JOURNAL OF MATERIALS SCIENCE 27 (1992) 391–398

Diffusion bonding of Ti–6Al–4V alloy at low temperature: metallurgical aspects

F. A. CALVO, J. M. GÓMEZ DE SALAZAR, A. UREÑA, J. G. CARRIÓN, F. PEROSANZ
Dept. Ciencia de los Materiales e Ingeniería Metalúrgica, Facultad de Ciencias Químicas, Universidad Complutense de Madrid, 28040 Madrid, Spain

The diffusion bonding of the Ti–6Al–4V alloy at low temperature (850 °C) has been studied. The principal objective of this investigation was the development of a diffusion bonding procedure suitable for Ti–6Al–4V alloy and capable of being used as part of a superplastic forming/diffusion bonding process. It was found that high-quality joints can be obtained by bonding at 850 °C, with pressures of 4 MPa and times in the range 90–120 min. Mechanical properties of the joints were determined using cylindrical and plane test pieces. Tensile, shear and peeling tests were used to determine the strength of the joints. On bonding with the above conditions, the parent alloy strength was reached. Little reduction in these values was measured because the heat treatment was applied during bonding. A metallographic study by scanning electron microscopy and energy dispersive spectroscopy was performed to determinate the influence of the previous parameters on the microstructural changes that occur in the joint. Grain growth kinetics and ratio of bonding area were also studied. The results shows that a new method of diffusion bonding for Ti–6Al–4V alloy has been developed. This method can be carried out using lower bonding temperatures than in conventional processes.

1. Introduction

The study of diffusion bonding of Ti–6Al–4V alloy has developed as a result of experience obtained from the investigation on superplastic forming of this and other titanium alloys for application in the fabrication of aircraft structures. Ti–6Al–4V alloy [1] and most titanium alloys formed as sheets [2, 3] can be subjected to a combined process of diffusion bonding (DB) with superplastic forming (SPF) to produce complex components in one operation with substantial cost and weight savings. This process has been studied and different experimental conditions for SPF/DB of Ti–6Al–4V have been proposed [4–9], but they restricted the diffusion bonding temperature to a very narrow range (920–930 °C) where the SPF conditions of the material are the best [3], providing the optimum volumetric equilibrium between the microstructural phases that are present in the alloy [10].

However, the requirement that both bonding and forming must be carried out at high temperatures is one factor which limits the application of SPF/DB to titanium alloys. For these reasons, there is a considerable interest in the extension of SPF/DB technology in titanium alloys to lower temperatures.

The present paper describes the results of experimental work in which the main objective was to develop a solid state bonding process for titanium alloys using bonding temperatures of 850 °C which approximately corresponds to half the melting point of titanium alloys.

The diffusion bonds obtained in this research were studied by metallographic techniques to determine the microstructural changes in the alloy after the thermomechanical treatment involved in the diffusion bonding process. Both the formation of new phases and grain growth of the parent alloy were investigated. The quality of the bonds was estimated using various mechanical methods (tensile, lap shear and peel strength tests) with different bonding conditions. Fractography of tested bonds made it possible to determine the relation between the bonded area and the mechanical properties.

2. Experimental procedure

2.1. Materials and preparation

The parent materials used in this investigation were received in the form of sheets with different thicknesses (from 1.0–2.5 mm), and in 28 mm thick plate, both in the annealed condition. The chemical compositions are given in Table I.

Metallographic studies and mechanical tests were carried out on the bonded joints. Different types of specimen were designed and prepared for each kind of test. For microstructural studies, pairs of rectangular specimens, 20 mm × 15 mm, were cut from the 1 mm thick sheet and bonded at different conditions. The microstructure of the as-received material was characterized in one of these specimens using different metallographic techniques. This microstructure is shown in Fig. 1. It is principally α-phase that forms the alloy matrix with β aggregates present on the grain boundaries of the α matrix. A very fine precipitate can be observed inside a grain. It was identified as α_2 phase.

0022–2461/92 $03.00 + .12 © 1992 Chapman & Hall

TABLE I Chemical composition of parent materials

Materials	Chemical composition (%)							
	Al	V	Fe	O	C	N	H	Ti
Sheets	6.20	4.30	0.08	–	0.05	0.009	0.005	Bal.
Plate	6.15	4.40	0.09	–	0.05	0.010	0.005	Bal.

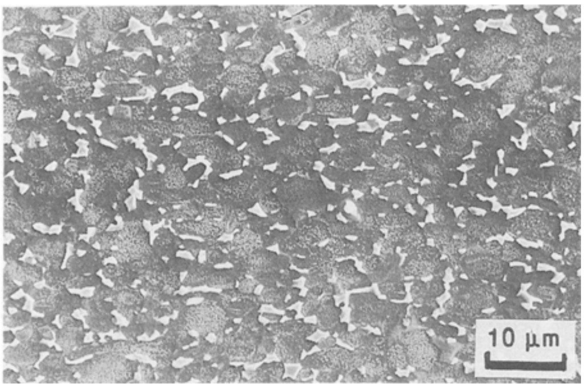

Figure 1 Parent alloy microstructure.

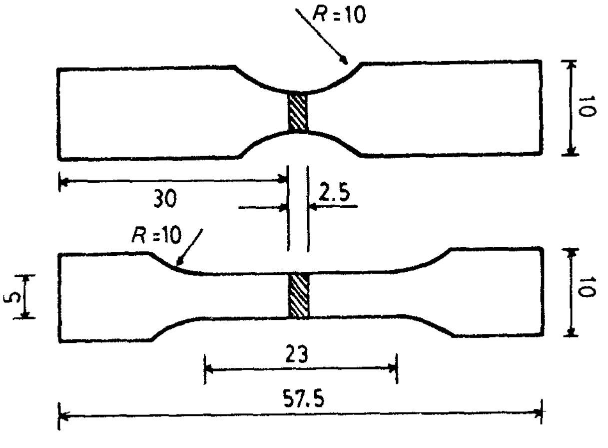

Figure 2 Dimensions (mm) of shear specimens for diffusion bonded sheets.

