

Investigation into the microstructure and mechanical properties of diffusion bonded TiAl alloys

G. Çam · G. İpekoğlu · K.-H. Bohm · M. Koçak

Received: 12 July 2005 / Accepted: 24 October 2005 / Published online: 6 June 2006
© Springer Science+Business Media, LLC 2006

Abstract TiAl alloys are potential candidates for replacing conventional Ti-alloys in gas turbine applications in the relatively lower temperature sections, owing to their low density and excellent high temperature properties. However, their intolerable ambient temperature brittleness hinders their use in such applications. Recently, TiAl alloys with some room temperature ductility were developed through alloy development programmes using special production routes such as powder metallurgy. However, the room temperature brittleness of these alloys could not be overcome.

Sound joining of these alloys is a fundamental prerequisite for their successful integration into high temperature aerospace applications. It has been well demonstrated that diffusion bonding, a commonly used joining technology in conventional Ti-alloys, can successfully be used in joining of TiAl alloys both in as-cast or special-rolled conditions. In this study, diffusion bondability of a recently developed C containing TiAl alloy with a duplex microstructure using bonding parameters in the range of commercially available equipments was studied. Microstructural

investigations in the joint area of the bonds were conducted to observe the presence of any weld defect. Additionally, the mechanical behaviour of the bonds was determined by shear testing to find out the optimum bonding parameters. Furthermore, the effect of post-bond heat treatment on the mechanical properties was investigated.

Introduction

There have been numerous studies for the use of light weight titanium aluminides (TiAl alloys) in high temperature applications owing to their excellent combination of mechanical properties. The promising properties of these alloys are low density (3.7–3.9 g/cm³), high specific stiffness, excellent high temperature strength retention, excellent creep and oxidation resistance and burn resistance [1–6]. However, the rolling, forging and extrusion processes can be applied to these materials only at elevated temperatures due to their excessive brittleness at ambient temperature.

In recent years, a considerable number of studies have been carried out to improve the room temperature ductility of these alloys. But, despite all the attempts their room temperature ductility and formability could not be brought to a desirable level. Thus, the development of a suitable joining technology is the key factor for successful integration of these alloys to engineering applications. Therefore, numerous research programmes have recently been initiated on the joinability of these alloys. However, fusion welding processes, such as electron beam welding, are very complicated and present some difficulties like solidification cracking due to the high brittleness of these alloys [7, 8]. Fusion welding can only be applied to these materials with adequate precautions,

G. Çam (✉)
Faculty of Engineering and Architecture, Mechanical Eng. Dept.,
Mustafa Kemal University, Tayfur Sökmen Campus,
31034 Antakya/Hatay, Turkey
e-mail: gcam@mku.edu.tr

G. İpekoğlu
Faculty of Engineering, Mechanical Eng. Dept.,
Dokuz Eylül University, Ege University Campus,
35100 Bornova/Izmir, Turkey

K.-H. Bohm · M. Koçak
GKSS Research Center, Institute of Materials Research,
Max-Planck Str., D-21502 Geesthacht, Germany

such as pre-heating and controlled cooling after welding. On the other hand, brazing and solid state joining processes, such as diffusion bonding and friction welding, avoid many of these problems and are attractive for such brittle materials. Promising results have been achieved in solid state joining of these alloys [9–22]. It is well known that the most critical parameter in diffusion bonding of TiAl alloys is the grain size and the fine grained TiAl alloys can be bonded at lower temperatures. Recent studies have shown that sound joints could be obtained at lower temperatures by grain refinement on the surfaces to be joined using a laser beam [23, 24].

In this study, the bondability of a new generation C-containing duplex TiAl alloy has been investigated using diffusion bonding parameters in the range of those of commercial bonding equipments. The bonding parameters (i.e., temperature, pressure and time) have systematically been altered to determine the optimum parameters for achieving sound bonds. Optical and scanning electron microscopy were employed to investigate the microstructural evolution and the presence of any weld defects along the bond line. Shear testing was conducted to evaluate the joint performance of the bonds obtained using different parameters. Moreover, the effect of post-bond heat treatment on mechanical behaviour of the joints was investigated.

Experimental procedure

The material used in this study is a new generation C-containing duplex TiAl alloy and its chemical composition is Ti-48 at% Al-3.7 at% (Nb, Cr, C).

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 remelting 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,

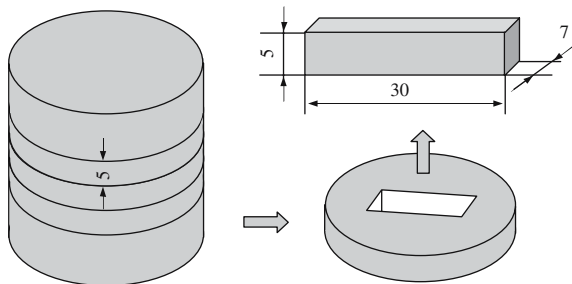


Fig. 1 Schematic showing the preparation of diffusion bond specimens (the cutting of 5 mm thick slices from TiAl bar and the extraction of the specimens from these slices)

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 24 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 slow cooling ($1,527$ °C/h = 1,800 K/h). After this two-stage heat treatment, diffusion bonding specimens of $30 \times 7 \times 5$ mm were extracted from these slices by EDM technique, Fig. 1.

