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Diffusion Welding of Al-α-Si_3N_4 Composite Materials

Araştırma Makalesi / Research Article

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ABSTRACT

In this study, Al-α-Si_3N_4 composite, produced by powder metallurgy method, were joined at 2.5 MPa pressure with various welding temperatures (620, 630, and 640 °C) and durations (1,1.5, and 2 h.) by diffusion welding method. Optical microscopy examination was carried out from welded interfaces and shear tests were also conducted to the sample interfaces to find out the effect of welding parameters and amount of α-Si_3N_4 reinforcement on the weldability properties of composite materials. The test results show that increase in the welding temperature and duration resulted in increase shear resistance of the welded zone. Results indicated that optimum parameters (welding temperatures, durations and amount of α-Si_3N_4 are 640 °C, 2 h., and 15wt % respectively) MMCs produced in this study could be joined by diffusion welding technique successfully with the 97.5 % strength of base material.

Keywords: Composite, Al-Si_3N_4, diffusion welding, shear strength.

Al-α-Si_3N_4 Kompozit Malzemelerin Difüzyon Kaynağı

ÖZ

Bu çalışmada toz metalurjisi metodu ile ile üretilen Al-Si_3N_4 kompozit malzeme değişen sürelerde, değişen sıcaklıklarda ve 2,5 MPa basınç altında difüzyon kaynağı ile birleştirilmiştir. Kompozit malzemeninin kaynaklanabilirliği üzerine α-Si_3N_4 miktarı ve kaynak parametrelerinin etkilerini belirlemek için numune kaynak arayüzeyleri üzerinden optic mikroskop incelemeleri ve kesme testleri yapılmıştır. Test sonuçları kaynak süresi ve kaynak sıcaklığı artarken kaynak arayüzeylerinin kesilme mukavetlerinin de artttığını göstermektedir. Numunelerin kesilme testi sonuçlarına optimum kaynak parametrelerinde üretilen (% 15 α-Si_3N_4 içeren numunede 2 saat süreli 640 °C sıcaklıkta üretilen numunede) metal matrisli kompozit malzemelerin difüzyon kaynak tekniği ile matris yapının % 97, 5' i kadar mukavemette birleştirilebilir olduğunu göstermiştir.

Anahtar kelimeler: Kompozit, Al-Si_3N_4, difüzyon kaynağı, kesilme mukavemet.

1. INTRODUCTION

Due to its low density, high corrosion resistance, high ductility and easy to formability properties, aluminum and its alloys are widely consumed in the aerospace and aerospace sectors, particularly in the automotive sector. For this reason, the studies that scientists are doing on improving the properties of these materials continue to increase. [1-2]. In order to improve the mechanical properties of this materials, different strengthening methods such as mechanical alloying, in-situ, extrusion and reinforcing with small and hard refractory particles have been using. [3-5]. By applying one of those processes, we can produce composite materials having better mechanical properties than those of obtained at plain alloys especially at high temperatures. [6-8]. Metal matrix composites are produced by liquid or solid state method. In the liquid state, ceramic particles are added to liquid metal by stirring before casting, but the resulting distribution of reinforcing elements in matrix structure is generally inhomogeneous. In the solid-state method, known as the powder metallurgy technique, reinforcing ceramic particles such as oxide, carbide and nitride are added into the metal powders, and then respectively compaction and sintering processes were done to produce block parts from mixed powder. One of the most important problems encountered in this method, diffusion bonding of metal and ceramic powders to each other may be inadequate [9-10]. When adjoining of these kinds of composite materials to each other is required, weldability properties gains particular importance. In generally, these materials can be joined by either fusion welding (arc welding, shielding gas welding, laser welding and electron beam welding etc.) or solid-state welding (soldering, explosive welding, friction welding and diffusion welding). In fusion welding, some problems may be seen at the vicinity of welding where particularly at the heat-affected zone [11-13]. During the welding process of aluminum material an oxide film occurs, which covers the surface of Al and makes the welding process difficult [14] In this case, we can use a protective gas reducing negative effect of oxide film on the welding zone in this way it can be achieved demanded welding quality. Diffusion welding is an effective and potential joining process especially for non-ferrous metals or composite materials. Using this method, we can reduce the problems such as cracking, distortion and

