Effects of Al₂O₃ nanoparticles volume fractions on microstructural and mechanical characteristics of friction stir welded nanocomposites

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ABSTRACT

The objective of the present study is to investigate the effect of Al₂O₃ nanoparticles volume percentage on tribological, mechanical and microstructural characteristics of 6061-T6 aluminum alloy based particulate-nanocomposite (P-NCs) fabricated using friction stir welding (FSW) process. Optical microscopy (OM) and scanning electron microscopy (SEM) was employed to evaluate the (a) microstructures of the produced nanocomposites to ascertain the distribution of Al₂O₃ nanoparticles in the nugget zone; (b) nanocomposite depth formed on Al-alloy matrix, and (c) fractured and wear characteristics. Results reveal that the produced P-NCs had a depth of 3286 μm across the perpendicular x-section of the weld nugget zone of P-NCs. With the increase in a volume percentage of Al₂O₃ nanoparticles there was a tremendous increment in the microhardness up to 125 HV which is higher than as-received AA6061-T6. It was also noticed that the tensile strength and the wear resistance of produced P-NCs were significantly increased at 0.3 vol% of Al₂O₃ nanoparticles as compared to 0.2 and 0.4 vol%. The corresponding mechanical and wear properties results were correlated to microstructure and fractography results.

1. Introduction

Nowadays, weight reduction and reduced fuel consumption are the two most important demands in the marine, aerospace, and automobile industries. To fulfill such demands, light metals as aluminum alloys are the most suitable [1, 2]. Heat treatable Al-alloys especially AA6061-T6 are most commonly employed in marine frames, pipelines, aircraft, marine, and construction, etc., due to their high corrosive resistance, and high material strength to weight ratio, and ability of welding [3, 4].

Metal matrix composites (MMCs), particularly particulate matrix composites (PMCs) that are fabricated by adding reinforcement particles (RPs), either in fiber form (boron, silicon carbide, fiber, etc.) or in particle form (aluminum oxide, titanium oxide, etc.) into their material matrix (Al, Mg, Ti, Cu alloys, etc.) are the latest materials [5, 6]. These
PMCs have exceptionally high thermal conductivity, high strength/weight ratios, high corrosive resistance, and high stiffness compared to the base alloy which makes them the center of attraction to many industries such as, automobile, aerospace, marine, nuclear, transportation, and so on [7].

However, uniform distribution of RPs in the aluminum substrate and their control is complicated and difficult to achieve via conventional methods for base matrix modifications [8, 9]. Previously, thermal spraying and laser beam methods were adopted by numerous researchers to produce P-NCs which result in degradation of mechanical properties due to the occurrence of undesirable secondary phases [10, 11]. Because these methods were operated at very high temperatures due to which it is very difficult to stop the unwanted chemical reactions between the RPs and the material matrix that forms the detrimental secondary phases [12, 13]. To avoid these limitations, a process is required for the production of P-NCs that do not reach the melting point (m.p.) of the base material (i.e. operates below m.p). In correspondence to the above problems, Friction Stir Welding (FSW) is the most suitable process for the fabrication of P-NCs on Al-matrices.

FSW process was patented and experimentally developed at The Welding Institute (TWI) in the UK in 1991 [14]. In the FSW process, a rotating FSW tool, once it touches the workpiece surface, is plunged into the joint interface utilizing an applied mechanical force. As a result, the material softens by the amount of heat generated by the friction between the FSW tool and the base material. With the transverse movement of the rotating tool more recirculation of plastically deformed material is forged in the forefront to the rear of the tool and this induced plastic deformation that results in solid-state joining [15, 16].

In contrast to conventional techniques used for the fabrication of P-NCs, FSW provides enormous advantages such as; (a) due to the solid-state joining no melting as well generation of chemical reactions will occur between RPs and the matrix, (b) due to severe plastic deformation FSW leads to vigorous mixing and refinement of microstructural characteristics in the parent alloy, (c) with the proper consolidation of material due to adequate amounts of localized heat produced and tool stirring action during FSW leads to the generation of a uniform P-NCs on an aluminum alloy matrix.

Attempts were made in the present study to produce aluminum oxide (Al₂O₃) nanoparticles based AA6061-T6 particulate-nanocomposites (P-NCs) on an Al-matrix via FSW. The effect of volume fractions of Al₂O₃ nanoparticles on microstructure and mechanical characteristics of AA6061-T6 based P-NCs produced using friction stir welding was also studied.

