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Microstructural and mechanical characterization of diffusion bonded hybrid joints

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Abstract Ti-alloys, particularly TiAl, are becoming attractive for the use in the production of high-temperature components such as turbine blades and exhaust valves, owing to their low density. However, these components may not be cost-effectively cast totally from TiAl alloys and casting defects may occur in investment casting of these complex parts. Other manufacturing technologies, such as machining, cannot be economically employed in these very hard and brittle materials. Production of bi-material or even multi-material TiAl components can therefore offer an alternative fabrication route provided that the joining and joint properties of these materials are well understood. In this study, the diffusion bondability and joint characteristics of TiAl and Ti–6Al–4V alloys were studied. These two different materials were joined by using various bonding parameters. Metallographic investigations were conducted for characterization of the interface region of these dissimilar joints. Furthermore, the mechanical behavior of the bond interface was evaluated by shear testing. Both results on the microstructural and mechanical

characterization provided the optimum bonding conditions for the production of TiAl–Ti6Al4V hybrid joints.

Introduction

Modern manufacturing technologies require the joining of dissimilar materials for fabrication of multi-material components. Exhaust valves, for example, can be produced by friction welding of the cap section to the stem section, both of which are made of different materials. The production of exhaust valves from light-weight materials has recently been attracting a great interest as it offers a number of advantages, such as reduction in fuel consumption, less vibration, longer service life and less maintenance. Light-weight intermetallic TiAl alloys ($3.7\text{--}3.9\text{ g/cm}^3$) are potential candidates in such applications. These alloys possess unique mechanical properties, such as low density leading to high specific stiffness, excellent creep and oxidation resistance, strength retention at elevated temperatures and burn resistance [1–3]. However, the very high brittleness of these alloys at ambient temperature makes the forming of thick parts possible and economical only by investment casting, although some wrought TiAl alloys in the form of sheet are also commercially available. The investment casting of complex parts from these alloys is either not possible since incomplete castings are very common or not economical. Furthermore, design and service requirements of the component may pose further limitations on the use of single material for the whole component. For example, areas with sharp notch type (high stress concentration) geometries may require materials with higher fracture toughness than intermetallic TiAl alloy at these locations. In the case of exhaust valves, this

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problem can, for example, be overcome by producing the cap section from intermetallic TiAl alloy via investment casting and the stem section from more ductile commercial Ti-alloys via machining and finally joining these two parts. However, convenient and economical joining processes must be determined in order to produce multi-material (hybrid) components by welding. Moreover, the joint performance must be acceptable for such applications.

Fusion welding processes cannot be economically applied in joining of intermetallic TiAl alloys due to their high susceptibility for cracking owing to their extreme room temperature brittleness [4–6]. On the other hand, low heat input solid state joining processes, such as friction welding, diffusion bonding and brazing, can be used in joining of multi-material systems, one of which is very susceptible to cracking. It has been reported that TiAl and Ti–6Al–4V alloys are successfully joined by conventional friction welding [5–9] and further investigations are on-going. Diffusion bonding, which is commercially applied in joining commercial Ti-alloys in aerospace industry with success, has also been demonstrated to successfully join TiAl alloy to itself [10–13]. This joining process obviously can also be used in joining multi-material systems. The successful joining of TiAl alloys to other materials opens up the possibility of producing dual property advanced hybrid components, such as exhaust valves, compressor and low-pressure turbine blisks. Recently, numerous research programs have been initiated on the joinability of Ti-alloys for hybrid components to increase the use of these alloys in various innovative engineering applications [14–16]. Similarly, numerous work on the joinability of TiAl alloys to various steel grades are also being currently conducted [17, 18]. It should be noted that the mechanical properties of the interfaces of hybrid joints can strongly be influenced by the bulk properties of each joint constituent. Presence of high strength mis-match between two materials may provide shielding of brittle phases (if exists at the interface) against applied deformation. Hence, the design of hybrid components together with the knowledge of the resulting interfacial properties is of great importance.

In this study, a series of hybrid joints between Ti–6Al–4V alloy and a new generation C-containing TiAl-alloy were produced by diffusion bonding using different bonding

parameters. In these bonding trials, the bonding parameters (i.e. temperature, pressure, and time) were systematically varied in order to determine, particularly, the effect of bonding time on the joint performance for a given set of temperature and pressure. Optical and scanning electron microscopies were employed to investigate the microstructural evolution and the presence of any weld defects along the bond interface. Moreover, the joint performances of the bonds produced were evaluated by conducting microhardness measurements, detailed energy dispersive spectrometry (EDS) analyses and shear testing. Thus, the optimum bonding parameters to produce sound hybrid joints between these two alloys were determined.

