

Superplasticity and Superplastic Diffusion Bonding of a Fine-Grained TiAl Alloy

Hanliang Zhu¹, Bing Zhao², Zhiqiang Li² and Kouichi Maruyama¹

¹Graduate School of Environmental Studies, Tohoku University, 02 Aobayama, Aobaku, Sendai 980-8579, Japan

²Beijing Aeronautical Manufacturing Technology Research Institute, Beijing 100024, P. R. China

Superplasticity and superplastic diffusion bonding in a TiAl alloy with a fine-grained duplex microstructure have been investigated in order to fabricate TiAl alloy products using a combination process of superplastic forming with diffusion bonding. Superplastic tensile tests were carried out at temperatures ranging from 1000 to 1100°C, and at strain rates ranging from 10^{-5} to 10^{-3} s⁻¹. A low superplastic flow stress of less than 25 MPa was observed at 1100°C and at a strain rate of 8.3×10^{-5} s⁻¹. Under this condition, a tensile elongation of 300% and a strain rate sensitivity coefficient of over 0.5 were obtained. Furthermore, superplastic diffusion bonding under a low pressure of 10 MPa was conducted. Defect-free bonds were achieved at 1100°C for holding 1 h. It is suggested that the low superplastic flow stress at 1100°C allows a significant plastic flow between the two contacted surfaces, resulting in a full contact of the surfaces, which is necessary for the sound bonds. Finally, a two-sheet part of spherical dome was successfully produced from this alloy.

(Received May 24, 2005; Accepted August 2, 2005; Published October 15, 2005)

Keywords: TiAl alloy, microstructure, superplasticity, diffusion bonding

1. Introduction

TiAl-based alloys are promising candidates for application in advanced aerospace programs because of their attractive high temperature strength, low density, good creep and oxidation resistance.¹⁻⁵⁾ However, the major drawbacks of these materials are their inadequate ambient ductility and formability, which restrict their applications. These shortcomings make the conventional manufacturing operations such as rolling, forging, or drawing to be difficult. As a result, superplastic forming combined with diffusion bonding (SPF/DB) appears to be a promising route for fabricating structure component of TiAl-based alloys.⁶⁾ The superplastic behavior and deformation mechanisms of various alloys have been extensively studied,⁷⁻¹³⁾ and the superplastic behavior of TiAl-based alloys has been demonstrated in the past few years.¹⁴⁻¹⁷⁾ Significant superplastic flow requires a fine, randomly distributed equiaxed grain microstructure, with grain size in the range of 1–10 μm.¹⁸⁾ Several TiAl-based alloys have been thermomechanically processed to develop a fine-grained duplex microstructure that exhibits superplastic behavior at temperatures ranging from 800 to 1310°C.^{15-17,19)} However, limited work has been reported on studies of superplastic deformation and diffusion bonding behavior of fine-grained duplex TiAl-based alloys taking account of the industrial conditions for the SPF/DB process.

The SPF/DB process for applications to advanced structure in conventional Ti alloys, *e.g.* Ti–6Al–4V, is approaching the stage of production practice after years of development and demonstration.²⁰⁾ Ti–6Al–4V alloy exhibits good superplasticity and excellent diffusion bondability at same temperatures ranging from 880 to 940°C. By selectively bonding only specific areas of two or more thin sheets of alloy and then internally expanding the resulting sandwich into a mold, using a low gas pressure, components with a predefined external shape and an internal cellular structure can be fabricated. Therefore, the SPF/DB process is a suitable technology for fabricating parts with complex shapes for Ti–6Al–4V alloy. However, the SPF/DB process through gas-pressure forming technology for conventional Ti alloys is

limited for application in TiAl-based alloys since the temperature and pressure for SPF and DB of TiAl-based alloys increase greatly. The SPF/DB process for TiAl-based alloys has not yet been developed for producing intricate components.

In this study, a fine-grained duplex microstructure of Ti–48Al–2Cr–2Nb alloy was prepared from a coarse-grained fully lamellar microstructure by thermomechanical process. Superplastic deformation behavior derived from this microstructure and diffusion bonding behavior for this material were investigated. During diffusion bonding process, a low pressure was applied taking account of the maximum temperature of 1100°C and the maximum pressure of about 10 MPa of industrial equipment and gas-pressure forming technology. The mechanism of diffusion bonding under a low pressure was discussed. On the basis of these studies, a primary two-sheet workpiece of spherical dome was fabricated in order to develop the SPF/DB process for TiAl-based alloys.

