Microstructure of diffusion-bonded joints in Al–Li 8090 alloy

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Joints were produced between Al–Li 8090 alloy sheet by solid state (SSDB) and transient liquid-phase (TLPDB) diffusion-bonding techniques. The bond interface was a planar, thermally stable, large-angle grain boundary in the SSDB joint. Non-planar grain boundaries in a band of coarse grains were present in the TLPDB joint. The origin of these microstructures and the measured shear strengths of the joints relative to that of the parent sheet are discussed.

1. Introduction

Diffusion bonding is an attractive joining technique for many advanced materials [1, 2], particularly when these are degraded by traditional fusion welding processes and when the need for environmental or thermal stability in thin-sheet structures precludes adhesive bonding. There is also the possibility of combining diffusion bonding with superplastic forming (SPF/DB) to manufacture complex sheet structures with reduced weight and fabrication costs compared with mechanically fastened structures.

Although SPF/DB techniques have been successfully applied to Ti-alloy structures [3] the application to Al-alloys has been limited by the difficulty in diffusion bonding these alloys [2, 4]. This is caused largely by the stable surface oxide film on Al-alloys. This film can prevent diffusion [4], contaminate the bond interface and lead to low bond strengths and unacceptable scatter in the strength data [5]. However, the oxide films on Li- and Mg-containing Alalloys are less stable [6], especially under diffusionbonding conditions [7] and some high-strength bonded joints have been made in Al-Li 8090 alloy by solid state [8, 9] and liquid-phase [9-12] bonding techniques. A detailed examination of the microstructures in solid state and liquid-phase diffusion bonded joints in 8090 alloy has been made using transmission electron microscopy (TEM) and energy dispersive Xray analysis (EDS). The results of these studies are reported in this paper.

2. Experimental procedure

Al–Li alloy sheet with a composition (wt %) Al–2.5 Li–0.6 Cu–0.8 Mg–0.12 Zr–0.1 Fe–0.05 Si was used in thicknesses of 2.5 and 4 mm. The surfaces to be bonded were mechanically polished to 1 μ m diamond or 1200 grit surface finish, then washed and ultrasonically cleaned in acetone. Surfaces to be liquid-phase diffusion bonded were first argon ion sputter cleaned and sputter coated with 1 μ m thick Cu layers before bonding with an 8 μ m thick Cu interlayer.

Joints were made in the form of overlap shear test pieces with a central step to control precisely the bond area and hence the bonding pressure. Bonding was carried out in a hot vacuum press at temperatures of $560 \,^{\circ}$ C for 4 h to give an overall through thickness deformation of 6%-12% and then slowly cooled, as described elsewhere [8]. Bonds were examined in the as-bonded and STA (20 min at $530 \,^{\circ}$ C, 5 h at 185 $^{\circ}$ C) conditions. TEM specimens were prepared by electropolishing and ion beam thinning.

3. Results

3.1. Solid state DB (SSDB) joints

Pancake-shaped grains elongated in the rolling direction are characteristic of unrecrystallized 8090 sheet [13]. In a polished and etched vertical section through a bonded joint in this sheet it was difficult to distinguish the planar bond interface A-A from the surrounding grain boundaries in the sheet, as shown in Fig. 1.

A transmission electron micrograph of the planar bond interface A–A is shown in Fig. 2; the only displacements of the interface occurred at grain-boundary triple points, e.g. at B. That this was the bond interface was confirmed by following this interface across the complete 2 mm width of the specimen by surface imaging using secondary electrons. The appearance of the bond interface at high magnification showed no evidence of porosity or continuous interfacial oxide (Fig. 3); occasional discrete particles were either complex oxides or identified as the icosahedral T2 phase (Al₆CuLi₃). The number and distribution of particles appeared to be similar in the bond interface and in the grain boundaries in the sheet.

In Fig. 2 the misorientations across the boundaries were measured as approximately 60° between grains 3 and 1, 58° between grains 3 and 2, and 6° for grains 2 and 1.

