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ABSTRACT: This study presents a diffusion bonding process of commercially pure aluminum to Ti-6Al-4V alloy. Prepared samples were exposed to temperature of 560, 600 and 640 °C for the bonding time of 30, 45 and 60 min at the atmosphere of argon gas and non-argon. Diffusion bonding is a dissimilar metal welding process which can be applied to the materials without causing any physical deformations. The processed samples were also metallographically prepared, optically examined followed by Vickers microhardness test in order to determine joint strength. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) were used in this work to investigate the compositional changes in order to observe the influence of atmosphere shielding in the transition zone. The result of tests and analyses were tried to be compared with the effect of argon shielding. The significant influences have been observed in the argon shielding during diffusion bonding process.

KEYWORDS: Argon gas shielding; Diffusion bonding; Dissimilar metal bonding; Solid-state bonding; Ti-6Al-4V alloy

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RESUMEN: Influencia del argón como gas protector en la difusión durante el proceso de unión de la aleación Ti6Al4V con el aluminio. Este estudio presenta los procesos de difusión durante la unión de aluminio puro con la aleación Ti6Al4V. Se expusieron probetas a las temperaturas de 560, 600 y 640 °C durante un tiempo de unión de 30, 45 y 60 min en una atmósfera en presencia y ausencia de gas argón. La unión por difusión es un proceso de soldadura entre metales distintos que puede ser aplicado a los materiales sin causar deformaciones físicas. Las probetas procesadas fueron preparadas también metalográficamente, examinadas por miscroscopía óptica, seguido de ensayos de microdureza Vickers para determinar el límite elástico. Se utilizó microscopía electrónica de barrido (SEM) y espectroscopía de energias dispersivas (EDS) para determinar los cambios en la composición y estudiar la influencia del argón como gas protector en la zona de transición. La influencia más importante se ha observado durante el proceso de difusión en estado sólido.

PALABRAS CLAVE: Aleación Ti6Al4V; Argón gas protector; Difusión y unión; Unión de metales distintos; Unión en estado sólido

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1. INTRODUCTION

Titanium is a strong metal with low density that is quite ductile and lustrous. The relatively high melting point (1660 °C) makes it useful as refractory metal. Aluminum is remarkable material for the metal's lower density and for its ability to resist corrosion due to the phenomenon of passivation (Leyens and Peters, 2003). Structural components made from titanium and aluminum plays a significant role in the aerospace and defense industries. These materials are also important in other applications such as transportation, structural materials, aircraft structural parts, automotive, medical prostheses, orthopedic implants, dental implants, sporting goods, jewelry, mobile phones, etc. Joining of these materials would reduce the weight and cost of final products with the high strength. Joining commercially pure aluminum to Ti-6Al-4V alloy is also desirable particularly for applications in the aerospace industry (Kahraman et al., 2007).

Titanium has many alloys for different purposes, however Ti-6Al-4V (Grade 5) alloy is more applicable for heat treatments and it is easy to be found in market (RMI, 2000; James, 2004), and it is an excellent combination of strength, corrosion resistance, weldability and fabricability. Large difference in the physical properties of commercially pure aluminum and Ti-6Al-4V alloy which includes the melting point, heat conductivity, and coefficient of thermal expansion prevents the use of conventional fusion welding to join the dissimilar metals (Kicukov and Gursel, 2015; Akca and Gursel, 2016). However, the joining of dissimilar metals is possible with diffusion bonding which is a solid-state bonding method (Messler, 1999). According to a literature review, many other metals are joined by diffusion bonding (Hoppin and Berry, 1970; Avery, 1991), however, joining of commercially pure aluminum and Ti-6Al-4V alloy does not have its place in the reported literatures, and influence of argon gas shielding is not studied previously. In diffusion bonding, the bond strength is achieved by pressure, temperature, time of contact and cleanness of the surfaces, and these combinations are called as diffusion parameters (Rusnaldy, 2001). During welding processes, titanium alloy picks up oxygen and nitrogen from the atmosphere easily (Sun et al., 2003; Kahraman et al., 2007). For this reason, the process is shielded by argon gas.

