Evolution of Interface Microstructure and Mechanical Properties of Titanium/304 Stainless Steel Diffusion Bonded Joint Using Nb Interlayer

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In the present investigation, the evolution of interface microstructure and mechanical properties of diffusion bonded joints of titanium to Type 304 stainless steel with a niobium interlayer were studied. The joints were processed in the temperature range of 800 to 1000°C for 0.5 h in vacuum. The stainless steel/niobium interface was free from intermetallic phase up to 900°C; however, Fe2Nb + Fe7Nb6 phase mixture was observed at and above a processing temperature of 950°C. The niobium/titanium interface was free from intermetallic compounds at all processing temperatures. A maximum tensile strength of 297 MPa (~93% of Ti) and shear strength of 217 MPa (~75% of Ti) along with a 6.9% ductility were achieved, when processed at 900°C processing temperature. The failure of bonded samples took place through the stainless steel–niobium interface at all processing temperatures during loading.

KEY WORDS: diffusion bonding; niobium interlayer; electron probe microanalyser; scanning electron microscope; X-ray diffraction.

1. Introduction

The increasing use of Ti or Ti alloys in aerospace, power generation, transportation and chemical industries due to their high strength to weight ratio and excellent corrosion resistance, there is an obvious requirement to join them with stainless steel.1–4) Chemical, mechanical and structural heterogeneities can develop, when titanium (Ti) is bonded to stainless steel by fusion welding. Corrosive media can attack defects such as segregation of chemical species, stress concentration sites and intermetallic phases leading to premature failure of the components.5–7) Direct bonding of these materials can promote residual stresses at the weld interface due to the differences in thermal expansion and metallurgical microstructures between them, and formation of brittle phases and intermetallic compounds at the diffusion zone.8) There are indications in the open literature9–11) that atomic migration can occur during diffusion bonding of Ti to stainless steel, leading to the formation of δ, χ, FeTi, Fe3Ti, Cr7Ti, βTi, Fe7Ti6O phases in the reaction zone. These intermetallic compounds can impair the mechanical properties of the bonded joints. Kato et al.12) reported that diffusion bonding of Ti to stainless steel provides a maximum bond strength of ~60% of that of titanium along with ~2.2% breaking strain when processed with an range of 877–927°C.

In an earlier investigation, solid state direct bonding between titanium and 304 stainless steel was carried out in the temperature range of 800–950°C for 5.4 ks under a uni-axial pressure of 3 MPa and a bond strength of 242 MPa along with ductility of 5% was achieved though Fe3Ti5O, Fe7Ti6 and Cr7Ti were formed at the interface.13) The interlayer is primarily used to minimize the formation of brittle intermetallic phases, besides minimizing mismatch due to thermal expansion and reducing bond temperature and/or pressure.14) In a prior research work, the present authors improved the strength and ductility of the diffusion bonded joints between titanium and type 304 stainless steel using nickel (Ni) or copper (Cu) individually as an intermediate materials.15,16) In this respect niobium can also be considered as a intermediate materials to improve the mechanical properties. Phase diagram shows that pure titanium and pure niobium does not form any intermetallic compound. However, it is known that intermetallic compounds such as NbNi3 and NbNi can be formed with increasing Ni content and intermetallic compound of Cr–Nb can form at 63–69 at% of Cr. The binary phase diagram of Fe–Nb shows that the intermetallic compounds including Fe2Nb and Fe7Nb6 can be formed at 22–42 at% of Nb and 47–50 at% of Nb, respectively.17)

The present study reports the diffusion bonding between Ti and type 304 stainless steel using niobium as an intermediate material and also presents the interface microstructure and the mechanical properties of the bonded joints. The bonding temperature was varied to observe the change in interface microstructure, which in turn influences the mechanical properties of the diffusion bonded region.
2. Experimental

2.1. Base Materials

Type 304 stainless steel (SS) and commercially pure titanium (Ti) were used in the form of cylindrical rod having a 35 mm diameter in hot rolled condition. Pure Nb foil (99.4 wt%) having a 300 μm thickness was used as an intermediate material. The chemical composition of commercially pure titanium (Ti) is Ti–0.02C–0.10Fe–0.15O–0.02N–0.0011H and that of 304 stainless steel (SS) is Fe–0.06C–1.38Mn–0.37Si–0.03P–18.15Cr–8.50Ni–0.005N (wt%). The room temperature mechanical properties of the base metals are presented in Table 1. The optical micrographs and X-ray diffraction of the base materials are given in Fig. 1. Commercially pure titanium has a (hcp) structure and the stainless steel has austenite (fcc) containing twins. Cylindrical samples having length and diameter of 30 mm and 15 mm, respectively were machined from 35 mm diameter rods of the parent metals.

