JOURNAL OF MATERIALS SCIENCE 40 (2005) 6385–6390

# An experimental investigation of phase transformation superplastic diffusion bonding of titanium alloy to stainless steel

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**Published online:** 5 October 2005

The solid-state direct diffusion bonding of a near $\alpha$-phase titanium alloy to an austenitic stainless steel by means of the phase transformation superplasticity (PTSP) caused by the cycles of heating and cooling has been carried out. The test results showed that, under the conditions of $T_{max} = 890^{\circ}C$, $T_{min} = 800^{\circ}C$, cyclic number of heating and cooling $N = 10$ cycles, specific pressure $P = 5$ MPa, heating rate $V_h = 30^{\circ}C/s$ and cooling rate $V_c = 10^{\circ}C/s$, the ultimate tensile strength of the joint reached its maximum value (307 MPa), and the bonding time was only 120 s. In the phase transformation superplastic state, the deformation of titanium alloy has a character of ratcheting effect and it accumulates with the cycles of heating and cooling. The observations of tensile fracture interface showed that both the brittle intermetallic compound (FeTi) and the solid solution based on $\beta$-Ti were formed on the interface, and the more in quantity and the smaller in size the solid solutions are, the higher the ultimate tensile strength is.


## 1. Introduction

Due to the excellent combination between the mechanical behaviors and corrosion resistance, more and more joints made from titanium alloy and stainless steel are used in many fields of industry, such as the space and nuclear industry [1]. In the fusion welding processing of dissimilar metals such as titanium alloys and stainless steels, brittle intermetallic compounds are formed in the vicinity of joints, which are detrimental to the mechanical properties of the couples and results in the decrement of the reliability of application [2, 3]. However, diffusion bonding is suitable for the joining of the titanium and stainless steels [4, 5]. In diffusion bonding, in order to produce a metallurgical joint between dissimilar metals, a higher bonding temperature and longer bonding time are needed to speed up the interdiffusion rate of relative elements. In such a situation, the growth of grains of parental metals and an incre- ment in the thickness of intermetallic compounds is difficulty to avoid [3, 4]. In addition to this, the thermo-stress as developed in the joints due to the differences in the coefficient of linear expansion between the titanium alloy and stainless steel is harmful to both the mechanical properties and corrosion resistance of the joints [3]. So it is very significant to bond the dissimilar metals at relatively lower bonding temperatures and shorter bonding times.

It is well known that very fine crystalline materials show superplasticity, and that in the superplastic state, they can be diffusion bonded easily [6, 7]. Improvement of weldability is also found in materials showing another type of superplasticity. When the materials are repeatedly heated and cooled in a temperature range which includes their phase transformation temperature, they can be greatly deformed. This phenomenon is known as dynamic superplasticity [8], and in this superplastic state material can be diffusion bonded as easily as can very fine grained materials. Although austenitic stainless steel cannot be phase-transformed from room temperature to its melting point, titanium and its

0022-2461 
DOI: 10.1007/s10853-005-1629-0

TABLE I Chemical composition of TA17 and 0Cr18Ni9Ti(wt%)

| | C | Si | Mn | P | S | Cr | Ni | Ti | Al | V | Fe |
|---|---|---|---|---|---|---|---|---|---|---|---|
| TA17 | 0.01 | 0.04 | | | | | | Bal. | 4.5 | 2.2 | 0.069 |
| 0Cr18Ni9Ti | 0.04 | 0.47 | 1.21 | 0.034 | 0.02 | 17.22 | 8.37 | 0.29 | | | Bal. |

alloys show the dynamic superplasticity related the phase transformation from $\alpha$-phase with HCP structure to $\beta$-phase with BCC structure [9, 10]. In present work, the near $\alpha$-phase titanium alloy and 18-8 type austenite stainless steel were bonded using the phase transformation superplastic (PTSP) diffusion bonding processing, and the optimum technical parameters were obtained with the aim of lowing the bonding temperature and shortening the bonding time.

