Diffusion bonding in stainless steel

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Abstract. Vacuum diffusion bonding is a joining technique whose success depends on the optimization of a number of parameters. In the present work a study of these parameters and bonding mechanisms is undertaken on a 304L stainless steel. The usual parameters (temperature and pressure) were chosen for bonding, during different times. Three different surface finishes were produced, mechanical strength was measured by tensile tests, and a number of characterization techniques were employed in order to obtain a complete description of the interface reaction products.
The best process condition in terms of joint mechanical strength is 1000°C, 17MPa during 40 minutes corresponding toa tension of 552 MPa. Interfacial fracture did not reduce the resistance level. Specimens in stainless steel 304L were treated during welding operations and did not present susceptibility to intergranular corrosion.
The obtained surfaces presented the following characteristics(i)a quality of Ra= 1 to 6 µm, that can be done during machining ;(ii)it will be avoid surface machining with friction operations;and (iii)superficial preparation must be done previous to the diffusion bonding.The tensile strength of the bond joint realized in these conditions is comparable to mechanical properties of the parent metal.

Keywords: diffusion bonding, stainless steel, parameters of the bonding, surface finishing, intergranular corrosion.

1. Introduction

Diffusion bonding is a solid state welding process which is applicable to the same or different types of materials under the yield strength and over the recrystalization temperatures (Guo et al,1987; Dunkerton ,1991). Diffusion welding is accomplished by bringing the surface to be welded together under moderate pressure and elevated temperature in a controlled atmosphere so that a coalescence at the interfaces or faying surfaces can occur (Garmong, 1975). Melting or fusion is not associated with either process. Being a solid state joining process, diffusion bonding eliminates problems of segregation, cracking and distortion stresses, which are generally encountered in liquid phase welding techniques (Owczarski et al, 1981; Spriggs, 1982).

A diffusion weld is created when the surfaces of materials are brought sufficiently close together so that short-range interatomic forces operate. If the surfaces are free from contamination, the driving force for the weld is a lowering of surface energy. Because real surfaces can never be perfectly smooth, initial contact on the faying surfaces is made between surface roughness when the load is applied (Yilmaz,2002,2003). Further contact is made by plastic yielding and creep deformation. At the same time, diffusion at the clean surface eliminates the remaining interfacial boundary.

Many models for diffusion welding have been proposed. The first conceptual process model by King and Owczarski (King ,1968; Derby ,1982, 1983a, b) had four stages- Fig.1: 1) initial contact, 2)attainment of intimate interfacial contact, 3) grain boundary diffusion/migration, and, finally, 4) volume diffusion. Initial contact is limited to a few roughness , followed by Stage 1, in which the asperities are crushed. Stage 2 involves grain boundary diffusion and migration, whereas Stage 3 consists of volume diffusion to isolated voids. Another early work by Hamilton(1973) proposes that the diffusion welding process consists of four steps: 1) development of intimate interfacial contact, 2) formation of the metallic bond, 3) interdiffusion, and 4) recrystalization/grain growth. In general, for a certain required level of weld quality, the optimum bonding conditions for diffusion bonding depends on pressure, temperature, time, and surface finishing.

 In the present work a study of these parameters is undertaken on a 304L stainless steel.
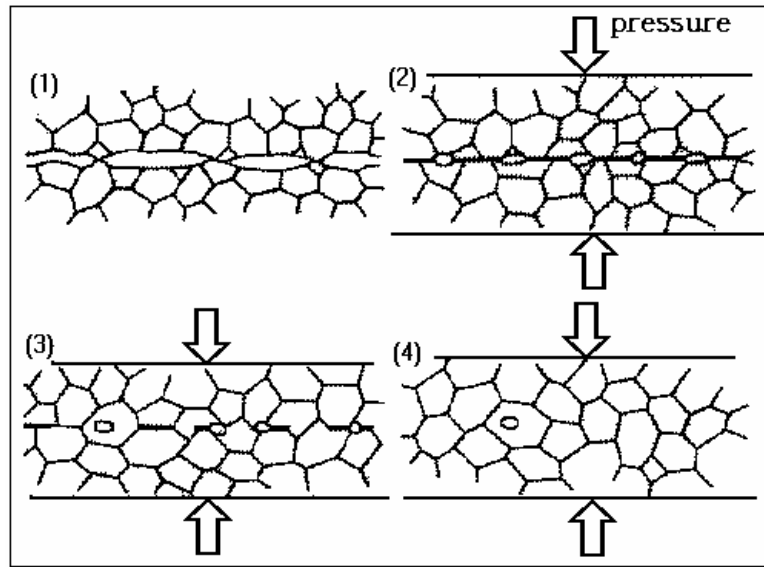
Figure1. Schematic illustrating stages of diffusion welding (Gimenes,2004).

