Study of Diffusion Bonding of Fine Grain TC21 Titanium Alloy

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Abstract: The diffusion bonding of TC21 titanium alloy with initial grain size of 2 μm was performed at 780–980 °C for 5–90 min. The microstructure, bonding quality, microhardness and deformation ratio of the joints were analyzed. It is found that the interface bonding ratio can achieve 100% and the deformation ratio can be controlled within 10% when diffusion bonding is performed at 880 °C for 15–30 min. The microhardness of the joints increases with increasing of bonding temperature, but it shows a peak value as the bonding time is prolonged. When the joint is bonded at 880–930 °C, fully equiaxed structures are observed, and with increasing of bonding temperature, the sizes of α and β phases are increased; but when bonding temperature is up to 980 °C, fully lamellar structures are obtained. When the joint is bonded at 880 °C for 5–60 min, the size of α and β phases increases with prolonging of bonding time. However, when the bonding time is prolonged to 90 min, the sizes of α and β phases decrease slightly.

Key words: TC21 titanium alloy; diffusion bonding; microstructure; bonding ratio; microhardness; deformation ratio

TC21 titanium alloy is an attractive type of ($\alpha+\beta$) titanium alloy with high strength, high toughness and high damage-tolerance^[1]. Because of wide application prospect in modern industries, it has received much attention from many materials scientists. The main research efforts have been focused on the microstructure, phase transformation and mechanical properties of TC21 titanium alloy^[2–5], which provide a basic foundation for applications. However, when TC21 titanium alloy is applied to structural parts, joining it to itself or dissimilar materials is inevitable. Therefore, it is an urgent task for materials-joining researchers to investigate the joining technology of TC21 titanium alloy.

Researches on joining of titanium alloys have been focused on fusion welding, brazing and diffusion bonding. Due to the reactive nature of titanium alloys at high temperatures, it is hard to avoid oxidation during welding, and the reactions between titanium alloys and brazing filler metals are difficult to be controlled. Diffusion bonding is an attractive joining technique for joining of similar materials, and has been successfully applied in joining of other kinds of titanium alloys^[6–8]. This should be a reference for the diffusion bonding of TC21 titanium alloy. In the present study, diffusion bonding of fine

grain TC21 titanium alloy was carried out. The microstructures were characterized, and the interfacial bonding quality, deformation ratio and microhardness of the diffusion bonded joints were studied by changing bonding technological parameters.

1 Experimental

The experimental material used in this study was 2 mm thick milled annealed TC21 titanium alloy sheet with average initial grain size of 2 μm . It is a typical fine grain titanium alloy. The nominal chemical composition of the alloy is shown in Table 1 and the initial microstructure is shown in Fig. 1. Prior to diffusion bonding, the sheet was cut into the samples with size of 10 mm×10 mm×2 mm using a spark discharge machine. Subsequently the intended bonding surfaces were ground using SiC papers and cleaned in acetone. Specimens were then assembled, and the initial thickness of the specimens (h_0) was measured with a vernier caliper, which was used to determine the deformation ratio. Diffusion bonding was performed in the vacuum furnace (Centorr-M60) with a vacuum degree of 6.6×10^{-3} Pa, adopting the joining parameters of temperature ranging from 780 to 980 °C, pressure

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Table 1 Chemical composition of fine grain TC21 titanium alloy (wt%)

Elements	Al	Sn	Zr	Mo	Cr	Nb	Si	Fe	C	N	H	O	Ti
Content	6.0-5.5	2.0	2.0	2.5-3.0	1.7	2.0	0.1	≤0.15	≤0.08	≤0.05	≤0.015	≤0.15	Balance

Fig.1 Microstructure of fine grain TC21 titanium alloy: (a) OM metallograph and (b) SEM image

of 10 MPa for 5 min to 90 min. A layer of yttrium oxide powder was coated between the specimens and the pressure bars to avoid bonding of them. After bonding process was completed the specimens were cooled to room temperature before removal from the chamber.

After bonding, the thickness of the bonded specimens (h) was measured in order to calculate the deformation ratio (k): $k = (h_0 - h)/h_0$, where h_0 denotes the thickness before joining, and h the thickness after joining. And then the joints were cross-sectioned, perpendicular to the interfaces, using a spark discharge machine. The cross-sections of these joints were polished to 1 μm diamond finish. They were then etched with a mixture agent of HF (1 mL), HNO₃ (2 mL) and H₂O (47 mL) for 3 to 5 s. Optical Microscope (MPG3) and Scanning Electron Microscopy (S-4700) were used to observe interfacial behaviors. Microhardness was measured on a microhardness tester (HVS-1000) under the test load of 500 g for 10 s. Interfacial bonding quality was evaluated by the interfacial bonding ratio (p), which is the ratio of the total length of the voids along the interface to the total length of the interface.