## 2. Experimental materials and procedures

The titanium alloy (TA17) and stainless steel (0Cr18Ni9Ti) were received in the form of rods with a diameter of 12 mm. The TA17 has the nominal composition of Ti-4Al-2V, containing less than 10% $\beta$ phase. The TA17 rods used in present work were recrystallised at 750°C for 1 h after hot rolling. The phase transformation temperature of TA17 measured by DSC is in the range of 888 to 939°C. The 0Cr18Ni9Ti is a Chinese designation of stainless steel with a nominal composition of less than 0.08C, 18%Cr, 9%Ni and less than 0.5%Ti (in wt%). The chemical compositions of TA17 and 0Cr18Ni9Ti are given in Table I.

The titanium alloy and stainless steel rods were cut into cylinders with 12 mm in diameter and 30 mm in length for the bonded specimen. Before bonded, the two faying surfaces were prepared by conventional grinding and polishing techniques, then chemically cleaned to remove the oxide film and grease. The PTSP diffusion bonding experiments were carried out in the Gleeble 1500-D thermo-mechanical simulator. The temperature of bonding was controlled and monitored by Ni–Cr and Ni–Al thermo-electrical couples which were welded in the stainless steel side approximately 1 mm away from the interface. The vacuum used was $5\times10^{-2}$ Pa. Several bonding variations, such as the maximum and minimum bonding temperature ($T_{max}$ and $T_{min}$), the cyclic number of heating and cooling, the specific pressures, and the heating rates are tested in this work.

The joint strength of bonded couples was determined by tensile experiments with a cross-heading speed of 2 mm/min. The micro-region compositions of Fe, Ti, Ni and Cr elements on the tensile fracture interface were measured by the energy dispersive spectroscope (VOYAGER-2) analysis. The tensile fractural interfaces were observed under the scanning electrical microscope (AMRAY-1845E) with an operating voltage of 25 KV.

## 3. Experimental results and discussion

### 3.1. The deformation of specimens during bonding

The basic character of PTSP diffusion bonding is the superplastic deformation of specimens, which places the elements in an activity state, and accelerates the diffusion of them. A typical curve of temperature ($T$) vs. time ($t$) and strain vs. time during PTSP bonding is shown in Fig. 1.

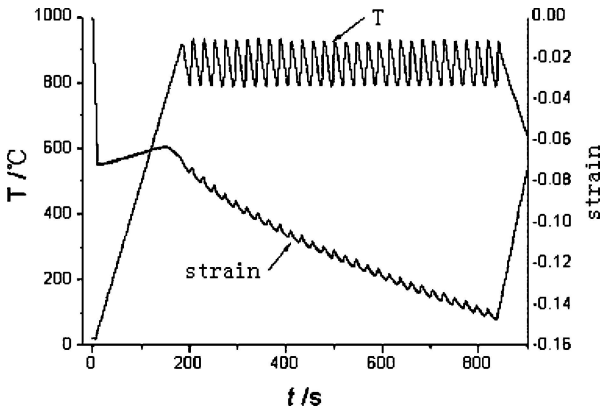


*Figure 1* The deformation of specimen during bonding ($T_{max} = 890°C$, $T_{min} = 800°C$, cycles = 30 times, $V_h = 30°C/s$, $V_c = 10°C/s$).

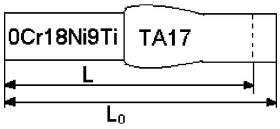


*Figure 2* Schematic drawing of the outline of couples bonded. $L_0$ and $L$ are the length before and after bonding respectively. Strain is defined as $\ln L/L_0$.

From Fig. 1, it can be shown that, when bonding temperature varies periodically and symmetrically during bonding, the response of strain to bonding temperature has a feature of "ratcheting effect," and it accumulates with the increment of cycles of heating and cooling. The measurement result of the couples (Fig. 2) bonded indicated that in the total strain, about 80–85% strain is concentrated in the TA17 side, which suggests that the superplastic behaviors really occur in the titanium alloy side.

