

Influences of diffusion bonding process parameters on bond characteristics of Mg-Cu dissimilar joints

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Abstract: In many circumstances, dissimilar metals have to be bonded together and the resulting joint interfaces must typically sustain mechanical and/or electrical forces without failure, which is not possible by fusion welding processes. The melting points of magnesium (Mg) and copper (Cu) have a significant difference (nearly 400 °C) and this may lead to a large difference in the microstructure and joint performance of Mg-Cu joints. However, diffusion bonding can be used to join these alloys without much difficulty. This work analyses the effect of parameters on diffusion layer thickness, hardness and strength of magnesium-copper dissimilar joints. The experiments were conducted using three-factor, five-level, central composite rotatable design matrix. Empirical relationships were developed to predict diffusion layer thickness, hardness and strength using response surface methodology. It is found that bonding temperature has predominant effect on bond characteristics. Joints fabricated at a bonding temperature of 450 °C, bonding pressure of 12 MPa and bonding time of 30 min exhibited maximum shear strength and bonding strength of 66 and 81 MPa, respectively.

Key words: diffusion bonding; magnesium alloy; copper alloy; shear strength; bonding strength; diffusion layer thickness; interface hardness

1 Introduction

The 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 are quite different, so it is necessary to control the melting on both sides of weld joints simultaneously. Even if this criterion is met, it may be not possible to have an appropriate joint when the two materials are metallurgically incompatible. This may lead to a weld zone and heat affected zone without adequate mechanical strength[1]. By means of diffusion bonding, it is possible to bond all the materials whose chemical and metallurgical properties are different. In particular, the bonding of advanced materials is not possible by classical welding methods because of unexpected phase propagation at the bond interface[2]. Hence, diffusion bonding introduces convenience to the

bonding of dissimilar materials which are not possible to bond by conventional welding methods and it is preferred by the materials in which the formation of brittle phase is unavoidable.

The quality of a joint is based on its strength. To obtain the maximum strength, it is essential to control the relevant process parameters completely. Therefore, it is very important to select and control the welding process parameters[3]. Various prediction methods are applied to define the desired output variables through developing mathematical models to specify the relationship between the input parameters and output variables. It was proved by several researchers[4–5] that efficient use of statistical design of experimental techniques can maximize the output variables. It 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-Cu alloys, no systematic study was reported to

correlate the process parameters and mechanical properties of diffusion bonded Mg-Cu dissimilar joints. Hence, an investigation was carried out to understand the effect of diffusion bonding process variables on bond characteristics.

2 Experimental

Square shaped specimens (50 mm×50 mm) were machined from magnesium and commercial grade copper alloys which were rolled to plates of 5 mm thick. The chemical compositions of the base metals are shown in Tables 1 and 2.

Table 1 Chemical composition of commercial grade copper alloy (mass fraction, %)

Al	O	Fe	Pb	B	S	Cu
0.14	0.092	0.007	0.001	0.018	<0.001	Bal.

Table 2 Chemical composition of AZ31B magnesium alloy (mass fraction, %)

Al	Mn	Zn	Mg
3.0	0.20	1.00	Bal.

The polished and chemically treated specimens were stacked in a die made of 316L stainless steel, and the entire diffusion bonding setup (see Fig.1) was inserted into a vacuum chamber (pressure of 0.13 Pa was maintained). From Ref.[6] and the previous work done in our laboratory[7], the independently controllable primary process parameters which affect the quality of diffusion bonded joint were identified. They are bonding temperature, bonding pressure and holding time. The working limits of each parameters were identified (Table 3) and reported[8].

Due to wider range of working limits of process parameters, a three-factor, five-level and central

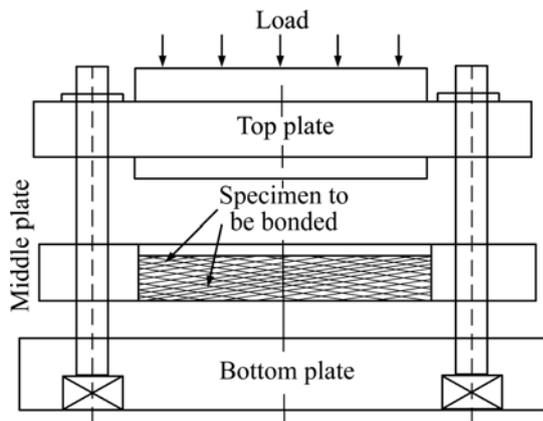


Fig.1 Schematic diagram of diffusion bonding setup

Table 3 Important diffusion bonding process parameters

Parameter	Level				
	-1.682	-1	0	+1	+1.682
Bonding temperature/°C	425	450	475	500	525
Bonding pressure/MPa	4	8	12	16	20
Holding time/min	10	20	30	40	50

composite rotatable centered design was chosen to conduct the experiments, as presented in Table 4. The specimens were heated up to the bonding temperature in an induction furnace at a heating rate of 25 °C/min and the required pressure was applied. After 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 illustrated in Fig.2, were prepared from the Mg/Cu diffusion bonded joints by wire-electrode cutting. Tests were carried out in a 100 kN capacity servo controlled Universal Testing Machine. Vicker’s microhardness testing machine was used to measure the diffusion layer hardness with a load of 0.49 N. Hardness and diffusion layer thickness were measured at five different locations of interface region and the average value is presented in Table 4. Microstructural examination was carried out at interface region using a optical microscope incorporated with an image analyzing software.

