Effect of Microstructure on Mechanical Properties of Friction-Welded Joints between Ti and AISI 321 Stainless Steel

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The microstructures and mechanical properties of the friction welded pure Ti/AISI 321 stainless steel have been investigated. From the Ti side from the interface to the Ti base metal, the sequence of recrystallized grain, elongated grain and many twin embedded grain structures were observed. The reaction layers were formed within 0.2 μm thickness at the interface under the conditions of relatively longer friction time ($t_1$) and lower upset pressure ($P_2$). These reaction layers formed at the central interface were identified as FeCr ($\sigma$ phase), (Fe, Cr)$_2$Ti, FeTi and $\beta$-Ti. The $\sigma$ phase was restrictedly formed at the peripheral interface. Higher mechanical properties were acquired under higher upset pressure condition due to higher compressive force between bonded materials, smaller grain size and narrower thickness of reaction layer. Therefore, maximum ultimate tensile strength of these joints was approximately 420 MPa with the conditions of 400 MPa of $P_2$ and 0.5 s of $t_1$.

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1. Introduction

Ti and its alloys have high specific strength and good erosion resistance, thus, they have been widely used in aerospace, chemical and nuclear industries.1,2,3 With increased use of Ti and its alloys, the bonding of Ti and its alloys to structural steels become more and more important. Especially, the dissimilar joint between Ti and stainless steel has been widely applied in nuclear industries and as accessories in oil-rig of refineries.1,3,4 The bonded couples should have leak tightness, easy fabricability and adequate strength for their efficacious use.4

The conventional fusion welding techniques between Ti and stainless steel have resulted in the segregation of chemical species, stress concentration and formation of intermetallics. Especially, the combination between Ti and Fe alloys makes lots of intermetallic compounds to form at the interface because Ti and Fe are not completely soluble in solid state.5,6 Fusion welding methods also involve melting and solidification of the base metals, which results in shape distortion of the welded metals and excessive generation of strain at the interface due to the difference in the coefficient of linear expansion in case of joining between dissimilar metals. Therefore, solid bonding techniques, diffusion bonding and friction welding should be applied to acquire the sound joints of the dissimilar metals.

Although sound joints between Ti and stainless steel were successfully achieved using diffusion bonding method,3,4,7 the brittle intermetallic compounds for example, Fe$_2$Ti, FeTi and Fe$_2$Ti$_2$O, were widely formed in the interface. The diffusion bonded joints represented a lower strength than those of the base metals due to thicker intermetallic compounds.

Friction welding carried out in the solid state has represented the superior joint strength when joining dissimilar materials9–12) because this can minimize the formation of the brittle intermetallic compound at the interface. In the friction welding process, a frictional heat is generated by the conversion of mechanical energy (rotation of specimens, compressive force) into thermal energy at the contact area of the workpieces without the use of the electrical energy or heat from other sources.

Friction welding has locally introduced deformed microstructures near the weld zone because the surrounding of the joints received the different thermo-mechanical effects and microstructural change can be observed mainly in more ductile materials.10–12) However, microstructural analysis near the weld zone has not been clearly developed yet. Therefore, this study aims to observe microstructures near the friction welded Ti/AISI 321 interface and to evaluate the effect of the microstructural issues on the mechanical properties.

2. Experimental Procedure

The materials used in the present work were pure Ti (grade II) and commercial available AISI 321 stainless steel, which were machined to 16 mm in diameter and 100 mm in length. The chemical compositions are shown in Table 1.

The surfaces of the parts were ground with SiC paper (#100) and cleaned with acetone before welding. Friction welding was carried out using a brake type friction welding machine (Nitto Seike Co. Ltd), which has various welding parameters such as rotating speed $N$, friction time $t_1$, upset time $t_2$, friction pressure $P_1$ and upset pressure $P_2$. In this present work, $P_1$, $t_2$, and $N$ were fixed at 100 MPa, 5 s and 2000 min$^{-1}$, respectively, while $t_1$ was varied from 0.1 s to 2.0 s and $P_2$ was changed from 100 to 475 MPa.

The resultant welds were sliced by EDM (electron discharge machine) to provide cross-section and then ground with SiC paper (#100-#2000), and finally micro polished using 0.3 μm AI$_2$O$_3$ powder. The microstructures of friction welded interfaces were observed by OM (Optical Microsco-
of incidence of 10°. These thin specimens were perforated by ion milling at 5 kv and using initially an angle of incidence of 10°, which was decreased after perforation to 5° to obtain an electron transparent region at the interface. These thin foils were observed using a 300 Kv high resolution TEM. The phase of the reaction products was analyzed by EDS (Energy Dispersive Spectroscopy) equipped in TEM and SAED (Selected Area Electron Diffraction) method.