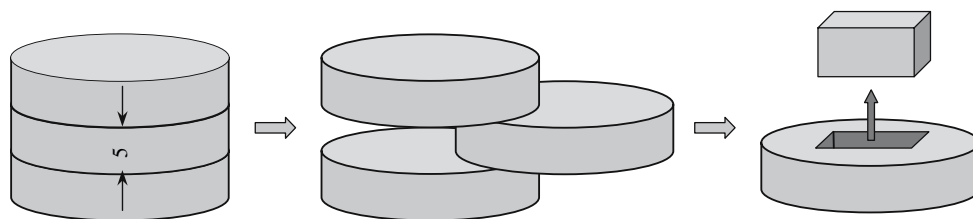
Experimental procedure

The materials used in this study are namely a new generation C-containing duplex TiAl alloy (Ti-48 at.% Al-3.7 at.% (Nb, Cr, C)) and Ti–6Al–4V alloy (designated as Ti64 hereafter).

TiAl alloy was received in the form of a cylindrical block of 90 mm in length and 70 mm in diameter, produced by vacuum arc re-melting and HIPped at $\sim 1,185$ °C (2,165 F) for 4 h using a pressure of ~ 172 MPa (25 ksi) to avoid any internal porosity. Slices of 5 mm in thickness were cut from this block by electro-discharge machining (EDM) technique, Fig. 1. These slices were then subjected to a two-stage heat treatment for the homogenization of the microstructure and carbide precipitation. The first stage of this heat treatment, i.e. homogenization, involved heating to 1,250 °C (1,523 K) with a heating rate of 20 °C/min, and holding at this temperature for 4 h, followed by oil quenching. The second stage, i.e. carbide precipitation, involved heating to 752 °C (1,025 K) with a heating rate of 20 °C/min, and holding at this temperature for 24 h, followed by a controlled cooling slow (1,527 °C/h = 1,800 K/h). After this two-stage heat treatment, diffusion bonding specimens of 30 mm \times 7 mm \times 5 mm were extracted from these slices by EDM technique, Fig. 1.

Ti64 alloy was received in the form of a cylindrical bar with a diameter of 40 mm. Slices of 5 mm in thickness were cut from this bar and then diffusion bonding specimens of 30 mm \times 7 mm \times 5 mm were extracted from these slices by EDM technique.

Fig. 1 Schematic showing the preparation of diffusion bond specimens



The surfaces of diffusion bonding specimens extracted by EDM technique were machined parallel. The mating surfaces of TiAl-alloy specimens were only ground with SiC paper down to grit 1,200 and ultrasonically cleaned using acetone. On the other hand, the mating surfaces of Ti64-alloy specimens were ground with SiC paper down to grit 4000 and then polished down to 1 μm using Al₂O₃ solution, followed by ultrasonic washing using acetone. After this surface preparation procedure, one TiAl-alloy specimen and one Ti64-alloy specimen were placed on top of each other and diffusion bonded, as schematically shown in Fig. 2, in order to obtain 5 shear test specimens produced with the same parameters. Diffusion bonding trials were carried out under a minimum vacuum level of 3×10^{-2} Pa (3×10^{-4} mbar) using different bonding parameters, Table 1.

In order to carry out microstructural investigations and to evaluate joint performance, metallography specimens (one specimen for each joint) and shear test specimens (minimum four specimens for each joint) were extracted from the joints produced by EDM technique, Fig. 2. Metallography specimens were mounted in bakelite, ground with SiC paper down to grit 4000 and then polished down to 1 μm using Al₂O₃ solution. No etching was used and polarized light was employed for optical microscopy. Detailed microstructural investigations along the bond lines of the joints produced were conducted by optical and scanning electron microscopy to determine the presence of any interfacial defect. Detailed line and point EDS analyses

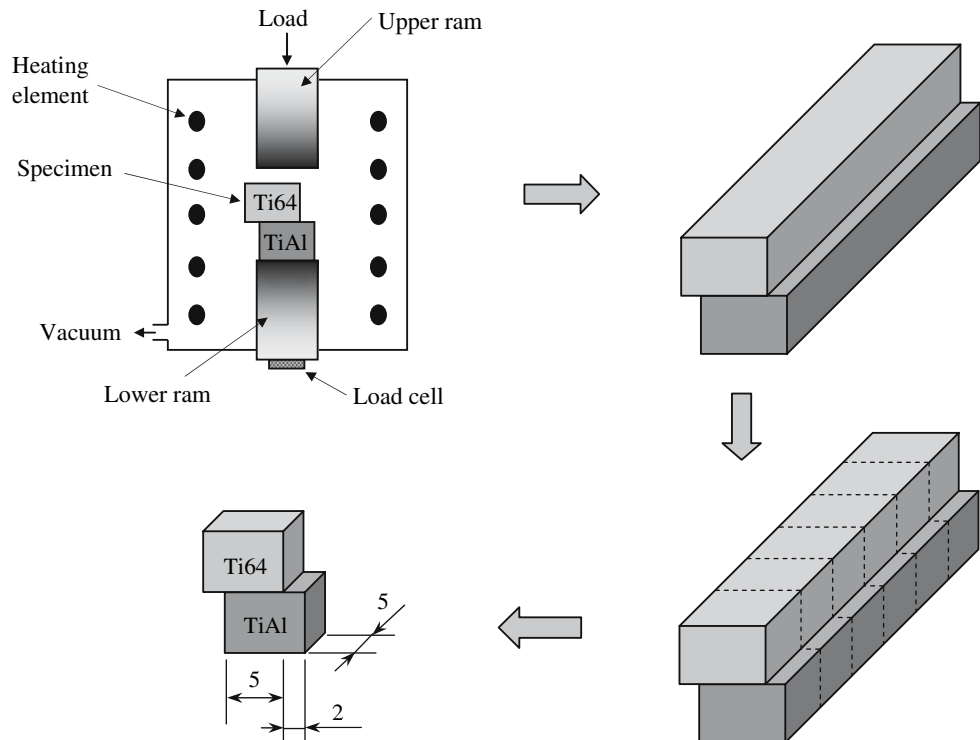
along and across the bond interfaces were also conducted to determine the chemical composition profiles, the diffusion mechanism, and the formation of new phases. Furthermore, hardness profiles were determined by carrying out microhardness measurements across the bond interfaces.

