

# Vacuum-free diffusion bonding of aluminium metal matrix composite

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## Abstract

Owing to their high specific strength aluminium metal–matrix composites (Al-MMC) are becoming popular for many critical engineering applications. These materials are prepared by controlled melting and solidification processing. In order to broaden their applications further, it is necessary to develop suitable joining techniques. Due to the metallurgical nature of these materials, conventional fusion welding techniques such as arc welding cannot be used. Diffusion bonding is one of the processes that has the potential for the joining of these materials without degrading their properties. At present, these techniques are used in vacuum, which is a time-consuming and expensive operation. It also puts a limit on the size of the component that can be bonded successfully. Therefore, a novel technique of vacuum-free diffusion bonding was investigated, where bonding was achieved in the open air with the aid of in situ surface treatment. The aim of this treatment was to disrupt and disperse the oxide layer and maintain a clean interface during diffusion bonding. The results of the investigation have shown that a joint efficiency of 90% and a percentage bonded area of over 75% can be achieved. This study involves the determination of the experimental set-up and the evaluation of the diffusion-bonding parameters. Metallographic studies and other evaluations show that this in situ surface treatment process is able to provide a clean joining surface despite the presence of a tenacious oxide layer. © 1999 Elsevier Science S.A. All rights reserved.

**Keywords:** Diffusion bonding; Al-MMC; Vacuum-free

## 1. Introduction

Diffusion bonding, also known as diffusion welding, has been used widely by ancient civilizations since the dawn of time. It was used to make artifacts such as gold cups and chalices in Euphrates during 3200 BC [1]. Diffusion bonding appears to be a simple, effective and ancient process as shown by its widespread usage in ancient times. In spite of its humble origin, the modern diffusion process is in the forefront of technology, as it is a joining method used for a wide range of advance alloys and composites [2–4].

Modern diffusion bonding processes were developed mainly for heat-sensitive materials; for materials with a tenacious oxide layer, and for composites that provide a high degree of difficulty for joining by other processes. This process is also used in materials that are used for structural application. Moreover, as a viable fabrication process, it has to be relatively low-cost, easy to apply, highly productive and applicable to diverse situations. In addition, the resultant joint material should have uniform properties that are similar to those of the original material. Consequently, the demands

on the modern diffusion bonding process have increased greatly.

Diffusion bonding is a type of pressure welding method that involves the inter-diffusion of atoms across the interface of the weld. The interface may be separated by an inter-layer that can be either in the solid or liquid state. Hence, diffusion bonding requires that the pieces to be joined are brought to within the atomic distance through localized plastic deformation in the weld zone, and that the mating surfaces are clean [5].

Diffusion bonding is a relatively simple joining process, which is controlled by three important process parameters. These are bonding temperature, pressure and holding or dwell time. In addition, these three parameters are inter-related and thus have an effect on each other. The bonding temperature should be between 50 and 70% of the melting point (in absolute temperature) of either the lowest melting temperature or major phase. The use of elevated temperature will aid the inter-diffusion of atoms across the interface of the weld and assist surface deformation. The bonding pressure should be high enough to ensure a tight contact between the joining surfaces. Moreover, it should be sufficient to aid in the deformation of surface asperities and to fill all the voids in the weld zone. Also, the holding time should be

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optimized to achieve the desired joint quality. It should be sufficient for an intimate contact to be formed and for the diffusion process to take place. However, excessive holding time may lead to degradation of physical and chemical properties.

Diffusion bonding process parameters such as temperature, pressure, holding time and surface roughness can be controlled accurately with relative ease and at low cost. However, the difficulties and high cost of removing the oxide layer have limited the usage of the diffusion-bonding process in many industrial applications. The cost of oxide removal and the maintenance of a clean surface are due to the requirement of a vacuum system, or dangerous chemicals that need special handling and disposal.

## 2. Experimental work

The base metal used for the experimental work contains 20% volume of aluminium oxide, while the matrix was an aluminium alloy 6061 that has undergone T6 heat treatment. The reinforcement particle diameter was 28  $\mu\text{m}$  and the median reinforcement particle spacing was 42  $\mu\text{m}$  in the Al-MMC. The chemical composition of the metal matrix is given as 0.63% Si, 0.85% Mg, 0.24% Cu, 0.45% Fe, 0.15%, Cr, 0.11% Mn, 0.04% Zn, 0.04% Ti, 0.005% Ni, with the remainder being Al. The test specimen is cylindrical in shape with a diameter of  $12 \pm 0.3$  mm and a height of  $20 \pm 0.3$  mm. The initial joint surface was determined as having an average peak-to-valley height of 12.5684  $\mu\text{m}$ , on average distance between two peaks of 1.0865  $\mu\text{m}$  and an average frequency of the peaks of 0.7735  $\mu\text{m}$ , with a variance of  $\pm 2\%$ . The compressive yield stress of the Al-MMC is 10.69 MPa at 450°C.