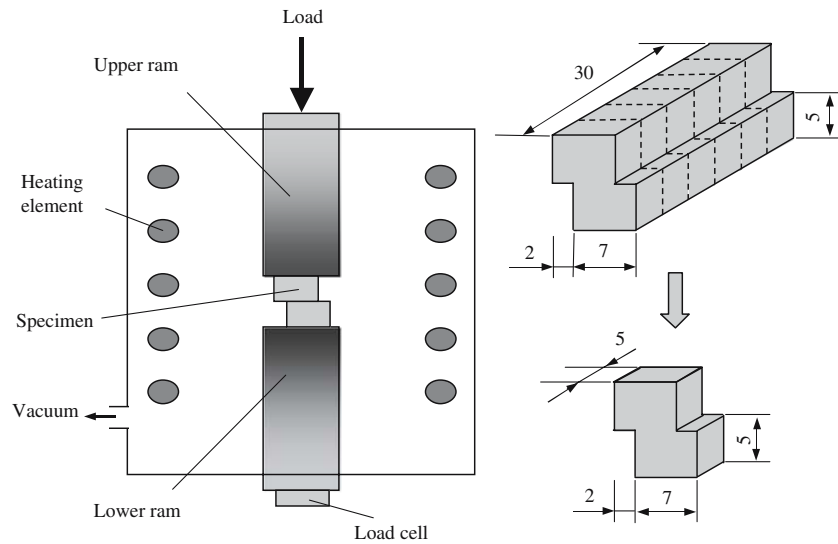
The surfaces of diffusion bonding specimens extracted by EDM technique were machined parallel. The mating surfaces of the specimens were only ground with SiC paper down to grit 1200 and ultrasonically cleaned using acetone. Fig. 2a schematically shows the diffusion bonding procedure in which two specimens were placed on top of each other and bonded 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^{-4} mbar using different combination of bonding parameters, Table 1. All the bonds were produced in two sets and one set was post-bond heat treatment. The post-bond heat treatment was carried out at 1,430 °C for 30 min.

In order to carry out microstructural investigations and to evaluate joint performance, metallography specimens (one specimen for each joint) and shear test specimens (minimum 4 specimens for each joint) were extracted from the joints both in the as-bonded and post-bond heat treated conditions by EDM technique, Fig. 2b. Metallography specimens were mounted in bakelite, ground with SiC paper down to grit 4000 and then polished down to 1 μ m using Al_2O_3 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 weld defect. Detailed point EDS analyses along the bond lines were also conducted to identify the new phases formed along the bond interface.

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 modes. Results of all these investigations were used to determine the optimum bonding parameters for achieving sound bonds in TiAl alloy. Moreover, the effect of post-bond heat treatment on joint quality was investigated.

Fig 2 Schematic illustrating (a) diffusion bonding of TiAl alloys and (b) the extraction of the shear specimens from the bonds produced



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).

Table 1 gives the bonding parameters employed in this study. Optical and scanning electron microscopy conducted on the bonds produced exhibited that the bond interface is clearly visible due to the discontinuity of the grains in both

Table 1 Diffusion bonding parameters used in this study

Materials bonded	Bond parameters		
	Temperature ($^{\circ}\text{C}$)	Pressure (MPa)	Time (h)
TiAl–TiAl	950	10	3
TiAl–TiAl	950	10	5
TiAl–TiAl	1,000	5	5
TiAl–TiAl	1,000	10	1
TiAl–TiAl	1,000	10	3
TiAl–TiAl	1,000	10	5
TiAl–TiAl	1,000	20	1
TiAl–TiAl	1,000	20	3

Fig. 3 Schematic showing the shear testing

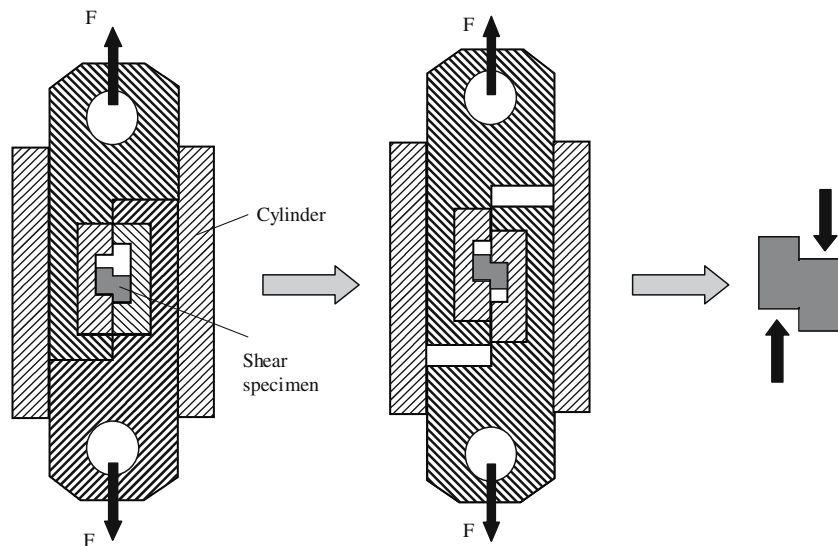
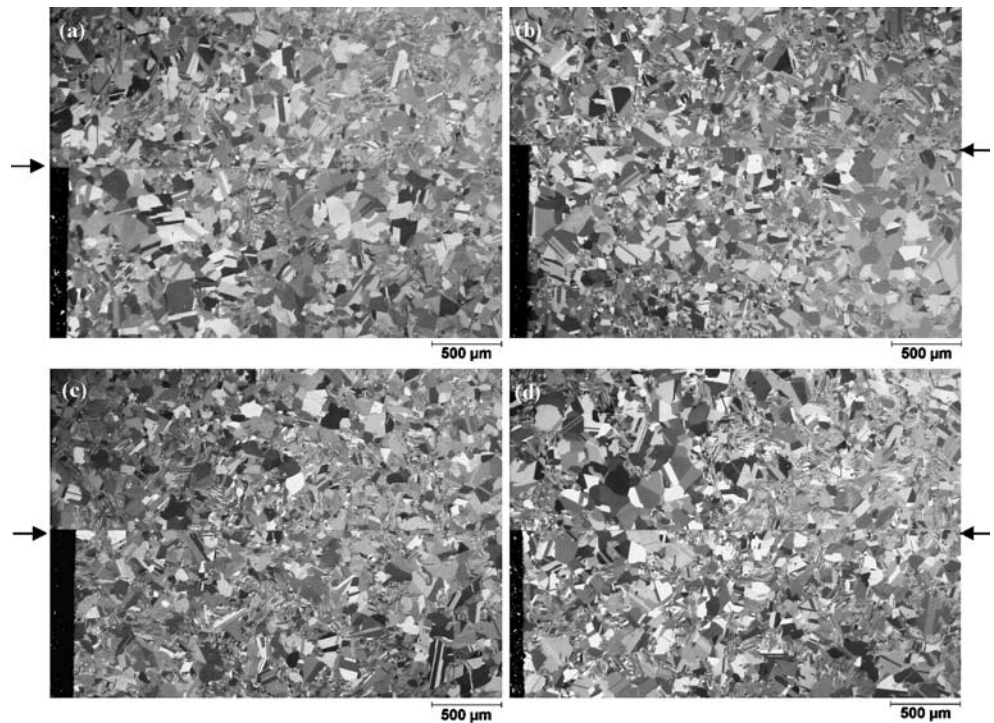


