22}(P^2) + b_{33}(t^2) + b_{12}(TP) + b_{13}(Tt) + b_{23}(Pt) \quad \text{(Eq 2)}$$

All the coefficients were obtained applying central composite rotatable design using the Stat-Ease Design-Expert DX7® statistical software package. After determining the significant coefficients (at 95% confidence level), the empirical relationships were developed using only these coefficients, The final relationships obtained to estimate diffusion layer thickness, hardness, shear strength and bonding strength are given as follows.

For diffusion layer thickness

$$\mathrm{DL} = \{32.85 + 2.84 * T + 1.20 * P + 1.32 * t\}\ \mu\mathrm{m} \quad \text{(Eq 3)}$$

For interface hardness

$$\mathrm{IH} = \left\{134.78 + 13.97 * T + 3.26 * P + 7.55 * t - 2.23 * P^2 + 2.19 * t^2\right\}\ \mathrm{Hv} \quad \text{(Eq 4)}$$

For shear strength

$$\mathrm{SS} = \left\{42.61 + 4.36 * T + 3.18 * P + 3.94 * t - 1.25 * Tt - 1.26 * T^2 - 0.90 * P^2 - 1.43 * t^2\right\}\ \mathrm{MPa} \quad \text{(Eq 5)}$$

Table 3 Experimental design matrix and results

	Coded values			Original values			Diffusion layer thickness, μm	Interface hardness, Hv	Shear strength, MPa	Bonding strength, MPa
Expt. no	*T*	*P*	*t*	Bonding temperature, °C	Bonding pressure, MPa	Holding time, min	DL	IH	SS	BS
1	−1	−1	−1	420	8	30	27	112	25	45
2	+1	−1	−1	460	8	30	33	137	38	58
3	−1	+1	−1	420	16	30	29	120	35	55
4	+1	+1	−1	460	16	30	35	145	45	65
5	−1	−1	+1	420	8	70	30	125	36	56
6	+1	−1	+1	460	8	70	34	155	45	65
7	−1	+1	+1	420	16	70	31	130	45	65
8	+1	+1	+1	460	16	70	33	160	49	69
9	−1.682	0	0	400	12	50	27	110	31	51
10	+1.682	0	0	480	12	50	37	158	45	66
11	0	−1.682	0	440	4	50	30	122	35	55
12	0	+1.682	0	440	20	50	35	133	43	64
13	0	0	−1.682	440	12	10	30	126	31	51
14	0	0	+1.682	440	12	90	36	154	44	63
15	0	0	0	440	12	50	34	137	44	64
16	0	0	0	440	12	50	33	134	42	62
17	0	0	0	440	12	50	31	133	42	61
18	0	0	0	440	12	50	33	136	44	64
19	0	0	0	440	12	50	34	134	43	61
20	0	0	0	440	12	50	33	135	42	62

