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Corrosion and microstructural aspects of dissimilar joints of titanium and type 304L stainless steel

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Abstract

To link titanium and zirconium metal based (Ti, Zr-2, Ti–5%Ta, Ti–5%Ta–1.8Nb) dissolver vessels containing highly radioactive and concentrated corrosive nitric acid solution to other nuclear fuel reprocessing plant components made of AISI type 304L stainless steel (SS), high integrity and corrosion resistant dissimilar joints between them are necessary. Fusion welding processes produce secondary precipitates which dissolve in nitric acid, and hence solid-state processes are proposed. In this work, various dissimilar joining processes available for producing titanium-304L SS joints with adequate strength, ductility and corrosion resistance for this critical application are highlighted. Developmental efforts made at IGCAR, Kalpakkam are outlined. The possible methods and the microstructural–metallurgical properties of the joints along with corrosion results obtained with three phase (liquid, vapour, condensate) corrosion testing are discussed. Based on the results, dissimilar joint produced by the explosive joining process was adopted for plant application.

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1. Introduction

In the reprocessing plant at Kalpakkam, spent mixed (U,Pu)C carbide fuel of the fast breeder test reactor (FBTR) will be reprocessed to retrieve the useful fissile material and remove the wastes and other unwanted fission products [1]. The first unit process in the reprocessing of spent nuclear fuel for recovering uranium and plutonium is dissolution. For the dissolution of fast reactor carbide fuels, the electro-oxidative dissolution technique (EODT) has to be employed to destroy the organic compounds generated as well to increase the dissolution rate of high plutonium-containing fuel [2].

American Iron and Steel Institute (AISI) type 304L austenitic stainless steels (SS) are extensively used in the construction of the reprocessing plants where nitric acid is the main process medium. However, the electrolytic dissolver cannot be fabricated from type 304L SS as the severely corrosive (11.5 N HNO₃, boiling, redox ions) conditions of dissolution lead to unacceptable high corrosion rates ($\cong 43180 \mu\text{m}/\text{y}$). Based on extensive studies [3–5], titanium has been chosen as the material of construction for the electrolytic dissolver as it shows acceptable low corrosion rates ($< 127 \mu\text{m}/\text{y}$). However, the electrolytic dissolver unit made of titanium has to be connected to the rest of process vessels and piping made of AISI type 304L SS. Flanges and mechanical joints cannot be used for such purposes because of zero failure requirements for this critical unit process involving highly corrosive and radioactive environments. Metallic joints of Ti/AISI type 304L SS prepared using solid state joining processes are the best option for such a situation. The fabrication and qualification of this

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dissimilar metal weld joint is crucial in the reprocessing plant. High corrosion resistance in severely corrosive and radioactive nitric acid with adequate mechanical strength and bent ductility are the minimum requirements for this purpose.

Dissimilar joints to link two different materials with entirely different physical and mechanical properties can be prepared by either fusion or solid state welding depending on the dissimilar materials and the desired properties for the application [6]. Several dissimilar joints are made by fusion welding methods as they produce joints with desired physical and mechanical properties. However, in a corrosive environment, the secondary precipitates formed during fusion and solidification corrode severely and affect the integrity of the joints. Dissimilar joining by solid state processes are considered for materials with extremely different physical and mechanical properties. Commonly employed dissimilar joining processes are roll bonding, pressure welding, friction welding, explosive joining, ultrasonic welding, diffusion bonding and laser forming. Since filler wires and electrodes are not required for solid state joining processes, the risk of hydrogen cracking and embrittlement of joints do not arise in the service. Also, since no melting happens at the interface, secondary precipitates are not formed and hence the corrosion resistance is not affected significantly during service. However, these processes are generally applicable for small components thus restricting the choice to make only selected critical components. The best dissimilar joining method for the particular application is chosen based on the joint properties, environmental considerations and process parameters. The good bond created between the two joining materials depends on their surface preparation including good surface finish, freedom from contamination, wettability, scales and process layers, and the joining process used. In a dissimilar joining process, basically two clean surfaces are brought together in intimate contact to produce strong adhesion through the force applied through several means. The difficulties in getting a sound joint are the presence of thin non-metallic layers and adsorbed gases on the bonding surfaces, and to maintain a total matching surface over the entire joining area. By applying pressure a bond can be produced but the lateral movement of the parts during the application of pressure can yield clean surfaces by asperity shearing.