For the shear test, pairs of rectangular specimens, 30 mm x 10 mm, were cut from a 2.0 mm thick sheet and bonded as shown in Fig. 2. The overlap length was 2.5 mm, and after bonding they were machined to make two different test pieces, one a standard single overlap specimen and the other with a variable width in the overlap zone. The overlap length of both test pieces was 12.65 mm.

Peel test piece blanks, 100 mm x 17 mm, made from 2.5 mm thick sheet, were bonded at one end, with a bonding length of 17 mm as shown in Fig. 3. They were bending into a suitable shape for peel testing.

Butt joints between cylindrical specimens of 10 mm diameter and 20 mm length, made from the plate material, were obtained. The tensile strength of these joints was measured using cylindrical tensile test pieces (ASTM A370). They were machined from bonded specimens with the bond interface positioned in the middle of the gauge length. The dimensions of these specimens are shown in Fig. 4.

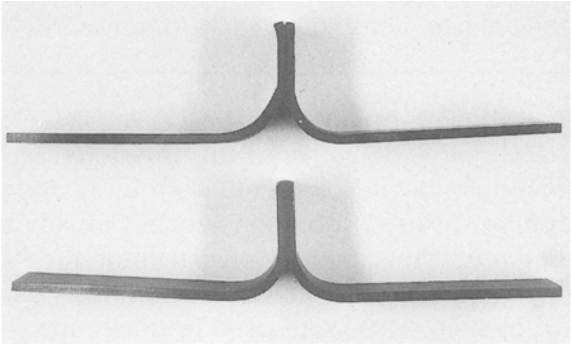

Figure 3 Peel test pieces.

For cylindrical specimens, the areas to be bonded were prepared following two different methods. Some of them were machined (turned) to 15 μm average roughness and the others were ground with 600 grade emery paper to 0.1 μm. The flat specimens (sheet) were only prepared in the second way. Just before bonding, all specimens were cleaned with acetone in an ultrasonic bath.

2.2. Bonding procedure

Diffusion joints were made using two different bonding machines. One used induction heating and applied the load by a mechanical screw feed system; the other applied the pressure by a lever system and used external resistance heating. Both types of equipment had high vacuum systems and all diffusion bonds were produced in the vacuum range 10^{-2}–10^{-3} Pa. Diffusion bonding tests for all types of specimen were made at 850 °C. Pressures of 2 and 4 MPa and bonding times between 30 and 120 min were studied with the above surface conditions.

The thermal bonding cycles used for the Ti–6Al–4V alloy were similar to those used for the different specimens tested. The heating speed was 0.4 $°C\,s^{-1}$ and after the set bonding time, the specimens were cooled in the vacuum chamber at slow cooling rates in the range 0.07–0.1 $°C\,s^{-1}$.

3. Results and discussions

3.1. Microstructure

A metallographical study of transverse sections of the diffusion bonds was carried out using light and scanning electron microscopy. In the as-bonded condition the two-phase structure of the parent alloy is preserved, although the α-phase grain grew. Although grain growth was observed during bonding (Fig. 5), for the bonding times tested, the grain size always

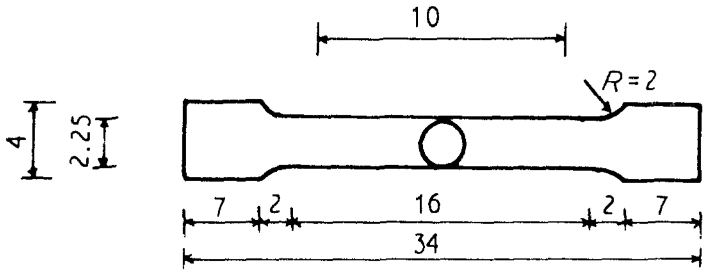

Figure 4 Dimensions (mm) of tensile specimens for diffusion bonded cylinders.

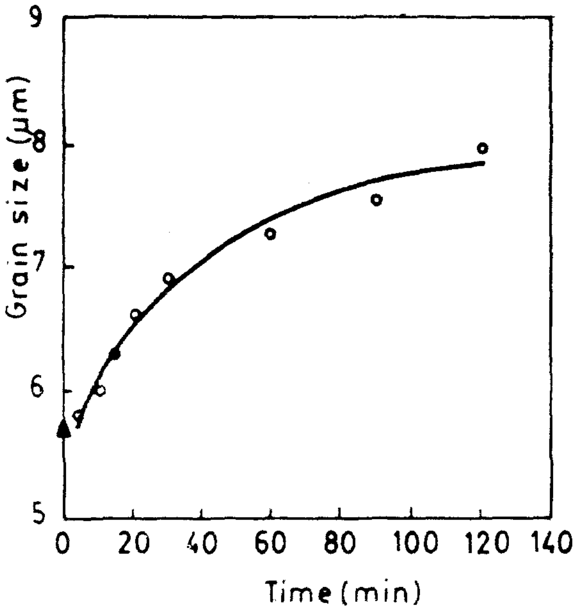

Figure 5 Grain size growth with bonding time: (▲) parent alloy, (○) diffusion bonds.

remained in the range required to develop superplastic properties. This grain growth is linked to the transformation of β phase into acicular α during cooling after the bonding procedure.