During any bonding process, argon and other atmospheric gas can react with the molten metal, causing defects that weaken the weld. The primary function of a shielding gas is to protect the molten weld metal form atmospheric contamination and the resulting imperfections. The primary gases used for welding and cutting are argon, helium, hydrogen, nitrogen, oxygen and carbon dioxide. The composition of the gas can and should be tailored to meet the process, material, and application requirements. Shielding gases are used in either a pure form or in blends of varying components.

The thermal conductivity of a gas is its ability to conduct heat. It influences the radial heat loss from the center to the periphery of the arc column. Argon, which has a low thermal conductivity, produces an arc which has two zones: a narrow hot core and a considerably cooler outer zone (Nils, 1998).

In this study, the diffusion parameters were determined at temperatures of 560, 600 and 640 °C, and the bonding time of 30, 45 and 60 min at the atmosphere of argon gas and non-argon. The effect of diffusion parameters on joint microstructural, the element compositions in the transition zone, and their corresponding developments by argon gas shielding were investigated.

2. MATERIALS AND METHODS

Ti-6Al-4V and commercially pure aluminum, with chemical compositions shown in Table 1 (Krishnaiah *et al.*, 2008), were bonded by diffusion bonding method. Ti-6Al-4V and aluminum samples were prepared for different tests in the dimensions given in Fig. 1. All the sample's surfaces were ground with SiC paper grade 120-280. The cleaning process of the surfaces might be carried out in two ways by acetone or carbon tetra chlorine. Even though cleaning with carbon tetra chlorine improves 14% joining strength than acetone cleaning process (Kazakov, 1985), surface cleaning with linen gives successful result in diffusion bonding, as well.

Properly controlled and monitored atmospheric furnace whose internal volume is 15 liters was used for the bonding process. A pressure of 3 MPa was applied to the bonding surfaces to improve the interfacial diffusion. In order to eliminate the oxidation problem, the bonding furnace was completely filled with Ar gas at a flow rate of 6 L min⁻¹. The bonding furnace was programmed to be heated at a

TABLE 1. Chemical composition of Ti-6Al-4V alloy and commercially pure aluminum (wt.%)

	Ti	Al	V	Ν	0	Н	Fe	Y	Si	Mn	Mg	Cu
Titanium	Balance	6.75	4.5	0.5	0.2	0.0125	0.3	0.005	-	-	-	-
Aluminum	-	Balance	-	-	-	-	0.437	-	0.124	0.129	0.004	0.026



FIGURE 1. Dimensions of test samples for microstructure and microhardness (All dimensions are in mm).

 TABLE 2.
 Diffusion bonding parameters

Sample N°	Bonding Temperature (°C)	Bonding Time (min)	Bonding Atmosphere
01	560	30	Non-argon
02	560	45	"
03	560	60	~~
04	600	30	cc
05	600	45	cc
06	600	60	66
07	640	30	66
08	640	45	66
09	640	60	66
A1	560	30	Argon
A2	560	45	66
A3	560	60	66
A4	600	30	~~
A5	600	45	~~
A6	600	60	~~
A7	640	30	~~
A8	640	45	~~
A9	640	60	66

rate of 30 °C min⁻¹ until the test temperatures (560, 600, and 640 °C) were achieved for holding times of 30, 45, and 60 min. At the end of the process the samples were allowed to cool in the bonding furnace. Also, other series of samples were bonded at the atmosphere of non-argon. The temperature of the process was monitored using a Pt/Rh thermocouple located close to the bonded samples. The bonding processes were completed and 18 samples were prepared with different bonding parameters shown in Table 2.

The bonded samples were firstly sectioned perpendicular to the bonding surface. In order to be able to hold the samples for grinding and polishing, afterwards the sectioned samples were mounted in bakelite as shown in Fig. 2, and the mounting operations were carried out for 9 min heating and 3 min cooling at a temperature of



FIGURE 2. Sample mounted in bakelite.

