

Research on superplastic diffusion bonding of 2205 duplex stainless steel

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Abstract

Aiming at the requirement of lightweight and corrosion resistance, superplastic diffusion bonding of 2205 duplex stainless steel was studied. The results showed that during the pre-treatment procedure, with the increase of pre-solution temperature from 1050 to 1350 °C, cold roll reduction from 50 to 85 %, the joint shear strength was improved after bonded under the same condition. The pretreatment of solution at 1350 °C and cold rolled by 85 % was a better choice for the diffusion bonding of 2205 duplex stainless steel. During the bonding process, the critical value of holding time, holding pressure, and bonding temperature were determined as 5 min, 10 MPa, and 1000 °C, before which the joint shear strength was fast increased and after which it was slowly increased or even decreased. Due to the σ -phase dispersed along with the interface after bonded, post-solution treatment was considered, and after post-solution at 1050 °C for 10 min, the best joint shear strength of 685 MPa was obtained.

Key words: stainless steels, bonding, precipitation, scanning electron microscopy (SEM)

1. Introduction

The duplex stainless steel usually has two phases of ferrite and austenite. Thus, it has excellent corrosion resistance and high strength combined with high toughness, and it is commonly known for its excellent performance [1, 2]. During the last several decades, the superplasticity property of duplex stainless steel has become a hot research topic since it can be widely used in the various forming process such as forging, blow-forming, extrusion [3–5]. The first investigation on the superplasticity of duplex stainless steel was reported by Hayden et al. [6]. Then a maximum elongation value of 1510 % was obtained by Sagradi et al. [7]. Diffusion bonding, which is an important solid-state welding process, joins two faying surfaces by holding them at an elevated temperature (0.5–0.8 melting point of the materials) for a certain time under a selected pressure [8, 9]. Microstructure of the specimens and technical parameters dur-

ing the bonding process always affected the welding results and joint properties. For example, Han et al. successfully manufactured a honeycomb structure of Ti-6Al-4V alloy by superplastic forming and diffusion bonding at 930 °C for 60 min under a pressure of 0.6 MPa [10]. Diffusion bonding joints of Ti-6Al-4V alloy and Cr22Ni5Mo3MnSi duplex stainless steel were successfully performed without any discontinuity along with the interface by Velmurugan et al. through low temperature (650–800 °C) diffusion bonding [11]. Sharma et al. [12, 13] used impulse pressure to investigate the diffusion bonding of ferritic stainless steels (Cr11Ni2MnSi), and the results showed that impulse pressure could accelerate the bonding process by grain refinement along with the bonding interface.

For the past few years, with the rapid development of the aviation and marine industry, the superplastic diffusion bonding components have been applied widely due to the advantages of the combining technique [14–16]. The requirement of resistance to

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Table 1. The chemical composition of 2205 duplex stainless steel (wt.%)

Cr	Ni	Mo	Mn	Si	C	S	P	N	Cu	V	Fe
22.05	5.37	3.22	1.10	0.42	0.017	0.006	0.024	0.15	0.043	0.039	67.56

Table 2. The experimental details of the superplastic diffusion bonding

No.	Solution temperature/time (°C/min)	Cold roll reduction (%)	Bonding temperature (°C)	Holding pressure (MPa)	Holding time (min)	Post-solution temperature (°C)	Post-solution time (min)
1	1050/40	85	1000	10	5	–	–
2	1100/40	85	1000	10	5	–	–
3	1150/40	85	1000	10	5	–	–
4	1200/40	85	1000	10	5	–	–
5	1250/40	85	1000	10	5	–	–
6	1300/40	85	1000	10	5	–	–
7	1350/40	85	1000	10	5	–	–
8	1350/40	50	1000	10	5	–	–
9	1350/40	60	1000	10	5	–	–
10	1350/40	70	1000	10	5	–	–
11	1350/40	80	1000	10	5	–	–
12	1350/40	85	1000	10	1	–	–
13	1350/40	85	1000	10	2	–	–
14	1350/40	85	1000	10	3	–	–
15	1350/40	85	1000	10	7	–	–
16	1350/40	85	1000	10	10	–	–
17	1350/40	85	1000	2	5	–	–
18	1350/40	85	1000	5	5	–	–
19	1350/40	85	1000	7	5	–	–
20	1350/40	85	1000	20	5	–	–
21	1350/40	85	900	10	5	–	–
22	1350/40	85	950	10	5	–	–
23	1350/40	85	1050	10	5	–	–
24	1350/40	85	1100	10	5	–	–
25	1350/40	85	1150	10	5	–	–
26	1350/40	50	3 cycles	10	5	–	–
27	1350/40	60	3 cycles	10	5	–	–
28	1350/40	70	3 cycles	10	5	–	–
29	1350/40	80	3 cycles	10	5	–	–
30	1350/40	85	3 cycles	10	5	–	–
31	1350/40	85	1000	10	5	1350	3
32	1350/40	85	1000	10	5	1350	5
33	1350/40	85	1000	10	5	1350	7
34	1350/40	85	1000	10	5	1350	10
35	1350/40	85	1000	10	5	1350	15
36	1350/40	85	1000	10	5	1350	20
37	1350/40	85	1000	10	5	1050	10
38	1350/40	85	1000	10	5	1100	10
39	1350/40	85	1000	10	5	1150	10
40	1350/40	85	1000	10	5	1200	10
41	1350/40	85	1000	10	5	1250	10
42	1350/40	85	1000	10	5	1300	10

chloride corrosion for materials used in the field of ship and ocean development machinery has garnered more extensive attention on the application of diffusion bonding in duplex stainless steel [17–19]. Ridley et al. [20] and Islam et al. [21] just reported

similar diffusion bonding of Cr25Ni7Mo3MnCu and Cr22Ni5Mo3MnCu duplex stainless steels, respectively. Yeh et al. [22] improved the welding properties of duplex stainless steel (Cr23Ni6MoMnCu) by superplastic diffusion bonding. Noticeably, based on

