Effect of bonding temperature and holding time on properties of hollow structure diffusion bonded joints of TC4 alloy

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Abstract

The hollow structure commonly used in aerospace field is generally formed by the SPF/DB, but its disadvantages lead to surface groove and complex internal structure difficult to form. In this paper, the method of milling combined with diffusion bonding is used to improve the defect. Diffusion bonding of TC4 titanium alloy hollow structure was carried out at temperature ranging from 800 °C to 900 °C for 10 ~ 30 min. The interfacial bonding ratio, deformation ratio, microstructures, microhardness and mechanical properties of the diffusion bonded joints were investigated. The results show that joints with high bonding quality can be obtained when bonded at 850 °C and 900 °C for 20 ~ 30 min. The joint bonding ratio is more than 92%, the tensile strength is 952 MPa, and the deformation ratio is maintained at about 10%. The microhardness of the TC4 alloy hollow structural joint increases with the increase in bonding temperature, showing a trend of "high in the middle and low on both sides", and it has a peak value (HV472) when bonding time is prolonged up to 30 min. The findings indicate that qualified workpiece can be obtained by the process.

Keywords TC4 titanium alloy · Hollow structure · Diffusion bonding · Microstructure · Mechanical properties

1 Introduction

Titanium alloys are used as structural materials in many engineering fields especially in aerospace and military fields [1–3] due to their excellent properties such as low density, high specific strength, excellent corrosion resistance and heat resistance [1, 4]. TC4 (Ti-6Al-4 V) alloy is the most widely used titanium alloy at present, which accounts for about half of the total amount of titanium alloy [5, 6]. It has a strong comprehensive performance, known as the “flagship alloy” of the titanium alloy industry, and many other titanium alloys can be regarded as its modified [7]. However, TC4 alloy has been recognized as one of the most difficult-to-machine materials [8]. Its low thermal conductivity and high processing cost have been the bottleneck of its development [9–11]. Therefore,

the new technology to solve the problem of titanium alloy processing has become the focus of researches [8]. Diffusion bonding (DB) is a solid-phase joining technique with few defects and high precision [12–14]. In the process, there is no melting and macroscopic plastic deformation of metals, and the bonding is achieved directly by the diffusion of solid phase atoms. From the view of bondability, diffusion bonding enables reliable bonding between various materials and some special structures, especially dissimilar materials and hollow structure [15–17]. Lin et al. [18] through a large number of experiments, put forward the general technical parameters of diffusion bonding of TC4 titanium alloy range: suitable bonding temperature 900–950 °C, bonding pressure 1–5 MPa, holding time 30 ~ 180 minutes. Zhang et al. [19] studied the diffusion bonding of TC4 alloy under small axial pressure

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of 0.5 MPa. The bonding temperature was 860 °C, 890 °C and 920 °C, holding time was 1 h. Tensile test and Charpy impact test were conducted to evaluate the joint strength and toughness. Yuan et al. [20] conducted the diffusion bonding experiment, and the bonding temperature was 775, 800, 850 and 900 °C, bonding time was 5, 10, 15 and 30 min, and the pressure was 10 MPa. The results show that the plastic deformation and creep mechanisms of void closure are dominated in the early stage or at low bonding temperature, with increasing bonding time or temperature, the surface diffusion and interface diffusion mechanisms act as the dominated matter for voids elimination. E. Yakushina et al. [21] studied the influence of ultra-fine-grained (UFG) and coarse-grained (CG) microstructure of Ti-6Al-4 V alloy on the strength of DB joint. The results indicate that the best quality of the samples bonded at 725 °C was obtained for UFG samples, which demonstrated notably higher shear strength for both 2 and 4 h cycles when compared with CG samples. Han et al. [22] studied the SPF/DB of four-layer plate structures by pneumatic forming. The optimum parameters of diffusion bonding were 930 °C, 30 min and 10 MPa.

