



## Investigation Regarding Al-Al<sub>2</sub>O<sub>3</sub> Composite Material Production and Its Weldability to Aluminum by Diffusion Welding

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### Abstract

In this study, firstly, a composite material aluminum based and containing 10 % Al<sub>2</sub>O<sub>3</sub> by weight is produced by powder metallurgy method. In the production of composite samples being 10 mm in diameter and 15 mm in length, first Al and Al<sub>2</sub>O<sub>3</sub> powders were mixed for 2 hours in a high – energy attritor and then pressed in a unidirectional axial die at 800 MPa pressure. The pressed samples were sintered in a tube furnace in a protective argon gas ambiance at 650°C for 2 hours. As for the second stage, the composite samples have been welded by diffusion welding to an Al block material in 99.5% purity and in the same dimensions at 5 MPa – constant load at different temperatures and durations by diffusion welding. The strength values of welded samples were determined by making shear tests from the welding zones versus composite material.

## 1. INTRODUCTION

Aluminum is a metal contained in the earth's crust in the most amounts and occupies the second row following iron and steel in terms of annual production and consumption values in the world. The facts that it is light and transmits heat and electricity easily and has higher strength against its density and higher oxidation resistance and possesses a good mouldability property are the leading features that cause us to over-produce and consume aluminum [1]. The largest consumption areas are the automotive and construction sectors, mainly aerospace and aviation industry. An important part of the studies accomplished towards improvement of its disadvantageous aspects, such as hardness and low melting point of aluminum is composed of the production of aluminum-matrix ceramic reinforced composite materials [2]. The reinforcement element consumed is in form of particulate fiber, continuous fiber or particulate and the particulate – reinforced Al - MMK production has a significant share in this area in terms of suitability for the secondary operations such as machining, welding mainly manufacturing ease. In the production of Al MMK, the applications are generally divided into two groups as melt and solid state method. The solid state production method is composed of MMK production with powder metallurgy at a large ratio. The reasons for this method to be basically preferred can be mentioned as the homogeneous distribution of the reinforcement element in the matrix structure, which offers ability of making production at relatively lower temperatures, and in desired chemical composition [3]. While Al MMK part production has become prevalent in space and aviation sectors at industrial scale, particularly in the automotive sector, the ability of these materials to be welded to similar materials as a post – production secondary process has gained even more importance [4-6].

While significant problems making the application difficult occurring when the aluminum – based materials are being welded to each other by the melt – welding method such as formation of oxide film at the heat – affected zone are experienced, when being welded to each other by solid state welding method being some kind of non – melting welding, high temperature – dependent significant problems such as the changes and oxidations in the material structure are not being lived [7,8]. Diffusion welding (DW) is one of the solid state welding processes, which two metallic surfaces are joined without local fusion by the application of

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pressure and temperature in vacuum or controlled atmosphere. The dissimilar metals, complex shaped parts, and a laminated plates can be welded by diffusion welding method. The diffusion welding is widely used in jewellery, automotive, machine, defence and aerospace industry [9]

According to the definition accepted by International Institute of Welding (IIW) at the proposal of Kazakov, the diffusion welding is a solid state bonding method in which atomic – level bonds are formed between the interface layers of the parts to be joined at as high temperatures as possible which would provide the diffusion when the intermediary cavities are taken up with the plastic deformation. Furthermore, according to the British Standard BS 499, the bonding with diffusion is a welding method applied to two matched surfaces to be joined by holding them at a pressure which does not lead to a detectable plastic flow until a metallurgical bond happens between the materials through solid state diffusion without affecting the material properties significantly [4,10]. In this study, the alteration in the shearing strength of welded zone has been analyzed depending on changing welding parameters by welding the produced  $Al_2O_3$  reinforced and aluminum matrix powder – metal parts to the pure aluminum by diffusion welding.