In conventional roll bonding process, two plates of the materials are placed together, heated and rolled to the desired thickness. It is generally used to clad one material with another. The high performance nitric acid grade type 304L SS with very low corrosion rates are joined/cladded with conventional type 304L SS to provide the benefit of corrosion resistance and extended life [6]. Forge welding and hammer welding are typical processes in which materials are compressed to form

bonds with little recrystallisation across the bonded surface [1]. A typical example is the hammer welding of swords in which high carbon steel is attached to low carbon steel. In cold pressure welding process, pressure brings together the mating surfaces, disturbs the native surface film and allows chemical bonding of clean surfaces without using any heat or flux. Soft and ductile metals like aluminium, lead, indium and tin can only be joined by this process. In the case of solid-state resistance pressure welding, heating is produced by passing current through the metals (Joule heating $\sim I^2R$). All the above processes have not been utilized for the present work due to their limitations in the dissimilar joining of titanium and stainless steels. Laser pressure beam welding is a newly developed method [7] by which metals can be joined at very high speed (up to 240 m/min) with minimum defects. The basic principle is that melting is suppressed or the molten metal is squeezed out before its resolidification, and this is possible when heating up the seam region of the welding parts with the focused laser beam and pressing them together simultaneously. The method is under development, and is not attempted for the present work.

In diffusion bonding, components are generally pressed together at a moderate pressure (≈ 10 MPa) and heated to approximately 0.6 times melting point (T_m) of the metals for the atoms to diffuse across the interface to form diffusion bonding [8]. Surface roughness and contamination are very critical to produce clean bonding between the surfaces. Generally, a roughness parameter Ra less than $2\ \mu\text{m}$ and waviness less than $400\ \mu\text{m}$ are preferred for the surfaces to be joined. This is a slow process as diffusion of atoms across the interfaces takes place over a long period of time. The strength of the joint produced depends on the clean atmosphere with no oxidation. Sometimes fillers/interlayers are needed to achieve good bond strength. Generally, this process is used for joining ceramics and a range of components based on titanium for aerospace applications. During the diffusion bonding process, initially only the surface asperities are in contact, but the application of pressure causes a metal-to-metal contact. With time, the contact regions grow as the atoms of the materials diffuse across the junction at an elevated temperature, $\approx 0.6 T_m$ (T_m – melting point). The pores developed during the diffusion process are eventually reduced to very low levels.

The friction welding process [9] is based on rotating one part at relatively high controlled speed and pressure (≈ 40 MPa) against the stationary part to which it is joined. The contacting surfaces are thus heated by this frictional contact to a high temperature and forged together to produce a reliable high strength weld. The weld is completed within seconds or a fraction thereof, after making contact. High pressure and low speed, or low pressure and high speed lead to improper joining geometries, and an optimum pressure and speed are

necessary to produce good joint geometry. The main parameters that directly influence the strength and bond quality of the joint are friction pressure, friction time, forging pressure and rotational speed. The frictional power, $F_p = \mu Lv$, where μ is the coefficient of friction, L is the applied load and v is the sliding velocity. When welding either hard-to-hard or hard-to-soft dissimilar metal combinations, the extent of deformation on individual metals differs, and for some metals the surface condition is also important. The process is mainly useful for joining rods, tubes, etc., and no special surface preparation is necessary. In the inertial friction welding, the energy for frictional heating is supplied by the kinetic energy of a flywheel. The process is very fast, and a 10 mm shaft can be welded onto impellers at the rate of four pieces per minute.

In the explosive joining process, two metal surfaces to be joined are kept in close contact and an explosive charge is detonated against one of them [10]. This process uses an explosive force to create an electron sharing metallurgical bond between two metal surfaces. The impact generates sufficient energy to cause the colliding metal surfaces to flow hydrodynamically when they intimately contact and promote solid state bonding. The jet is ejected outward from the collision apex between the metals, and this produces a cleaning action by scarfing the metal surfaces. Fig. 1 shows the schematic of the arrangements for the explosive joining process. The most important action of explosive joining is the ‘flyer plate acceleration’ which results in a dynamic bonding action. The dynamic bond angle β results in oblique impact because the flyer and the base plates promote the hydrodynamic flow of the metal surfaces and a resulting jetting action. In general, bonds produced by extreme metal deformation results in a wavy interface resulting in optimum strength.

Although the explosive detonation generates heat, there is no time for heat transfer to the metal surfaces.

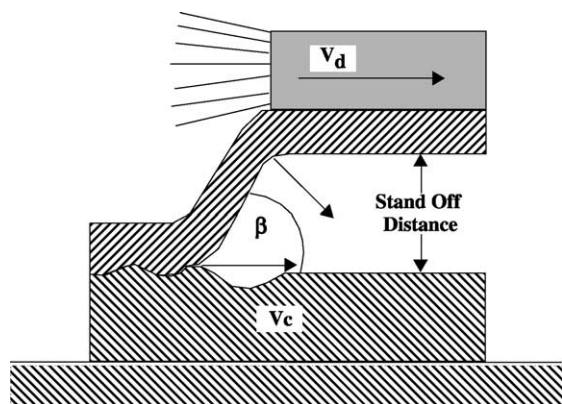


Fig. 1. Schematic of the arrangements for the explosive joining process.