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segregation usually occurs during the liquid-phase welding proces. [15]. In this method, a temperature between 50 % - 80 % of the melting point of the materials is chosen as welding temperature. Therefore, some problems caused by high welding temperature such as phase transformation or microstructural change can not occur during welding process [16-20]. According to some research results, it is indicated that the expected strength of a joint can be obtained by a correct selection of the welding temperature, duration, pressure and protective atmosphere [1]. In this study, initially Al- α-Si_3N_4 composite material were produced by using powder metallurgy method and then the samples were joined by diffusion welding method under the various welding parameters. Finally, mechanical and microstructural properties of weld interface were examined. Hence, to optimize optimum welding parameters for this study, shear test and microstructure examinations were performed on the welded sample.

2. EXPERIMENTAL PROCEDURE

2.1 Production of Composite Material

In present study, to produce composite material atomize aluminum powder (mean particle size of 104.8 μm) as matrix material and α-Si_3N_4 powders as a reinforcing material were used **(**Table 1)**.** Al and α-Si_3N_4 powders were mixed together according to the proportions of Al-α-Si_3N_4 (5; 10; 15 % wt.) and then mechanical mixing process were carried out for 2 hours with steel balls in a mechanical mixing device. (Figure 1).

Table 1. Properties of α-Si_3N_4 powder

Grade	SN-E-10
Morphology	Equiaxed
Particle size (μm)	0.1 ~ 0.3
Specific surface area (m^2/g)	10 ~ 14
Purity	N > 38 % Fe < 100 ppm O < 2.0 % Ca < 50 ppm C < 2.0 % Al < 50 ppm
Degree of crystallinity (%)	~100
Phase composition	α-Phase 95 % β-Phase 5 %

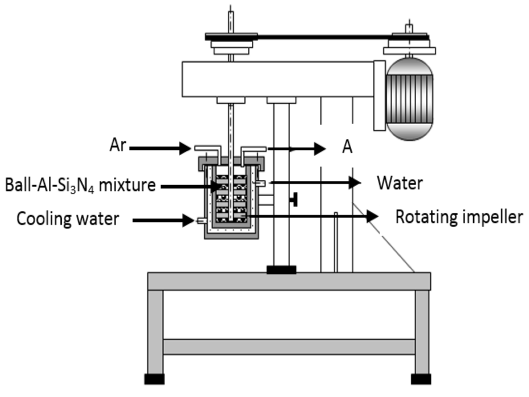

Figure.1. Mechanical mixing device

The parameters chosen for mechanical mixing process are as shown in Table 2. The first propose of the use of argon gas was to prevent possible oxidation of the new surfaces of Al particles created by fracturing Al particles during the mechanical mixing process. In addition, in order to minimize the welding tendency of aluminum particles to each other during mechanical milling process, stearic acid (1.5 wt %) as process control agent (PCA) was added to the mixed powder Then, to determine the effects of mechanical milling process on the morphology (as shape and particle size) of mixed powder, characterization studies were performed by using optic microscope and Malvern Master Sizer E version 1.2b laser scattering machine.

Table 2. Parameters of mechanical mixing process

Mixture Powders	Al+5 % Si_3N_4
	Al+10 % Si_3N_4
	Al+15 % Si_3N_4
PCA (%)	1.5
Charge ratio (balls: powder)	10:1
Mixing time (h)	2
Mix. Atm.	Ar
Rotor speed (rev. min^{-1})	450

After the characterization of particle size and morphology of mixed powder, to produce powder metal block samples with dimensions Ø10 x 15 mm in size, compaction processes were performed at 500 MPa pressure in a single action die. Block specimens were then put into graphite boats and then the boats were placed in an atmosphere controlled tube furnace. The furnace was heated to test temperature for predetermed duration under flowing argon atmosphere. Sintering was performed at sintering temperature for two hours. After the sintering process composite specimens were characterized by measuring the densities, hardness, shear strength, and doing the optic microscope examinations.