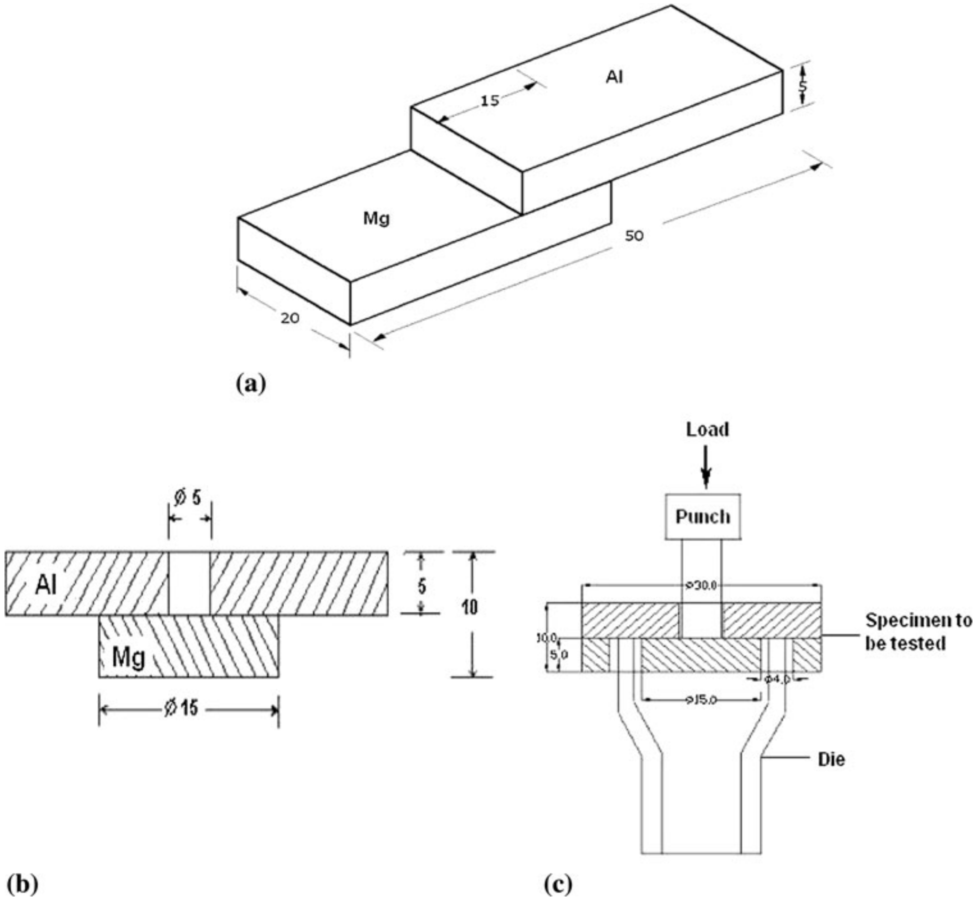

Fig. 2 Dimensions of test specimens: (a) lap shear tensile specimen; (b) ram tensile specimen; (c) ram tensile test setup

