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Development of Intermediate Layer for Diffusion Bonding of Continuous Alumina Fiber Reinforced Aluminum Matrix Composite†

Kenji IKEUCHI* and Kazuhiko ASANO**

Abstract

In order to develop an intermediate layer for improving the diffusion bondability of a fiber reinforced aluminum matrix composite, an investigation has been made of critical factors controlling the diffusion bondability on a joint of a continuous alumina fiber reinforced aluminum matrix composite to a commercially pure aluminum and of effects of intermediate layers on the bondability. Al-Mg-Cu alloys containing 0.5-2% Mg and 3-4.5% Cu were employed as the intermediate layer, since Mg and Cu were reported to improve effectively the bondability of aluminum alloys. On the electropolished faying surface of the composite, fibers protruded from the matrix were formed, and had very harmful effects on the diffusion bondability of the composite, interfering with the intimate contact at the bond interface. The diffusion bondability of the composite to the aluminum was improved remarkably by the use of the intermediate layer of the Al-Cu-Mg alloy. The joint strength was increased significantly with Mg content of the intermediate layer.

KEY WORDS: (Aluminum Matrix Composite) (Alumina Fiber) (Aluminum) (Diffusion Bonding) (Intermediate Layer) (Al-Mg-Cu Alloy)

1. Introduction

The aluminum and its alloy, most prevalent metallic matrix of the fiber reinforced metal matrix composite, have very poor diffusion bondability because of the presence of the very stable and tenacious oxide film on the faying surface[1]. For this, it has been said that bonding temperatures as high as the solidus of the alloy and bonding pressures high enough to produce heavy plastic deformation at the bond interface are necessary to obtain 100% joint efficiency by the diffusion bonding of the aluminum alloys[2]. However, as well known[3], to expose the metal matrix composite to high temperatures and high pressures causes in many cases serious impairment of mechanical properties of FRM. For this reason, only little attention has been paid to the diffusion bonding of the aluminum alloy matrix composite, though the diffusion bonding is widely applied to fabricating the metal matrix composite itself[4]. However, we have reported in previous papers[5,6] that the bonding temperature and plastic deformation required to obtain high joint efficiency of aluminum alloys can be decreased by the addition of alloying elements; e.g., Mg can promote breakup of the oxide film by reacting with the oxide film to form $MgAl_2O_4$[5], and Cu can promote the intimate contact and the breakup of the oxide film by forming a liquid phase at lower bonding temperatures[6] (by decreasing the solidus temperature).

The present investigation, therefore, is aimed at developing an intermediate layer of aluminum alloys for facilitating the diffusion bonding of the aluminum alloy matrix composite. For this, we have investigated critical factors controlling the diffusion bondability of a continuous fiber reinforced aluminum matrix composite to a commercially pure aluminum, applied Al-Mg-Cu alloys of various Mg and Cu contents as the intermediate layer, and discussed the mechanism explaining the effect of the intermediate layer on the bondability.

2. Experimental Details

The aluminum matrix composite used was prepared from a continuous alumina fiber reinforced aluminum matrix composite (abbreviated as FRM). The sections of the FRM specimen perpendicular and parallel to the fiber are shown in **Figs. 1(a)** and **1(b)**, respectively. The alumina fiber included 15% SiO_2, and its volume

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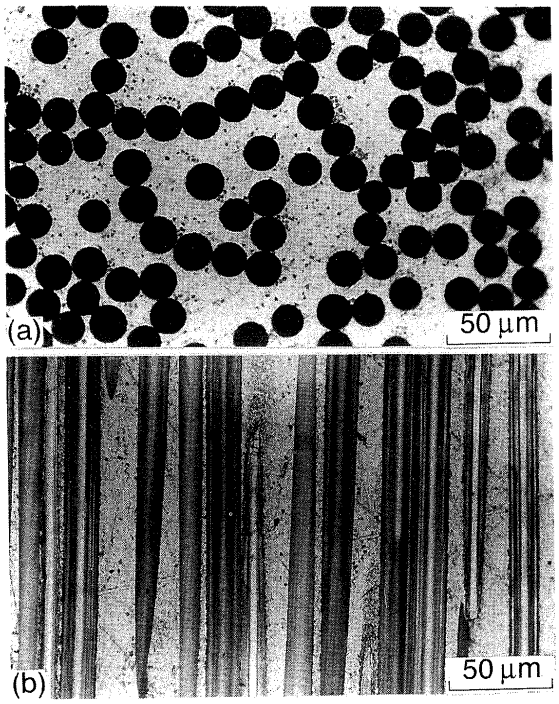


Fig. 1 Microstructures of the base metal of FRM: (a) perpendicular and (b) parallel sections to the reinforcement fiber.