The optical micrographs of interface region of diffusion bonded joints are shown in Fig.3.

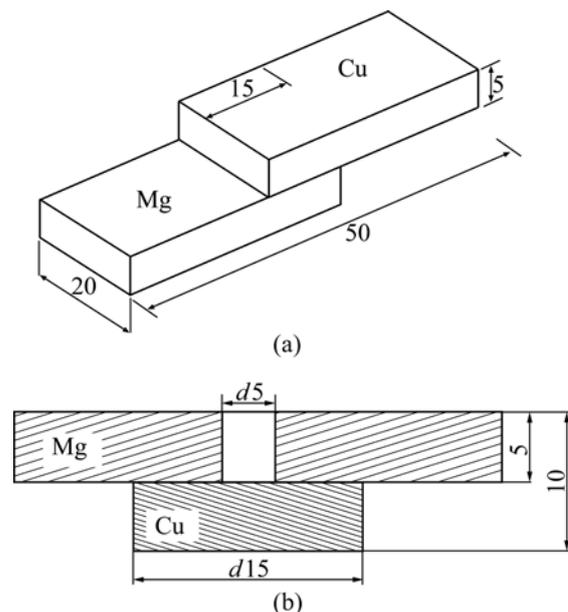
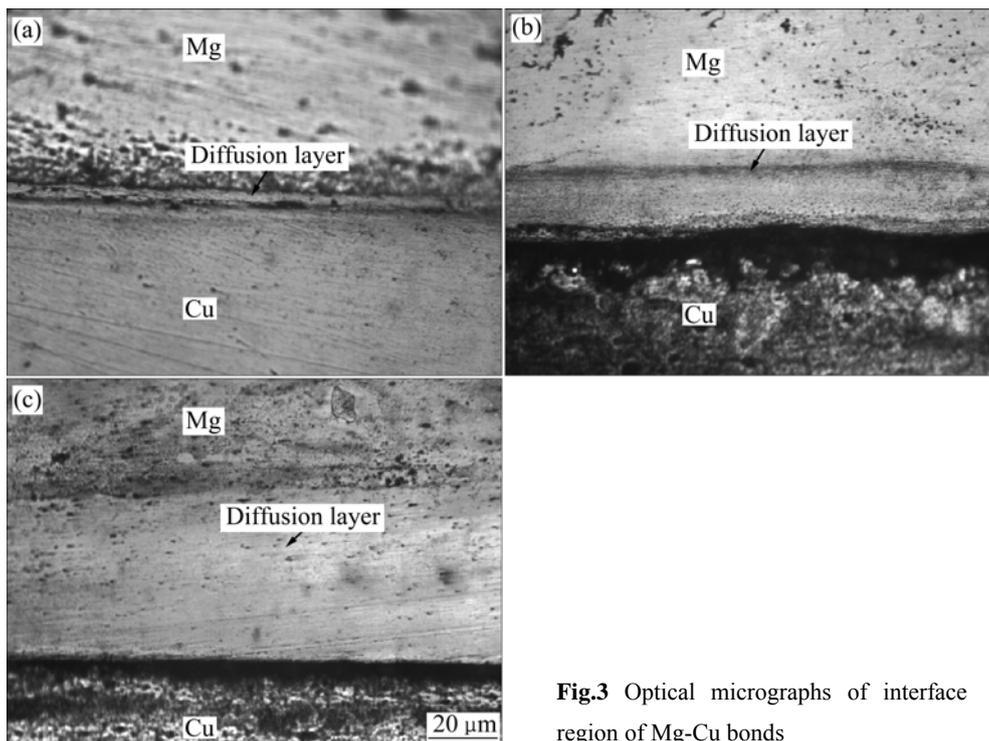


Fig.2 Dimensions of test specimens (Unit: mm): (a) Lap shear tensile specimen; (b) Ram tensile specimen