The precipitates observed inside the α-grain in the as-received material, also appeared after the bonding cycle. However, increasing bonding time did favour the homogenization of the alloy elements in the metallic matrix and the dissolution of those aggregates. After 120 min bonding, precipitates could not be observed (Fig. 6a and b).

Energy dispersive spectroscopic (EDS) microanalysis proved that the intercrystalline precipitate was a Ti–Al compound (Fig. 7), probably the ordered solid solution Ti_3Al (α_2 phase). This α_2 phase produces a strengthening of the α-matrix, and a decrease of the parent alloy ductility [11].

All the bonded specimens shown superficial enrichment in α-phase, because of the high solubility of oxygen in α-titanium. The high hardness of the α-phase makes the following machining process difficult. The elimination of this α-phase layer is necessary to apply a chemical milling to the bonded pieces [12].

The diffusion bonds exhibited a very high microstructural quality for all bonding times tested. The original bond interface disappeared and no voids were observed for the bonding conditions used. Complete recrystallization was observed through the bond interface. However, most of the bonded specimens showed a lack of union at their edges. This effect was due to the microscopical pressure gradient in this part of the bond interface, and it explains the presence of a very high proportion of cavities at the edges of some specimens which decrease in number and size towards the centre of the specimens (Fig. 8). The evolution of the interfacial voids confirmed the theoretical model proposed by Guo and Ridley [13] based on the shrinkage of lenticular voids (Fig. 9a and b) by diffusion and creep mechanisms.

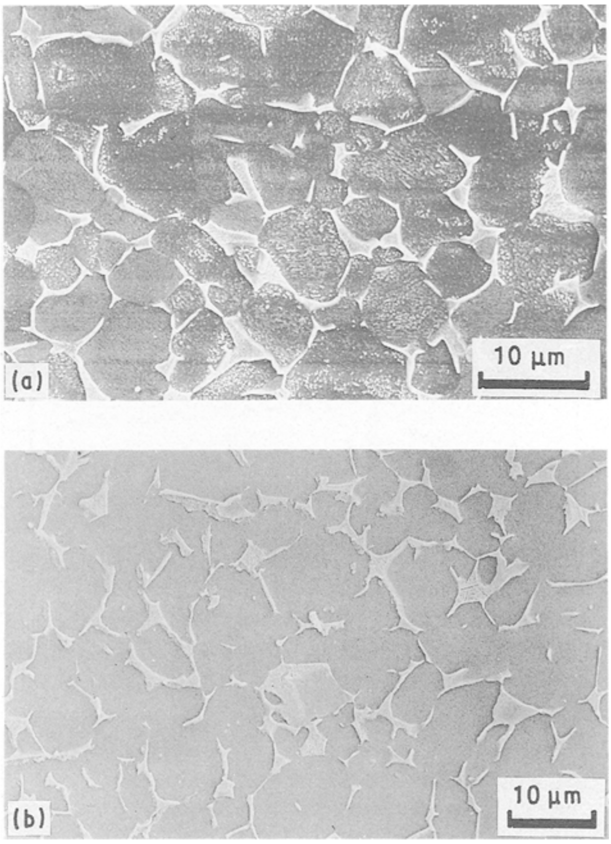

Figure 6 Diffusion bonds in Ti–6Al–4V sheet at 850 °C and 4 MPa. Bonding time: (a) 60 min, (b) 120 min.

3.2. Tensile tests

The results of tensile tests on cylindrical bonded pieces showed high-strength bonds could be obtained for Ti–6Al–4V joints when a bonding temperature of 850 °C was used. The maximum tensile strength of 960 MPa (approximately 90% parent alloy tensile strength) was reached for bonding conditions of 850 °C, 4 MPa and 90 min, using ground surfaces

