A Study on the Microstructure, Mechanical Properties, and Corrosion Behavior for Friction Stir Welded Ti-6Al-4V Alloys

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Abstract—**Friction stir welding is an emerging severe plastic deformation technology and has been used to produce a butt-joint on Ti-6Al-4V alloys sheet with a thickness of 2.5mm at rotation speeds of (1500 and 2000 rpm) and a constant travel speed of 200 mm/min. The effect of welding and rotation speeds on microstructure, tensile strength, and corrosion resistance property has been investigated. Micro-structural study of welded joint specimens has been performed by scanning electron microscopy for both stir zone and thermo-mechanically affected zone. Tensile property and micro-hardness studies have been done to identify the joint strength. Micro-structure correlates with the mechanical property. XRD has been investigated to distinguish between α and β phases. Finally, the EBSD has been conducted to identify the recrystallization phenomenon in the stir zone. A corrosion study has been performed for friction stir welded joints in Ringer's solution which is equivalent to body fluid solution. Finally, the Tafel plot analysis has been employed to determine the corrosion rate for SZ and TMAZ. We found 96% of joint strength achieved for friction stir welded joint than the base metal. This study promises applications not only in the welding industry but also in other fields of engineering technologies and our practical life.**

Index Terms— **Friction Stir Welding (FSW), Ti-6Al-4V alloys, Stir Zone (SZ), Thermo-Mechanically Affected Zone (TMAZ), tensile strength**

I. Introduction

The Friction stir welding - FSW technique is a new solid-state patented solid-state joining process in 1991 by the Welding Institute (TWI). During the welding process, a rotating welding tool with a pin at its end placed between two plates is welded into a butt joint and then moves along the welding line. Besides, the peak temperature is often lower than the melting points of the material, therefore, many drawbacks can be minimized or prevented, such as large distortions, hot cracks, and coarse microstructure [1-7]. Titanium and titanium ~~alloys~~ like particularly Ti-6Al-4V alloy, are widely used in many industrial fields such as aerospace, chemical, nuclear industries, and most importantly for biomedical applications due to their high specific strength, erosion with the benefits of high specific strength and good corrosion resistance. Microstructure and mechanical properties of Ti-6Al-4V alloy joints, comparable to aluminum alloy friction stir welding joints have been extensively researched [8-14]. The restricted availability of titanium alloy on Friction stir alloy data could be attributed to the elevated process-related temperatures which makes it considerably complex to conduct. The low thermal conductivity results of titanium at very high temperatures are produced at the base metal interface of the instrument, requiring high strength tool materials and a cooling system to protect the equipment. Besides, the elevated chemical reactivity of titanium to oxygen at elevated temperatures requires protective measures to prevent the diffusion of oxygen and hydrogen within the joint area from being changed. Therefore, the previous work on titanium alloy by friction stir welding focused primarily on tool materials and geometry or optimization of process parameters [15, 16].

For several recent decades, the need for titanium and their alloys has been increased in the aerospace sector in terms of resource scarcity and their growing costs. Titanium and its alloys are employed commonly in the welding industry. They are welded using conventional fusion welding processes. But the applied fusion welding to titanium and their alloys forms a brittle microstructure, large deformation, and high residual stresses after welding [17]. Friction Stir Welding used to avoid many

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problems related to the fusion welding process, the weld formed with fewer defects and having excellent properties, even in some materials with poor welding properties [18]. Low and high melting point materials have been widely investigated such as Aluminum, Copper, Magnesium, Steel, Stainless steel, etc could be a promising solution for joining similar and dissimilar metals using friction stir welding [19-24]. The suitable welding technique for manufacturing a large component or a small component for the biomedical application of Ti-6Al-4V is indispensable. Fusion welding of Ti-6Al-4V has created many difficulties, such as the formation of the brittle cast and coarse microstructure, severe deformation, and high residual stress with contamination in the joints due to the large thermal cycle. Moreover, it produced fumes and sputtering [25]. With this success among light metal alloys, research on friction stir welding of high strength steel and titanium has been extensively studied by various researchers to observe the feasibility of the joint, as well as observation of the microstructure and mechanical properties of the joints [26].