These results can be compared with grain misorientation data obtained for 8090 alloy using an electron back scattering device $\lceil 13 \rceil$. The peaks in

Figure 1 Optical micrograph of vertical section through a solid state diffusion bonded (SSDB) joint at A-A.



Figure 3 A high magnification transmission electron micrograph of SSDB interface at A–A.



Figure 2 Transmission electron micrograph of SSDB interface at A-A, showing displacement of bond interface at B.

frequency versus grain misorientation occurred around 7° and 58° corresponding to "low"- and "high"-angle boundaries, respectively. Thus the above results indicate that the bond interface is a conventional large-angle grain boundary similar to those present in the sheet [13].

The δ' (Al₃Li) precipitate distribution adjacent to the bond interface, shown in Fig. 4a and b indicates no Li depletion occurred during diffusion bonding; the larger particle present at B is T2 phase.

The thermal stability of the grain boundaries in unrecrystallized sheet is caused partly by the pinning effect of Al_3Zr particles in the boundaries and partly by the solute present, particularly Li. Surface Li loss or critical amounts of plastic strain (produced, for example, at cut edges of the sheet) can lead to grain



Figure 4 Transmission electron micrograph showing δ' (AlLi) phase near SSDB interface: (a) bright-field, (b) dark-field δ' diffracted beam.

coarsening at elevated temperatures [6]. The reduction in the number of Al_3Zr particles in the bond interface may be compensated by the increase in the number of oxide particles, and because the deformation during diffusion bonding is below the critical strain, this interface boundary is also very resistant to migration.

3.2. Transient liquid-phase DB (TLPDB) joints

The TLPDB process has been described elsewhere [14] and modelled in some detail for the Cu–Ag system [15]. In the present experiments interdiffusion of Cu from the interlayer and elements from the 8090 alloy led to phases with melting points below 550 °C, e.g. Al–Cu eutectic melts at 548 °C and Al–Li–Cu–Mg phases below this temperature. Complete dissolution of the Cu layer coincided with a maximum in the width of the liquid phase in the joint and was followed by progressive solidification and final homogenization of the composition across the bond region.

In the bond interface region, a $60 \ \mu m$ wide band of large (about $10 \ \mu m$ wide and $60 \ \mu m$ long) recrystallized grains formed from the liquid (Fig. 5). In the asbonded state there was no evidence of residual Cu layers, but some large grains contained residual coarse acicular $CuAl_2$ phase at B and grain-boundary $CuAl_2$ phase at C in Fig. 5a; after reheat-treatment no $CuAl_2$ phase was detected (Fig. 5b). The Cu distribution across a grain boundary at the centre of this region is shown in Fig. 6; the Cu concentration reached a maximum of 5.8 wt % at the grain boundary, falling to < 4.5 wt % about 2 µm from the boundary. This Cu distribution was similar across the whole coarse-grained band.

A feature of the coarse-grained region were clusters or rows of particles in some grain boundaries (Figs 5b, 7a-c); only a few isolated particles were found within the grains (Fig. 7c). Irrespective of the site, the individual particle size was in the range 60-100 nm. The particles were identified by EDS analysis (Fig. 8) as Al₃Zr with significant amounts of Cu and Ti. A series of microdiffraction patterns taken from a single particle which gave the EDS result above is shown in Fig. 9a; the patterns could be indexed as shown in Fig. 9b. They were consistent with the metastable L1₂ ordered cubic structure having a unit cell a = 0.4013 nm. This Al₃Zr phase is stable up to 600 °C and found in alloys containing Zr below the peritectic value of 0.18 wt % Zr [16]. The Al₃Zr particles can nucleate T_2 phase and were sometimes embedded in T_2 phase at the grain boundaries as shown in Fig. 10.



Figure 5 Optical micrograph of vertical section through a liquid-phase diffusion bonded (TLPDB) joint: (a) as-bonded, (b) after reheat-treatment to STA condition. Al_3Zr particles at A.



