

Dinesh Kumar\*, Satnam Singh

NIT Kurukshetra, Department of Mechanical Engineering, India  
\*Corresponding author: E-mail: dinesh\_61900120@nitkkr.ac.in

Received (Otrzymano) 12.10.2023

## EFFECT OF GRAPHITE NANOPARTICLES ON MECHANICAL PROPERTIES OF FSPed ALUMINUM SURFACE COMPOSITE

<https://doi.org/10.62753/ctp.2024.05.1.1>

Friction stir processing (FSP) is a manufacturing technique that can be employed to produce aluminum 6082 surface composites (ASCs). These ASCs display considerable increases in hardness and tensile strength, which makes them ideal for a wide variety of automotive applications. One example is piston skirts that are used in the cylinder chamber. The primary emphasis of this research is to investigate the accumulative impact that several passes have on Al 6082 surface composites that were filled with graphite nanopowder. The mechanical properties and microstructure of the fabricated composites were studied in order to accomplish this goal. The microstructural investigation showed that the graphite nanopowder particles were evenly distributed throughout the Al-6082 alloy. In addition, better dispersion of the graphite nanopowder was seen throughout the matrix material as the number of passes made during friction stir processing was increased. This may be explained by the reduction in grain size that occurs inside the aluminum metal matrix composites (AMMCs) that are produced as a consequence. According to the results of the research, the microhardness of the material grew to 105.3 HV after the third pass of the tool, and its maximum tensile strength rose to 215±3 MPa. In the ASCs that fabricated after three passes of friction stir processing, the smallest grain size that was measured was 24 micrometers.

**Keywords:** Al 6082, graphite nanopowder, FSP, microhardness, tensile strength

### INTRODUCTION

Surface composites made of aluminum are used extensively in a diverse selection of applications. The manufacture of composites often makes use of procedures such as stir casting and infiltration, both of which belong to the category of liquid-state processing techniques. The FSP method that was used produced the desired outcome of enhancing mechanical properties without introducing unwanted intermetallic phases [1]. However, in order to overcome these drawbacks such as the formation of undesirable intermetallic phases, solid-state processing was used known as friction stir processing (FSP). When compared to the base material, the ASCs created with FSP exhibit improved mechanical qualities as a result of the intermetallic phases being altered by FSP [2].

Friction is generated between the FSP tool and the workpiece during this processing method, which softens the starting material (in this case, Al 6082). In addition, the plasticity brought about by the tool rotation and the transverse speed facilitates movement of the material from the advancing side to the retreating side [3]. There are three zones, as shown in Figure 1, resulting from the aforementioned factors: the stir zone (SZ), the thermo-mechanically affected zone (TMAZ), and the heat affected zone (HAZ) [4]. Because the microstructure is improved as a consequence of using ceramic reinforce-

ments in ASCs, the mechanical properties of the surface composite material, including its strength, hardness, and elasticity, are all improved as well. The body of research that is now available indicates how different reinforcements, such as B<sub>4</sub>C, TiC, Al<sub>2</sub>O<sub>3</sub>, SiC, TiB<sub>2</sub>, ZrB<sub>2</sub>, and TiO<sub>2</sub>, may be added to the matrix material (aluminum alloy) in order to attain the required properties of the composites that are created [5]. The research carried out by Bourkhani et al. investigated the usage of Al<sub>2</sub>O<sub>3</sub> particles as a reinforcing material in an AA1050 aluminum alloy matrix via the friction stir processing method [6].

The authors observed at the interaction between the FSP tool pin and the Al<sub>2</sub>O<sub>3</sub> particles. The researcher noted clustering of the reinforcing particles during the first pass; nevertheless, further observations revealed enhancement in the mechanical properties and wear behavior during the second pass. During the second pass in FSP, the consistency of the microstructure provides further evidence to support even dispersion of the reinforcement and matrix material. Hardness and tensile strength are important parameters that play a role in determining the degree to which the properties increase in the surface composites fabricated by FSP [6]. In their work, Rathee et al. [7] compared the efficacy of the grooving method employed in FSP compared to the approach of filling a pre-prepared groove with reinforcement.