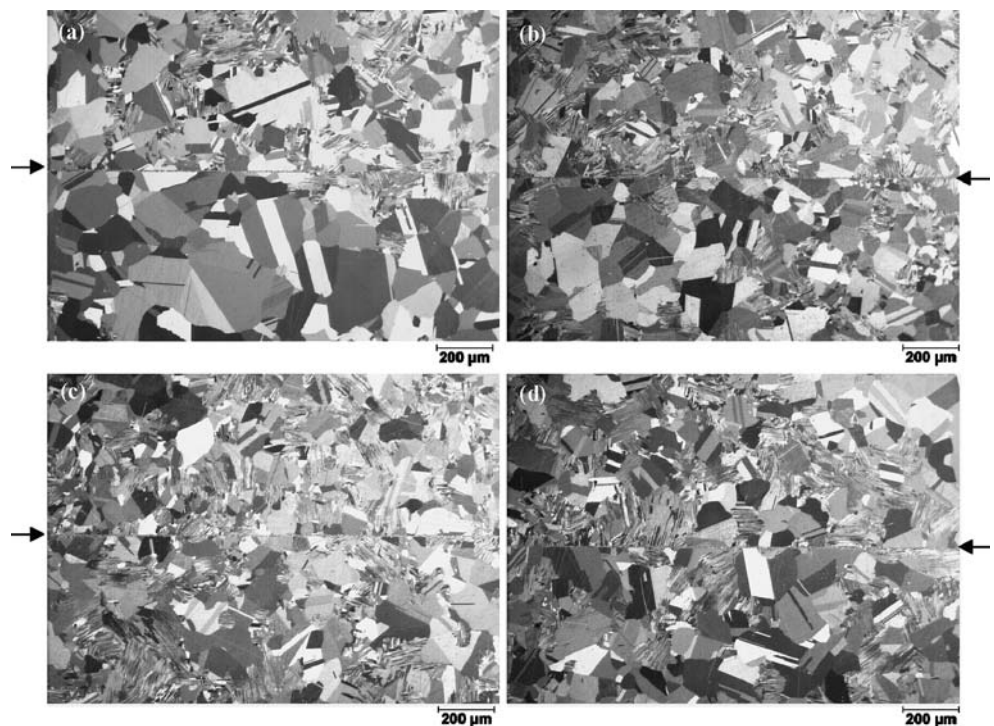
Fig. 4 Polarized light micrographs showing the joint area of some of bonds produced: (a) 1,000 °C/10 MPa/1 h, (b) 1,000 °C/10 MPa/3 h, (c) 1,000 °C/10 MPa/5 h and (d) 1,000 °C/20 MPa/3 h. (Note that the arrows indicate the bond interfaces)



sides joined. But, no weld defect was detected along the joint interfaces, indicating that sound bonds were achieved, Fig. 4 and 5. The electron microscopy conducted on the metallography specimens extracted from the bonds produced showed that a newly formed phase was detected in the bond interfaces of all the joints, Fig. 6. The detailed EDS analyses conducted in these newly formed phases

indicated that it is probably α_2 phase (Ti_3Al) having a chemical composition of 67–77% Ti and 21–26% Al, Fig. 7. This can be attributed to the fact that the oxygen contamination of the mating surfaces cannot be completely avoided, which is a strong stabilizer of the α_2 phase. A detailed electron microscopy conducted by Buque and Appel [22] on diffusion bonded as-cast TiAl alloy with a

Fig. 5 Higher magnification polarized light micrographs of some of the bonds obtained: (a) 1,000 °C/5 MPa/5 h, (b) 1,000 °C/10 MPa/1 h and (c) 1,000 °C/10 MPa/5 h and (d) 1,000 °C/20 MPa/3 h. (Note that the arrows indicate the bond interfaces)



