Interfacial microstructure and mechanical properties of diffusion-bonded joints of titanium TC4 (Ti-6Al-4V) and Kovar (Fe-29Ni-17Co) alloys

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ARTICLE INFO	ABSTRACT
	Diffusion bonding is a near net shape forming process that can join dissimilar materials through atomic diffusion under a high pressure at a high temperature. Titanium alloy TC4 (Ti-6Al-4V) and 4J29 Kovar alloy (Fe-29Ni-17Co) were diffusely bonded by a vacuum hot-press sintering process in the temperature range of 700—850 ℃ and bonding time of 120 min, under a pressure of 34. 66 MPa. Interfacial microstructures and intermetallic compounds of the diffusion-bonded joints were characterized by optical microscopy, scanning electron microscopy, X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS). The elemental diffusion across the interface was revealed by electron probe microanalysis. Mechanical properties of joints were investigated by micro Vickers hardness and tensile strength. Results of EDS and XRD indicated that (Fe, Co, Ni)-Ti, TiNi, Ti_2Ni, $TiNi_2$, Fe_2Ti, $Ti_{17}Mn_3$ and Al_6Ti_{19} were formed at the interface. When the bonding temperature was raised from 700 to 850 ℃, the voids of interface were reduced and intermetallic layers were widened. Maximum tensile strength of joints at 53. 5 MPa was recorded by the sintering process at 850 ℃ for 120 min. Fracture surface of the joint indicated brittle nature, and failure took place through interface of intermetallic compounds. Based on the mechanical properties and microstructure of the diffusion-bonded joints, diffusion mechanisms between Ti-6Al-4V titanium and Fe-29Ni-17Co Kovar alloys were analyzed in terms of elemental diffusion, nucleation and growth of grains, plastic deformation and formation of intermetallic compounds near the interface.

1. Introduction

Titanium alloys possess low densities, high strengths, and excellent corrosion resistances, enabling them for a wide range of applications in petrochemical, aviation and space industries[1]. They can be bonded with many kinds of steels to achieve multi-functional applications, while brittle intermetallic compounds, such as Ti-Fe and Ti-Ni phases, were formed at the joints[2-6]. Wang et al.[2,3] suggested the formation of massive undesirable hard and brittle phases, mainly $TiFe_2$ and TiFe, at the welded region when titanium alloy TA15 and stainless steel (SS) 304SS were welded by electron-beam welding. Chen et al.[4] conducted laser welding of Ti-6Al-4V with 201SS, which yielded a large amount of brittle intermetallics,

such as FeAl, FeTi, Fe_2Ti and $Ti_5Fe_{17}Cr_5$. Due to the existence of these hard and brittle intermetallics, residual stresses originated from the mismatch of thermal expansion of base metal cannot be released by plastic deformation, leading to cracks and spontaneous brittle fracture at the interface[3,5,6].

Kovar alloys possess advantages of similar thermal expansion properties with hard (borosilicate) glass and soft-magnetic properties, and thus they have been used for a wide range of applications in electronics industry[7-9]. Zhang et al.[10] brazed an aluminum alloy 5005 and 4J34 Kovar alloy with an Al-Si-Mg filler foil. It was found that FeAl and $FeAl_3$ were formed at the interface where fracture occurred. Wang et al.[11] prepared alumina ceramic/Kovar alloy joint brazed with Ag-35. 2Cu-1. 8Ti (wt. %).

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They showed that Ti element had interaction with Kovar alloy to form TiNi$_3$ and TiFe$_2$ in the welding area. Yang et al.[12] investigated the interaction between 4J33 Kovar alloy and Ag-Cu-8Ti active brazing alloy. In the vicinity of interface near the 4J33 Kovar side, (Fe, Co, Ni)-Ti intermetallic compounds were identified[12,13].

Developments of aerospace and electronic packaging industries have demanded for multi-functional components with high mechanical and physical properties, and flexibilities in design while at a low cost. Bonding titanium with Kovar alloys can achieve a combination of their advantages; however, intermetallic compounds aforementioned tend to form at their joint. Most of them are hard and brittle phases that are detrimental to the mechanical properties of the joint. Therefore, in this research, diffusion bonding was used to weld these two alloys.

Diffusion welding can enable contacted surfaces to achieve porosity closure by creep and atomic diffusion at a certain pressure and high temperature below their melting points[14-17]. It is a near net shape forming process that is suitable for joining dissimilar materials. Diffusion bonding of Ti-6Al-4V alloy and micro-duplex stainless steel was successfully made when processing was carried out at 900 ℃ for 45 min under a load of 4 MPa[18]. So far, the available studies have been done on the joining of titanium alloys and stainless steel or low-carbon steel, while there are no results reported on the welding of titanium and Kovar alloys.