Few studies on the microstructure and mechanical properties of friction stir welding Ti-6Al-4V have been reported globally. H.J. Liu et al [27] found friction stir welding of 2 mm thick Ti–6Al–4V, at a constant tool rotation speed of 400 rpm with varying welding speeds from 25 to 100 mm min^{-1} and maximum joint strength of approximately 95% that was achieved for 400 rpm and 100 mm min^{-1} combination of a parameter. They showed that the stir zone had a lower hardness than that of the base material due to the annealing effect caused by friction heating as well as softening during recovery and dynamic recrystallization. Zhang et al [28] pointed out friction stir welding with the thickness of 3 mm - Ti–6Al–4V plates under different rotational speeds and obtained defect-free welds successfully at rotational speeds of 400 and 500 rpm. But they reported that the greatest hardness values were obtained at the stir zone, and they commented that the stir zone formed finer grains than HAZ due to the dynamic recrystallization. Kitamura et al [29] conducted FSW on a 2mm thick plate of Ti-6Al-4V and observed the effect of the thermal cycle by varying the rotational speed from 300rpm to 1000rpm, and a welding speed that varied from 25 to 400 mm.min^{-1}. They observed when the peak temperature was below the beta transfuse temperature, the microstructure consisted of fully equiaxed primary alpha grains and no lamellar alpha-beta. When the peak temperature surpassed the beta transfuse temperature, there was an increase of the stir zone strength concurrent with increasing welding speed due to the reduction of alpha-beta lamella size. The energy absorbed by the stir zone which contained the lamellar structure was higher than that by the equiaxed structure.

S. Mironov et al [30] studied the microstructural evolution of the FSW welded Ti alloy and established the reasons for the texture development in the development in the β grain structure using EBSD. A.L. Pilchak et al [31] investigated the microstructure and texture of two representative welds made just above and high above the β transfuse with scanning electron microscopy and electron backscatter diffraction (EBSD). L. Zhou et al [32] found the friction stir welding of two-phase (α-β) titanium alloy (Ti–6Al–4V) and reported that defect-free welds were produced with proper welding parameters using a welding pin tool. Moreover, they observed a bimodal microstructure in the stir zone due to dynamic recrystallization and phase transformation as the peak temperature was less than the β transfuse temperature. The dynamic recrystallization phenomenon was found to be the predominant factor for microstructural refinement in the stir zone. Titanium and its alloy have been widely used in biomedical applications for a long time. Researchers reported that Ti-6Al-4V alloy has several positive features leading to long term ill-effects such as peripheral neuropathy, osteomalacia, and Alzheimer’s disease due to the release of aluminum and vanadium ions from the alloy. Besides, vanadium which is present both in the elemental state and in oxides (V_2O_5) is also toxic [33]. Furthermore, Ti-6Al-4V alloy has a good wear resistance property and higher elastic modulus than human bone which leads to a ‘‘stress shielding effect’’ [29]. A report [34] showed that the nitrogen ion implantation and heat treatment procedures enhanced the corrosion fatigue of Ti-6Al-4V alloy.

In the present research, the success of the Ti-6Al-4V sheet by employing friction stir welding and the microstructure evolution, micro-hardness, tensile strength of the welding seam, corrosion properties in body fluid solutions were investigated to elucidate how the welding parameters influences the microstructure and mechanical properties of the weld joints.

II. Materials and Methods

The materials used in this study are Ti-6Al-4V alloys sheets with the base metal tensile strength of 1052 MPa and a sheet thickness of 2.5 mm. The chemical composition of Ti-6Al-4V alloys with the following nominal: Al 6.15, V 4.00, C 0.10, Fe 0.30, N 0.05, O 0.20, H 0.015 and balanced Ti (all in mass%) [27].

A milling machine was used for all the friction stir butt-welding, as modified from a DECKEL MAHO DMU 60T milling machine (German manufacture) at CNC workshop, Hung Yen University of Technology and Education, Hung Yen province, Vietnam. Welding experiments were performed using a special welding system equipped with a Tungsten carbide tool with a shoulder diameter of 12 mm and a pin height of 1.8 mm being used for the FSW butt joining. A depth of penetration of 2.2 mm has been used. The rotary speeds of 1500 and 2000 rpm were varied with a constant travel speed of 200 mm.min^{-1}. FS welding was done successfully without any defects as shown in Fig. 1 (For clear view zoomed FS welded portion).

The tensile specimens were prepared as per the ASTM E8 specification [35]. Tensile testing was done in universal tensile testing m.c^{-1} at a crosshead speed of 3 mm.min^{-1}. For corrosion measurement, Ringer’s solution was used as a solvent (6.5g NaCl, 0.42g KCl, 0.25g $CaCl_2$, and 0.2g of sodium bicarbonate were dissolved in one liter of distilled water) and followed the standard procedure to get the Tafel's plot [36].