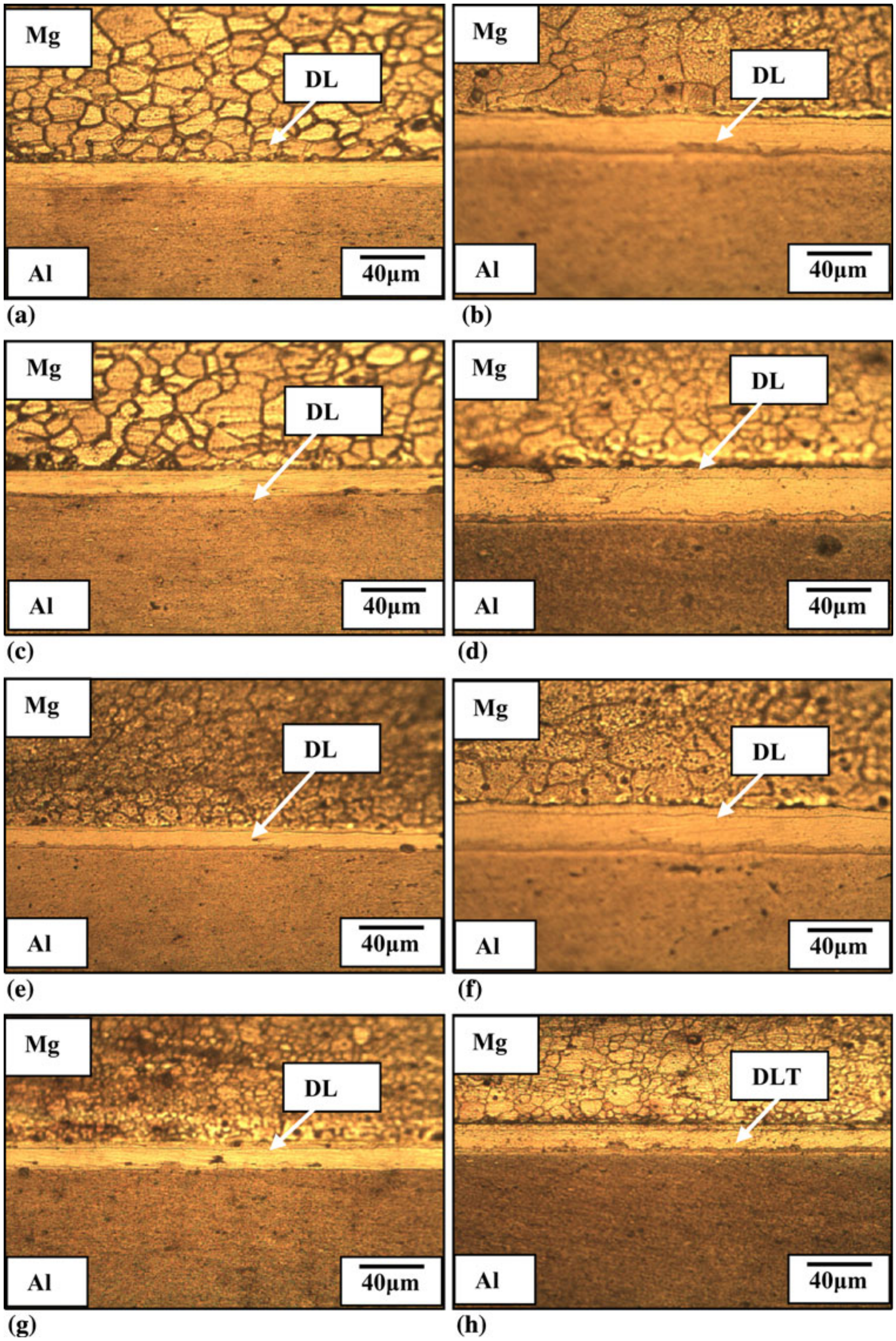

Fig. 3 Optical micrographs of interface region of Mg-Al bonds

For bonding strength

$$\begin{aligned} BS = \{&62.28 + 4.48 * T + 3.31 * P + 3.82 * t - 1.25 * Tt \\ &-1.01 * T^2 - 1.54 * t^2\} \text{ MPa} \end{aligned} \qquad \text{(Eq 6)}$$

The determination coefficient (R^2) indicates the goodness of fit for the model (Table 4). In this case, the value of the determination coefficient (R^2) indicates that the model does not explain only less than 3% of the total variations. The value of adjusted determination coefficient (adjusted R^2) is also high, which indicates a high significance of the model.

Using the above-developed empirical relationships, the shear strength and bonding strength were estimated for different combinations of temperature, pressure and holding time. The estimated values of shear strength and bonding strength of diffusion bonded Mg-Al joints are presented in graphical form as shown in Fig. 4 and 5.