### 2. Materials and methods

AA6061-T6 thin Al-sheets of thickness 2.5 mm were used for FSW. AA6061-T6 chemical composition was obtained by electro-dispersive X-ray spectroscopy that is given in Table 1. The reinforcing particles, such as Al₂O₃ nanoparticles were used in different volume percentages, such as 0.2, 0.3, and 0.4 vol%. Nano Partech, Chandigarh, India, supplied Al₂O₃ nanoparticles with 99.99% purity and >30 specific surface area.

Al₂O₃ nanoparticles were incorporated into the cylindrical holes of varying diameter and fixed depth 3 mm drilled in a zigzag pattern at an equal distance on the faying surface of the adjoining faces of AA6061-T6 sheets as reported in the previous research by Singh et al. [17-19]. The average grain size of as-received Al₂O₃ nanoparticles was 15 nm which is assessed by using TEM as shown in Figure 1.

The FSW tool with the cylindrical shoulder of 15 mm diameter and cylindrical pin (with a round bottom to reduce wear and increase tool life) of length and diameter 2.30 mm and 5 mm were used. The upper surface of the adjoining faces was closed using a pin-less tool to avoid the scattering of RPs from the drilled holes during the FSW process. The selected process parameters range along the central joint line used in FSW is mentioned in Table 2.

FSW was conducted on a dedicated vertical milling machine (HMT: VMC-TC-1200). Once the FSW was finalized, a small cross-section of 10 mm x 10 mm samples was extracted from the weld nugget zone of P-NCs (normal to the FSW direction) for microstructural characterization and evolutions. Samples were prepared by employing the standard procedure of metallography using Keller’s reagent and then examined under an optical microscope (OM: Leica DM 2700 M) and scanning electron microscope (SEM: Jeol-124Nx, Japan). Micro-hardness testing was done along the central portion of the nugget zone (NZ) of P-NCs (normal to the FSW direction) at a distance of 0.5 mm below the top surface of P-NCs by employing 200 g load for 15 s. Tensile and wear properties of the produced P-NCs were also evaluated. Tensile test samples were cut according to ASTM-E8M-11 from the P-NCs by using wire electrical discharge machining (WEDM) and performed using a cross-head velocity of 2 mm/min. The wear test for P-NCs was performed by employing a pin-on-disk arrangement as per ASTM: G99-05 standard. The rounded shape pins of diameter 5 mm were cut from the NZ (nugget zone) for which the pin was positioned normal to the FSW.

| Alloying elements | Mg | Si | Cu | Mn | Cr | Zn | Ti | Al |
|-------------------|----|----|----|----|----|----|----|----|
| Wt.%              | 0.901 | 0.615 | 0.258 | 0.0454 | 0.19 | 0.054 | 0.0195 | Bal |

Table 1. Chemical composition of AA6061-T6.
direction. Disc employed for wear testing was made up of EN31 steel with 62 HRC and the sliding track diameter of the disc surface was 100 mm. Tests were performed under the dry-sliding condition with 15 N of normal load along with 450 and 0.07 of disc rotational (rpm) and sliding velocities (mm/s).

3. Results and discussions

3.1. Microstructural characterization

The size of the friction stirred nugget zone (NZ) was observed analogous to the FSW cylindrical tool pin diameter and length of 5 mm and 2.30 mm respectively which is attributed to the higher amount of frictional localized heat occurred between the rotating tool shoulder and base Al-matrix, such that these results are concurrent with the findings of Eftekharinia et al. [20]. The cross-sectioned NZ depth of fabricated P-NCs perpendicular to the FSW direction was calculated as 3286 μm as depicted in Figure 2. SEM photomicrographs of Al-Al₂O₃ based P-NCs and optical micrograph of as-received aluminum parent matrix are illustrated in Figure 3.