Four specimens extracted from each joint were subjected to shear test at room temperature, as schematically shown in Fig. 3, to determine shear strength of the joints. Fractography was also conducted on the fracture surfaces of the shear specimens by scanning electron microscopy to determine the fracture features.

Table 1 Diffusion bonding parameters used in this work

Material combination	Bonding parameters		
	Temperature (°C)	Pressure (MPa)	Time (min)
TiAl–Ti64	825	5	15
TiAl–Ti64	825	5	30
TiAl–Ti64	825	5	45
TiAl–Ti64	850	5	15
TiAl–Ti64	850	5	30
TiAl–Ti64	850	5	45
TiAl–Ti64	850	8	15
TiAl–Ti64	875	5	15

Fig. 2 Schematic illustrating diffusion bonding of TiAl and Ti64 alloys and extraction of the shear specimens from the bonds produced



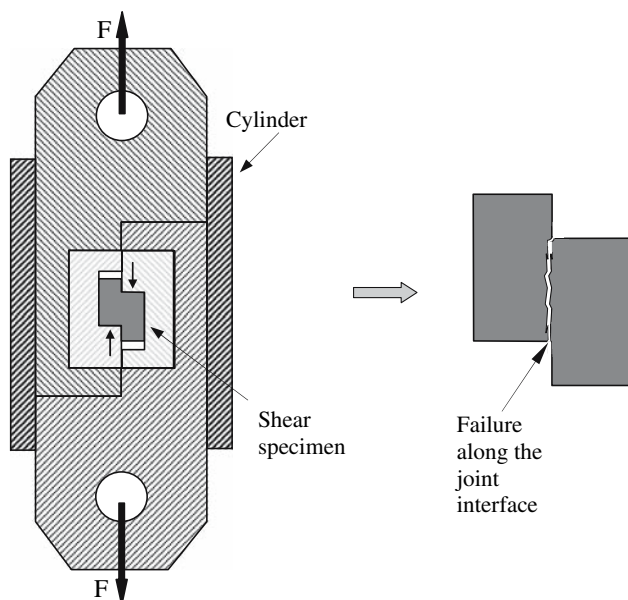


Fig. 3 Schematic illustration of the shear testing

Results and discussion

Microstructural aspects

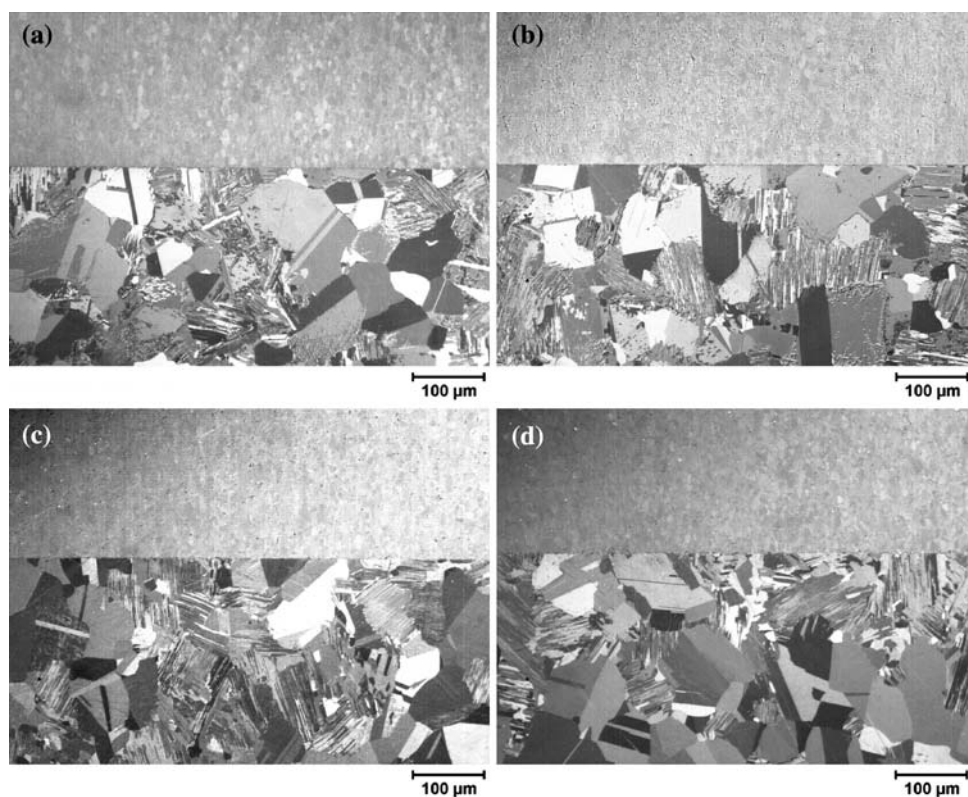
TiAl alloy used in this study has a fine duplex microstructure consisting of gamma (γ) grains and lamellar grains (γ and α_2

layers). Average grain size is relatively fine (about 50–100 μm). The other material used, i.e. Ti64 alloy, has a fine duplex microstructure consisting of α and β phases.