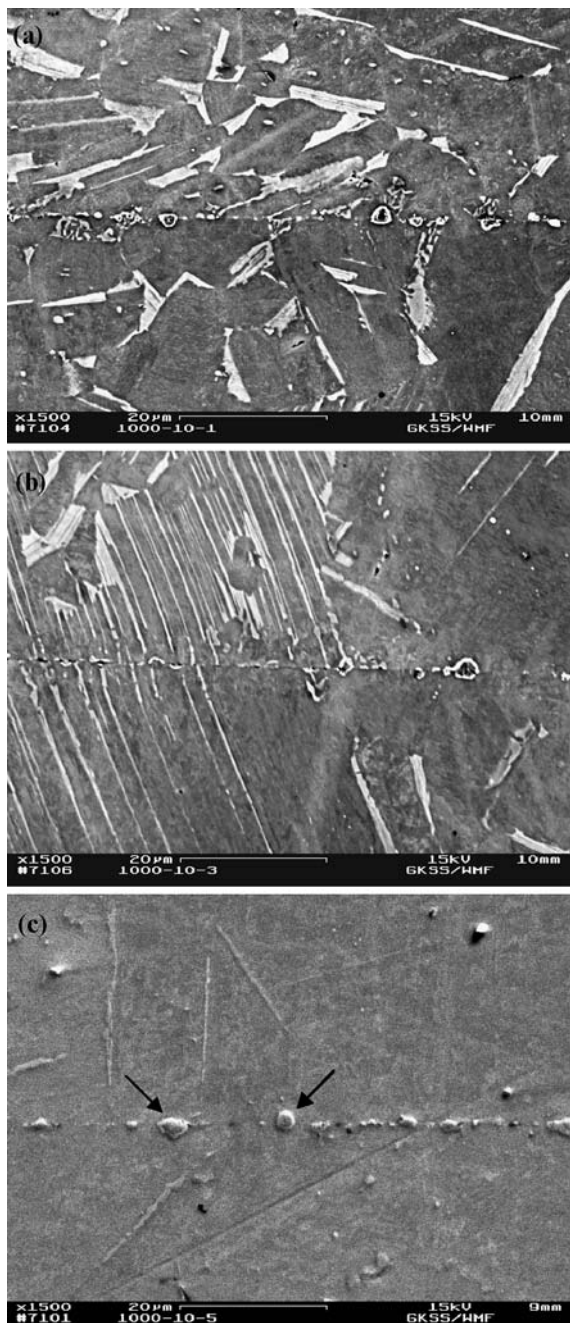


Fig. 6 Back-scattered micrographs (SEM) of the bonds produced at 1,000 °C with a pressure of 10 MPa using different bond times. (a) 1 h, (b) 3 h and (c) 5 h (Note that the newly formed α_2 phases along the bond interface indicated by arrows coarsen and become isolated as the bonding time increases)

composition of Ti-45 at% Al also displayed that the α_2 phase forms along the bond interface. Dark regions along the bond interfaces shown in Fig. 6 are not porosity. The EDS analyses conducted in these regions indicated that they were brittle α_2 (Ti_3Al) phases originally present in these areas and fallen out during mechanical surface polishing. It was also observed that the size of the α_2 phases forming during bonding increases with the increase in the

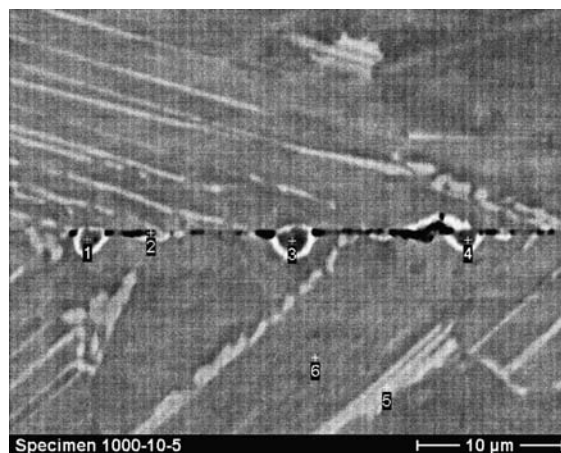


Fig. 7 The chemical analyses (EDS) results conducted in the bond area of the joint produced with the parameters of 1,000 °C/10 MPa/5 h and the micrograph (SEM) showing the corresponding points where the analyses were carried out

bonding time for a given set of temperature and pressure, Fig. 6. Furthermore, optical and electron microscopy investigations also revealed that the grain coarsening in TiAl alloy takes place with the increase in bond time, Figs. 6 and 8.

A coarse-grained fully lamellar microstructure (average grain size is about 500 μm) was observed in all the bonds after the post-bond heat treatment given at 1,430 °C for 30 min, Fig. 9. This phase transformation led to the fact that the original bond interface becomes invisible. As seen in Fig. 9, the bond interface can only be found through following the edges of the specimens.