Table 4 Experimental design matrix and results

Expt. No.	Coded value			Original value			Diffusion layer thickness/ μm	Interface hardness, Hv	Shear strength/MPa	Bonding strength/MPa
	Bonding temperature/ $^{\circ}\text{C}$	Bonding pressure/MPa	Holding time/min	Bonding temperature/ $^{\circ}\text{C}$	Bonding pressure/MPa	Holding time/min				
1	-1	-1	-1	450	8	20	12	77	66	81
2	+1	-1	-1	500	8	20	18	102	58	73
3	-1	+1	-1	450	16	20	14	85	60	75
4	+1	+1	-1	500	16	20	20	110	50	65
5	-1	-1	+1	450	8	40	15	90	59	74
6	+1	-1	+1	500	8	40	19	120	52	67
7	-1	+1	+1	450	16	40	16	95	54	69
8	+1	+1	+1	500	16	40	22	125	40	55
9	-1.682	0	0	425	12	30	12	75	60	76
10	+1.682	0	0	525	12	30	24	120	48	62
11	0	-1.682	0	475	4	30	15	90	58	73
12	0	+1.682	0	475	20	30	19	95	50	64
13	0	0	-1.682	475	12	10	15	98	60	75
14	0	0	+1.682	475	12	50	20	120	50	64
15	0	0	0	475	12	30	19	102	57	74
16	0	0	0	475	12	30	18	99	59	75
17	0	0	0	475	12	30	16	98	60	75
18	0	0	0	475	12	30	18	101	61	76
19	0	0	0	475	12	30	18	99	59	74
20	0	0	0	475	12	30	18	100	58	73

**Fig.3** Optical micrographs of interface region of Mg-Cu bonds

3 Developing empirical relationships

The responses, diffusion layer thickness (D), interface hardness (H), shear strength (S_S) and bonding strength (S_B) are the functions of bonding temperature (θ), bonding pressure (p) and holding time (t), they can be expressed as

$$D=f\{\theta, p, t\}; H=f\{\theta, p, t\}; S_S=f\{\theta, p, t\}$$

$$\text{and } S_B=f\{\theta, p, t\} \tag{1}$$

The second order polynomial (regression) equation is used to represent the response surface Y . The selected polynomial could be expressed as[9]

$$Y=b_0+b_1(\theta)+b_2(p)+b_3(t)+b_{11}(\theta^2)+b_{22}(p^2)+b_{33}(t^2)+b_{12}(\theta p)+b_{13}(\theta t)+b_{23}(pt) \tag{2}$$

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

For diffusion layer thickness:

$$D=17.85+3.09\theta+1.08p+1.20t \tag{3}$$

For interface hardness:

$$H=99.81+13.60\theta+2.52p+6.81t+1.25\theta t+2.41p^2+3.42t^2 \tag{4}$$

For shear strength:

$$S_S=58.97-4.33\theta-3.26p-3.35t+1.59\theta^2-1.59p^2-1.24t^2 \tag{5}$$

For bonding strength:

$$S_B=74.46-4.58\theta-3.38p-3.48t-1.12\theta p-1.69\theta^2-1.87p^2-1.51t^2 \tag{6}$$

The determination coefficient (R^2) indicates the fitness of the model (Table 5). 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.

4 Results and discussion

Using the empirical relationships developed above, the shear strength and bonding strength were estimated for different combinations of temperature, pressure and holding time. Figs.4–5 represent estimated values of shear strength and bonding strength of diffusion bonded Mg-Cu joints for different combinations of temperature, pressure and holding time. However, shear strength cannot be used to evaluate the extent of bonding because the specimens may have different tempered conditions. It was reported[10] that the actual shear strength requirements of the bonds for aircraft structures are generally in an order of 10–20 MPa. Therefore, 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.

4.1 Effect of bonding temperature on shear strength and bonding strength

Figs.4–5 reveal that shear strength and bonding strength increase with increasing the bonding temperature[11]. It is also evident that the shear strength

Table 5 ANOVA test results for responses

Term	First order terms			Second order terms			Probability > F	R ²	Model
	Sum of squares	Degrees of freedom	Mean square	Sum of squares	Degrees of freedom	Mean square			
Diffusion layer	130.28	3	43.43	1.19	6	0.20			
Interface hardness	2 524.54	3	841.51	168.45	6	28.08			
Shear strength	256.46	3	85.49	32.29	6	5.38			
Bonding strength	286.44	3	95.48	43.10	6	7.18			
Term	Error terms			Lack of fit			Fraction		Model
	Sum of squares	Degrees of freedom	Mean square	Sum of squares	Degrees of freedom	Mean square	Probability > F	R ²	
Diffusion layer	4.83	5	0.97	4.35	5	0.87	<0.000 1	0.948 6	Significant
Interface hardness	10.83	5	2.17	13.73	5	2.75	<0.000 1	0.993 1	Significant
Shear strength	10.00	5	2.00	19.79	5	3.96	<0.000 1	0.956 0	Significant
Bonding strength	5.50	5	1.10	14.66	5	2.93	<0.000 1	0.972 9	Significant