The objective of this research is to investigate the diffusion bonding of a titanium TC4 (Ti-6Al-4V) and a Kovar (Fe-29Ni-17Co) alloys. In this work, solid-state diffusion bonding was done using a titanium alloy and a Kovar alloy at various temperatures for a constant period of time. The research focused on the evolution of interfacial microstructure at different temperatures and the bonding strength of diffusion-bonded joints. Based on mechanical properties and microstructure of diffusion bonded joints of titanium TC4 (Ti-6Al-4V) and Kovar (Fe-29Ni-17Co) alloys, mechanisms of joint formation, diffusion and fracture were investigated.

2. Materials and Methods

2.1. Materials and processing parameters

Chemical compositions and physical properties of TC4 (Ti-6Al-4V) and 4J29 (Fe-29Ni-17Co) base metals are listed in Tables 1 and 2, respectively. Cylindrical specimens of 30 mm in diameter and 25 mm in length were machined from the base metals. Prior to bonding, the surfaces were ground, polished, and then cleaned in an ultrasonic bath containing acetone, washed with ethyl alcohol and dried in air. The experiments were conducted in the temperature range of 700—850 ℃ with an interval of 50 ℃, for a bonding period of 120 min under an uniaxial load of 2.5 T (34.66 MPa pressure) in 0.2—0.8 vacuum, which was applied by hot pressing. During this process, heating was started at a constant rate of 10 ℃/min and after joining, furnace was naturally cooled to room temperature.

Table 1
Chemical composition of base metals (wt. %)

Alloy	Mn	Si	C	Fe	Co	Ni	Ti	Al	V	N	H	O
TC4	—	0.15	0.10	0.3	—	—	Balance	6	4	0.05	0.015	0.2
4J29	0.4	0.20	0.02	Balance	17.3	29	—	—	—	—	—	—

Table 2
Physical properties of base metals

Alloy	Density/ (g · cm^{-3})	Melting point/℃	Expansion coefficient/ (10^{-6} K^{-1})	Ultimate tensile strength/MPa
TC4	4.55	1725	7.14	978
4J29	8.10	1460	4.70	548

2.2. Interfacial microstructure characterization

Bonded joints were cut longitudinally, then ground, polished, and etched with two separated reagents for metallographic observation. The titanium alloy side was etched by an aqueous mixture solution of 8 mL HF, 10 mL HNO$_3$, and 82 mL H$_2$O, and the Kovar alloy side was etched by a mixture solution of 2.5 g CuCl$_2$, 50 mL ethyl alcohol, and 50 mL HCl. Microstructure owing to diffusion was revealed in a light microscope (AxioCam MRc 5). Line scan analyses crossing interfaces were carried out to evaluate diffusion of elements at the interface using a scanning electron microscope (SEM, JEOL JSM-7001F at 15 kV) equipped with an X-ray energy-dispersive spectrometer (EDS), and an electron probe micro analyzer (EPMA, JEOL JXA-8530F field-emission Hyperprobe at 15 kV) equipped with both an EDS and wavelength-dispersive spectrometers (WDSs).

2.3. Evaluation of mechanical properties

Tensile properties of joints were evaluated using a microcomputer-controlled electronic universal testing machine (WDW-3100) at a loading rate of 0.1 mm/min

at room temperature and four samples were tested for each parameter. The hardness measurements were carried out using a micro-Vickers hardness tester (HXD-100TM/LCD). The test load was 1.98 and 4.90 N for Kovar and titanium alloys, respectively, with dwell time of 15 s.