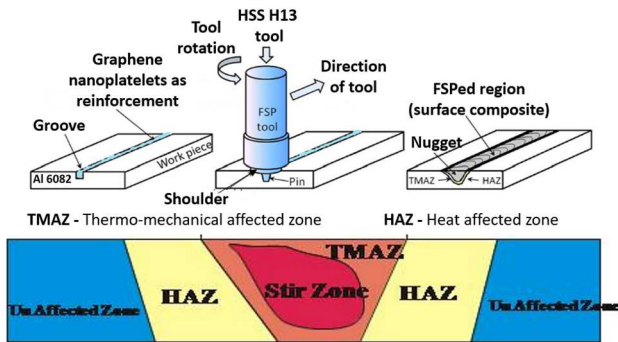


Fig. 1. Different zones in FSP

The researchers came to the conclusion that in comparison to the other methods such as stir-casting, infiltration etc., the grooving method produced much better outcomes. The use of a tool offset from the center towards the workpiece's retreating side allowed the fabrication of a surface composite that was devoid of any defects. An offset of 1.5 mm, which is equal to half the radius of the cylindrical tool pin, was found to be ideal in order to obtain the desired results [7]. Evidence of the phenomenon known as grain strengthening was presented by Khodabaksh et al. in their work on the fabrication of AA5052/graphite composites [8]. In their research, Patil et al. developed an FSP surface composite fabricated from Al 7075 with TiC and graphite serving as the reinforcing components. The EDS examination confirmed the presence of graphite and titanium carbide (TiC) particles inside the surface composite. Homogeneous distribution of the reinforcement in the produced composites was further supported by investigations conducted using field emission scanning electron microscopy (FESEM) [9]. Abushanab and Moustafa conducted a study in which they used the FSP approach to fabricate a surface composite of Al 6082/Al<sub>2</sub>O<sub>3</sub>. The researchers found that dynamic recrystallization and frictional heat may be generated in substantial amounts, especially after the third pass. In addition to that, the result of this technique was the creation of equiaxial grains with fine shapes [10]. Kandasamy et al. conducted a study in which they used the FSP technique to produce a surface composite of Al 7075 with SiC particle reinforcement. The FSP surface composite exhibited a notable increase of 18% in hardness and 24% in tensile strength, respectively. The SEM study of the FSP surface composites revealed the presence of uniformly distributed reinforcing particles and a significant reduction in grain size, indicating essential grain refinement [11].

Kavery et al. used the FSP approach to fabricate a composite material consisting of an AA-6082 matrix reinforced with TiC particles. Their findings indicate that the incorporation of the TiC reinforcement significantly enhances the hardness properties of the composite material. The hardness value of an aluminum composite reinforced with Mg<sub>2</sub>Si is enhanced by consecutive FSP passes, leading to a reduction in porosity

and improved molecular continuity [12]. Meanwhile, increasing the number of FSP passes results in a more uniform dispersion of the matrix and reinforcing particles, which in turn improves the mechanical properties of composites [13]. According to the research by Khan et al., the hardness of the AA-5083 alloy was improved by incorporating TiB<sub>2</sub> particles into the surface layer by the FSP method [14]. Hardness and wear resistance testing was conducted on the A380-SiC surface composite produced by means of the FSP technique. Maryam and Subhi [15] determined that the optimal values of hardness (177 HV) were seen at the tool rotation speed of 1460 rpm during surface composite production by FSP. Additionally, they reported that the wear rate was  $4 \times 10^{-8}$  gm/cm under a load of 15 N.