It can be noted that the dispersion of Al₂O₃ nanoparticles in NZ is more evident and uniform during FSW. This was attributed to the following reasons, (a) occurrence of severe plastic deformation (SPD) and continuous dynamic recrystallization (CDRX) in the weld zone [21], (b) presence of Al₂O₃ nanoparticles that strongly influenced the size of the grains in the NZ by providing obstruction to the motion of grain boundaries via Zener-pinning effect [15, 22]. It was also noticed that at higher tool rotational speed of 2000 rpm it causes more homogeneous nanoparticles distribution in the NZ, while the transverse speed doesn’t significantly affect the distribution of RPs, these results are in good agreement with the studies reported by Eskandari et al. [23], Gou et al. [24] and Faradonbeh et al. [25]. However, it is inferred from the Figure 3 that the Al₂O₃ nanoparticles are more homogeneously distributed in the NZ at 0.3 vol% in comparison

Table 2. Process parameters window for FSW.

| FSW process parameter | Rotating speed r/min | Traveling speed mm/min | Tilt angle (°) | Pin profile | Concave shoulder diameter (S_d) mm | Cylindrical pin diameter (P_d) mm | S_d/P_d | FSW passes | Al₂O₃ nanoparticles volume % |
|-----------------------|----------------------|------------------------|---------------|-------------|----------------------------------|----------------------------------|---------|-------------|-----------------------------|
| Respective values     | 2000                 | 70                     | 0             | Cylindrical round bottom          | 15                               | 5                                | 3       | 1           | 0.2, 0.3, 0.4                |

Figure 1. (a) Schematic for incorporating RPs into the base matrix, (b) TEM micrograph of Al₂O₃ nanoparticle.

Figure 2. SEM photomicrograph depicts the depth of fabricated P-NCs on Al-matrix.
to 0.2 and 0.4 vol% for Al/Al₂O₃ based P-NCs fabricated via FSW. This was due to the vigorous stir action induced by the FSW tool, as a result, an adequate amount of heat and mechanical force was generated which is sufficient for the uniform dispersal of RPs in the wide area of nugget zone [21, 26, 27]. These findings are in better agreement with the studies conducted by Keneshloo et al. [28] and Paidar et al. [29] according to which the area/volume fraction as well the size of the reinforcement particles are inversely proportional to each other.

3.2. Micro-hardness measurements

The micro-hardness profiles extracted from the NZ of Al-Al₂O₃–0.2%, Al-Al₂O₃–0.3%, and Al-Al₂O₃–0.4% based P-NCs and base alloy matrix (i.e. AA6061-T6) is shown in Figure 4. It is well understood that the micro-hardness variation in the processed nugget zone merely depends upon the existence of Al₂O₃ nanoparticles and also their uniform dispersion [30]. It is worth mentioning that, due to the large volume fraction of Al₂O₃ nanoparticles in the NZ their expansion to volume rate is quite high which leads to an

Figure 3. SEM micrographs of produced Al/Al₂O₃ based P-NCs, (a) 0.2 vol%, (b) 0.3 vol%, (c) 0.4 vol%, and (d) As-received AA6061-T6.

Figure 4. Micro-hardness measured transversely from weld centreline for Al/Al₂O₃ based P-NCs and base material.
increase in micro-hardness to 125 HV which is higher as compared to the as-received base material. This was attributed to the high rotational speed (2000 rpm) of the FSW tool due to which shoulder produced a sufficient amount of localized heat and mechanical plunge force which is required to disperse RPs more homogeneously and also to consolidate soft deformed plasticized material circulated about the FSW tool [18, 19]. These results are in better concurrence with Paidar et al. [31] according to which the WC nanoparticles addition during FSW of AA5182 alloy remarkably increases the hardness of the weldments compared to unreinforced welds that are attributed to the grain growth inhibition via the existence of RPs in the NZ (according to Hall–Petch relationship according to which small grain size leads to better microhardness).

It is noticed that with the increase in the volume fraction of Al2O3 nanoparticles it causes more grain refinement in the NZ which is attributed to the more softening of base material that results in a decrease in ductility of P-NCs as a result of ductile matrix [32]. Moreover, the microhardness values were significantly increased with the loading of Al2O3 nanoparticles. The addition of Al2O3 nanoparticles has a significant pinning effect on the migration of grain boundaries, which results in the refinement of grains in NZ, as the same was suggested by Paidar et al. [33]. They stated that with the increase in stirring action and vigorous plasticized material flow strain (which is a function directed by rotation tool pin) it helps in to break and disperse the reinforcement particles during FSW. Based on this, it was concluded that in NZ the grain size was dependent on the transverse as well as tool rotational speed and the presence of Al2O3 nanoparticles in the NZ. As the FSW process is more similar to SPD, where the heat generated and the rate of strain has an important effect on the refinement of grains, such that the dynamic recrystallization (DRX) grains size keeps on decreasing by a decrement in the process temperature or increment in the rate of strain [3, 17].