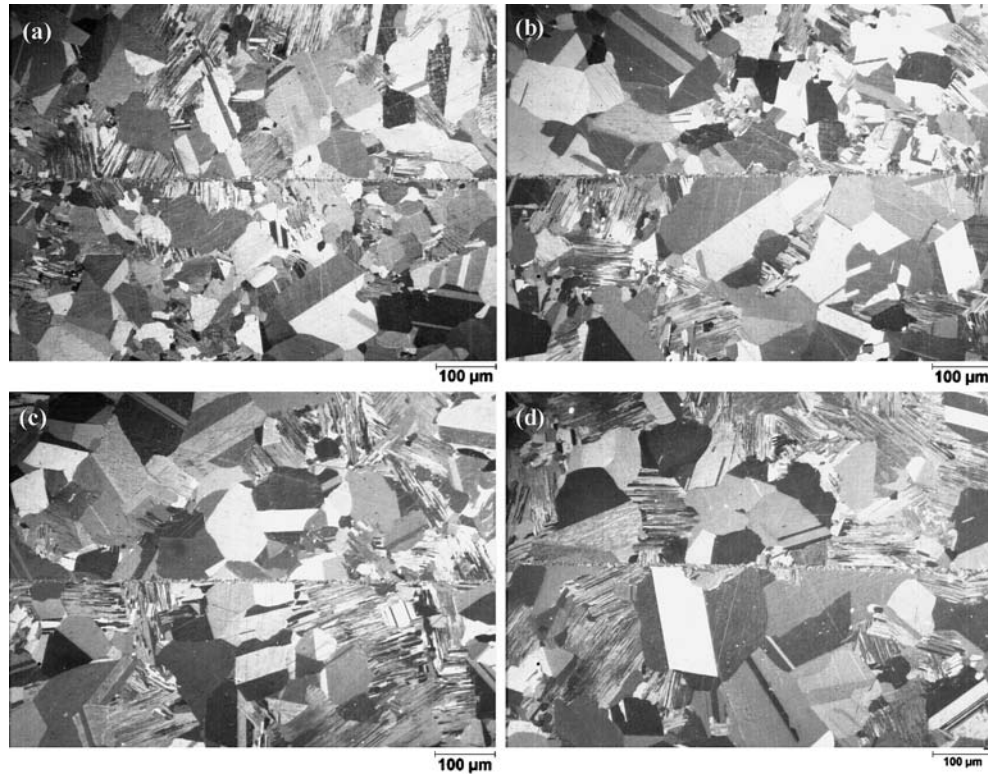
Joint performance

The joint performances of the bonds produced using different parameters were determined by shear testing both in the as-bonded and post-bond heat treated conditions. The results of these tests were given in Table 2 and illustrated as a block diagram in Fig. 10. The average shear strength values of the bonds produced in the as-bonded condition are shown in Fig. 11.

The highest average shear strength value, i.e., 388 MPa, was exhibited by the bond produced with the parameters of 1,000 °C/10 MPa/5 h in the as-bonded condition. Similar shear strength values were also reported both for diffusion bonded special hot-rolled and cast TiAl alloys, special hot-rolled alloys exhibiting slightly higher values [16, 19]. 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. 10. However, the shear strength values of all the bonds obtained are significantly lower than those of the base material, Table 2, although sound bonds were achieved in

Fig. 8 Grain coarsening around the bond interface in TiAl with the increasing bond time.

(a) 1,000 °C/10 MPa/1 h, (b) 1,000 °C/10 MPa/3 h, (c) 1,000/5 h and (d) 1,000 °C/20 MPa/3 h. °C/10 MPa



all the cases. The reason for this is the formation of the brittle α_2 phase along the joint interface, Fig. 7, tendering the bond interface brittle. The second highest average shear strength value, i.e., 383 MPa, was exhibited by the bond

produced with the parameters of 1,000 °C/20 MPa/3 h. However, some of the specimens extracted from this bond displayed relatively low shear strength, i.e., 322 MPa, Table 2. Nevertheless, the shear strength level of

Fig. 9 Evolution of a coarse-grained fully lamellar microstructure in the bonds after post-bond heat treatment.

(a) 950 °C/10 MPa/3 h, (b) 1,000 °C/10 MPa/1 h, (c) 1,000 °C/10 MPa/3 h and (d) 1,000 °C/10 MPa/5 h

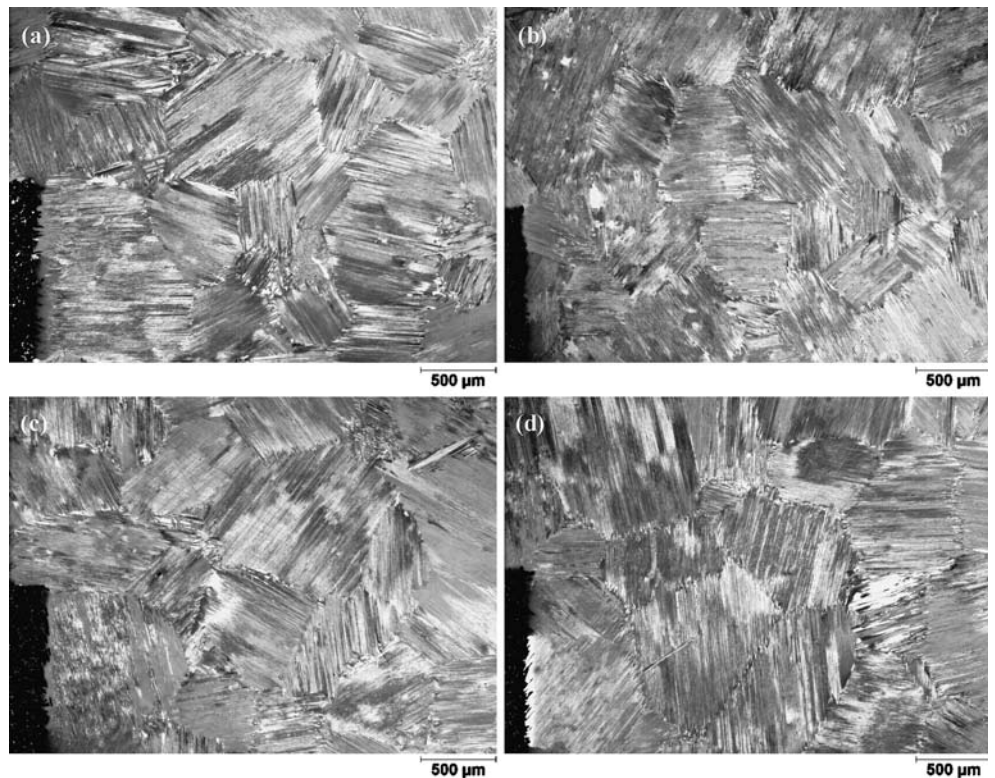
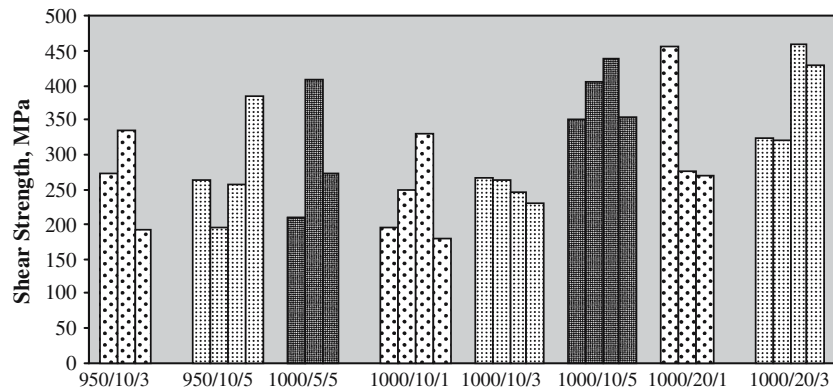