To the best of the authors' knowledge, there is little research on the fabrication of Al 6082/graphite nanopowder surface composites utilizing the FSP approach. The Al 6082 alloy is considered to possess excellent specific strength and reduced weight characteristics [16-19]. This work aims to investigate the production of Al 6082/graphite nanopowder surface composites (SCs) by comparing several properties in relation to the number of performed FSP passes, specifically for the piston skirt material in engine cylinders for automotive applications. The purpose of the current research is to manufacture aluminum surface composites (ASCs) that are reinforced with graphite nanoplatelets in order to improve the mechanical properties of the composites [20]. Piston skirts are an essential component in the automotive industry, and one of the goals of this project was to develop a surface composite made of Al 6082 that could serve as a suitable replacement for the material that had previously been used in production [21]. It is essential that the manufactured composites that are devoid of any flaws in order to attain high tensile strength and a high level of hardness [22].

## METHODS AND MATERIALS

In the current study, friction stir processing was used to create an aluminum surface composite. Plates made of the Al 6082 alloy, with the dimensions 120 mm x 50 mm x 6 mm, were used as the base to produce the ASCs.

In order to obtain the desired level of strength, graphite nanoplatelet powder was used, purchased from Mech-Tech Automation Systems, New Delhi. A scanning electron microscopy micrograph and EDS analysis results of the base alloy (Fig. 2a and c) as well as an SEM micrograph and XRD analysis results of the graphite nanopowder (Fig. 2b and d) are shown. The graphite nanopowder average particle size of 10  $\mu$ m was a deliberate choice when determining these parameters such as materials properties and processing conditions. In order to eliminate any moisture that may have been present in the reinforcement, an additional process of preheating the graphite nanopowder at the temperature of 300 °C was carried out.

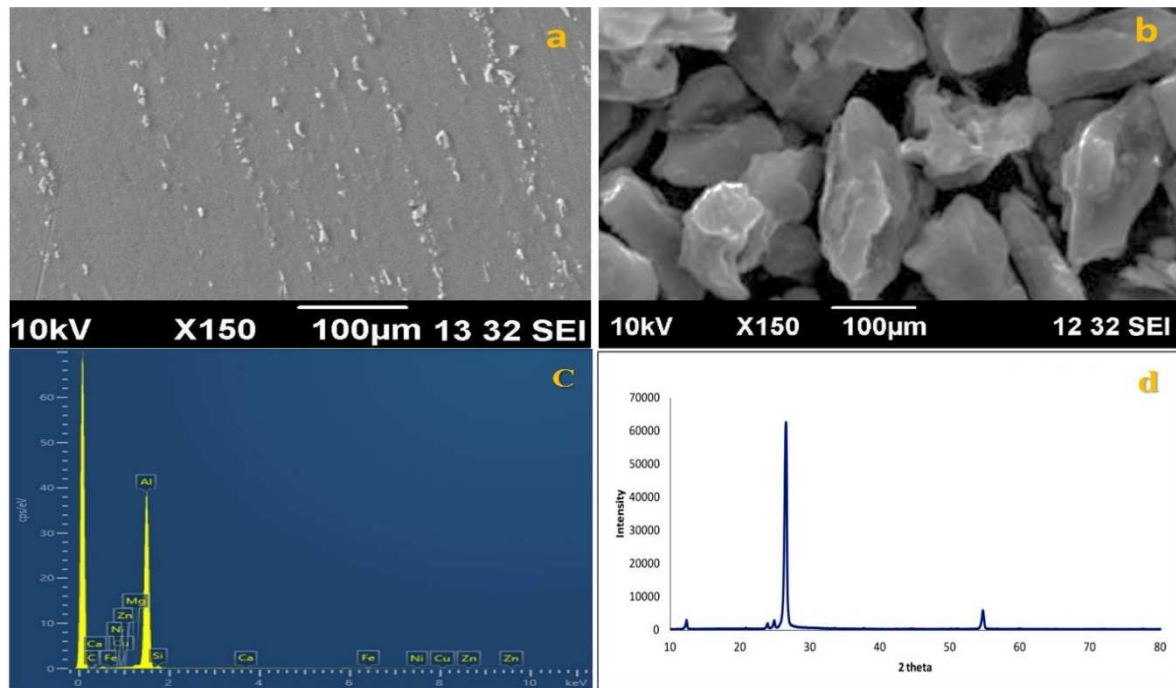


Fig. 2. SEM micrographs of: a) Al 6082 alloy and b) graphite nanopowder; c) EDS analysis of Al 6082 alloy and d) XRD analysis of graphite nanopowder