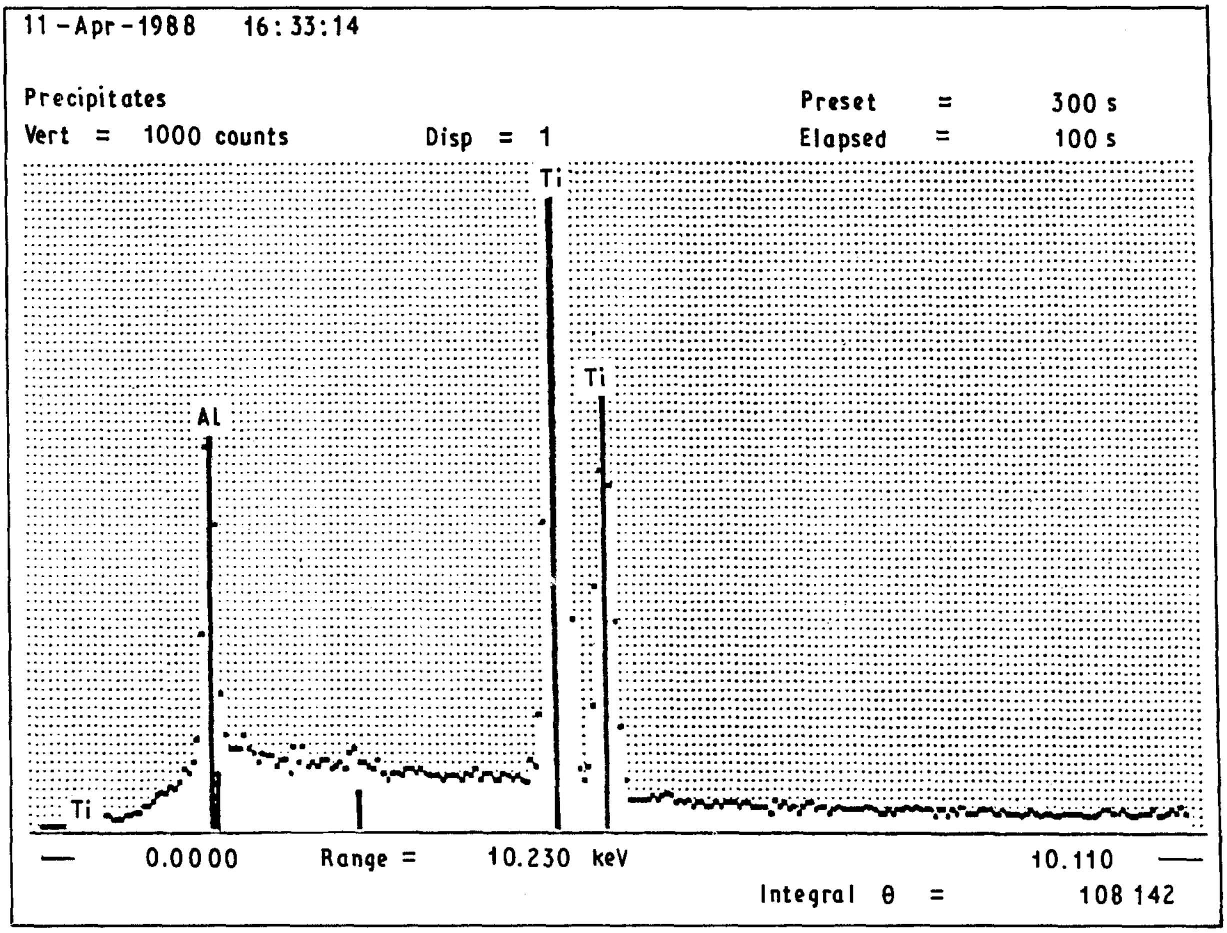

Figure 7 EDS microanalysis of Ti_3Al precipitates.

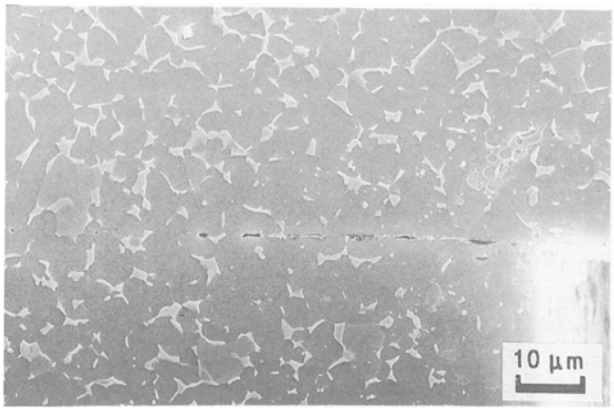

Figure 8 Edge effect in diffusion bonded Ti–6Al–4V sheet.

(0.1 µm). Increasing the bonding pressure from 2 MPa to 4 MPa had little effect on the tensile strength, but the increase of bonding time from 90 min to 120 min produced a light softening of the bonded alloy. It was related with the dissolution of α_2 precipitates and the grain growth of α-matrix observed for longer bonding times. These results are graphically shown in Fig. 10a and b.

It is interesting to note the effect on the mechanical properties of the surface roughness of the bonded areas. Although the tensile strength of turned specimens is only slightly lower than the value obtained for

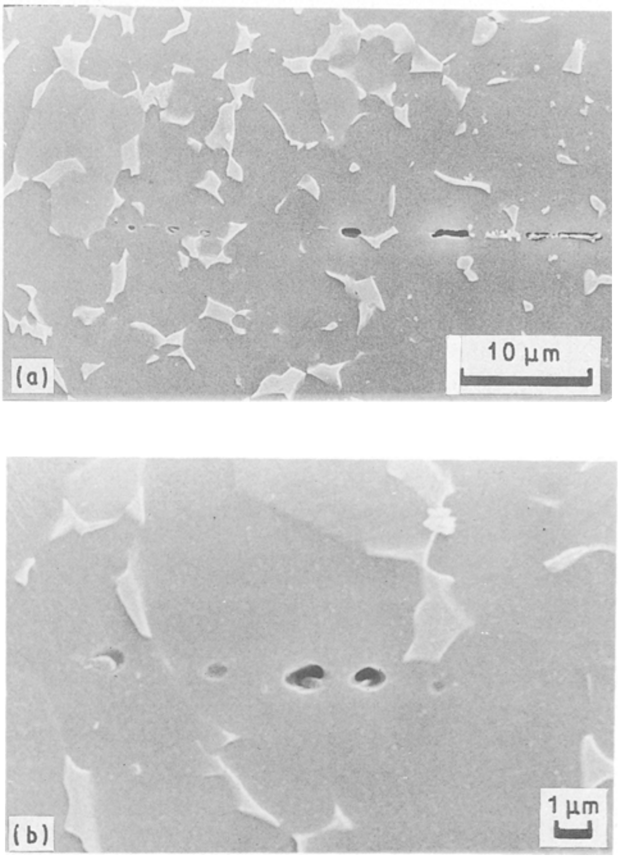

Figure 9 (a) Interfacial voids close to the specimen edge; (b) detail of lenticular voids.

