

A two-step transient liquid phase diffusion bonding process of T91 steels *

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Abstract In this study, a two-step heating process is introduced for transient liquid phase (TLP) diffusion bonding for sound joints with T91 heat resistant steels. At first, a short-time higher temperature heating step is addressed to melt the interlayer, followed by the second step to complete isothermal solidification at a low temperature. The most critical feature of our new method is producing a non-planar interface at the T91 heat resistant steels joint. We propose a transitional liquid phase bonding of T91 heat resistant steels by this approach. Since joint microstructures have been studied, we tested the tensile strength to assess joint mechanical property. The result indicates that the solidified bond may contain a primary solid-solution, similar composition to the parent metal and free from precipitates. Joint tensile strength of the joint is not lower than parent materials. Joint bend's strengths are enhanced due to the higher metal-to-metal junction producing a non-planar bond lines. Nevertheless, the traditional transient liquid phase diffusion bonding produces planar ones. Bonding parameters of new process are 1 260 °C for 0.5 min and 1 230 °C for 4 min.

Key words T91, transient liquid phase diffusion bonding, two-step heating process, scanning electron microscopy

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0 Introduction

The T91 heat resistant steels is a readily available industrial material (stabilized with vanadium and niobium additions), used as a steam generator material for power plants. It bears high strength, low thermal stress and low ductile-brittle transition temperature (DBTT) shift even under irradiation. But it has the poor weld ability, due to its relatively high containing of alloy elements^[1-2]. Transient liquid-phase bonding has been especially successful for joining such materials as Ni-base alloys, Al-base alloys, intermetallic and steels^[3-9]. It has less residual stress and fewer cracks in the bonded region than traditional fusion welding approaches^[5, 8-10]. In the TLP diffusion bonding, a suitable interlayer containing a melting-point depressant (such as B, Si and P) is inserted be-

tween pieces to be joined and formed of a low-melting point liquid phase at the bonding temperature.

A major challenge is that traditional methods may yield a planar bond line, together with agglomerated oxide, to weaken bond strength^[8, 10]. Previous study showed that addressing a temperature gradient across the joint boundary helps to solve such a problem^[10]. In this study, we describe a new approach to get a preferable TLP bonding joint by a two-step heating method. The key steps of TLP bonding to get anticipated joints are melting and isothermal solidifying interlayer^[5]. Elevating the bonding temperature fully melted and improved the wettability of liquid interlayer. We propose a novel two-step process of TLP bonding to further study the superiorities of T91 heat resistant steel.

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1 Experimental procedure

The material used in our study was a T91 heat resistant steel, and a Fe – Ni – B – Si amorphous alloy foil in

35 – 40 μm thick. The interlayer melting point is 1 080 – 1 130 $^{\circ}\text{C}$. The compositions of T91 steel and interlayer are described in Table 1.

Table 1 Chemical compositions of T91 steel and interlayer used (wt. %)

Element	C	Si	Mn	P	S	Ni	Cr	Mo	Nb	B	Fe
T91	0.08 – 0.12	0.20 – 0.50	0.30 – 0.60	0.02	0.01	0.4	8.00 – 9.50	0.85 – 1.05	0.06 – 0.10	—	Balance
Interlayer	—	6 – 8	—	—	—	46	4 – 5	—	—	7 – 8	Balance

The specifications and mechanical properties of T91 heat resistant steels tubes are: 63.5 mm diameter, 4.6 mm wall thickness, tensile strength (R_m) is 585 MPa. The contacting surfaces of the base metals were polished by 800 grade emery paper and further cleaned in ethanol and acetone before diffusion bonding. Interlayer metal was cut into rings and washed with acetone after be placed to the bonding space. Sample piece was treated by high-frequency induction heating and argon was used as the protective gas. The TLP bonding process was performed using a purpose built bonding apparatus which is shown in Fig. 1. The bonding pressure was 6 MPa. In the traditional method, firstly 900 $^{\circ}\text{C}$ was reached and maintained for 10 seconds as transition phase in order to make the workpiece temperature uniformity inside and outside. Secondly raise the temperature to 1 230 $^{\circ}\text{C}$ for 4 minutes for welding phase. Afterwards, samples were cooled to 100 $^{\circ}\text{C}$ then reheated to 780 $^{\circ}\text{C}$ immediately and maintained for six minutes in order to reduce the welding stress (Fig. 2a). In our approach, we raised the temperature to 1 260 $^{\circ}\text{C}$ and

