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Diffusion bonding of two phase y-TiAl alloys with duplex microstructure

G. Çam, J. Müllauer, and M. Koçak

Solid state diffusion bonding of TiAl was carried out with different bonding parameters within the superplastic temperature range. The effect of post-bond heat treatment (PBHT) on the mechanical properties of the bonds was also studied. Defect free sound bonds were achieved within the temperature range 925–1150°C and the pressure range 20–40 MPa. Transverse microtensile specimens extracted from the bonds were tested to evaluate room temperature tensile properties of the bonds and to correlate them with bonding parameters. The bond strength increased with an increase in bonding temperature and pressure. All the specimens of the bond made at $925^{\circ}C$ and 40 MPa, and most of the specimens of bonds made at $1000^{\circ}C$ and 30 MPa and at $1100^{\circ}C$ and 20 MPa, failed in the bond area; all the specimens of bonds made at $1100^{\circ}C$ and 30 MPa and at 1150°C and 20 MPa failed in the base metal. Post-bond heat treatment at $1350^{\circ}C$ for 1 h led to the transformation of the recrystallised γ grains at the bond interface formed during bonding to a lamellar microstructure, resulting in an indiscernible bond line in all cases. This resulted in an improvement in the bond strength in most of cases. Moreover, the recrystallised y grains were also formed away from the bond area in the bonds made at 1100°C and 30 MPa and at 1150°C and 20 MPa. After PBHT, these bonds exhibited slightly lower tensile strength values owing to the change in the base metal microstructure away from the bond area.

The authors are at the GKSS Research Center, Institute of Materials Research, D-21502 Geesthacht, Germany. Manuscript received 10 February 1997.

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INTRODUCTION

Intermetallic TiAl is now being considered as a potential material for aerospace applications owing to its excellent combination of high temperature properties and low density. There has been intensive research activity on two phase intermetallic y-TiAl based alloys in recent years and remarkable improvements in high temperature creep properties and processing technology for these alloys during recent years have made them potential engineering alloys for high temperature structural applications in the aerospace industry.¹ These advanced alloys exhibit a desirable combination of high modulus retention, creep and oxidation resistance at elevated temperatures, and low density.²⁻⁴ However, the limited ductility and fracture toughness of the alloys at room temperature are significant drawbacks, which may limit their wider use in some applications. Therefore, the development of appropriate joining techniques, including solid state bonding and fusion welding, is indispensable for wider practical use of this material in other structural

applications. There is also growing interest in superplastic forming and diffusion bonding (SPF–DB) of these materials in production of some aerospace components.

Although there have been remarkable developments in the understanding of alloy development, processing, microstructural control, and microstructure-property relationships,⁵⁻⁷ there have only been a few reports aimed at establishing fabrication/processing techniques for these materials, such as joining,8-13 and their superplastic behaviour in the temperature range 925-1150°C.14-18 These reports are discussed in detail elsewhere.^{19,20} Joining these materials by fusion welding processes presents some complications, such as solid state cracking. However, solid state diffusion bonding with or without interlayers can be successfully used to join these materials. Solid state diffusion bonding in conjunction with superplastic forming is a potential technique for producing even thin walled complex structural components from γ based TiAl for aerospace applications. However, there is still a need to establish appropriate bonding conditions for these materials, particularly for the assessment of superplastic forming parameters for industrial production processes.

Based on the extensive experience on the development of γ -TiAl alloys and creep, fracture, and fatigue mechanisms^{7,21-23} at the GKSS Research Center, studies on DB and laser beam welding of these alloys have recently been started. The present paper reports the first set of results of this project on DB. The aim of this preliminary study is to establish an appropriate solid state DB procedure within the industrial superplastic forming temperature range of conventional titanium alloys, in order to successfully apply SPF–DB processes to an alloy termed γ -TAB. The DB parameters which have been assessed in this work will be transferred to an industrial project which involves SPF–DB processes for manufacturing structural parts from thin γ -TAB sheets.