In aerospace industry, TC4 alloy hollow structure is often used to achieve the purpose of lightweight [23–25]. Hollow structural parts are often used in aircraft panel, cabin door, access cover, missile rudder, engine blade and other parts [26]. The structure is not completely hollow, and the interior has stiffeners, which can meet the requirements of strength and realize lightweight targets [23]. The advantages of diffusion bonding are very suitable for solving the internal bonding problem of the hollow structure. At present, the use of SPF/DB to form the TC4 hollow structure is the mainstream research. It has the advantages of high efficiency and the integrity of forming parts. However, it is easy to produce groove defects on the surface of parts [22, 27–30]. Therefore, in order to overcome the defects, the method of forming TC4 hollow structures by diffusion bonding is studied in this paper. Taking the rudder surface as the object, the structure design and diffusion bonding experiment are studied. The proper process parameters of the bonding joint are obtained and their effects on diffusion bonding forming quality are discussed.

2 Design and experimental

2.1 Structural design

The research object of this paper is the rudder surface, and it is a hollow structure. It has strict requirements for mass and center of mass, as shown in Table 1. Therefore, it is necessary to design the position, width and depth of

Table 1 Design requirements and results of the rudder surface

Requirements	Mass	440~500 g
	Center of mass	Within the – 1~4 mm range of the axle wire
Design results	Milling depth	3.75 mm
	The skin thickness	1.25 mm
	Stiffener width	1 mm
	Mass	474 g
	Center-of-mass coordinate	58.12, 83.69, – 2.84 mm

stiffener so as to meet the requirements and the using strength. Build the rudder surface part model by using 3D modeling software, adjust the width and depth of model to meet the requirements. The design results are shown in Table 1 and Fig. 1.

According to the design result of the rudder surface, the test parts are designed and manufactured (Fig. 2b). The width and depth of the stiffener are consistent with the design of the rudder surface, in order to ensure the reliability of the test results. The size of specimen is $\phi 50 \times 5$ mm, and the width of the diffusion bonding joint is 1 mm (Fig. 2a).

2.2 Experimental

2.2.1 Material and treatment

The material used in the experiment is a rolled and annealed TC4 (Ti–6Al–4 V) titanium alloy, supplied by AVIC Beijing aeronautical manufacturing technology research institute. TC4 alloy is the phase titanium alloy for medium strength, in which the Al element is a stable element of phase structure, and the V element is a stable element of phase structure. Its chemical composition and mechanical properties are shown in Table 2. The microstructure of the material is observed by optical microscope, which consists of bright α and black β phases, as shown in Fig. 3.

Specimen pretreatment should be carried out before the diffusion bonding experiment. The first step is to use abrasive paper to grind, because the surface of specimen is machined by finish turning, the roughness is Ra0.8, so it is smoother. Use 1000–3000# abrasive paper grinding surface in turns, until the macro-surface is bright and no scratches, and then washed with ethyl alcohol to remove impurities. Put the specimen into the ultrasonic cleaner to clean, the cleaning time is 15 min, and finally dry it off. When the specimen is fixed with a cylindrical pin, put it into the graphite mold and ready for diffusion bonding experiment, as shown in Fig. 4a.

Fig. 1 Design result drawing of rudder surface

Fig. 2 Diffusion bonding specimen for diffusion bonding: **a** specimen drawings; **b** actual specimens

Table 2 Chemical composition and mechanical properties of TC4 alloy used in the experiment

Chemical composition of TC4 (wt.%)									Mechanical properties			
Elements	Al	V	Ti	Fe	C	N	H	O	σ_b /MPa	$\sigma_{0.2}$ /MPa	ψ /%	δ /%
Nominal	5.5~6.8	3.5~4.5	Bal.	0.30	0.10	0.05	0.015	0.20	980	≥ 825	25	10