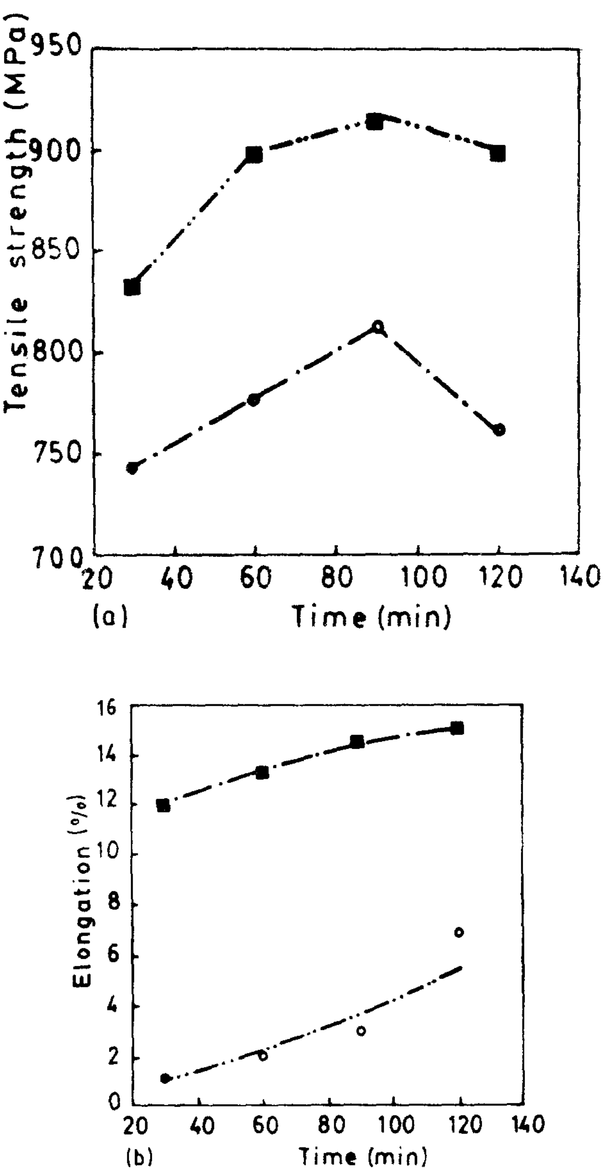

Figure 10 Effect of the bonding time and surface roughness on the mechanical tensile properties of diffusion bonded Ti–6Al–4V cylinder: (a) tensile strength; (b) elongation. (■) Ground surface, (○) turned surface.

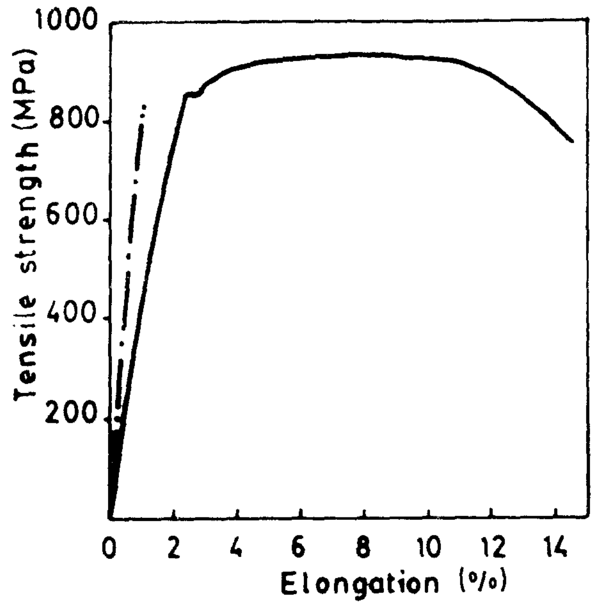

Figure 11 Tensile strength versus elongation curves for different surface microroughnesses (——) ground; (–·–) turned specimens.

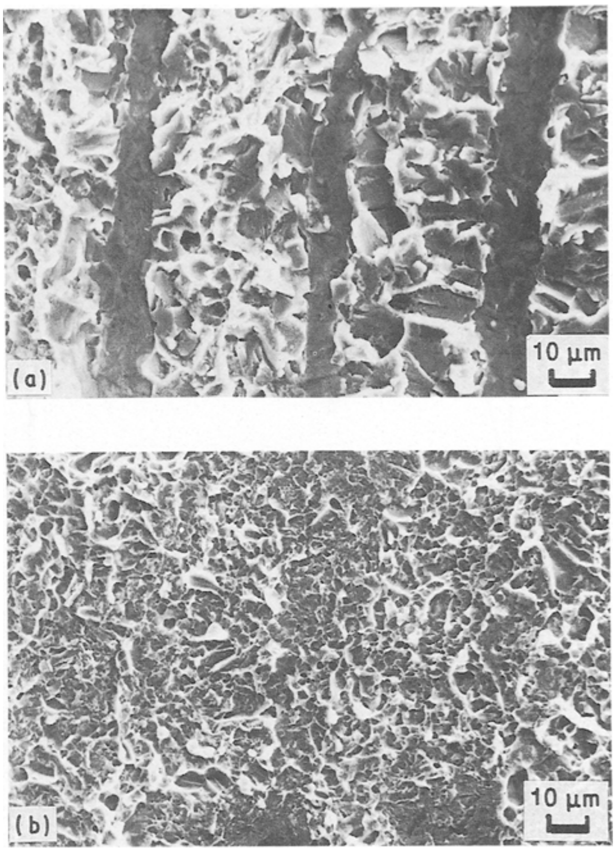

Figure 12 Fracture surfaces of diffusion bonded cylinders: (a) high-strength bond; (b) low-strength bond with unbonded areas.

ground specimens (Fig. 10a), a high dependence between elongation and surface finish was observed (Fig. 10b). While joints with a surface roughness of 0.1 μm failed through the parent alloy with a great elongation, similar to in the as-received material (≈ 15%), joints bonded with higher roughness failed through the bond interface with virtually no necking. The load–elongation curve exhibited by a high-quality bond during a tensile test is compared with the curve obtained for a high-roughness bonded specimen in Fig. 11. In the first case a significant plastic deformation has occurred in the bond region prior to tensile fracture. This plasticity was absent in poor bonds and these interfaces were subsequently found to have unbonded areas. The fractographic study of both kind of tested joint showed this fact (Fig. 12a and b). This study also made it possible to determine the evolution of the bonded area proportion and its dependence on the bonding conditions (Fig. 13).