1 Schematic diagram of geometry of microtensile specimens tested by loading at shoulders of specimens



a as received; *b* after PBHT at 1350°C for 1 h

2 Polarised light micrographs of base metal

EXPERIMENTAL

A series of diffusion bonds with different bonding parameters was made using Ti-47Al-4.5(Cr,Mn,Nb,Si,B) (at.-%), referred to as γ -TAB,¹ specimens of approximate size 11 × 15 × 21 mm. The material was vacuum arc melted, investment cast, and hipped at 1185°C and 140 MPa for 4 h. Bonding temperatures within the industrial superplastic forming temperature range of γ alloys, i.e. 925–1150°C, and bonding pressures in the range 20–40 MPa were used in this study, bonding time being 1 h in all cases. Surfaces were machined and ground with 1200 grade SiC paper followed by rinsing with alcohol before bonding. The pressure was lower than 4.5 × 10⁻⁵ torr (6.0 × 10⁻⁵ mbar) for all the bonds made. The bonds made with these bonding parameters were also given a post-bond heat treatment (PBHT) at 1350°C for 1 h, the cooling rate being 900 K h⁻¹.

Optical metallography and scanning electron microscopy were used to investigate the microstructural development during diffusion bonding and to assess the quality of the



a 925°C-40 MPa; b 1000°C-30 MPa; c 1100°C-30 MPa

3 Polarised light micrographs of specimens bonded under given conditions

bonds made under various bonding conditions. Extensive microhardness measurements were also made to determine the hardness values of different phases encountered and the effect of PBHT on hardness. Microtensile specimens $21 \times 2 \times 0.5$ mm in size with a 9 mm gauge length were machined using spark erosion cutting. Tensile tests were carried out by loading the specimens at the shoulders, as shown in Fig. 1, at room temperature.

RESULTS AND DISCUSSION Microstructural aspects

The as received base material has a duplex microstructure comprising ~90 vol.-% lamellar $\gamma + \alpha_2$ colonies and ~10 vol.-% equiaxed γ grains. The microstructures of the investment cast base material in the as received condition and after a PBHT at 1350°C for 1 h are shown in Fig. 2.



a before bonding; b during bonding; c after bonding

4 Schematic representation of microstructural development at original interface during DB of γ -TAB alloy

Grain growth after the heat treatment at 1350°C is clearly visible.

Under all the bonding conditions studied, the formation of equiaxed γ grains at the original interface during bonding owing to dynamic recrystallisation induced by asperity deformation on mating surfaces under the applied bonding pressure was observed as reported earlier.24-27 In the bonds made at relatively low temperature and pressure combinations (925°C-40 MPa, 1000°C-30 MPa, and 1100°C-20 MPa), the original interfaces are visible after the nucleation of equiaxed γ grains as a result of their insufficient growth into both mating parts (Fig. 3a and b). In contrast, the original interfaces are no longer discernible owing to nucleation and sufficient growth of recrystallised γ grains into the mating parts in the bonds made at higher temperature-pressure combinations (1100°C-30 MPa and 1150°C-20 MPa) (Fig. 3c). This dynamic recrystallisation is expected to improve bond strength by removing planar boundaries at the original interface. The microstructural development sequence at the original interface is shown schematically in Fig. 4. The hardness value of the recrysallised γ grains formed at the bond interface lies between the hardness of lamellar grains and γ grains present in the original y-TAB base material.^{24,26} Cross-sectional microstructures of bonds produced using different bonding conditions are shown in Fig. 3. As may be seen from Fig. 3, the



a bonded at 925°C-40 MPa; *b* bonded at 1000°C-30 MPa; *c* bonded at 1100°C-30 MPa

5 Polarised light micrographs of specimens after PBHT at 1350°C for 1 h

size of the recrystallised γ grains increases with increasing bonding temperature. Consequently, the strength of the bonds made at higher temperatures is expected to be higher.

Moreover, the bonding at 1100°C and 30 MPa resulted in some locally recrystallised γ grains in the base material away from the bond region (Fig. 3c). The number of locally recrystallised γ grains increased with increasing temperature in the bond made at 1150°C and 20 MPa. Dynamic recrystallisation in γ alloys deformed at elevated temperatures, i.e. above 850°C, has also been observed by other researchers.^{28–30}

A PBHT at 1350°C for 1 h was also carried out to improve bond quality. The recrystallised γ grains formed at the original interface during diffusion bonding transformed to lamellar colonies on PBHT, leading to an indiscernible bond interface in all the specimens regardless of the chosen bonding parameters (Fig. 5). Furthermore, the recrystallised



 $a \gamma$ grains formed in base metal away from bond region by dynamic recrystallisation; b transformation of recrystallised γ grains to near lamellar microstructure on PBHT at 1350°C for 1 h

6 Microstructures of bond made at 1150°C-20 MPa

 γ grains formed in the base material away from the bond interface in the bonds made at 1100°C–30 MPa and 1150°C–20 MPa also transformed to a duplex structure containing lamellae and γ grains on PBHT (Fig. 6).