2.2. Preparation of Joints

In all cases, mating surfaces of Type 304 SS and Ti samples were prepared by conventional grinding and polishing technique to obtain a surface roughness of ~0.6 μm. Nb foil having a thickness of 300 μm was used as an intermediate material, and both surfaces of the interlayer were polished in a same fashion. The mating surfaces were cleaned in acetone and dried in air. The SS–Nb–Ti assembly (SS is in lower and Ti is in upper side) was kept in contact in a fixture made of stainless steel and was then inserted in a vacuum chamber. Diffusion bonding of this assembly was carried out at temperatures ranging from of 800 to 1000°C in steps of 50°C for 0.5 h in (2–5)×10⁻⁴ Pa vacuum. A compressive stress of 3 MPa was applied along the longitudinal direction of the sample. Heating was done at a rate of 0.24°C s⁻¹ at the time of processing and after the operation, and the samples were allowed to cool in vacuum at a cooling rate of 0.1°C s⁻¹ up to 300°C.

2.3. Microstructural Characterizations

For metallographic examination, the bonded joints were sectioned in a transverse direction to the bond line, grounded and polished. The polished surfaces of the bonded couples were examined in a scanning electron microscope (Leica S440) in back scattered mode (SEM-BSE) to obtain the finer structural details in the diffusion zone. The electron probe microanalyser (CAMECA SX 100) was used to get the elemental concentration profiles across the diffusion interfaces. The $k_\alpha$ lines of Ti, Fe, Ni and Cr were generated at an operating voltage of 15 kV and a current of 12–18 µA. LiF crystal was used to diffract the corresponding characteristic X-ray radiation. The presence of intermetallic phases on the fracture surfaces was confirmed by the X-ray diffraction technique (Philips PW 1840) using a Co target. A scanning range of 30–100° with a step size of 0.01° ($2\theta$) was used during the diffraction study. A fracture surface of the bonded samples were observed in secondary electron mode of SEM using energy dispersive spectroscopy (Oxford 5431) to reveal the nature and location of failure under loading.

2.4. Evaluation of Mechanical Properties

Tensile properties of the bonded joints were evaluated in a universal testing machine (Instron 4204) at a crosshead speed of 8.33×10⁻⁴ ms⁻¹ at room temperature. Cylindrical specimens were machined according to the ASTM specification E 8M-96 with gauge diameter and length of 4 mm and 20 mm, respectively. The interlayer was at the centre of the gauge length. Shear strength of the bonded joint was evaluated at room temperature using a screw tensile testing machine set at a crosshead speed of 8.33×10⁻³ ms⁻¹. The specimens used in shear testing were machined to a diameter of 10 mm. The intermediate material of the specimen was at the centre of the two jaws. The design of the jaws is given in Fig. 2. Four samples were tested at each process parameter to check the reproducibility of results. The micro-hardness measurement of the polished bonded samples were carried out on a diamond micro-indenteter using a 15 gf load for 15 s duration.

Table 1. Mechanical properties of the base metals at room temperature.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Shear strength (MPa)</th>
<th>0.2% Yield stress (MPa)</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Fracture elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium</td>
<td>296.6 ± 2.5</td>
<td>205 ± 2</td>
<td>319 ± 3</td>
<td>23 ± 0.9</td>
</tr>
<tr>
<td>Stainless Steel</td>
<td>573.0 ± 3</td>
<td>740 ± 4</td>
<td>822.5 ± 6</td>
<td>42 ± 2</td>
</tr>
</tbody>
</table>

Fig. 1. Characteristics of the base metals (a) optical micrograph of titanium (b) optical micrograph of stainless steel, (c) XRD patterns.
3. Results and Discussion

3.1. Microstructural Characterization of the Diffusion Interface

SEM micrographs of the diffusion bonds are shown in Fig. 3. The diffusion zone is free from cracks for all the bonded joints. It is observed that the diffusion interfaces of the bonded joints are clearly visible without any discontinuity and diffusion occurs between the interlayer and the two substrates. The SEM micrographs at higher magnification and elemental concentration of SS–Nb and Nb–Ti interfaces of bonded joints are given in Fig. 4 and Fig. 5, respectively. The SS–Nb interface is free from intermetallics up to the processing temperature of 900°C and above this temperature a thin diffusion layer has been observed at the Nb side. Nb has bcc crystal structure at all temperatures. Due to more open crystallography of bcc structure, iron atoms can travel a longer distance in Nb lattice compared to the distance traveled in the stainless steel matrix. The thin diffusion layer was found to be of Fe (37.1–38.9 wt%) and Nb (56.8–60.9 wt%) with a small amount of Cr (1.2–2.5 wt%) and Ni (bal). Hence, the Fe–Nb binary phase diagram indicates that, this layer is perhaps the Fe9Nb3+Fe2NbB phase mixture. The width of this layer is 1.1 μm at 950°C and is increased to ~2.7 μm at the processing temperature of 1000°C.

The Nb–Ti interface is characterised by the presence of a light shaded reaction zone which is Widmanstätten α–β Ti shown in the SEM-BSE images (Fig. 4). Nb is a β-stabilising element and after its migration in the Ti lattice, it can lower the eutectoid transformation temperature of Ti formed β titanium and the acicular α–β Ti due to the decomposition of β-Ti during cooling. The Nb–Ti interface is free from any other intermetallics. The presence of Ti (~9.1–19.2 wt%) in Nb side (near the interface) indicates that substantial diffusion of Ti in to Nb take place at bonding temperatures. Similarly, Nb also migrated to the Ti substrate in larger quantity (~21.2–35.9 wt%) and formed the β-Ti phase.