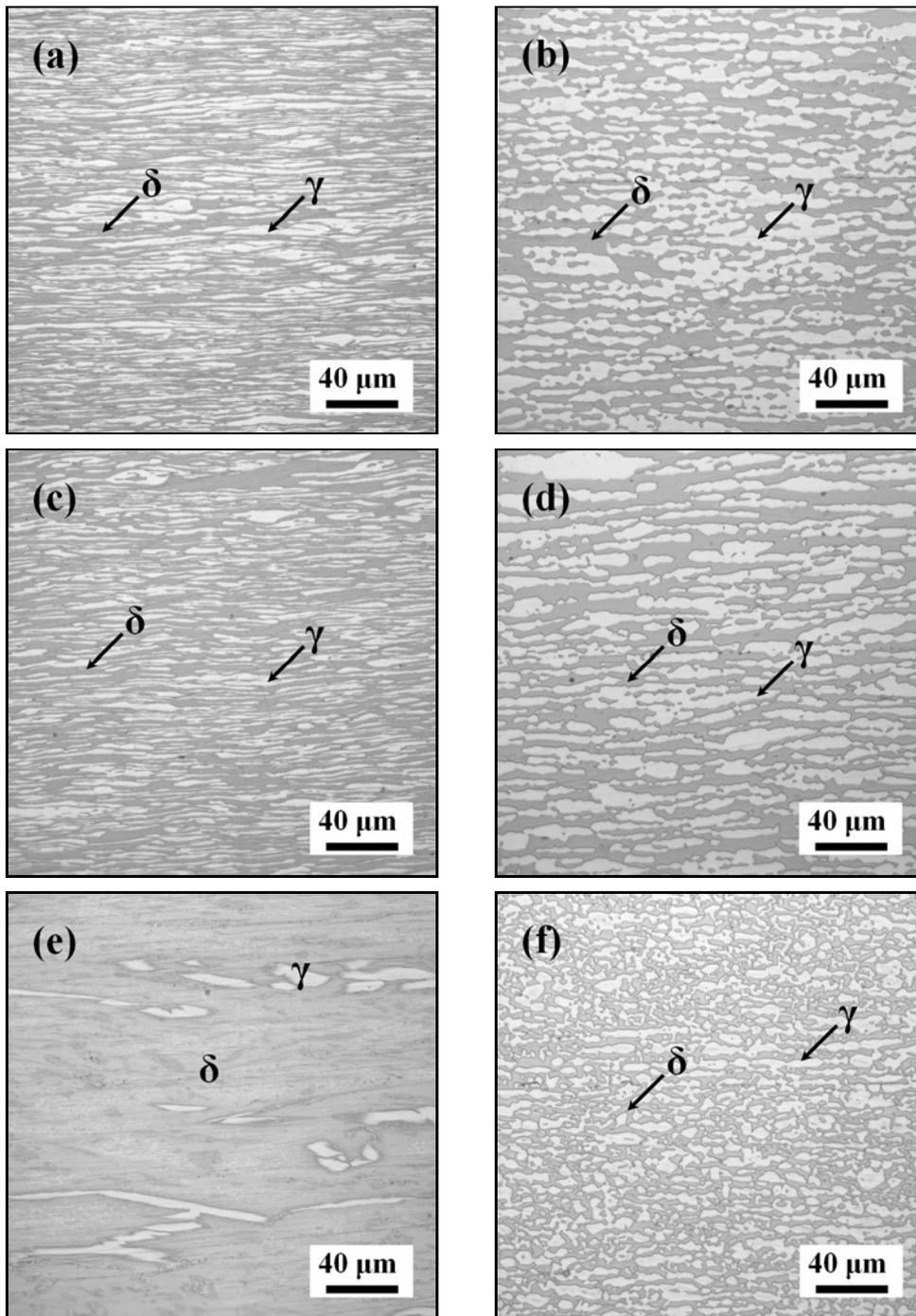


Fig. 1. Microstructures after cold rolled with a reduction of 85% and after diffusion bonded under the pre-solution temperature of 1050 °C (a, b), 1150 °C (c, d), and 1350 °C (e, f).

the literature review, only Komizo et al. [23] briefly studied the superplastic diffusion bonding between Cr18Ni4Mn3CuSi, SAE2205, and Cr25Ni7Mo3MnCu duplex stainless steels. However, the study and analysis of the diffusion bonding of duplex stainless steels are few and unsystematic.

Based on the above, in this paper, superplastic diffusion bonding of 2205 duplex stainless steel was studied systematically, effects of pretreatment and techni-

cal parameters during the bonding process were discussed, and methods of improving the joints' performance were put forward.

2. Experimental material and procedures

The duplex stainless steel used in this research was SAE2205, whose chemical compositions are listed in

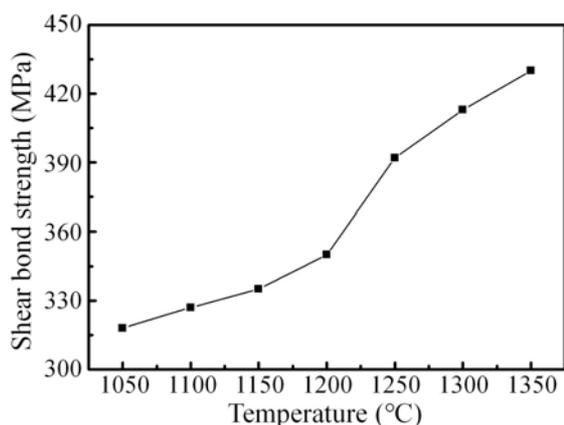


Fig. 2. The evolution of joint shear bond strength with pre-solution temperature.

Table 1. The 2205 duplex stainless steels as received were then solution-treated at 1050–1350 °C for 40 min using resistance furnace (SRJX-8-13A) having an accuracy of ± 5 °C, followed by water quenching, and temperature interval was 50 °C. They were then cold-rolled into thinner sheets by reduction of 50, 60, 70, 80, and 85 %. Specimens used for superplastic diffusion bonding were cut along the rolling direction with the dimensions of $15 \times 10 \text{ mm}^2$ by using an electric discharge machine. All the diffusion bonding experiments were carried out on the Gleeble-1500 thermal simulator and in the Ar protective atmosphere. Experimental steps were as follows: the specimens with the same pre-solution treatment were heated to the setting temperatures (900, 950, 1000, 1050, 1100, and 1150 °C) at a rate of 20 °C s^{-1} , followed by adding the setting pressure (2, 5, 7, 10, and 20 MPa) in 5 s and holding at this pressure for different times (1, 2, 3, 5, 7, and 10 min). To explore the effects of pretreatment on diffusion bonding, specimens with different pretreatment state were bonded under the same experimental condition. To improve the shear bond strength of the joints, during the bonding process, the temperature was changed between 1000 and 900 °C for 3 cycles, each cycle was 100 s, when the bonding time was 5 min and holding pressure was 10 MPa. The post-solution treatment was performed between 1050 and 1350 °C for 3–20 min using the resistance furnace. The experimental details are listed in Table 2.

After the diffusion bonding experiments, the shear test was carried out on the WDW-50E microcomputer controlled electronic universal testing machine at a constant speed of 10 mm min^{-1} . Shear bond strength of two specimens at least was tested for each condition, and the average value was calculated. The detailed dimensions of the shear test specimens were illustrated in [24, 25], which ensured the fracture along with the bonding interface. For observing microstructure after experiments, the specimens were cut per-

pendicular to the bonding interface along the rolling direction. Metallographic samples were prepared by conventional metallographic polishing and etching in a NaOH electrolyte (40 g NaOH, 100 ml water) with a voltage of 6 V for approximately 9 s. Thin foils with a thickness of 0.5 μm for observation with a transmission electron microscope (TEM) were machined from the bonding surface. These foils were thinned to 40 μm by sandpaper before cut into $\varnothing 3 \text{ mm}$ wafers, and then were twin jet electro polished in 10 ml $\text{HClO}_4 + 90 \text{ ml C}_2\text{H}_5\text{OH}$ solution, and then examined in the Tecnai G2 F30 operating at 300 kV.