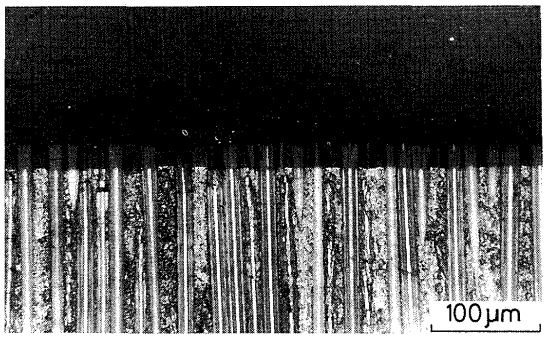


Fig. 2 Section perpendicular to a faying surface of FRM.

fraction was 50%. The matrix of the FRM was a commercially pure aluminum. The faying surface was the section perpendicular to the direction of the fiber, viz., the direction of the maximum strength, as shown in **Fig. 2.** The faying surface was finished by electropolishing in a solution of 20% hyperchloric acid in ethylalcohol. As shown in Fig. 2, fibers were protruded from the matrix surface, since the matrix was dissolved preferentially by the electropolishing. The protruded height of the fiber was increased proportionately with electropolishing time. The FRM specimen was bonded to a commercially pure aluminum 1050 (abbreviated as Al) and an Al-Mg-Si series 6063 alloy (abbreviated as 6063 alloy) which was often used as a matrix of composites reinforced with alumina fibers. The Al and 6063 alloy specimens were cut from rods of 20 mm diameter having chemical compositions as shown in **Table 1.** Faying surfaces of these specimens were finished by electropolishing. The electrolytes were a solution of 20% hyperchloric acid in ethylalcohol for the Al specimen and a solution of 10% hyperchloric acid in ethylalcohol for the 6063 alloy specimen.

Table 1 Chemical compositions of the base metals of a commercially pure aluminum 1050 and an Al-Mg-Si alloy 6063 (mass%).

Specimen	Cu	Si	Fe	Mn	Mg	Zn	Cr	Ti	Al
Al (1050)	0.01	0.08	0.12	tr.	tr.	tr.	0.01	0.01	Bal.
6063 alloy	tr.	0.43	0.18	0.01	0.54	0.01	tr.	0.01	Bal.

In order to improve the diffusion bondability of FRM, intermediate layers of Al-Mg-Cu alloys having chemical compositions as shown in **Table 2** were applied. For the Al-(2-0.5%)Mg-(3-4.5%)Cu alloys, the Cu content was decreased as the Mg content was increased in order for the solidus temperature to be kept almost constant. The solidus temperatures of these alloys were estimated from electric resistance - temperature curves. The solidus temperatures of the Al-2%Mg-3%Cu, Al-1.5%Mg-3.5%Cu, and Al-1%Mg-4%Cu alloys lay around 843-853 K, and that of the Al-0.5%Mg-4.5%Cu alloy around 833-843 K. Intermediate layers of Al-Cu-Mg series 2017, Al-6%Cu, and Al-2%Mg alloys were also employed for comparison. The solidus temperature of the Al-6%Cu alloy lay around 803-813 K, and that of the Al-2%Mg alloy around 893 K. The thickness of the intermediate layers was 60-80 μm.

Table 2 Chemical compositions of intermediate layers employed (mass%).