Table 1 gives the bonding parameters employed in this study. With the practical view in mind, the bonding temperature was intentionally kept as low as possible, i.e. between 825 and 875 $^{\circ}\text{C}$. No apparent weld defects (i.e. porosity or cracks) were detected by optical microscopy indicating that sound hybrid bonds have been achieved. The bond line is clearly visible due to the different microstructures of the two materials joined, Fig. 4. Optical microscopy also revealed that the increase in bonding time for a given set of temperature and pressure resulted in excessive creeping of Ti64 alloy, which has a lower yield point than that of TiAl alloy, Fig. 5.

The electron microscopy conducted on the metallography specimens extracted from the bonds produced, on the other hand, showed that there are some isolated unbonded areas (porosity) along the bond interface in the bond obtained with the parameters 825 $^{\circ}\text{C}/5\text{ MPa}/15\text{ min}$, Fig. 6a. All other bonds, except the one produced with the parameters 825 $^{\circ}\text{C}/5\text{ MPa}/15\text{ min}$, also exhibited some porosity-like dark regions along the bond interface, Fig. 6. The EDS analyses conducted in these regions indicated that they are not porosity, and they were brittle α_2 (Ti_3Al) phases originally present in these areas, and fallen out during mechanical surface polishing.

Fig. 4 Polarized light micrographs showing the joint area of some of bonds produced: (a) 825 $^{\circ}\text{C}/5\text{ MPa}/15\text{ min}$, (b) 825 $^{\circ}\text{C}/5\text{ MPa}/30\text{ min}$, (c) 850 $^{\circ}\text{C}/5\text{ MPa}/15\text{ min}$, and (d) 850 $^{\circ}\text{C}/5\text{ MPa}/45\text{ min}$



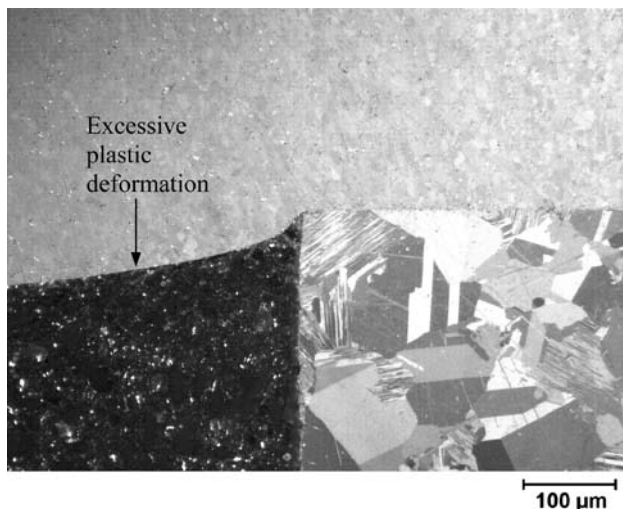


Fig. 5 Micrograph showing the excessive plastic deformation in the lower yield point Ti64 alloy side due to creep in the bond produced with the parameters of 850 °C/5 MPa/45 min

Moreover, a reaction zone (a newly formed phase seen as gray reaction layer at the interface) was detected in the bond interfaces of all the joints produced as identified by electron microscopy, Fig. 6. The detailed EDS point analyses conducted in these zones indicated that this newly formed phase is probably α_2 phase (Ti_3Al) having a chemical composition of 72–77 at.% Ti and 16–20 at.% Al, Table 2. It was also observed that the width of this

reaction zone increases with the increase in the bonding time for a given set of temperature and pressure, Fig. 6.

The chemical composition profiles for 5 elements, namely Ti, Al, Nb, Cr, and V, were also obtained using EDS analysis across the bonds produced to determine diffusion mechanism. As seen from Fig. 7, there is a diffusion flux of Al atoms from TiAl alloy side toward Ti64 alloy side and of Ti atoms in the opposite direction. Similar observations in hot isostatic diffusion bonding of IHI 01A TiAl alloy to Ti64 alloy were also reported by Holmquist et al. [14, 15]. Furthermore, it was also observed that some Nb-enrichment from TiAl alloy and V-enrichment from Ti64 alloy in the reaction zone take place throughout the joints, Fig. 7. No evidence of Cr diffusion was detected in the joints produced.