3. Results and discussion

3.1. Effect of pretreatment on superplastic diffusion bonding of 2205 duplex stainless steel

3.1.1. Effect of pre-solution temperature

After pre-solution treated under different temperatures, Fig. 1 shows the microstructures after cold rolled with a reduction of 85 % and after diffusion bonded by holding at a pressure of 10 MPa under 1000 °C for 5 min. In the optical metallographs, γ and δ were distinguished by the contrast of white and dark, respectively. Figure 2 showed the evolution of joint shear bond strength with pre-solution temperature. Seen from Figs. 1a,c,e, with the cold rolling reduction, the content of ferrite and austenite in 2205 duplex stainless steel changed with the increase of pre-solution temperature. As pre-solution temperature became higher, ferrite content became more. After diffusion bonding, recrystallization occurred in the specimens. There was a clear straight line dispersed with many voids after diffusion bonded (Fig. 1b), indicating the position of bonding interface. In this case, the shear bond strength was lower (Fig. 2). With the increase of pre-solution temperature, the weld-line gradually became less pronounced, voids amount became lower, and most of these voids were in the interior of ferrite or austenite (Fig. 1d); meanwhile, the shear bond strength also increased. As shown in Fig. 1f, when the pre-solution temperature was 1350 °C, the bonding interface was clean and almost did not contain any voids, and the shear bond strength became better. Thus, joint shear bond strength increased from 318 to 430 MPa along with the increment of pre-solution temperature increased from 1050 to 1350 °C.

3.1.2. Effect of cold roll reduction

After pre-solution treated at 1350 °C, specimens with different cold roll reduction were bonded by holding at a pressure of 10 MPa under 1000 °C for 5 min,

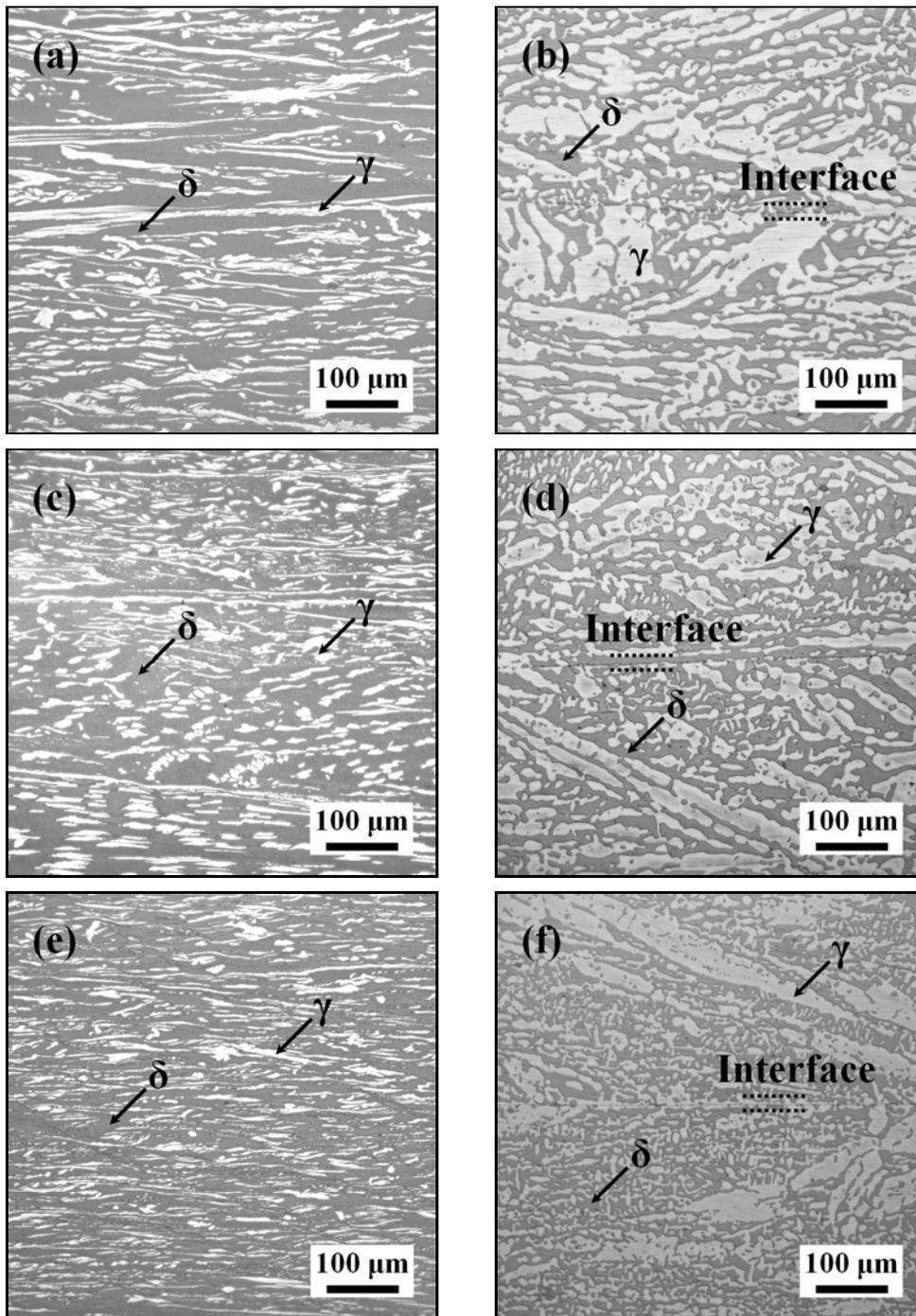


Fig. 3. Microstructures after cold-rolled and after diffusion bonded with the reduction of 60 % (a, b), 70 % (c, d), and 80 % (e, f).

and the microstructures before and after diffusion bonded are shown in Fig. 3. Figure 4 shows the evolution of joint shear bond strength along with the increment of cold roll reduction. Seen from Fig. 3, finer $\gamma + \delta$ microstructure was obtained when the cold roll reduction increased both before and after bonding. However, recrystallization could be observed, similar to the results of 3.1.1., and there were voids dispersed along the bonding interface. The larger cold rolling reduc-

tion led to fewer voids and a more uniform microstructure, which is shown in Figs. 3b,d,f. When the cold roll reduction was 85 %, the voids almost disappeared, and the two specimens were nearly integrated into one after diffusion bonded (Fig. 1f). The microstructure evolution illustrated the reason why the joint shear bond strength increased from 329 to 430 MPa when the cold roll reduction increased from 50 to 85 %, as can be seen in Fig. 4.

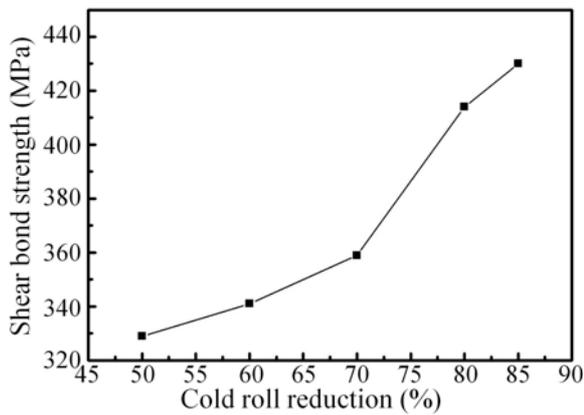


Fig. 4. The evolution of joint shear bond strength with cold roll reduction.

3.2. Effects of diffusion bonding parameters

It was well known that the most important parameters during the superplastic bonding process are holding time, holding pressure, and bonding temperature. Thus, specimens with the same pretreatment of $1350^{\circ}\text{C} + 85\%$ were used to study the effect of the three parameters. Figure 5 shows the influence of a single variable on the joint shear bond strength, while the other two parameters were fixed during the bonding process.