3. Results and Discussion

3.1. Microstructure and elemental line profiles

Fig. 1(a—d) shows micrographs of diffusion-bonded samples processed at different temperatures. It is found that diffusion interfaces are free of cracks and interface lines are clearly visible. Base metal microstructures of TC4 and 4J29 were not obviously changed with temperature varying from 700 to 850 ℃. Generally, as bonding temperature increases, the interfacial reaction zone is extended because of more binary or ternary intermetallic compounds formed due to diffusion of various elements, such as Ti, Al, V, Fe, Co and Ni, across the interface. The width of diffusion layer at the joint interface widens simultaneously, as a result of increased diffusion coefficient at a higher temperature. When the temperature reaches 850 ℃, amount of body-centered cubic (bcc) β-Ti phase increases obviously on the titanium alloy side near the interface. As β-stabilizing elements, Fe, Co and Ni migrate from the Kovar alloy side to the titanium alloy side, which significantly decrease the transition temperature of bcc β-Ti to hexagonal close-packed (hcp) α-Ti from 890 to 590 ℃ or even lower[19], and thus higher amount of high-temperature β phase is retained at room temperature. Therefore, the degree of diffusion bonding could be judged according to the width of intermetallic compounds and the amount of β phase near the interface.
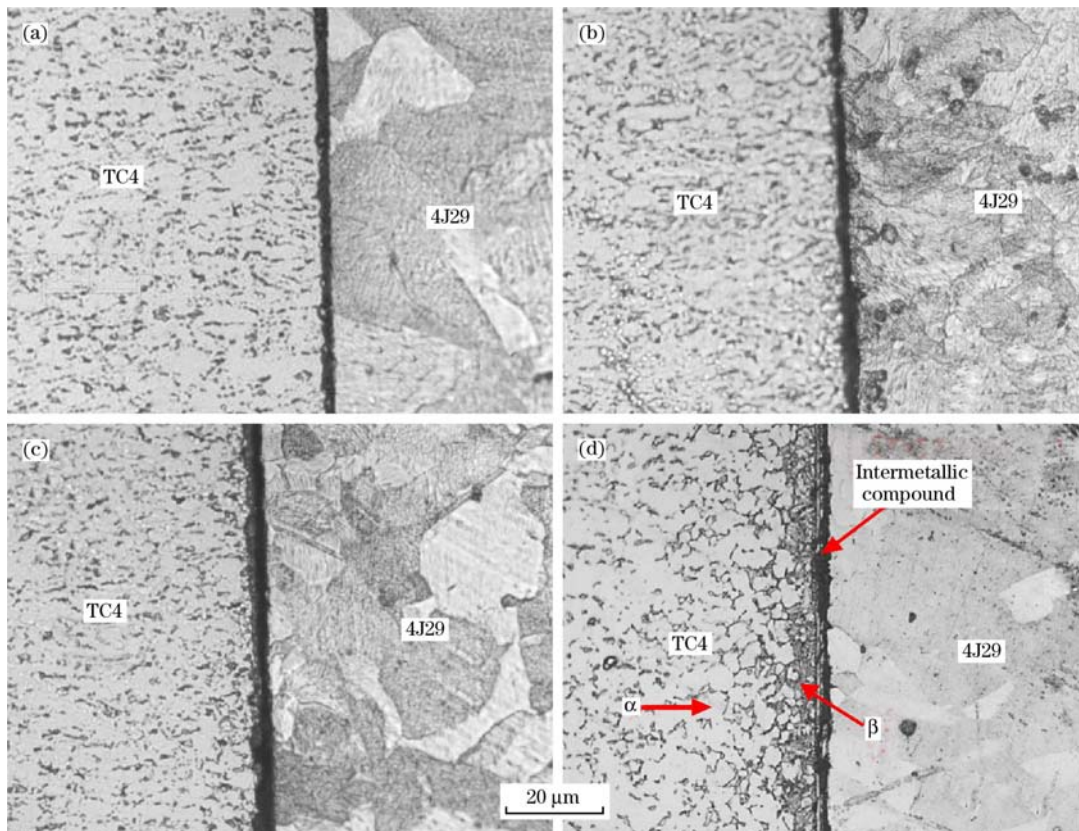

On TC4 side, white grains are α phase, while gray grains are β phase.
Fig. 1.   Optical micrograph of diffusion-bonded joints for 120 min at 700 ℃ (a), 750 ℃ (b), 800 ℃ (c) and 850 ℃ (d).

Concentration profiles of elements, namely, Fe, Co, Ni, Ti and Al of the joints bonded at 800 ℃ for 120 min are measured by EDS line scans, as shown in Fig. 2. It shows again that the amount of β-Ti phase in the vinicity of interface is more than that of parent metal. The dark line in Fig. 2(a) is the track of the electron beam after line scanning. In Fig. 2 (b), concentration of Ti, Fe and Co descends slowly at the center of interface, which means that intermetallic compounds are formed at this area, and the specific compounds will be demonstrated by XRD tests. Besides, concentration of Al is increased in the vinicity of interface. Al is an α-stabilizing element, which mainly exists in α phase in the form of substitutional solid solution. As β-stabilizing elements Fe, Co and Ni diffuse into titanium alloy from Kovar alloy
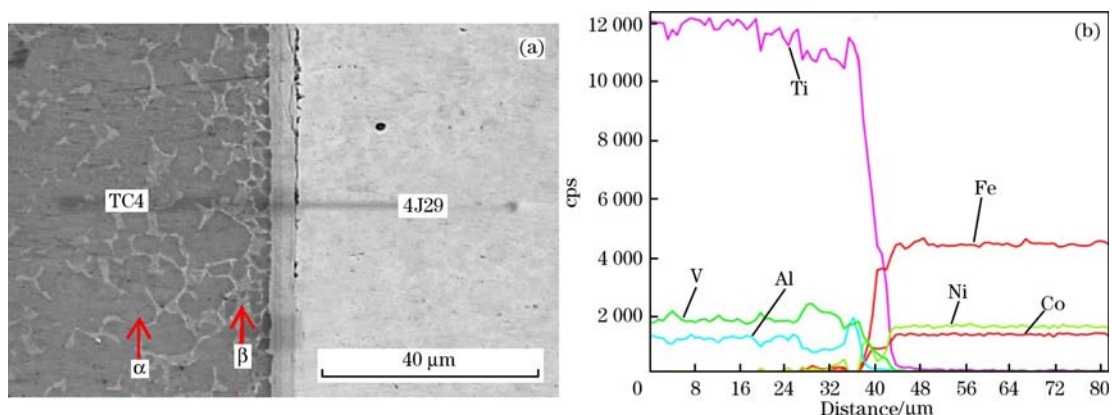
(a) SEM image (large dark grains are α phase, while other bright regions are β phase);   (b) Line scanning.
Fig. 2.   Interface element distribution of TC4 and 4J29 diffusion bonded at 800 ℃.

at 850 ℃, the amount of α phase is reduced when it reaches room temperature. When the concentration of Al adjacent to the interface is above 5.5％, titanium aluminides (TiAl and Ti_3Al) form[20]. According to the above analysis, the bonded joint mainly contains Fe-Ti, Ti-Co and Ti-Al phases at the interface.