There is a need for this study to modify known diffusion bonding methods as there are no prior studies on the vacuum-free solid state diffusion bonding process of Al MMC. The diffusion bonding process used by Li and Zuruzi [6] is modified and conducted in the following manner. The selections of the bonding process parameters are based on results obtained from studies on the diffusion bonding of Al T6061 alloy made by Li and Zuruzi [6], and literature research and on results of a trial diffusion bonding process.

Firstly, the recommended diffusion bonding temperature range was 466K (193°C)–653K (380°C), as Al has a melting point of 933K (660°C). This is based on a literature search, which recommended 0.5–0.7 of the melting point in degrees absolute. However, a diffusion bonding temperature of 400–450°C is found to be more suitable for the diffusion bonding of aluminium alloy 6061, as shown by results from trial runs and from the work of Li and Zuruzi [6]. Consequently, a diffusion bonding temperature of 450°C is used in this study.

The diffusion bonding time ranges from 3 to 90 min as has been commonly used in other studies. In this study, diffusion bonding times of between 6 and 20 min are used and are based on results from the trial run.

The selected initial pressure is based on the compressive yield stress of the material, as recommended by other diffusion studies. However, the use of pressure equal to the yield stress was found to be unsatisfactory and pressure higher than the yield stress value was used for most of the specimens. Nevertheless, it should be pointed out that insufficient numbers of specimens were tested at the yield stress for any conclusion to be drawn.

The selection of the surface condition was hampered by the fact that no recommended surface roughness value was found for Al-MMC. Consequently, the surface roughness is based on a conservative estimation of the expected material flow and literature research. First, other diffusion studies recommended a surface roughness of not more than 40  $\mu\text{m}$  for a soft material such as aluminium and 2.5  $\mu\text{m}$  for a hard material such as ceramics [5]. Accordingly, Al-MMC, which is harder than an Al alloy but softer than ceramic, should require a surface roughness that is between that of these two materials. However, a simple mixture rule cannot be applied, as the flow of material (creep) will be contributed to, mainly by the soft aluminium matrix instead of by the reinforcement. Hence, the estimated allowable surface roughness should not be greater than 20  $\mu\text{m}$  for the diffusion bonding process of Al-MMC in this study. Results from the trial run have shown that the surface preparation used is sufficient.

Lastly, the oxide layer removal was conducted by the in situ surface treatment that is mentioned in the study conducted by Li and Zuruzi [6]. The in situ surface treatment requires the use of freshly ground specimens that were subjected to a load before bonding. This loading is followed by the rotation (turning) of the specimens with respect to one another in a direction parallel to the bond plane under an applied load.

The furnace was pre-heated to the required temperature (450°C) and allowed to stabilize for at least half an hour. The specimens were then placed into the furnace and a load of 1 kN (about 60% of the yield stress) was applied for a pre-determined time of 6 min. The specimen was given the in situ surface treatment by four 90° rotations of the top specimen with respect to the bottom specimen. A bonding load of 2 kN was used to achieve the required bonding pressure of about 160% yield stress. The load was applied for a holding time of either 6 or 8 min. After the holding time, the load was released slowly and the specimen was removed from the furnace. Finally, the specimen was cooled in open air to room temperature. The diffusion bonding cycle is shown in Fig. 1.

Bond assessments were carried through microscopic observation and mechanical testing. Microscopic studies involve the observation of the bond line and the surrounding region of bonded specimens. In addition, fracture surface of tensile specimens were conducted for the determination of the percentage bonded area and the type of fracture. The metallographic specimens were etched in freshly prepared Poulant's reagent for 5 s. Mechanical testing involved the