As shown in Fig. 5, shear bond strength of joint increased when prolonging the holding time, increasing the holding pressure, and raising the bonding temperature. Under these conditions, the bonding line gradually became indistinct, and the void distributed along with the interface gradually decreased, which can be seen in Fig. 6. However, after the holding time reached 5 min, and the pressure added to 10 MPa, shear bond strength became stable with the increment of the holding and pressure (Figs. 5a,b). Meanwhile, when the bonding temperature was higher than 1050°C , the increase of bonding temperature would decrease the shear bond strength (Fig. 5c), and the coarser microstructure was observed (Fig. 6f).

The fracture surface after holding for a short time and less pressure exhibited inhomogeneous dimples where some of them were pretty shallow (Figs. 7a–c), indicating unbonded areas, which indicated that interface combination of the joints was weaker. With an increase in holding time up to 5 min and holding pressure up to 10 MPa, as shown in Fig. 7d, the dimples became more and more homogeneous, meaning that interface combination was strengthened. Also, the σ -phase precipitation distributed along with the bonding interface, which acts as void nucleation sites, was observed inside the dimples and was identified by the

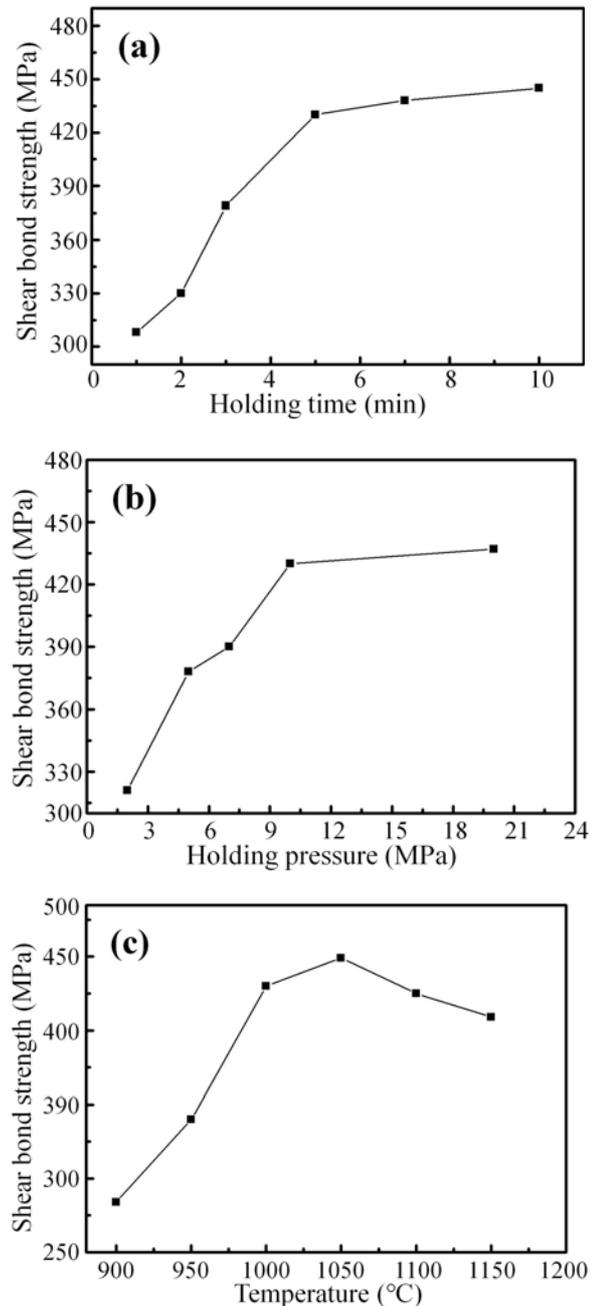


Fig. 5. Evolution of joint shear bond strength along with holding time (a), holding pressure (b), and bonding temperature (c) while the other two were fixed as $1000^{\circ}\text{C} + 10\text{ MPa}$, $1000^{\circ}\text{C} + 5\text{ min}$, and $10\text{ MPa} + 5\text{ min}$, respectively.

energy dispersed spectrum (Fig. 8). Meanwhile, as shown in Fig. 7e, when the bonding temperature was 900°C , there were lots of σ -phase with larger size dispersed at the interface. In Fig. 7f, after bonded under higher temperature, the dimples became bigger, and the joint shear bond strength decreased.