## 2. EXPERIMENTAL STUDIES

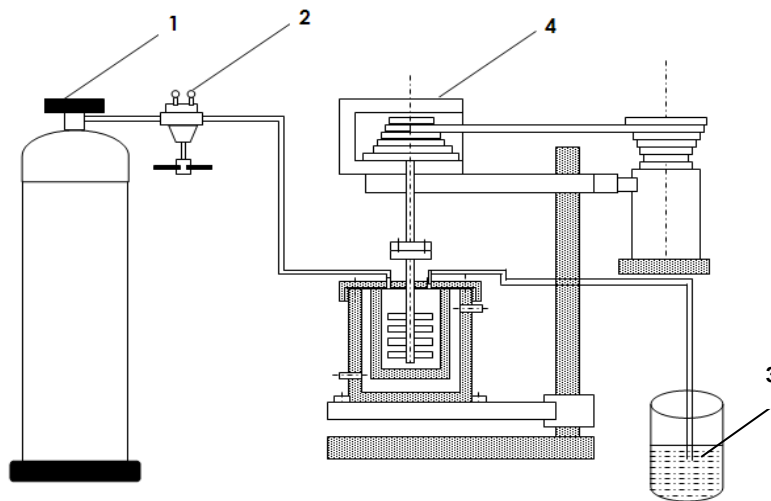
### 2.1. Production of Composite Material

In this study, Al powder with an average particle size  $56.95 \mu m$ , obtained from ECKART Dorn company, produced by gas – atomization method with code number “ECKA Aluminum AS 91/S” A 91/S in 99.5% purity has been used as a matrix material. As the reinforcement material,  $Al_2O_3$  powder in 99.52% purity and particle size  $> 25 \mu m$  has been used. The aluminum to be welded to the sample composites are in 99.56% purity and with diameter 10 mm and length 15 mm.

For the production of aluminum – matrix, alumina – reinforced composite material, firstly a mechanical mixing has been made to provide homogeneous distribution of alumina powders. The mechanical mixing process has been performed in a high – energy SZEGVARI brand attritor (Fig. 1). For mixing process, the steel balls being 10 mm in diameter and weighing totally 500 g. have been placed into the cylinder of attritor with volume  $1200 cm^3$ . Then 50 g. of mixture powder and 1% of Zn-Sterat lubricant have been added to prevent the aluminum powder from sticking to the inner surfaces of the cylinder and steel balls throughout mixing process (Tab. 1). Ensuring that the high – purity argon gas continuously passed through the cylinder of attritor, the medium has been put into inert state to avoid oxidation of aluminum throughout the mixing process. In order to prevent temperature rise throughout mixing process, environment has been constantly cooled by a water cooling system.

**Table 1.** Parameters of mixture powder production.

Powder mixture ratio (%)	90 Al + 10 $Al_2O_3$
Mixture powder quantity (g)	50
Quantity of balls (g)	400
Ball diameter (mm)	10
Mixture / ball ratio	1 / 8
Mixing period (h)	2
Protective gas	Argon
Mixing rate (Rev / Min)	450
Lubricant type	Zn-sterat
Lubricant ratio (by % weight)	1

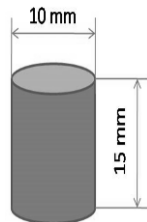


**Figure 1.** Mechanical mixing facility.

1) Argon cylinder, 2) Manometer, 3) Water container, 4) Attritor

In order to determine powder shape, the photos of the samples prepared prior to mixing Al and Al<sub>2</sub>O<sub>3</sub> powders and post – mixing of mixture powder have been taken at various magnifications on an optical microscope. First, the powder samples have been prepared for the metallographic with cold embedding and then rubbed down with respectively 240 – 1200 sandpapers. Finally, 1 μm Al<sub>2</sub>O<sub>3</sub> paste has been applied on the sample surfaces and then polished on the polishing disc ensure their readiness for a metallographic examination. By studying on an optical microscope, transformation of powders happened in the original particle morphology prior to being mixed by a mechanical mixing of the powders has been revealed.