### 3.3. Mechanical property evaluations

The calculated values of tensile properties for Al-Al2O3–0.2%, Al-Al2O3–0.3%, and Al-Al2O3–0.4% based P-NCs and base alloy matrix (i.e. AA6061-T6) are graphically shown in Figure 5. The tensile properties of Al-based P-NCs depend on the (a) size of the grains, (b) dislocation density, (c) percentage of agglomerated particles in the welded zone, and (d) bonding quality. [34] as also reported by Faradonbeh et al. [25], Eftekharinia et al. [20] and Paidar et al. [31]. It can also be observed that the tensile properties (ultimate tensile strength (UTS), yield strength (YS), and elongation at break (EL) of the produced P-NCs were decreased as compared to the as-received base material. This was attributed due to the presence of Al2O3 nanoparticles that confined the grain boundaries sliding and prevent dislocations motion that results in degradation of tensile properties [35, 36].

Also, the findings showed that the combination of lower travel speed and medium Al2O3 nanoparticles volume percentage (0.3 vol%) leads to increase in tensile strength significantly which is related to the more adequate uniform dispersion of nanoparticles in the NZ, these results are in better agreement with the findings of Faradonbeh et al. [25] according to which the decrease in tool travel speed results in finer B4C particles clusters and gradual improvement in the poor distribution of B4C particles in the NZ.

It is worth mentioning that the increment in Al2O3 nanoparticles volume percentages, the UTS, YS, and EL% was significantly decreased. Moreover, increment in Al2O3 nanoparticles loadings results in (a) increment in the inter-particle spacing area between the Al2O3 nanoparticles and the Al-matrix, as a result, lower interfacial area were created which leads to the accumulation of Al2O3 nanoparticles and results in deterioration of tensile properties [37], (b) less load transferred from the weak Al-

![Figure 5. Tensile properties of Al/Al2O3 based P-NCs and base material.](image-url)
matrix across the matrix/particle interface to the high stiffness and hard ceramic Al₂O₃ nanoparticles may occur that leads to a decrease in strength of the Al-matrix [36, 38], (c) decrement in work hardening rate which leads to a decrease in elastic modulus, macroscopic yielding, and tensile strength [39], (d) increment in a distance of effective slip during deformation that leads to the lower ductility due to early onset of void nucleation with an increase in the volume fraction of Al₂O₃ nanoparticles [38, 39].

Similarly, Barmouz et al. [40] and Paidar et al. [29] also stated that agglomeration of reinforcements, peak temperature, and large strain rate is the governing main parameters for the tensile behavior that results in crack nucleation and propagation due to which the failure of the welds could take place along the AS than RS. The SEM photomicrographs for fractured portions of Al-Al₂O₃–0.2%, Al-Al₂O₃–0.3%, and Al-Al₂O₃–0.4% based P-NCs and base alloy matrix (i.e. AA6061-T6) is shown in Figure 6. Fracture appearance of the base matrix (AA6061-T6) reveals the occurrence of deep small dimples which indicates the mode of ductile fracture and affirms the improvement in ductility [41]. The decrease in tensile properties of Al-Al₂O₃–0.2% and Al-Al₂O₃–0.4% based P-NCs is attributed to the presence of large shallow dimples with overlay and separation of RPs layer as same was reported by Faradonbeh et al. [25] according to which it is due to de-cohesion between B₄C particles and matrix, whereas for Al-Al₂O₃–0.3% based P-NCs it consists of small size dimples that results in an increase of tensile strength as similarly reported by Paidar et al. [31] according to which the occurrence of small dimples is due to strong deformation of the NZ as well the uniform dispersion of WC reinforcement at lower travel speed. These outcomes justify the variations in tensile strength of Al-Al₂O₃–0.2%, Al-Al₂O₃–0.3%, and Al-Al₂O₃–0.4% based P-NCs and the cause for a decrease in tensile strength in comparison with the base Al-alloy.

Figure 6. SEM photomicrographs of fractured surface for Al/Al₂O₃ based P-NCs, (a) 0.2%, (b) 0.3%, (c) 0.4%, and (d) As-received AA6061-T6.

