Effect of SiC/Si₃N₄ micro-reinforcement on mechanical and wear properties of friction stir welded AA6061-T6 aluminum alloy

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Abstract

Friction stir welding (FSW) is extensively used to join aluminum alloys components for applications in structural, aviation and automotive sectors. In this investigation, the mechanical properties (tensile strength, percentage elongation, and microhardness), abrasive wear properties and microstructure of friction stir welded joint of AA6061-T6 were studied by inter-plate reinforcing of silicon carbide microparticles (μSiC) and silicon nitride microparticles ($\mu\text{Si}_3\text{N}_4$). The friction stir welding was performed at different tool rotational speed (TRS) (750, 1000 and 1400 rpm) and welding speed of 56, 80 and 112 mm/min. The friction stir welded joint without reinforcement and with μSiC exhibited maximum tensile strength at two combinations of welding speed and tool rotational speed; one at welding speed of 112 mm/min, TRS of 1000 RPM and other at welding speed of 56 mm/min and TRS of 1400 rpm. It was realized that the reinforcement of μSiC and $\mu\text{Si}_3\text{N}_4$ decreased the tensile properties of the welded joint but improved the abrasive wear and microhardness properties considerably in the stir zone. The reinforcement of μSiC during FSW demonstrated better tensile, microhardness and abrasive wear resistant properties than the FSW joints with $\mu\text{Si}_3\text{N}_4$ reinforcement. The improvement in microhardness of the FSW joint reinforced with SiC and Si₃N₄ microparticles is attributed to the pinning effect produced by the hard particles inclusions.

Keywords Friction stir welding · Mechanical properties · Silicon nitride · Silicon carbide · Aluminum alloy

1 Introduction

Friction stir welding is a solid-state welding technique mostly employed in the welding of aluminium alloys which is free from major weldability problems such as cracking and porosity encountered in fusion joining [1, 2]. In friction stir welding, a non-consumable tool is used with especially designed pin which is plunged into and along the adjoining line of two pieces of the similar or dissimilar materials. Tool rotational speed, speed of welding and tilting angle are the main parameters which affect the joint properties in FSW [3–5]. Although FSW is a good welding process for Al alloys as it is free from many welding defects yet FSW of

aluminium alloy decreases the mechanical properties significantly in the weld area due to partial annealing, overheating, overaging and coarse weld microstructure and wide heat affected zone (HAZ). A recent advancement in FSW is reported on the usage of sensor for controlling and monitoring of FSW process and a roadmap is developed to use industry 4.0. Sensors are being used to control the force, torque, temperature, current power vibration etc. during FSW process [6].

AA6061 series aluminum alloys being of light weight reflect moderate strength and are widely employed in structural, aviation and automotive sectors. The heat treatable aluminum alloy AA6061-T6 is not subjected to full

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annealing during the welding operation but subjected to partial annealing and overaging process. This Al alloy is very gullible to time at temperature; higher temperature and long duration; which leads to decay in strength of the base material as well as adjacent to the weld zone. Hence, it becomes imperative to control the various parameters such as preheating, interpass temperatures and overall heat input during FSW of the heat-treatable alloys. Usually, the most common heat-treatable alloys like AA6061T6, lose a large proportion of their mechanical strength after friction stir welding. For example, AA6061T6 normally owns a tensile strength of 310.26 MPa before welding and after welding, its strength reduces to 186.15 MPa [7]. This problem can be overcome by two ways: one technique is the post heat treatment of the alloy after the welding to acquire its mechanical strength back to original state. Some authors performed post weld heat treatment (PWHT) on friction stir welded AA6061T6 and observed that the welding efficiency was decreased by 65% as compared to base metal due to Portevin–Le Chatelier (PLC) effect, but the post weld heat treatment diminished the PLC effect and consequently improved the properties back to original state [8, 9].