Thus, there is no appreciable temperature increase in the metals, and also very little diffusion occurs at the interface. The detonation velocity is generally directly proportional to the explosive density, and is normally between 2000 and 5000 m/s. Typical detonation energy is about 3.5–6.5 kJ, pressure is 100–400 kbar and the temperature of explosion is 3000–6000 K. The surface finish of the joining surfaces plays a major role in determining the integrity of the bond developed.

Solid-state welding processes, viz. explosive bonding, diffusion bonding and friction welding can be employed for fabricating this DMW (dissimilar metal welding) joint to achieve adequate mechanical properties and corrosion resistance [11]. Literature on the joint preparation and corrosion performance in nitric acid for the dissimilar joints of titanium to type 304L SS is very scarce. In Japanese reprocessing plants, both explosion joined and diffusion joined sections are used for plant applications [12]. A satisfactory diffusion joint between zirconium and stainless steel at a strength level of Zr could be achieved using annealed tantalum foils inserted into the joint surfaces. This avoided the formation of brittle and soluble intermetallic compound layers and favoured the full contact of interfaces during joining. A direct bonding between titanium and type 304L SS could be achieved by vacuum diffusion bonding with the pre-cleaning of the surfaces by the ion sputtering method. The diffusion bonded specimens showed satisfactory performance only in 3 N HNO₃ containing Cr⁶⁺ ions at 333 K. In the present paper dissimilar joints produced by solid state processes like explosive joining and friction joining are characterized and evaluated for applications in severely corrosive nitric acid conditions employed in reprocessing plants.

2. Experimental work

2.1. Selection of dissimilar joining processes

Considering the geometry and size of the tubular joint required, solid-state welding processes like diffusion bonding, friction welding and explosive joining are considered for the present investigation. It is reported that the tensile strength of the diffusion bonded joint is only 150 MPa which is considerably lower than that of both titanium and 304L SS [13]. Hence, only friction welding and explosive joining processes are pursued in our work.

Titanium and stainless steel have different forging characteristics, with titanium undergoing relatively higher deformation at elevated temperatures as compared to the stainless steel. Hence, the smoothness of the stainless steel surface is very important for good metallurgical bonding during friction joining [14]. The typical parameters maintained during friction welding of

titanium to 304L SS for the present investigation are: rotational speed = 1560 rpm, frictional pressure = 196 MPa, friction time = 3 s, upsetting pressure = 294 MPa, and upsetting time = 10 s [11].

The explosive bonding process produces a bonding by high velocity impact of the work pieces caused by a controlled explosion/detonation. The bonding is produced within a fraction of a second. The important interrelated variables in explosive bonding are (1) collision velocity (2000–5000 m/s), (2) collision angle β ($\cong 45^\circ$), (3) flyer plate velocity, and (4) nature of explosive. Smooth surface finish of titanium and type 304L SS are obtained in order to create a better interface after explosive joining operation.

2.2. Joint preparation for dissimilar joining

Titanium Grade-2 and type 304L SS rods, each of 22 mm diameter and 100 mm length, were friction-welded using a continuous-driven friction-welding machine at Welding Research Institute (WRI), Trichirapalli, India. The nominal chemical composition and mechanical properties of the materials are given in Table 1. The friction-welded rods are machined to pipes of sizes 13.7 mm (OD) \times 2.2 mm (thickness) and 21.3 mm (OD) \times 2.77 mm (thickness). Transverse-welded specimens for tensile, bend and other tests are prepared from these machined pipes.

ASTM A262 practice C qualified 25 mm thick 304L SS plate is explosively joined with a 12 mm thick titanium Grade-1 material (direct joining) using plates of size 450 \times 450 mm², and a 'sound-bond' area of size of about 300 \times 300 mm² is obtained after ultrasonic examination. In another set of experiments, an intermediate step of first cladding a 3 mm thin titanium is carried out prior to the normal explosive joining with

12 mm titanium plate (buffer joining, Ti–Ti-304L SS). The nominal chemical composition and mechanical properties of the titanium and 304L SS plates used for explosive bonding are given in Table 2. The cladded plate is subjected to a stress-relieving heat treatment at (540 \pm 10) °C for 1.5 h. From the 'sound-bond' region of the direct Ti-304L SS cladded plate, a 37 mm long cladded sleeve of size 21.3 mm (OD) \times 2.77 mm (thickness) is machined. Subsequently, a titanium Grade-2 pipe and a 15 NB Schedule 40 304L SS pipe, each of length 75 mm, are butt-welded to the Ti- and SS-side of the cladded sleeve, respectively. Transverse-welded specimens for the various tests are prepared from this piece.