Fig. 3 Microstructure of TC4 alloy used

The equipment used in diffusion bonding experiment is vacuum hot-pressing sintering furnace, as shown in Fig. 4b, the model is zt-40-20y. The maximum pressure of the equipment is 20 T, the ultimate cold vacuum degree is 6.67×10^{-3} Pa, the size of work area is $\phi 160 \times 160$ mm, the internal pressure head diameter is $\phi 85$ mm, the pressure and displacement can be tracked and controlled automatically, and the displacement precision is 0.05 mm. The heating element is graphite carbon tube, its highest heating temperature can reach 2000 °C and the continuous working temperature is 1850 °C. The temperature measuring element is tungsten-rhenium thermocouple, and its temperature control precision is ± 2 °C. The equipment has the advantages of fast heating rate and uniform temperature field, and is mainly used for vacuum sintering, high temperature hot pressing, etc. [31]. It can meet the requirements of temperature, pressure and vacuum in diffusion bonding experiment, and accurately control the heating

Table 3 Diffusion bonding experimental scheme

Number	Temperature (°C)	Time (min)	Pressure (MPa)
1	800	30	12
2	850	10	12
3	850	20	12
4	850	30	12
5	900	30	12

rate, heating temperature and holding time through the computer program. Put the assembled specimen and mold into the furnace, and enter the temperature control curve by computer programming according to each test parameter in Table 3. The temperature control curve is shown in Fig. 5 (Take temperature curve of bonding temperature 850 °C, holding time 30 min and pressure 12 MPa as an example). When the degree of vacuum reaches 10 Pa, the program starts and begins to heat up. According to Table 3, after heating to the test temperature, keep the temperature constant and apply pressure for the set time. Then, the specimen was furnace-cooled to room temperature. The whole test process lasted about 12 h.

2.2.2 Microstructure analysis and mechanical properties testing

After the diffusion bonding experiment, the stiffener of the specimen was cut off by WEDM, and the microstructure analysis and mechanical properties testing were carried out. Before analysis and testing, specimens were grinded by 240–7000# abrasive paper in turns and the surface of specimen was polished into a mirror surface and then corroded. The ratio of corrosion liquid is $\text{HF}:\text{HNO}_3:\text{H}_2\text{O} = 1:3:10$ (volume ratio), and the corrosion

Fig. 4 **a** Schematic diagram of specimen assembly; **b** Diffusion bonding equipment

Fig. 5 Temperature control curve

time is 10 s. The microstructure of diffusion bonding joint was observed by optical microscope (OM), and the bonding ratio was calculated by Image-Pro Plus software. The bonding ratio is an important index to evaluate the quality of diffusion bonding. The higher the bonding ratio, the better the quality of diffusion bonding and the formula of bonding ratio is as follows:

$$\varepsilon_L = \frac{L_0 - L_1}{L_0} \times 100\% \quad (1)$$

where ε_L is joint bonding ratio, L_0 is bonding interface length, L_1 is unbonding and defect section length.

Tensile tests are carried out with a Zwick micro-tensile tester. The stretching speed is set to 2.4×10^{-1} mm/min, and the preload is 0.1 N. After tests, the tensile fracture was observed by SEM. Microhardness was measured by an instrumented universal hardness tester under the test load of 1 N for 15 s. Take the diffusion bonding interface as the center, measure microhardness values every 0.5 mm and compare the changes. The deformation ratio is obtained by measuring the thickness of bonding specimens. The calculation formula of deformation ratio is:

$$\varepsilon_h = \frac{h_0 - h_1}{h_0} \times 100\% \quad (2)$$

where ε_h is deformation ratio, h_0 is height before bonding, h_1 is height after bonding.

3 Results and discussion

3.1 Microstructure

In general, the quality of diffusion bonding is mainly affected by the following three factors: bonding temperature, holding

Fig. 6 Macroscopic picture of diffusion bonding specimens joints

time and bonding pressure. In particular, temperature and time are the most important factors [32]. This paper focuses on the effect of these two main factors on diffusion bonding quality of TC4 alloy hollow structures.

The 5 sets of experiment specimens in Table 3, have reached a good bonding on the macro-scale without any macroscopic defects. Cut the specimens by WEDM, and it is observed that the macroscopic interface has disappeared (Fig. 6). The preliminary stress test was carried out, and the specimens were well bonded.