2.2. Diffusion Welding

Before the welding process surfaces of sintered samples, with the size of Ø10x15 mm, were polished by 0.4 Ra surface roughness (Figure 2) and then cleaned by alcohol. Surface roughness of the samples were measured by using Taylor Hobson instrument. In order to obtain suitable weld couples, surfaces of the samples were fully connected to each other as shown in Fig 3.

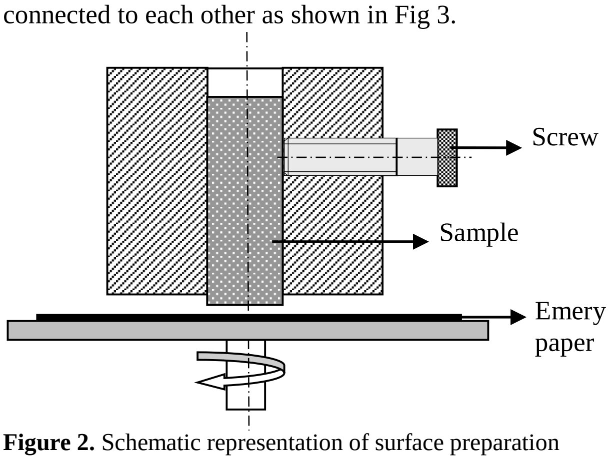

Figure 2. Schematic representation of surface preparation apparatus.

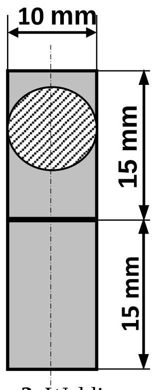

Figure 3. Welding couple

Samples were then placed in the center of the diffusion welding device as showed Fig. 4. In order to prevent the oxidation of welding couple Ar gas was introduced into the test chamber before welding. Then 2.5 MPa pressure was applied to the samples before welding. Ambient temperature was risen up to test temperature with a heating rate of 5 °C.min-1 and samples were isothermally soaked at desired durations then cooled down to room temperature in Ar atmosphere. Welding parameters for this experiment is shown in Table 3.

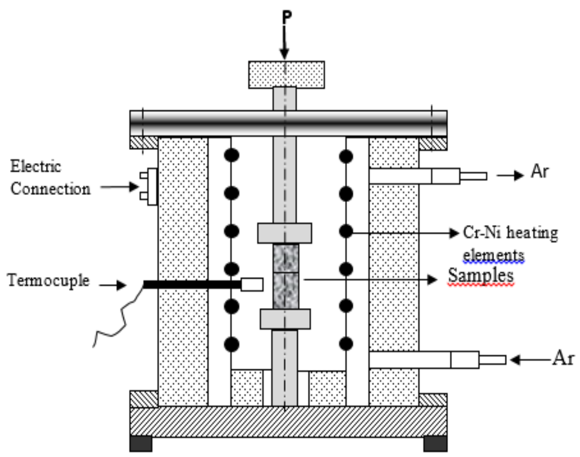

Figure 4. Diffusion welding device

Table 3. Welding parameters

Welding temperature (°C)	620 – 630 - 640
Applied pressure (MPa)	2.5
Protective gas	Ar
Surface roughness Ra (µm)	0.4
Welding time (h.)	1-1.5-2

2.3. Shear Test

One of the samples had different amount of Si_3N_4 and welded at different welding parameters were prepared metallograpically for microstructure determination. Remains were subjected to shear test with the speed of 1.5 mm/sec load applying by using shear test apparatus (Figure 5).