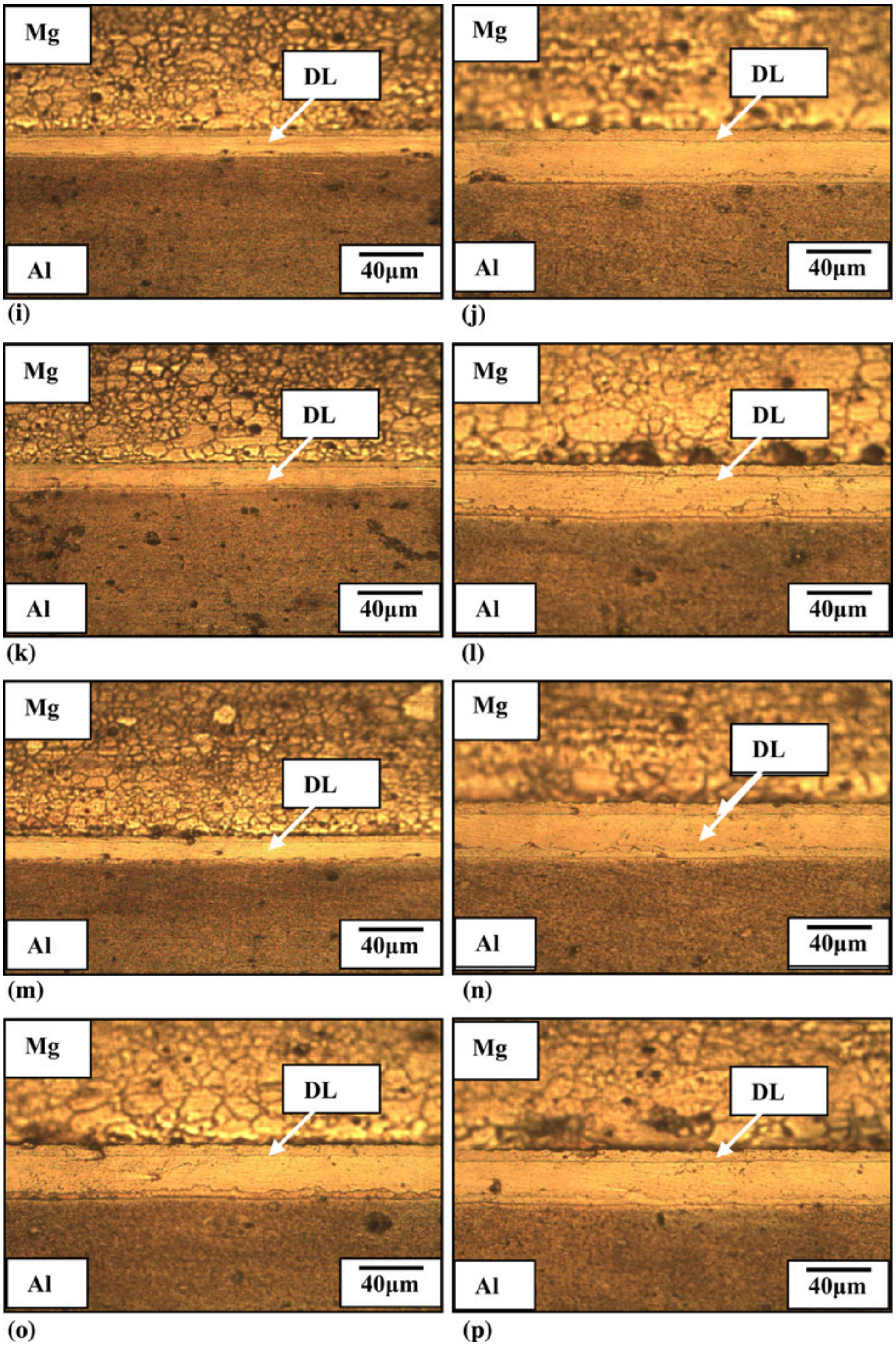

Fig. 3 Continued

From the results, it is understood that shear strength of the bonds depends on bonding temperature, bonding pressure and holding time. However, shear strength cannot be used to evaluate the extent of bonding because the specimens may have different tempered conditions. But it was reported (Ref 10) that the actual shear strength requirements of the bonds for aircraft structures are generally in the order of 10-20 MPa. Hence, in this investigation, the shear strength of the bonds was evaluated by conducting lap shear tensile strength and bonding strength was evaluated by conducting ram tensile test. From the strength values, it could be inferred that all the bonds satisfy the above requirements and falls under 'good bonds' category.

3.2 Effect of Bonding Temperature on Shear Strength and Bonding Strength

Surface conditions also change with temperature. The asperities deform at higher temperature providing yielding

Table 4 ANOVA test results for responses

Terms	Diffusion Layer thickness (DL)	Interface hardness (IH)	Shear strength (SS)	Bonding strength (BS)
First order terms				
Sum of squares (SS)	154.02	3586.79	610.33	628.05
Degrees of freedom (df)	3	3	3	3
Mean square (MS)	51.34	1195.60	203.44	207.68
Second order terms				
Sum of squares (SS)	3.98	157.82	86.78	77.15
Degrees of freedom (df)	6	6	6	6
Mean square (MS)	0.66	26.30	14.46	12.86
Error terms				
Sum of squares (SS)	4.83	10.83	5.33	9.33
Degrees of freedom (df)	5	5	5	5
Mean square (MS)	0.97	2.17	1.07	1.87
Lack of fit				
Sum of squares (SS)	4.22	16.24	18.18	13.74
Degrees of freedom (df)	5	5	5	5
Mean square (MS)	0.84	3.25	3.64	2.75
F ratio				
Prob $>$ *F* (a)	<0.0001	<0.0001	<0.0001	<0.0001
R^2	0.9457	0.9929	0.9669	0.9678
Model	Significant	Significant	Significant	Significant

(a) Prob $> F < 0.0500$ indicate model terms are significant

and stress. It is evident from Fig. 4 that the shear strength and bonding strength are increasing with increasing bonding temperature (Ref 9). At a low temperature of 400 °C, the shear strength and bond strength of the diffusion-bonded joint are low (31 and 51 MPa, respectively). This is due to poor contact of the bonded surface and insufficient thermal excitation. Also, at low temperature, the flowability of metal is substantial, yet yield strength of the base materials still remains high, which leads to an incomplete coalescence of the mating surfaces (Ref 11). Moreover, the grain boundary surface tension and grain boundary mobility are less at low temperatures which controls the initial movement of interface grain boundaries.