Joint performance

Hardness profiles were determined by conducting detailed microhardness measurements across the bonds produced. Figure 8 illustrates the hardness profile of the bond produced with the parameters of 850 °C/5 MPa/45 min as an example, which represents all the joints produced. As seen from the figure, it was not possible to determine the hardness of the newly formed α_2 phase along the joint interface. This can be attributed to the fact that this newly formed phase is not wide enough, i.e. 3 μm (Fig. 7), for microhardness determination.

Fig. 6 Back-scattered micrographs (SEM) showing the bond regions of some of the joints produced: (a) 825 °C/5 MPa/15 min, (b) 825 °C/5 MPa/45 min, (c) 850 °C/5 MPa/15 min, and (d) 850 °C/5 MPa/30 min. (Note the presence of some isolated unbonded areas in the bond interface of the bond shown in a, the dark regions seen in the micrographs of other bonds are not porosity)

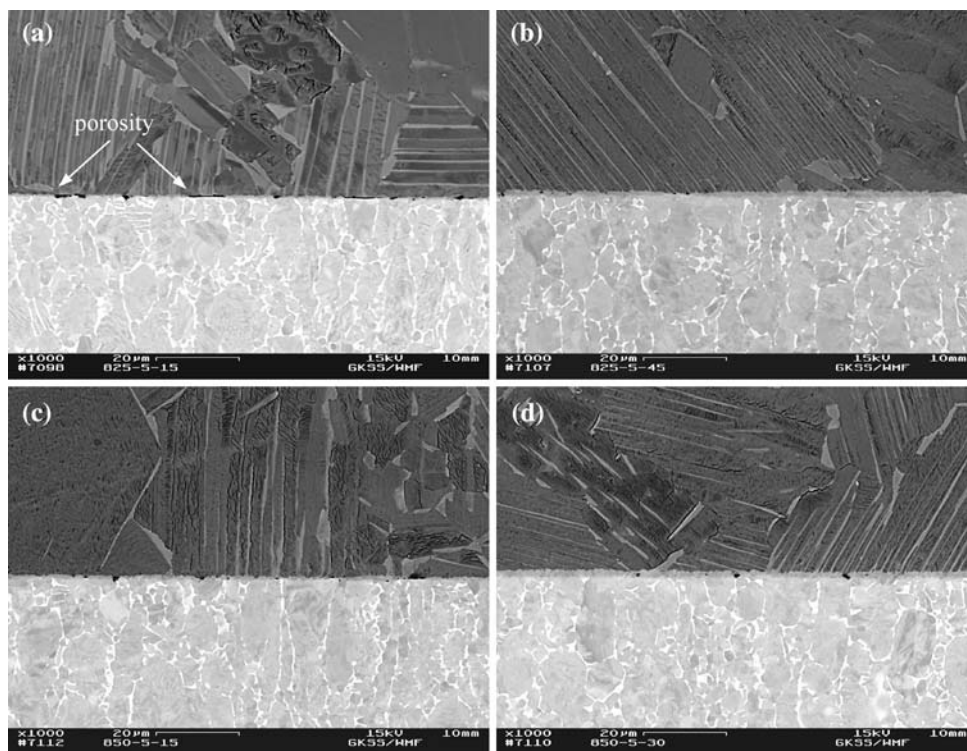


Table 2 The chemical analyses (EDS) results conducted in the bond area of the joint produced with the parameters of 825 °C/5 MPa/45 min

Position ^a	Al (at.%)	Ti (at.%)	Cr (at.%)	Nb (at.%)	V (at.%)
1	4.3	75.9	1.7	0.3	17.7
2	7.0	87.7	0.2	0.2	5.3
3	30.2	61.4	2.6	3.7	2.0
4	34.8	56.7	1.7	4.7	2.2
5	16.5	77.1	0.2	1.2	5.2
6	17.0	75.4	0.2	1.9	5.7
7	16.8	75.5	0.2	1.7	6.0
8	18.7	72.8	0.4	2.9	5.2
9	20.1	71.8	0.2	2.7	5.4

^a See Fig. 7 for the positions where EDS point analyses conducted

As seen from Table 3, the nominal shear strengths of the base materials, namely TiAl and Ti64, were found to be 1,398 and 1,399 MPa, respectively, obtained dividing the recorded forces by cross-sectional areas of the specimens tested, which do not represent the inherent shear strength values. Shear strength of a material is normally 0.6 times tensile (or compression) strength of that material. Thus, nominal shear strength values obtained are higher than expected due to the fact that areas of the fracture surfaces were much larger than the cross-sectional areas of the specimens.

The joint performances of the bonds produced were determined by shear testing. The results of these tests were given in Table 3 and Fig. 9. The highest average shear strength value, i.e. 483 MPa, was exhibited by the bond produced with the parameters of 850 °C/5 MPa/15 min. Furthermore, four specimens extracted from this bond showed very similar values, indicating that the bond quality is homogeneous throughout the joint, Table 2 and Fig. 9. As expected, the shear strength values of all the bonds obtained are significantly lower than those of both base materials, i.e. TiAl and Ti64 alloys, Table 2, although sound bonds were achieved in most of the cases. The reason for this is the presence of the brittle α_2 phase along the joint interface, Fig. 6, promoting brittle failure along the interface between TiAl alloy (higher strength) and the reaction zone.