Fig. 3 shows EPMA analysis of TC4 and 4J29 joint interface bonded at 850 ℃, which displays diffusion of elements at the interface by elemental maps. It is found that Co (Fig. 3(c)) and Ni (Fig. 3(d)) elements are diffused into the adjacent areas near the interface on titanium alloy. This area is consistent with the metallographic observation where β-Ti phase is enriched, which indicates that the formation of β-Ti phase is mainly due to the diffusion of Ni and

Co elements. By comparison, Fe (Fig. 3(b)) and Ti (Fig. 3(e)) elements are mainly concentrated in the vicinity of the interface, which is in good agreement with previous study[18]. Diffusion of Ti, Fe, Ni and Co elements to interface results in the formation of intermetallic compounds, such as Fe_2Ti, $TiNi_3$, Ti_2Ni, TiNi and Ti_2Co. It has been illustrated that Al atoms segregate at the interface and the remaining α phase, which is consistent with the line scanning results. Segregation of Al (Fig. 3(f)) leads to the formation of Ti-Al intermetallic, such as TiAl, Ti_3Al and Al_6Ti_{19}. The formation of intermetallic will be confirmed by the XRD measurements taken at the interface. From EPMA distribution map of each element, it is obvious that there is a sharp decline in content at the interface.
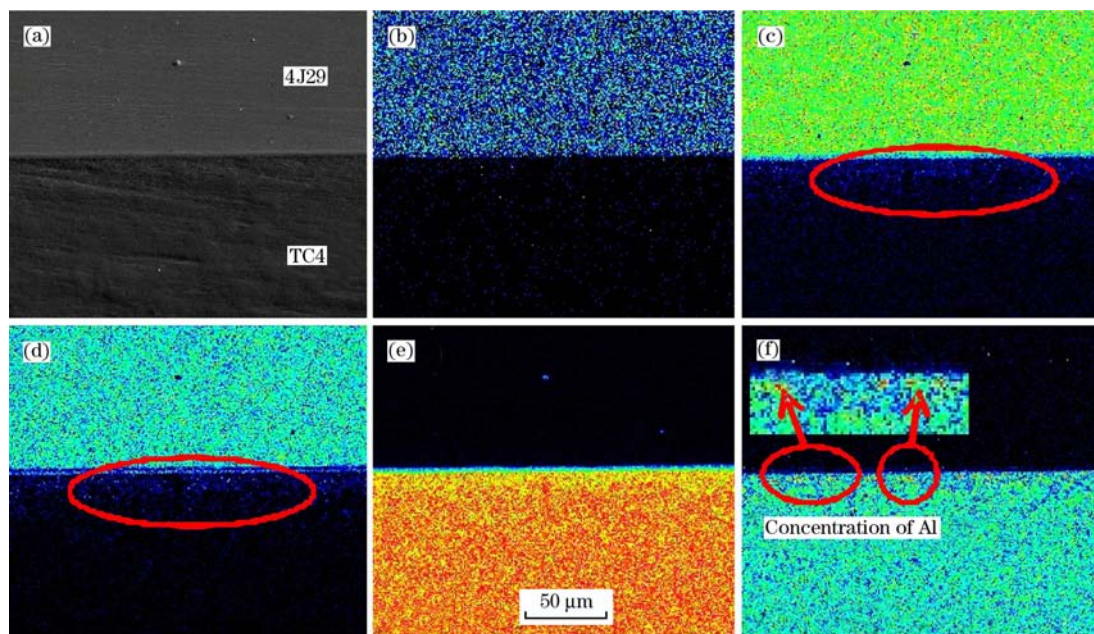
(a) BSE image;   (b) Fe map;   (c) Co map;   (d) Ni map;   (e) Ti map;   (f) Al map.
Fig. 3.   Concentration profiles from EPMA analysis of Fe, Co, Ni, Ti and Al elements along with a BSE (backscattered electron) image of a region at TC4 and 4J29 joint interface bonded at 850 ℃.

According to Fick's second law, in infinite length diffusion, relationship between elemental concentration and distance from the interface can be written as follows:

$$c = \frac{c_1 + c_2}{2} + \frac{c_1 - c_2}{2} \left[\mathrm{erf} \left(\frac{x}{2\sqrt{Dt}} \right) \right] \quad (1)$$

where, c_1 and c_2 are initial concentrations of element in two contact materials, respectively; x is the distance from the interface; t is diffusion time; and D is diffusion coefficient[21]. Diffusion coefficient of Fe into titanium alloy at 800 ℃, 8 MPa ($D_{Fe} = 3.37 \times 10^{-14}$ m^2/s) was reported in Ref. [22]. Based on the results, diffusion distance of Fe into titanium is calculated as 26.25 μm by Eq. (1), which is wider than the result of ~18 μm in Fig. 2. This is due to the fact that in the process of atomic diffusion, the diffusion coefficient decreases with increasing pressure[23].