Figure 7 shows the microstructure of the TC4 alloy hollow structure at diffusion bonding temperature of 800 °C, 850 °C and 900 °C. The magnification is 200 times, pressure of diffusion bonding is 12 MPa, and holding time is 30 min. The pictures show that the quality of diffusion bonding is getting better as the bonding temperature increases. The interface of joint is obvious at 800 °C. The voids are dense and large in size, and they were observed into strips at low magnification. At 850 °C, the joint quality has been obviously better. There are only a few voids at the joint and the size is smaller. When the bonding temperature is 900 °C, no void was observed under the microscope, which indicates that the material on both sides of the joint has been well bonded. The effect of temperature on diffusion bonding can be expressed by Arrhenius formula in kinetic theory [32, 33]:

$$D = D_0 \exp(-Q/RT) \quad (3)$$

where D is diffusion coefficient, D_0 is pre-exponential factor, Q is volume diffusion activation energy, R is gas constant, and T is bonding temperature. From Eq. (3), the higher diffusion bonding temperature, the greater diffusion coefficient, this means diffusion rate is greater. However, excessive temperature will reduce the performance of joint, so it is necessary to obtain appropriate temperature parameters according to the specific conditions by test.

Figure 8 shows the microstructure of the TC4 alloy hollow structure at holding time of 10, 20 and 30 min. The magnification is 500 times, bonding temperature is 850 °C,

Fig. 7 Microstructures of diffusion bonding joints under different temperature: **a** 800 °C; **b** 850 °C; **c** 900 °C; **d** Microstructure of the edge of diffusion bonding specimen

and pressure is 12 MPa. As shown in the figure, the quality of diffusion bonding joint has improved as the holding time becomes longer. When the holding time is 10 min, there are some larger size voids at the interface of bonding joint, and the voids are connected into strips. These strip-shaped and unbounded defects have a great bad effect on the mechanical properties of products, so the defects must be avoided in production. When the holding time is 20 min, the number of voids at bonding joint is decrease. The voids are round and the distribution is dispersed. There were no strip-shaped and unbounded defects found. This indicates that the quality of diffusion bonding is getting better. When the holding time is 30 min, only a small amount of voids exist at the joint, and the quality of diffusion joint is good. From the comparison of Fig. 8, with the longer holding time, the grain of the material has a certain growth, but it is not very obvious. The effect of holding time on diffusion bonding can be expressed by the following equation [33, 34]:

$$X = k(t - t_D)^{\frac{1}{2}} \quad (4)$$

where X is atomic diffusion distance, k is rate constant, t is holding time, t_D is diffusion delayed reaction time.

The change of bonding ratio with diffusion bonding temperature and holding time is shown in Fig. 9 [Calculated by Eq. (1)]. It is more obvious from Fig. 9 that with the increase in bonding temperature and holding time, the joint interfacial bonding ratio is improved to a certain extent. At the conditions of bonding temperature 900 °C, holding time 30 min, pressure 12 MPa, the joint bonding ratio has reached 98%. Under the same conditions, the bonding ratio of 850 °C reached 92%. It is observed by optical microscope that under the influence of pretreatment, some voids are not closed the edge of the specimen due to insufficient contact between the bonding interfaces (Fig. 7d). Therefore, even under the proper process parameters, the bonding ratio of whole specimen does not achieve 100%. However, the joint interfacial bonding ratio is high after leaving the edge and the voids at the interface disappear completely. The bonding ratio is basically 100%. In addition, under the conditions of 850 °C, 20 min and 12 MPa, joint bonding ratio reached over 86%. Only a few voids were observed under microscope, the whole bonding quality of joint was better.

Fig. 8 Microstructures of diffusion bonding joints under holding time: **a**10min; **b**20min; **c**30min

By the above analysis of the experiments show that when the diffusion bonding temperature is more than 850 °C, holding time is more than 20 min and pressure is proper, the TC4 alloy hollow structure specimen can get a good bonding quality on the basis of joint interfacial bonding ratio. Because the specimen has unavoidable error in the mechanical processing, especially in the pretreatment stage, the quality of the edge of joint is poor. Therefore, a certain allowance should be left when processing in order to trim the edge of product after diffusion bonding. The bonding ratio can reach 96%–100% after the treatment.