### 3.2. The relationship between the technique parameter and tensile strength

#### 3.2.1. Maximum temperature ($T_{max}$)

Under condition of minimum temperature $T_{min} = 800°C$, cyclic number of heating and cooling $N = 30$, heating rate $V_h = 30°C/s$, cooling rate $V_c = 10°C/s$, and specific pressure $P = 5$ MPa, the dependence of $\sigma_b$ (ultimate tensile strength) of joint on $T_{max}$ was determined (see Fig. 3). The Maximum temperatures were chosen as $T_{max} = 850$, 870, 910, 930, 950 and 970°C.

In the $T_{max}$ range of 850 to 890°C, $\sigma_b$ increases slightly with the increment of $T_{max}$, whereas when $T_{max}$ is higher than 890°C, $\sigma_b$ reduces with the increment of $T_{max}$, especially when $T_{max}$ exceeds 930°C. At 890°C, the highest $\sigma_b$ value is 276 MPa. Therefore, the optimum $T_{max}$ is 890°C, which is just above the phase transformation beginning temperature (888°C) of TA17.

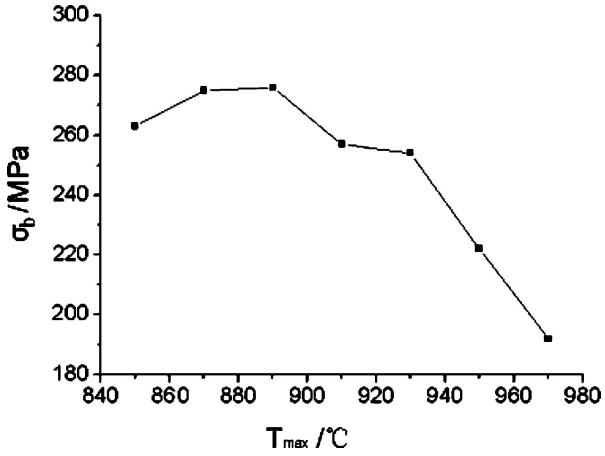


*Figure 3* Dependence of $\sigma_b$ (ultimate tensile strength) of joint on $T_{max}$.

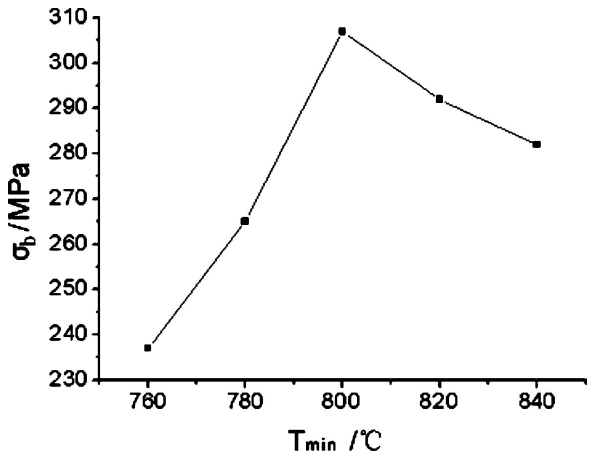


*Figure 5* Dependence of $\sigma_b$ (ultimate tensile strength) of joint on $T_{min}$.

### 3.2.2. Cyclic number of heating and cooling, N

Under a condition of maximum temperature $T_{min}$ = 890°C, minimum temperature $T_{min}$ = 800°C, heating rate $V_h$ = 30°C/s, cooling rate $V_c$ = 10°C/s, and specific pressure $P$ = 5 MPa, the dependence of $\sigma_b$ (ultimate tensile strength) of joint on $N$ was determined (see Fig. 4). The Ns were chosen as $N$ = 2, 10, 15, 20, 30 and 45 cycles.

From Fig. 4, the optimum $N$ value is 10 cycles, at which the $\sigma_b$ reaches its highest value, 307 MPa. It should be noticed that the total holding time at high temperature of 10 cycles is only 120 s.

### 3.2.3. Minimum temperature, $T_{min}$

Under condition of maximum temperature $T_{min}$ = 890°C, heating rate $V_h$ = 30°C/s, cooling rate $V_c$ = 10°C/s, cyclic number of heating and cooling $N$ = 10, and specific pressure $P$ = 5 MPa, the dependence of $\sigma_b$ (ultimate tensile strength) of joint on $T_{min}$ was determined (see Fig. 5). The minimum temperatures were chosen as $T_{min}$ = 760, 780, 800, 820 and 840°C.