3.2. Phase identification

Fig. 4 reveals a crack of the sample bonded at 850 ℃. Crack is formed at the interface and propagates along compounds layer. EDS result of 4J29 side is shown in Fig. 4(a). It is found that Ti diffuses into 4J29 side. Based on Fe-Ni-Ti ternary phase diagram, when the concentration of Ti is higher than 0.82 at.%, intermetallic compounds of Fe-Ti and Ti-Ni can be formed[14-19]. It is observed that S is concentrated in the interface. Existence of higher percent of sulfur weakens crystal boundary and decreases the strength of joints[24]. The signal of C is from carbon coating during sample preparation. By EDS analysis, it is confirmed that crack forms in intermetallic compounds layer where Fe-Ti, Ti-Ni, Fe-V and Ti-Al phases are present. These intermetallic compounds are hard and
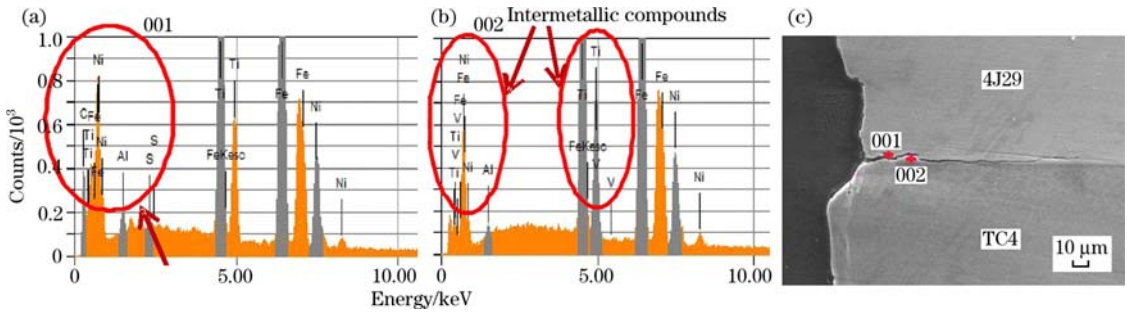
(a) 4J29 side from point 001;   (b) TC4 side from point 002;   (c) Secondary electron image of crack.

Fig. 4. EDS analysis of interface crack of sample bonded at 850 ℃.

brittle phases which do not release the stress that formed owing to different coefficients of linear expansion between TC4 (7.14×10^{-6} K^{-1}, 20 ℃), intermetallic compounds and 4J29 (4.7×10^{-6} K^{-1}, 20 ℃) during cooling[25]. Therefore, crack emerges at the interface under a small external force.

X-ray diffraction patterns of the fracture surfaces of diffusion bonded joints at 800 and 850 ℃ are shown in Fig. 5. It is found that intermetallic phases such as TiNi, Ti$_2$Ni, TiNi$_2$, Fe$_2$Ti, Ti$_{17}$Mn$_3$ and Al$_6$Ti$_{19}$ are present in reaction zone. The rest of the

Fig. 5. X-ray diffraction patterns of fracture surfaces of diffusion bonded joints at 800 and 850 ℃.

compounds are not observed because their contents are not high enough. Compared with diffusion products of titanium alloy and stainless steel, there is a little difference between reaction products[3,17,18,26,27]. Because concentrations of Co and Ni are higher in Kovar alloy, it is more liable to form Ti-Ni phases than Ti-Fe phases at the interface.