Figure 7. Frictional coefficient values for Al/Al₂O₃ based P-NCs and as-received AA6061-T6.
3.4. Wear characteristics

The corresponding results of wear resistance for Al-Al$_2$O$_3$ based P-NCs and base material are illustrated in Figure 7. It is inferred from Figure 7 that there is a decrease in wear rate with the increment in Al$_2$O$_3$ nanoparticles loadings which is attributed to an enhancement in hardness profile due to the uniform distribution of Al$_2$O$_3$ nanoparticles, and behave as applied load-carrying nanoparticles [42]. It was also observed that the wear resistance was increased at 0.3 vol% of Al$_2$O$_3$ nanoparticles is due to a reduction of frictional coefficient provided that these nanoparticles are distributed uniformly within the aluminum matrix, whereas wear rate was increased due to the presence of hard Al$_2$O$_3$ nanoparticles, which comes out during the wear test by the prismatic pin, laying on the disc and acting as a barrier. These results are in better agreement with the findings of Paidar et al. [31] and Eftekharinia et al. [20] according to which the wear rate is directly proportional to or as a function of RPs distribution and the average grain size. It is noted that an increase in tool rotational speed with a decrease in travel speed leads to better Al$_2$O$_3$ nanoparticle distribution in NZ, in turn, increases the wear rate of the reinforced (AA6061-T6) base alloy.

Moreover, in the case of Al-Al$_2$O$_3$–0.2%, Al-Al$_2$O$_3$–0.4% based P-NCs due to the increase in Al$_2$O$_3$ nanoparticles loadings it leads to a conversion of adhesive wear into abrasive wear, that results in separation, and overlay of P-NCs, instead of erosion which leads to large weight loss compared to parent material and Al-Al$_2$O$_3$–0.3% based P-NCs. However, the least wear rate was observed at 0.3 vol% of Al$_2$O$_3$ nanoparticles based P-NCs.

The morphological characteristics of wear surface of Al-Al$_2$O$_3$ based P-NCs and parent material are depicted in Figure 8. It can be observed from Figure 8 that due to the presence of mechanically mixed tribo-layer that functions as a solid lubricant could transform the behavior of wear from dual to trilogy wear which leads to the reduction in wear rate [38–40], as these results are analogous to the findings by Paidar et al. [33] according to which the sufficient distribution of SiC particulates, small SiC particles, and fine equiaxed grains size in NZ are utmost factors that significantly influence the worn surface of reinforced FSW welds. However, due to the existence of strong Al$_2$O$_3$ nanoparticles, the path of wear is a little larger for the base material; in comparison with Al-Al$_2$O$_3$ based P-NCs. Moreover, in the absence of Al$_2$O$_3$ nanoparticles during the wear testing, it leads to the cultivation of micro-surface upon contact and turns into uneven
surfaces [42] as also reported by Eftekharinia et al. [20] and Paidar et al. [29].

4. Conclusions

Using Al₂O₃ nanoparticles as reinforcements an Al/Al₂O₃ based P-NCs were successfully produced on AA6061-T6 using friction stir welding (FSW). The impact of Al₂O₃ nanoparticles on microstructural, mechanical, and tribological characteristics of AA6061-T6/Al₂O₃ based P-NCs fabricated via FSW were examined and the major outcomes of the present study are given below:-

1. Al₂O₃ nanoparticles were homogeneously distributed in the nugget zone (NZ) at 0.3vol% as compared to 0.2 and 0.4vol% of Al/Al₂O₃ based P-NCs fabricated via FSW which is due to the vigorous stir action, the adequate amount of heat and the mechanical force produced by the FSW tool, that results in a uniform dispersal of RPs in the wide area of the NZ with depth 3286μm.
2. Due to the large volume fraction of Al₂O₃ nanoparticles in the NZ their expansion to volume rate is quite high which leads to an increase in microhardness to 125 HV which is significantly higher than the as-received base material.
3. The tensile properties of the produced AA6061-T6/Al₂O₃ based P-NCs were increased with the increase in the maximum 0.3vol% of Al₂O₃ nanoparticles.
4. Wear resistance was increased for 0.3vol% based P-NCs as compared to 0.2 and 0.4vol% due to the reduction in frictional coefficient provided that Al₂O₃ nanoparticles were distributed uniformly within the aluminum matrix.
5. The formation of wear eroded particles is high at 0.4vol% in comparison to 0.2 and 0.3vol% for Al-Al₂O₃ based P-NCs.

Disclosure statement

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