The result of X-ray diffraction studies confirmed the presence of different intermetallics at the fracture interfaces as shown in Fig. 6. The X-ray diffraction spectra identified the formation of intermetallic phases like Fe2Nb, Fe6Nb7, FeNbO4 and NbO2 which are present for all the processing temperatures. SEM micrographs shown in Fig. 4, did not reveal the presence of Fe2Nb and Fe6Nb7 intermetallic phases at the SS–Nb interface formed at temperature at or below 900°C due to very low volume fraction. Formation of FeNbO4 and NbO2 observed in the X-ray diffraction pattern presumably due to the higher affinity of Nb for oxygen. SEM micrographs did not reveal them due to their low volume fraction. Ti oxide has not been detected in XRD pattern due to the fact that fracture occurred place through the SS–Nb interface.

3.2. Mechanical Properties of the Diffusion Bonded Joints

The variations in mechanical properties of the diffusion bonded joints with the change in bonding temperature are shown in Fig. 7. It is observed that the bond strength gradually increases with the increase in temperature up to 900°C and again decreases with the increase in the bonding temperature. At a lower processing temperature of 800°C, bond strength is low due to the less contact between the faying surfaces as the yield stress of the material still remains high and the plastic collapse of the mating surfaces is also minimum. With the increase in bonding temperature, contact between the mating surfaces increases due to the increase in plastic collapse and the tensile strength of the bond materials gradually increases. Maximum tensile strength of ~296.5 MPa and shear strength of ~216.5 MPa along with ~6.9% elongation are obtained at 900°C due to the increased contact area between the mating surfaces and the absence of intermetallic phases. There are indications in the open literatures that the strength and ductility simultaneously drop with the increase in bonding temperature. The tensile strength exhibits a substantial increase with respect to the values of those of directly bonded commercially pure titanium and stainless steel joints.

With a further rise in bonding temperature to 950°C and above, the coalescence of mating surfaces increases. However, Fe2Nb+Fe6Nb7 intermetallics phase mixture is formed at the SS–Nb interface and their thickness increases with the increase in bonding temperature. So, the bond strength gradually drops and attains lowest value at 1000°C processing temperature (tensile and shear strength are ~237.2 and ~166.2 MPa, respectively). Figure 8 shows the micro hardness of the bond interfaces at various bonding temperatures. At 950°C and above bonded samples have maximum hardness values at the SS–Nb interface as compared to the other bonded samples due to the presence of Fe–Nb based intermetallic compound at the bond interface. However the hardness values are more or less same at the Nb–Ti interface for all the processing temperature. Hardness values at
the Nb–Ti interface are higher as compared to the base materials presumable due to the presence of $\beta$-Ti phase.

3.3. Fracture Surface of Bonded Joints

Figure 9 shows the fracture surfaces of the diffusion bonded joints. Inter-granular fracture has been observed in all the fracture surfaces. Fracture surface of the sample processed at lower temperature (Fig. 9(a)), voids are observed and these voids decrease with the increase in bonding temperature due to the larger contact between the mating surface increases. The bright and shaded regions have been observed in fracture images. The bright region of the fracture surface at higher processing temperature is Fe$_x$Nb+$\alpha$-Ti phase mixture with the composition of Nb ($\sim$56.8–60.9 wt%) and Fe ($\sim$37.1–38.9 wt%), Cr (1.2–2.5 wt%) and Ni (bal.). The shaded region is a solid solution of

![Fig. 4. SEM-BSE images of diffusion bonded joints at higher magnification processed at (a) and (b) 850°C; (c) and (d) 900°C; (e) and (f) 950°C; (g) and (h) 1000°C.](image)

![Fig. 5. Quantitative elemental concentration profiles of the bonded samples processed for 0.5 h at (a) 900°C, SS–Nb interface; (b) 900°C, Nb–Ti interface; (c) 1000°C, SS–Nb interface; (d) 1000°C, Nb–Ti interface.](image)
Fe and Cr in niobium with Fe (6–8 wt%) and Cr (1.5–3 wt%). From the fracture surface, it is revealed that failure takes place through the stainless steel–niobium interface.

4. Summary and Conclusions

The evolution of interface microstructure and mechanical properties of diffusion bonded joints of titanium to type 304 stainless steel with a niobium interlayer were investigated and the following conclusions are drawn.
1) The SS–Nb interface is free from intermetallic compounds up to 900°C processing temperature; however, the layer wise Fe$_2$Nb+Fe$_7$Nb$_6$ phase mixture is observed at 950°C and above processing temperatures. The Nb–Ti interface is free from intermetallic compound for all the processing temperatures.
2) The maximum tensile strength of 297 MPa and shear strength of 217 MPa along with 6.9% ductility have been achieved, when bonded joints processed at 900°C temperature.
3) X-ray diffraction and fracture surface studies reveal that failure takes place through the stainless steel–niobium interface.

REFERENCES