The shear test results of the specimens extracted from the bonds produced at temperatures of 825 and 875 °C using a pressure of 5 MPa for 15 min exhibited lower shear strength values and large deviations, indicating inhomogeneous bond quality. This can be attributed to the presence of porosity along the joint interface in the case of the bond obtained at 825 °C and possibly to the thickening of the brittle α_2 phase forming along the joint interface in the case of the bond produced at 875 °C, tendering the

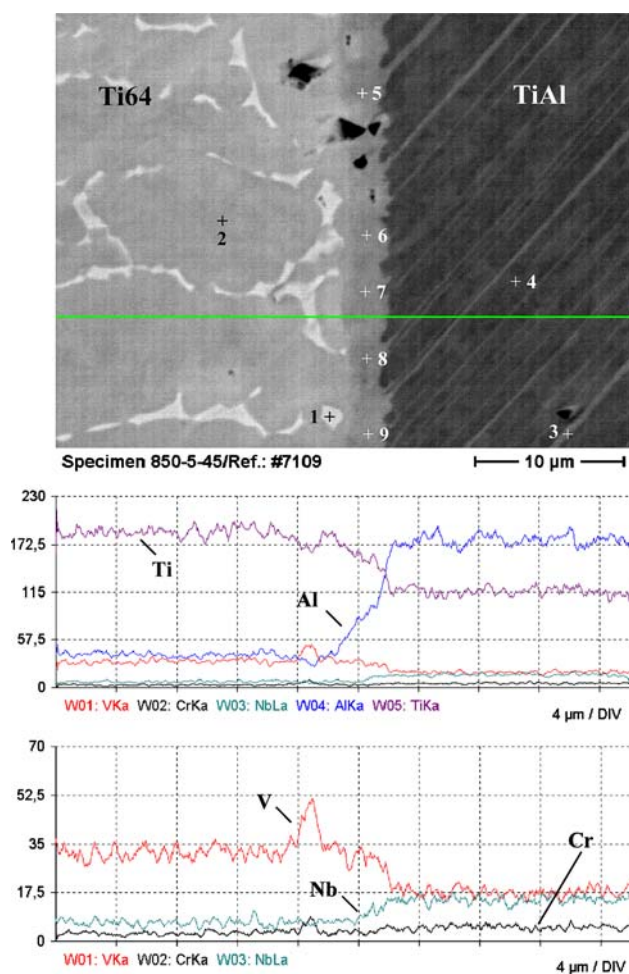


Fig. 7 The chemical composition profiles for Ti, Al, Nb, Cr, and V obtained across the bond interface produced with the parameters of 825 °C/5 MPa/45 min and the micrograph (SEM) showing the corresponding position where the line analysis was conducted. (Note the flux of Al atoms from TiAl alloy side toward Ti64 alloy side and of Ti atoms in the opposite direction, and also V enrichment from Ti64 alloy and Nb enrichment from TiAl alloy) (the numbers in the micrograph indicate the positions where EDS point analyses were conducted)

interface between TiAl alloy and the reaction zone brittle. These results clearly indicate that the ideal bonding temperature is 850 °C, Fig. 10.

It was also determined that the increasing bond time results in a decrease in the shear strength for the bonds produced at temperatures of 825 and 850 °C using a pressure of 5 MPa, Figs. 11 and 12. This is due to the increase in the thickness of the brittle α_2 phase forming along the joint interface with increasing time (>1.5–2 μm, Fig. 6d), giving rise to brittle failure along the interface between TiAl alloy and the newly formed brittle α_2 phase. This in turn degrades the bond quality. The shear test results of the specimens extracted from the bonds produced at 850 °C with a bonding time of 15 min using different

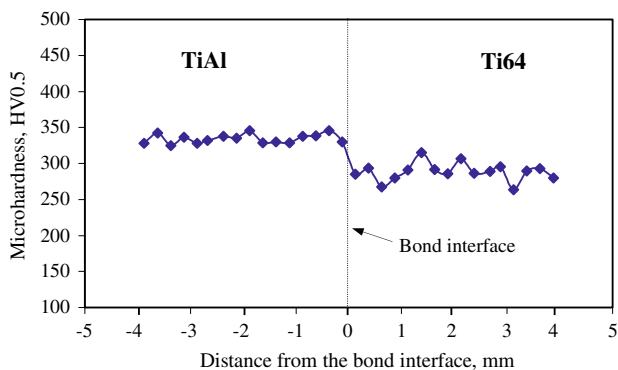


Fig. 8 Hardness profile of the bond produced with the parameters of 850 °C/5 MPa/45 min

Table 3 Results of shear testing

Bonding parameters (°C/MPa/min)	Shear strength values (Mpa)	Average shear strength (MPa)
825/5/15	358; 423; 402; 368	388
825/5/30	284; 348; 336; 385	338
825/5/45	280; 213; 173; 172	210
850/5/15	490; 487; 472; 481	483
850/5/30	290; 303; 271	288
850/5/45	97; 98; 100; 99	99
850/8/15	263; 444; 376; 312	349
875/5/15	280; 323; 273; 352	307
Base material Ti64	1298; 1426; 1473	1399
Base material TiAl	1536; 1393; 1446	1398