When temperature reaches to 420 °C, it results in a considerable improvement in shear strength and bonding strength. Higher temperature improves the contact ratio, because the atom diffusion is higher with higher temperature leads to effective bonding. At a bonding temperature of 460 °C, maximum shear strength of 49 MPa and a bonding strength of 69 MPa are obtained. With increase in temperature, the yield strength of the joint decreases, which leads to larger interfacial deformation and hence the atom diffusivity increases, resulting in easier and speeder chemical bonding. As in the initial stages of bonding at high temperatures, migration of interface grain boundaries was involved as above, the higher rate of grain growth would lead to rapid removal of evidence of the bond line and increases the strength nearer to the parent metal (Ref 12).

Further increase in temperature to 480 °C leads to reduction in strength, because increase in temperature generally promotes mass transfer of alloying elements across the interface, which is responsible for the increase in volume fraction of the reaction products such as $MgAl_2O_4$, Mg_2SiO_4, Al_2SiO_5, $Mg_2Al_2O_4$ and Mg_2Al_3 causes more embrittlement of joints. However, plastic collapse of the mating surface asperities leads to intimate contact, which counter balances the embrittlement phenomena due to intermetallic phases. Higher rate of diffusion causes higher amount of intermetallic compound formation, which leads to increase in brittleness of the joint and decrease in joint strength (Ref 13).

3.3 Effect of Bonding Pressure on Shear Strength and Bonding Strength

As the initial pressure decreases or relaxes with time and temperature, a constant pressure of 10^{-3} mmHg was maintained inside the vacuum chamber to prevent oxidation. From Fig. 4 it can be inferred that the shear strength and bonding strength of the joints are increasing with increase in bonding pressure, irrespective of bonding temperature and holding time. The effect of bonding pressure on strength is less when compared to temperature and time. At a bonding pressure of 4 MPa, minimum shear strength and bonding strength are obtained. Because, at low bonding pressure, contact is only at the protrusions on the bonded surface, so the contact rates and the strength of the bonded joint are lower. Generally, when the bonding pressure is applied, the points of contact between the two surfaces will expand almost instantaneously. It is also observed that when bonding temperature increases the initial set pressure also increased, which may be due to expansion of metals. After diffusion bonding, when the set up was cooled to room temperature, the pressure was reduced to initial set pressure automatically.

Higher shear strength is obtained at a pressure of 12 MPa. Increase in pressure develops higher rate of plastic deformation at contact sites, which increases the contact areas of clean surfaces and hence diffusion rate changes abruptly. Increase in pressure influences re-crystallization temperature, and deformation tends to enhance the contact of bond surface and rapid growth of re-crystallization. This will obviously increase the rate of interface contact, and atoms are made to pass through this bonding interface. So, more diffusion paths are created due to movement of atoms (Ref 14).

Further increase of pressure to 20 MPa resulted in small increase of shear strength and bonding strength. The property