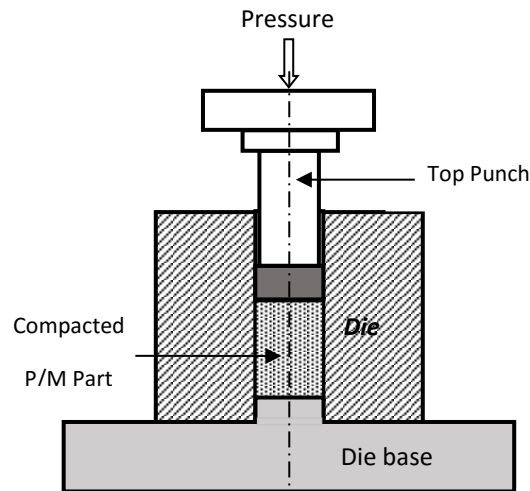
Block parts have been produced from the post – mixing powders of mixture in dimensions required for the diffusion welding process (Fig. 2).



**Figure 2.** Sample of diffusion welding.

The samples required for the diffusion welding has been obtained by causing the mixture powder to be pressed at 800 MPa pressing pressure at 1.5 mm / min. loading rate inside unidirectional axial die. Pressing operation has been performed at the axial die provided in Figure 3. While the compression was being executed at such die, the internal surfaces of die and bottom and top surfaces of the punch have been lubricated with Zn-Sterat + ethyl alcohol mixture. The purpose of this process is to prevent abrasion of die surfaces by decreasing the friction between the surfaces of mixture powder and die inside the die and to ensure easy removal of samples from the die. Another objective of this application is also to improve the surface quality of the powder metal part obtained.

In order to be able to obtain the welding sample being 15 mm in length and 10 mm in diameter, 3.32 grams of powder mixture have been loaded in the die at each time.



**Figure 3.** Die components.

After accomplishment of making the raw density measurements for the samples, they have been sintered in an atmosphere controlled tube furnace at 650 °C in an argon gas atmosphere for 2 hours. The main purpose of sintering is to materialize the inter-particle chemical bonding through atomic diffusion in the block parts and thus to achieve the intended strength and density in the block part. As to another aim of sintering is to remove the lubricant (Zn-sterat) used in the block samples from the medium. Block samples in a graphite boat have been placed into the center of a furnace while at an ambient temperature. Later on, an internal temperature of furnace was raised to sintering temperature by 5 °C / min – the heating rate was held at this temperature for 2 hours. The furnace was cooled down to ambient temperature by 5 °C / min – the cooling rate was controlled following the sintering process.

Post-sintering density measurement of each of the three samples, as with the raw density measurement, was accomplished on a precision scale by using the density kit according to the Archimedes principles. The average of density values for three individual samples has been taken as the raw density value.

Depending on sintering, the metallographic studies were conducted in order to be able to observe becoming sintered situation and microstructure in the inner structures of block samples. Cold – sink process (polyester) was implemented to the sintered sample blocks for the optical examinations. The photographs of samples have been taken at different magnifications in an optical microscope, as with their optical microscope studies, ensuring them to be passed through processes in the same manner.

In the diffusion welding efforts, the chemical composition of which is provided in Table – 2, the aluminum material being 10 mm in diameter and 15 mm in length has been prepared a by being cut perpendicularly so as to have a super finishing.

**Table 2.** Chemical composition of aluminum.

Chemical composition (%)							
Si	Fe	Cu	Mn	Ti	Zn	Mg	Al
0.25	0.40	0.05	0.05	0.05	0.05	0.05	Rest

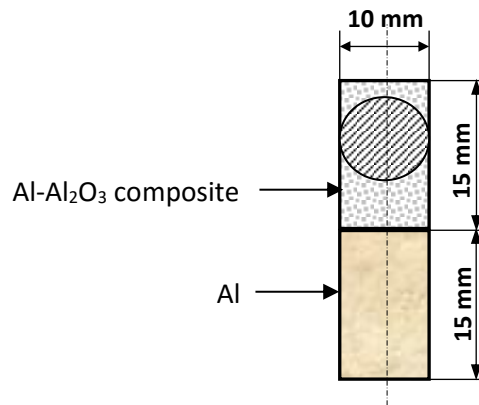
## 2.2. Preparing of Diffusion Welding of Samples

As the surface quality of sample in the diffusion welding is an important parameter, in this study, the sample surfaces were carefully sanded and made ready for the welding process. The surfaces of the samples to be welded to each other were sanded respectively with 240, 500, 800, 1000, 1200 sandpaper. Later on, they were cleaned with alcohol and dried in a drying machine and placed in a diffusion furnace without losing