3.2 Mechanical properties

3.2.1 Tensile mechanical properties

Figure 10a shows the change in the tensile strength of joint when the bonding temperature is 800 °C, 850 °C and 900 °C. As shown in Fig. 10a, the tensile strength of the joint increases as the bonding temperature increases

in the range of 800 °C–900 °C. When the temperature is 900 °C, the tensile strength reached 979 MPa, which is basically the same as the tensile strength (980 MPa) of the base metal. And at 850 °C, the tensile strength is 952 MPa, which is more than 97% of tensile strength of the base metal. The tensile tests and microstructural observation show that the joints obtained under these two parameters not only achieved almost complete diffusion bonding on the micro-scale, but also have good mechanical properties. The tensile strength is basically the same as the base metal and has met the product requirements. When the bonding temperature is 800 °C, the tensile strength of diffusion bonding specimen is only 588 MPa. The reason is that the diffusion bonding is not complete enough at this temperature, and there are many voids at the interface. However, when the temperature rises from 800 °C to 850 °C, the tensile strength of the specimen increases greatly and basically reaches the tensile strength of the base metal. This further proves that the 850–900 °C is the suitable temperature for diffusion bonding.

Fig. 9 Bonding ratio of joints under different conditions: **a** At different temperatures for 30 min, 12 MPa; **b** At 850 °C, 12 MPa for different time

Fig. 10 Tensile strength of joints under different conditions: **a** At different temperatures for 30 min, 12 MPa; **b** At 850 °C, 12 MPa for different time

Figure 10b shows the change in the tensile strength of joint when the bonding time is 10, 20 and 30 min. From Fig. 10b, the tensile strength of the joints increases gradually with the prolonging of holding time in 10–30 min. When the holding time is 10 min, the tensile strength of the diffusion joint specimen is 837 MPa, which has reached 85% of tensile strength of the base metal. When the holding time is 20 min and 30 min, the tensile strength of joints is 917 MPa and 952 MPa, respectively. Within a certain range, the prolonged holding time leads to more sufficient diffusion bonding. It causes the tensile strength of the joints to rise steadily.

Figure 11 is true stress–strain curves of the diffusion bonding specimens obtained by tensile tests. The diffusion bonding process parameters are temperature 900 °C and 850 °C, holding time 30 min and pressure 12 MPa. The stress–strain curves show that the specimens do not yield significantly during the tensile test. Figure 12 is the macroscopic morphology and microstructure of tensile fracture of specimens. It can be seen from the diagram that the dimples are deep and dense, and the fracture presents typical ductile fracture.

Fig. 11 Stress–strain curves

3.2.2 Microhardness profiles

Figure 13 shows the change in microhardness at the interface and on both sides of the joint. Microhardness measurements were performed at each point of the 0.5 mm at the center of the interface of the diffusion bonding joint (see Fig. 14). Three points are taken in parallel in

Fig. 12 SEM fractographs of tensile specimens: **a** Macroscopic morphology; **b** Micro-morphology

Fig. 13 Microhardness values of joints under different conditions: **a** At different temperatures for 30 min, 12 MPa; **b** At 850 °C, 12 MPa for different time

each position, and their average values are calculated to obtain the microhardness of the position. It can be seen from Fig. 12, even under the effect of different bonding temperature and holding time, the peak value of microhardness of diffusion bonding joint also occurs at the joint interface. And the closer to the joint interface, the higher the microhardness value. The main reason for this phenomenon is likely to be the pressure applied during the holding stage, which results in a large microscopic plastic deformation of the grain near the interface, lead to grain refinement occurs. The secondary reason is likely to be that impurities and oxides are not completely cleaned up at the interface, which results in a locally increase in microhardness after diffusion bonding.