Intermediate layer	Cu	Si	Fe	Mn	Mg	Zn	Cr	Ti	Al
2017 alloy	3.92	0.55	0.30	0.68	0.55	0.08	0.04	0.04	Bal.
Al-2.0%Mg-3.0%Cu	3.06	0.02	tr.	0.01	2.17	tr.	tr.	tr.	Bal.
Al-1.5%Mg-3.5%Cu	3.44	0.02	tr.	0.01	1.67	tr.	tr.	tr.	Bal.
Al-1.0%Mg-4.0%Cu	3.98	0.02	tr.	0.01	1.03	tr.	tr.	tr.	Bal.
Al-0.5%Mg-4.5%Cu	4.32	0.03	tr.	0.01	0.54	tr.	tr.	tr.	Bal.
Al-6.0%Cu	6.04	0.02	0.01	0.01	tr.	tr.	tr.	tr.	Bal.
Al-2.0%Mg	0.01	0.01	tr.	0.01	2.03	tr.	tr.	tr.	Bal.

In the present investigation, the true contact area at the bond interface was estimated from the electric resistance across the bond interface. Dimensions of the specimens for the electric resistance measurement are shown in **Fig. 3.** The electric resistance measurement permitted to evaluate the bondability using very small

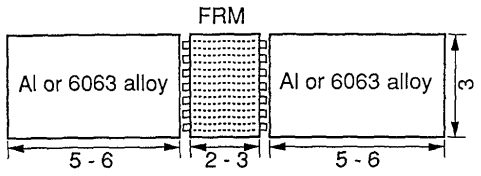


Fig. 3 Dimensions of the specimens for the electric resistance measurement across the bond interface.

specimens. The electric resistance measurement was carried out during heating these specimens from room temperature at a constant heating rate and constant bonding pressure in a vacuum of the order of 10^{-2} Pa (see ref.(7) for details). A vacuum furnace equipped with a spring to apply a bonding pressure was used for heating the specimens as shown in **Fig. 4**. A conventional potentiometric method was used for the electric resistance measurement as shown in Fig. 4.

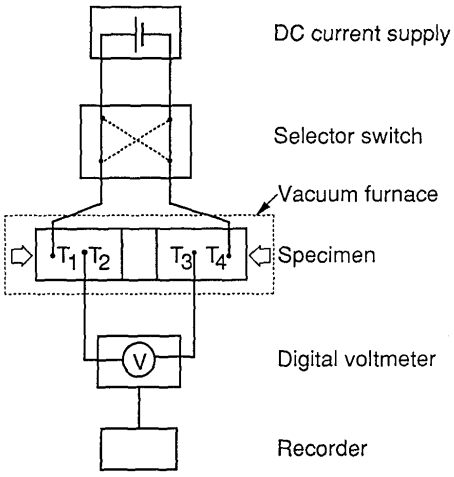


Fig. 4 Circuit for the electrical resistance measurement across the bond interface.

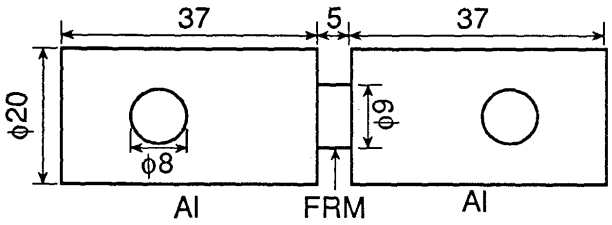


Fig. 5 Dimensions of specimens for tensile test of the joint.

The strength of joints was estimated from the tensile strength of specimens as shown in **Fig. 5**. The specimen was subjected to the tensile test without machining after diffusion bonding. The diffusion bonding of the tensile test specimen was carried out in a vacuum of the order of 10^{-2} Pa using an apparatus reported in a previous paper[8]; i.e., the bonding zone was heated with a radiant resistance heater of molybdenum foil, the bonding pressure was applied with a hydraulic press, and the temperature of the bond interface was monitored with a C-A thermocouple percussion-welded on the side surface of the Al specimen about 2 mm away from the bond interface and kept constant within an accuracy of ±1 K.

3. Results and Discussion

In order to evaluate the diffusion bondability of FRM to Al, change in the true contact area during heating the specimens shown in Fig. 3 were examined by the electric resistance measurement across the bond interface. The electric resistance across the bond interface is shown as a function of temperature in **Fig. 6**. Open marks in Fig. 6 stand for the electric resistance during heating the specimens from room temperature at bonding pressures of 1.5-3 MPa. Closed marks stand for the electric resistance during the cooling process subsequent to the heating. As shown in Fig. 6, the electric resistance across the bond interface was decreased, as the temperature and pressure were increased. However, the