The metallurgical effects of temper condition and post weld heat treatment has been widely studied on the friction stir weldability of similar and dissimilar aluminum alloy. İpekoğlu et al. [10] studied the effect of both O and T6 temper conditions on friction stir weldability of AA6061 aluminum alloy and observed that the O temper condition displayed an increment in hardness in the weld region due to grain refinement whereas those welded in T6 temper condition revealed a significant hardness drop in the weld region resulting from over-aging. Further, İpekoğlu et al. [11] investigated the effect of temper condition prior the joining and PWHT on microstructural and mechanical properties of friction stir butt-welded AA7075 aluminum alloy and revealed that the FSW resulted in a strength under-matching when the AA7075 was welded in T6 condition while a significant strength over-matching was recorded in the joints produced in O temper condition. The PWHT resulted in a significant recovery in the strength and thus joint performance when welded in T6 condition. There was a decrease in the joint performance value of the joints welded in O temper condition although the strength was increased. In another study of PWHT of FSWed AA6061 Al-alloy of different temper condition, İpekoğlu et al. [12, 13] found that the PWHT enhanced the mechanical properties of the AA6061 alloy in both O and T6-temper conditions much higher than those of respective as-welded plates and comparable to those of the respective base plates.

Liu et al. [14] reported an improvement in the strength of ZK60 alloy by post heat compression and ageing

treatment. Microstructure and mechanical behavior of friction stir welded Mg alloy were analyzed before and after heat treatment and it was reported that the tensile strength, efficiency and elongation were improved by heat treatment [15]. If option of PWHT is selected, the ability of filler alloy in response of the heat-treatment should be assessed. Generally, the filler alloys do not respond to PWHT if there is not significant dilution with the heat-treatable aluminum alloy. This cannot be easily achieved and it is difficult to control it normally. For this reason, filler alloys have been developed to independently respond to heat-treatment. The second option is to control the process parameter such as tool rotational speed and feed which is responsible for heat generation during welding and thus the reduction in mechanical properties can be avoided. The influence of welding parameters on mechanical and microstructural properties of friction stir welded AA6061 joint was analyzed and maximum hardness 85 HV was measured at 950 RPM and 3 passes. It was also found that number of weld passes and TRS had significant effect on the grain size [16]. Kumar et al. [17] estimated the heat input and process forces as function of different process parameters in FSW and found that the tool speed, welding speed, probe diameter and tool shoulder diameter were the critical process parameters which affected the heat input and FSW process forces. Kumar et al. [17] observed that the axial thrust (Z-force) was generally affected by the rotational speed, tool diameter, and welding speed. Astarita et al. [18] performed an experiment to find the mechanical properties of the welded joints of AA6061T6 Aluminum alloy made by arc and FSW and found that the joints produced by FSW had greater mechanical strength than the joints that was welded by manual argon-arc welding. The mechanical properties of the welded joints obtained by FSW process was very much dependent on the sizes of the tool and the size of the rubbing shoulder; when the tool size was larger, the better properties were obtained. Various researchers proposed different methods to decrease the heat input for example decreasing of TRS to travel speed ratio, cooling of plates by water during FSW [19], and using of backing plate for cooling purpose [20, 21] but most of these methods were found effective in reducing the heat input only in HAZ and TMAZ and had not much effect on the stir zone [22, 23]. Accordingly, a different method is required to regain the hardness lost during FSW due to partial annealing of the joint which can be achieved by using a reinforcement powder such as silicon carbide (SiC) and silicon nitride (Si_3N_4) in the welding zone which is believed to enhance the hardness and wear resistance properties of aluminum alloy. Eftekharinia et al. [24] studied the wear properties and microstructure of AA6061/SiC surface