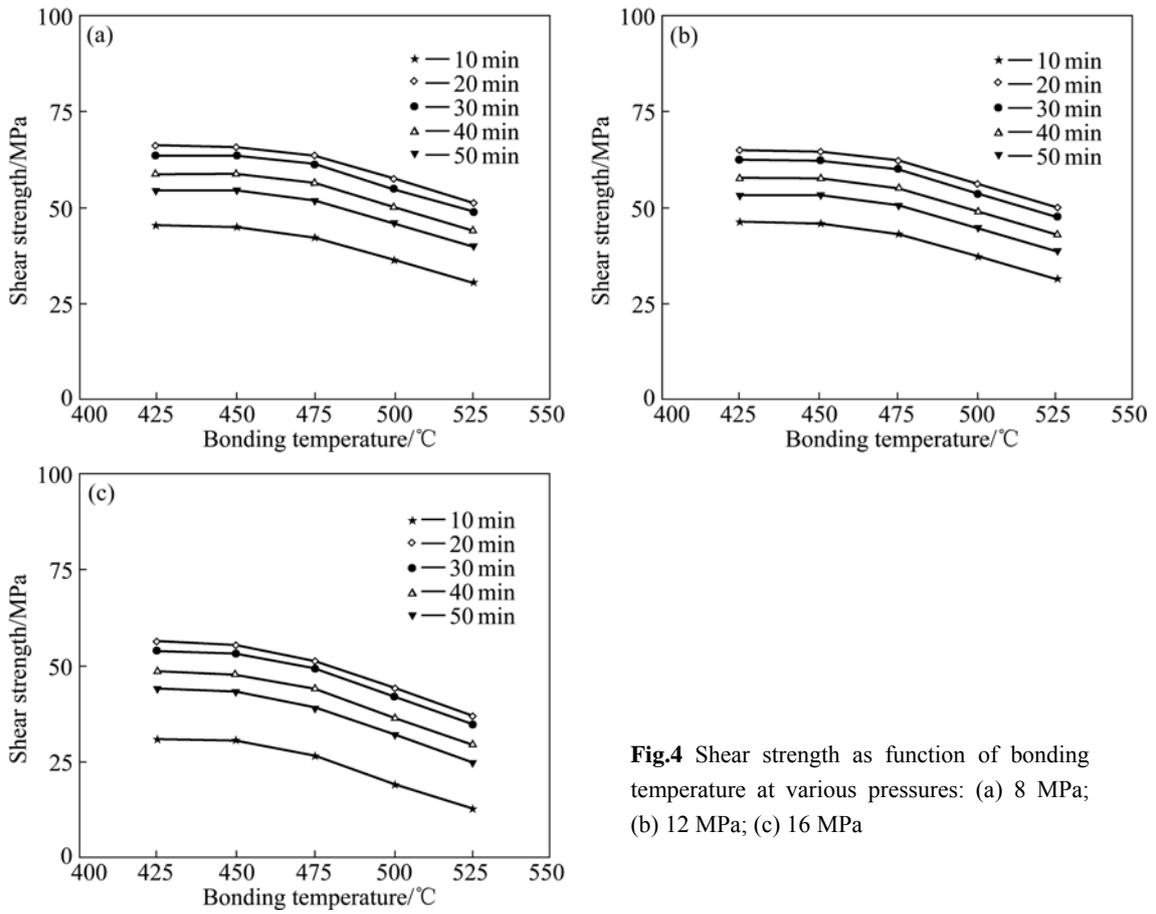


Fig.4 Shear strength as function of bonding temperature at various pressures: (a) 8 MPa; (b) 12 MPa; (c) 16 MPa