A square-shaped groove measuring 3 mm on each side and exactly aligned with the direction of FSP tool movement was cut in the plate made of the Al 6082 alloy. An orbital vertical milling machine with a loading capacity of 10 tones was used for this process.

The HSS (H13) tool used to produce the ASCs was cylindrical in shape and had the following dimensions: shoulder diameter of 30 mm, height of 5.8 mm, and a shoulder pin diameter of 4 mm. The reinforcing material was introduced into the groove, and then the groove was sealed using a tool without a pin. This was done with the purpose of preventing the graphite powder from escaping from the groove and being dispersed. In order to successfully create the ASCs specimens, the process parameters had to be meticulously selected based on a pilot run. These specifications include a rotational speed of 1500 rpm, transverse speed of 45 mm/min, penetration depth of 1.5 mm, and a tilt angle of 0.5 degrees. The selection of these particular values was done with the intention of ensuring that the produced surfaces would be completely free of any imperfections. A schematical diagram of the sequential steps involved in the fabrication of the ASCs can be seen in Figure 3.

The number of passes was between one and three. Nevertheless, after the third run, it was found that no considerable defects were produced, leading to an enhancement in the mechanical qualities of the ASCs. This improvement resulted from the third pass of FSP. The ASCs that were subjected to one, two, or three passes of the tool were given the corresponding designations: single pass (SP FSPed) ASCs, double pass (DP FSPed) ASCs, and triple pass (TP FSPed) ASCs, respectively. For the purpose of making a comparison,

the FSPed (Al 6082) specimen does not have any reinforcing particles. The experimental setup for the production of the FSPed SCs is shown in Figure 3.

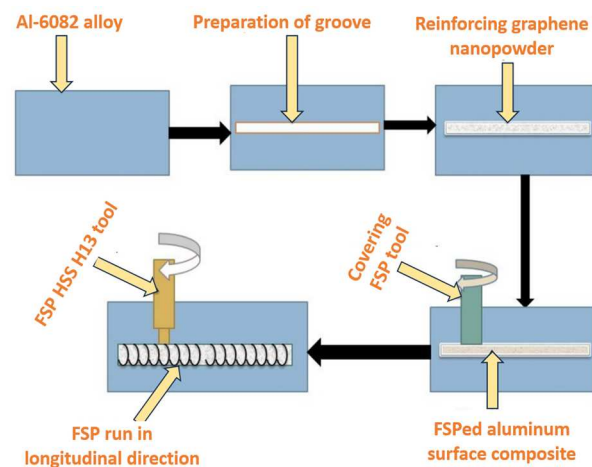


Fig. 3. Schematical diagram of steps in FSP

A groove 4 mm wide was produced as a result of the machining operation. As can be seen in Figure 3, this groove was made parallel to the direction that the FSP path travels. After the groove was made in the specimen, it went through a cleaning procedure that included acetone being applied to it. The groove was then polished using varied grit levels in order to remove any imperfections. In order to achieve a shiny finish which necessitates a high level of surface smoothness, a powerful reagent was applied to it.