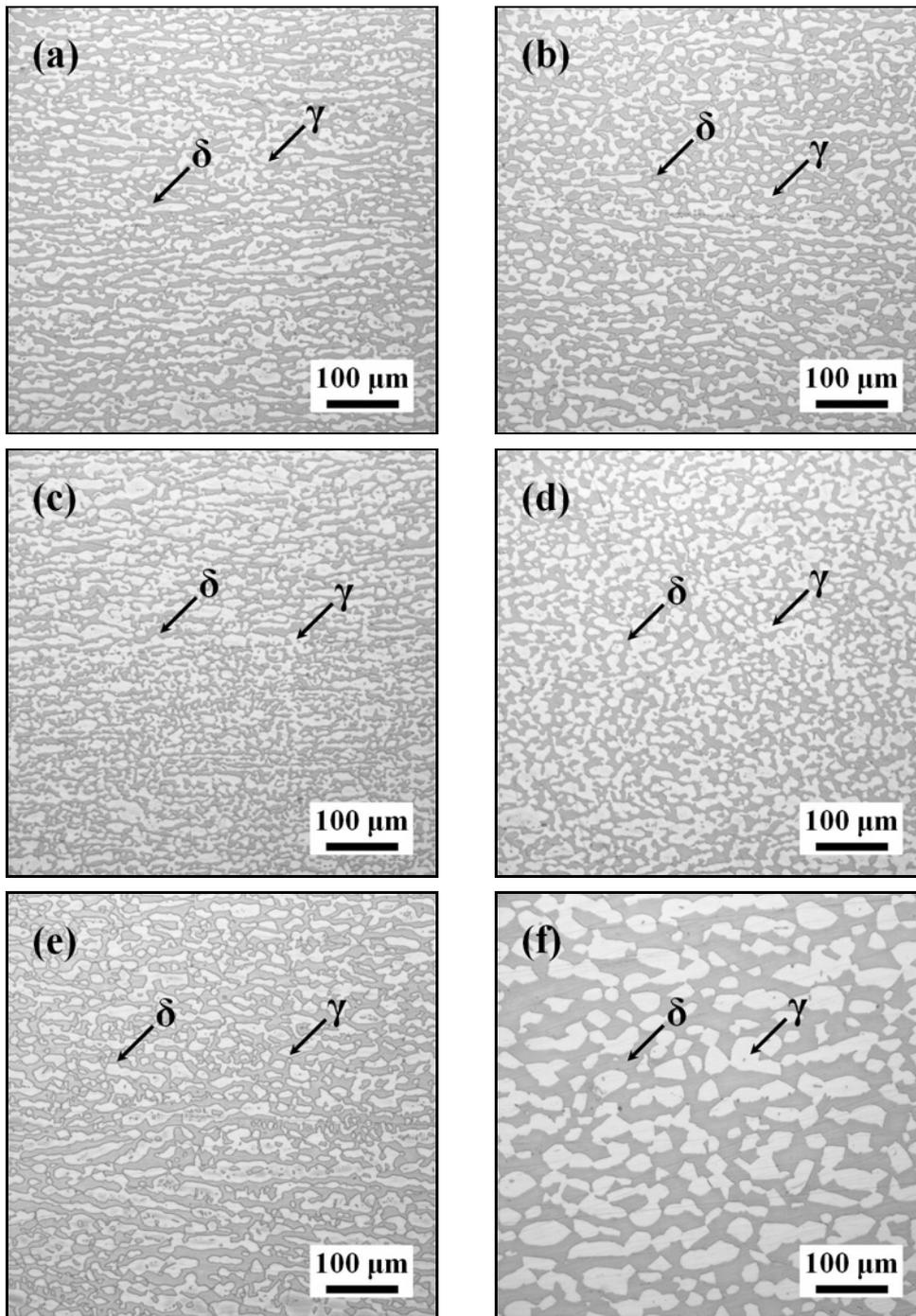


Fig. 6. Microstructures after diffusion bonded changing a single variable of (a) 2 min, (b) 7 min, (c) 5 MPa, (d) 20 MPa, (e) 1050°C, and (f) 1150°C.

3.3. Improvement of bonding interface in 2205 diffusion bonding joints

3.3.1. Improvement during the bonding process

Using the same specimens diffusion bonding test, when the holding pressure was both 10 MPa, after variation between 1000 and 900°C for 3 cycles during

5 min, shear bond strength was all improved compared with results of 3.1.2. in which the joints were bonded under a constant temperature 1000°C for 5 min, as shown in Fig. 9. When the cold roll reduction was 85 %, the shear bond strength was improved up to 625 MPa during the changing temperature bonding process, which was a 45 % increment compared to that of 430 MPa under the constant temperature condition.

Metallurgical structure and TEM graphs of the

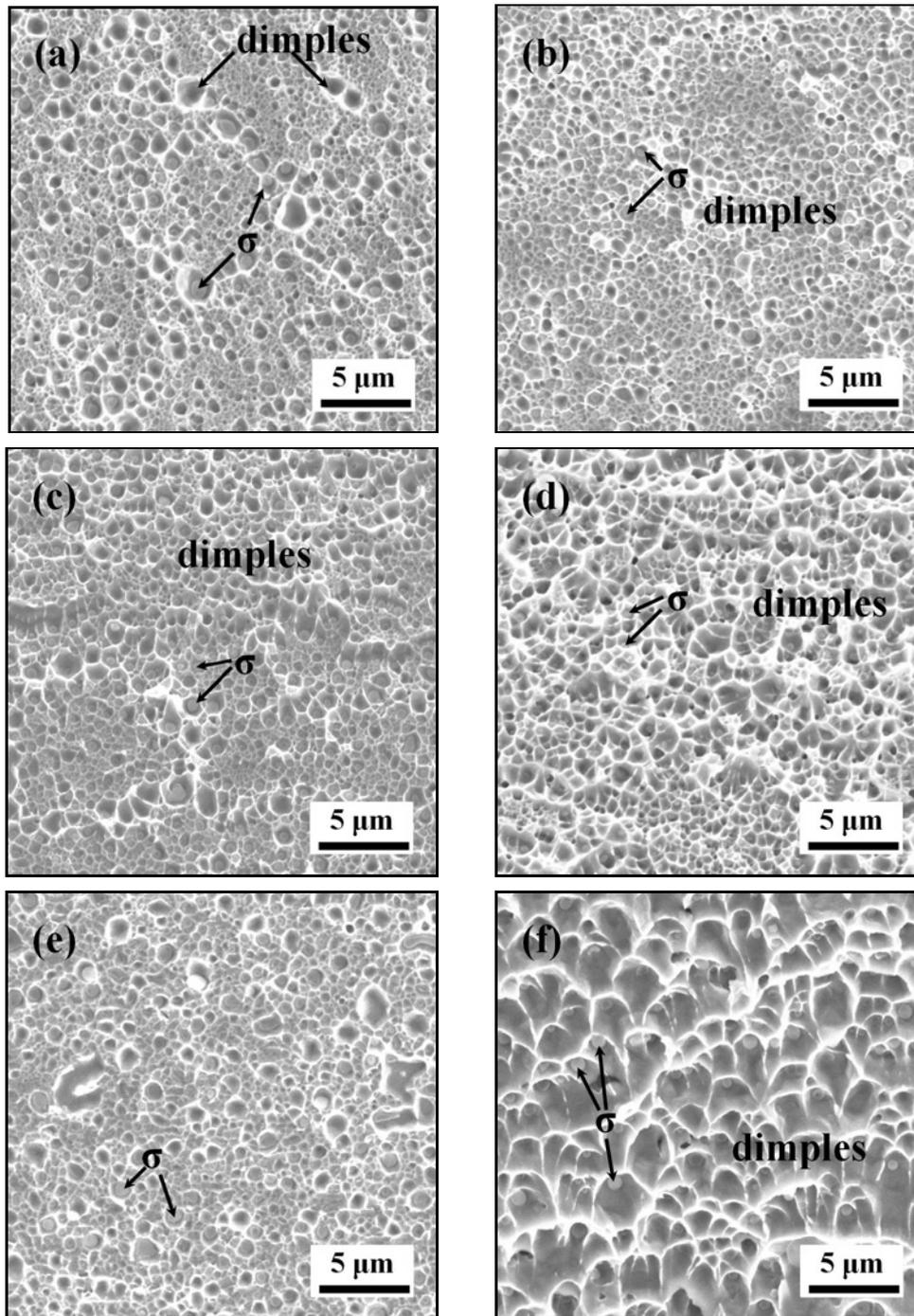


Fig. 7. The fracture surfaces after bonded by changing a single variable of (a) 2 min, (b) 3 min, (c) 5 MPa, (d) 5 min + 10 MPa, (e) 900 °C, and (f) 1150 °C.

joints bonded under the two conditions were observed, as can be seen in Fig. 10. The average grain size of the sample bonded at constant temperature is 2.44 μm (Fig. 10a), while the average grain size of the sample bonded at changed temperature is 1.54 μm (Fig. 10b) measured by the intercept method. Compared with the experiments carried out under constant temperature (Figs. 10a,c), when there was temperature changing during the bonding process, the activity of atoms

and grain boundaries was improved, microstructure and grains were refined (Figs. 10b,d). These were favorable for the bonding process, and this was the reason why the shear bond strength of the joint increased.