From Fig. 13a, the microhardness of the joint increases integrally as the temperature increases. When the temperature is 800 °C, the range of microhardness changes from HV347–403. At 850 °C, the microhardness range is HV349–457. At 900 °C, the microhardness range is HV364–472, and the maximum microhardness is reached at the interface area. The value of microhardness of the

Fig. 14 Distribution of microhardness values measurement points

joint interface is obviously low at 800 °C. This is because the diffusion bonding is not sufficiently at this temperature, so that there are many voids at the interface, which leads to lower microhardness. The increase in temperature accelerated the microscopic plastic deformation of the interface and the contact area became larger. Therefore, the diffusion speed of the interface atoms is enhanced, and the grain refinement is further refined so that the microhardness increases. From Fig. 13b, the microhardness of the joint decreases as the holding time increases. The maximum microhardness value is HV365 at the holding time of 30 min. Prolonging holding time leads to the

Fig. 15 Deformation ratio of joints under different conditions: **a** At different temperatures for 30 min, 12 MPa; **b** At 850 °C, 12 MPa for different time; **c** The physical picture of diffused bonding specimens

growth of metal grain, which results in the decrease in microhardness. However, the growth of grain is not very serious because the range of holding time is small in this experiment. Therefore, the change of microhardness value is not very large. Through the microhardness test, it can be concluded that the bonding temperature has more effect on the microhardness value than the holding time.

3.2.3 Deformation ratio

Figure 15 shows the deformation ratio of the joint under different diffusion bonding parameters [Calculated by Eq. (2)]. The clearance between the upper and lower die of the diffusion bonding die is 2 mm in the experiment, which means the maximum deformation of specimens is 2 mm. Due to these deformations are all irreversible, the maximum deformation ratio can reach 20%. From Fig. 15, it is found that the deformation ratio of specimen increases with the increase in bonding temperature and holding time, but their effect degree is different. As shown in Fig. 15a, bonding temperature has a great effect on the deformation ratio. With the increase in temperature, the resistance to deformation of alloy decreases rapidly. The deformation ratio is only 4% at 800 °C and 13% at 850 °C. At 900 °C, the maximum deformation rate has reached 20%. It indicates that the deformation ratio has been too large at this temperature, which will have a bad effect on the forming of the products. As shown in Fig. 15b, with the increase in holding time, the deformation ratio increases

gradually and reaches the maximum value of 13% at 30 min. It is indicated that the effect of holding time on deformation is not large. Under the condition of proper bonding temperature, prolonging the holding time properly will not cause excessive deformation ratio of products.

4 Conclusions

- (1) TC4 alloy hollow structure was designed based on the rudder surface, and its diffusion bonding experiment was carried out. In the process of diffusion bonding temperature 850 °C, holding time 20–30 min and bonding pressure 12 MPa, a good quality diffusion bonding joint has been obtained. When the holding time is 30 min, the joint bonding ratio is more than 92%, the tensile strength is 952 MPa, and the deformation ratio is maintained at about 10%.
- (2) Diffusion bonding temperature is the main factor affecting the diffusion bonding quality of TC4 hollow structure. The higher bonding temperature is, the higher bonding ratio is, and the tensile strength is also improved with it. However, the excessive temperature will increase the deformation ratio of hollow structural parts. The experimental results show that the bonding temperature should not exceed 900 °C, when this kind of TC4 hollow structure is formed by diffusion bonding.

- (3) Diffusion bonding holding time has a great effect on joint bonding quality. As the holding time increases, the joint bonding ratio increases, which results in the increase in tensile strength. However, if the holding time is too long, it will lead to grain growth, lower hardness, and have a bad effect on tensile strength. Meanwhile, excessive holding time reduces the production efficiency, so the holding time should be shortened as much as possible.
- (4) The microhardness of the TC4 alloy hollow structural joint shows the trend of "high in the middle and low on both sides" after diffusion bonding. It is because the process of diffusion bonding leads to grain refinement at the interface. When the temperature increases, the whole microhardness of the joint increases, and when the holding time is prolonged, the whole microhardness decreases.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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