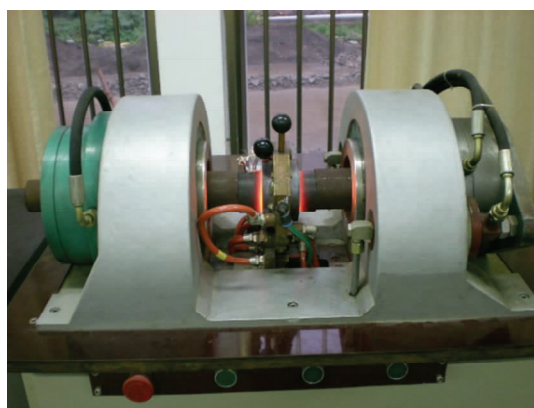
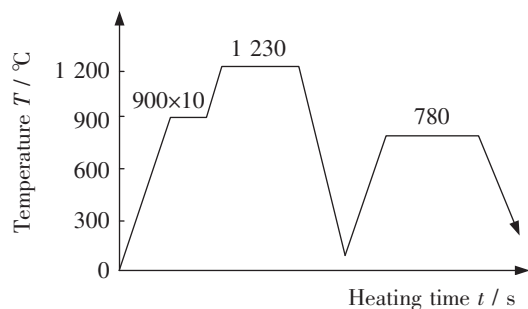
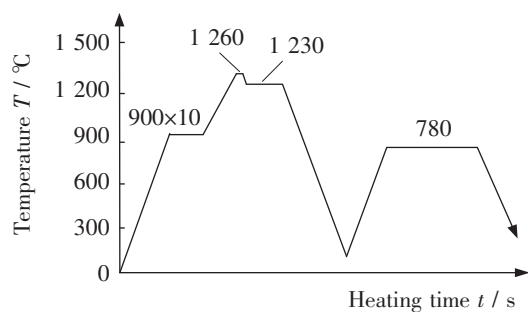


Fig. 1 Transient liquid-phase bonding equipment used in the test



(a) Traditional TLP bonding



(b) Two-step TLP bonding process

Fig. 2 Schematic diagram of technological parameters curve during TLP bonding

kept it for 30 seconds between transition phase and welding phase (Fig. 2b).

After the whole process, we cut the welded samples into transversal sections by electro-discharge machining (EDM) for samples examination. The microstructures were checked by GX71 optical microscopy and a JXA-8800R scanning electron microscopy (SEM) equipped with energy dispersion X-ray (EDX) spectrometer. Meanwhile, the strength and bending test of the TLP bonding joints were also carried out by a universal testing machine WES-600 at room temperature. The T91 TLP bonding joint were cut into 100 mm \times 10 mm \times 4.5 mm and 100

mm \times 8 mm \times 4.5 mm discs by milling for strength testing and bend testing. Fig. 3 shows the sampling location of tensile test specimens (L) and bend specimens (W). Each pipe joint can take four tensile specimens and eight bend specimens.