3.3. Mechanical properties

Fig. 6(a−d) shows the variation in micro-hardness of bonded joint at different temperatures in the range of 700−850 ℃. It is found that in the adjacent areas near the interface at the titanium alloy side, hardness is higher compared to base materials. The reason comes from diffusion of Fe, Co and Ni elements into titanium alloy, leading to the formation of β-Ti and brittle intermetallic phases such as Ti$_2$Ni, Al$_6$Ti$_{19}$, and Fe$_2$Ti at the bonded joint. The increase in the maximal hardness value is directly proportional to increase in temperature, 364 HV, 388 HV, and 400 HV in sequence. Maximum hardness value of (415±13) HV is obtained at 850 ℃ which is about 136 HV higher than that of titanium alloy. In the adjacent areas near the interface at the Kovar alloy side, hardness of 4J29 is lower than that of base ma-
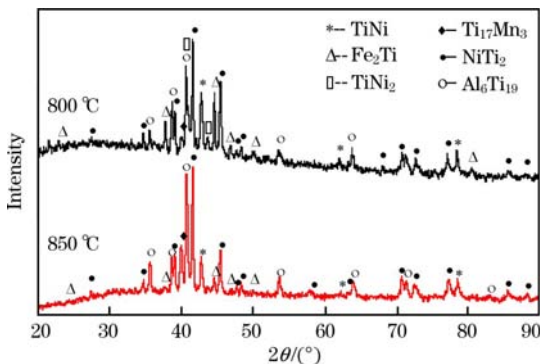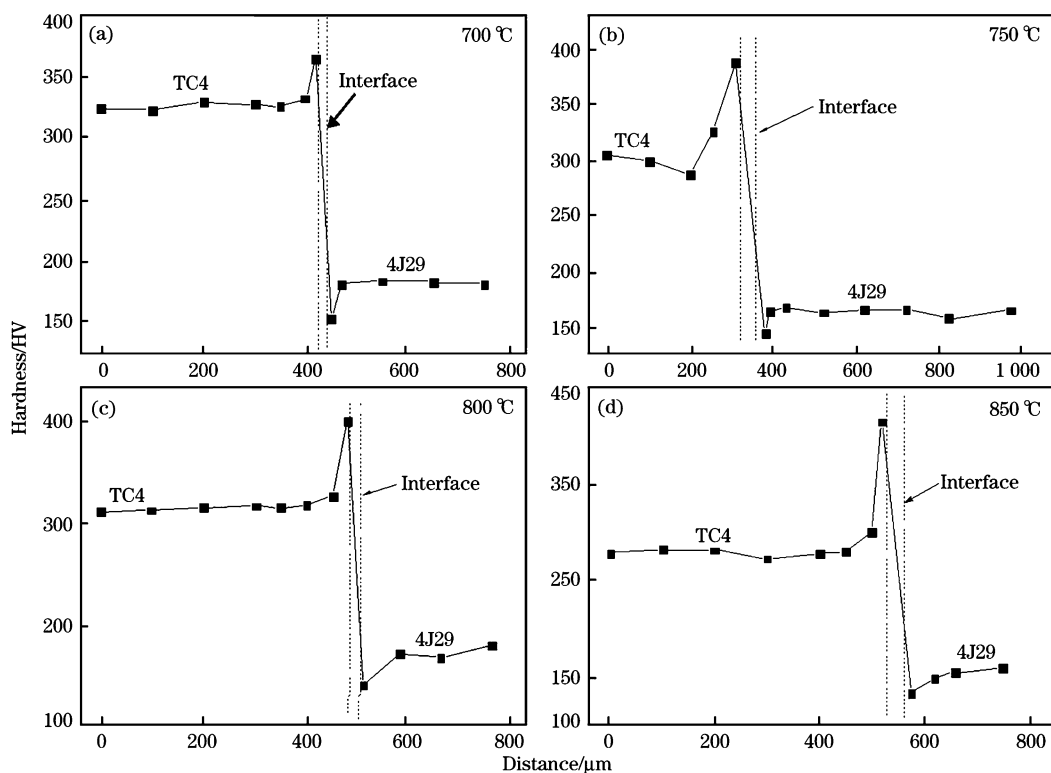
Fig. 6.　Micro-hardness of TC4 and 4J29 diffusion bonded at different temperatures.

terial, because Ti and Al elements are mainly concentrated at the interface. Amount of diffused Fe, Co and Ni into the titanium alloy side is more than that of the diffused Ti and Al into Kovar alloy, resulting in unbalanced mass flow across the interface[15,17]. Significant difference of hardness between interface and base metal leads to mechanical heterogeneity, which is detrimental to releasing the stress and mechanical property.

Variation in mechanical properties of the bonded joints in temperature range of 700−850 ℃ at an interval of 50 ℃ for 120 min is shown in Fig. 7. Diffusion-bonded joint between titanium and Kovar alloys prepared at 700 ℃ for 120 min separated during machining the sample, and thus tensile strength is assigned as zero. At 700 ℃, the interface is barely connected, owing to low temperature which limits the activities of atoms with low diffusion coefficients. When bonding temperature rises, bond strength gradually increases due to enhanced diffusion, and thus the compound layer at the interface is widened. Besides, as bonding temperature rises, yield strength of titanium alloy is decreased sharply for superplastic property, which is beneficial to connection of the interface by plastic deformation. By comparison between mechanical properties and interfacial diffusion width, it is obvious that tensile strength gradually increases with interfacial width at 700 − 850 ℃. When bonding temperature increases to 850 ℃, tensile strength of joint reaches 53.5 MPa with interfacial width of ∼6 μm.
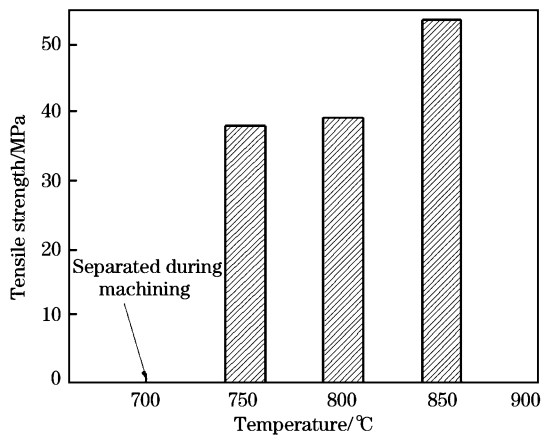

Fig. 7.　Mechanical properties of diffusion bonded joints processed for 120 min at different temperatures.