180 °C, and a force of 40 kN applied. The grinding processes were done with SiC paper grade 180, 500, 800, 1200, 2000, and 2500, respectively, the ground samples were subjected to polishing operation with 1 μ m alumina suspension. Both grinding and polishing processes were done with Struers LaboPol-5 at angular velocity of 500 rev min⁻¹. Samples were etched with a chemical solution; 1% HF - 1.5% HCL - 2.5% HNO3 - 95% H2O (Aydın *et al.*, 2012). At the end, Metallographic examinations were done to the bonded samples.

Microstructure was evaluated by optic microscopy (Nikon LV100DA-U), and scanning electron microscopy (SEM, Philips XL30S FEG). Changes in joint compositions across the joints were examined using energy dispersive spectroscopy (EDS). Mechanical properties were evaluated by microhardness tests. The microhardness test was carried out in an Instron Wolpert Testor 2100. One of the microhardness measurements was taken in the interface of the bonded samples and two sides of bonded joints by the interval of 100 μ m, and Micro HV method at 50 gram force (gf) was used.

3. RESULTS AND DISCUSSION

The results of diffusion bonding process showed the importance of the effect diffusion parameters. Bonding at 560 °C for the bonding time of 30 min at the atmosphere of argon gas and non-argon could not be achieved. The failure of bonding of this parameter can be attributed to the insufficient bonding time. When bonding time was increased to 60 min, bonding could be achieved. However, when bonding time was increased to 45 min without any increment in bonding temperature, bonding at the atmosphere of argon gas was achieved, but not at non-argon. In order for atoms to diffuse in the diffusion bonding method, adequate temperature and time are required. In fact, combinations of temperature and time have to be decided carefully. Longer bonding time is not only disadvantageous for bonding economy, but also it can create some undesirable effects which damage the mechanical properties of bonding (Itharaju, 2004). Luo and Acoff (2000) have also applied 4 h of bonding time and as a result, diffusion interfaces were extremely discontinuous.

3.1. Optic microscopy studies

The purpose of microstructural studies with optical microscope is to investigate phase transformations of the bonded samples. The optical micrographs of some samples were published in Figs. 3, 4, 5, and 6.

When the differences between argon gas shielding and non-argon were compared, the samples bonded at the atmosphere of argon gas are appeared to be bonded regularly, but the samples bonded at the atmosphere of non-argon were bonded irregularly. Small voids were also occurred in Fig. 3b and Fig. 4b due to non-argon atmosphere. On the other hand, diffusion interfaces of Fig. 3a and Fig. 4a are quite regular and continuous.

When the atmosphere shielding is compared in Fig. 4, the oxidation and burning appeared in the diffusion interface of both aluminum and titanium sides in Fig. 4b. Nevertheless the diffusion interface of Fig. 4a indicates the regular and clear transition zone. Figure 5b shows the carburization as a result of non-argon atmosphere. The comparison of argon gas shielding has been made with small scales of 100 and 400 μ m in Figs. 3, 4, and 5, because the most differences were observed in those scales. Nevertheless, Fig. 6 was examined in large scale of 40 μ m.

When it is compared in large scale, it was observed that the aluminum structures more clear in the samples bonded at the atmosphere of argon gas in Fig. 6a, besides aluminum grains were burned due to non-argon atmosphere in Fig. 6b. The comparison of atmosphere effect in transition zone has been shown. It is observed that the samples bonded at the atmosphere of argon gas pose more regular and distinctive transition zone. The carburization, rifts and burning have been occurred in the samples bonded at non-argon. Another observation is that diffusion interface or bonding line became discontinuous and irregular.

3.2. SEM and EDS analyses

The SEM and EDS analyses were performed on the transition zone of all the bonded samples to investigate the influence of bonding atmosphere



FIGURE 3. Optical micrograph of the sample bonded at 560 °C for 60 min at the atmosphere of: (a) argon gas and (b) non-argon.