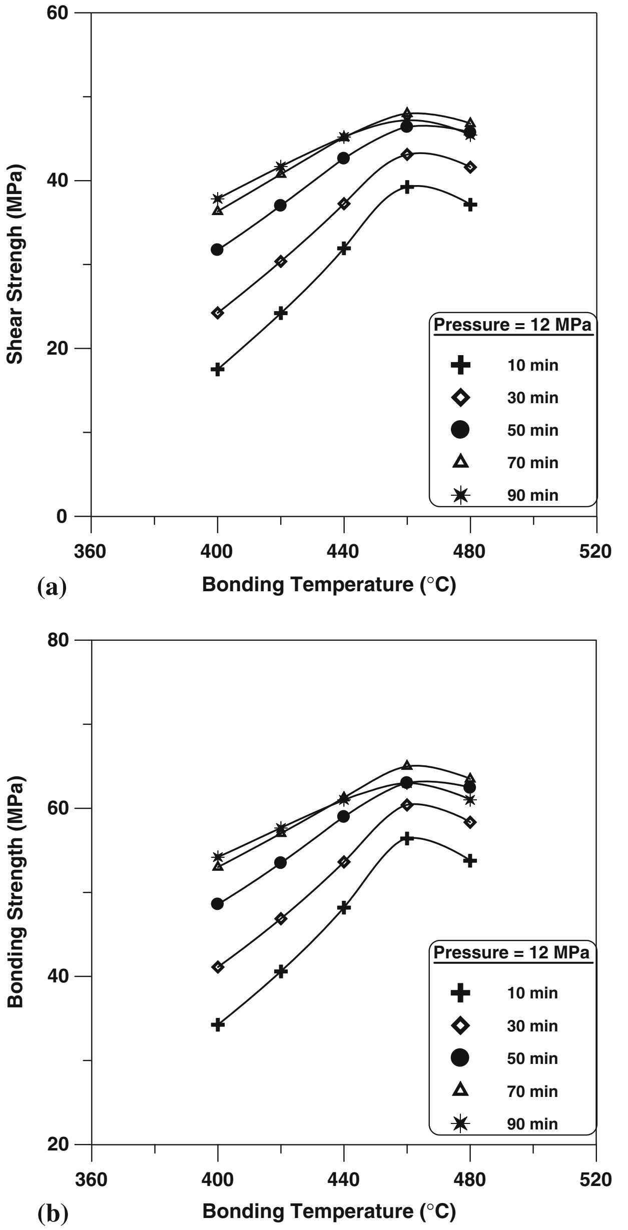

Fig. 4 Effect of diffusion bonding parameters on (a) shear strength and (b) bonding strength

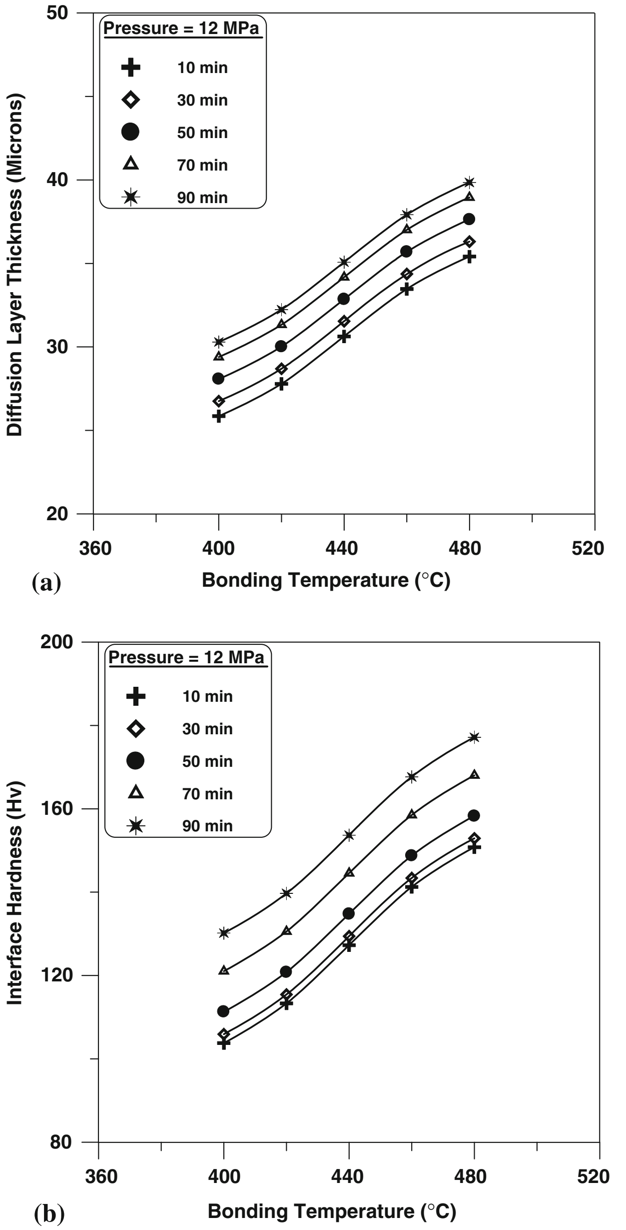

Fig. 5 Effect of diffusion bonding parameters on (a) diffusion layer thickness and (b) interface hardness