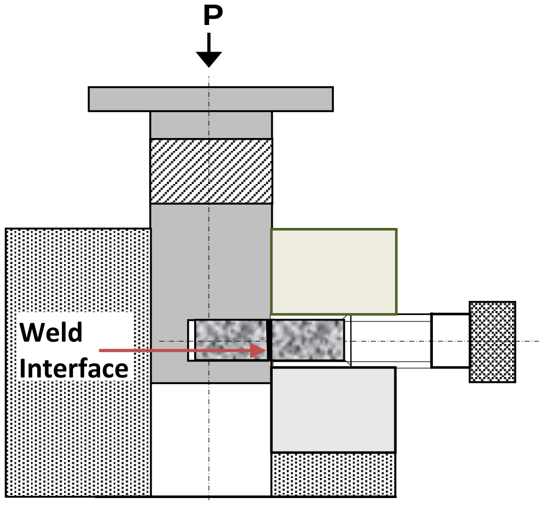

Figure 5. Shear test apparatus

3. RESULT AND DISCUSSION

After the compaction, green densities of the composite materials are given in figure 6. The density results show that maximum density was obtained in the composite sample containing 10 wt. % α-Si_3N_4. The main reason for this may be related to the particle size of the used mixture powder. After the mixing process, because of the increase in amount of Si_3N_4in the aluminum powders, mean particle size of mixed powders was calculated as 97.87, 42.23, and 61.02 µm respectively. It is seen that the green density of powder metal composite materials increase while the mean particle size of mixed powder decreases. After the sintering, a considerable increment was seen in final density (Figure 6) for all the composite samples.

The test results show that composite material containing 15 % α-Si_3N_4 have maximum density value. The hardness values of the composite materials had different amount of α-Si_3N_4 reinforcing materials were given in Figure. 7. Similar to density maximum hardness value was taken from composite materials containing 15 % α-Si_3N_4. Test results show that increasing of the amount of Si_3N_4 had a considerable positive effect on the hardness of composite materials and there was a good concord between the density and hardness values of the all of the composite materials.

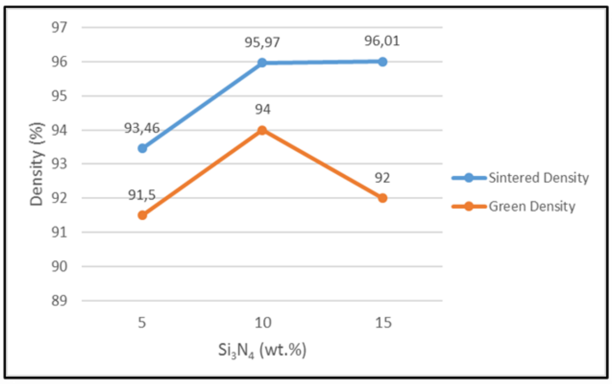

Figure 6. Chang of green and full density of composite materials with increasing of α-Si_3N_4 content.

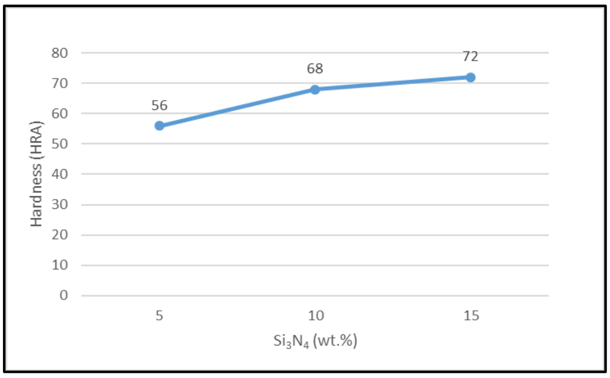

Figure 7. Hardness values of composite materials containing different amount of α-Si_3N_4

The shear test results given in Figure 8 show that shear resistance values increase while amount ofα-Si_3N_4 in composite material increase. As a result of the tests made, the best results in terms of density, hardness and cut resistance were obtained from the composite sample containing 15% α-Si_3N_4.

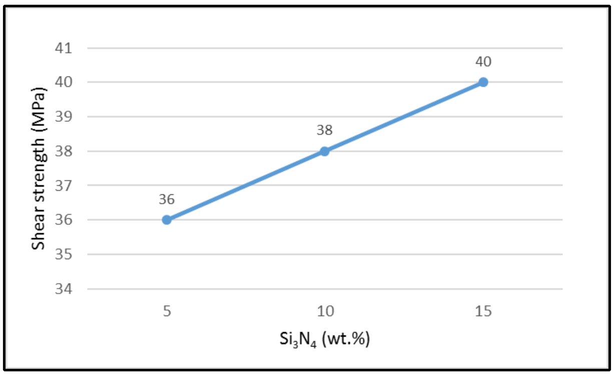

Figure 8. Shear strength values of composite materials containing of different amount of α-Si_3N_4