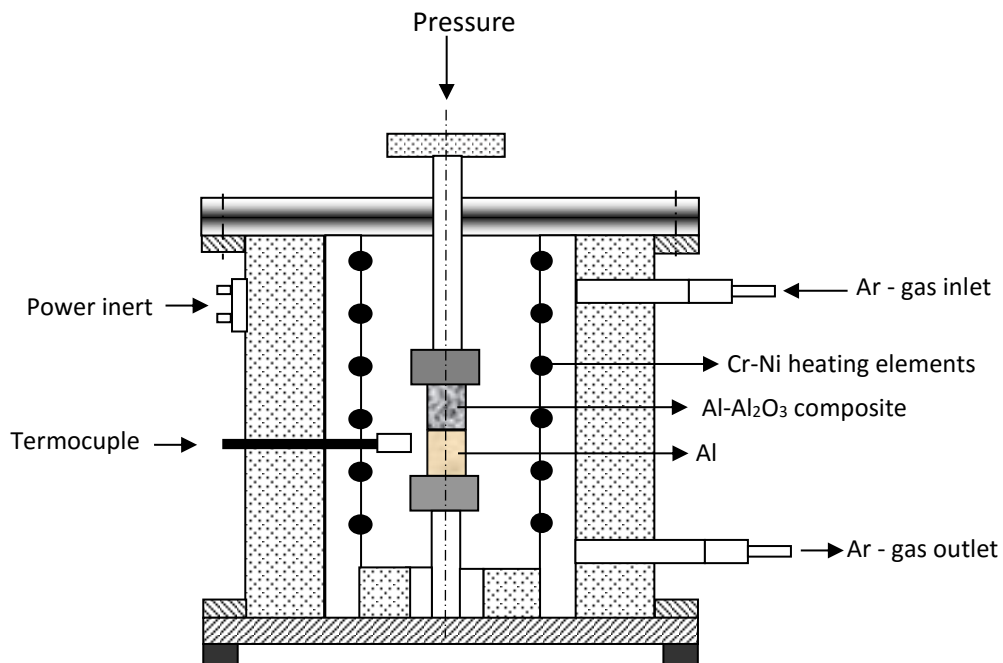
time (Fig. 4). Before the diffusion welding process was started, the welding medium is caused to be filled with high – purity argon gas.

To do so, the welding medium was made inert by sending protective gas to the medium for 5 minutes before activating the furnace to be heated. Later on, argon gas flow through the medium throughout welding operation has been maintained. When the welding process was terminated, the gas flow through medium continued under current burden until the ambient temperature reached 100 °C (Fig. 5).

Pressure from the pressure, temperature and time parameters of the diffusion welding has been kept steady in this study. The temperature and time have been modified. In the welding processes, 5 MPa pressure, 590 °C, 610 °C and 630 °C – welding temperature values and 30, 60 and 90 minute – welding times have been applied.