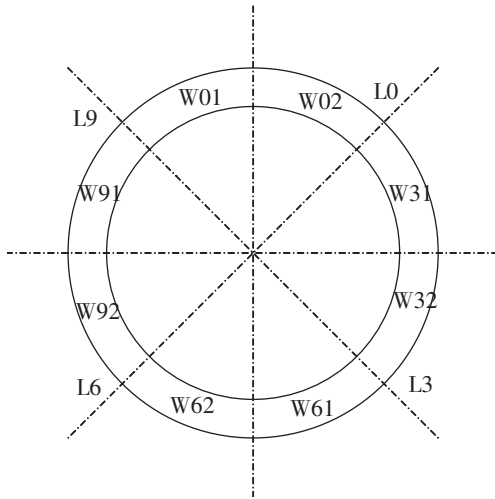
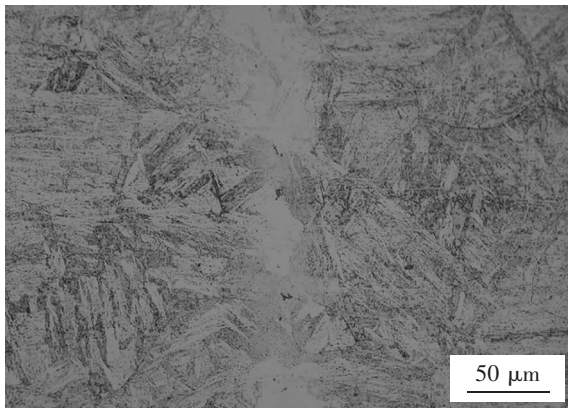
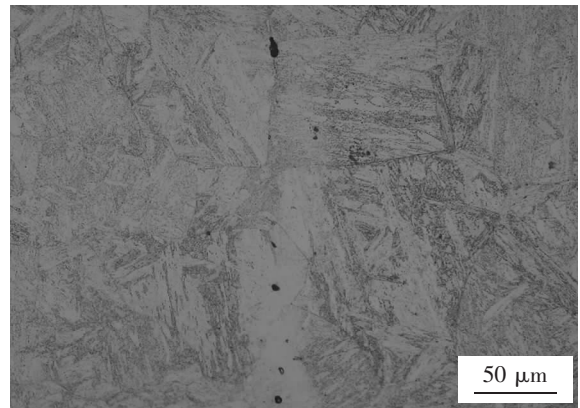


Fig. 3 Schematic diagram of the steel pipes sampling used to tensile and bend test



(a) Two-step TLP bonding



(b) Traditional TLP bonding

Fig. 4 Optical micrographs of the interface of the TLP bonding joints

2.2 Joint composition

In order to determine the homogeneity, we analyzed the distribution of elements in the joint interface. An electron probe micro-analyzers (EPMA) analysis using JXA-8800R scanning electron microscopy was employed to monitor element quantitative changes at joint's interfaces. In the concentration profile of traditional TLP bonding process, Cr and B did not fully diffuse into the bonding

2 Results and discussion

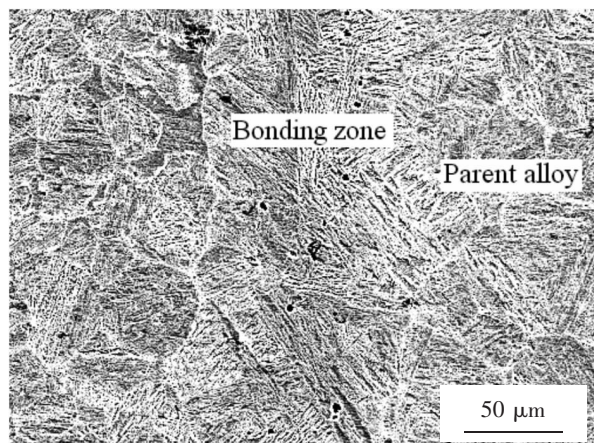
2.1 Microstructure

The microstructure of the T91 TLP bonding joint is shown in Fig. 4 and Fig. 5. We observed the similar microstructure of TLP bonding to the parent alloy by using the two-step heating process (Fig. 4a, Fig. 5a). By comparing the TLP bonding microstructure produced in the new process with the product by using the traditional process, the thickness in both bonding zones was visualized in a quite diverged way (Fig. 5). By our two-step heating process, a wider bond-zone 56 μ m was observed near the joint line in the contacting interface. Moreover, the bond-line was indistinguishable from the parent alloy (Fig. 5a). In the products by traditional TLP bonding process, a narrow bond-zone 48 μ m was seen at the joint (Fig. 5b). In addition, microstructures developments indicate that boron and chromium compounds were observed in the traditional TLP bonding process, which might weaken the joint (Fig. 5b).