3.3.2. Improvement by post-solution treatment

According to above investigation, a joint with a shear strength of 430 MPa could be obtained when

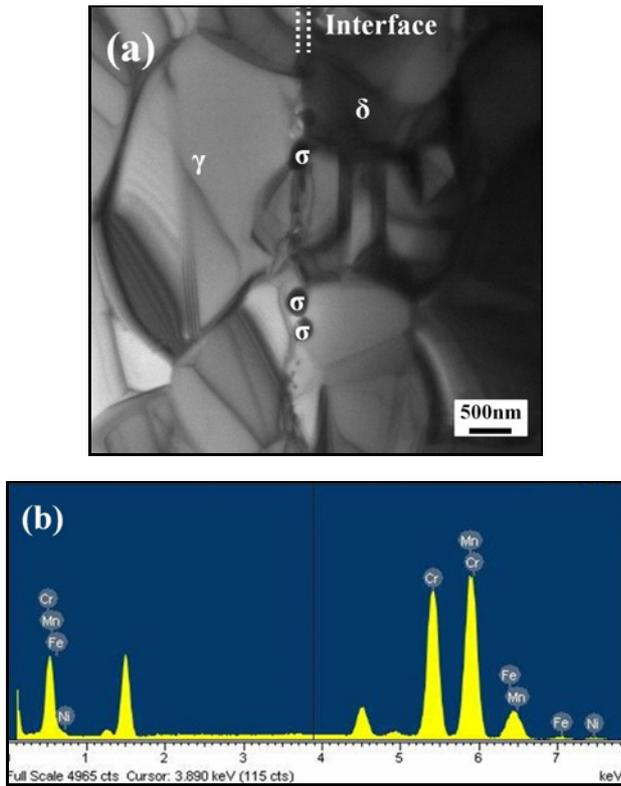


Fig. 8. The σ -phase precipitation distributed along with the bonding interface after bonded under the condition of 5 min + 10 MPa + 1000 °C.

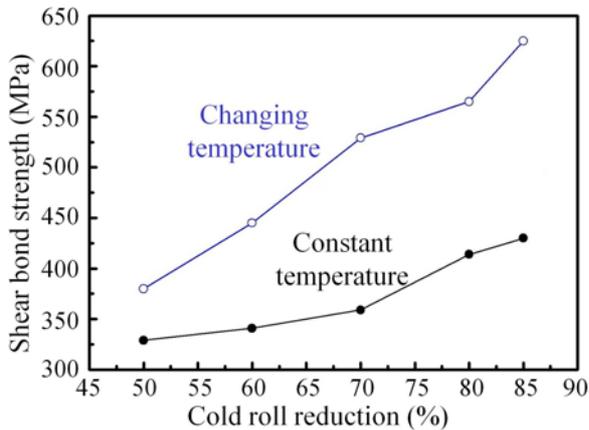


Fig. 9. Shear bond strength of the joints after bonded at constant temperature and changing temperature while the holding time was 5 min and the pressure was 10 MPa.

the diffusion bonding processed by holding at a pressure of 10 MPa for 5 min under 1000 °C, and σ precipitation distributed along the bonding interface could be observed clearly, which was harmful to the joint shear strength. Thus post-solution treatment was considered. Two parameters of solution temperature and

solution time were studied, and their effects on the shear bond strength were shown in Figs. 11, 12. Compared with 430 MPa after diffusion bonded, shear bond strength of the joints after post-solution treated was all improved.

Figure 13 shows the ductile fracture in all conditions. The σ -phase was observed inside the dimples only when the solution time was between 0 and 7 min (Figs. 13a,b). Also, with an increase in solution time from 0 to 7 min, the number and size of the σ -phase were reduced. When the holding time reached 10 min and above, there was almost no observation of the σ -phase (Figs. 13c,d). These were corresponding to the variation of shear strength shown in Fig. 11. When the solution time increased from 0 to 10 min, the shear strength sharply increased from 430 to 530 MPa. When the solution time continuously increased up to 20 min, the shear strength slowly increased only up to 538 MPa because of almost full partitioning by holding for 10 min. Thus, 10 min for solution treatment was selected in the post-solution temperature experiments.

As shown in Fig. 12 and Figs. 14b,d,f, after post-solution treated under 1050–1350 °C for 10 min, the shear bond strength was significantly increased than that of after diffusion bonded, predominantly due to the dissolution of σ -phase. However, when the temperature continuously increased from 1050 to 1350 °C, the shear strength slowly decreased due to the microstructure coarsening (Figs. 14a,c,e). When the post-solution temperature was 1050 °C, the shear strength was increased up to 685 MPa.

4. Discussion

Based on the above experiments, shear bond strength of 2205 duplex stainless could be affected by pre-solution treatment, cold roll reduction, technological parameters during the bonding process, and post-solution treatment.

4.1. Analysis of pretreatment

When the pre-solution temperature increased from 1050 to 1350 °C, the phase ratio between ferrite and austenite had been changed. As can be seen in Figs. 1a,e, ferrite content increased along with the increment of pre-solution temperature, and ferrite had better plasticity than austenite. During the following bonding process, when a certain pressure was given, plastic deformation was easier to occur, which could facilitate effective physical contact of the two specimens. Also, recrystallization occurred during the bonding process. Finer γ_{new} -phase formed in the original ferrite phase, and phase transformation of $\delta \rightarrow \gamma_{\text{new}}$ occurred. The newly formed $\gamma + \delta$ mi-

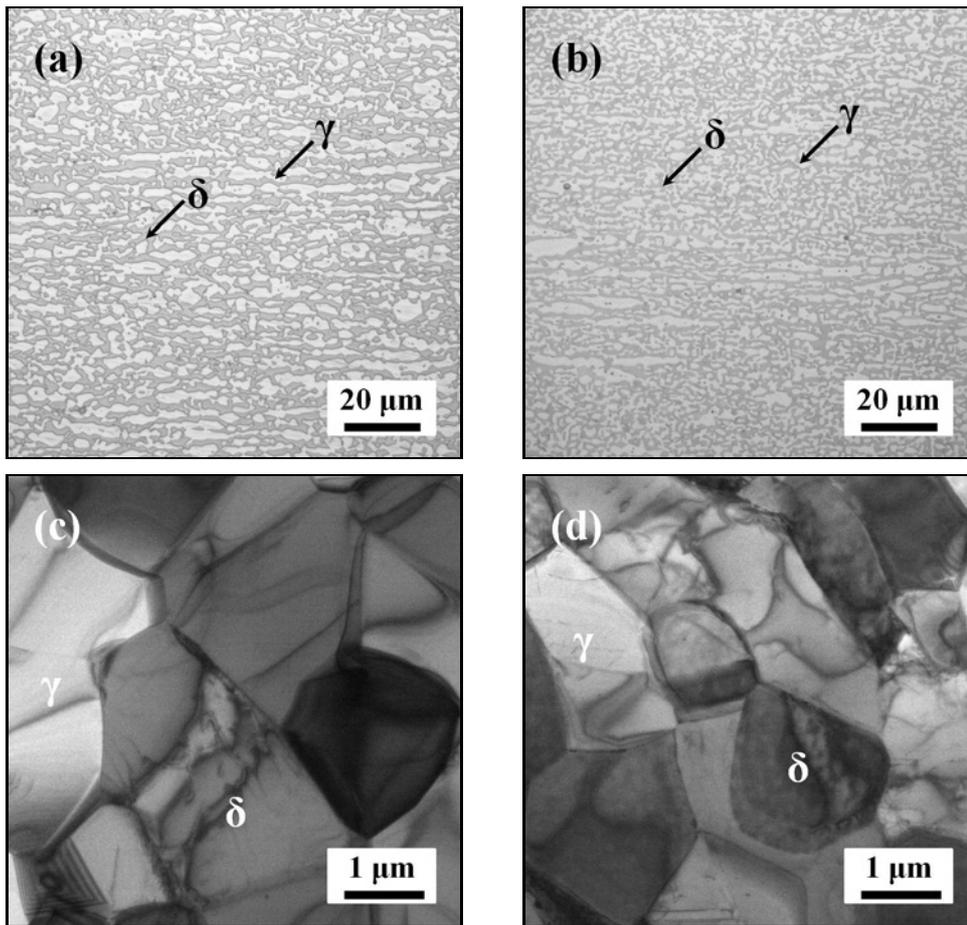


Fig. 10. Metallurgical structure and TEM graphs of the joints bonded at constant temperature (a), (c), and changing temperature (b), (d).