3.3. Lap shear tests

The results of lap shear tests on diffusion bonds of Ti–6Al–4V were strongly dependent on test piece configuration. To obtain precise results it is necessary to use testing jigs, when working without these, large bending and peel stresses are produced in the specimen edges during testing which produce a decrease in the measured shear strength. Therefore, the test pieces were clamped to rigid supports, reducing the scatter in the failure load values obtained, and increasing the calculated medium strength.

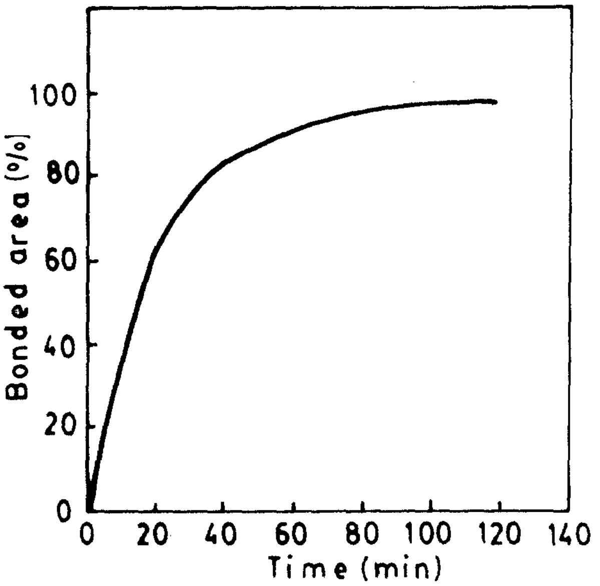

Figure 13 Evolution of the bonded area with bonding time for turned specimens.

However, to measure the true shear strength, using single overlap specimens, it is necessary to work with small overlap lengths, (approximately 1.6 times the sheet thickness). It is necessary to produce failure through the bond interface and not through the parent materials. Nevertheless, working with such small lengths introduces important errors in measuring the overlap area. For these reasons, when standard overlap pieces were tested, tensile failure generally occurred through the parent alloy, in the zone close to the overlap edges. The shear strength of these specimens, calculated as the ratio between the parent material tensile load and the overlap area, was always close to 350 MPa and these values did not show a clear dependence on the bonding time or pressure (Fig. 14). This is because the values were lower than the true shear strength. In these test pieces failure always initiated at the edge of the overlap area, with the initial crack growing through the bond interface. However, when the crack tip hit a high-integrity bond, it progressed through the parent sheet, which had a reduced thickness because of the plastic deformation during bonding.

To avoid the previous effects, a new type of shear test piece was designed for diffusion bonding sheets. This piece avoids failure through the parent alloy by increasing its width in this zone. It is a non-parallel shear test specimen with a variable width from 5–10 mm, being the minimum value in the middle of the overlap zone. These types of specimen induced failure by shear through the bond interface, and only when the thickness reduction of the parent sheet by plastic deformation is higher than 10%, could failure occur in the bulk alloy.

Maximum shear strengths of 550 MPa, close to the parent material shear strength for similar thermal conditions, were measured with the second type of test piece. These values were obtained for joints bonded with pressures of 4 MPa using bonding times longer than 90 min (Fig. 14a). The decrease in the bonding pressure (2 MPa) produced an important reduction of the shear strength, so that it is necessary to use bonding times longer than 120 min to obtain strengths close to 470 MPa (Fig. 14b).