2.Experimental procedure

The materials used in experiments were received from 11 mm diameter commercial rods. The chemical composition of the materials has been represented in Tab. 1. Specimens have been prepared in pieces 10 mm long for metallographic investigations and 30mm long for tensile testing. Bonding temperatures were ranged from 850ºC to 1000ºC (with heating rate 20ºC/min and cooling rate 6ºC/min) with protective atmosphere of argon .Bonding pressures used were varied from 14 to 20 MPa , times from 30 to 60 min, with different surface finishes:

1) pieces were cut from the rods (machining), and their surfaces ultrasonically degreased in acetone bath;

2) pieces were cut from the rods (machining) for diffusion bonding and their surfaces were polished to a 1200 mesh , ultrasonically degreased in acetone bath;and

3) pieces were cut from the rods (machining), and with interval 2 hours were ultrasonically degreased in acetone bath.

Diffusion bonding conditions are detailed in Tab. 2.

For metallographic investigations, specimens were cut longitudinally and prepared by conventional techniques. Investigationjs were performed with optical and scanning electron microscopy (SEM)

Oxalic acid etch test was used to provide a rapid method for identifying these specimens that were certain to be free of susceptibility to rapid integranular attack. Each practice is related in a table showing the classifications of etch structures on a given stainless steel grade are equivalent to acceptable, or possibly nonacceptable performance. Specimens having acceptable etch structures need not be subjected to the hot acid test. Tests were realized according to ASTM A262-02a and after etch structure was classificated.

Tensile strengths of the bonded samples were detected by tensile testing prepared according to ASTM E8-89, and three samples were tested for each bonding condition.

Table 1.Chemical composition of the used material (as received).

Material	Elements(w%)								
	C	Si	Mn	P	S	Cr	Ni	Mo	Fe
Stainless steel (AISI 304L)	0,03	0,37	1,99	0,038	0,02	18,03	8,02	0,40	Other

Table 2. Bonding conditions for diffusion bonding.

Sample #	Welding temperature (ºC)	Applied pressure (MPa)	Welding time (min)	Protective gas	Surface finish #
S1	850	14	30	argon	1
S2	850	17	30	argon	1
S3	850	20	30	argon	1
S4	1000	14	30	argon	1
S5	1000	15	30	argon	1
S6	1000	17	30	argon	1
S7	1000	17	30	argon	2
S8	1000	17	30	argon	3
S9	1000	17	40	argon	1
S10	1000	17	60	argon	1

3. Results and discussions

3.1 Welding parameters

In general, diffusion welding involves little bulk deformation and is conducted at elevated temperature. Therefore temperature and pressure are importants characteristics of the process. Correct selection of these parameters permits to extend intimate interfacial contact and to increase interdiffusion.Another parameter ´time´ is one variable which detects minimal necessary duration of the welding for completion of the grain boundary diffusion, interdiffusion and volume diffusion.   Choice of the variable is described in 3.3 Mechanical properties.

Mechanical properties of the joint can reveal a reach of the completion of the grain boundary diffusion, interdiffusion and volume diffusion.

In addition to define the basic variables it is examined influence of the surface roughness and of the surface contaminant in order to obtain the diffusion bonding.

The 304L stainless steel was diffusion-bonded in the conditions indicated as in Tab. 2.

By investigation of the bond region on samples S1-S5, it is deduced that is not remaining interfacial boundary ,and welding parameters or set variables were selected incorrect.

In figure 2,as an example, it is observed a central profile of sample 6 welded with 1000°C , 17 MPa and 30 min . It is seen that the material surfaces were brought close enough together that stort-range interatomic forces operated and interdiffusion and grain growth took place (Coble,1970,Baluffi,1981). It is considered that the determination of the welding parameters (temperature and pressure)producing a bond region is realized to perform a successful welding (Hill,1987;Takahashi,1992; Davé, 2003)
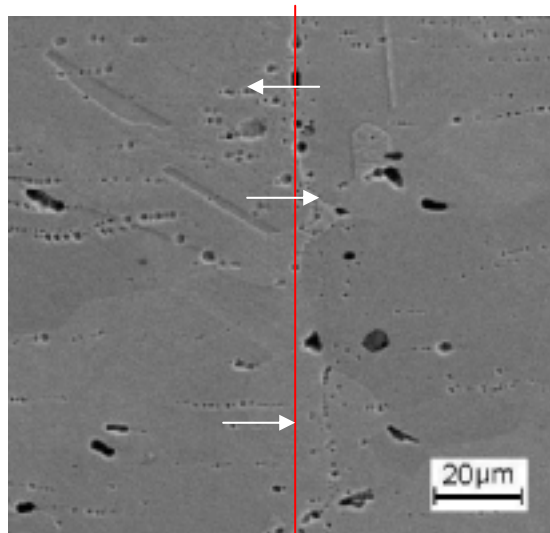