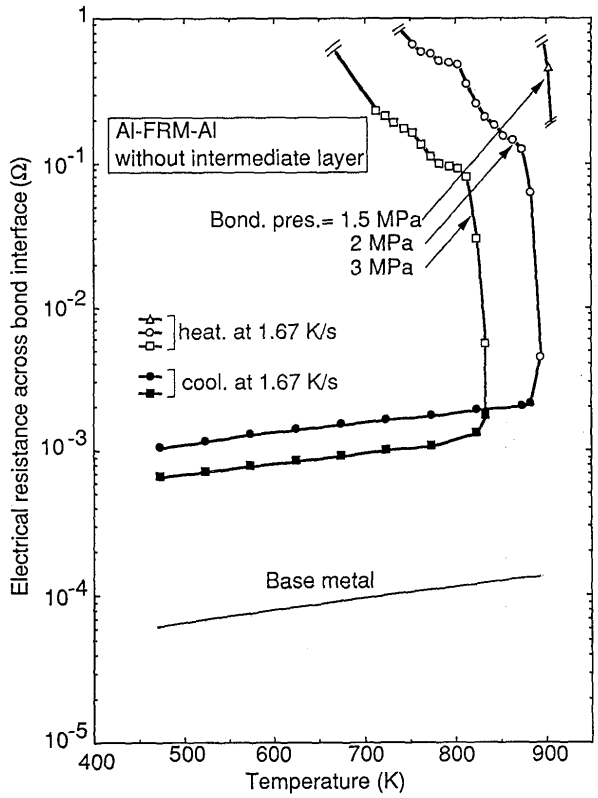


Fig. 6 Electrical resistance across the bond interface of FRM to Al vs. temperature during heating and cooling at a rate of 1.67 K/s.

electric resistance across the bond interface was about an order of magnitude greater than that of the base metal even after heating to the highest temperature, above which the Al specimens were deformed too heavily to continue the further measurements. According to the constriction resistance theory[7], the true contact area S_M at the bond interface is given by the following equation:

$$S_M / S = \pi \cdot \rho_M^2 / (4 \cdot n \cdot R_S^2) \quad (1),$$

where S is the apparent contact area, n the number of true contact spots per a unit area, R_S the resistance of the bond interface of a unit area, and ρ_M the resistivity of the base metal. Since the electric resistance across the bond interface R consists of the electric resistance of the bond interface and that of the base metal between terminals T_2 and T_3 as shown in Fig. 4,

$$R = 2 \cdot R_S / S + \rho_M \cdot l / S \quad (2),$$

where l is the distance between terminals T_2 and T_3. By eliminating R_S from eqs. (1) and (2), the true contact area was estimated from the electric resistance across the bond interface R. The estimated true contact area was less than 0.5% of the apparent contact area even at the highest heating temperature possible.

In contrast to this, the diffusion bonding of Al to Al, as shown in **Fig. 7**, the electric resistance across the bond interface became almost equivalent to the base metal resistance at temperatures above 853 K (Since the electric resistance across the bond interface was very close to the base metal resistance, they are indistinguishable in Fig. 7). These results suggest that the diffusion bondability of FRM to Al is extremely poor compared with that of Al to Al.

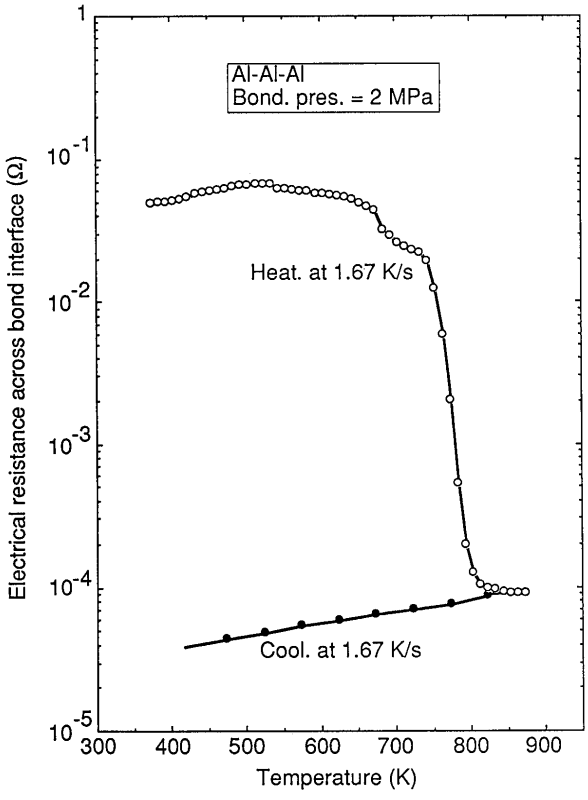


Fig. 7 Electrical resistance across the bond interface of Al to Al vs. temperarture during heating and cooling at a rate of 1.67 K/s.