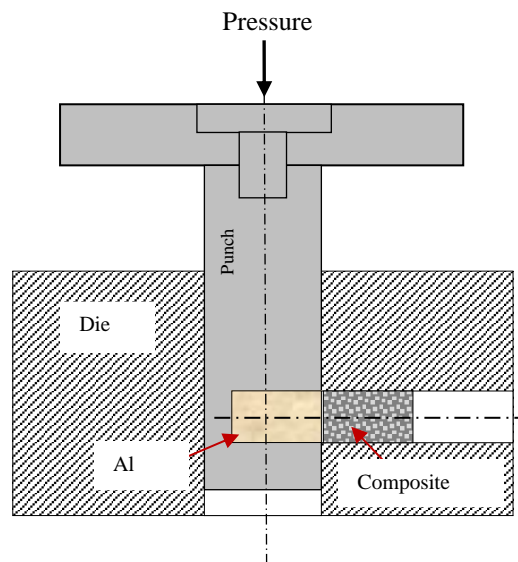
*Figure 5. Pre-welding weld pair.*



*Figure 5. Schematic view of diffusion welding furnace.*

### 2.3. Shear Test

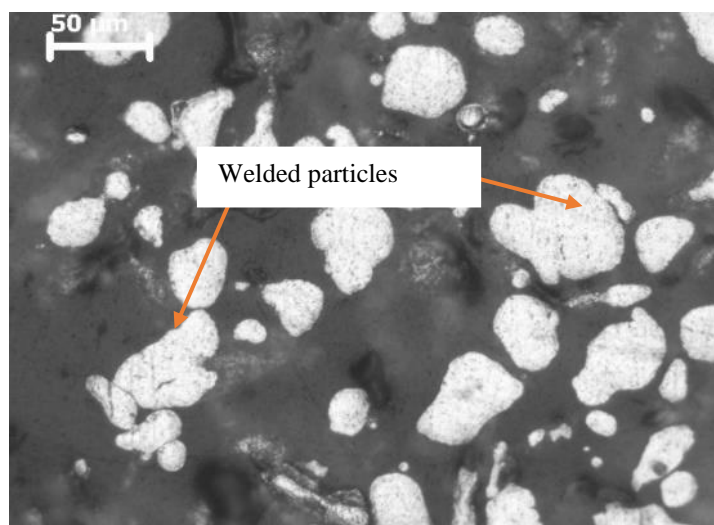
Upon completion of the diffusion welding operation, three welded samples each being taken from every group welded with different welding time and temperature were subjected to a shear test. The samples for which diffusion welding process performed have been subjected to shear test using a shear apparatus (Fig. 6). Firstly, the shear tests of aluminum and composite as the main materials has been performed and then welded samples have been cut from their welded joining places.



**Figure 6.** Schematic view shear test in the mold.

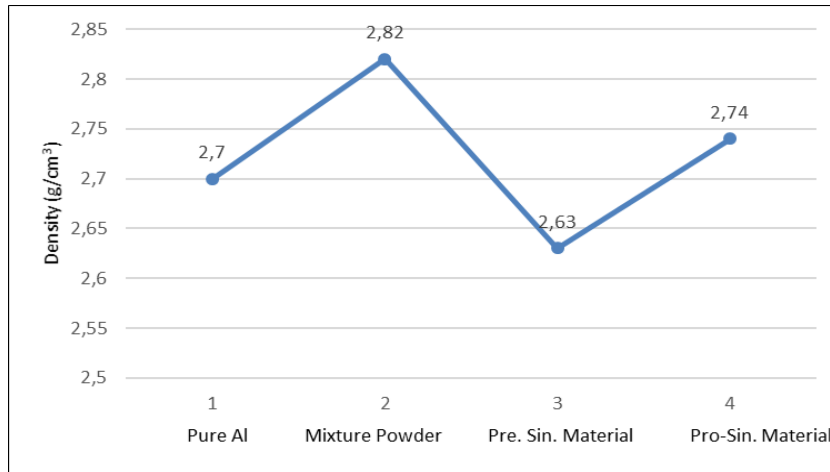
### 3. DISCUSSION AND CONCLUSIONS

Post – mechanical mixing change in powder grain shape which makes up the matrix structure in the composite material is shown in Figure 7. It is seen that the powder grain shape of aluminum powder produced by the atomization technique initially appeared to be almost spherical has been deformed in the mechanical mixing process and divided into smaller pieces and has even been welded to each other.



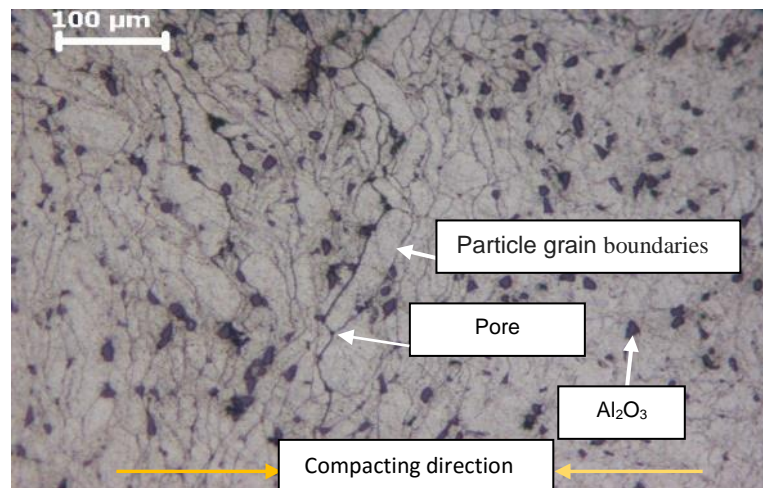
**Figure 7.** Post – mechanical mixing change in the size and shape of powder grain.