Figure2. SEM micrography of sample S6.

Surface finishing is one important welding parameter. Diffusion welding was realized on samples S7 and S8 with different surface finishing.

Sample S7 was cut from the rods for diffusion bonding and their surfaces were polished to a 1200 mesh , ultrasonically degreased in acetone bath. By investigations on bond regions, it can be deduced that during process the attainment of intimate interfacial contact and formation of the metallic bond were suspensed (Kazakov, 2002). Probably, material particles derived during preparation of specimens slowed down rate of diffusion.

The corrosion resistance of 304L stainless steel results from the presence of a thin hydrous oxide film on the surface of the metal (Sritharan ,1980; Chen, 1981; Munir ,1983). For stainless steel, this film, stabilized by chromium, is considered to be continuous, nonporous, insoluble and self-healing. During machining the surface that is going to be worked has not oxide films. But the film repaired itself when it is re-exposed to air. In sample S8 interval between cutting and welding was sufficient for recovery the oxide film.It is deduced that in sample S8, and a pressure 17MP is not sufficient for breaking this oxide film.

In figure 2.1 ,it is seen an example, to get a comparison of micrographes of a centrals profiles of samples 6,7 and 8 welded with 1000°C , 17 MPa and 30 min,but surface roughness and surface contaminant are differents.
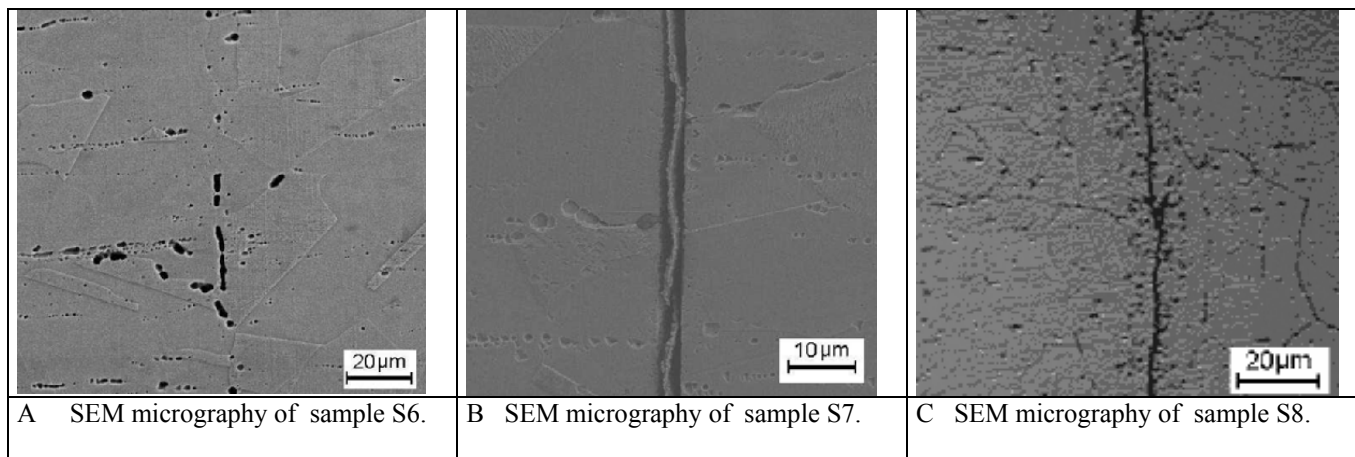
A SEM micrography of sample S6.	B SEM micrography of sample S7.	C SEM micrography of sample S8.

Figure2. 1 SEM micrography of samples S6,S7,S8.