2. Experimental Procedure

TiAl-based alloy with a nominal composition of Ti–48Al–2Cr–2Nb was prepared by the water-cooling crucible vacuum induction melting technique.²¹⁻²³⁾ The ingot was subjected to a hot isostatic pressing (HIP) at 1250°C and 150 MPa for 4 h to remove cast porosity. For producing a fine-grained microstructure, the HIPed material was then subjected to a multi-step thermomechanical process involving multiple steps of forging. Tensile specimens with a gauge section of 15 mm × 6 mm × 2 mm and diffusion bonding specimens with a size of 20 mm × 20 mm × 2 mm were cut from the heat-treated materials by electrical discharge machining.

The superplastic tensile tests were conducted in air at temperatures ranging from 1000 to 1100°C and strain rates from 1×10^{-5} to 1×10^{-3} s⁻¹. For the diffusion bonding trials, the surfaces of the specimens have been grounded with 600 to 1000 grade SiC paper. The bonding temperature and time varied between 900 to 1100°C and 0.5 to 2 h, respectively. The diffusion bonding pressure of 10 MPa was

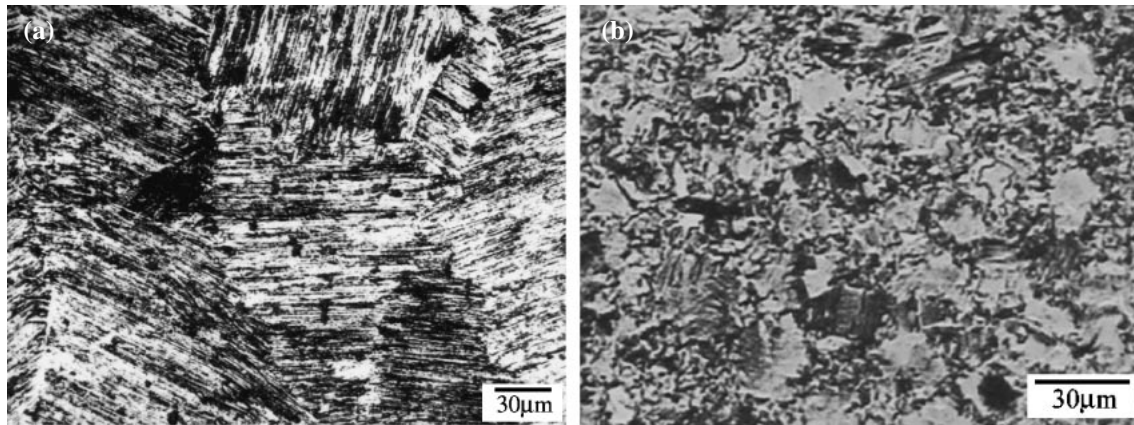


Fig. 1 Microstructure of Ti-48Al-2Cr-2Nb alloy: (a) as-cast fully lamellar microstructure, (b) fine-grained duplex microstructure after thermomechanical process.

used and all bonds were produced under a vacuum of lower than 6.6×10^{-3} Pa. Superplastic forming and diffusion bonding for a two-sheet workpiece was carried out in a one-hundred ton press equipped with vacuum furnace.

Specimens for optical metallography were etched in a solution of 10 ml HF + 5 ml HNO₃ + 85 ml H₂O. The fracture surfaces after superplastic deformation and the diffusion bonding interfaces etched in the solution described above were examined using a scanning electron microscope (SEM).

3. Results and Discussion

3.1 Initial microstructure

The as-cast and thermomechanically processed microstructures of the Ti-48Al-2Cr-2Nb alloy are shown in Fig. 1. The as-cast TiAl alloy has a fully lamellar microstructure consisting of lamellar α_2 and γ plates. The lamellar colonies have a size of about 1 mm. After hot isostatic pressing and multi-step thermomechanical processing, the microstructure exhibits a duplex morphology consisting of equiaxed γ grains, spheroidized α_2 phase and fine lamellar colonies [Fig. 1(b)]. The grain size appears to be much finer with an average value of about 4 μ m. It is speculated that the coarse lamellar colonies in the cast alloy were broken up and the microstructure was significantly refined during the thermomechanical process. This Ti-48Al-2Cr-2Nb alloy with fine-grained microstructure was used in the study on superplastic deformation and diffusion bonding behavior of TiAl alloy.