Tensile tests were carried out to evaluate the mechanical properties of the joints. Tensile test was performed at room temperature using an Instron type testing machine with a cross head speed. The Vickers hardness was measured with a load of 1.96 N, for 10 s.

### 3. Results and Discussion

Figure 1 represents macroimages near the interfaces of Ti/AISI 321 with different friction welding conditions. The flashes (or welding burr) were restrictedly formed at the pure Ti side due to more ductile property at high welding temperature. On Ti side, microstructural deformed regions are observed within 0.5 mm from the interface and the widths are changed with welding conditions, while AISI 321 isn’t macroscopically deformed. It can be observed that the width of deformed region increases from the central to peripheral region because the peripheral region experienced more severe plastic deformation and reached higher temperature.

### Table 1 Chemical compositions of the starting metal.

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<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
<th>Cr</th>
<th>Fe</th>
<th>H</th>
<th>O</th>
<th>N</th>
<th>Ti</th>
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<tr>
<td>Ti</td>
<td>0.08</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>0.25</td>
<td>0.015</td>
<td>0.2</td>
<td>0.03</td>
<td>Bal</td>
<td></td>
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<tr>
<td>321</td>
<td>0.08</td>
<td>1.0</td>
<td>2.0</td>
<td>0.045</td>
<td>0.03</td>
<td>11.3</td>
<td>17.32</td>
<td>Bal</td>
<td>—</td>
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To observe details of the deformed structure on Ti side, higher magnifications of optical microstructures are shown in Fig. 2. Figure 2(a) represents the microstructure of the peripheral interface. The grain structure of Ti at the interface is finer compared to that of Ti base metal (d) and equaxed. The grain size of Ti at the interface is 22 μm which is approximately ten times smaller than that of the base metal having 186 μm of grain size. These fine and equaxed grain structures were caused by the recrystallization process during the welding because the welded materials near the interface simultaneously received both frictional heat and plastic deformation. Therefore, original grains were severely broken and a dynamic recrystallized grains were generated and statically grew after welding process. The reaction layer between Ti and AISI 321 couldn’t be discerned by the limited resolution of optical microscopy due to its thin thickness. Region (b), where is located next to recrystallized structure, shows a perpendicularly elongated grain structure against the direction of the compressive force and twin structure in some grains. This structure seemed to be shear band structure which was observed in the plastically deformed materials. This region didn’t receive sufficient thermo-mechanical effect enough to form recrystallized grains. Grains in region (c) have lots of twin embedded in the grain with a similar grain size with that of Ti base metal. These twin structures are more clearly observed in the peripheral side. The formation of these shear band and twin structure during the friction welding can be explained by the HCP crystal structure of Ti because shear band and twin structure cannot be observed in case of friction welded Al alloy joints which has higher stacking fault energy (SFE). HCP structured materials such as Ti and Mg which have relatively lower stacking fault energy, represents the remarkably developed twin and shear band structure near the interface.

The recrystallized grains formed during the welding might statically grow after welding during air cooling. Therefore, the Ti grain size near the interface depends on the temperature gradient with each weld region and welding parameters. Figure 3 shows the grain size variation according to the central and peripheral interface with welding parameters (P1 and t1). The recrystallized grain size of Ti at the central and peripheral interface shows 21.9 and 31.2 μm, respectively, with condition of P1 = 150 MPa and t1 = 0.5 s. A longer friction time (t1 = 2.0 s) condition promoted grain growth at both regions and grain of the central and peripheral interface grew to be 34.6 and 42.5 μm, respectively, while a higher P2 (300 MPa) repressed a grain growth at the interface. At the peripheral region, the recrystallized grain size is larger than that of the central region. Because the
peripheral region received a higher degree of plastic deformation and reached a higher temperature, there existed a higher driving force for recrystallization and grain growth in order to eliminate the plastic strain energy.\textsuperscript{15} A longer $t_1$ also elevated the temperature of the interface and accelerated the grain growth.