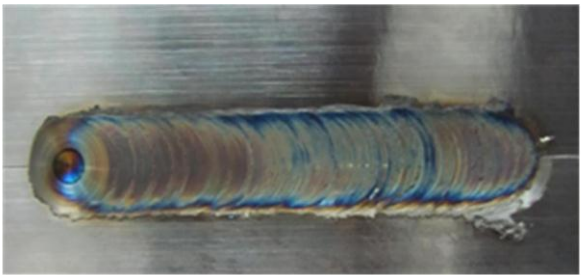

Figure 1. Friction stir welded Ti-6Al-4V alloy plate

III. Results and Discussions

The microstructure of the stir zone (SZ) for 1500 rpm, 200 $mm.min^{-1}$; 2000 rpm, 200 $mm.min^{-1}$ is presented in Fig. 2. Fig. 3 shows the thermal metal affect zone (TMAZ) for 1500 rpm, 200 $mm.min^{-1}$, and 2000 rpm, 200 $mm.min^{-1}$. The microstructure at the stir below on the top surface is the fully lamellar structure. The SZ experiences high temperatures and severe plastic deformation, which is the main reason for dynamic recrystallization in the SZ. The existence of a large amount of dislocation-free equiaxed grains confirms the dynamic recrystallization phenomenon in the SZ. Moreover, $\alpha \rightarrow \beta$ transformation was observed due to the high temperature arise (above β transfer temperature) during the FSW process. During the cooling process, $\beta \rightarrow \alpha + \beta$ transformation occurs, enhances the formation of the alternate lamellar structure of $\alpha + \beta$. It indicates that the bimodal microstructure in SZ is formed due to both dynamic recrystallization and phase transformation [25, 27]. However, the severe plastic deformation at such a high temperature also gives the stir zone leading to the nucleation of very fine strain-free grains. Due to speedy cooling, this β phase transforms back to the α-β phase region but since the cooling rate was very high, we got α-β lamellar structure inside the prior β grain boundaries. However, TMAZ grain size was ~~is~~ slightly coarser than that of dynamic recrystallized SZ easily evidenced by Figs. 1 - 2. With increasing the speeds at constant travel speed the heat input would increase. That is why a slower cooling rate gave a higher time to grain growth in 2000 rpm for both SZ and TMAZ.

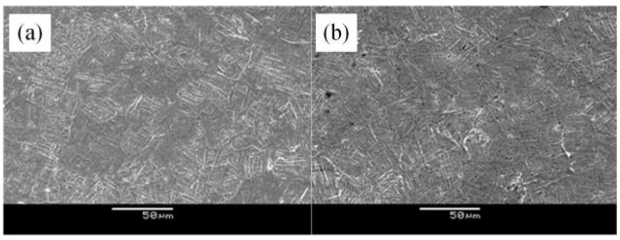

Figure 2. Microstructure of SZ for (a) 1500 rpm 200 $mm \times min^{-1}$; (b) 2000 rpm 200 $mm \times min^{-1}$

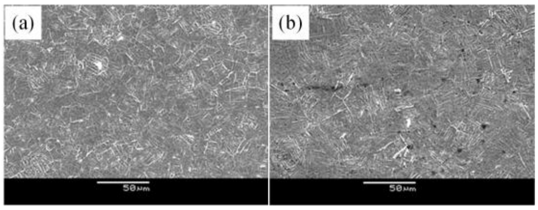

Figure 3. Microstructure of TMAZ for (a) 1500 rpm 200 $mm \times min^{-1}$ (b) 2000 rpm 200 $mm \times min^{-1}$

For better identification and more concrete confirmation, XRD was done to distinguish between α and β phases (see Fig. 4). It proved the presence of α and the β phase in the welded joint. EBSD was done to identify the recrystallization phenomenon at the stir zone. It clearly shows that the fine grains were illustrated at SZ in Fig. 5.