of the bonded joints also mainly depends on thickness of the intermetallic compounds, which was unaffected as pressure increases. The voids formed at the original interface will disappear as the contact area expands with time, because the stress within the contact zone will cause a plastic flow by either conventional creep or super plasticity. The smaller voids would be removed rapidly by diffuse ion.

3.4 Effect of Holding Time on Shear Strength and Bonding Strength

From Fig. 4 it can be observed that the shear strength and bonding strength of the joints are increasing with increase in holding time, irrespective of temperature and pressure. High shear strength is obtained at a holding time of 70 min. This is mainly because the holding time has an effect on the creep of the protrusions and the quantity of atomic diffusion. The strength is increasing more rapidly with increase in holding time from 10 to 70 min and then it decreases sharply. Longer holding times show a continuing grain growth accompanied by a small increase in specific strength. At 90 min of holding time, a sharp decrease in strength was attributed due to the growth of intermetallic compounds. The thickness of the intermetallic compound increases remarkably with holding time and the strength of the bond decreases. Lower shear strength and bonding strength of 31 and 51 MPa are obtained at a holding time of 10 min. It is evident that the lower holding time led to the weak bonds and this may be due to insufficient time allowed for the diffusion of atoms across the bond interface from the mating surfaces (Ref 15).

It is generally considered that a sound bond has been obtained when there is no metallographic evidence of the bond line in the interface. However, contamination in surfaces to be joined is often unavoidable in real bonding situations. Therefore, neither an interface-free bond nor the strength of the bond is problematic. It is known that the bond quality was basically dependent on the extent of removal of the initial bond interface (Ref 16). The mechanism(s) by which the bond interfaces eliminated is that recrystallization which was initiated in the bond region caused the elimination of the bond interface and led to high bond strengths. Increase in bonding temperature will speed up grain boundary re-crystallization and grain growth. The elimination of bond interface based on the temperature, time dependent, grain growth, and on the bond microstructures, it is reasonable to conclude that grain growth is an important process in the removal of the initial bond interface leading to full bonding.

3.5 Effect of Process Parameters on Diffusion Layer Thickness

It is necessary to analyze the role of diffusion layer on bonding characteristics, because the formation of diffusion layer at the interface influences the strength of the bond. Optical micrographs were taken at the interface region of all the bonds to understand the effect of diffusion bonding process parameters on the formation of diffusion layer and they are presented in Fig. 3. It can be seen that the diffusion layer thickness is mainly influenced by bonding temperature. At a temperature of 400 °C, the diffusion layer thickness is 27 μm and it is increased gradually with an increase in bonding temperature. When the bonding temperature reaches 480 °C, diffusion layer thickness increased to a maximum of 37 μm. While correlating strength with diffusion layer thickness, it is understood that both very narrow diffusion layer thickness of 27 μm (Fig. 3a) and very wide diffusion layer thickness of 37 μm (Fig. 3j) led to the lower shear and bonding strength. A diffusion layer thickness of 33 μm is found to be optimum to attain higher shear strength and bonding strength (Fig. 3h).

Figure 5(a) reveals the effect of process parameters on diffusion layer thickness of diffusion bonded joints. From the figure, the following inferences can be obtained: (i) diffusion layer thickness is increasing with increase in bonding temperature and bonding pressure; (ii) the effect of holding time has the least effect when compared to bonding temperature and bonding pressure; (iii) an optimum diffusion layer thickness of 33 μm is obtained (Fig. 3h) at a temperature of 460 °C, pressure of 16 MPa and holding time of 70 min.