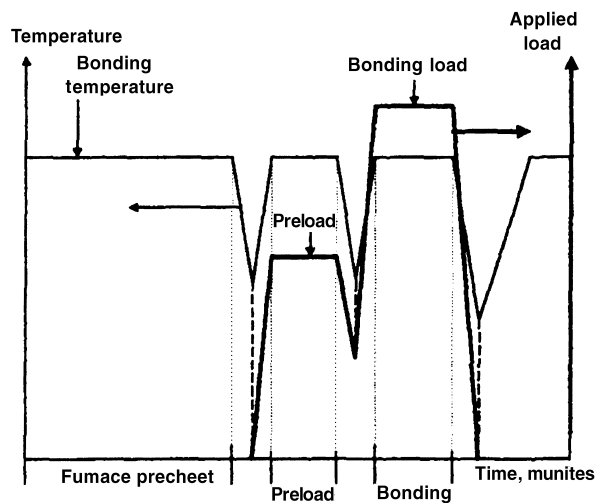


Fig. 1. Graphical description of the diffusion bonding-process cycle, with time vs. temperature and applied load.

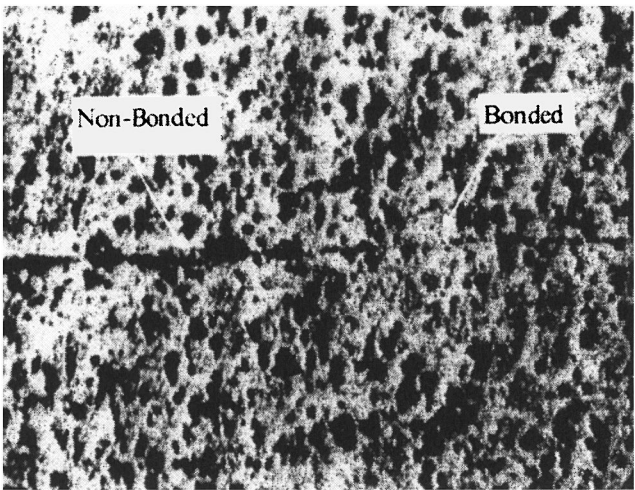


Fig. 2. Bonded and non-bonded region at the bond line of the specimen (100×).

determination of the ultimate tensile strength (UTS) of the specimen.

3. Results and discussion

Bond assessment was carried out to determine if the specimens had been joined successfully by the diffusion bonding process. The quality of the joint was assessed by metallographic studies, joint efficiency and percentage of bonded area. The equation used to calculate the joint efficiency is given as:

Joint efficiency =  $\frac{\text{UTS of bonded specimen}}{\text{UTS of reference specimen}} \times 100$  (1)

Reinforcement particles and matrix that straddle across the bond line characterize the bonded region. Reinforcement particles are irregular in shape whilst air void tends to be elongated and planar. On the other hand, the unbonded region is recognized by the elongated voids that are parallel to the bond line. This is shown in Fig. 2.

Fig. 3 shows that bonding had taken place across the entire bond line of the specimen. In addition, there is segregation of the reinforcement panicles on both sides of the bond line region, the areas of segregation being parallel to each other. The width of this parallel segregation micro-structure is defined as the bond-line region. Moreover, the segregated reinforcement particles have a preferred orientation in the direction parallel to the bond plane. Furthermore, the center of the bond line has a lower average particle density and size. Hence, there is segregation at the boundary of the bond-line region and reverse segregation at the center of the bond line.

Contrarily, segregation in the microstructure is not observed across the entire bond line for most of the specimens. Fig. 4 shows that the bond line cannot be distinctively

identified from the parent material at the bond-line region. This observation is desirable and, hence, diffusion bonding is thus suitable for the joining of metal–matrix composites. Consequently, this shows that in situ surface treatment and diffusion bonding have been conducted successfully on the specimen.

In addition, the specimen contains longitudinal cracks that straddle across the bond line as is shown in Fig. 5. These longitudinal cracks are due to the application of excessive compressive stress and are similar to those observed commonly in friction welding. These longitudinal cracks can only propagate across the bond line if there is an absence of voids at the bond interface. This is because the presence of voids can arrest the growth of the cracks as governed by the T-junction rule of fracture mechanics. Consequently, the presence of longitudinal cracks across the bond line can be used to confirm that diffusion bonding has occurred. Also,

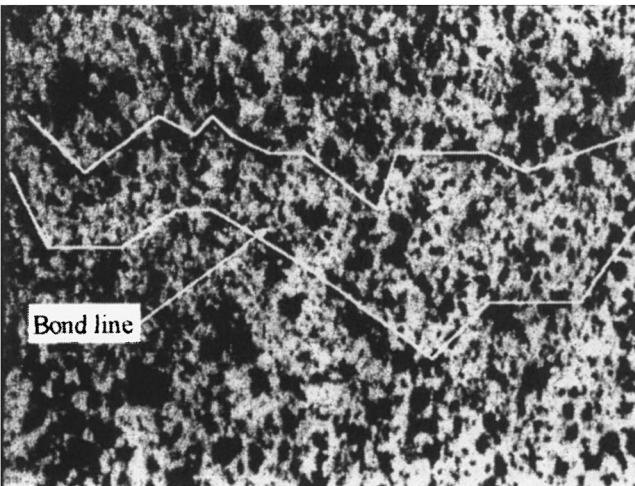


Fig. 3. Complete bonding at the bond line of specimen (100×).