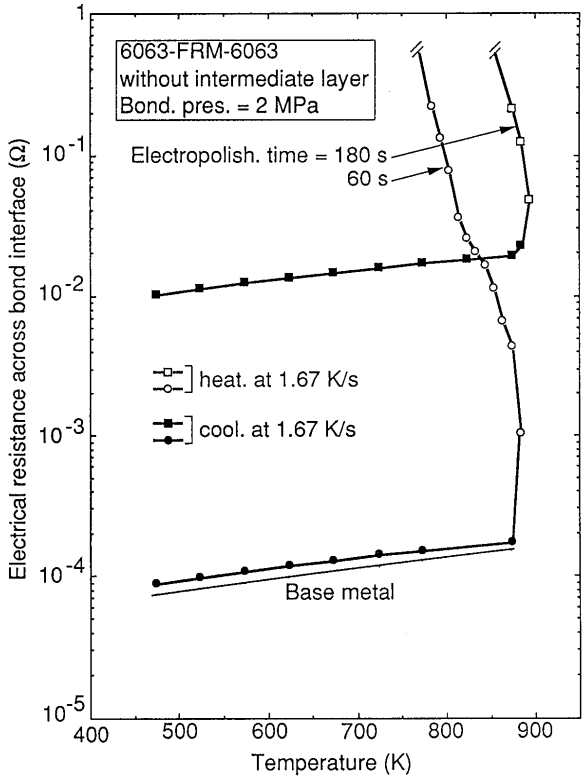
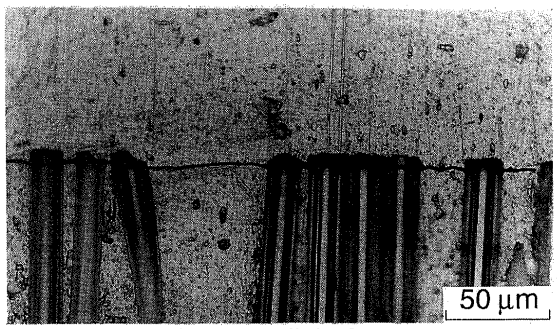


Fig. 8 Electrical resistance across the bond interface of FRM to 6063 alloy vs. temperarture during heating and cooling at a rate of 1.67 K/s.

In order to examine the effect of alloying elements on the bondability, the electric resistance measurement was carried out on joints of FRM to 6063 alloy. As can be seen from **Fig. 8**, the electric resistance across the FRM-6063 alloy bond interface was significantly lower than that of the FRM-Al bond interface at all temperatures tested. This suggests that the addition of Mg, a major alloying element of 6063 alloy, is effective in improving the bondability. On the other hand, the effect of the electropolishing of the FRM faying surface on the bondability is also shown in Fig. 8; i.e., the electric resistance across the bond interface became much higher when the electropolishing time was increased. In



(a) Electropolish. for 60 s

(b) Electropolish. for 180 s

Fig. 9 Effect of electropolishing time of the FRM faying surface on the microstructure of FRM-6063 alloy joints.

order to explain this result, we observed the microstructure of the joints after the electric resistance measurement.

As shown in **Fig. 9**, the protruded height of the fiber was increased, as the electropolishing time was increased, and uncontacted area at the bond interface was much increased. We also observed similar phenomena for the joint of FRM to Al. These results suggest that the protruded fiber interfered with the intimate contact between the FRM and Al specimens. We think that the very poor bondability of FRM to Al is due to the interference of the intimate contact by the protruded fiber.

In order to improve the poor bondability of FRM to Al, we applied intermediate layers of aluminum alloys. One of the alloying elements for the intermediate layer was Mg, since the electric resistance measurement suggests that Mg can improve the bondability. Another alloying element was Cu in order to take advantage of the liquid phase forming at lower bonding temperatures.