It can be seen clearly from Fig. 5 that there is a sharp peak at $T_{min}$ = 800°C, which corresponds to the highest ultimate tensile strength (307 MPa).

### 3.2.4. Specific pressure, P

Under condition of maximum temperature $T_{min}$ = 890°C, minimum temperature $T_{min}$ = 800°C, cyclic number of heating and cooling $N$ = 10, heating rate $V_h$ = 30°C/s, cooling rate $V_c$ = 10°C/s, the dependence of $\sigma_b$ (ultimate tensile strength) of joint on $P$ was determined (see Fig. 6). The specify pressures were chosen as $P$ = 3, 5, 8 and 10 MPa.

When the specific pressure increases from 3 to 5 MPa, the ultimate tensile strength increases rapidly, and when $P$ is 5–10 MPa, it changes little. So, the optimum $P$ value is 5 MPa.

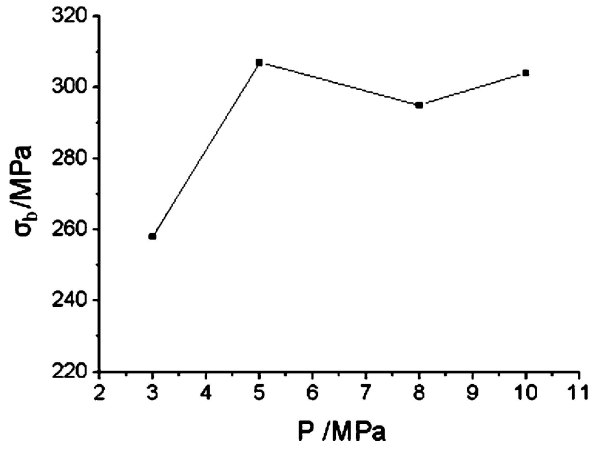


*Figure 6* Dependence of $\sigma_b$ (ultimate tensile strength) of joint on $P$.

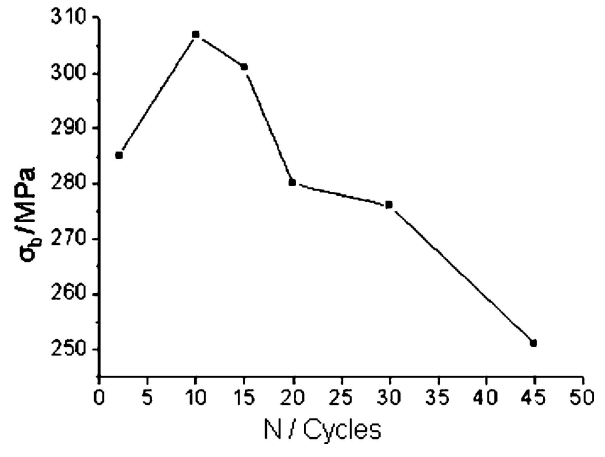


*Figure 4* Dependence of $\sigma_b$ (ultimate tensile strength) of joint on $N$.

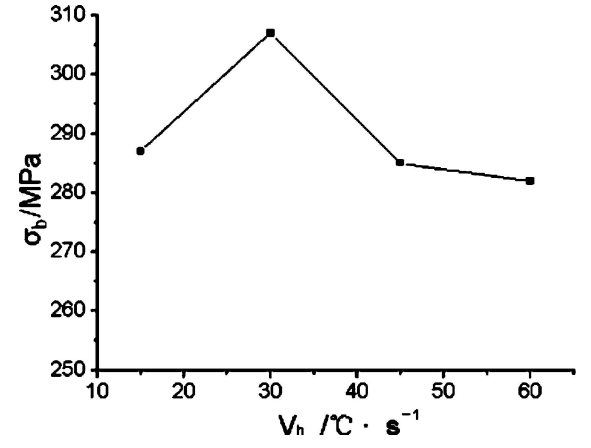


*Figure 7* Dependence of $\sigma_b$ (ultimate tensile strength) of joint on $V_h$.