3.2 Superplastic deformation behavior of the fine-grained microstructure

Figure 2 shows plots of true stress and true strain for tensile specimens with the fine-grained microstructure tested at a strain rate of $8.3 \times 10^{-5} \text{ s}^{-1}$ and temperatures ranging from 1000 to 1100°C. In all cases the true stress-strain curves exhibit strain hardening to peak stresses, and then followed by strain softening until rupture occurs. From Fig. 2, it is also found that the true strain corresponding to the peak stress increases with an increase of temperature. The peak flow stress decreases greatly with an increase of temperature. It is noticeable that the flow stress for 1100°C is lower than

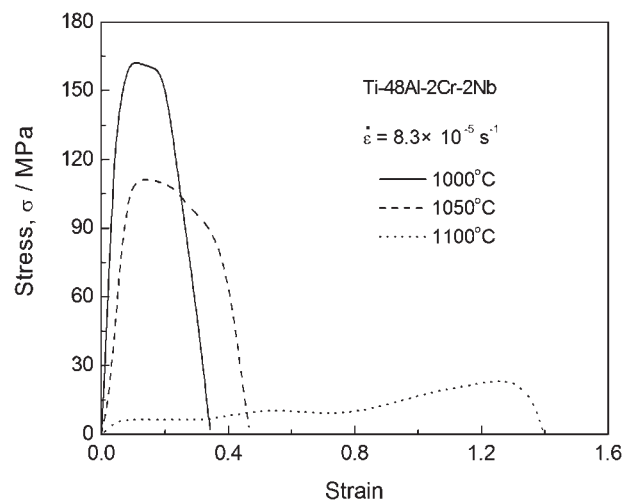


Fig. 2 True stress-true strain curves at different temperatures for the fine-grained Ti-48Al-2Cr-2Nb alloy.

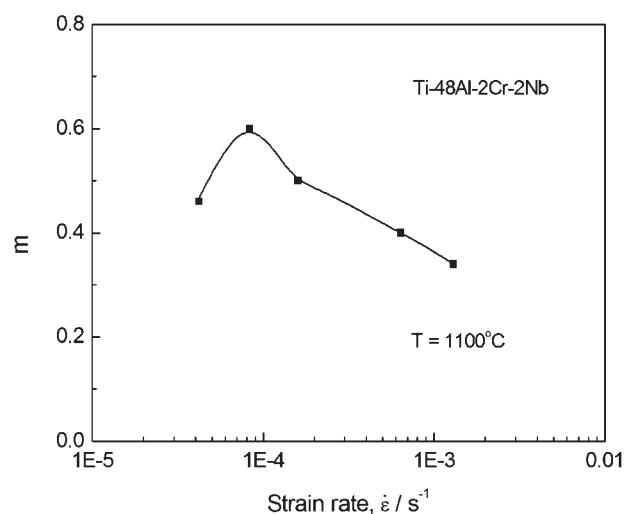


Fig. 3 Effect of strain rate on the value of m for the fine-grained Ti-48Al-2Cr-2Nb alloy.

10 MPa in the early part of the curve and a peak stress is lower than 25 MPa, which is close to a flow stress of 5–20 MPa for Ti-47Al-4.8X (Nb, Cr, Si, Mn) alloys with a