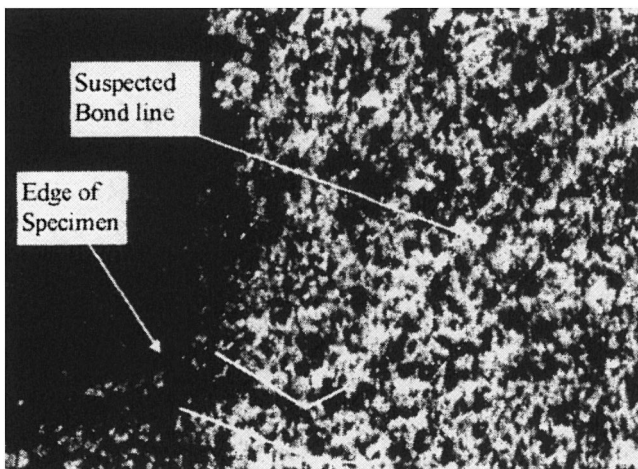


Fig. 4. Difficulty in identification of the exact location of the bond line from the edge of the specimen (100 $\times$ ).

the presence of the longitudinal crack deflection in the bond-line region can be due to bonding voids or hard particles across the crack propagation path. Crack deflection occurs at a dark area that has an irregular shape. Consequently, it is likely that the dark area is a relatively large particle. Also, there is a relatively high density of particles around the dark area, which could be the fragments of the particle as it interacts with the propagating crack.

Finally, Fig. 6 shows an SEM micrograph of the bond line of the specimen. It is observed that the bond line appears to be slightly darker than the matrix, but lighter than the reinforcement particle of the specimen. It should be noted that the etchant would preferentially remove the aluminium oxide (the reinforcement particle) as compared to the matrix material. Hence, it can be deduced that the bond line contains aluminium oxide particles in small amounts of fine particle size, and uniformly distributed, as shown by the uniform and shallow etch marks. The fine aluminium oxide

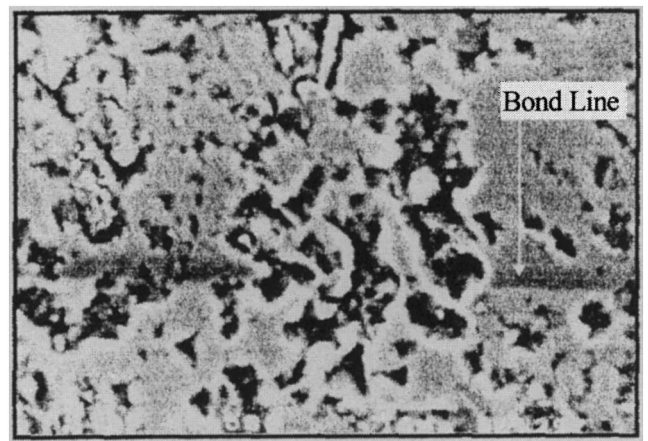


Fig. 6. SEM micrograph of the bond line of specimen (15 kV, 1500 $\times$ ).

particles can originate from break up or shearing of the original oxide layer or of reinforcement particles during the diffusion bonding process.

The fracture surfaces of the tensile specimens were studied to determine the percentage bonded area, and whether ductile fracture had occurred. The fracture surfaces of the tensile specimens were determined to be ductile as characterized by the observation of necking and dimples on the fracture surface. This observation can be used to confirm that the specimen had been diffusion-bonded.

#### 4. Mechanical testing

Finally, mechanical testing involved tensile testing and determination of joint eminency and a percentage bonded area. The UTS results obtained from tensile testing are given in Table 1 along with the estimated percentage bonded area. The reference UTS specimens had been soaked at the bonding temperature for either 14 or 16 min, so as to achieve a similar thermal history for both the reference and the test specimens. The reference specimen for 14 min dwell time achieved a UTS of 148.7 MPa and 16 min dwell time achieved UTS of 163.9 MPa.