As shown in **Fig. 10**, when intermediate layers of Al-Cu-Mg alloys were applied, the electric resistance across the FRM-Al bond interface became almost equivalent to the base metal resistance at temperatures

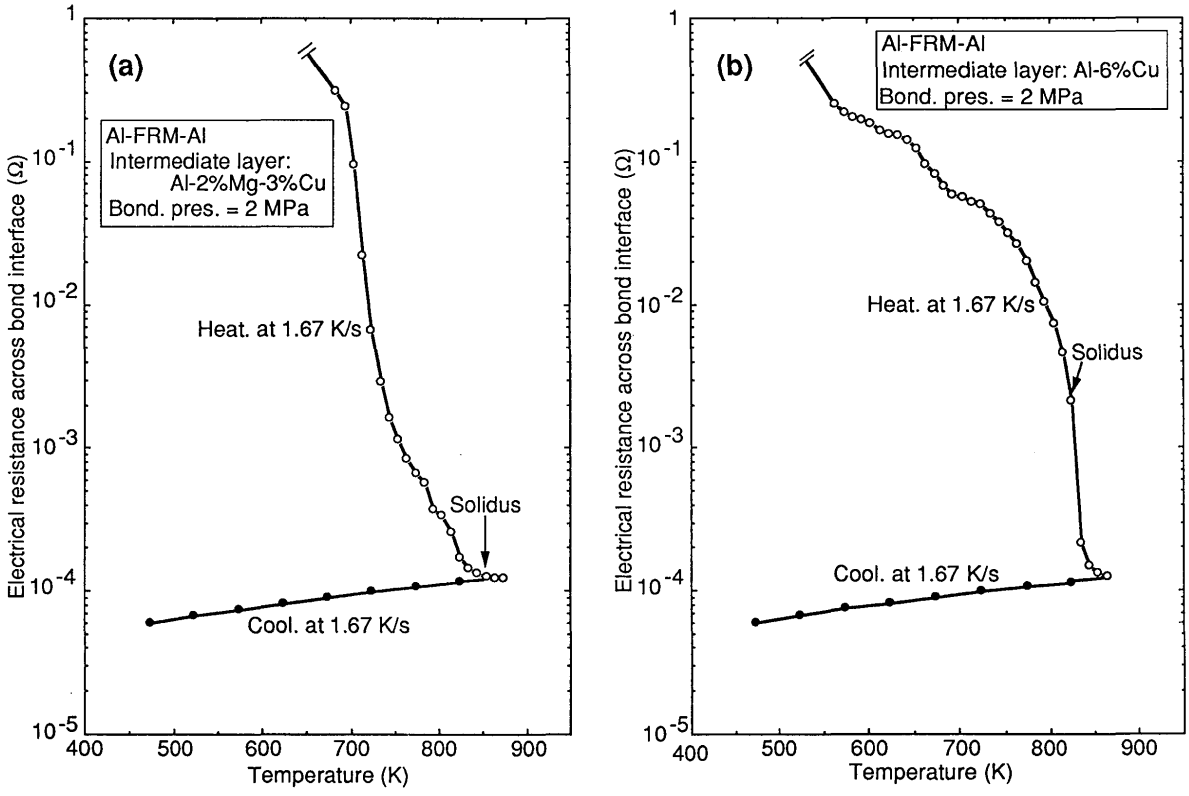


Fig. 10 Effect of the intermediate layer on the relation between electric resistance across the bond inetrface and temperature: (a) intermediate layer of Al-2%Mg-3%Cu alloy, and (b) intermediate layer of Al-6%Cu alloy.