### 3.2.5. Heating rate, $V_h$

Under a condition of maximum temperature $T_{min}$ = 890°C, minimum temperature $T_{min}$ = 800°C, cyclic number of heating and cooling $N$ = 10, cooling rate $V_c$ = 10°C/s, and specific pressure $P$ = 5 MPa, the dependence of $\sigma_b$ (ultimate tensile strength) of joint on $V_h$ was determined (see Fig. 7). The heating rates were chosen as $V_h$ = 15, 30, 45 and 60°C/s.

From Fig. 7 the suitable heating rate for PTSP diffusion bonding of TA17 to 0Cr18Ni9Ti is 30°C/s.

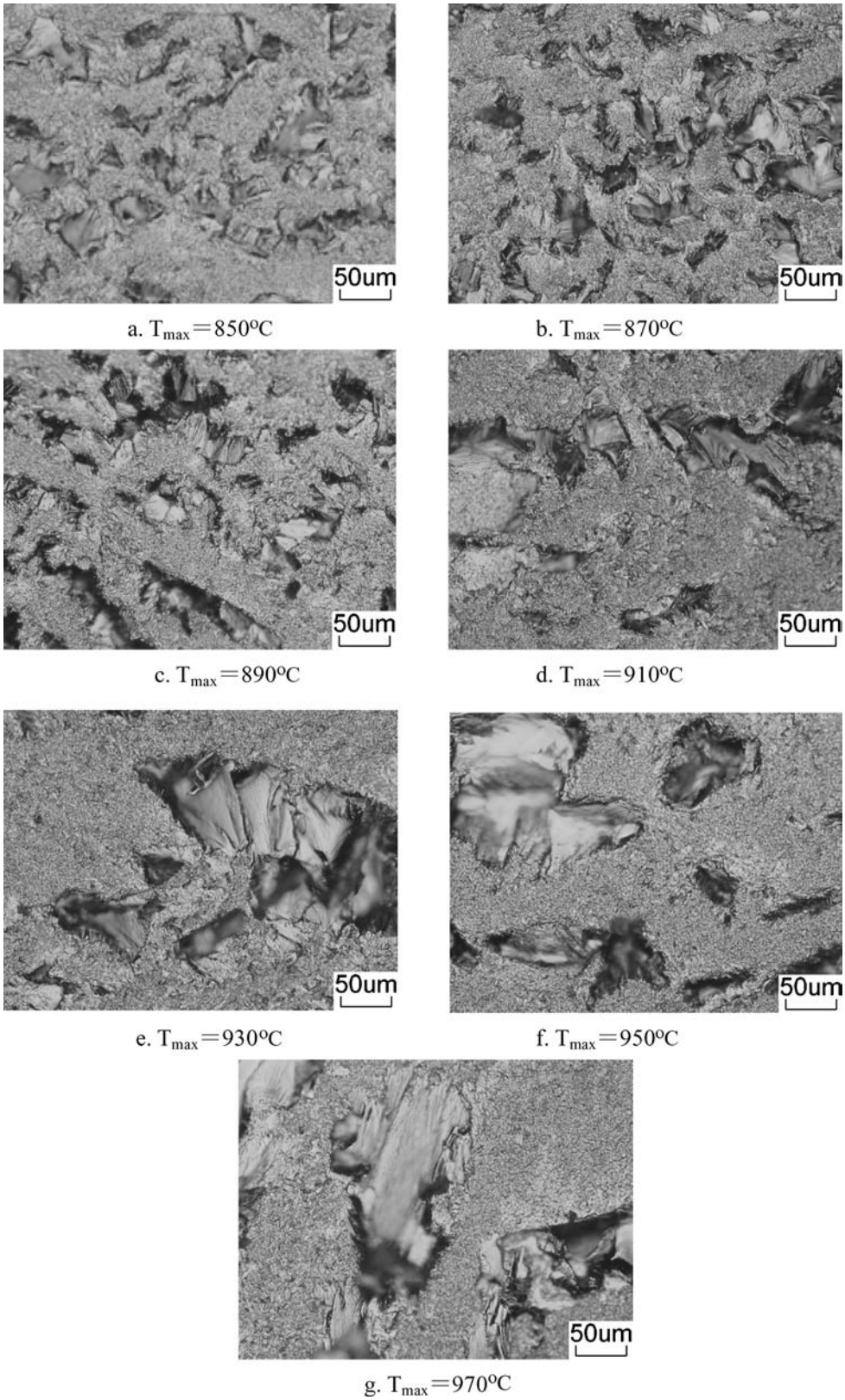


*Figure 8* Fracture interfaces on TA17 side of tensile specimen bonded at different $T_{max}$ ($T_{min}$ = 800°C, $N$ = 30 cycles, $P$ = 5 Mpa, $V_h$ = 30°C/s, $V_c$ = 10°C/s).

### 3.3. Analysis of tensile fracture interfaces

It is well known that the mechanical properties of bonding joint is closely retated to the microstructures formed in the interface of couples. Fig. 8 is the fracture interfaces of tensile specimen on the TA17 side bonded at different $T_{max}$ which reflects the microstructures in the interface. All the fracture surfaces consist of grey matrix and relatively dark blocks with different size and distribution. Generally speaking, with the increment of $T_{max}$, the size of the blocks grows. When $T_{max}$ is below 890°C, the blocks grow very slowly, but at the temperatures higher than 890°C, they become much bigger. The measurement results of imaging analysis showed that the area fraction of the blocks in the fracture surface was dependent on the $T_{max}$, which is shown in Table II.

From Fig. 3 and Table II, one can know that the variation tendency with $T_{max}$ of both the ultimate tensile strength and the area percent of blocks in the fracture surface is generally identical, for example, at 890°C, the ultimate tensile strength and the area percent of blocks reach their maximum value, 276 Mpa and 27.1% respectively.

TABLE II Relationship between area percent of blocks and $T_{max}$

| $T_{max}$/°C | 850 | 870 | 890 | 910 | 930 | 950 | 970 |
|---|---|---|---|---|---|---|---|
| %* | 23.6 | 22.5 | 27.1 | 21.1 | 24.0 | 18.8 | 19.1 |

*Average value of more than 10 measurements at different fields.

In order to investigate the nature of the matrix and blocks in the fracture surface, a scanning electrical microscope (SEM) was used to observe the detail of the facture surface, and the micro-region composition within different part was measured by the energy dispersive spectroscopy (EDS) analysis, see Fig. 9 and Table III, respectively.

Carefully observing Fig. 9, it shows that the matrix has no traces of deformation during tensile testing, it breaks along the original interface, see Fig. 9a,c and e. Correspondently, the blocks show a distinct character of deformation, such as cleavage planes and stages,