DeXel metallography imaging software (Version SIGMA, UK) was used in order to examine the grain size of the dispersed particles present in the FSPed sur-



face composites (SCs). The ASTM E03-01 standard was used in order to conduct microstructural analysis of the Al 6082 alloy and the FSPed surface composites. A JOEL scanning electron microscope (SEM) was employed in order to investigate the morphology of the base alloy as well as the FSPed surface composites. The Vickers hardness tests were carried out by means of a Vickers hardness tester that had a precision of 0.01 mm. The applied load was 200 gf and the dwell length was 15 seconds. In order to determine the average value of the material hardness, three separate measurements were recorded as required by the ASTM E92 standard. The principles described in the ASTM E8 standard were used to conduct tensile strength testing of the specimens, each of which had the dimensions 75 mm x 10 mm x 5 mm. Tensile testing was carried out on a FASNE Universal Testing Machine. Determination of the tensile strength of the base alloy and the FSPed surface composites was the average of three tests on each type of specimen. Figure 4 presents a graphic representation of the specimens.

TABLE 1. FSP parameters

Sample	Rotation speed [rpm]	Transverse speed [mm/min]	Tilt angle (degree)
SP FSPed ASCs	1500	45	0.5
DP FSPed ASCs			
TP FSPed ASCs			

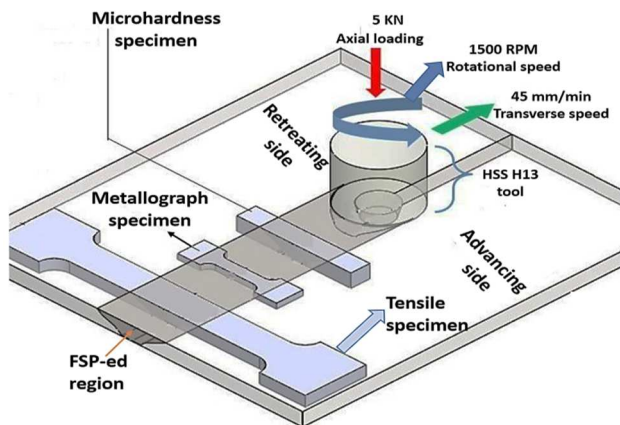


Fig. 4. Specimens for conducted tests

## RESULTS AND DISCUSSION

### Evolution of morphology

Identification of the graphite nanopowder particles inside the matrix was conducted using SEM, as seen in Figure 5. Figure 5a presents the results of an SEM investigation of the Al 6082 alloy after undergoing FSP. Figure 5a shows cracks, scratches, and dents that reduced the mechanical properties of the FSPed material. Figure 5b shows particle clustering on the surface layer of the SP FSPed SC. Clustering also lowers the mechanical properties of the surface composite. Suresh

Kumar et al. found similar behavior in a single pass FSPed Al6082/Si3N4/Cu alloy. This is due to insufficient frictional heat and plastic deformation after one tool pass [22]. A second pass resulted in a better interface between the base alloy Al 6082 and / the graphite nanoplatelets. Figure 5c shows nucleation and decreased particle agglomeration from the improved contact. Figure 5d presents the surface composite morphology after the third tool pass. This figure shows a flat surface with homogeneous reinforcing particle dispersion in the matrix material. The observed decrease in grain size to 24  $\mu\text{m}$  serves as further evidence supporting the presence of a robust link between the graphite nanoplatelets and the Al 6082 alloy after the third pass.