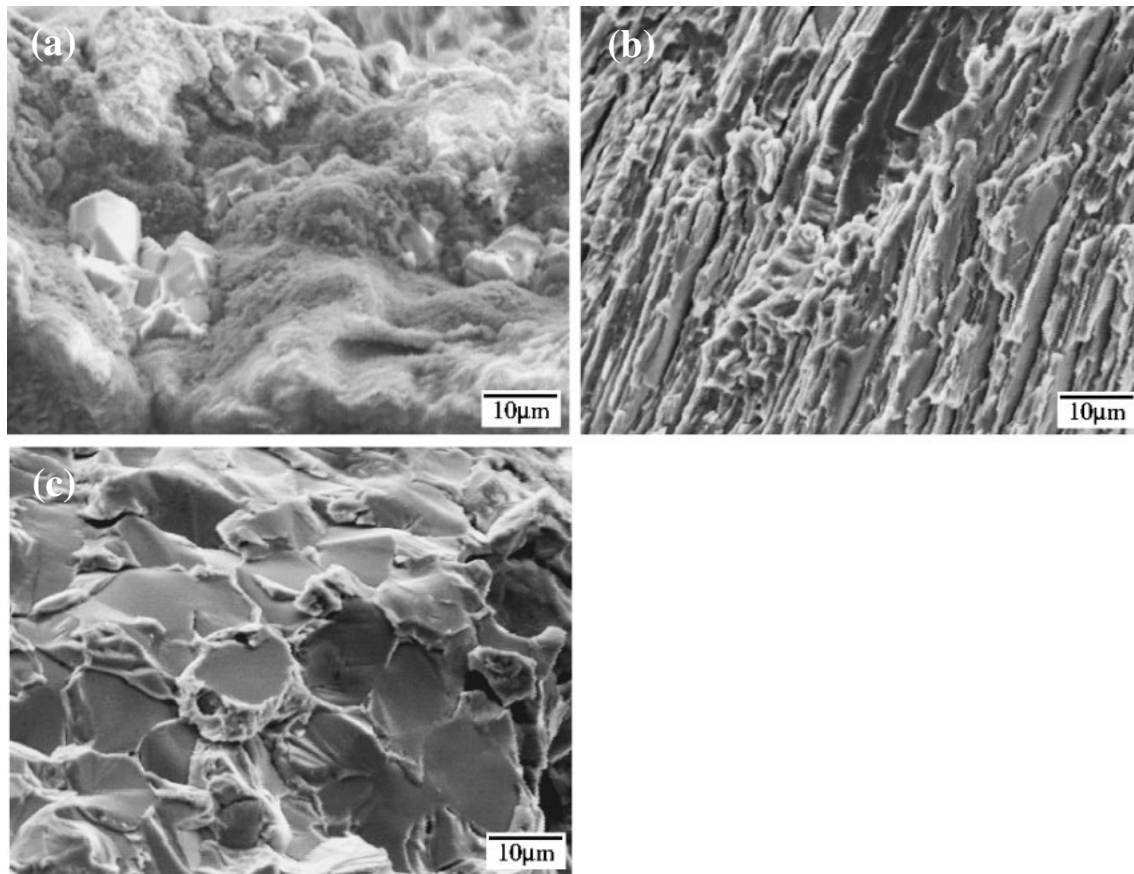


Fig. 4 Fracture surfaces of samples tested at different temperatures: (a) 1000°C, (b) 1050°C and (c) 1100°C.

grain size of 15–20 μm at temperatures ranging from 1250 to 1310°C.²⁴⁾ The flow stress is much lower than a flow stress of 60–300 MPa reported for most superplastic TiAl alloys.²⁵⁾ The tensile elongation reaches about 300% at 1100°C and at a strain rate of $8.3 \times 10^{-5} \text{ s}^{-1}$.

The strain rate sensitivity coefficient (m) versus strain rate of the material was determined at 1100°C (Fig. 3). All the m values at 1100°C and strain rates ranging from 1×10^{-5} to $1 \times 10^{-3} \text{ s}^{-1}$ are higher than 0.33, which is a critical value for superplastic deformation.¹⁴⁾ At a strain rate of $8.3 \times 10^{-5} \text{ s}^{-1}$, the value of m is as high as 0.6, which is higher than the typical value of 0.5 for many fine-grained superplastic alloys.^{18,26)} These results indicate that the fine-grained TiAl alloy exhibits a good superplasticity, which gives sufficient ductility to manufacture this alloy into intricate sheet components.²⁷⁾

Figure 4 shows fracture surfaces of samples tested at different temperatures. Fracture occurs predominantly along grain boundaries at all temperatures. However, transgranular fracture mode also occurs at low temperatures [Figs. 4(a), (b)]. Lamellar delamination within the lamellar colonies is observed at 1050°C [Fig. 4(b)]. With increasing deformation temperature, the fraction of intergranular cracks and cavities increases. Fig. 4(c) clearly shows that cavities nucleate at grain boundaries at 1100°C and the propagation of the intergranular cavities results in fracture of the sample.