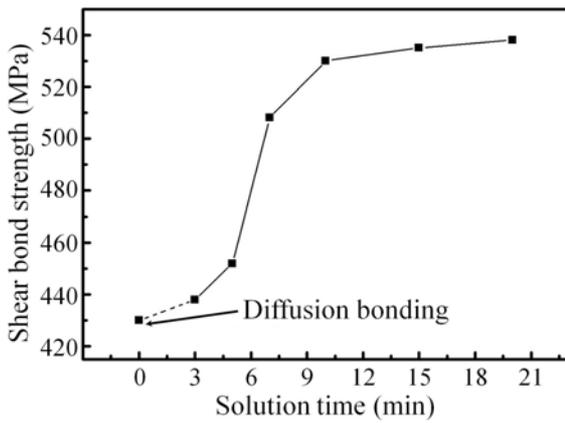


Fig. 11. Shear bond strength after post-solution treated for a different time at 1350°C.

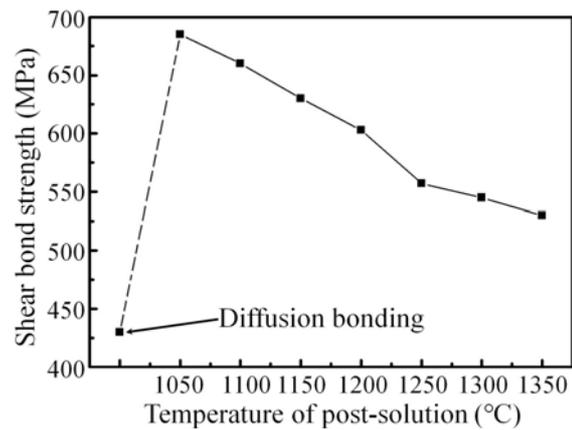


Fig. 12. Shear bond strength after post-solution treated under different temperatures for 10 min.

crostructure was more uniform. This would be made the grain rotation and grain boundary sliding occur easier, which would provide convenience to the generation of the continuous interface. Thus, shear bond strength could be increased. Similar to these processes and performance, the larger the cold roll re-

duction was, the finer the $\gamma + \delta$ microstructure was after the bonded experiment. This was favorable for the bonding process. Furthermore, when the cold roll reduction was bigger, there was more energy stored in the microstructure, which was beneficial to recrystallization during the following bonding process. Thus,

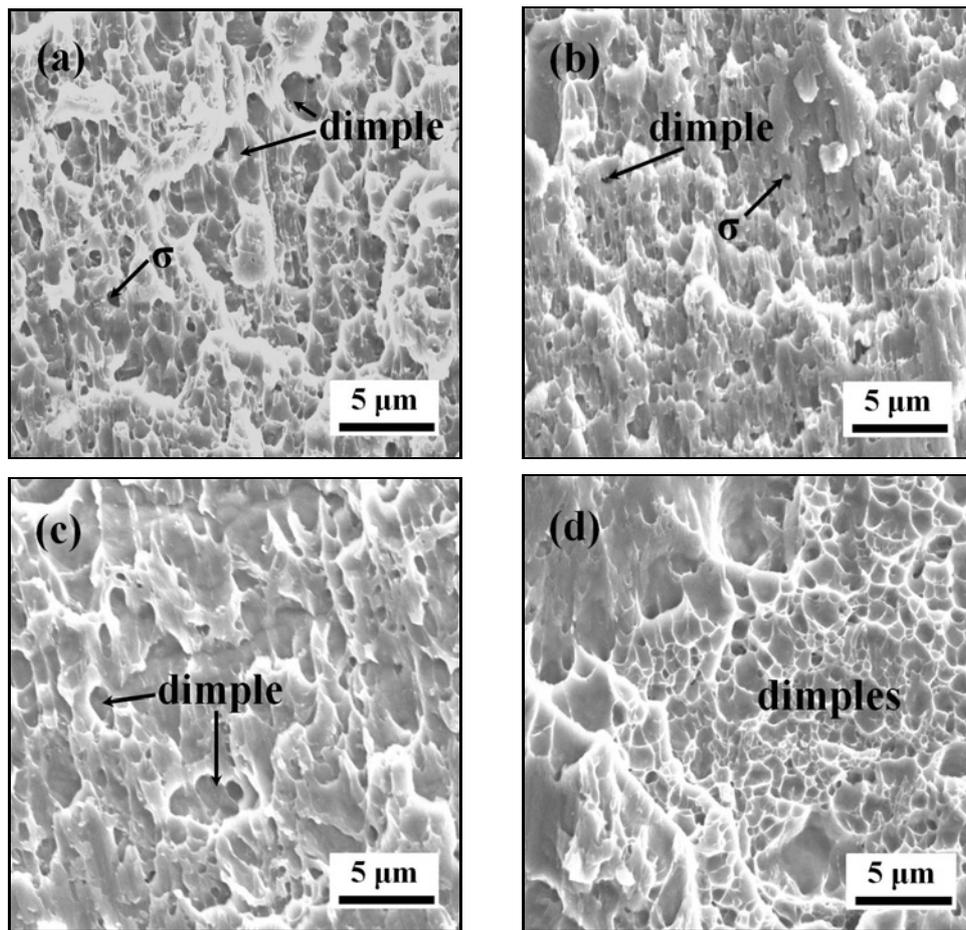


Fig. 13. Fracture after post-solution treated for (a) 3 min, (b) 7 min, (c) 10 min, and (d) 20 min at 1350°C.

based on these experiments, the pretreatment of solution treated at 1350°C and cold rolled by the reduction of 85% was a better choice for the diffusion bonding of 2205 duplex stainless steel.

4.2. Analysis of technological parameters during the diffusion bonding process

According to the results in 3.2, three parameters of holding time, holding pressure, and bonding temperature could affect the shear bond strength of the joints. During holding at a certain pressure, the creep/superplasticity induced and accelerated grain boundary migration [26]. In these experiments, there were voids along grain boundaries when the holding time was short (Fig. 6a), and the holding pressure was lower (Fig. 6c). The voids were in the interior of ferrite and/or austenite when the holding time and pressure increased (Figs. 6b,d). This grain boundary migration was beneficial to healing the bonding interface and reducing voids along with the interface [27]. Thus, the joint shear strength of 430 MPa could be obtained when the holding time was 5 min, and the pressure was 10 MPa. Both the size and amount of voids

were decreased. A similar phenomenon was reported in [27, 28]. As to the bonding temperature, it should be set above 1000°C since there was a large amount of σ -phase with a higher dimension (Fig. 7e), which was harmful to the bonding interface. The shear bond strength increased from 284 to 449 MPa (Fig. 5c). However, after the holding time and pressure were increased up to 5 min and 10 MPa, the increment of these two had little contribution to the shear bond strength. Meanwhile, due to the microstructure coarsening, shear bond strength even decreased when the temperature changed from 1050 to 1350°C. Therefore, during the 2205 diffusion bonding process, a great selection would be held for 5 min at 1000°C under the pressure of 10 MPa.