Figure 6 Copper distribution across grain boundary in coarse-grained region in TLPDB joint: (a) Transmission electron micrograph showing position of EDS analysis, (b) Cu distribution.







Figure 8 EDS analysis of Al₃Zr particle.

4. Discussion

Excellent quality joints were obtained by both solid state and liquid-phase bonding techniques, with no evidence of Li depletion or of continuous oxide films in the joints. The absence of oxide films is consistent



Figure 7 Transmission electron micrograph of TLPDB joint: (a) Al_3Zr particles in rows at A and in clusters at B in a grain boundary. (b) Al_3Zr particles at A in a cluster. (c) Rows of Al_3Zr particles in a grain boundary at A and within a grain at B. Inset shows isolated Al_3Zr particles within a grain.

with the results of Maddrell *et al.* [7] that indicated Li and Mg present in 8090 alloy reduced surface oxide films to spinels at the bond interface where they were present as isolated particles.

After liquid-phase bonding, a dramatic increase in grain size (to about ten times the initial sheet grain size) was apparent in a band extending to about 60 μ m (six times the initial width of the Cu interlayers) in the bond interface region. TLPDB using relatively thick (100 μ m) Zn interlayers produced via clad sheet [10, 11] led to a larger coarse-grained band 200 μ m wide with Zn-rich particles containing Cu and Mg present in the grain boundaries; these particles were very difficult to dissolve and were unchanged after 24 h at 540 °C. Solidification cracking was also found in these bonds. Using Zn interlayers produced by evaporation it was found that the bond strength decreased when the interlayer thickness exceeded 6 μ m [12].

In a study of the mechanism of liquid-phase bonding in 8090 alloy [17], the liquid was observed to penetrate the base metal along grain boundaries. Because the Al₃Zr phase was present as insoluble particles and was no longer effective in pinning these boundaries, rapid grain growth occurred. The present results suggest that during subsequent diffusion bonding and resolidification in the joint the Al₃Zr particles are swept ahead of the solid/liquid interface to become clusters or sheets of particles in the boundaries of the coarse grains. The lack of coarsening in the Al₃Zr particles is consistent with the reported [16]stability of the Al₃Zr phase below 600 °C. The uniformity of the Cu concentration in the coarse-grained region was caused by rapid diffusion in the liquid state. The bond thermal cycles appeared to be insufficient for complete homogenization of the Cu; a similar lack of homogenization was observed in LPDB joints made with Zn interlayers [10-12].

The different microstructures produced by these two bonding techniques is reflected in the fracture behaviour of the joints [8, 9]. The planar bond inter-



Figure 9(a) Micro-diffraction patterns for various zones in Al_3Zr particles. (b) Schematic drawing of indexed patterns.



Figure 10 Al₃Zr particle embedded in T_2 phase.

face characteristic of solid state bonds favours rapid crack growth in the bond plane at ambient temperatures analogous to failure in the short transverse direction in 8090 plate. The planar fracture path is absent in the liquid-phase bonded joint and a much rougher fracture surface is obtained for this joint. However, both bonding techniques have produced joints with a high resistance to peel under superplastic forming conditions [8, 9].

A bulk shear strength of about 210 MPa was obtained for 8090 alloy in the form of 4 mm thick sheet [8]. The planar interface and the coarse-grained band in the above solid and liquid-phase bonded joints, respectively, led to slightly lower shear strengths of 190 MPa [9]. Further work is therefore required on LPDB joints to define the minimum thickness of Cu or Zn interlayers compatible with a non-planar interface, minimum residual interlayer elements and preferred bonding parameters.

5. Conclusions

In solid state diffusion-bonded joints between 8090 alloy sheets the planar bond interface was a thermally stable large-angle grain boundary and the microstructure in the adjacent bond region was similar to that in the parent sheet. In transient liquid-phase diffusion-bonded joints a band of coarse grains was produced in the bond region with clusters of Al_3Zr phase in the grain boundaries. The planar bond interface and the coarse grain region were responsible for slightly lower bond shear strengths compared with the strength of the parent sheet.

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