It has been proposed that the rate-controlling deformation mechanism is grain boundary sliding (GBS) accommodated by slip controlled by lattice diffusion in superplastic TiAl

alloys.^{19,28)} The stress exponent (n) of the investigated alloy is approximately 2 ($n = 1/m$) at 1100°C and low strain rates, indicating that GBS is also the dominant deformation process in the present fine-grained TiAl alloy. GBS raises the stress in the grain and causes stress concentration at the grain boundaries,²⁹⁾ resulting in nucleation of cavities at the grain boundaries. However, relatively coarse lamellar colonies in the duplex microstructure should restrict GBS as suggested in Ref. 30, leading to lamellar delamination in the lamellar colonies and transgranular fracture at low temperature [Fig. 2(a), (b)]. With increasing deformation temperature, the restriction effect of the coarse lamellar colonies on GBS decreases greatly, resulting in a low flow stress and intergranular fracture at 1100°C [Fig. 2(c)]. Therefore, the fine grain size is suggested to be responsible for the low flow stress and good superplasticity at 1100°C for the duplex microstructure. From Fig. 4(c), it is found that the grain size is still less than 10 μm after the superplastic deformation tests.

3.3 Superplastic diffusion bonding behavior of the fine-grained microstructure under a low pressure

As mentioned earlier, for the gas-pressure forming to make intricate parts from TiAl alloys, the maximum pressure was limited to the order of 10 MPa due to the limitation of the equipment and the technology. Therefore, in this study, the diffusion bonding process was carried out under a pressure of 10 MPa, which is close to a bonding pressure of 5–20 MPa for a fine-grained TiAl sheet,³¹⁾ but much lower than a bonding