composite obtained from friction stir processing by using different types of pins and number of passes and concluded that the geometry of the pin had major role in the distribution of the particle and it also affected the microhardness of stir zone. They found that the stir zone hardness was maximum when square shaped tool was used due to better dispersion of silicon carbide particle resulting in uniform microhardness variation. The number of pass also affected the microhardness and maximum microhardness was found with four passes. Ahmed et al. [25] investigated the effect of welding parameters on friction stir welded joint of AA6082 reinforced with aluminium oxide (Al_2O_3). Different TRS and reinforced percentage of Al_2O_3 was taken. Marzoli et al. [26] investigated the properties of FSW of $\text{Al}_2\text{O}_3/20\text{p}$ reinforced AA6061 alloy and concluded that the stirring effect of the tool influenced the distribution of reinforcement particles and its shape. Kumar et al. [27] studied the effect of Zn as an alloying element at the joint interface and found that the phase constituents were important in deciding the soundness of the welded joints. Li et al. [28] investigated the effect of different welding parameters on mechanical properties and microstructure of AA6061-T6 butt welded joints by a stationary type shoulder and concluded that the hardness profiles in both HAZ and TMAZ of AA6061T6 SSFSW joints revealed the softening regions along the both retreating side and advancing side. The lowest hardness value was observed as 60% of the base metal hardness and the welding speeds had more influence on the hardness profile as compared to the tool rotation speeds. Tool pin profile had also an effect on tensile strength of the joint. Marzbanrad et al. [29] performed experiments with different tool pin profiles and evaluated the microstructural and mechanical properties of friction stir welded joint and concluded that the peak temperature was higher in square pin profile than any other tool pin profile, and the higher temperature favored the plasticizing of the mixture and solid diffusion and the superior tensile properties were found with square pin profile.

Balasubramanian et. al [30]. demonstrated optimized process parameters of TRS of 1100 rpm and welding speed of 100 mm/min for producing defect-free friction welded joints in AA6061 aluminum alloy. Similarly, Rajakumar et al. [31] found the optimized process parameter of TRS of 1178 rpm and welding speed of 115 mm/min. In another study, Rajkumar et. al [32]. obtained the optimized process parameter of TRS of 1150 rpm and welding speed of 84.3 mm/min for producing the greatest tensile strength in FSW of 6061 aluminum alloy. In a similar study, Heidarzadeh et al. [33] demonstrated that the optimum process parameters to get maximum tensile strength of FSW joints of AA6061 alloy were 920 rpm of TRS, 78 mm/

min of welding speed while for maximum tensile elongation the parameters were obtained at TRS of 1300 rpm and welding speed of 60 mm/min. These finding has been used for the parameter selection process of this experiment.

This study aims to perform the FSW of AA6061-T6 with a different combination of tool rotational speed and feed to identify the best combination of these two parameters at which proper heat is generated to produce sound welding. Further, the objective is to find the effect of reinforcement of silicon carbide and silicon nitride in the welding zone on tribo-mechanical properties of the friction stir welded joints.

2 Material and experimental setup

In this experiment, AA6061-T6 aluminum alloy sheets of dimension 60 mm × 150 mm with 5 mm thickness were used for friction stir welding. The micropowders of silicon carbide (average particle size ~ 20 μm) and silicon nitride (average particle size ~ 45 μm) were introduced in-between the AA6061-T6 aluminum alloy sheets during the FSW process. Based on the earlier optimized parameters for defect-free weld joints as discussed in the introduction section, for maximum tensile strength and maximum tensile elongation, the process parameters in this experiment have been selected as TRS of 750, 1000 and 1400 rpm and welding speed of 56, 80 and 112 mm/min to conform the earlier findings as well as the limitations of FSW machine.

At first, a total of 9 different samples were welded with different combinations of TRS and travel speed without any kind of micro-powder addition. A rectangular groove of width and depth of 1.4 mm and 2.0 mm respectively was machined out along the top center-line of two adjoining plates using a shaper machine for accommodating 1.2 g of SiC and 1.0 g Si_3N_4 microparticles. The FSW tool (Fig. 1) was prepared on a lathe machine from mild steel and further tempered at 900 °C to increase its strength and hardness. A square-pin (6 mm x 6 mm) geometry was used for better dispersion of silicon carbide and silicon nitride microparticles in the welding zone. The shoulder diameter and pin length of the FSW tool were kept 18 mm and 4.5 mm respectively. A total 27 FSW joints were prepared as shown in Table 1.

The samples for the tensile testing were prepared by cutting the welding joint in the direction perpendicular to the welding direction by using a power hacksaw. The tensile test specimen was prepared according to the ASTM-E8 [34] standard consisting the stir zone in the center of gage length and the tensile test was performed on universal testing machine (UTM). The microhardness of the welding zone was measured by a Vickers microhardness tester with a load of 5 kgf for 10 s. The samples were also prepared for

Fig. 1 The FSW tool

sand abrasion test to find the wear behavior of the joint. The specimens for microstructure observation were prepared by using Keller's reagent as etching reagent. The distribution of silicon carbide was observed by scanning electron microscopy (SEM) and the microstructure of various regions was evaluated by optical microscopy.