3. Results and discussion

3.1. Evaluation of properties of titanium-type 304L SS joints

Both the friction-welded and explosive-joined pipe sections are non-destructively examined using liquid penetrant, radiography and ultrasonic testing. Both the joints passed all the NDT tests with no significant defect indications (Table 3).

Tensile testing is carried out at room temperature on two transverse-welded specimens each from both the joints. While for the friction-welded joint, the failures occurred at the joint interface with negligible ductility, the failure occurred in the titanium base metal for the explosive-bonded joint with significantly higher ductility of about 20% total elongation. However, the strength of the explosive-bonded joint (with average UTS of about 430 MPa) is slightly lower than that of the friction-welded joint (with average UTS of about 480 MPa).

Table 1

Nominal chemical composition and mechanical properties of the titanium and 304L stainless steel rods used for friction joining

Material	Weight %						YS (MPa)	UTS (MPa)	TE (%)
Ti Gr.-2	C	H	N	O	Fe	Ti	294	476	33
	0.011	0.001	0.011	0.127	0.05	Bal.			
304L SS	C	Mn	Si	S	P	Cr	225	537	71
	0.025	1.43	0.5	0.0047	0.008	18.4			

Table 2

Nominal chemical composition and mechanical properties of the Titanium and 304L stainless steel plates used for explosive joining

Material	Weight %						YS (MPa)	UTS (MPa)	TE (%)
Ti Gr.-1	C	H	N	O	Fe	Ti	280	432	36
	0.01	0.002	0.012	0.0948	0.03	Bal.			
304L SS	C	Mn	Si	S	P	Cr	225	537	71
	0.025	1.12	0.54	0.008	0.02	18.5			

Table 3
Results of non-destructive, mechanical and three-phase corrosion tests

Tests	Friction welded joint		Explosive bonded joint	
	Test 1	Test 2	Test 1	Test 2
<i>Non-destructive tests</i>				
Liquid penetrant testing	Passed		Passed	
Radiography	Passed		Passed	
Ultrasonic testing	Passed		Passed	
<i>Mechanical tests</i>				
<i>Tensile tests</i>				
Ultimate tensile strength (MPa)	462	495	397	457
Elongation (%)	Negligible	Negligible	21.4	19.7
<i>Bend tests</i>				
Root bend (<i>bend angle at failure</i>)	<5°	<5°	60°	76°
Face bend (<i>bend angle at failure</i>)	<5°	<5°	80°	106°
<i>Three phase corrosion tests</i>				
			Ti + 304L SS	Ti + Ti + 304L SS
Liquid phase <i>avg. corrosion rate</i> (µm/y)	12		11	38
Vapour phase <i>avg. corrosion rate</i> (µm/y)	18		38	114
Condensate phase <i>avg. corrosion rate</i> (µm/y)	256		305	137

Root- and face-bend tests were also carried out on two transverse-weld specimens each from both the joints. While the friction-welded joint shows very poor bend-ductility with all the root- and bend test specimens failing within 5° of bending, the direct Ti-304L SS explosive-joined specimen shows significantly improved bend-ductility with the root- and face-bend specimens

failing after 60°–76° and 80°–106° of bending (Table 3). Though the Ti–Ti-304L SS joint passed liquid penetrant testing, radiography and ultrasonic examinations, the joint failed poorly in the tensile and bend tests.

Three-phase corrosion tests are conducted in 11.5 N boiling nitric acid for specimens obtained from both the joints. The test involves exposure of the specimens to the