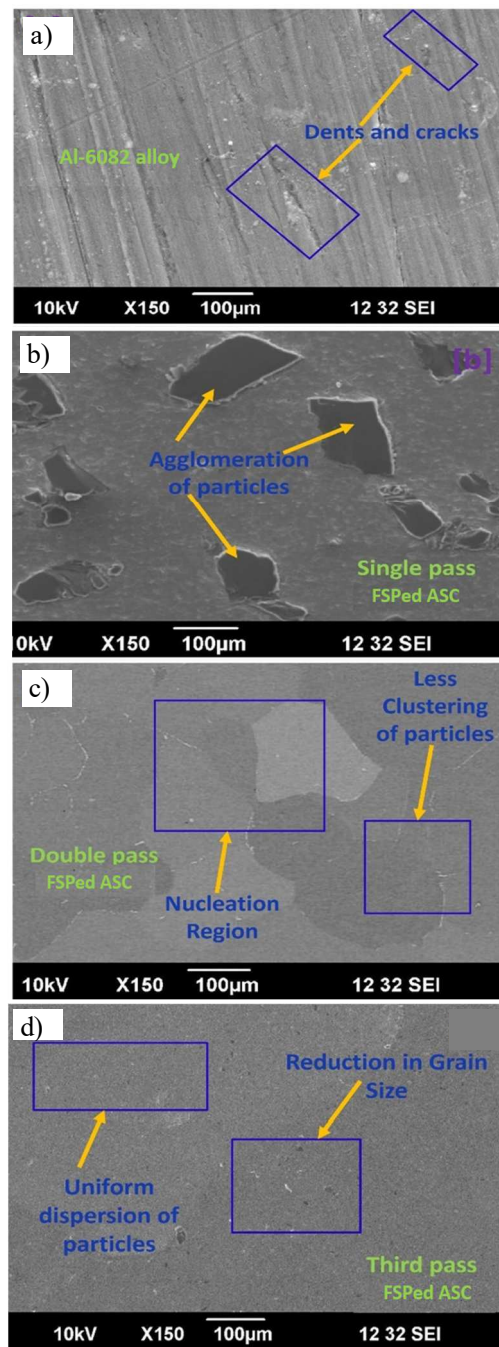


Fig. 5. Comparison of Al6082 and FSPed SC microstructures at different passes (a-d)

## Microhardness measurements

Figure 6 presents the hardness values of the surface composites produced using FSP as well as the base material Al 6082. The initial hardness value of the Al 6082 alloy in its as-received state was measured to be 68.3 HV. However, with the introduction of graphite nanoplatelets, a significant increase in hardness was detected. The improvement in hardness ranged from 25.45% to 62.25% for each consecutive pass of the tool. The surface composite subjected to three passes had the highest recorded hardness value of 105.3 HV. The improvement in hardness seen in the created surface composite following the third pass of the tool may be attributed to the reduction in porosity, less clustering of the reinforcement particles, and smaller mean values of grain size. The increase in hardness of the surface composites produced by FSP may be attributed to many factors. Firstly, the incorporation of graphite nanoplatelets into the Al 6082 alloy contributes to the ability of the resulting surface composite to withstand indentation loads. The three pass FSP produced a surface composite exhibiting enhanced mechanical characteristics. This improvement is attributed to the uniform dispersion of particles inside the created surface composite. Table 2 displays the average values (three repetitions for each test) of the mechanical properties.

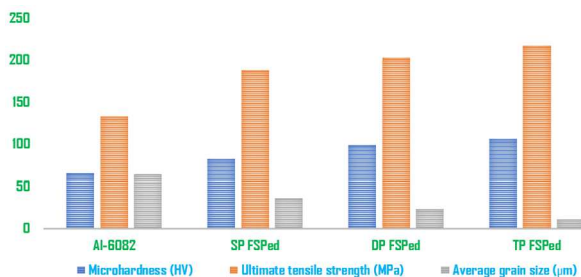


Fig. 6. Mechanical properties of FSPed aluminum surface composites

TABLE 2. Average values of mechanical properties

Specimen	Mean grain size [μm]	Microhardness [HV]	Tensile strength [MPa]
Al-6082 alloy	96	68.3	134 ± 3
SP FSPed	65	83.4	187 ± 2
DP FSPed	35	97.8	204 ± 5
TP FSPed	24	105.3	215 ± 3

## Measurement of tensile strength

In order to ascertain the tensile strength of the surface composite manufactured using FSP, the specimens were tested under ambient conditions. The tensile strength was evaluated by maintaining the strain rate at 5 mm/s, which is significant because the testing conditions are designed to simulate or represent real-world scenarios where materials experience a specific rate of force or strain. The tensile test is used in different commercial applications such as automotive, aerospace, and marine industries.