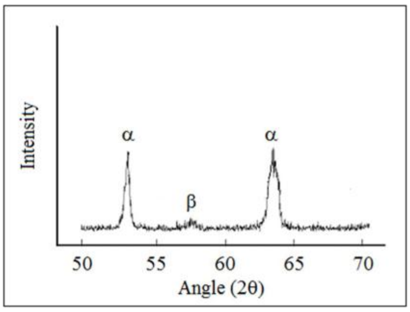

Figure 4. XRD analysis for 1500 rpm 200 $mm \times min^{-1}$

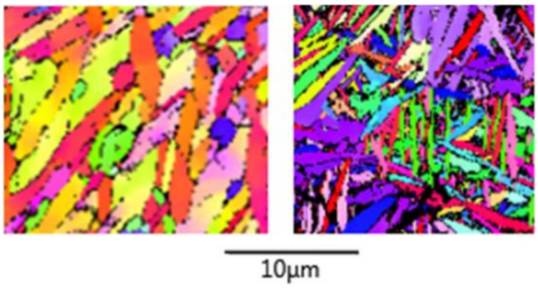

Figure 5. EBSD analysis at the stir zone

Vicker's micro-hardness was taken across the cross-section of the weld (see Fig. 6(a)). In general, the Stir Zone had the highest hardness and it decreased away from the SZ towards the base material having the lowest hardness. Based on phase transformation and deformation mechanisms, they would be taken place in different zones. It is seen that 1500×200^{-1} has higher hardness than that of 2000×200^{-1}. This is quite obvious due to the grain growth incase of 2000×200^{-1}. Micro-hardness is an indication of tensile strength. The higher the tool rotation speed was, the more mechanical mixing occurred. Thus the greater would be the heat generated due to higher friction levels and the higher the temperature reached, after crossing the β transfer, it was in fully β. Hence, the finer β grain boundaries ensure the smaller grains in 1500×200^{-1} giving the highest strength (see Fig. 6(b)) and toughness. Micro-structure, micro-hardness, and tensile strength justify each other well.

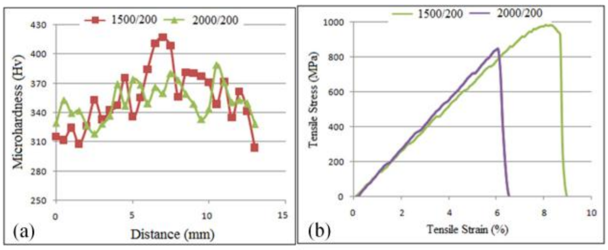

Figure 6. (a) Micro-hardness plot and (b) Stress-Strain Curve

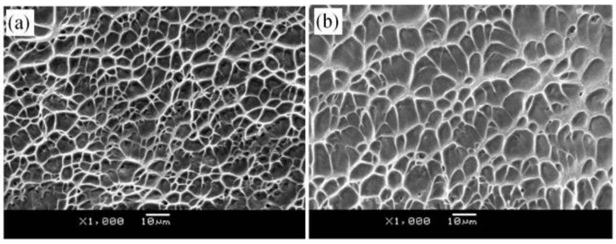

Figure 7. Fracture surface by SEM for (a) 1500 rpm 200 mm/min; (b) 2000 rpm 200mm/min

The fracture surface was studied by scanning electron microscopy for both combinations of the parameter (see Fig. 7). Mostly ductile type of failure was observed for both the cases. Interestingly, the difference in grain size, also prominent, occurred in the dimple size. 2000.200^{-1} with higher heat input shows a larger dimple size. This results in similar those of the reports [19, 20]. The ductility depends on the deformation capacity of grain size. The large grain size will common slip easilier than the smaller grain size does with the oriented boundaries. The friction stir welding created a disruption of grain structure in weld-joint, and during tensile testing, the results show that the joint non-uniform deformation and the lower elongation of the weld-joint were comparable in the base metal.

A potentiostatic polarization study was performed for both SZ and TMAZ individually for two different weld parameter combinations in the Ringer's solution (see Fig. 8). Corrosion resistance property for both SZ and TMAZ increased with the raise of the rotational speed. Moreover, in comparing to the corrosion rate between SZ and TMAZ for the given welded joint, TMAZ shows poor corrosion resistance compared to SZ. It indicated that corrosion property was influenced by the welding parameters such as rotation speed. Performances of the higher heat input and higher grain growth in 2000.200^{-1} seemed to be better than those of $150 .200^{-1}$. In the SZ of both cases, passivation is shown. In the case of titanium, passivity was provided by the oxide film (TiO2) and followed by a breakdown that must occur in this oxide film. In this case, pits did not appear due probably to the metallurgical heterogeneity that arises from the fact that the nucleation events did not disappear with a time of exposure. The ion Ti^4+ formed by the attack of the solution to titanium leaving the metal (Ti) for the solution and consequently, there was a migration of H+ ions to the metal surface forming the atomic hydrogen (H) and soon, then molecular hydrogen (H2) according to the reactions:

Ti^4+ (anodic reaction) -Ti - 4e - 2e -→ H_2- 2e - $2H^+$ → 2H (cathodic reaction).