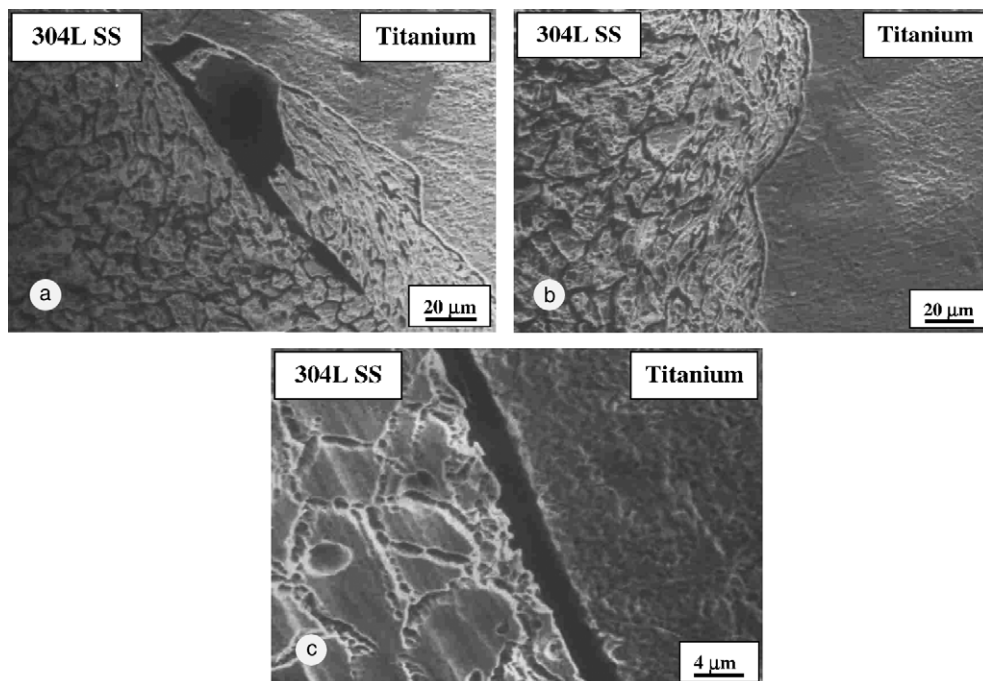


Fig. 2. SEM micrographs of (a) corrosion attack at vortex region, (b) perfect joint, for explosive joint, and (c) 'trench' formation due to corrosion attack in friction joint.

liquid, vapour and condensate phases of the boiling nitric acid medium for five periods of 48 h each, followed by measurement of the corrosion rates in each of the three phases. Table 3 gives the average corrosion rate, in $\mu\text{m}/\text{y}$, in the three phases. It is observed that for both the joints, the corrosion rates in the condensate phase is markedly higher than in the other two phases. It is also observed that the average corrosion rate of the explosive-bonded joint in all the phases, except the liquid phase, is marginally higher than the friction-weld joint (Table 3). In comparison with direct Ti-304L joint, the buffer joints with thin Ti sheet (Ti-Ti-304L SS) shows better corrosion rates in all the three phases as compared to direct Ti-304L SS joints. Detailed optical and scanning electron microscopy examination of specimens exposed to the condensate phase indicates that the friction-welded joint undergoes severe corrosion attack with 'trench' formation at the joint interface, while the explosive-welded joint has severe corrosion attack on the 304L SS with selective attack at the vortex region of the joint interface (Fig. 2). The vortices formed in Ti-SS joint appear to contain a mix of both metals arranged in an eddy pattern that is 'frozen in' by rapid solidification. This phenomenon may be responsible for higher corrosion at such locations leading to high corrosion rates for

explosive joints in comparison with friction joints. In the case of Ti-Ti-304L SS joints, the interface between Ti (6 mm) and Ti (3 mm), and between Ti (3 mm) and type 304L SS is found to be generally very smooth and perfect without significant defective zones (Fig. 3(a)–(d)). However, some defects in the form of cracks and vortex attack could be noticed (Fig. 3(c) and (d)) at both interfaces between Ti (6 mm) and Ti (3 mm), and between Ti (3 mm) and type 304L SS after the corrosion test, in spite of low corrosion rates obtained by this buffer joint as compared to the direct Ti-304L SS joint. The surface appearance of the stainless steel portion is significantly different between the direct and buffer joined pieces after the corrosion tests. Very smooth surface with insignificant intergranular corrosion is noticed for the SS portion of the buffer joint as compared to severe intergranular corrosion and deformed structure exhibited at the SS portion of the direct Ti-304L SS joint (Fig. 4). Profilometric measurement across the interface of the corroded specimens clearly shows the presence of a 2500 μm wide gap due to corrosion attack of the friction joint tested in the condensate zone, while there was no such deep and wide gap is present at the interface for explosive joints due to corrosion attack (Figs. 5 and 6). In view of the observation that the 304L SS region only