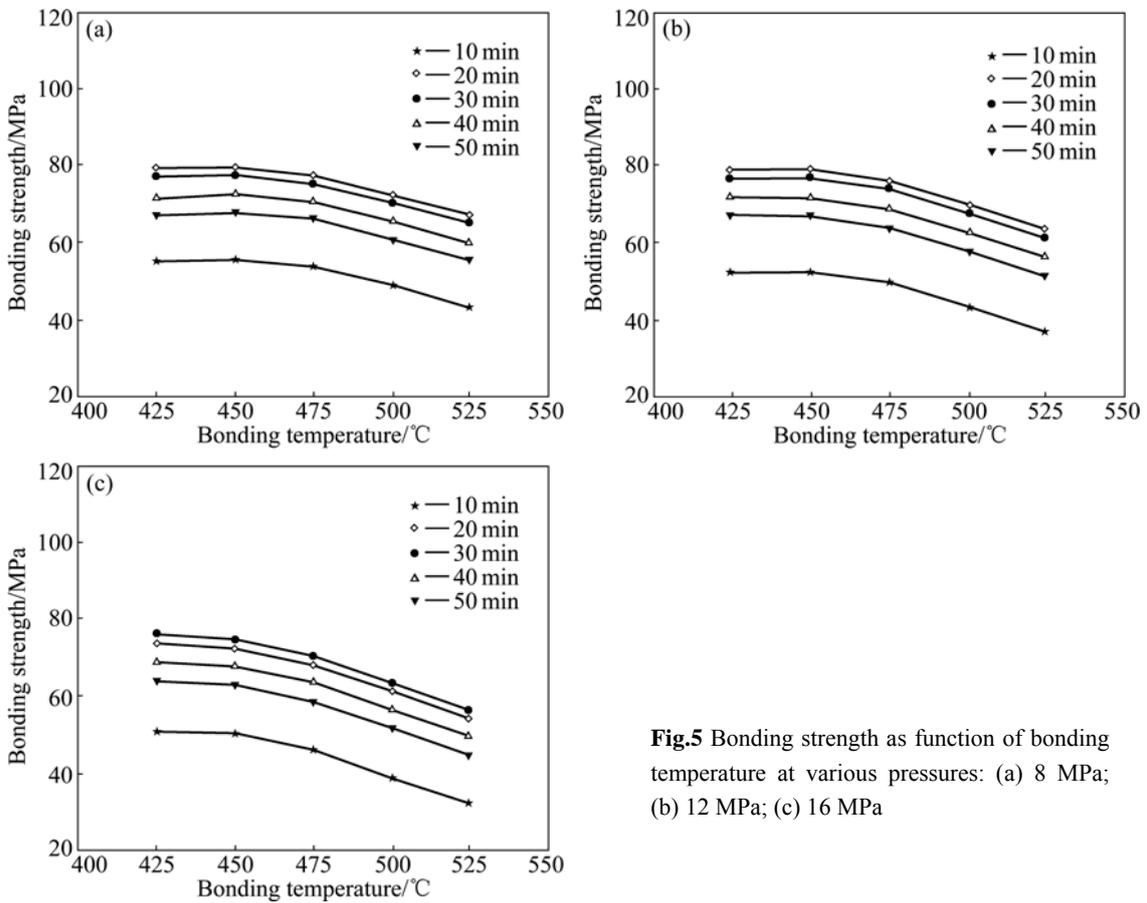


Fig.5 Bonding strength as function of bonding temperature at various pressures: (a) 8 MPa; (b) 12 MPa; (c) 16 MPa

and bonding strength of the joints closely depend on bonding temperature. At a low temperature of 425 °C, the shear strength and bonding strength of the joints are low (40 and 55 MPa, respectively). Generally, atoms on either side of the interface can diffuse into the opposite side during diffusion process only if the temperature is sufficiently high. So, necessary levels of temperatures are usually in the range of 0.6–0.8 T_m (where T_m represents the melting points of the materials involved). At 400 °C, only a small number of Cu atoms might diffuse into the Mg side. This may be the reason for low shear strength and bonding strength. Also, at low temperature, the flowability of metal is substantial yet the yield strength of the base materials remains high, which leads to an incomplete coalescence of the bonding surfaces[12]. Increasing diffusion-bonding temperature to 450 °C results in a considerable improvement in shear strength and bonding strength. Increase in diffusion bonding temperature promotes mass transfer of alloying elements across the interface, which is responsible for the increase in volume fraction of the reaction products and leads to more brittle joints.

However, plastic collapse of the bonding surface asperities leads to intimate contact, which counter-balances the embrittlement due to the intermetallic phases; so, shear strength and bond strength naturally improve and attain their maximum values of 66 and 81 MPa, respectively. In contrast, at high temperature, the initial stages of bonding could involve migration of interface grain boundaries, and the higher rate of grain growth leads to rapid removal of the bond line and increases the strength near the parent metal[13].

When the bonding temperature reaches 525 °C, the eutectic liquid appears in the interface, and a great quantity of Cu elements diffuse into base Mg and form the intermetallic compound, which leads the thickness of the intermetallic compound to increase quickly. Quick increase in thickness of intermetallic compound leads to a decrease in the strength and an increase in the brittleness of the joint. With temperature increasing, the width of brittle intermetallics considerably increases and the embrittlement effect over-balances the positive effect obtained due to the improvement in coalescence of faying surfaces. To confirm the presence of intermetallic compounds at the joint interface of optimum diffusion layer, SEM (Fig.6), XRD (Fig.7) and EDS analysis were carried out and the results are presented in Table 6. As Mg-Cu diffusion bonded joint is a dissimilar joint, the reaction products with new phases are found in the diffusion layer. It is understood that the presence of intermetallic $MgAl_2O_4$, Al_2O_3 , and SiO_2 is responsible for poor strength.