3 Results

3.1 Microstructure

The microstructural examination of the FSW weld reveals four distinct sections i.e. base metal (BM), heat-affected zone (HAZ), thermomechanical-affected zone (TMAZ) and stir zone (SZ). The typical microstructure of these regions of the sample without any reinforcement having maximum tensile strength without reinforcement welded at a

welding speed of 112 mm/min and 1000 rpm is shown in Fig. 2a–d respectively.

Figure 3 shows the optical and SEM images of the stir zone of FSW specimen welded at welding speed of 112 mm/min and 1000 rpm without reinforcement (a and b), reinforced with the silicon carbide particles (c and d) and reinforced with silicon nitride (e and f) microparticles. The EDS spectra of friction stir welded joints with the inter-layer reinforcement of SiC microparticles and Si_3N_4 microparticles are shown in Fig. 4a, b respectively. The EDS spectrum reveals the presence of SiC microparticles in Fig. 4a and presence of Si_3N_4 microparticles in Fig. 4b.

3.2 Effect of process parameters on tensile strength

The tensile test specimen and fractured tensile specimen are shown in Fig. 5a, b. The effect of process parameters (rotational speed, welding speed) on ultimate tensile strength and percentage elongation is presented in Figs. 6 and 7 respectively.

3.3 Evaluation of microhardness variation

Vickers microhardness test was performed to measure the effect of reinforcement of SiC and Si_3N_4 microparticles in the welding zone. The samples possessing higher tensile strength were selected for the microhardness test. Hardness variation in the different welding zone like SZ, TMAZ, and HAZ was observed from the result obtained through Vickers microhardness test. Figure 8 shows the microhardness variation in the different zone for a welding speed of 56 mm/min and TRS of 1400 rpm.

Figure 9 shows the microhardness variation in welding zone at welding speed of 112 mm/min and TRS of 1000 rpm at which higher tensile strength was observed.

The maximum tensile strength of the joint produced by using silicon nitride as reinforcement was found at welding speed of 56 mm/min and TRS of 750 rpm so this sample

Table 1 Sample with specific sand abrasion wear rate at sliding distance of 1046.15 m, counter of 3000, wheel speed of 320 rpm and applied load of 10 N

Sample no.	Welding speed/ RPM	Tensile strength (Avg)	Powder used	Specific wear rate ($\text{mm}^3/\text{N m}$)
S1	56/1400	204.57	No	0.0223832
S2	80/1400	186.67	No	0.0324208
S3	112/1000	204.25	No	0.0253891
S4	56/1400	173.71	SiC	0.0191626
S5	80/1400	171.71	SiC	0.0142780
S6	112/1000	173.76	SiC	0.0098228
S7	56/750	171.43	Si_3N_4	0.0118089
S8	80/1000	163.71	Si_3N_4	0.0152979
S9	112/750	165.75	Si_3N_4	0.0141706

Fig. 2 Typical microstructure of friction stir welded joint without any reinforcement at welding speed of 112 mm/min and tool rotational speed of 1000 rpm **a** BM, **b** HAZ, **c** TMAZ, **d** SZ

was also selected for microhardness evaluation as shown in Fig. 10.

3.4 Effect on sand abrasion wear rate

Out of 27 samples, 9 samples were selected having a maximum tensile strength in their respective group for the sand abrasion test (3 samples from FSW without powder, 3 sample from FSW with silicon carbide powder, and 3 samples from FSW with silicon nitride powder). Each standard test was conducted for 1046.15 m of sliding distance. A multiple of tests was conducted keeping constant parameters with Counter of 3000, RPM of 210, Load of 10 N, and sliding distance: 1046.15 m (Fig. 11).

Specific wear rate of the selected samples is shown in Table 1 and Fig. 12.