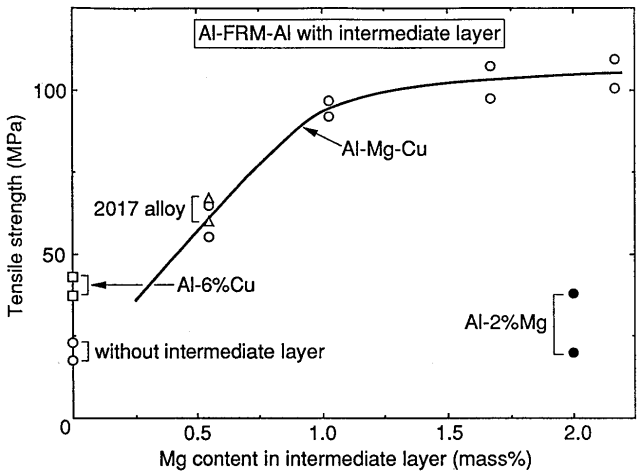


Fig. 11 Tensile strength vs. Mg content of the intermediate layer for FRM-Al joints with intermediate laters of Al-Cu-Mg alloys.

around 873 K. This means that the decrease in the electric resistance across the bond interface during heating was accelerated remarkably by the use of intermediate layers of Al-Mg-Cu alloys. Comparing the electric resistance-temperature curves shown in Figs. 10(a) and 10(b), we can see that the electric resistance across the bond interface approached the base metal resistance at lower heating temperatures when Mg content of the intermediate layer was higher. These results indicate that

(1) the intermediate layer of the Al-Mg-Cu alloy can improve remarkably the bondability of FRM to Al,

(2) the intermediate layer is more effective as Mg content was increased, and

(3) bonding temperatures above the solidus of the intermediate layer is necessary to obtain a sound joint.

In order to test the effectiveness of the intermediate layer on the actual diffusion-bonded joint, tensile tests

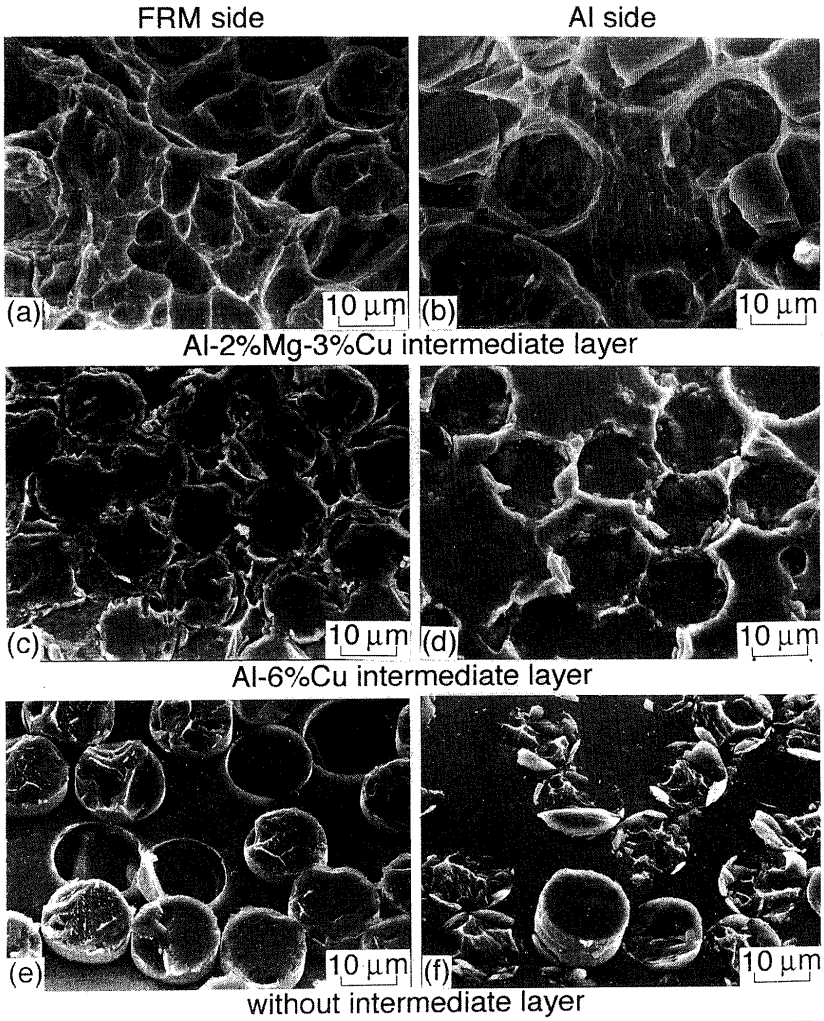


Fig. 12 Fractured surfaces of FRM-Al joints subjected to tensile test: (a) and (b) intermediate layer of the Al-2%-3%Cu alloy , (c) and (d) intermediate layer of the Al-6%Cu alloy, and (e) and (f) no intermediate layer.

were carried out on joints of FRM to Al bonded with the intermediate layer listed in Table 2 at a temperature of 873 K (see Fig. 10). As shown in **Fig. 11**, though bonding parameters for all the joints tested were the same (temperature = 873 K, pressure = 2 MPa, time = 1.8 ks), the tensile strength of the joint was increased significantly by the use of the intermediate layer of Al-Mg-Cu alloys, and the strength was increased with Mg content of the intermediate layer in accordance with the results of electric resistance measurement.