Table 2 Results of shear testing

Bond parameters (°C/MPa/h)	Shear Strength, MPa									
	As-bonded					Post-bond heat treated				
	1	2	3	4	Ave.	1	2	3	Ave.	
950/10/3	272.5	335.0	191.4	–	266.3	433.6	412.1	450.2	432.0	
950/10/5	262.7	197.3	257.8	384.8	275.7	394.5	333.0	456.1	394.5	
1,000/5/5	209.0	407.2	272.5	–	296.2	508.8	559.6	287.1	534.2	
1,000/10/1	196.3	251.0	330.1	180.7	239.5	543.0	509.8	539.0	530.6	
1,000/10/3	267.6	263.7	246.1	230.5	252.0	459.0	408.2	439.5	435.6	
1,000/10/5	352.5	405.3	440.4	355.5	388.4	411.1	561.5	492.2	488.3	
1,000/20/1	455.1	276.4	268.6	–	333.4	663.1	535.2	542.0	580.0	
1,000/20/3	323.2	322.3	458.0	429.7	383.3	450.2	444.3	464.8	453.1	
Base material	1,536.4	1,392.6	1,446.3	–	1,398.4	–	–	–	–	

Fig. 10 Shear strength values obtained from all the bonds produced in the as-bonded condition. (Bond parameters: °C/MPa/h)



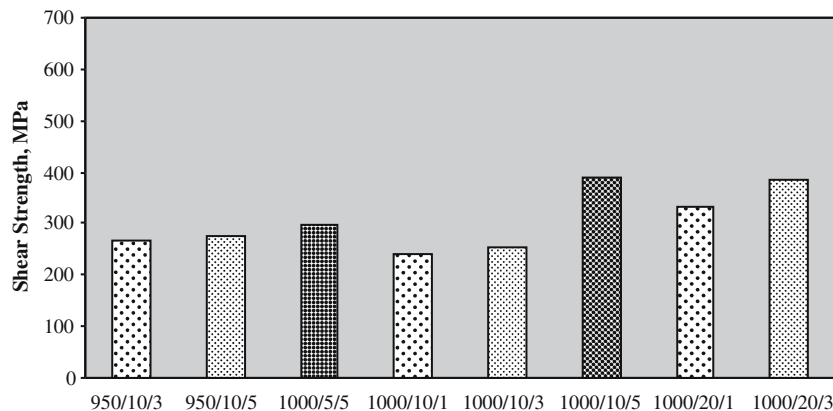
homogeneous as-cast TiAl alloy base material has been achieved in these bonds [19].

A relatively high deviation in the shear strength values of four specimens extracted from each bond was observed in the joints, except the ones produced with the parameters namely 1,000 °C/10 MPa/3 h and 1,000 °C/10 MPa/5 h, Table 2. These results indicate that with a given set of other parameters a temperature of 950 °C and a pressure of 5 MPa are not adequate to achieve a sound bond with homogeneous bond quality throughout the joint. Moreover,

the bond time, i.e., 3 h, used at 1,000 °C/20 MPa was found to be insufficient for obtaining optimum shear strength, Table 2.

It was determined that the increasing bond time improves the shear strength for the bonds produced at 1,000 °C using a pressure of 10 MPa, Fig. 12. This can be attributed to the formation of fine grained and almost continuous brittle α_2 phase along the joint interface in the bonds produced using shorter bond times, Fig. 6a and b, giving rise to brittle failure. This in turn degrades the bond

Fig. 11 Average shear strength values of the bonds produced in the as-bonded condition. (Bond parameters: °C/MPa/h)



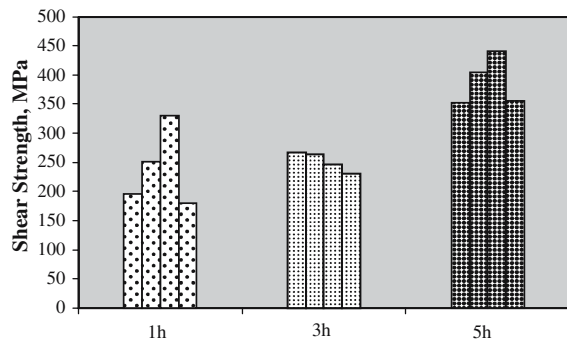


Fig. 12 The effect of time on the joint performance in the bonds produced at 1,000 °C/10 MPa

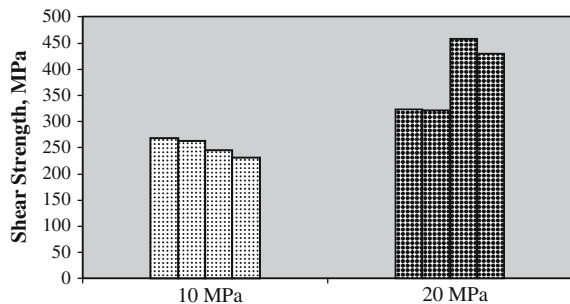


Fig. 13 The effect of pressure on the joint performance in the bonds produced at 1,000 °C/3 h