Post – pressing raw density value of composite material was measured as  $2.63 \text{ g/cm}^3$ . This value represents a 93% - density against the average theoretical density of mixture powder ( $2.82 \text{ g/cm}^3$ ). As for the post – sintering density, it has been measured to be  $2.74 \text{ g/cm}^3$  and this output corresponds to a 97% - density. Upon sintering, a 4% - density increment has been obtained in comparison to the raw density in the composite material (Fig. 8). The reason for this rise appears to be that a diffusing – induced chemical bonding has happened at every contact point of individual powder particles to each other in process of sintering operation. While the positive intrinsic energy gained by the aluminum powder particles during mechanical mixing by being deformed makes a positive contribution to sintering and density increase – despite all kinds of protection measures – partial oxidation occurring on the surface of powder particles before sintering come across us as a factor adversely affecting the sintering and density increase.



**Figure 8.** Alteration graphic in the density measurement results.

When the post – sintering internal structure of composite material is examined, it is seen that the boundary lines constituting the contact areas of powder grains which makes up the matrix structure are very clear. This image shows that the internal sintering of composite material produced itself is not at the desired level. This situation has also caused the shear strength of composite to come out lower. However, in general, the homogeneous distribution of reinforcement element  $\text{Al}_2\text{O}_3$  and pores seen in the matrix structure is a positive situation. It is seen that the existing pores are small in size and in shape close to a spherical form and that the pores are in patches between the matrix structure and the reinforcement element. This is a condition that weakens the bonding between matrix reinforcement elements and negatively affects the mechanical properties of the composite.



**Figure 9.** Composite material micro structures.

The shear test – applied samples are of three groups, pure Al group, composite group and the welded group. The shear tests have been performed firstly with pure Al, then the composite material produced (90% Al + 10% Al<sub>2</sub>O<sub>3</sub>) and finally the samples welded from their welded zone of the samples.

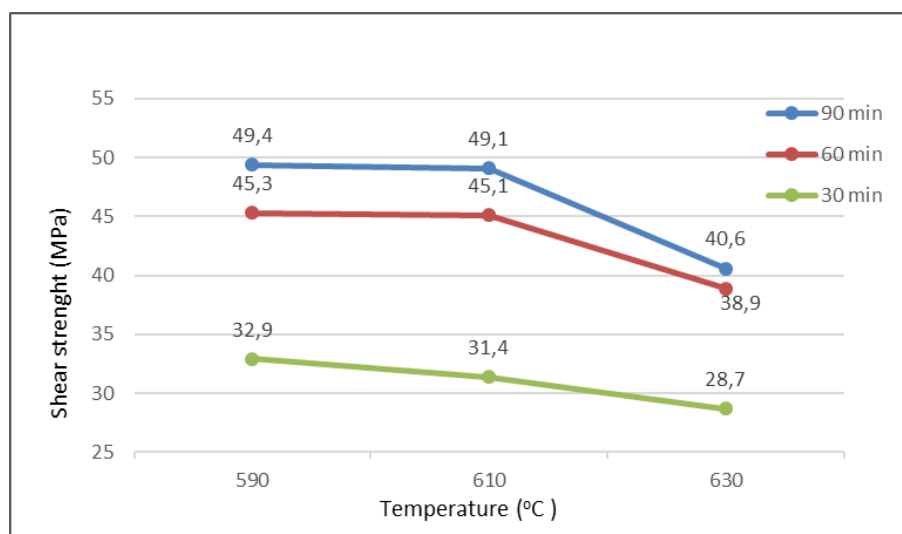
**Table 3.** Aluminum and composite cutting strength values.