2 Results and Discussion

2.1 Experimental results

2.1.1 Diffusion bonding quality

To achieve high quality bonding, interface voids should be removed in the joints. Fig.2 shows the interface morphologies of the joints, which were obtained at different bonding temperatures for 30 min. It can be seen that bonding temperature is a primary parameter which plays an important role in the bonding process. When bonded at 830 °C, voids with average size of about 1 μm can be observed in the interface of the joint. When bonded at 880-980 °C, voids disappear completely.

Fig.3 shows the interface morphologies of the joints for different bonding time. From Fig.3, it can be seen that when bonded at 880 °C for 5 min, voids with average size of about 2.4 μm can be found in the interface. As bonding time increases to 15 min and/or more, voids get closed completely.

Fig.2 Interface morphologies of joints bonded at different temperatures ($t=30$ min): (a) 830 °C, (b) 880 °C, (c) 930 °C, and (d) 980 °C **Fig.3** Interface morphologies of joints bonded for different holding time ($T=880$ °C): (a) 5 min, (b) 15 min, (c) 60 min, and (d) 90 min

The evaluated interfacial bonding ratio is shown in Fig.4. From Fig.4a, when bonded at 780 °C for 30 min, the interfacial bonding ratio is 87%. With increasing of bonding temperature, the interfacial bonding ratio increases. This is evidenced by the fact that when bonded at 830 °C, the interfacial bonding ratio is 91%; but when bonded at 880-980 °C, the interfacial bonding ratio achieves 100%. From Fig.4b, with respect to the bonding temperature of 880 °C, when bonded for 5 min, the interfacial bonding ratio is 90.5%. As bonding time is prolonged to 15-90 min, the interfacial bonding ratio reaches 100%.

Fig.4 Interface bonding ratio of joints bonded at different temperatures or for different time: (a) $t = 30$ min and (b) $T = 880$ °C

Fig.5 Microhardness of joints bonded at different temperatures or for different time: (a) $t = 30$ min and (b) $T = 880$ °C

2.1.2 Microhardness

Microhardness of diffusion bonded joints of fine grain TC21 titanium alloy was tested to evaluate the mechanical properties of the joints (shown in Fig.5). From Fig.5a, when bonded at 830 °C, microhardness of the joint is 3480 MPa. As bonding temperature increases to 930 °C and 980 °C, the microhardness is 3700 MPa and 3950 MPa respectively. From Fig.5b, when bonded for 5 min, the microhardness of the joint is 3400 MPa. With the bonding time prolonged within 15-60 min, the microhardness increases and achieves a peak value (3560 MPa) when bonded for 60 min. When bonded for 90 min, the microhardness decreases slightly.

2.1.3 Deformation ratio

Serious deformation of diffusion bonded joint may lead to poor application of the joint. Therefore, deformation ratio usually should be controlled within 10%. Fig.6 shows the deformation ratio of the joint under different bonding conditions. From Fig.6a, the deformation ratio increases with increasing of bonding temperature. When bonded at 930 °C, the deformation ratio comes to 25%. From Fig.6b, as bonding time is prolonged, deformation ratio increases. When bonded for longer than 30 min, the deformation ratio exceeds 10%, especially when bonded for 90 min, it comes to 33%. Consequently, the diffusion bonding of fine grain TC21 titanium alloy should be conducted below 880 °C for less than 30 min.

2.2 Discussion

2.2.1 Microstructure characterization

Fig.6 Deformation ratio of joints bonded at different temperatures or for different time: (a) $t = 30$ min and (b) $T = 880$ °C

Fig.2 indicates that when bonded at a temperature of lower than 930 °C for 30 min, the fully equiaxed structures of the joints can be obtained; while when bonded at 980 °C for 30 min, only the fully lamellar microstructure is observed. The main differences between the equiaxed microstructures of these joints are the size and shape of α and β phases. When bonded at 830 °C, small α and β strips are observed (Fig.2a). As the bonding temperature comes to 880 °C, the sizes of α and β phases increase, and α phase becomes equiaxed (Fig.2b). When bonding temperature increases to 930 °C, more equiaxed α phase is obtained, and α and β phases grow bigger (Fig.2c). Fully lamellar microstructure is composed of fully lamellar α and β phases without any equiaxed primary α phase (Fig. 2d). Meanwhile, large β grains with clear α phase grain boundaries and different orientation colonies, which consist of parallel lamellar α and β phases, have been found (shown in Fig.7)

From Fig.3, it can be seen that fully equiaxed microstructures with different sizes and shapes of α and β phases are obtained when bonded at 880 °C for different bonding time. For a short bonding time (5-15 min), most α and β phases have small size and strip shape (Fig.3a, 3b). As the bonding time increases to 30 min, many α and β phases become bigger and equiaxed (Fig.2b). This tendency will be continued when bonded for 60 min (Fig.3c). But when bonded for 90 min, many small sized β phases emerge, which leads to the decrement of α phase (Fig.3d).