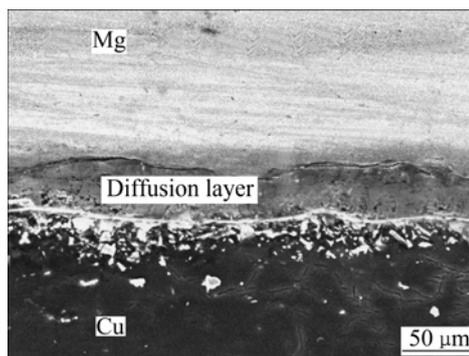


Fig.6 SEM images of Mg/Cu bonds

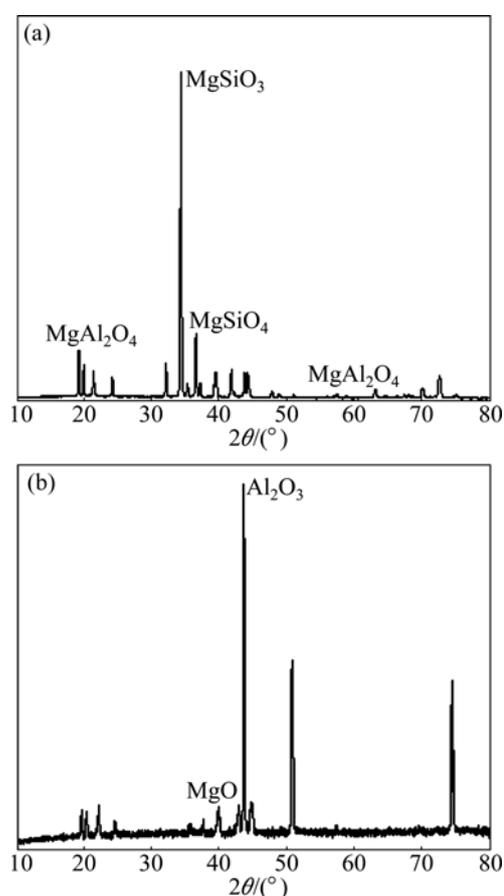


Fig.7 XRD patterns of Mg/Cu bonds: (a) Magnesium side; (b) Copper side

4.2 Effect of bonding pressure on shear strength and bonding strength

Graphically, the dependence of joint strength on bonding pressure can be depicted by a curve as shown in Figs.4–5. The bonding pressure was applied in order to secure a tight contact between the bonding surfaces and a vital condition for the inter-diffusion of atoms of the metals joined. If the bonding pressure is less than the optimum value, the bonding decreases. It can be inferred that the shear strength and bonding strength of the joints increase with increasing the bonding pressure, and they

Table 6 XRD and EDS results of Mg/Cu bond at joint interface

$\theta/^\circ\text{C}$	p/MPa	t/min	$D/\mu\text{m}$	H (Hv)	S_S/MPa	S_B/MPa	EDS results		XRD results
							$w(\text{Mg})/\%$	$w(\text{Cu})/\%$	
450	12	30	18	102	66	81	24.60	51.16	Mg_2SiO_4 , MgAl_2O_4 , MgSiO_2 , SiO_2 , Al_2O_3 ,

irrespective of bonding temperature and holding time. The effect of bonding pressure on strength is less when being compared with temperature and time. Higher shear strength is obtained at a pressure of 12 MPa.

At low bonding pressure of 8 MPa, shear strength and bonding strength are minimum. The explanation is that the bonding surfaces are never perfectly smooth; they are always rough to some extent. When such surfaces are brought together, they contact 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 expand almost instantaneously. When it is increased to 12 MPa, plastic deformation develops at contact sites to increase the contact areas of clean surfaces and hence the joint rate increases a little. Further increase of pressure to 20 MPa, small increase in shear strength and bonding strength is observed. Increase in pressure influences re-crystallization temperature and deformation, and then tends to enhance the contact of bond surface and rapid growth of recrystallization. This obviously increases the interface contact rate and the atoms pass through this bonding interface. Therefore, more diffusion paths are created due to the movement of atoms. The property of the bonded joints also mainly depends on the thickness of the intermetallic compounds, which is not affected by the pressure. The voids formed at the original interface disappear as the contact area expands with time, because the stress within the contact zone causes a plastic flow by either conventional creep or super plasticity. Smaller voids are removed rapidly by diffusion[14].

4.3 Effect of holding time on shear strength and bonding strength

Shear strength and bonding strength of the joints increase with increasing the holding time, and are irrespective of temperature and pressure. High shear strength is obtained at a holding time of 20 min. Holding time has an effect on the creep of the protrusions and the quantity of atomic diffusion. When holding time is not long enough, bonding strength decreases and this may be due to the fact that the holding time is insufficient to cause diffusion of atoms across the bond interface from the bonding surfaces. The strength increases more rapidly with increasing the holding time

up to 50 min and then it decreases sharply. Longer holding time leads to a continuing grain growth accompanied with a small increase in specific strength. The sharp decrease in strength is attributed to the growth of intermetallic compounds. The thickness of the intermetallic compound increases remarkably with holding time, while the tensile strength of the bond joint decreases. In order to obtain high strength, a longer holding time or a higher bonding pressure is required[15].