4 Discussion

It was observed that the low welding speed and higher tool rotational speed provided a good amount of heat for sound welding. From Fig. 6, the maximum tensile strength of 6061-T6 aluminum alloy welded joints without the addition of powder was observed at two combinations of welding speed and tool rotational speed; one at 56 mm/min, 1400 rpm and another at 112 mm/min and 1000 rpm. Hanapi et. al did similar experiments and observed the maximum tensile strength of nearly 300 MPa. The difference in the result of this experiment and the authors result was due to the thickness and process parameters of the experiment that the author had taken [35]. Lakshminarayanan et. al [36] also performed

Fig. 3 Typical microstructure (optical and SEM image) of FSW joint at welding speed of 112 mm/min and tool rotational speed of 1000 rpm **a** and **b** without using any reinforcement, **c** and **d** with SiC as reinforcement, **e** and **f** with Si₃N₄ as reinforcement

similar experiments and found that the maximum tensile strength of FSW joint was around 240 MPa and the difference in result occurred due to the thickness of the plates, tool type and process parameter that author had taken. Exactly similar trends were observed in the welding joint by using SiC micropowder. The maximum tensile strength of 173 MPa was observed at two combinations of welding speed and tool rotational speed; one at 56 mm/min, 1400 rpm and another at 112 mm/min and 1000 rpm (Fig. 6). The tensile strength was decreased by

a small value of around 31 MPa by adding SiC powder. The tensile strength of the welding joints produced by using Si₃N₄ micropowder showed a totally different trend and the maximum tensile strength of these joints was observed at welding speed of 56 mm/min and 750 rpm. The tensile strength was much lower as compared to the tensile strength of the joint without powder. The greater decrement in tensile strength of the joints occurred due to bigger particle size of silicon nitrite which was envisioned to introduce brittleness in the joint. It was

Fig. 4 EDS spectrum of FSW joint with **a** μSiC reinforcement, **b** $\mu\text{Si}_3\text{N}_4$ reinforcement

observed from Fig. 7 that for the joint without reinforcement, the maximum percentage elongation of 7.39% and 7.38% were realized at a welding speed of 56 mm/min and TRS of 1400 rpm, and at welding speed of 112 mm/min and TRS of 1000 rpm respectively. At lower transverse speed, a high rotational speed provides sufficient energy for material flow and produces sound welding.

Fig. 5 Tensile test specimen **a** before and **b** after fracture

The ductile properties of FSW joints of samples without treatments were found to decrease with the reinforcement of SiC and Si_3N_4 microparticles except at welding parameter of 1400 rpm and 80 mm/min with the greater reduction realized with silicon nitride microparticles.

The average microhardness value of as-obtained base material (AA6061-T6) was HV108. It was observed from Fig. 8 that for a welding speed of 56 mm/min and tool rotational speed of 1400 rpm, the microhardness value in the stir zone increased up to HV95 which was maximum in case of silicon carbide as reinforcement. The microhardness was also found to be enhanced with silicon nitride reinforcement up to the maximum value of HV 85. The microhardness in stir zone was about HV72 in case of welding without any reinforcement. Abbasi et al. [37] observed that when AZ31 Mg alloy was reinforced with μSiC particles during friction stir welding, the SiC microparticles created pinning effect due to which grain growth was retarded and hardness was increased. Karthikeyan and Mahdevan [38] found that the addition of SiC particle in the weld zone during FSW of AA6351 alloy increased the microhardness of the welding zone. The higher hardness realized with SiC microparticles can be attributed to the higher hardness of SiC than Si_3N_4 and also the finer grain size of SiC than Si_3N_4 .