Among the intermediate layer used, the effect of the Al-2%Mg intermediate layer was relatively weak as shown in Fig. 11. At a bonding temperature of 873 K, while other intermediate layers included a liquid phase, only the Al-2%Mg alloy was in the solid state. Therefore, this result suggests that the addition of Mg is not effective unless a liquid phase is included in the intermediate layer.

In order to explain the effect of the intermediate layer on the joint strength, fracture surfaces of joints subjected to tensile test were observed. When no intermediate layer was applied, as shown in **Figs. 12(e)** and **12(f)**, fragments of alumina fibers were observed on the fractured surface of the Al side, but the rest of the fracture surface of the Al side and the surface of the FRM matrix were quite smooth without any dimple pattern. Only small pieces of Al were observed to adhere to the top of the protruded fiber on the fractured surface of the FRM side. These results suggest that in this case the intimate contact between the surfaces of the FRM matrix and Al was not attained and that the bond was formed only between the top of the protruded fiber and Al surface.

In the case of the intermediate layer of Al-6%Cu alloy, as shown in **Figs. 12(c)** and **12(d)**, the profile of the protruded fiber was transferred to the fractured surface of the Al side, but ductile fracture morphology could hardly be observed. This means that in this case the intimate contact between the FRM surface and the intermediate layer was achieved, but the bond strength of the interfaces between the intermediate layer and the fiber and between the intermediate layer and matrix of FRM was rather weak. The weak bond strength of these interfaces can probably explain why the intermediate layer of the Al-6%Cu alloy was less effective in improving the joint strength than those of the alloys of higher Mg contents (see Fig. 11). The coexisting liquid phase in the Al-6%Cu alloy at the bonding temperature probably contributed to the promotion of the intimate contact at the bond interface.

On the other hand, in the case of the intermediate layer of the Al-2%Mg-3%Cu alloy with which the highest joint strength was obtained, dimple patterns were observed both on the fiber surface and on the matrix, with many fiber fragments embedded in the fractured surface of the Al side, as shown in **Figs. 12(a)** and **12(b)**. These results suggest that in this case, the intimate contact between the FRM and intermediate layer surfaces was attained, and the intermediate layer was bonded rather strongly both to the FRM matrix and to the fiber. For this, we think, the highest bond strength was obtained by using the intermediate layer of the Al-2%Mg-3%Cu alloy. The high bond strength of this intermediate layer both to the FRM matrix and to the fiber can probably be attributed to the reaction of Mg with the fiber and the superficial oxide film to form $MgAl_2O_4$ as reported in the previous paper[5].

4. Conclusions

In order to develop an intermediate layer for facilitating the diffusion bonding of FRM to Al, an investigation has been made of the critical factors controlling the bondability, effects of intermediate layers of Al-Cu-Mg alloys on the joint strength, and the mechanism by which the intermediate layer influenced the joint strength. Results obtained are summarized as follows:

(1) The protruded fiber on the electropolished faying surface of FRM is a major factor which makes the diffusion bondability of FRM very poor compared with monolithic aluminum alloys.

(2) The diffusion bondability of FRM to Al was improved significantly by the use of the intermediate layer of the Al-Mg-Cu alloy. The joint strength was increased with Mg content of the intermediate layer.

(3) Observation of the fractured morphology of the joint suggests that the effect of the Cu addition to the intermediate layer is to promote the intimate contact by producing a liquid phase at lower bonding temperatures and that the effect of the Mg addition is to increase the bond strength of the intermediate layer both to the FRM matrix and to the fiber. We think these effects of the Cu and Mg addition can explain the improvement of the joint strength by the use of the intermediate layer of Al-Mg-Cu alloys.

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