4.4 Effect of process parameters on diffusion layer thickness

Since the formation of diffusion layer at the interface influences the strength of the bond, it is necessary to analyze the role of diffusion layer on bonding characteristics. 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. While correlating shear strength results and diffusion layer thickness, it is concluded that a narrow diffusion layer thickness of 12 μm (Fig.3(a)) and a relatively wide diffusion layer thickness of 24 μm (Fig.3(c)) both lead to lower shear strength. A diffusion layer thickness of 17.77 μm is found to be optimum to obtain higher shear strength (Fig.3(b)).

Fig.8 reveals the effect of process parameters on diffusion layer thickness of diffusion bonded joints. From the curves, the following inferences can be obtained: 1) Diffusion layer thickness increases with increasing the bonding temperature and holding time; (2) Bonding pressure has the least effect when being compared with bonding temperature and holding time; 3) An optimum diffusion layer thickness of 17.77 μm is obtained (Fig.3(b)) at 475 $^\circ\text{C}$, 12 MPa and a holding time of 30 min. When bonding temperature is taken into consideration, it is seen that diffusion layer thickness depends mainly on bonding temperature. At 425 $^\circ\text{C}$, the diffusion layer thickness is 12 μm , and it increases gradually with increasing the bonding temperature. When the bonding temperature reaches to 525 $^\circ\text{C}$, diffusion layer thickness reaches the maximum. The thickness of diffusion layer depends on atom diffusion [16]. When the bonding temperature increases to over 525 $^\circ\text{C}$, the joining process allows the diffusion of all elements from both metals, which promotes the chemical

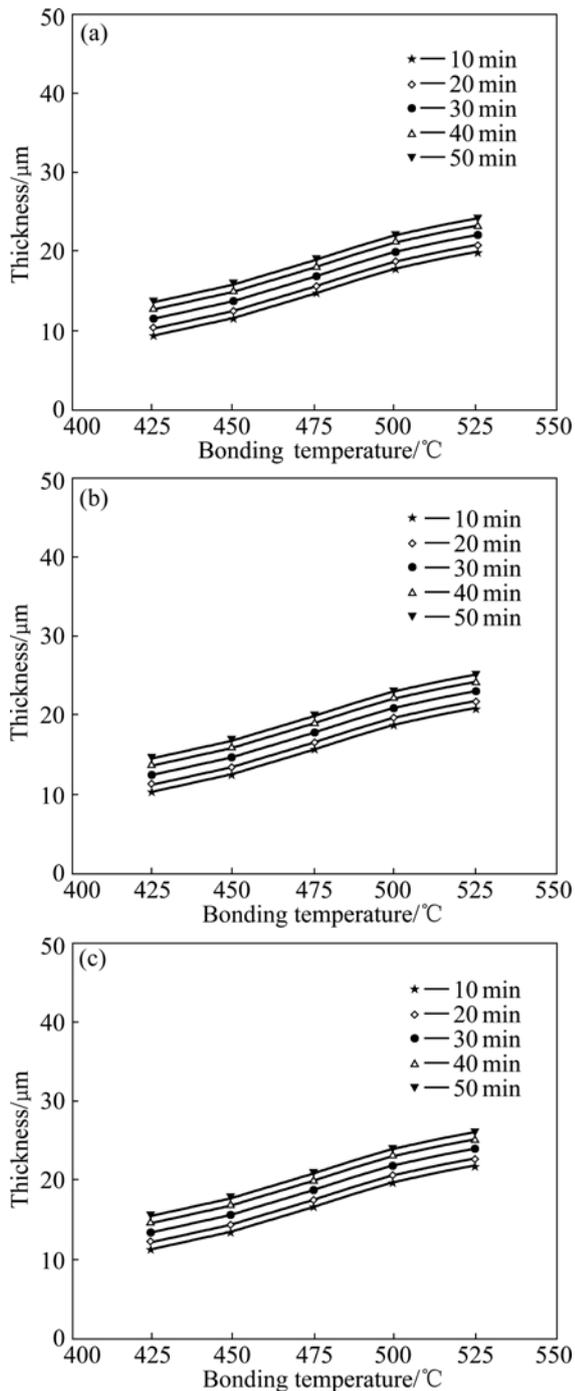


Fig.8 Diffusion layer thickness as function of bonding temperature at various pressures: (a) 8 MPa; (b) 12 MPa; (c) 16 MPa

joint (in all welding condition) between materials when inter-diffusion between the materials is provided without the formation of voids and brittle phases such as intermetallic compounds. These findings are in agreement with Fick's second law, a partial differential equation[17] describing 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 control the resulting mechanical properties. The intermetallic compound grows steadily and gradually at the bond region of dissimilar metal joints with increasing the temperature. The particle distribution of intermetallic compounds has no harmful effects on the joint performances; moreover, it strengthens the joints.