At welding speed of 112 mm/min and TRS of 1000 rpm, it was evident again that the microhardness of the joint produced by reinforcing SiC microparticles was much higher with HV104 magnitude in the stir zone. Microhardness profile of joint produced by using silicon nitride microparticles did not reflect any appreciable improvement in the hardness. The bigger size of silicon nitride particles led to the formation of coarse grain in the stir

Fig. 6 Tensile strength of FSW joints

Fig. 7 Percentage Elongation of FSW joints

Fig. 8 Hardness of FSW joint at welding speed of 56 mm/min and 1400 RPM

Fig. 10 Hardness of FSW joint at welding speed of 56 mm/min and 750 RPM

Fig. 11 Sand abrasion test specimen **a** before wear test, **b** after wear test

Fig. 12 Specific wear rate of FSW samples

from the Fig. 3c, d that SiC particles are uniformly distributed in the stir zone. The square-shaped tool pin was envisioned to increase the material flow and also caused

breaking of agglomeration and dispersing the SiC particles. From optical and SEM image of FSW joint welded with reinforcement of Si_3N_4 particles as shown in Fig. 3e, f, it was observed that due to bigger particle size of Si_3N_4 particles presence, the dent was seen in the SEM image which might lead to deterioration in tensile properties of the friction stir welded joints.

From the SEM image of FSW joint without reinforcement shown in Fig. 3b, the surface of SZ produced was uniform and no dent or fracture was detected in the sample which led to higher tensile strength. The SEM image of the SZ welded with SiC reinforcement (Fig. 3d) reflected that the surface structure was not as uniform as the sample with no reinforcement and the presence of uniform small dents led to a decrease in the tensile strength of the FSW joint. From the SEM image of the SZ welded with Si_3N_4 as reinforcement i.e. Fig. 3f, the surface showed non-uniform structure and there were too many dents present on the surfaces which transformed the structure matrix toward more brittle due to bigger size of Si_3N_4 particles which further led to a deterioration in the tensile strength of the joint.

5 Conclusions

The aim of this research was to enhance the microhardness and abrasive wear properties of AA6061-T6 aluminum alloy with minimum sacrifice in tensile properties at optimized FSW parameters for two different microparticles reinforcements along the adjoining surface. In the present study, AA6061-T6 aluminum alloy is joined by friction stir welding with/without silicon carbide and silicon nitride microparticles reinforcement in the welding zone. The tensile strength of the FSW joint without reinforcement was found to be maximum at two combinations of the tool rotational speed and welding speed; one at 1400 rpm and 56 mm/min and other at 1000 rpm and 112 mm/min. The joint produced using μSiC as reinforcement showed exactly similar trend and exhibited maximum tensile strength at same welding parameters to that of without reinforcement but the FSW joint produced with silicon nitride as reinforcement showed a totally different trend due to bigger particle size of silicon nitride. Tensile strength and percentage elongation of the FSW joint were found to decrease by adding μSiC and $\mu\text{Si}_3\text{N}_4$ as reinforcement and the decrement in tensile properties with $\mu\text{Si}_3\text{N}_4$ was more than the FSW joint with μSiC reinforcement because the reinforcement of $\mu\text{Si}_3\text{N}_4$ caused coarser grain structure of welded zone than those samples with μSiC reinforcement. The maximum tensile strength of FSW joint without any reinforcement, with μSiC reinforcement and with $\mu\text{Si}_3\text{N}_4$ reinforcement were measured to be 204.57 MPa, 173.60 MPa, and 171.51 MPa respectively. Microhardness of the FSW joint was increased by using SiC and Si_3N_4 microparticle reinforcement due to the pinning effect of hard particles reinforcements. The hardness of SZ was found to increase from HV72 (without reinforcement) to HV85 and HV95 with $\mu\text{Si}_3\text{N}_4$ and μSiC reinforcement respectively. The FSW joint produced by using reinforcement demonstrated a much lower abrasive wear rate as compared to the joints produced without any reinforcement. The reinforcement of SiC and Si_3N_4 microparticles served as load-bearing element and the reinforcement of SiC microparticles in FSW joints exhibited better abrasive wear resistant properties than the FSW joint with $\mu\text{Si}_3\text{N}_4$ reinforcement. The bigger size of the reinforcement particle led to the formation of brittle matrix which was envisaged to decrease the tensile strength and percentage elongation. Henceforth, it is envisioned from this research that the reinforcement of smaller and harder particles addition along the contacting interface enhances the microhardness and wear resistant properties with lesser sacrifice in tensile properties. This further directs the research community a future vision

to investigate the effect of different nanoparticles reinforcements to arrive best microhardness and tribological properties with least or no sacrifice in tensile properties.

Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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