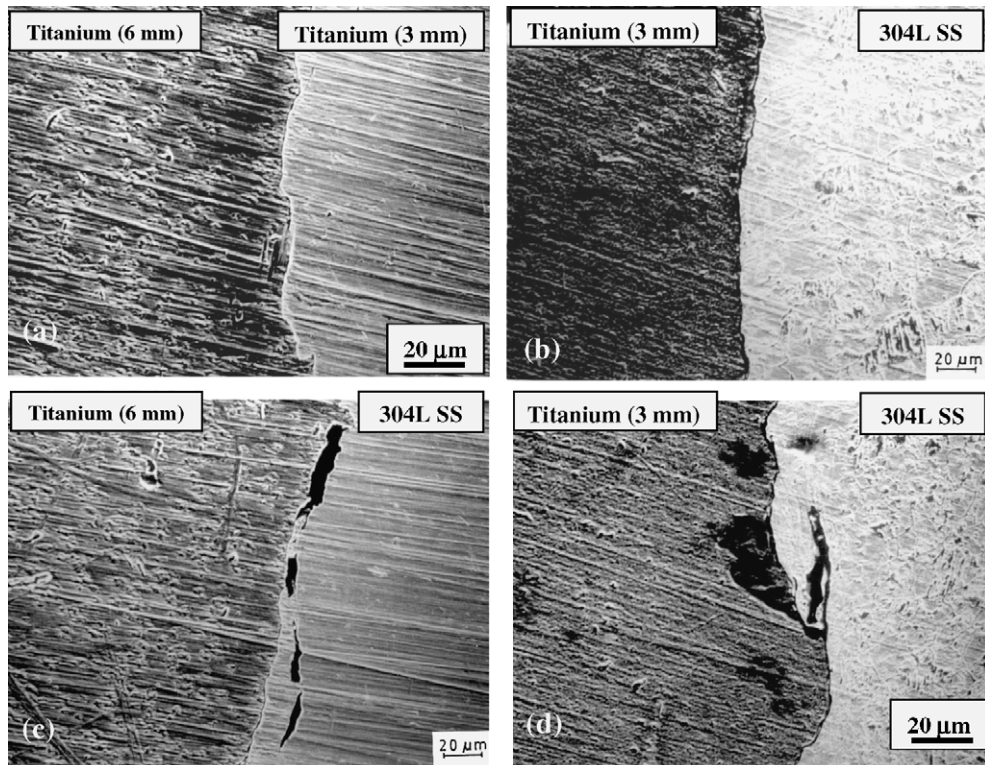


Fig. 3. SEM micrographs of interface of Ti-Ti-304L SS joint showing, (a) smooth joining of Ti (6 mm) and Ti (3 mm), and (b) smooth joining of Ti (3 mm) and type 304L SS (c) corrosion attack at Ti (6 mm) and Ti (3 mm) interface, and (d) corrosion attack at Ti (3 mm) and type 304L SS interface, after the corrosion test.

mainly corrodes for explosive joints in comparison with the severe corrosion attack at the interface of friction joints leading to ‘trench’ formation, explosive joints are chosen for this critical application. Between direct and buffer joints of Ti-304L SS, direct joints are considered superior due to better mechanical properties and bend-ductility.

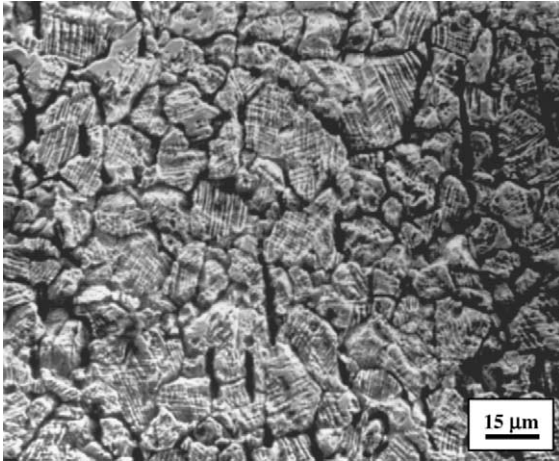


Fig. 4. SEM micrograph showing severe deformation and intergranular corrosion attack on SS portion of direct joint.

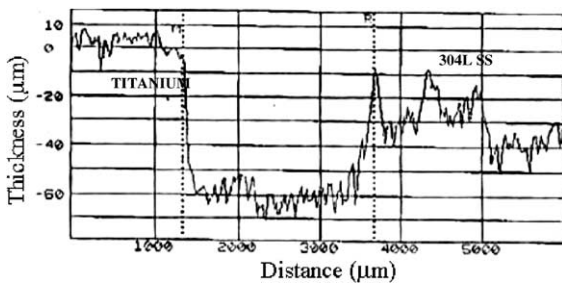


Fig. 5. Thickness profile of Ti+304L friction joint in condensate phase.

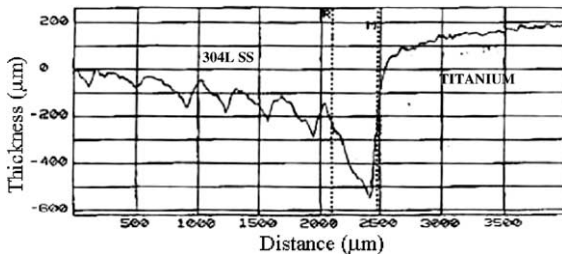


Fig. 6. Thickness profile of Ti+304L explosive joint in condensate phase.