FIGURE 4. Optical micrograph of the sample bonded at 600 °C for 30 min at the atmosphere of: (a) argon gas and (b) non-argon.

on microstructure and elemental compositional changes during the process. The differences of the microstructure of the transition zone between argon gas shielding and non-argon were compared, the samples bonded at the atmosphere of argon gas were bonded regularly, but the samples bonded at the atmosphere of non-argon were bonded irregularly (see Fig. 7). A Figures 7 and 8 indicates the bonding differences between argon gas shielding and non-argon. While the sample in Fig. 7a was bonded quite regular and acceptable, but there are tiny gaps in the transition zone of Fig. 7b in a nano-scale. Small rifts were also occurred in Fig. 8b due to non-argon atmosphere. On the other hand, diffusion interface of Fig. 8a is quite regular, continuous, and filled with interface containing Ti/Al/V. Figure 8b and Fig. 9b show the carburization as a result of non-argon atmosphere. Nevertheless, Fig. 8a and Fig. 9a represent the transition zones of the sample bonded at 600 °C for 30 and 45 min, respectively. Although Fig. 9 (a and b) have the same scale and magnification, there are many differences such as gaps, rifts, dross, and slag. Dross is a mass of solid impurities floating on a molten metal or dispersed in the metal. It forms on the surface of low-melting-point metal. Slag is

also usually a mixture of metal oxides and silicon dioxide (Tsakiridis, 2012). There are also many differences between Fig. 10 (a and b) which were bonded at the atmosphere of argon gas and nonargon, respectively. In Fig. 10b, a rift occurred clearly as line, it indicates the bonding error.

Three selected areas from the sample bonded at 600 °C for 60 min at the atmosphere of argon gas and non-argon were marked, and elemental composition in the transition zone were obtained by EDS analysis in Fig. 11. Figure 11a shows the elemental composition of the sample bonded at the atmosphere of argon gas. Area 1 consist of 100 wt.% of aluminum. Spot 1 (diffusion interface) is rich in titanium (67.79 wt.%) and has 28.69 wt.% of aluminum, and 3.52 wt.% of vanadium. Area 4 shows the elemental composition of Ti-6Al-4V alloy. Fig. 11b shows the elemental composition of the sample bonded at the atmosphere of nonargon, and it is observed that aluminum composition of spot 1 (diffusion interface) decreased to 8.55 wt.%. Thus, the transition was sharply occurred, and directly going from aluminum to Ti-6Al-4V. Line elemental analysis of the sample bonded at 600 °C for 60 min at the atmosphere of argon gas and non-argon was also obtained for



FIGURE 5. Optical micrograph of the sample bonded at 600 °C for 45 min at the atmosphere of: (a) argon gas and (b) non-argon.

by EDS examination in Fig. 12. It is also possible to see the elemental differences between Fig. 12 (a and b). The transition was sharply occurred, and directly going from aluminum to Ti-6Al-4V. This observation can be attributed to non-argon atmosphere. Figure 13 shows the selected areas and EDS elemental composition investigated into the sample bonded at 640 °C for 45 min. When the samples bonded at 640 °C for 45 min and 600 °C for 60 min compared with EDS analyses results, it is observed that when the temperature increased, weight of aluminum element increased, but weight of titanium decreased in transition zone. In Fig. 13b, the oxidation occurred in the diffusion interface due to non-argon atmosphere. Figure 14 (a and b) show the EDS line element profiles for the samples bonded at 640 °C for 45 min at the atmosphere argon gas and non-argon.

3.3. Microhardness measurements

Microhardness measurement method and marks have been shown in Fig. 15. Figure 16 has been prepared according to the hardness test results obtained from the diffusion bonded joints. The results were grouped with respect to the bonded at the atmosphere of argon gas and non-argon for all parameters. It can be seen that the titanium sides have hardness value of 450 HV, while aluminum sides have hardness value of about 33 HV. Microhardness profiles of the bonded samples have been examined; basically, it is observed that the results are quite similar. The hardness values of aluminum sides are always lower than titanium sides as expected, and the hardness values in the transition zone are always higher than aluminum sides but lower than titanium sides.