Material	Shear strength (MPa)
Aluminum	121
Composite	50

At the moment of cutting, firstly the cutting load increases and despite the sample has not been completely cut at a certain stage, the load applied by the device for cutting decreases and after a while the press stops applying shearing load. The cause of this is just like post - maximum pull seen situations in the tensile samples. At the beginning, while the whole sample demonstrates resistance against being cut, the cross – section becomes smaller when certain amount of the sample is cut and the cutting of sample is maintained with plastic deformation without any rise in the applied load.

First, the shear resistance of the composite material was examined. The prime reason for the resistance of the composite come out to be low in comparison to the pure aluminum is – as mentioned earlier – non – occurrence of own internal sintering grade of composite structure itself is not at the desired level. In addition, it is the insufficiencies seen in bonding of the reinforcement element and the matrix structure to each other.

As a measure of being welded in similar studies conducted in the literature, the results of shearing resistance of composite (base material) and the shearing resistance of the region where the composite was welded to each other has been compared. In this study, the pure aluminum has been welded to the composite and the comparison of the welded material versus the shear resistance of the composite material. The average shearing resistance of composite material has been found to be 50 MPa following three separate measurements. When we look at the similar studies in literature, it seems that a shearing resistance of as much as up to 70-80% at the welded interfaces of the main material is an acceptable result [8- 12]. In this study accomplished, the maximum shearing resistance has been acquired at a sample welded at 590°C within a 90 minute – period. This shearing value 49.4 MPa obtained corresponds to 98.8% of the shearing resistance of the composite sample. This value is a value very close to the composite shear resistance (Fig. 10).



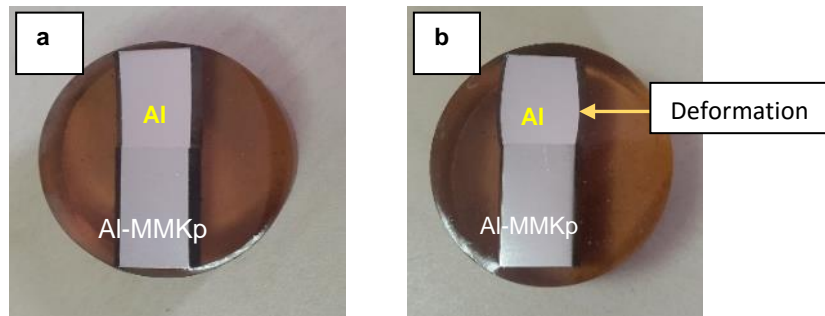
**Figure 10.** Temperature and time – dependent change in the shear strength of welded samples.

As seen in Figure 10, while the welding time increases in the joining processes made by weld at all three temperatures and the shearing resistance of the welding zone also increases. This result is also consistent



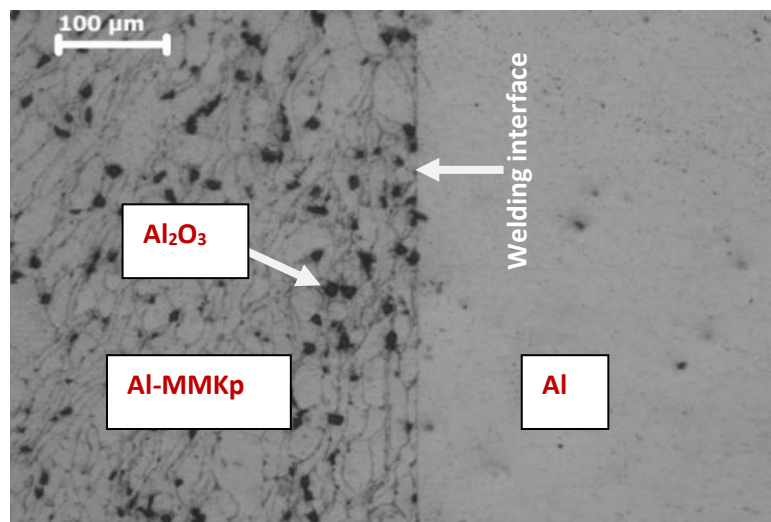
with similar studies in the literature and shows that the diffusion, namely getting welded, continues at the interface as the welding time increases [13].