The main variables of this study are the variation of α-Si_3N_4 ratio in terms of material, welding temperature and duration in welding process while the samples obtained from MMCs are welded to each other by diffusion welding. The highest shear strength values of all samples after welding at different temperatures and durations under constant load were obtained from composite materials containing 15% Si_3N_4 (Figure 9). These results show that the increase of the silicon nitride ratio in the composite material increases the shear strength of the weld zone. In some similar studies in the literature, it is stated that the amount of reinforcements increases while the weldability decreases in composite materials. However, the increase in Si_3N_4 reinforcement ratio in this study increased the weldability of the composite and the shear resistance of the welded area. The most important reason being that the size of the particles of Si_3N_4 used as reinforcing element is very small and is homogeneously distributed in the matrix structure [21].

The most important and effective parameter for this type of solid state diffusion is the welding temperature [22-23]. The increase in welding temperature in all joining process resulted in an significant increase in the cut-off resistance of the weld zone (Fig. 9; 10; 11).

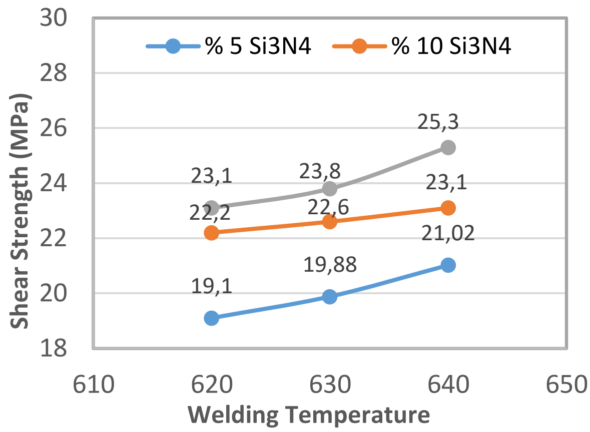

Figure 9. Comparing of shear strength values obtained from composite materials welded at different temperatures and for 1-hour duration.

Composite samples containing 15 % Si_3N_4 were welded at 620, 630, and 640 °C for 2h. The result showed the 83.2 %, 87 %, and 91 % of the shear strength of original un-welded material respectively.

The shear strength values show that while the welding temperature increases the diffusion at the welded sample interface continues to increase. According to the values seen in the literature, either the sintering temperature of the composite material production stage and the welding temperature values of the diffusion welding are selected to be slightly higher because of oxidation problem of especially Al powders.

During the production process of the powder metal part, the mixing of the powders in the mechanical mixing medium and especially during the pressing of the mixed powders in die after mixing, the partial oxidation of the aluminum powders could not be prevented completely. To prevent these oxidation problems, it is important to carry out that operations should be made in a protective atmosphere in glove box. In the mechanical mixing process, the aluminum powder particles are broken, the clean surfaces are exposed, and the internal energy that is loaded on the particles by deformation has an effect of accelerating the oxidation.

During sintering, the sintering temperature chosen is somewhat higher than the values given in the literature in order to allow diffusions to occur by exceeding the oxide film at the points of contact between the powder particles. In some applications, pressing process of powders is done at higher pressures than chosen in this process to try overcome negative effect of the oxide film been on the powder particles surface [24-26].

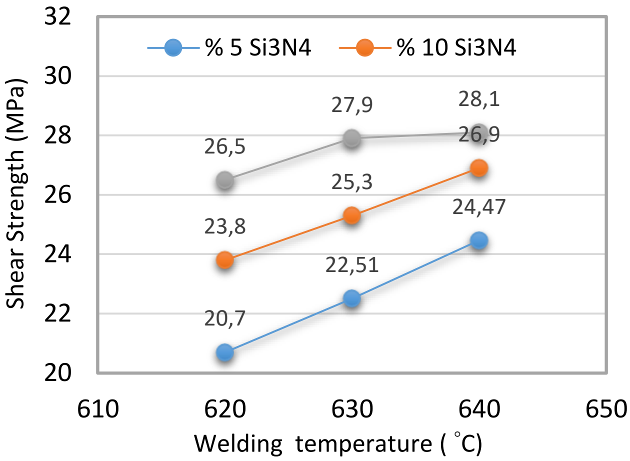

Figure 10. Comparing of shear strength values of composite materials welded at different temperatures for 1.5 h duration.