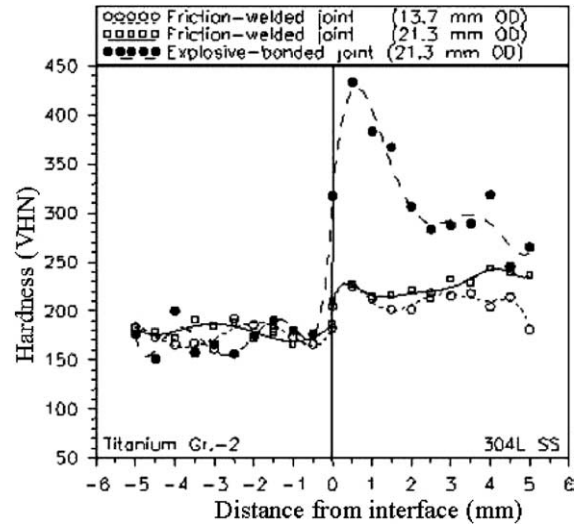


Fig. 7. Microhardness profile across the Ti+304L SS friction and explosive joints [11].

Microhardness measurements are also made across the titanium/304L SS interface of both the joints at an interval of 500 μm using a load of 200 g. The microhardness profiles for the joints are shown in Fig. 7. It is observed that the hardness of 304L SS near the interface increases by only about 50VHN for the friction-welded joint as compared to the increase of about 250 VHN for the explosive-bonded joint. The high hardness of 304L SS near the interface in the explosive-bonded joint can be attributed to the high degree of deformation/cold working of the SS surface during the explosive cladding operation. However, the titanium to 304L SS joint made with a buffer 3 mm titanium sheet shows an improvement with reduced hardness at the joint interface as shown in Fig. 8. This also supports the smooth and less intergranular corrosion attacked surface for the SS portion of the buffer joint as compared to severely deformed and corroded surface of SS portion of direct joint of Ti-304L SS shown in Fig. 4.

3.2. Joint preparation for equipment erection in plant

On comparison of the results of the various tests given in Table 3, it can be concluded that the explosive-bonded joint has significantly better bend- and tensile-ductility as compared to the friction-welded joint, with the other properties being comparable for both the joints. Since the joint did not have enough ductility (only 80°–100° bend was obtained as against 180° bend in the guided bend test), it is decided to have an additional outer Ti–Ti-304L SS sleeve over the dissimilar joint to protect it from stresses and strain. In addition to

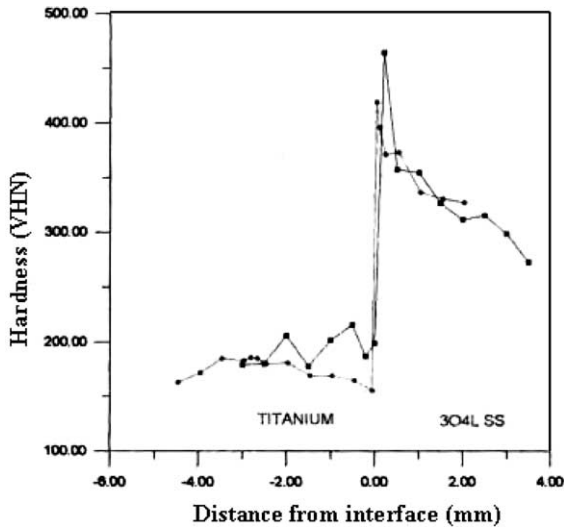


Fig. 8. Microhardness profile across explosively joined Ti + 304L SS and Ti + Ti + 304L SS (●—● Ti + Ti + 304L SS, ■—■ Ti + 304L SS).

catering the needs of strength requirements, the provision of Ti–Ti-type 304L SS sleeve provides extra corrosion allowances to the joint area. Since the titanium to titanium welding is close to the bonded area, additional precautions are taken such as: (1) providing a copper sink over the clad area for absorbing the heat and thus avoid the opening up of the bonded area, (2) follow sequence of machining, welding and boring as shown in

Fig. 9 to absorb the heat, and (3) carry out segment by segment welding, and after each segment and each pass of the welding, the temperature is brought down to room temperature as quickly as possible by forced air circulation cooling. After Stage 2 and 3 in Fig. 9, the DMW joint is inspected for radiography, fit up, visual and liquid penetrant examinations for ensuring the meeting of the specifications. Based on the results of the extensive investigations carried out, and considering corrosion resistance as the most critical requirement, the explosive-bonded titanium-304L SS joint is validated for connecting the titanium dissolver with other AISI type 304L SS process vessel piping.