When the variation of shearing resistance values according to the increase in the welding temperature is examined, the highest shearing resistance values for all welding times have been obtained from. Very close values have been obtained also with the joints made at 610 °C as with those made at 590°C. However, lower cutting strengths of the weld zones of the welded samples at 630 °C indicate that the weld temperature is higher for aluminum than for welds, although this does not create a problem for the composite. Although the welding temperature of shearing resistance of welding zones of samples welded at 630 °C in this performance made has come out to be lower does not lead to any problem for the composite from the welding pairs, it shows that it is high for the aluminum. When looked at in the literature, it is seen that while the melting point is taken generally between 0.50 – 0.70 of the welding point of the material welded, it is taken up to 0.90% in some studies [2, 14]. For this reason, in visual inspection of the pairs welded at 630 °C, it has been observed that the aluminum part was deformed (Fig. 11).

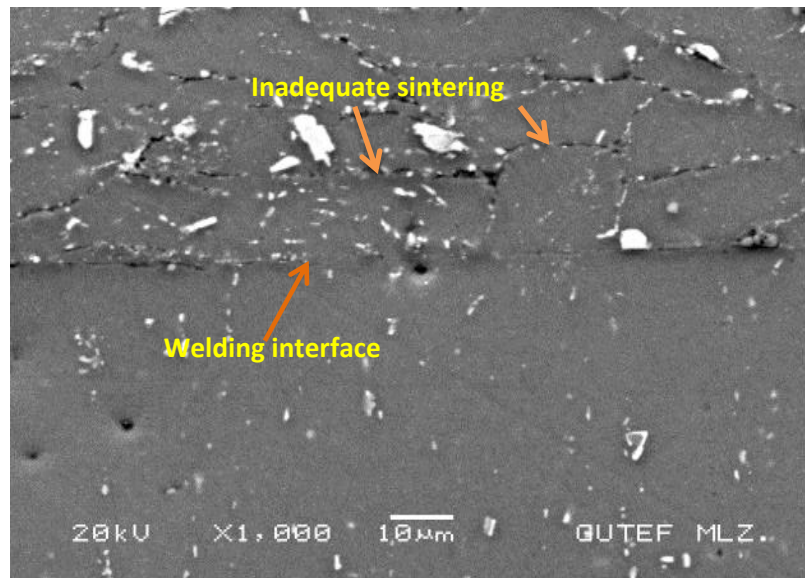


**Figure 11.** Post-welding view of welded pair. a) 590 °C - 60 min b) 630 °C - 60 min.

The welded zones of samples have been examined under an optical microscope following diffusion welding and their photos were taken and provided below. The joining line produced by the welding on the samples shows difference according to the temperature and duration of welding process. The clarity in the welding lines exhibited themselves being inversely proportional to the welding and diffusion. As the diffusion increases, the welding increased and the welding line has become less being inversely proportional hereto (Fig. 12 and 13). The increase in the welding time at all three temperatures has reduced the sharpness in the welding lines. The reason hereof has been seen that time and diffusion have proportionally augmented and promotion of diffusion has improved welding process and weakened the welding line.



**Figure 12.** The microstructure of a sample welded at 590 °C and in 90 minute duration.



**Figure 13.** SEM image of a sample welded at 590 °C–temperature and in a 90–minute period of time

**In conclusion;** it has been possible to make a successful diffusion welding of 10% Al<sub>2</sub>O<sub>3</sub> – reinforced MMK<sub>p</sub> structure produced by powder metallurgy method to a pure aluminum. According to the welding zone – provided shearing resistance, the best bonding between the diffusion welding and the welding pairs has been obtained in an application performed at 590°C for a 90 minute – period of time. With increased welding time, as the diffusion bonding increases in welding pairs, increased temperatures have led to visible deformation in the aluminum part of the welded pair made under current load (5 MPa). In the next process, the sintering behavior improvement – oriented studies can be conducted for the composite material produced.

### CONFLICT OF INTEREST

No conflict of interest was declared by the authors

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