3.4. Fracture morphology

Fracture morphology and EDS analysis of diffusion boned joints at different temperatures are shown in Fig. 8. At 750 ℃ bonding temperature, voids and secondary cracks are observed in Fig. 8(a). Presence of voids is attributed to less contact between the ground surfaces of TC4 and 4J29, which leads to the formation of secondary cracks under loading. All these defects determine strength of the joint bonded at 750 ℃. As shown in Fig. 8(b), (Fe, Co, Ni)-Ti intermetallic compounds[12] are formed with a composition of Ti 47.62 at.%, Fe 22.67 at.%, Ni

16. 38 at. %, and Co 13. 33 at. %. Brittle intermetallic phases promote the generation of cracks, resulting in fracture of joints. As temperature rises to 800 ℃, volume fraction of voids shown in Fig. 8(b) is reduced for a better connection to form intermetallic phases, which contributes to slight increase of joint strength. Cleavage planes are noticeable in Fig. 8(c), which indicate brittle nature of diffusion boned joints. Intermetallic phases, such as TiNi, Ti_2Ni, $TiNi_2$, Fe_2Ti, $Ti_{17}Mn_3$ and Al_6Ti_{19}, as confirmed by previous XRD results, are found on the fracture surface of joints bonded at 800 and 850 ℃ as shown in Fig. 8(d).

These compounds determine the brittle fracture of the joints. In Fig. 8(e), granular fracture zone and intergranular fracture area are observed. Intergranular fracture area is the part where mating surfaces of TC4 and 4J29 contact initially. In the process of diffusion bonding, there are some spaces to be eliminated by the growth of grains in the gap, leading to formation of granular fracture zone[28]. Afterward, a better connection is gained, which contributes to apparent increase in strength. EDS analysis of this area is shown in Fig. 8(f), indicating intermetallic compounds as mentioned above.
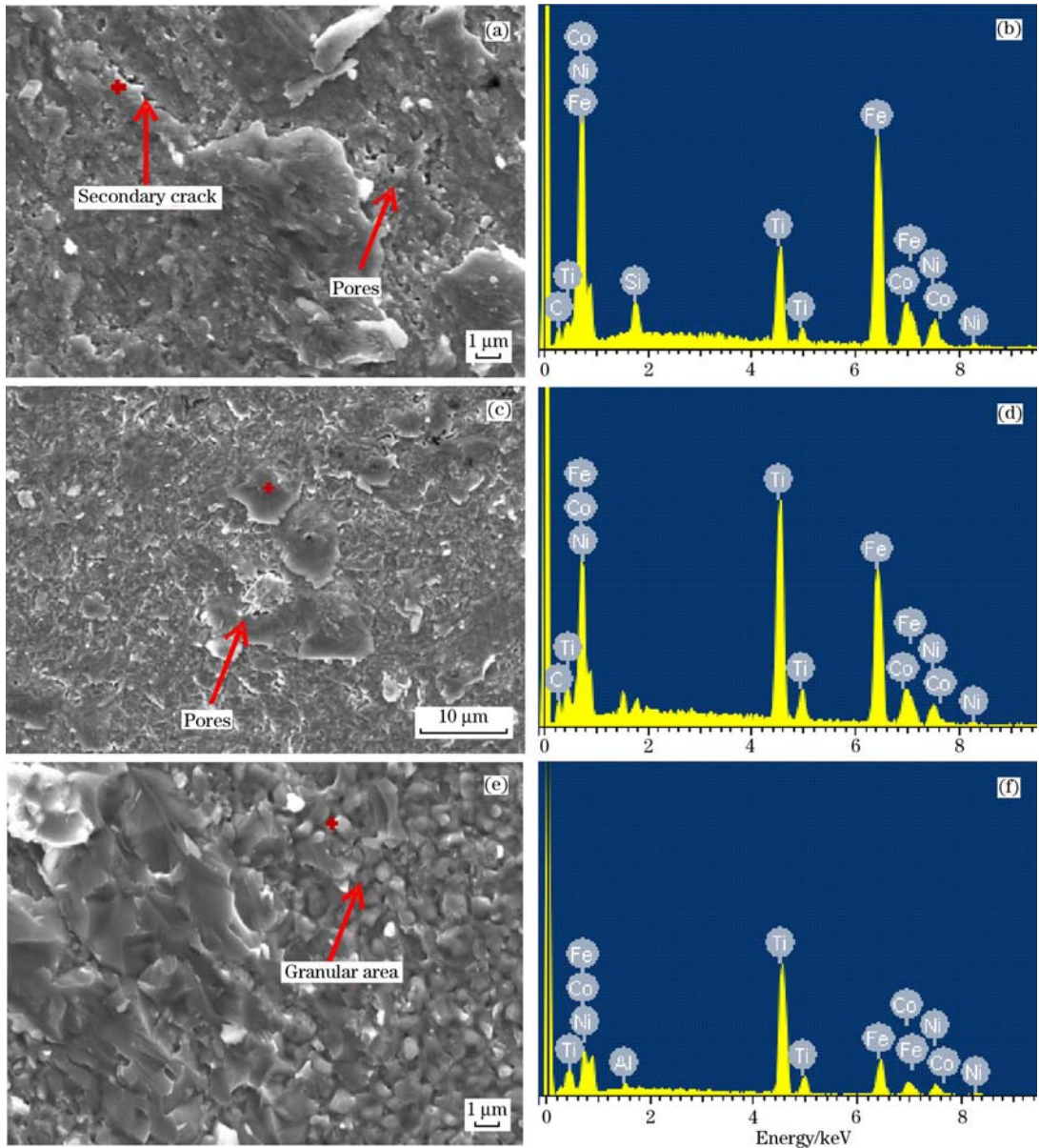