3.2 Intergranular corrosion

Heat treatment of stainless steels can lead to early failure under severe corrosive conditions and can be greatly reduce service life in many relatively mild environments. Microstructural changes produced by different heat treatments have considerable influence on the corrosion resistance of stainless steels. These steels normally exhibit greater resistance when all carbon is in solution, producing a homogeneous single-phase structure. Unstabilized austenitic stainless steels become subject to severe attack along grain boundaries at room temperature in a number of corrosive media if the metal is heated to temperatures in the range from 550°C to 850°C This attack is known as intergranular corrosion and results from precipitation of chromium carbide and consequent depletion of chromium in the areas adjacent to the grain boundaries, (Kamat ,1988; Kaganovskii et al.,1997).
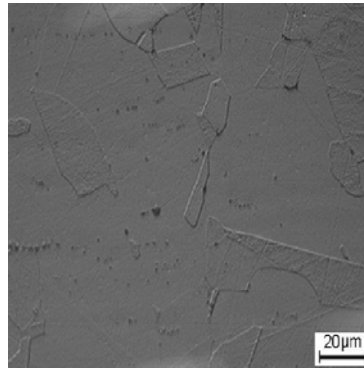
Among the methods available for detecting susceptibility to intergranular corrosion the oxalic acid etch test is choosen in this study.

The oxalic acid etch test is used to provide a rapid method for identifying the specimens S6, S9 and S10 that are certain to be free of susceptibility to rapid intergranular attack. Details for performing this test are given in ASTM 262-02 .
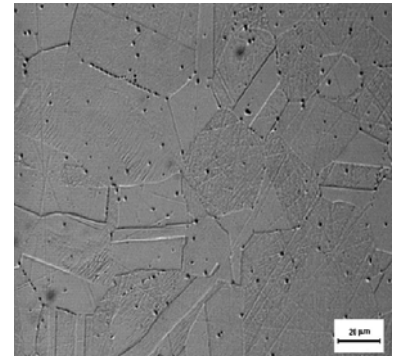
The etched surface was examined by complete transverse from inside to outside diameters of rods. The etch structure is classified as a step structure.- steps only between grains, no ditches at grain boundaries. Specimens have acceptable etch structures (fig.3) and need not be subjected to the hot acid test.
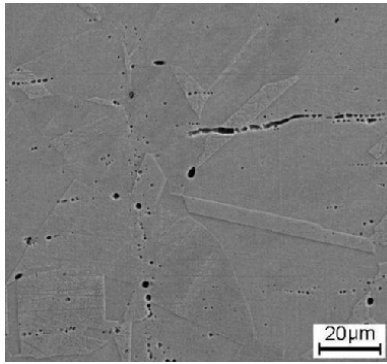
(a) Step structure of sample S6. (b) Step structure of sample S9. (c) Step structure of sample S10.

Figure 3. Etch structures.

3.3 Mechanical properties

The tension test was used to measure yield strength, tensile strengthand elongation. The tensile strengths of the bonded samples were detected by tensile testing prepared according to ASTM E8-89 L_o=4d The mechanical properties (Table 3) of the samples 6,9,10(three specimens each sample) were tested for each bonding condition.

Table 3. Mechanical properties of of joins.

	Δl, elongation (mm)	σ_e, yield strength (MPa)	$\sigma_{máx.}$, tensile strengh (MPa)	ϵ, strain
S10	11,1	229	546	0,28
S9	11,1	219	552	0,28
S6	7,7	199	549	0,19
Stainless steel 304L		210	564	0,58

The last line of Tab. 3 shows nominal tensile strength, yield strength and strain of 304L stainless steel. It is evident that tensile strengths and yield strengths of the samples 6,9,10 does not differ significantly from the nominal ones.

The strain of the sample S6, which are welded during 30 minutes, is 0,19 and differs significantly from the strains of the samples S9and10.This occurs because the joining time. Increasing time from 30 to 40 minutes permits to conclude the last stage of the welding process. Grain growth through the faying surfaces augments the diffusion bonded joint ,but increasing joining time from 40 to 60 minutes does not improve changes on mechanical properties. Under these circumstances, the effect of joining time is not impressive.

4. Conclusions

Optimum bonding conditions for diffusion welding of 304L stainless steel are in the temperature 1000°C, pressure of 17 MPa and times of 40 minutes. The effect of the pressure is higher than other conditions, because welding pressure deforms surface roughness plastically, breaks oxides whose solubility is not possible to attain and increases contact area. Bonding temperature 1000ºC (with heating rate 20ºC/min and cooling rate 6ºC/min) permits to avoid intergranular corrosion. The tensile strength of the bond joint realized in these conditions is comparable to mechanical properties of the parent metal.

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