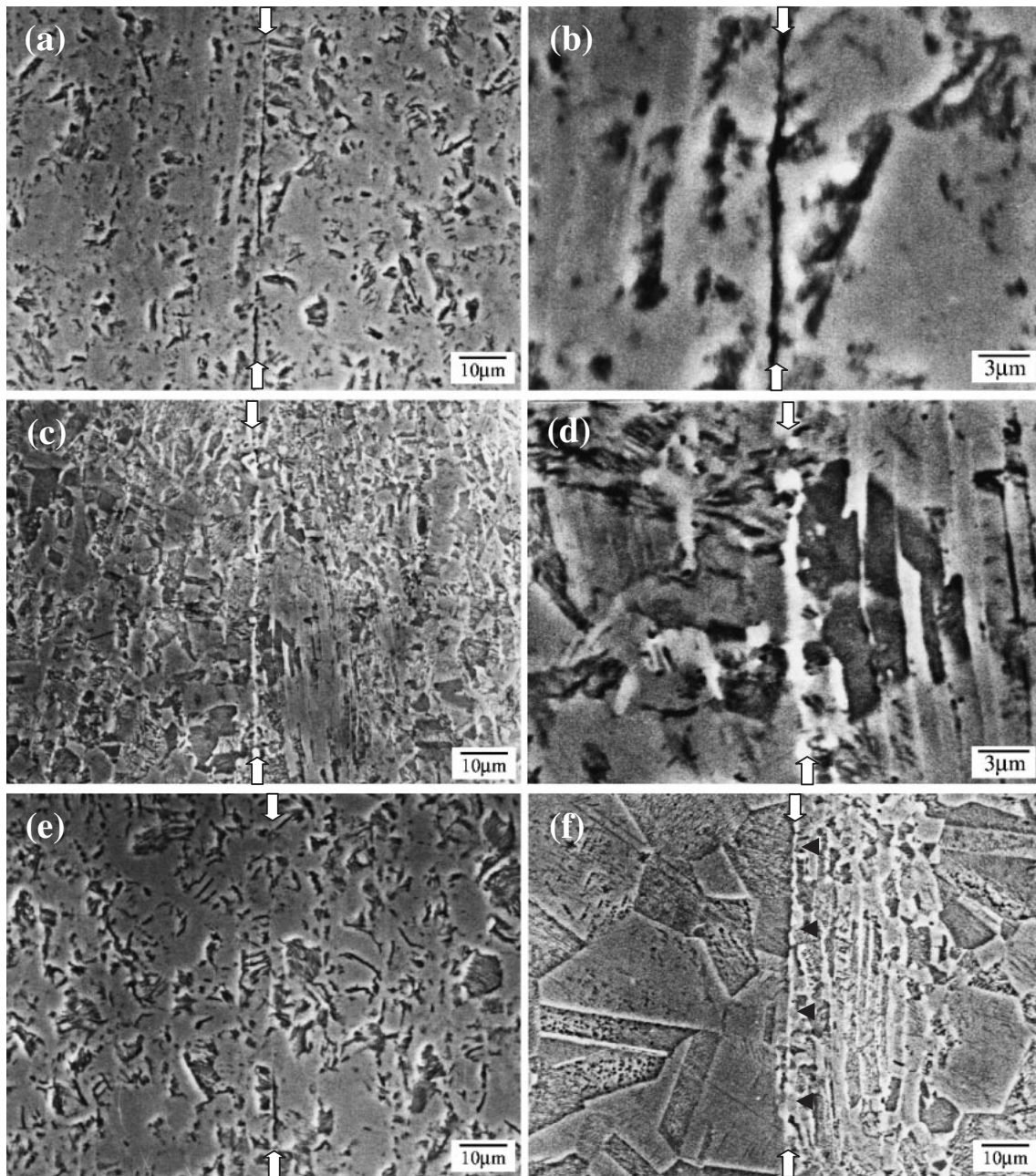


Fig. 5 SEM micrographs of TiAl bonds produced at (a) and (b) 900°C/1 h, (c) and (d) 1000°C/1 h, (e) 1000°C/2 h and (f) 1100°C/1 h. White arrows show the bond lines and arrowhead in (f) indicate fine grains.

pressure of 40–120 MPa reported for diffusion bonding of most TiAl alloys.^{32–34} The diffusion bonding trials were carried out under temperature-time range of 900 to 1100°C and 0.5 to 2 h.

The diffusion bonding interfaces are shown in Fig. 5. The bonds produced at 900°C/10 MP for a bonding time of 1 h exhibit unbonded interface [Fig. 5(a)]. At higher magnifications the unbonded areas appear as incompletely interfacial contact, resulting in interfacial voids [Fig. 5(b)]. The bonding ratio is only 18.8% under this condition (Fig. 6). These results indicate that the two surfaces to be joined could not achieve close contact under the low bonding pressure and at the low temperature. With increasing temperature to 1000°C for 1 h, the bonding ratio increases to 96% (Fig. 6), but the bond interface is still visible [Fig. 5(c)]. At higher magni-

fications [Fig. 5(d)] it can be found that the two surfaces to be bonded were brought into full contact, but the interface between the two contact surfaces could not disappear completely. This result indicates that the bonding time was not long enough to allow sufficient diffusion across the bond interface from the mating surfaces. With increasing bonding time to 2 h, the visible bond interface almost disappeared completely [Fig. 5(e)]. From previous study,³¹ the visible bond interface was observed in bonds produced at 1000°C/5 MPa with a bonding time of 5 h for a fine-grained TiAl sheet, and no visible bond interface was found when the bonding time was increased to 8 h. However, the average values of the shear strength calculated from the reported data are 269.5 and 264.8 MPa for the bonding time of 5 and 8 h, respectively. Therefore, the visible bond interface may have

less effect on the mechanical property of the joints. However, the voids in the bond interfaces certainly decrease the mechanical property. At bonding temperature of 1100°C for 1 h, the visible bond interface can also be found, but no void is observed. Moreover, some fine grains with grain size of about 2 μm in the bond interface are observed [Fig. 5(f)], whereas the grains in the matrix materials become evidently coarse under the isostatic diffusion bonding condition. The grain growth of matrix materials with temperature is shown in Fig. 6. From previous studies, it is known that the grain growth will degrade the SPF behavior,^{16,35)} but the coarse grain is advantageous to the creep resistance,^{36–38)} which is one important property for the TiAl alloy parts employed at elevated temperature.

During diffusion bonding process, two clean metallic surfaces are brought into contact at elevated temperature under a pressure. The two surfaces are far from smooth on the atomic scale and consist of asperities and voids in point to point contact (Fig. 7). Furthermore, the contact point between the two surfaces constitutes a small fraction of the total surface area as shown in Fig. 7. On application of pressure the contact areas will expand in order to support that pressure. The voids in the bond interface are filled by two processes: time dependent plastic collapse by superplastic flow³⁹⁾ and diffusive mass transfer, involving grain boundary diffusion and volume diffusion,^{39,40)} from the bond interface to the surface of the voids.⁴¹⁾ After full contact, the bond

interface disappears by subsequent diffusion between the two contact areas (Figs. 5(e) and 7).