It can be seen that both types of diffusion-bonding conditions for tensile specimens achieve a high average joint efficiency of 90.5% and a percentage bond area of 77.7%. These results are comparable to those obtained in other studies of open-air diffusion bonding of materials [2–6]. In addition, the diffusion-bonding process has not been optimized in this study and it is believed that the results will improve with process optimization. Hence, the vacuum-free diffusion bonding of Al-MMC can be conducted with the method used in this study.

Nevertheless, it should be mentioned that the tensile test was not conducted with standard size specimens and thus may be subject to some error. In addition, the UTS was calculated based on results of the maximum load reading from the tensile testing machine and the estimated bonded

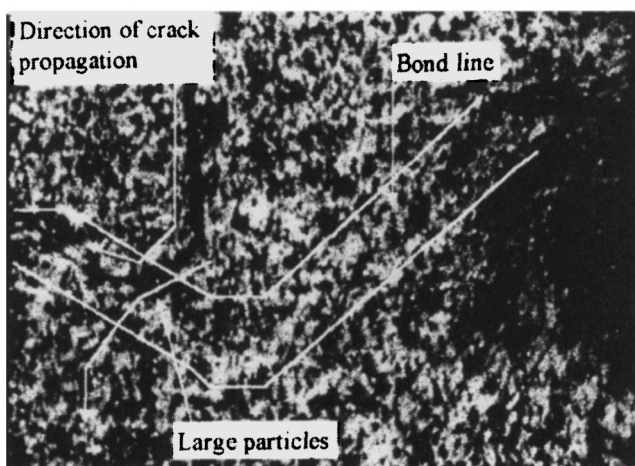


Fig. 5. The presence and deflection of longitudinal crack in the specimen (100 $\times$ ).

Table 1  
Results of ultimate tensile strength test (UTS) and percentage bonded area

Specimen	1	2	3	4	5	6
Total dwell time, (min)		14			16	
Load, kN	5.164	4.924	4.715	4.213	4.406	4.107
Cross section area, (x10 <sup>-5</sup> m <sup>2</sup> )	4.25	4.14	4.19	4.12	4.15	4.20
Estimated bonded area, (x10 <sup>-5</sup> m <sup>2</sup> )	3.49	3.21	3.30	3.02	3.24	3.19
UTS, (MPa)	147.9	153.6	142.7	139.6	136.0	128.6
Joint efficiency, (%)	90.2	93.7	87.1	93.8	91.5	86.5
Average joint efficiency, (%)		90.3			90.6	
Bonded area, (%)	82.1	77.5	78.8	73.3	78.1	76.0
Average bonded area, (%)		79.5			75.8	

area from the image analyzer. Consequently, the UTS value will be subject to errors in the determination of the bonded area. The determination of the bonded area of the fracture surface is based on the assumption that only the bonded region will be deformed during mechanical testing, and it is possible to identify the deformed area. Consequently, there are two possible sources of error in this method: the inability to differentiate the bond area from the unbonded area and the possible deformation of the unbonded area. Firstly, it has been suggested that the unbonded region that surrounds the bonded region may be deformed during the failure of the bonded regions. This can be due to the necking or yielding effect as a result of a tri-axial stress state that exists next to the unbonded region. Therefore, the measured bonded area will be larger than the actual area.

Secondly, the ability to differentiate between the bonded and unbonded region is subjective and depends on the skills of the investigator, the equipment and the methodology. At present, determination of the bonded area is based on an image analyzer that is attached to an optical microscope. This method has several limitations such as the lack of depth of focus, optical interference between the bonded and unbonded region, and the operator-dependent factor.

However, the results of percentage bonded area are inversely proportional to the UTS and this will act to reduce the effect of error in its estimation. This is because the underestimation of the percentage bonded area will cause overestimation of the UTS and vice versa. Hence, the net effect of error in estimation will be minimized if the bond assessment is based on the combined performance of both the UTS and the percentage bonded area.

5. Conclusions

The feasibility of the vacuum-free diffusion bonding of an aluminium matrix composite had been investigated and found to be viable, as an average joint efficiency of 90.5% and a percentage bonded area of 77.7% have been achieved. In addition, metallographic studies have shown that bonding has occurred across the bond line and ductile fracture is observed in tensile testing. Moreover, it was found that the in situ surface treatment process is able to provide a clean joining surface, despite the presence of a tenacious oxide layer.

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