Mechanical properties

The as received base material properties were determined by testing of the microtensile specimens which were extracted using spark erosion cutting. The as received base material was given the same thermal cycle as the bonding at 1100°C. Furthermore, the base material was also heat treated at 1350°C for 1 h to simulate the PBHT. Neither heat treatment showed any significant effect on the tensile strength of the base material (Fig. 7). A large scatter in tensile strength was observed under all the conditions which could be reduced by homogenising the as received microstructure. However, a homogenisation heat treatment was not given to the as received cast material in the present study since the main aim was to establish appropriate bonding conditions for this material by qualitatively comparing the bonds made using various bonding parameters.

Specimens extracted from the bond made at 925° C displayed poor tensile strength, as expected (Fig. 8). All the specimens failed along the bond interface. As can be seen from Fig. 3*a*, the original bond interface is clearly visible after diffusion bonding. The presence of this planar bound-



AR as received; HT 1 same treatment as bonding at 1100°C; HT 2 heat treated at 1350°C for 1 h

7 Range of tensile strength values of base metal in as received and heat treated conditions

ary presents a preferable crack path in the transverse tensile test.

As can be seen from Fig. 8, the specimens extracted from the bond made at 1000°C displayed better tensile strength values than those bonded at 925°C. However, most of the specimens failed again along the bond interface. The specimens extracted from the bond made at 1100°C with a pressure of 20 MPa displayed higher tensile strength (Fig. 8), but half of the specimens still failed along the bond interface. Figure 9 shows the failure location in a specimen in which failure took place along the bond interface. Investigations on the fracture surfaces of the specimens failing at the bond interface showed that fracture surface topography becomes rougher as the bond temperature increases, as discussed in Refs. 25–27. It was also observed







fracture location Polarised light micrograph of specimen bonded at 1100°C-20 MPa failing along bond region

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that brittle intercrystalline fracture took place along the bond interface in the specimens failing at the bond interface (Fig. 10*a*). The fracture surface of a specimen bonded at 1100°C and failed in the base material, a typical fracture of lamellar and globular γ grains, is shown in Fig. 10*b*. The fine, light grains seen in Fig. 10*a* were analysed by energy dispersive X-ray analysis and it was found that they were titanium rich γ grains containing chromium, niobium, and manganese, as reported in Ref. 27.

The specimens extracted from the bond made at 1100° C with the higher pressure of 30 MPa showed again higher tensile strength and all the specimens but one failed in the base material; in particular, the scatter of the tensile fracture strength data became more narrow. This was expected as the bond interface after bonding at 1100° C with a pressure of 30 MPa was almost indistinguishable (Fig. 3c). All the specimens extracted from the bond made at 1150° C-20 MPa also exhibited high tensile strength (Fig. 8) and failed away from the bond interface; bonds made under these conditions displayed the smallest scatter of the tensile fracture strength data. This might be a result of partial homogenisation of the as cast microstructure at higher temperatures (Fig. 6).

The lower tensile strengths of the bonds compared with the intrinsic tensile strength of the investment cast and hipped γ -TAB alloy determined by testing standard tensile specimens,⁷ as seen in Fig. 8, might be a result of the nonpolished surfaces of the microtensile specimens (surface microcracks can be present after spark erosion cutting of microtensile specimens). There is a distinct tendency for increasing cross tensile strength with increasing bonding temperature, as shown in Figs. 8 and 11. This may be caused by the increase in grain size of the recrystallised γ grains. As γ grains nucleated at the original bond interface grow into both mating parts, they destroy the originally flat interface which then is no longer discernible.