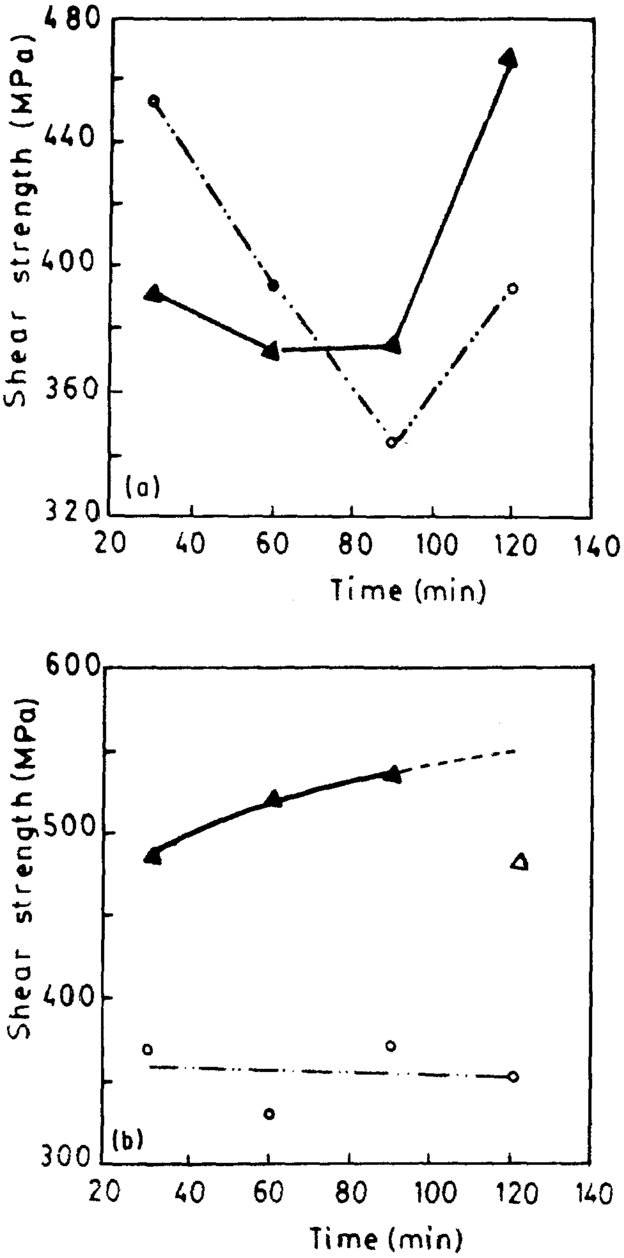

Figure 14 Effect of bonding time on shear strength of overlap bonds: (a) $P = 4$ MPa; (b) $P = 2$ MPa. (▲) standard test piece, (○) variable section test piece (open symbols indicate failure in the parent sheet).

Shear fractures for diffusion bonded joints of Ti–6Al–4V with high shear strength are shown in Fig. 15. Scanning electron micrographs at low magnification show that failure occurred by shear in the overlap zone (Fig. 15a). Although failure was initiated in the bond interface (A), it progresses by shear through the parent sheet (B). The failure mode in these zones is a ductile fracture which reveals a heavily dimpled surface, being the voids elongated in the shear direction (Fig. 15b).

3.4. Peel tests

The true peel strength of a diffusion bonded joint is very difficult to evaluate because of the strong dependence between this value and other design parameters,

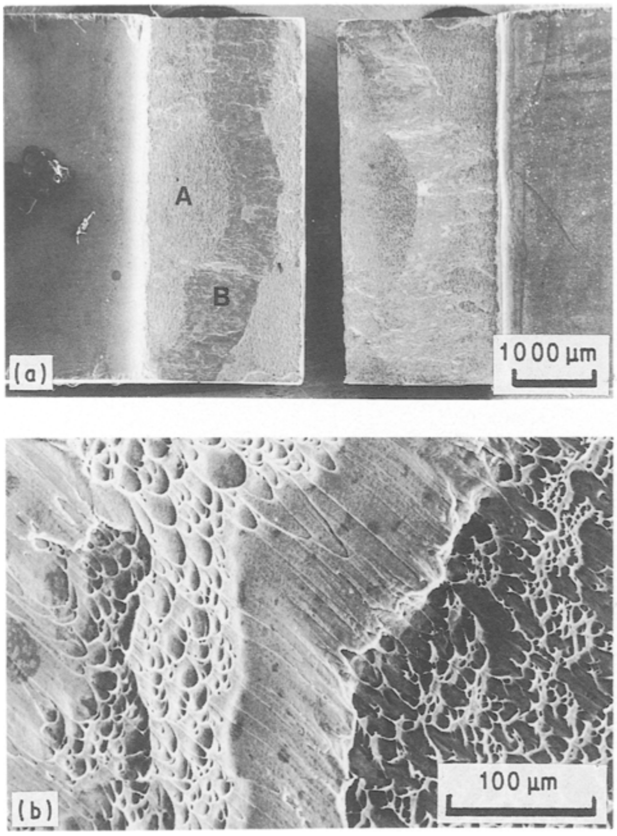

Figure 15 (a) Shear fracture surface of Ti–6Al–4V diffusion joint bonded at 850 °C, 4 MPa and 90 min. (b) Detail of ductile failure zone.

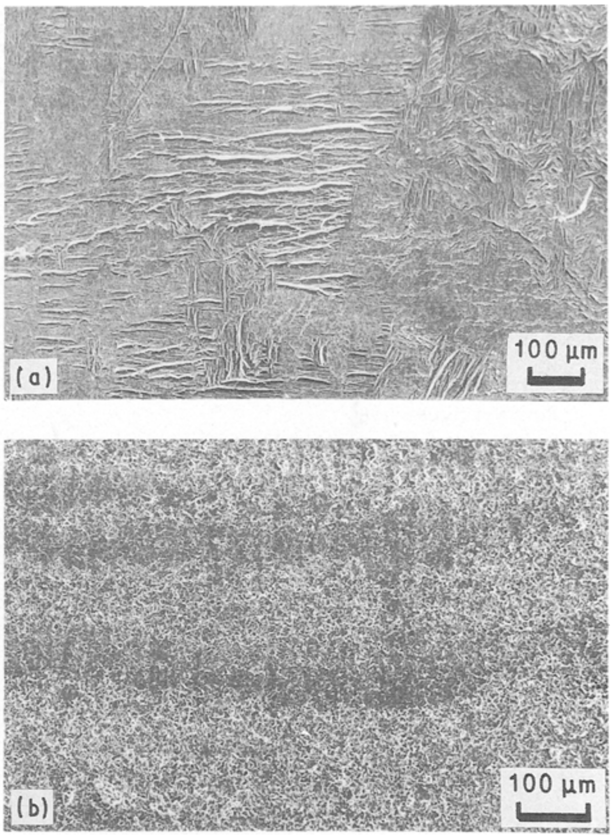

Figure 17 Fracture surfaces of diffusion bonded joint in a peel test. (a) $T_\mathrm{b} = 850\,^\circ\mathrm{C}$, $P_\mathrm{b} = 4$ MPa, $t_\mathrm{b} = 120$ min. (b) $T_\mathrm{b} = 850\,^\circ\mathrm{C}$, $P_\mathrm{b} = 2$ MPa, $t_\mathrm{b} = 30$ min.