Fig. 8. Fracture morphology and EDS analysis of diffusion-bonded joints at 750 ℃ (a, b), 800 ℃ (c, d) and 850 ℃ (e, f).

By comparing bonding results at different temperatures, the process of diffusion bonding between titanium alloy and Kovar alloy mainly includes three steps. Firstly, partial surfaces of base metals contact

with each other where Ti, Fe, Ni, Co, and Al elements are diffused and some pores exist at the interface, as shown in Fig. 9(a). Secondly, pores are gradually diminished by plastic deformation, nuclea-

tion and growth of grains at higher temperature with a certain pressure. Initially intermetallic phases, such as (Fe, Co, Ni)-Ti, TiNi$_2$, Ti$_{17}$Mn$_3$ and Fe$_2$Ti, grow to laminar microstructure, as shown in Fig. 9 (b). Meanwhile, near the contacted area, some grains grow towards the pores, filling the pores gradually. This process is accompanied with diffusion of elements. Thirdly, increased diffusion of Al, Ti and Ni elements promotes the formation of TiNi, Ti$_2$Ni, TiAl, Ti$_3$Al and Al$_6$Ti$_{19}$ at the interface. More β-Ti is

gained in the adjacent areas near the interface at titanium alloy side due to diffusion of Co and Ni into this area. A well contacted interface is gained and amount of intermetallic compounds is enhanced to achieve a connection between titanium and Kovar alloys, as shown in Fig. 9(c). Therefore, it can be concluded that connection of titanium and Kovar alloys is achieved by elemental diffusion, nucleation and growth of grains near contacted area, plastic deformation and formation of intermetallic compounds.
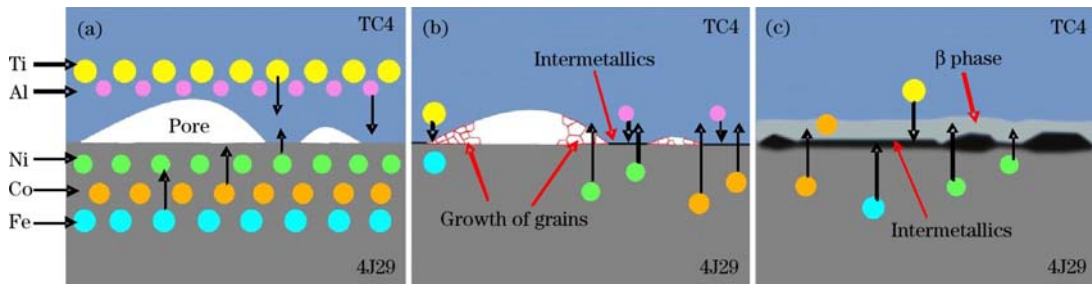
(a) Surface contact and element diffusion;   (b) Plastic deformation, nucleation and growth of grains;   (c) More intermetallic compounds nucleated at interface and more β phase formed in adjacent areas near interface at titanium alloy side.
Fig. 9.   Process of diffusion bonding between titanium alloy and Kovar alloy.

4. Conclusions

Diffusion bonding of TC4 titanium and 4J29 Kovar alloys has been carried out in vacuum in the temperature range of 700—850 °C for 120 min under 2.5 T (34.66 MPa) uniaxial pressure, which was applied along the longitudinal direction of the specimen.

(1) Titanium alloy (TC4) and Kovar alloy (4J29) could be connected by diffusion bonding, and the compounds such as (Fe, Co, Ni)-Ti, β-Ti, TiNi, Ti$_2$Ni, TiNi$_2$, Fe$_2$Ti, Ti$_{17}$Mn$_3$ and Al$_6$Ti$_{19}$ were formed at the interface.

(2) As bonding temperature was increased from 700 to 850 °C, the voids of interface were reduced with widened intermetallic layers, promoting the connection of joints. Maximum tensile strength of joints was 53.5 MPa, obtained at the process of 850 °C for 120 min.

(3) Fracture surface of joints indicated brittle nature, and failure took place through the interface of intermetallic compounds.

(4) Connection of titanium and Kovar alloys was achieved by elemental diffusion, nucleation and growth of grains near contacted area, plastic deformation and formation of intermetallic compounds.

Acknowledgment

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