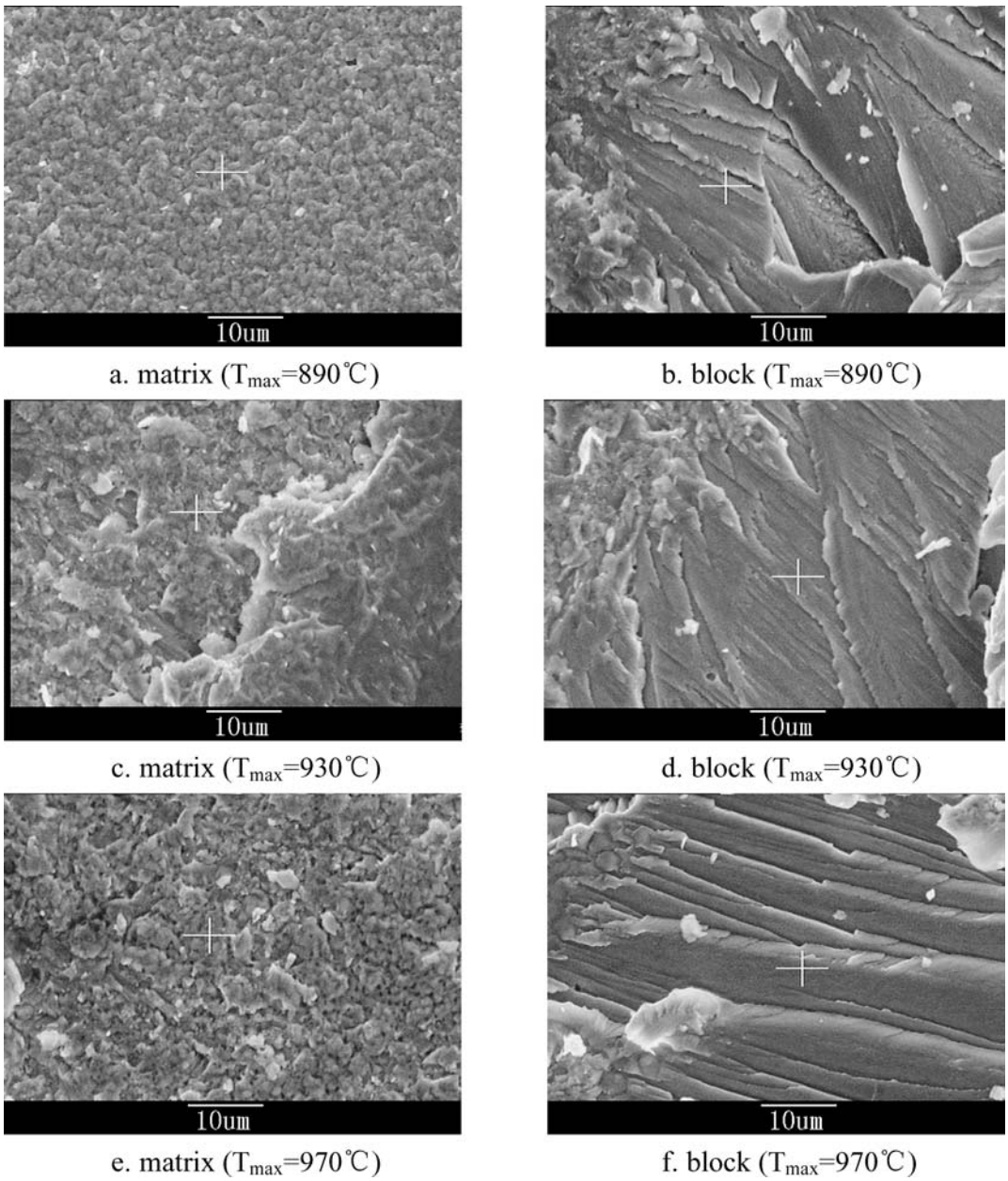


*Figure 9* SEM morphology of matrix and blocks in the fracture interfaces ($T_{min} = 800$°C, $N = 30$ cycles, $P = 5$ Mpa, $V_h = 30$°C/s, $V_c = 10$°C/s).

TABLE III micro-region composition of matrix and blocks (at%)

| | $T_{max} = 890°C$ | | $T_{max} = 930°C$ | | $T_{max} = 970°C$ | |
|---|---|---|---|---|---|---|
| Element | Matrix | Block | Matrix | Block | Matix | Block |
| Ti | 45.21 | 78.04 | 54.58 | 78.24 | 37.17 | 75.79 |
| Fe | 41.95 | 11.66 | 35.12 | 12.59 | 48.87 | 14.20 |
| Cr | 7.65 | 3.09 | 4.29 | 3.40 | 9.37 | 4.75 |
| Ni | 2.96 | 1.06 | 6.01 | 1.37 | 3.98 | 1.86 |

tearing ridges, as well as secondary cracks, see Fig. 9b, d and f. The SEM observations indicated that the tensile load was mainly subjected to the blocks, and the deformation during tensile testing was concentrated in them, which suggested the blocks have a higher jointing strength, whereas the jointing strength of the matrix is much lower.