In the presence of chloride ions in Ringer's solution, Cl^- can migrate in parallel across the passivating oxide. If chloride ions reached the interface of the metal/film, it would form metal chlorides like $TiCl_2$, $TiCl_3$, and $TiCl_4$ [37, 38]. Moreover, if sufficient metal chlorides were accumulated in place, the oxide could rupture explosively and reveal a bare metal surface, which can form a microscopically saturated chloride solution. The origin of the explosive film rupture is the fact that metal chlorides generally have greater molar volumes than the corresponding oxides, and a formation of the chloride salt at the metal or passive film interface must engender stresses which would tend to cause blistering in the passivating film. This explains why after passivation occurs, it returns to the active state. However, due to the characteristics of the subsequently and spontaneously formed thin oxide film which is mainly composed of TiO_2, this is the main reason behind the high corrosion resistance of this alloy. It also pointed out that the oxide film thickness on Ti implants, after some years in the human body, may reach larger values than those in the preliminary stage [38].

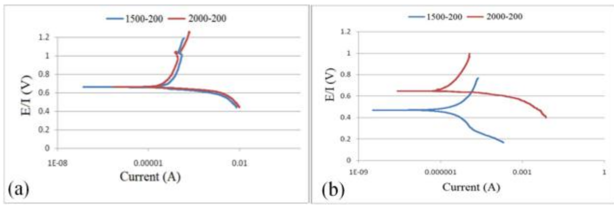

Figure 8. Tafel's plot for (a) Stir Zone and (b) Thermo-mechanically affected zone (TMAZ)

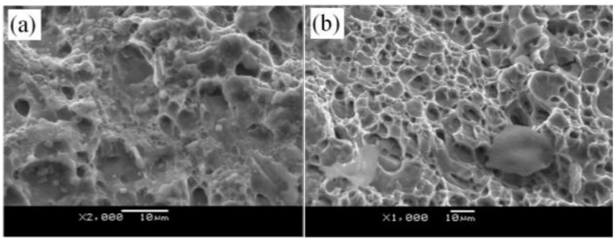

Figure 9. Corroded surface study by SEM for (a) 1500 rpm 200 $mm \times min^{-1}$; (b) 2000 rpm 200 $mm \times min^{-1}$

TABLE I. CORROSION RATE FOR SZ AND TMAZ OF 1500×200^{-1} AND 2000×200^{-1}

Samples	SZ Corrosion rate ($mm \times year^{-1}$)	TMAZ Corrosion rate ($mm \times year^{-1}$)
1500×200^{-1}	0.03	0.09
2000×200^{-1}	0.002	0.01

To evaluate the corrosion properties of the stir zone, the potentiodynamic test was performed with 5% HCl at the normal temperature. The nitrogen atmosphere condition was maintained during the experiments. These experiments were repeated four times and the results showed that the 2000×200^{-1} specimens have a higher corrosion rate than the 1500×200^{-1} specimens. The corrosion resistance studied by employing SEM (see Fig. 9) with 1500×200^{-1} was higher than that of 1500×200^{-1}, as presented in Table II. Although, for both the cases, the chlorides ion and TiO_2 layers were observed in the corroded surfaces.

IV. CONCLUSIONS

In the present research, Ti-6Al-4V titanium alloys were used as research subjects. The main issues have been discussed in detail such as cross-sections, microstructure characteristics, tensile properties of the stir zone. The following main conclusions could be drawn from the present study:

- 96% of joint strength achieved for friction stir welded joint was better than the base metal.
- After cooling, the structure grain of the formation of the weld with a small size and randomly oriented, this results in increased tensile strength and hardness at the stirring area of the weld joint.
- The microstructure of the Stir Zone for both cases showed a completely homogenous α-β lamellar structure inside the prior β grain boundaries as the peak temperature was higher than the β transferring temperature.
- The combined temperature effect is the primary responsible β phase that transforms back to the alpha-β phase, but since the cooling rate was very high, within the previous β grain boundaries, we got the alpha-β lamellar structure, resulting in the most effective condition for the mechanical efficiency of the joints.
- Corrosion results of 2000 rpm and 200 mm×min^{-1} travel speed combination is better than those of 1500 rpm and 200 mm×min^{-1} combination.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

The first author carried out the experiment, analyzed data, and wrote the manuscript. The second author and third author processed the data and edited the manuscript.

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