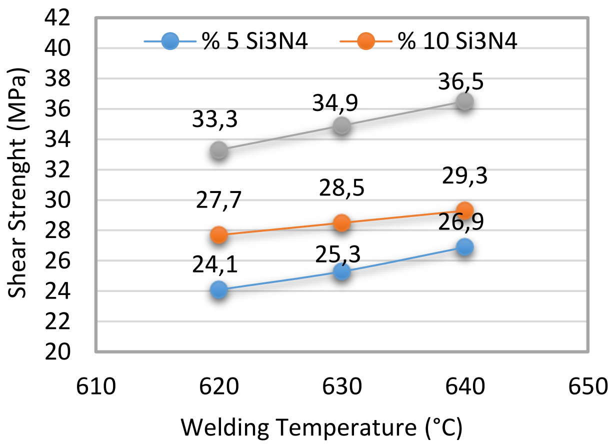

Figure 11. Comparing of shear strength values of composite materials containing different amount of Si_3N_4 and welded at different temperatures for 2 hours duration.

The welding double surfaces to be welded before the diffusion welding were degreased by sanding and welding was carried out in a protective gas environment. Thus, the oxide problem at the weld interfaces was removed considerably, and the desired welding was obtained by diffusing bonding at the weld interface of the samples (Fig. 12). As a result, in such samples, the weld zone shear resistance increased up to 91% of the original un-welded composite material. One of the main factors affecting the shear resistance of the welded zone of the welded specimens is the degree of contact between the to be welded surfaces before welding.

Especially for the interfaces of the samples to be welded, there should be full contact on the outer edge. Otherwise, edges that are not in contact with each other cause notch effect at this zone during the shear test, cause decreasing of shear resistance of the welding zone [Figure 13-14].

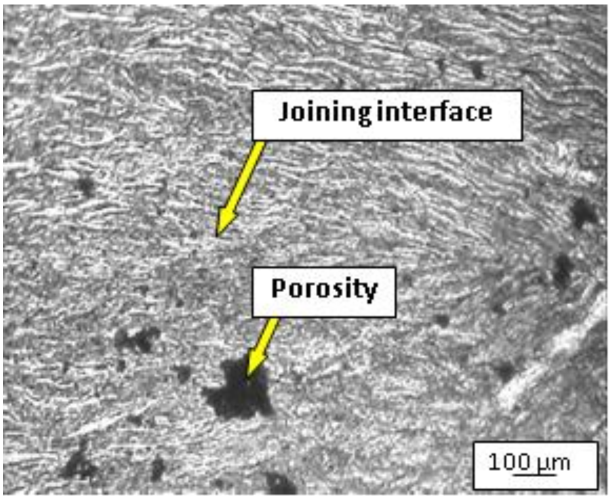

Figure 12. Composite: Al+15 % α-Si_3N_4 Welding Temperature: 640 ºC Welding duration:1 h.

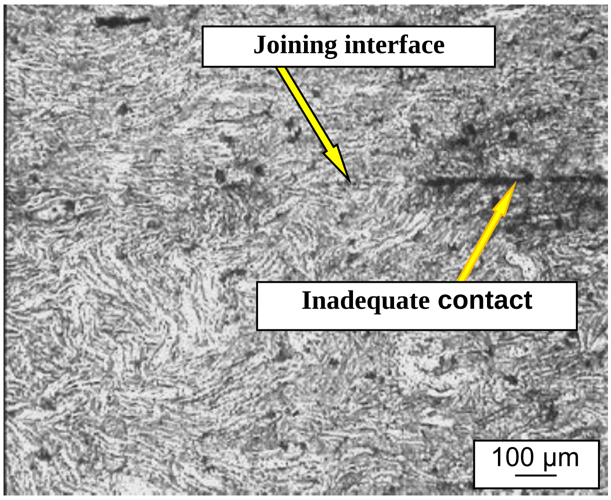

Figure 13. Composite: Al + 15% α-Si_3N_4 Welding Temperature: 640 ºC Welding Duration: 2 h.