4.3. Analysis of improvement to shear bond strength

Since the microstructure refinement during the bonding process and the σ precipitation dispersed in the bonding interface had a significant influence on the shear strength, methods of grain refining and post-solution treatment were considered.

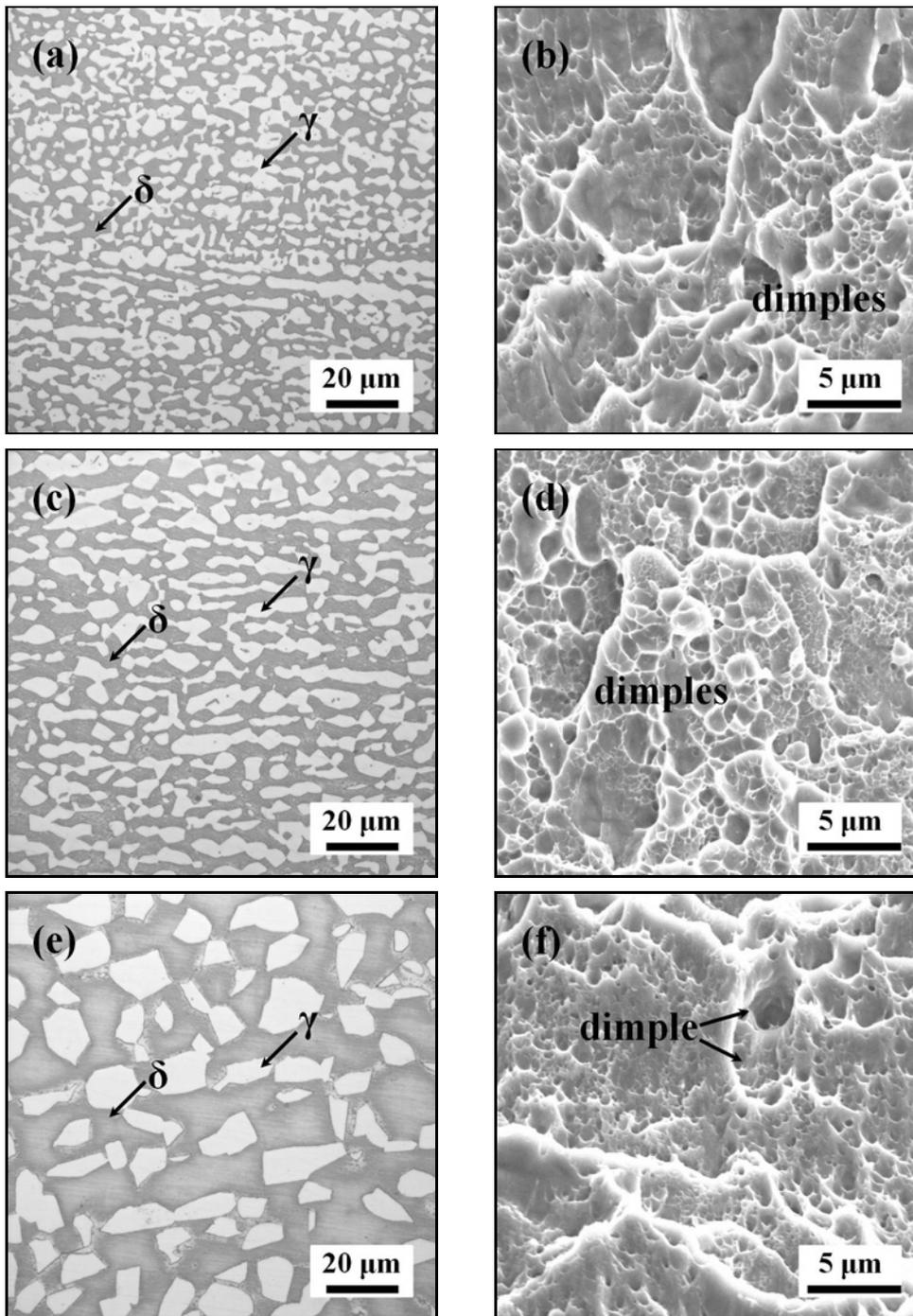


Fig. 14. Microstructure and fracture after post-solution treated for 10 min at (a, b) 1050°C, (c, d) 1100°C, and (e, f) 1250°C.

Based on the above experiments, during the superplastic diffusion bonding process, recrystallization occurred, and fine $\gamma + \delta$ microstructure was obtained. After changing temperature was introduced to the bonding process, all joints showed better performance, according to 3.3.1. The reason was that when the temperature was changed, more phase transformation occurred between γ and δ , and more fine and uniform grains were obtained (Figs. 10b,d). Due to the

change of temperature, the activity of atoms and grain boundaries was improved. The respective motion of grain boundary and finer grains under the condition of changing temperature formed a greater bonding interface and hence increased the shear bond strength of the joints.

To solve the problem that there was σ precipitation dispersed in the bonding interface, post-solution treatment was carried out. When the solution tem-

perature was at 1350°C, increasing the holding time from 0 to 10 min, the shear bond strength was significantly increased from 430 to 530 MPa. When prolonging the holding time up to 20 min, the shear strength became stable due to that σ -phase was fully dissolved in 10 min, shown in Figs. 11, 13. Meanwhile, since the microstructure became coarser (Fig. 14) when post-solution treated temperature varied between 1050 and 1350°C and shear strength decreased. Thus, the post-solution at 1050°C for 10 min was the best choice.

5. Conclusions

The effect of pretreatment, technological parameters during the bonding process on 2205 duplex stainless steel was studied, followed by research on the improvement of joint shear strength through post-solution treatment, and led to the following conclusions:

1. During the pretreatment procedure, with an increase in pre-solution temperature from 1050 to 1350°C, cold roll reduction from 50 to 85%, the joint shear strength was improved after bonded under a completely the same condition. The pretreatment of solution at 1350°C and cold rolled by 85% was a better choice for the diffusion bonding of 2205 duplex stainless steel.

2. During the bonding process, with an increase in the holding time, holding pressure, and bonding temperature, the joint shear strength was improved through local plastic deformation, void shrinkage by diffusion, and grain boundary migration across the bonding interface. Also, the critical values of holding time, holding pressure, and bonding temperature were determined as 5 min, 10 MPa, and 1000°C, before which the joint shear strength was fast increased and after which it was slowly increased or even decreased.

3. With the temperature changing during the bonding process while holding time and pressure remain unchanged, the activity of atoms and grain boundaries was improved, the microstructure was refined, and hence the joints shear strength was increased.

4. The joints shear bond strength decreased with the increment of temperature from 1050 to 1350°C due to the microstructure coarsening, increased fast before holding 10 min, and slowly after 10 min since the σ -phase was fully dissolved in 10 min. Thus, post-solution at 1050°C for 10 min achieved the best joint shear strength of 685 MPa.

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