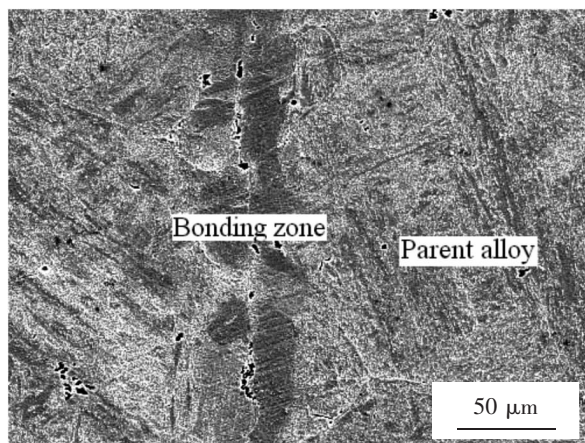
zone, visualized as an impulse in both Cr and B curves at the joints, which might be a chromium-boride compound (Fig. 6a). In our two-step TLP bonding approach, the profiles indicate that a significant compositional homogeneity was achieved at the joint's region (Fig. 6b). There were no residual brittle phases in the joints because the interlayer fully melted and MPD element diffused to enough extent (Fig. 6b). However, it is worth noticing that the

chromium-boride compound formation was suppressed by using the two-step TLP bonding approach. Meanwhile, a super-cooling of composition was formed on the solid/liquid interface to yield an even non-planar interlayer. The

two-step heating approach changes not only the solidification interface shape from planar to non-planar at isothermal solidification stage, but also facilitates melting element diffusion to avoid formation of brittle phase.

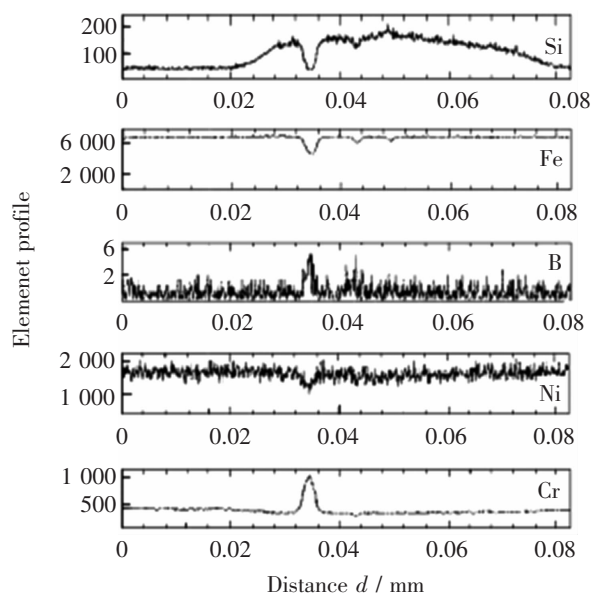


(a) Two-step TLP bonding

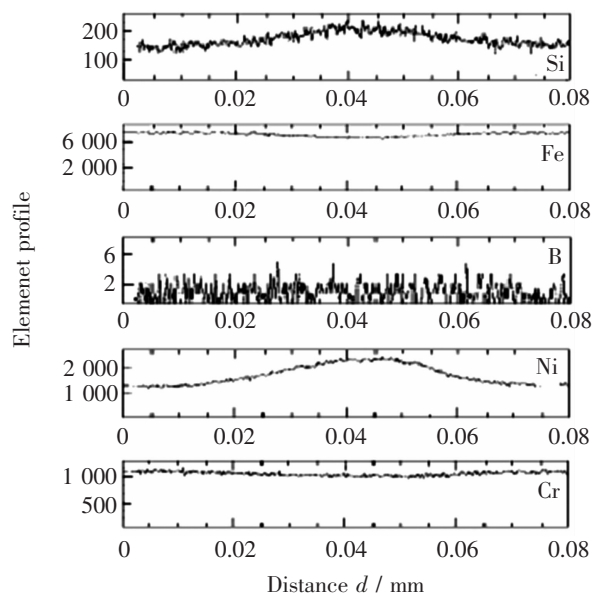


(b) Traditional TLP bonding

Fig. 5 SEM image of joint using different TLP bonding process



(a) Traditional TLP bonding



(b) Two-step TLP bonding

Fig. 6 Line scan for various elements of different TLP bonding process

2.3 Mechanical properties

To study the welding strength of the joints, we determined a tensile strength and bend angle. The bonds made by the two-step heating approach reached a tensile strength