The intermetallic compounds never join up and form a whole body; and they also have no effect on the plasticity and strength of joints. But once they connect and the thickness is more than 5 μm, because of their high brittleness and internal stress, the plasticity and strength of joints obviously decrease. In particular, because the diffusion rate of Mg atoms is much higher than that of Al atoms at high temperature, an excess of Mg atoms diffuses across the interface into the aluminium side and forms cavities according to Kirkendall effect[18], which leads to continuous cavities at the interface and results in a dramatic decrease in the interface bonding strength in the specimens bonded at 525 °C.

4.5 Effect of process parameters on interface hardness

Fig.9 reveals the effect of process parameters on the interface hardness of diffusion bonded Mg-Cu joints. According to the figure, the following inferences can be obtained: 1) The hardness increases with increasing the bonding temperature and holding time; 2) Applied pressure has less effect on hardness; 3) A higher hardness of HV 100 is obtained at a bonding temperature of 500 °C, pressure of 16 MPa and holding time of 40 min. It can be seen that the hardness increases with increasing the temperature and holding time; however, there is no evidence of change in hardness with changing pressure. Increase in hardness with increasing the temperature and holding time can be attributed to the formation of intermetallic compounds as discussed in the previous sections.

5 Conclusions

1) Empirical relationships are developed to predict the bonding strength, shear strength, diffusion layer thickness and interface hardness of the Mg-Cu diffusion bonded joints by incorporating diffusion bonding process parameters.

2) Bonding temperature has greater influence on shear strength and bonding strength, followed by holding time and bonding pressure.

3) Joints fabricated at a bonding temperature of 450 °C, bonding pressure of 12 MPa and holding time of 30 min exhibit maximum shear strength and bonding strength of 66 and 81 MPa, respectively. This may be due to the formation of optimum diffusion layer.

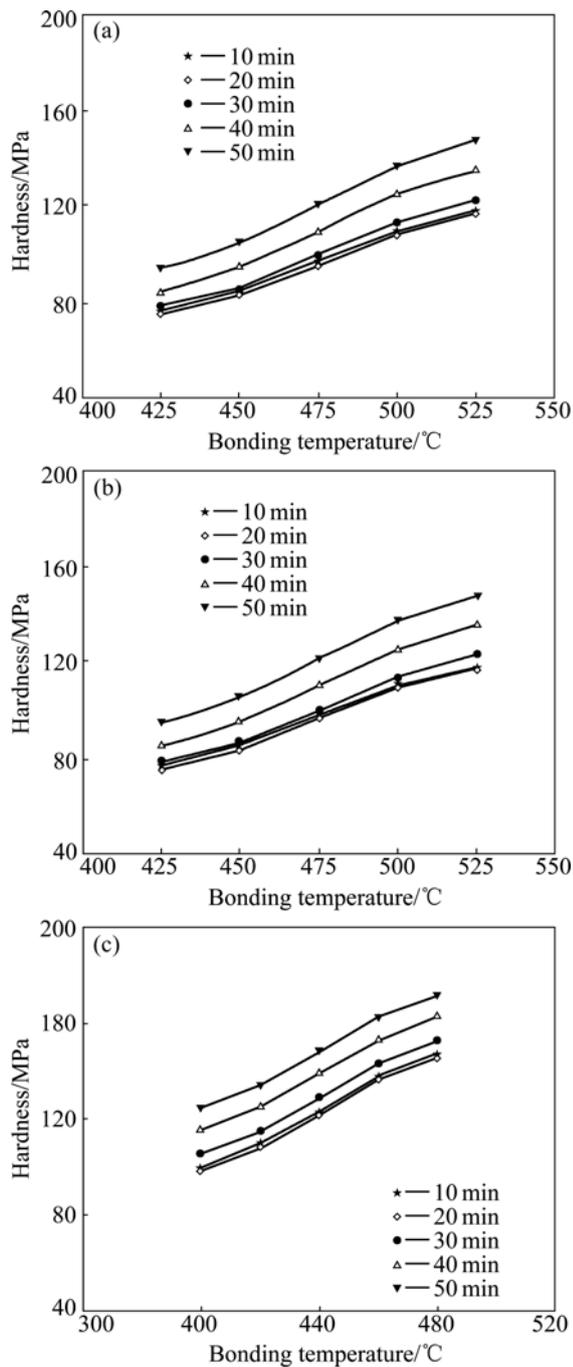


Fig.9 Interface hardness as function of bonding temperature at various pressures: (a) 8 MPa; (b) 12 MPa; (c) 16 MPa

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