In Fig. 16, the profiles show microhardness measurements of the samples bonded at the atmosphere of argon gas and non-argon. Microhardness profiles of diffusion couples were examined, the observations are; low temperature can be the cause not to have higher hardness values in titanium sides. In literature, both the microhardness results and bonding temperature are higher in titanium side, simultaneously (Barıs, 2007). The crystal structure of titanium is bodycentered-cubic, which is termed the β -phase, and a transformation occurs when the temperature achieved is higher than 885 °C, therefore materials can have higher hardness value with this transformation (RMI, 2000). The bonding atmosphere is



FIGURE 6. Optical micrograph of the sample bonded at 640 °C for 30 min at the atmosphere of: (a) argon gas and (b) non-argon.



FIGURE 7. SEM micrograph of the sample bonded at 560 °C for 60 min at the atmosphere of: (a) argon gas and (b) non-argon.

compared with the microhardness results, and it is observed that microhardness values of aluminum are higher on the samples bonded at the atmosphere of non-argon, however the microhardness values of titanium alloy are higher with small differences on the samples bonded at the atmosphere of argon gas.

4. CONCLUSIONS

The solid-state diffusion bonding was carried out between Ti-6Al-4V alloy and commercially pure aluminum using different diffusion parameter. The influence of argon gas shielding was investigated, and the results can be summarized as:

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FIGURE 8. SEM micrograph of the sample bonded at 600 °C for 30 min at the atmosphere of (a) argon gas and (b) non-argon.



FIGURE 9. SEM micrograph of the sample bonded at 600 °C for 45 min at the atmosphere of: (a) argon gas and (b) non-argon.



FIGURE 10. SEM micrograph of the sample bonded at 640 °C for 60 min at the atmosphere of: (a) argon gas and (b) non-argon.



FIGURE 11. EDS elemental composition of selected areas of the samples bonded at 600 °C for 60 min at the atmosphere of: (a) argon gas and (b) non-argon.



FIGURE 12. EDS line element analysis of the samples bonded at 600 °C for 60 min at the atmosphere of: (a) argon gas and (b) non-argon.

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FIGURE 13. EDS elemental composition of selected areas of the samples bonded at 640 °C for 45 min at the atmosphere of: (a) argon gas and (b) non-argon.



FIGURE 14. EDS line element analysis of the samples bonded at 640 °C for 45 min at the atmosphere of: (a) argon gas and (b) non-argon.



FIGURE 15. Microhardness measurement marks with; a) SEM and b) optical micrographs.

- Bonding at 560 °C for 30 min at the atmosphere of argon gas and non-argon could not be achieved. The failure of bonding of this parameter can be attributed to the insufficient bonding time. When bonding time was increased to 60 min, bonding could be achieved. However, when bonding time was increased to 45 min without any increment in bonding temperature, bonding at the atmosphere of argon gas was achieved, but not at non-argon. It shows the importance of argon gas shielding.

- According to the results, argon gas shielding during the bonding process was proven to be used in industry.

- A comparison between the samples bonded at the atmosphere of argon gas and non-argon can be made with EDS and SEM analyses results, and optic micrographs. Interfacial voids, carburization, corrosion, oxidation, slag and dross occurred due to non-argon atmosphere.

- The samples bonded at the atmosphere of argon gas have more powerful diffusion strength, because it was observed that the transition zones are more distinctive than the samples bonded at the atmosphere of non-argon.

- The bonding atmosphere is also compared with the microhardness results, and it was observed that microhardness values of aluminum are higher on the samples bonded at the atmosphere of non-argon. However, the microhardness values of titanium alloy are higher with small differences on the samples bonded at the atmosphere of argon gas.

- It was observed that the aluminum structures are clearer in the samples bonded at the atmosphere of argon gas, besides impurities in aluminum are burned in the samples bonded at the atmosphere of non-argon.

- A strong and homogeneous diffusion interface did not achieve on the samples bonded at the atmosphere non-argon, although it has been achieved on the samples bonded at the atmosphere argon gas. The transition was sharply occurred, and directly going from aluminum to Ti-6Al-4V according to EDS analysis results.

- Elemental composition in the transition zone must be distributed equally to have atomic bonding, and it has been proven that it is just possible with the process of argon shielding.

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FIGURE 16. Hardness profiles of all bonded samples.

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