at 850 MPa, whereas 830 MPa in traditional method (Table 2). The fracture location of all tensile samples is not in the joints.

Table 2 Process and mechanical properties of T91 steel TLP bond

TLP process	Temperature $T / ^\circ\text{C}$	Bonding time t / min	Tensile strength R_m / MPa	Bend angle $\varphi / (^\circ)$
Two-step	1 260	0.5	850	180
	1 230	4.0		
Traditional	1 230	4.0	830	5

In the bend strength test, all bend specimens did not fracture when bent 180° in the group by two-step heating approach, while all the fractures were identified at the joints in the group of the traditional TLP bonding process. Bending fracture morphology was shown in Fig. 7. We can see some of the granular second phase. The bend strength test indicates that the bend angle of the two-step TLP welded joints is 180° . In contrast, a joint welded by the traditional TLP bonding process is merely 5° . The relatively lower bend ductility made by traditional TLP bonding process was possibly attributed to the generation of intermetallic precipitates because of the borides.

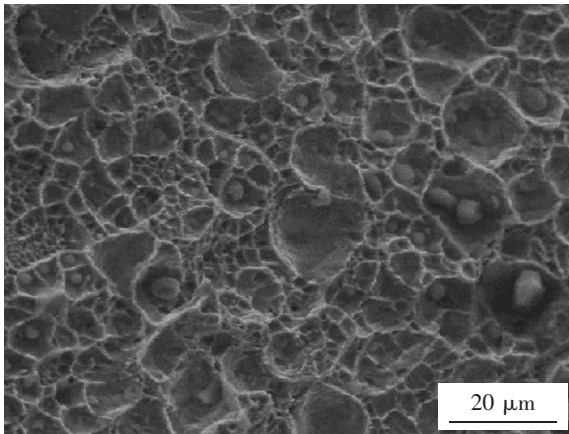


Fig. 7 The bend samples fractography of joint with traditional TLP bonding

2.4 Discussion

The traditional process will form a small amount of brittle phase in the TLP diffusion bonding process, due to the melting point depressant element is not sufficient diffusion. The bend samples fractography (Fig. 7) and EPMA line scan analysis found that the boron and chromium compounds generated. The two-step heating temperature process cannot be the formation of brittle phase, because

the process has a high temperature heating stage. When the heating temperature is higher, the melting temperature of the interlayer is increased. In this case, the inter diffusion between the base material and the interlayer was more fully. Therefore, the joints were diffusion uniform and did not form compounds. According to the alloy crystalline theory of crystallization, if this is a constitutional super cooling in the liquid-solid interface at the forefront of crystallization process, crystal growth pattern may be changed from planar to cellular. The short-term high temperature heating in the two-step heating temperature process may be caused a constitutional super cooling. So the two-step heating temperature process not only can change the interface shape of isothermal solidification phase, but also can accelerate the diffusion spread of melting point depressant elements and avoid the formation of brittle phase.

3 Conclusions

(1) Bonding parameters of new process are $1\ 260\ ^\circ\text{C}$ for 0.5 min and $1\ 230\ ^\circ\text{C}$ for 4 min. Product's microstructure of two-step heating process is similar to parent materials, producing non-planar interfaces, while by traditional one it produces planar interfaces.

(2) The bonds made by the two-step heating approach reached a tensile strength at 850 MPa, whereas 830 MPa in traditional method. Joints of two-step process bear not only higher tensile strength, but the better bending ability as well. The products made by the traditional process do not reach the anticipated level.

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