Figure 4 shows TEM microstructure of the central interface and EDS spectrums of each layer with the condition of $P_2 = 200$ MPa and $t_1 = 2.0$ s.
on Ti side is observed and dislocations are also found in the recrystallized grain. Two kinds of reaction layers (a, b) and spherical shaped particles (c) are observed at the interface between Ti and AISI 321. The thickness of reaction layer is approximately within 0.2 μm under the condition of relatively longer \( t_1 = 2.0 \) s and lower \( P_2 = 200 \) MPa, which make broader thickness of reaction layer to be formed in the interface. The formation of reaction layer should be minimized by applying relatively shorter \( t_1 \) and higher \( P_2 \) welding condition. The diameter of spherical shaped particles is about 0.02 μm. The layer (a) is composed of 54.07 at% of Fe, 29.1 at% of Ti, 9.23 at% of Cr and balanced Si. Therefore, this layer can be identified as \((\text{Fe, Cr})_2\text{Ti}\) intermetallic compound by binary phase diagrams of Fe-Cr and Fe-Ti. Layer (b) is composed of Ti (65.2%), Fe (28.2%) and some of Cr and Ni. At first sight this layer appears to correspond to some form of intermetallic \((\text{Fe, Ni})_2\text{Ti}\), but such a phase has not been referred in the Fe-Ti binary phase diagram. Some discussion about existence of \(\text{FeTi}_2\) phase could be found in the literature, but such a structure has been designated as \(\text{Fe}_3\text{Ti}_2\text{O}\) phase. Chemical composition of this layer could identify this layer as \(\text{FeTi}_2\) intermetallic. However, because there was some possibility of the existence of oxygen which cannot be escaped during applying compressive force in the central interface, this layer also might be identified as \(\text{Fe}_2\text{Ti}_2\text{O}\) oxides. Particle C is composed of 82.5% of Ti, 14% of Fe and 3.84% of Cr. These particles are identified as the transformed \(\beta\)-Ti (BCC) because Fe and Cr known for \(\beta\)-Ti stabilized elements\(^{3,16}\) diffused into Ti side. These particles are mainly distributed at the Ti side of the interface.

Figures 5 and 6 represent TEM microstructures of the peripheral interface and the electron diffraction patterns obtained from the each layer with condition of \( P_2 = 200 \) MPa and \( t_1 = 2.0 \) s. Three different layers were formed at the peripheral interface between Ti and AISI 321. Layer (a) formed near AISI 321 stainless steel side has the composition of 64.1% of Fe, 25.3% of Cr, 6.5% of Ti and balanced Ni. This layer can be identified as \(\sigma\) phase with tetragonal
Especially, the formation of $\sigma$ phase was limited at the peripheral interface of friction welded Ti/AISI 321 stainless steel. Layer (b) is composed of 55.9% of Fe, 14.6% of Cr and 25.9% of Ti, respectively and therefore identified by (Fe, Cr)$_2$Ti phase. Layer (c) formed near Ti side is FeTi compound because this layer has a similar at% of Fe (42.5%) and Ti (44.7%). SAEDs (Select Electron Diffraction Pattern) (Fig. 6(a)) obtained near AISI 321 side represent the overlapped $\gamma$ austenite structure and $\sigma$ phase. Those obtained (Fig. 6(b)) near the Ti side are also overlapped FeTi phase with simple cubic structure and $\beta$-Ti phase with BCC structure. It was difficult to discern (Fe, Cr)$_2$Ti phase with SAED patterns in this study because this layer was very thin layer and some diffraction positions were overlapped with other layers.

In general, $\sigma$ phase is formed by aging during long times at temperatures between 773 and 1073 K because the direct decomposition of austenite to $\sigma$ phase requires long times due to the accompanying redistribution of alloying elements by substitutional diffusion. For example, the start of $\sigma$ phase precipitation was detected in AISI 321 austenite stainless steel aged at 873 and 973 K for 72 hour. $^{17}$ The restricted formation of $\sigma$ phase at the peripheral interface of friction welded AISI 321/Ti was interesting result because the joints were maintained during very short time within approximately 10 s at high temperature. Therefore, there existed another factors to control the formation of $\sigma$ phase at the peripheral interface of friction welded AISI 321/Ti. Although the temperature near the weld zone cannot be easily measured due to the rotation of welded specimens, it might be less than 1473 K which corresponds to delta ferrite transformed temperature from the austenite phase. The formation of $\sigma$ phase can be accelerated in the duplex microstructure of delta-ferrite and austenite phase because the presence of delta-ferrite in the austenite stainless steel is an important factor for $\sigma$ phase kinetics. $^{18-20}$ Some studies reported that the severe plastic deformation and recrystallization accelerated the formation of $\sigma$ phase in the 308 stainless steel $^{21}$ microduplex steel $^{22,23}$ and 304 austenite stainless steel $^{24}$ in friction welding process, each central and peripheral interface experienced the different thermo-mechanical conditions due to the different regional velocity. $^{20}$ For example, the velocity of the central interface was 0 m/s, while that of the peripheral interface reached 3 m/s. Therefore, the peripheral interface received more severe plastic deformation and reached higher temperature than those of the central interface. The peripheral interface also experienced the dynamic or static recrystallization. The restricted formation of $\sigma$ phase at the peripheral interface without additional aging treatment can be explained by some reasons such as the severe plastic deformation, higher temperature and recrystallization during short welding time.