The analysis results (Table III) of micro-region composition at the point in Fig. 9 marked by a cross showed in the matrix the content of Fe Cr and Ni is higher than those on the block, and the atom percent of Fe has exceeded the solubility of Fe in $\alpha$-Ti and $\beta$-Ti (from the Ti-Fe binary phase diagram, at 900°C, the solubility of Fe in $\alpha$-Ti and $\beta$-Ti is 2–3% and 22–23% in atom percent respectively [11]). The blocks, in the other hand, have the atom percent of Fe within the solubility of Fe in $\beta$-Ti at the maximum bonding temperatures, and it increases with the increment of $T_{max}$, which is identical to the Ti-Fe binary phase diagram. Therefore, it can been concluded that the matrix is mainly the intermetallic compound, FeTi, and the block distributed randomly in the matrix is the solid solution based on $\beta$-Ti. During the range of bonding temperature, by means of the phase transformation superplastic mechanism, the solid solution formed because of the profuse diffusion of $\beta$-Ti stabilizer, such as Fe, Cr and Ni, into titanium alloy and is kept to room temperature. Increasing the $T_{max}$ results in enhancement of diffusion of Fe, Cr and Ni in the TA17 side and that of Ti in the 0Cr18Ni9Ti stainless steel side, and the intermelallic phase, which was formed at lower bonding temperature, grew in size and increase in quantity [3].

From the testing results shown above, the mechanical property of bonding joints is mainly dependent on the size and quantity of solid solution on the interface of the couple bonded. The more in quantity and the smaller in size the solid solutions are, the higher the joint strength. According to the area fraction of solid solution (i.e. the blocks in Table II) and the ultimate tensile strength of joints (see Fig. 3), the ultimate tensile strength of solid solution in the fracture surface can be calculated, which is in the range of 1005 to 1222 MPa. This higher strength was achieved by the solid solution strengthening effect of the $\beta$-Ti stabilizer's diffusion and dissolving in the body center cubic $\beta$-Ti.

The effect of cyclic number of heating and cooling on the microstructure and mechanical property of the joint is similar to that of bonding temperature. The specific pressure, $P$. also plays an important role in the PTSP diffusion bonding (see Fig. 6). Under lower $P$, saying 3 MPa, the contact and diffusion of atoms only occurs in the micro-protuberance of the bonded interface [5], leading to lower joint strength, and when $P$ is larger than 5 MPa, the state of contact becomes better, which increases the joint strength. The heating rate, $V_h$, has an effect of two-side. At relatively lower $V_h$, the creep effect dominates over the superplasticity induced by the phase transformation, and function of the latter does not work very well. When $V_h$ exceeds 30°C/s, the thermo stress resulted from the large difference in the coefficient of linear expansion of two parentl metals develops at their interface [3], which results in the decrease of the joint strength.

## 4. Conclusions

The solid-state direct diffusion bonding experiments of TA17 near $\alpha$-phase titanium alloy to 0Cr18Ni9Ti austenitic stainless steel by means of the phase transformation superplasticity caused by the cycles of heating and cooling have been carried out, and the relationship between the ultimate tensile strength of the joints and technical parameters has been investigated experimentally, as well as the governing factor of the mechanical property of the joint has been determined. From this investigation, the following conclusions can be drawn:

1. When the bonding temperature is cycled between the maximum and minimum temperature for phase transformation of the TA17, the strain response has a character of "ratcheting effect," and it accumulates with the increment of cycles of heating and cooling.
2. Under the conditions of $T_{max} = 890°C$, $T_{min} = 800°C$, cyclic number of heating and cooling $N = 10$ cycles, specific pressure $P = 5$ MPa, heating rate $V_h = 30°C/s$ and cooling rate $V_c = 10°C/s$, the ultimate tensile strength of the joint reached its maximum value (307 MPa), and the bonding time is only 120 s.
3. The results of tensile fracture interface observation show that both the brittle intermetallic compound (FeTi) and the solid solution based on $\beta$-Ti formed on the interface, and the more in quantity and the smaller in size the solid solution are, the higher the ultimate tensile strength is.
4. The Fe content in the solid solution increases with the increment of maximum bonding temperature, and ultimate tensile strength of the solid solution phase lays in the range of 1005 to 1222 MPa.

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*Received 17 February*
*and accepted 20 April 2005*