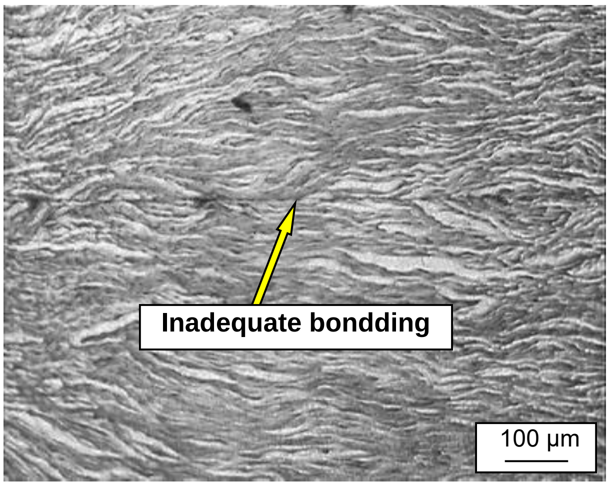

Figure. 14 Composite: Al + 5 % α-Si_3N_4 Welding temperature: 640 ºC Welding duration: 1 h.

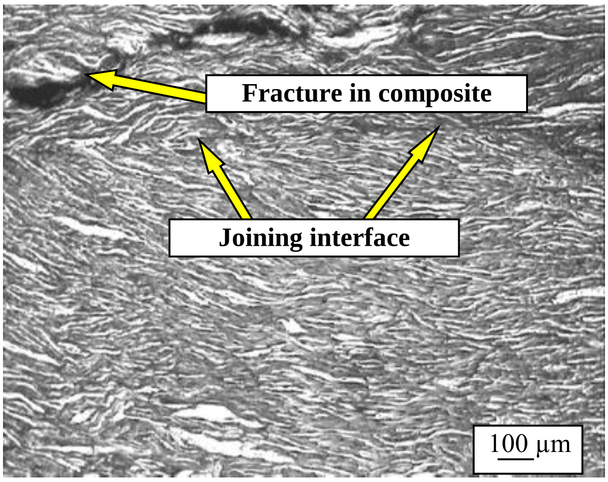

Figure 15. Composite: Al + 15% α-Si_3N_4 Welding Temperature: 640 ºC Welding Duration: 2 h.

For all samples with different composition and welded at different temperatures, test result show that increasing of the welding duration cause increasing of the shear resistance of welded zone (Fig. 9-11). Optical images obtained from microstructural studies show that increasing welding duration effects positively shear resistances of welded zone of samples.

Microstructural images show that diffusion bonding increases at the interface of welding couple when welding temperature and amount of α-Si_3N_4 increase (Fig.15).

CONCLUSION

1. In this study, composite specimens containing α-Si_3N_4 reinforcing elements at different ratios were successfully welded to each other with a diffusing welding under constant load and different temperatures and duration.
2. After the sintering maximum density increment was observed at powder metal composite sample containing 15 wt % α-Si_3N_4 and this reached 90 % density of theoretical density of mixture powders.
3. The shear resistance values show that the increasing welding temperature resulted in diffusion bonding at the interface of welding double.
4. Before welding process the surface must be fully contacted with each other. Non-contact surfaces are the most important barrier for diffusion that must be at the interface. When this problem is present on the outer surface of the welded double, the weld zone severely reduces the shear resistance by creating a notch effect during the shear test.
5. In the process of composite material production, it is necessary to form a more effective protective environment in order to prevent oxidation of mixture powders and pre-welding surfaces. Preventing oxidation during the production and welding of the composite material is important for the mechanical properties of the composite material and for the desired level of diffusion welding.
6. Powder metal composite sample containing 15 wt % α- Si_3N_4 and welded for 2 hour at 640 °C showed the best shear resistance result. During the shear test of this sample, 91 % of the shear strength of original composite material was reaced.

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