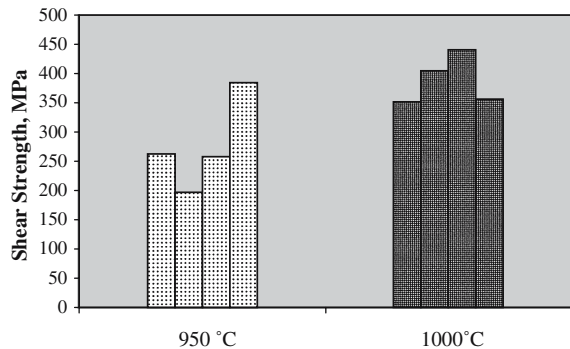


Fig. 14 The effect of temperature on the joint performance in the bonds produced using 10 MPa pressure for 5 h

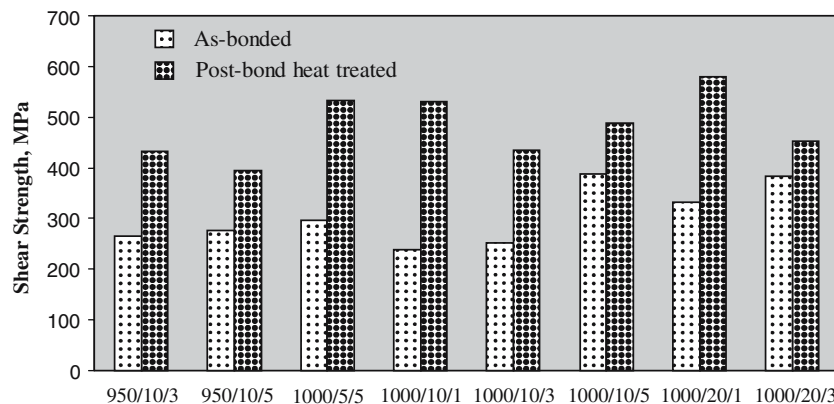


Fig. 15 The effect of post-bond heat treatment on the joint performance of the bonds produced with different bond parameters. (Bond parameters: °C/MPa/h)

quality. In the bonds produced with longer bonding times, i.e., 5 h, the brittle α_2 phase forming along the bond interface becomes coarse grained and in turn becomes isolated and discontinuous, Fig. 6c. Thus, the bond interface does not present any more a preferable path for propagation of a brittle failure through it.

The shear test results of the specimens extracted from the bonds produced at 1,000 °C with a bonding time of 3 h using different pressure levels showed that the increase of pressure from 10 MPa to 20 MPa also improves the bond performance, Fig. 13. The comparison of the shear strength values obtained from the bonds produced using a pressure of 10 MPa and a bonding time of 5 h at different temperatures indicates that 950 °C is not a sufficient temperature for achieving sound bonds with high joint performance, Fig. 14. Thus, the shear strength can further be improved by increasing the bond temperature, pressure or time.

Figure 15 shows the effect of post-bond heat treatment (at 1,430 °C for 30 min) on the shear strength values of the bonds. As seen from this figure, the post-bond heat treatment improves the shear strength for all the bonds produced. All the bonds exhibited similar shear strength values after post-bond heat treatment regardless of the bonding parameters with which they were produced. This is due to the disappearance of the original joint interface through the formation of coarse lamellar grains across the bond line in all the bonds, Fig. 9. The shear strength of post-bond heat treated specimens is, however, significantly lower than that of the base material. This is not unexpected since the original fine grained duplex microstructure is much more ductile and, therefore, exhibits a higher shear strength than the coarse-grained fully lamellar microstructure evolved in post-bond heat treatment.

Figure 16 shows the fracture surfaces of base material and some of the bonds produced in this study. As expected, TiAl alloy base material with a duplex microstructure exhibits a fracture surface containing ductile and cleavage