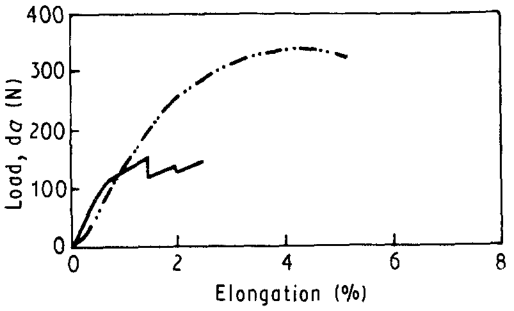

Figure 16 Load–elongation curve for peel test in bonded specimens. Bonding conditions: (a) (–··–) $T_\mathrm{b} = 850\,^\circ\mathrm{C}$, $P_\mathrm{b} = 4$ MPa, $t_\mathrm{b} = 120$ min.

such as the test piece configuration and testing technique. However, it was interesting to obtain some information on peel stress behaviour of Ti–6Al–4V diffusion bonds. Peel strengths obtained were in the range 90–200 N mm^{-1}.

Specimens with low peel strength (< 100 N mm^{-1}) presented a broken load versus deformation curve for the peel test, as shown in (b) bonding conditions in Fig. 16. This is because the peel crack appeared to grow in a series of jumps which induced a drop in the peel stress. Fractographic SEM studies of the joints showed this crack propagation: areas with plastic deformation alternated with large zones of low-quality joint, where the crack growth rate was very high (Fig. 17a).

Joints with high peel strength (≈ 200 N mm^{-1}) showed peel test curves where the load remained almost constant during crack growth (Fig. 16, (a) bonding conditions. Failure surfaces of these specimens show large plastic areas, developed during the slow crack growth period (Fig. 17b). These joints were obtained for bonding times longer than 60 min.

4. Conclusions

High-quality diffusion bonded joints can be produced for Ti–6Al–4V alloy using a bonding temperature of 850 °C, which is lower than those applied in the superplastic forming process for this alloy.

The best mechanical properties were obtained using bonding pressures of 4 MPa and times in the range 90–120 min. Under these conditions, the grain size of the parent alloy was kept in the range required for superplastic forming.

Both the tensile and shear strengths obtained for these bonding conditions were very close to the bulk material strength under the same bonding conditions. Failure in both kinds of test occurred through the parent material with a slight reduction in relation to the bulk strength in the as-received condition. This was because the thermal treatment applied during bonding produced a slight grain growth.

Despite the peel strength depending on a combination of factors, the results suggest that high peel strength can be obtained at 850 °C.

References

1. J. PILLING, D. W. LIVESEY, J. B. HAWKYARD and N. RIDLEY, *Metals Sci.* **18**(3) (1984) 117–122.

2. C. HAMMOND, "Superplastic Forming of Structural Alloys", Conference Proceedings, Warrendale, PA, 21–24 June 1982, edited by N. E. Paton and C. H. Hamilton (AIME, New York, 1982) pp. 131–145.
3. C. H. HAMILTON, "Superplastic Forming", Conference Proceedings, Los Angeles, CA, 22 March 1984, edited by S. P. Agrawal (American Society for Metals, Metals Park, OH, 1984) pp. 13–22.
4. I. E. BOTTOMLEY and B. GINTY, "Diffusion Bonding 2", Proceedings of the 2nd International Conference on Diffusion Bonding, Cranfield, UK, 28–29 March 1990, edited by D. J. Stephenson (Elsevier Applied Science) pp. 232–241.
5. B. KELLOCK, *Machinery Prod. Engng* **143**(3) (1985) 18–20.
6. M. OHSUMI, M. SHIMIZU, A. TAKAKASHI and T. TSUZUKU, Mitsubishi Heavy Industries Ltd, Technical Review, February 1984.
7. A. ARIELI and R. B. VASTAVA, "Superplastic Forming", Conference Proceedings, Los Angeles, CA, 22 March 1984, edited by S. P. Agrawal (American Society for Metals, Metals Park, OH, 1984) pp. 70–75.
8. R. SAWLE, "Superplastic Forming of Structural Alloys", Conference Proceedings, Warrendale, PA, 21–24 June 1982, edited by N. E. Paton and C. H. Hamilton (Metallurgical Society of AIME, New York, 1982) pp. 301–317
9. L. ASCANI, "Innovation in Materials and their Applications", Conference Proceedings, Chicago, 25–27 October 1977 (American Society for Metals, Metals Park, OH) pp. 19–36.
10. O. D. SHERBY and O. A. RUANO, "Superplastic Forming of Structural Alloys", Conference Proceedings, Warrendale, PA, 21–24 June 1982, edited by N. E. Paton and C. H. Hamilton (Metallurgical Society of AIME, New York, 1982) pp. 241–254.
11. ASM Committee on Titanium and Titanium Alloys, "Metals Handbook", Vol. 3, 9th Edn (American Society for Metals, Metals Park, OH, 1980) pp. 763–74.
12. W. T. HARRIS, "Chemical Milling" (Clarendon Press, Oxford, 1976).
13. Z. X. GUO and N. RIDLEY, *Mater. Sci. Technol.* **3** (1987) 945.

Received 18 July 1990
and accepted 6 February 1991