From previous study on a fine-grained Ti-based intermetallic compound,⁴²⁾ among different mechanisms for the contacting process, plastic collapse by superplastic flow is a dominant mechanism, especially in the initial stages of the bonding process, and the contributions from grain boundary diffusion and volume diffusion are negligibly small. Furthermore, the superplastic deformation is useful in breaking the surface oxide film of the two surfaces to be joined and leading the surfaces to contact. In the present study, the two metallic surfaces cannot contact fully at the low temperature of 900°C due to a high flow stress of the material compared to the low bonding pressure. With increasing the bonding temperature to 1100°C, much lower flow stress of less than 25 MPa allows a significant flow between the two contacted surfaces under the applied bonding pressure, resulting in a full contact of the surfaces in a short time. The large-scale superplastic deformation drives significantly dynamic recrystallization in this region, as indicated by the presence of fine grains in the bonding interfaces [Fig. 5(f)]. Therefore, good superplasticity of the fine-grained TiAl alloy is capable of diffusion bonding under a low pressure.

On the basis of above studies, the gas-pressure forming technology for the fine-grained TiAl alloy was carried out in a one-hundred ton press equipped with vacuum furnace for conventional titanium alloys. A two-sheet part of spherical dome (Fig. 8) was successfully produced. It is hoped that this technology could be further developed to produce more complex parts for TiAl alloys.

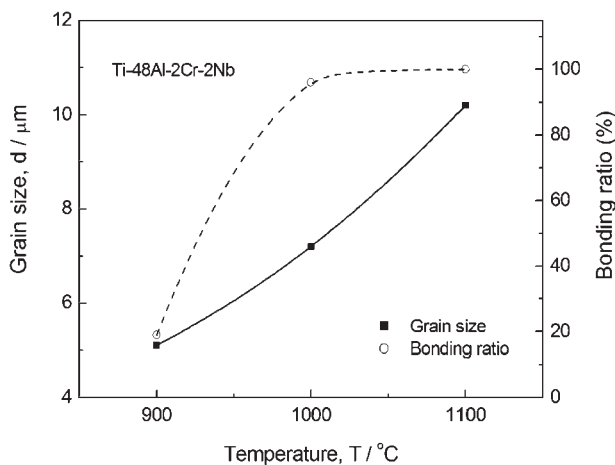


Fig. 6 Effect of bonding temperature on bonding ratio and grain growth under bonding pressure of 10 MPa and bonding duration of 1 h.



Fig. 8 Two-sheet part of spherical dome produced from the fine-grained Ti-48Al-2Cr-2Nb alloy.

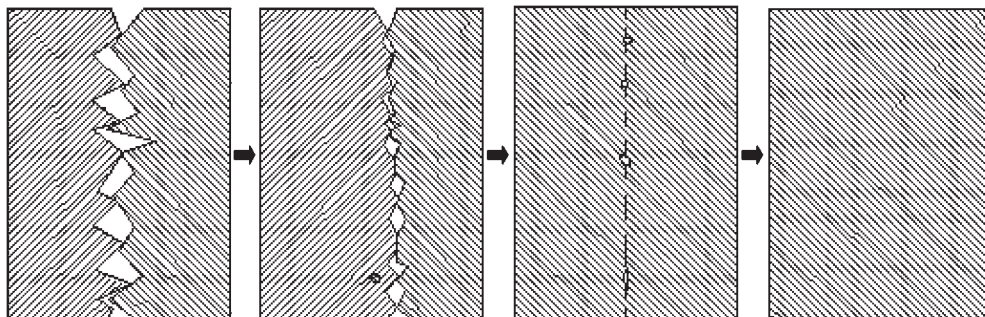


Fig. 7 Schematic illustration of superplastic diffusion bonding process.

4. Summary

The thermomechanically processed Ti-48Al-2Cr-2Nb alloy obtains a duplex microstructure and a fine grain size of about 4 μm . The duplex microstructure exhibits a low flow stress and a good superplasticity at elevated temperature due to the fine grain size. A maximum elongation of 300% was obtained at 1100°C and at a strain rate of $8.3 \times 10^{-5} \text{ s}^{-1}$. Moreover, the strain rate sensitivity coefficient at 1100°C is higher than 0.33. The low superplastic flow stress and good superplasticity of the material at elevated temperature permit a full contact of two clean surfaces under a low bonding pressure of 10 MPa during diffusion bonding process. Sound bonds were achieved at 1100°C for 1 h. On the basis of these studies, a two-sheet part of spherical dome was successfully produced from this alloy in industrial equipment for conventional Ti alloys. It is hoped that this technology could be further developed to produce more complex parts for TiAl alloys.

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