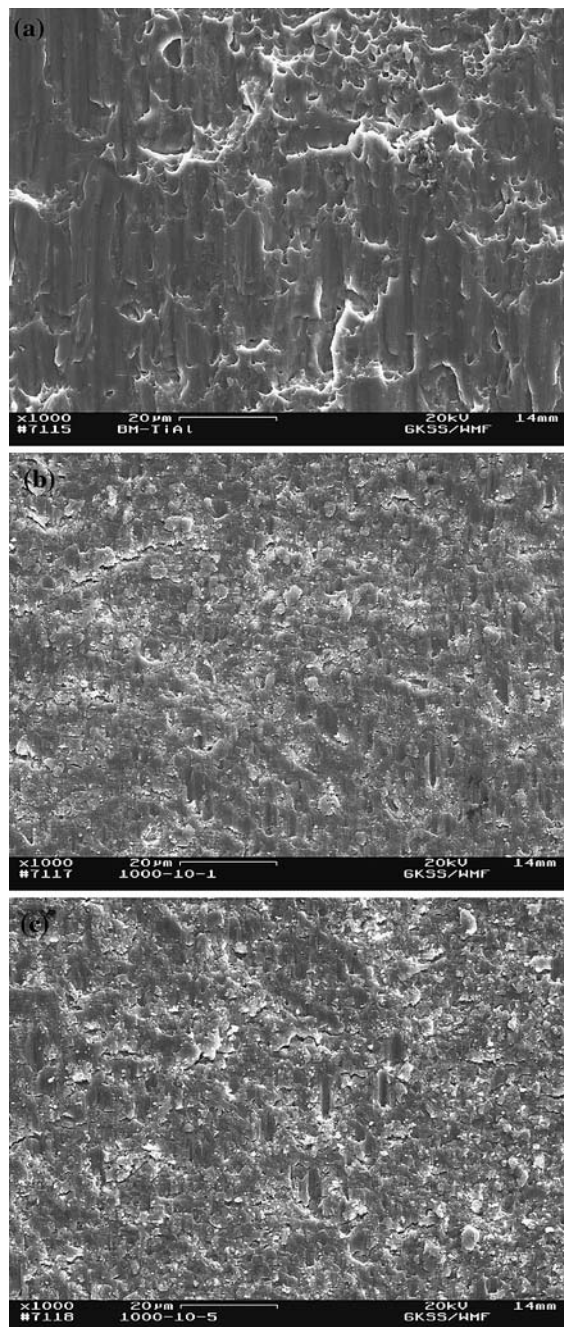


Fig. 16 Fracture surfaces of the base material and some of the bonds produced: (a) Base material, (b) 1,000 °C/10 MPa/1 h and (c) 1,000 °C/10 MPa/5 h

failure regions, Fig. 16a. The fracture surface of the bond produced with the parameters of 1,000 °C/10 MPa/5 h, which displayed higher shear strength values than the bond produced with the parameters of 1,000 °C/10 MPa/1 h, is rougher in appearance with significant failure in the γ -TiAl base material, Fig. 16b and c. As the bonding time decreases for a given set of temperature and pressure the

fracture surface becomes flat and failure occurs through the newly formed brittle α_2 phase along the joint interface as explained above, Fig. 16b.

Conclusions

The following conclusions have been drawn from the present work:

- Sound joints without any pores or cracks can be produced in a new generation C-containing TiAl alloy by diffusion bonding. In all the bonds obtained, the formation of a brittle α_2 phase along the bond interface was detected.
- The highest shear strength values were obtained with the parameter sets of 1,000 °C/10 MPa/5 h and 1,000 °C/20 MPa/3 h, namely ~390 and 383 MPa, respectively. 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 brittle phase along the joint interface promoting brittle failure.
- The results indicated that the bond performance improves with the increase in the bonding parameters. Thus, the shear strength can be further increased by using a longer bonding time at 1,000 °C/20 MPa.
- The continuity of the brittle α_2 phase along the bond interface, which forms during bonding, degrades the joint performance by presenting a favourable crack path in shear loading. With longer bonding times, this brittle phase grains join together and coarsen, in turn become discontinuous along the interface, thus the interface is no longer a favourable crack path for brittle fracture.
- Post-bond heat treatment leads to an improvement in the joint performance. Similar shear strength values were obtained from all post-bond heat treated specimens produced with different bonding parameters. This is due to the formation of coarse fully lamellar grains across the joint interface resulting in the disappearance of the original interface.
- Although the original joint interface disappears through the post-bond heat treatment, it cannot restore the shear strength of the bonds to the level of that of the base material. This can be attributed to the fact that the coarse-grained fully lamellar microstructure is more brittle than the original fine-grained duplex microstructure of the base material.

Acknowledgements This work was carried out within the German–Turkish joint project entitled “Solid State Joining of Advanced Light Weight High Temperature Materials for Aerospace and Automobile Applications” financed by KFA-Jülich Research Center, Germany

and Tübitak, Ankara, Turkey. The authors would like to thank both organisations for their financial support. The authors also thank Dr. F. Appel for supplying the new generation C-containing TiAl alloy, Mr. V. Ventzke for his help with SEM and Mrs. P.-M. Fischer for her help with optical microscopy.

References

1. Kim Y-W (1989) JOM 41:24
2. Kim Y-W, Dimiduk DM (1991) JOM 43:40
3. Kim Y-W. (1994) JOM 46:30
4. Yamaguchi M, Umakoshi Y (1990) Prog Mater Sci 34:1
5. Appel F, Wagner R (1998) Mater Sci Eng R 22:187
6. Appel F, Brossmann U, Christoph U, Eggert S, Janschek P, Lorenz U, Müllauer J, Oehring M, Paul JDH (2000) Adv Eng Mater 2(11):699
7. Patterson RA, Martin PL, Damkroger BK, Christodoulou L (1990) Weld J 69:39s
8. Threadgill PL (1995) Mater Sci Eng A192–193:640
9. Yan P, Wallach RE (1993) Intermetallics 1:83
10. Çam G., Bohm K-H, Müllauer J, Koçak M (1996) JOM 48(11):66
11. Çam G, Müllauer J, Koçak M (1997) Sci Technol Weld Join 2(5):213
12. Bohm K-H, Ventzke V, Çam G, Koçak M (1997) Schweißen und Schneiden 49(9):660
13. Glatz W, Clemens H (1997) Intermetallics 5:415
14. Çam G, Koçak M (1998) Int Mater Rev 43(1):1
15. Holmquist M, Recina V, Ockborn J, Pettersson B, Zumalde E (1998) Scripta Mater 39:1101
16. Çam G, Clemens H, Gerling R, Koçak M (1999) Intermetallics 7(9):1025
17. Çam G, Clemens H, Gerling R, Koçak M (1999) Z Metallkd 90 (4):284
18. Çam G, Bohm K-H, Koçak M (1999) Schweißn und Schneiden 51(8):470
19. Çam G, Koçak M (1999) J Mater Sci 34:3345
20. Lee SJ, Wu SK (1999) Intermetallics 7(1):11
21. Masahashi N, Hanada S, Mizuhara Y (2001) Mater Trans 42(6):1028
22. Buque C, Appel F (2002) Z Metallkd 93 (8) 784
23. Gremand M, Garrard M, Kurz W (1990) Acta Metall 38:2587
24. Lin JG, Yu GS, Wu GQ, Huang Z (2001) J Mater Sci Lett 20:1671