Since titanium cannot be welded directly to AISI type 304L SS, a methodology had to be evolved for the erection of titanium equipment in AISI type 304L SS containment box of the cell. The welding of titanium equipment to SS by appropriate design is a preferred one instead of mechanical fastening as welding avoids reduction of vacuum in the box which is always maintained at a higher negative pressure as compared to the cell space. Welding operation also prevents the leakage of liquid from the containment box, whereas the same is not guaranteed with mechanical fastening. A 6 mm Ti Grade 1 plate is clad with a 12 mm 304L SS plate, which is further machined and welded to the top flanges of the dissolver. It is essential that while welding the clad ring to the dissolver flange at the bottom, it must be welded in segment by segment sequence (Fig. 10). In addition, cooling the welded segment to room temperature to avoid overheating of the clad portion

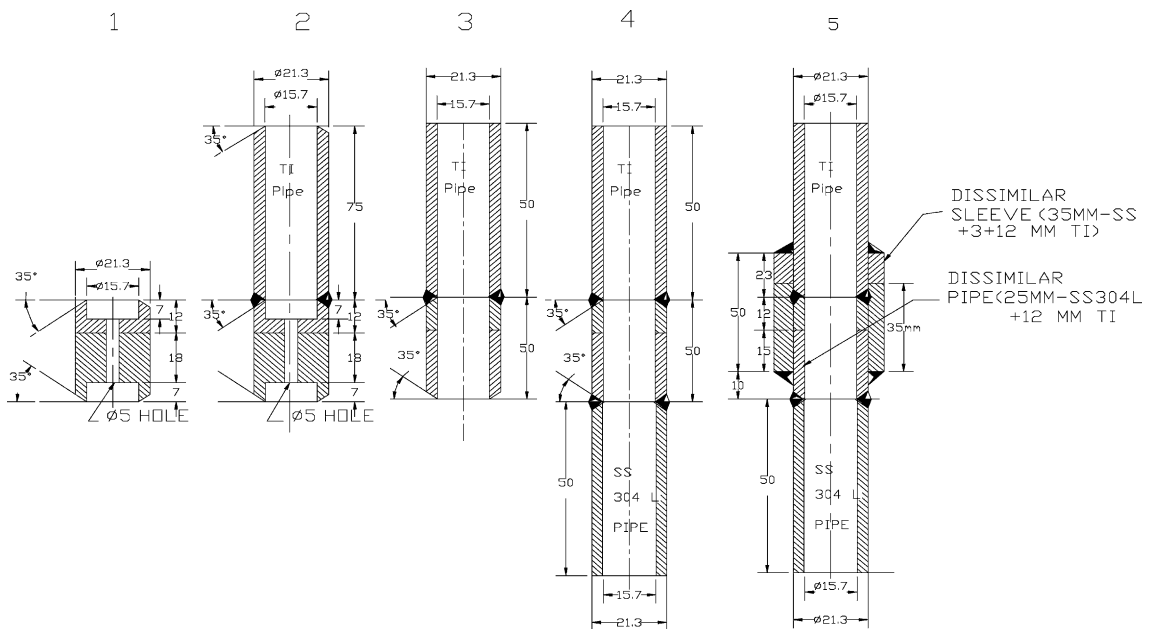


Fig. 9. Joint design and sequence of fabrication of Ti + 304L SS [11].

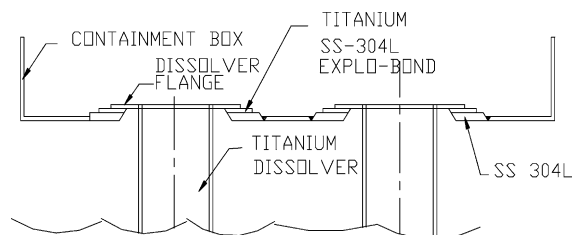


Fig. 10. Joint design for the erection of the dissolver.

which otherwise may lead to opening up of the bonded area is practised. Electrolytic dissolver system for the dissolution of the spent fuel from FBTR is thus linked to the rest of the plant made of type 304L SS through explosive joints of titanium and type 304L SS.

4. Summary

Among the various dissimilar joining processes discussed application of friction welding and explosive joining methods are considered with respect to joining of titanium and type 304L stainless steel. The dissimilar joints prepared are evaluated by non-destructive, mechanical and corrosion tests. The following are the main conclusions: (i) dissimilar joints made using the friction process failed in bend tests though the corrosion resistance in nitric acid is acceptable; however, the corrosion attack is very significant at the joint interface, (ii) dissimilar joints made using the explosive process are acceptable, as the joints possess adequate ductility during bend test; though the corrosion rate is high, it is acceptable as corrosion attack is on the stainless steel portion of the joint, (iii) dissimilar joints with an intermediate titanium sheet, made using the explosive process, show an acceptable corrosion rate, however, the joint failed in the bend test. Based on the results, direct titanium-304L SS joints manufactured using the explosive joining process is chosen for connecting the titanium

dissolver with 304L SS piping for application in the nuclear fuel reprocessing plant.

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