Wu and Lo (Ref 17) also revealed that the diffusion layer thickness depends on atom diffusion. When the bonding temperature was increased to over 480 °C, the joining processes have allowed the diffusion of all elements from both the magnesium and aluminium sides quickly. This fact promotes the chemical joint (in all welding condition) between materials is achieved when inter-diffusion between the materials is provided without the formation of voids and brittle phases such as intermetallic compounds. These findings were in agreement with Fick's second law, a partial differential equation (Ref 18) describes that the rates at which atoms are redistributed in a material by diffusion. The composition, extent, nature and properties of the phases originated during the welds will control the resulting mechanical properties. The intermetallic compound grows steadily and gradually via enhanced temperature at the bond region of dissimilar metal joints. The particle distribution of intermetallic compounds has no harmful effects on the joint performances; moreover, it can strengthen the joints.

The intermetallic compounds have never joined up and formed a whole body; they also have no effect on the plasticity and strength of joints. But once they connect and the thickness is higher, they lead to high brittleness and internal stress, and caused obvious decrease in the plasticity and strength of joints. In particular, because the diffusion rate for Al atoms is much higher than that for Mg atoms at this high temperature, an excess of Al atoms diffuses across the interface into the magnesium side and forms cavities according to Kirkendall effect (Ref 19). This Kirkendall effect leads to produce continuous cavities at the interface, resulting in a dramatic decrease in the interface bonding strength level in the specimens bonded at 480 °C. As the diffusion bonding occurs in an atomic level, it is difficult to locate the fracture area. A ductile type of fracture was observed and the location of failure was on the magnesium side. It also reveals that magnesium particles were adhered on the surface of the aluminium, which indicates that the failure was at magnesium side.

3.6 Effect of Process Parameters on Interface Hardness

Figure 5(b) reveals the effect of process parameters on interface hardness of diffusion bonded Mg-Al dissimilar joints. From the figure, the following inferences can be obtained: (i) hardness value is increasing with increase in bonding temperature and holding time; (ii) applied pressure has less effect on hardness; (iii) a higher hardness of 160 Hv is exhibited at a bonding temperature of 460 °C, pressure of 16 MPa and holding time of 70 min. It can be seen that the hardness increases with increasing temperature and holding time; however, there is no evidence of change in hardness due to change in pressure. Increase in hardness with increasing temperature and holding time can be attributed to the formation of intermetallic compounds as discussed earlier.

Mechanical properties of the joints were controlled by the composition, extent, nature and properties of the phases originated during the welds. In this investigation, strength of the joints were increased with increase in hardness to a certain level. The increase of hardness was due to the formation of intermetallic compounds formed at the interface. The intermetallic compound grows steadily and gradually via enhanced temperature at the bond region of dissimilar metal joints. The particle distribution of intermetallic compounds has no harmful effects on the joint performances; moreover, it can strengthen the joints. The intermetallic compounds have never joined up and formed a whole body; they also have no effect on the plasticity and strength of joints and hardness of the joint was high. But once they connect and thickness grows beyond 5 μm, the hardness was changed into brittleness. So, the plasticity and strength of joints will obviously be decreased. Because the diffusion rate for Al atoms is much higher than that of Mg atoms at high temperature, an excess of Al atoms diffuses across the interface into the magnesium side and forms cavities according to Kirkendall effect (Ref 18). This Kirkendall effect leads to produce continuous cavities at the interface, resulting in a drastic reduction in the interface bonding strength level in the specimens bonded at maximum temperature.

4. Conclusions

From this investigation, the following important conclusions are derived.

- Empirical relationships were developed to predict the bonding strength, shear strength, diffusion layer thickness and interface hardness of the Mg-Al diffusion bonded joints by incorporating diffusion bonding process parameters.
- The bonding temperature has greater influence on shear strength and bonding strength, followed by holding time and bonding pressure.
- Joints fabricated at a bonding temperature of 460 °C, bonding pressure of 16 MPa and holding time of 70 min exhibited maximum shear and bonding strength of 49 and 69 MPa, respectively. This may be due to the formation of optimum thick and hard diffusion layer at the interface.

Acknowledgments

The authors wish to place their sincere thanks to University Grants Commission (UGC), New Delhi, for financial support rendered through a Major Research Project No. F-31-51/2005(SR).

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