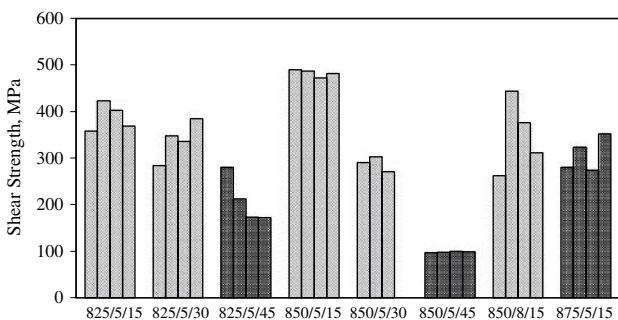


Fig. 9 Shear strength values of all the bonds produced. (Bond parameters: °C/MPa/min)

pressure levels showed that the increase of pressure from 5 to 8 MPa degrades the bond performance, Fig. 13.

Figure 14 shows the fracture surfaces of base materials and some of the bonds produced in this study. As expected, Ti64 alloy base material exhibits a more ductile failure than that of TiAl alloy, Fig. 14a, b. The fracture surface of the bond produced with the parameters of 850 °C/5 MPa/15 min, which displayed higher shear strength values than

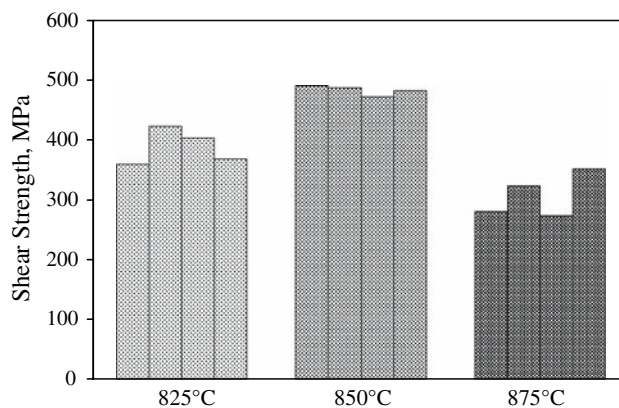


Fig. 10 The effect of temperature on the joint performance for the bonds produced using a pressure of 5 MPa for 15 min

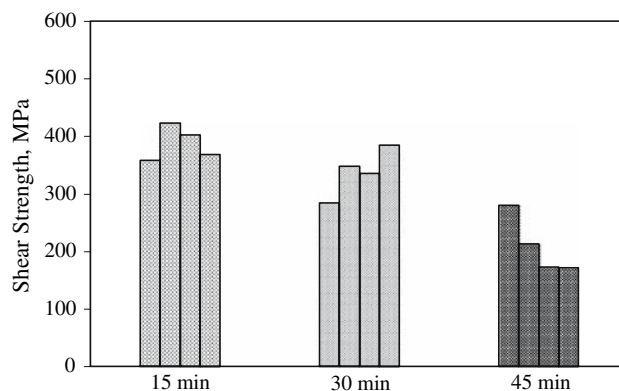


Fig. 11 The effect of time on the joint performance for the bonds produced at 825 °C/5 MPa

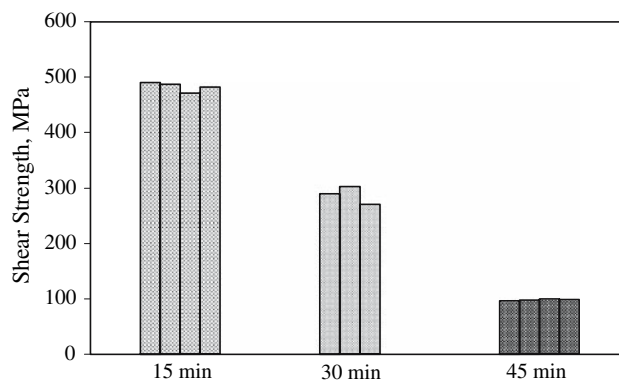


Fig. 12 The effect of time on the joint performance for the bonds produced at 850 °C/5 MPa

the bond produced with the parameters of 850 °C/5 MPa/45 min, is rougher in appearance with significant pull-out of the γ -TiAl material, Fig. 14c. As explained above, this can be attributed to the increase in the thickness of the newly formed brittle α_2 phase along the bond interface in the bonds produced with longer bonding times, i.e. 45 min,

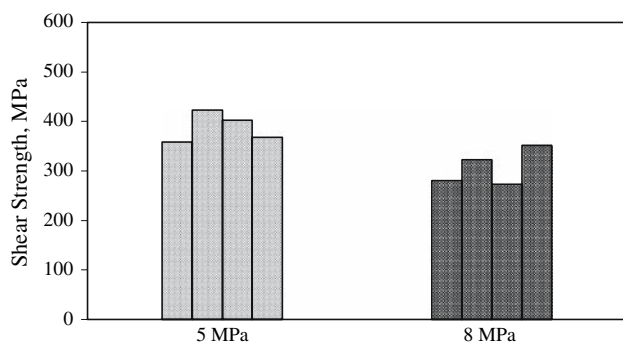


Fig. 13 The effect of pressure on the joint performance in the bonds produced at 850 °C/15 min

leading to brittle failure along the interface between TiAl alloy and this brittle phase. Thus, as the bonding time increases for a given set of temperature and pressure the fracture surface becomes very flat and featureless with some minor pull-out of the γ -TiAl material, Fig. 14d.