The microstructure evolution of the diffusion bonded joint is mainly determined by the heating process^[9]. When bonded at a temperature lower than 930 °C, the sample is heated to α and β phase coexistence region. In this region, some α phases transform into β phases, while most α phases will remain and grow together with β phases. With the increase of bonding temperature and time, the sizes of grown α and β phases increase. During cooling, these grown α and β phases are left, and they compose the further grown α and β phases together with the α phases transforming from β phases. But when bonded for 90 min, more α phases will be transformed into β phases during heating, and more small sized β phases are left during cooling, which leads to the decrease of the α and β phases.

When bonded at 980 °C, which is higher than the β transus temperature of TC21 titanium alloy, nearly all α phases transform into β phases, and they are mixed with the initial β phases. Meanwhile, big sized grown β grains are obtained during heating. When cooled, α phase first nucleates at the β grain boundaries and continuous α layer is formed, then some α phases grow along the previous α layer; the others grow into the prior β grains, and the colonies composed of parallel α and β lamellar are formed. The colonies continue to grow in the grains, until they meet other colonies different from them. Finally, big sized β grains with α phase grain boundaries and different colonies are obtained. It is indicated that the sizes of colonies increase with the decrease of cooling rate^[10].

Fig.7 Grain morphology of joint bonded at a higher temperature ($T=980$ °C, $t=30$ min): (a) OM morphology and (b) SEM image

2.2.2 Bonding quality and microhardness

The increase of interfacial bonding ratio with the bonding temperature and time is related to the diffusion mechanisms and the diffusion coefficient. The main diffusion bonding mechanisms include surface source mechanism, interface source mechanism and bulk deformation mechanism. The diffusion coefficients of these mechanisms increase with increasing of bonding temperature, which results in fast closure of voids. Because longer time creates a sufficient diffusion opportunity, the interfacial bonding ratio increases as the bonding time is prolonged.

The microhardness of the joint is mainly dependent on the microstructure characteristics. For the fully equiaxed microstructure, the microhardness is mainly dependent on the volume fraction of α phase. Therefore, according to the microstructure characteristic discussed above, the microhardness increases with the increase of bonding temperature, but shows a peak value as bonding time prolonged. For the fully lamellar microstructure, the colonies will lead to a short slip length. This results in increasing of the yield stress and microhardness.

The irreversible deformation of the diffusion bonded joints includes instantaneous plastic deformation and creep deformation. With increasing of bonding temperature, the resistance to deformation of the alloy becomes low, which makes the two kinds of deformation weakened. While prolonging bonding time leads to weakened creep deformation, thus resulting in higher deformation ratio. Therefore, bonding temperature and time should be controlled strictly.

3 Conclusions

1) The defect free diffusion bonded joint of fine grain TC21 titanium alloy (average initial grain size of 2 μm) with a low deformation ratio can be obtained at 880 °C for 15-30 min. With the increase of diffusion bonding temperature and time, the interfacial bonding ratio remains at 100% while the deformation ratio can exceed 10%.

2) For the bonding temperature ranging from 780 to 980 °C, the microhardness of the joint increases with the increase of the bonding temperature. For the bonding time between 5 to 90 min, the microhardness shows a peak value when bonded

for 60 min. This is determined by the microstructure evolution of the joint.

3) When bonded at lower than 930 °C for 30 min, the fully equiaxed structures are observed and the α and β phases grow as increase of bonding temperature. But when bonded at 980 °C, which is higher than the β transus temperature of TC21 titanium alloy, the fully lamellar structures with α phase grain boundaries and different colonies are obtained.

4) When bonded at 880 °C for 5-90 min, the sizes of α and β phases increase with prolonging of bonding time. But when bonded for 90 min, more β phases transforming from α phases are left during cooling, which decreases the sizes of primary α and β phases.

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TC21 钛合金的扩散连接研究

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摘要: 研究并分析 TC21 钛合金扩散连接接头的微观组织、界面结合质量、显微硬度和压缩变形率随连接工艺参数的变化规律。结果表明: 连接温度的提高及连接时间的延长分别导致扩散系数的增大和扩散程度的增加, 同时变形能力增大, 因而界面焊合率提高, 但接头变形率增大; 在 880 °C 的连接温度下, 只要连接时间介于 15~30 min, 界面焊合率就能达到 100%, 而接头变形率可控制在 10% 以内。接头显微硬度随连接温度的升高而明显提高, 但随连接时间的变化存在峰值。对接头微观组织特征的分析表明: 连接温度越高, 两相长大速率越快, 两相尺寸增大; 但当连接温度达到 980 °C 时, 被焊母材处于 β 单相区, 较快的冷却速度下 α 相不能完全析出, 接头微观组织因此变为层片状的两相组织。随着连接时间的延长, 两相尺寸先增大, 当连接时间延长到 90 min 后, 高温下由 α 相转变而来的 β 相增多并被保留下来, 割裂了原有的两相, 使两相尺寸减小。正是接头微观组织的这种变化特征, 决定了显微硬度的变化规律。

关键词: TC21 钛合金; 扩散连接; 微观组织; 焊合率; 显微硬度; 变形率

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