Post-bond heat treatment resulted in a slight decrease in the hardness of all the phases encountered. Moreover, the PBHT did not significantly change the tensile properties of the base material (Fig. 7). Therefore, it can be concluded that it improved the bond quality in most of the conditions studied, resulting in failure of all specimens in the base material rather than along the bond interface. The visible bond interfaces disappeared after the PBHT (Fig. 5), which in turn eliminated the presence of a favourable crack path along the originally flat bond interface. All the specimens extracted from the bond made at 925°C failed in the base



a bond region; *b* base metalMicrographs of fracture surfaces

material after PBHT whereas they all failed along the bond interface without being given PBHT. Similarly, all other specimens extracted from the bonds made using other bonding parameters failed in the base material after PBHT. Figure 12 shows the failure location away from the bond region in a specimen which was heat treated after bonding at 1000°C with a pressure of 30 MPa. However, the bond strength of the specimens made at 1100°C–30 MPa and 1150°C–20 MPa was decreased slightly after the PBHT (Fig. 13) owing to the microstructural change (increase in the volume of γ grains in the microstructure) in the base material structure away from the bond region.



11 Effect of bonding conditions on tensile strength



a fracture location and intact bond interface; b higher magnification showing intact bond interface indicated by arrow in Fig. 12a

12 Polarised light micrographs of failed specimen bonded at 1000°C-30 MPa and post-bond heat treated: failure occurred in base material away from bond interface

From an economic point of view, it is desirable to obtain bonds with acceptable performance at as low a temperature and pressure level as possible without any further PBHT. However, for this specific cast γ -TAB alloy the bonds produced with the bonding conditions 925°C-40 MPa, 1000°C-30 MPa, and 1100°C-20 MPa need a PBHT whereas the bonds produced with the bonding conditions 1100°C-30 MPa and 1150°C-20 MPa exhibit better bond performance and PBHT reduces the strength levels. It is therefore necessary to bond this material either using relatively low temperature and high pressure combinations (925°C-40 MPa, 1000°C-30 MPa, and 1100°C-20 MPa) and applying a PBHT, or using relatively high temperature and low pressure combinations (1100°C-30 MPa and 1150°C-20 MPa) without any further PBHT.

Work is in progress to determine the mechanical properties of the bonds made using homogenised as cast material. Fracture toughness properties of the bond region and the effect of microstructural/mechanical heterogeneity (mismatch) of the bond area on micromechanism of the fracture/deformation process will also be investigated, as will the use of thin interlayer material on deformation and fracture process at the DB interface.

CONCLUSIONS

1. Defect free sound bonds were achieved by solid state DB within the temperature range 925–1150°C and a



13 Effect of PBHT on tensile strength of bonds made at relatively higher temperatures: note slight decrease in tensile strength after PBHT

bonding pressure range 20-40 MPa, the bonding time being 1 h.

2. Equiaxed γ grains were formed at the bond interface during bonding owing to dynamic recrystallisation induced by asperity deformation on mating surfaces under applied bonding pressure. The size of the newly formed γ grains increases with increasing bonding temperature. Recrystallised γ grains were also formed in the base metal away from the bond interface with bonding conditions of $1100^{\circ}C-30$ MPa and $1150^{\circ}C-20$ MPa.

3. The original bond interface almost disappeared following nucleation and growth of the recrystallised γ grains in the bonds made with the bonding conditions 1100°C– 30 MPa and 1150°C–20 MPa.

4. Best tensile properties were displayed by the joints made at $1100^{\circ}C-30$ MPa and $1150^{\circ}C-20$ MPa, among the bonding conditions studied without PBHT. There is a trend for increasing tensile strength with increasing bonding temperature. The scatter in fracture strength data was significantly reduced at higher bonding temperature and pressure combinations.

5. Post-bond heat treatment at 1350° C for 1 h resulted in the transformation of the recrystallised γ grains to lamellar colonies at the bond interface. This renders the originally flat bond interface indiscernible for all the bonding conditions studied.

6. As a result of the disappearance of the bond interface after PBHT, the bond quality was found to improve for the bonds made at relatively low temperature and pressure levels. However, the tensile strength of the bonds made at $1100^{\circ}C-30$ MPa and $1150^{\circ}C-20$ MPa decreased slightly owing to the microstructural changes in the base material away from the bond area.

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