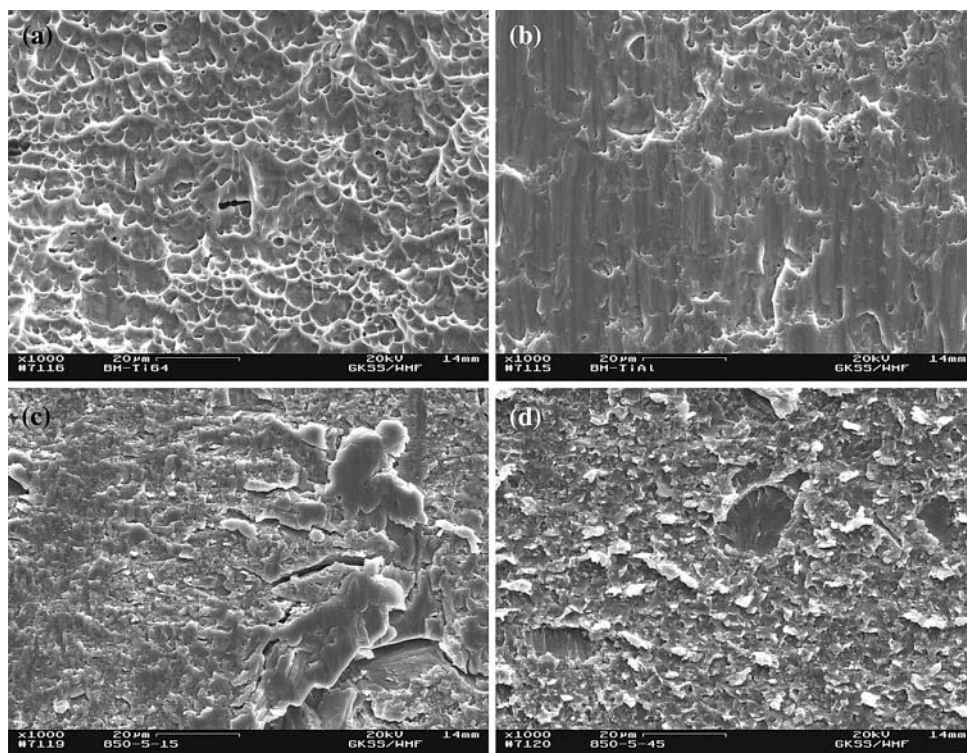
Conclusions

The following conclusions have been drawn from the present work:

- It was well demonstrated in this study that TiAl alloy can successfully be joined to Ti64 alloy by diffusion bonding.

- In all the bonds obtained, the formation of a brittle α_2 phase layer along the bond interface was detected. The thickness of this brittle layer increases with the increase of the bonding time.
- Although sound bonds were achieved, all the bonds produced exhibited lower shear strength values than those of both base materials joined due to the presence of a continuous brittle layer along the joint interface promoting brittle failure.
- Some pores were detected along the joint interface only in the bond produced with the parameters of 825 °C/ 5 MPa/15 min. Sound bonds without any pores or cracks were achieved with other parameter sets employed.
- The EDS chemical composition profiles exhibit a diffusion flux of Al-atoms from TiAl alloy toward Ti64 alloy and of Ti-atoms in the opposite direction. In addition to that, some Nb-enrichment from TiAl alloy and V-enrichment from Ti64 alloy in the reaction zone occur throughout the joints. No evidence of a diffusion flux of Cr was detected.
- The optimum bonding temperature was determined to be 850 °C for achieving the highest joint performance.
- Similarly, the optimum pressure was found to be 5 MPa. Further increase of pressure degrades the joint quality.
- It can be concluded that the bond strength is controlled by the interfacial porosity up to a maximum thickness of the brittle α_2 phase layer forming along the bond

Fig. 14 Fracture surfaces of the base materials and some of the bonds produced: (a) base material Ti64, (b) base material TiAl, (c) 850 °C/5 MPa/15 min, and (d) 850 °C/5 MPa/45 min. (Note: White areas in (d) indicate the remainings of the brittle reaction layer at the interface)



interface, i.e. 1.5–2 μm . The increase of the thickness of this brittle phase beyond this critical value results in a degradation of joint performance. Thus, beyond the critical thickness of the brittle phase forming at the interface, the presence of brittle reaction product becomes more critical in affecting the bond strength. The highest shear strength value has been obtained with a bonding time of 15 min for a given set of other parameters.

- The interface between TiAl alloy and the reaction zone seems to be more critical than that between Ti64 alloy and the reaction zone where the bonds failed in shear testing.

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