Figure 7 shows the hardness distributions near the interfaces with welding conditions. Figures 7(a) and (b) represent the central and peripheral region, respectively. Both AISI 321 and Ti near the interfaces show higher hardness compared to those of Ti and AISI 321 base metal. The hardness of AISI 321 at the interface is approximately 210 HV and the hardened region reaches 4 mm from the interface. This result might be caused by the partial formation of martensite phase. Fukumoto et al. $^{25}$ reported that the interface of austenite stainless steel friction welded to 1050 Al was hardened because strain-induced martensite phase was formed. Our previous studies also reported the martensite phases were formed at the interface of friction welded Cu/carbon steel $^{12}$ and TiAl/AISI 4140 steel $^{20}$ due to rapid cooling after welding process. More detail microstructural inspection on AISI 321 stainless steel side will be carried out in future work. Hardness distributions near the Ti side near the peripheral interface are shown in Fig. 8. The hardness of the unaffected Ti base metal is about 170 HV. The remarkably hardened region (a) is observed within 0.2 mm from the interface because this region contained a very fine recrystallized grain structure and brittle $\beta$-Ti. Hardness of these regions with welding conditions is closely related with the grain size and represents a range of 220–245 HV. Region (b) and (c) represent the regions with elongated grain and twin structure, respectively. These regions also show slightly higher hardness than that of the Ti base metal due to a work hardening effect.
Figure 9 shows the ultimate tensile strength with welding conditions and fractured specimens. Tensile strength totally increased with increasing \( P_2 \), while \( t_1 \) had a little influence on the tensile strength of the joints. A longer \( t_1 \) is not the recommended welding condition because of large materials consumption.

Ultimate tensile strength reached that (430 MPa) of Ti base metal when \( P_2 \) was over 400 MPa, and fracture occurred at the Ti base metal as shown in Fig. 9(b). Fractured image (c) obtained by SEM (scanning electron microscopy) represents a ductile mode which is a very similar to that of the Ti base metal (d). However, when \( P_2 \) was 300 MPa, fracture occurred both interface and Ti base metal and when \( P_2 \) was less than 200 MPa, fracture position was limited at the exact interface regardless of \( t_1 \). Maximum ultimate tensile strengths of friction welded Ti/AISI 321 stainless steel joints reach approximately 420 MPa over 400 MPa of \( P_2 \) within 2.0 s of \( t_1 \).

Higher \( P_2 \) acted as the positive effects on the joints strength due to many microstructural factors. First of all, higher pressure can give a higher compressive force to the bonded interface which enhanced the joinability between two dissimilar materials. Formation of brittle intermetallic compounds can be also restricted within narrow limits by higher upset pressure. Moreover, smaller size of recrystallized grain might increase the mechanical properties near the interface.

It has been already known that a large amount of the reaction layer can be formed under longer friction time condition. In the present study, there was little relationship between tensile strength and friction time at the constant \( P_2 \) compared to that of upset pressure at the constant \( t_1 \). That was because the experimental range of friction time was shortened within 1.5 s, therefore, the thickness of reaction layer was not much different. However, tensile strength was slightly higher at a shorter \( t_1 = 0.5 \) s.

Tensile strength of friction welded Ti/AISI 321 stainless steel shows a higher value than those of diffusion bonded Ti/stainless steel.\(^{3-9}\) A higher joints strength of friction welded Ti/AISI 321 could be explained by the narrower reaction layer within 0.2 \( \mu \)m, which enhanced the joinability of friction welded Ti/AISI 321 stainless steel.

4. Conclusions

One of solid state bonding techniques, friction welding was applied to join the Ti and AISI 321 stainless steel and superior joint strength was acquired. Main conclusions of this study are as followed

1. Microstructure of Ti side was severely deformed near the interface. The recrystallized grain, elongated structure and lots of twin structure in grains were formed in sequence from the interface to Ti base metal.

2. The reaction layers were thinly formed within 0.2 \( \mu \)m at relatively longer friction time (2.0 s) and lower upset pressure (200 MPa). The layer thickness was thinner than those of other welding techniques. These reaction layers were identified as \((\text{Fe, Cr})_2\text{Ti}, \text{FeTi}_2\) or \(\text{Fe}_2\text{Ti}_4\)O and \(\text{FeCr(C27 phase)}, (\text{Fe, Cr})_2\text{Ti}, \text{FeTi}\) and \(\beta\)-Ti at the peripheral region from AISI 321 stainless steel to Ti in sequence.

3. The restricted formation of \(\text{FeCr(C27 phase)}\) at the peripheral interface during a short welding time was explained by the severe plastic deformation, a higher
temperature and recrystallization during the process.

(4) The hardness near the interface increased comparing with those of Ti and AISI 321 stainless steel. Ultimate tensile strength of friction welded Ti/AISI 321 stainless steel joints showed about 420 MPa over 400 MPa of $P_2$ within 2.0 s of $t_1$. Tensile strength of friction welded Ti/AISI 321 stainless steel reached a higher value due to the prevention of the brittle reaction layers within the limited range.

REFERENCES