Table 2 presents the tensile strength results of the specimens, which were subjected to varying numbers of passes during processing and included reinforcing particles. The specimens subjected to FSP exhibit a higher level of tensile strength in comparison to the base material. Furthermore, it can be observed that the tensile strength is influenced by the number of passes. Based on the graphical representation, it can be inferred that there is a positive correlation between the tensile strength and the number of passes. The surface composite specimens subjected to three passes exhibited the highest tensile strength of  $215 \pm 3$ . The following factors may contribute to the observed increase in tensile strength: (i) the uniformity of graphite particles in the surface composites produced by friction stir-processing; (ii) the use of graphite particles also augments the material toughness; (iii) the decrease in the size of the grains and the formation of a robust interfacial connection between the matrix and reinforcement particles. The aforementioned favorable features contribute to the improvement in the mechanical properties of the surface composite produced using FSP technology.

## Fracture analysis of tensile test specimens

As can be seen from the fractured tensile specimens, the Al 6082 specimen (Fig. 7a) underwent ductile fracture, which ultimately resulted in ductile failure. The SP FSPed ASCs (Fig. 7b) are characterized by a large number of dimples, which serve as an indicator of the failure mode. It was also noticed that the specimen has a coarse average depression dimension. The fractured sample of the DP FSPed ASCs is the result of void nucleation followed by coalescence, as seen in Figure 7c. This is the phenomenon that caused the fractures. Within the group of ASCs that were subjected to DP FSP, it was noticed that the size of the dimples and the dominance of the cleavage face decreased.

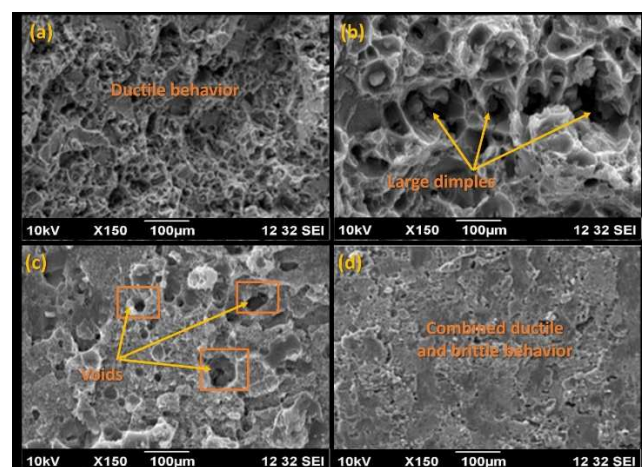


Fig. 7. Fractured tensile test specimens: a) Al 6082 s, b) SP FSPed ASC, c) DP FSPed ASC and d) TP FSP-ed ASC

Grain refinement and work hardening were the factors that led to the combined brittle and ductile fracture of the TP FSP-ed ASCs, as seen in Figure 7d.

## CONCLUSIONS

The current study investigated the hardness, tensile strength, grain size, and morphology in Al 6082 alloy surface composites reinforced with graphite nanoplatelets, which were produced utilizing the friction stir processing method. The primary conclusions are as follows:

1. Al 6082/graphite nanopowder surface composites were successfully fabricated using friction stir processing.
2. The TP FSPed surface composites exhibit the highest hardness of 105.3 HV and a tensile strength of  $215 \pm 3$  MPa. These improvements may be attributed to a decrease in the occurrence of dents, scratches, and fractures.
3. After three passes of friction stir processing, the grain size decreased by 20%, which results in the strong interfacial bonding between the reinforcement and matrix materials.
4. Graphite nanoplatelets were found throughout the Al 6082 alloy, as shown by the microstructural investigation. Furthermore, aluminum metal matrix composites (AMMCs) were formed when the number of passes performed by the FSP tool is increased.

## REFERENCES

- [1] Raja R., Jannet S., Rajesh Ruban S., George L., Mechanical, wear, and microstructural examination of copper surface composites reinforced with SiC nanoparticles done by FSP, *Materials Today: Proceedings* 2023, 92, 1, 376-381, DOI: 10.1016/j.mater.2023.05.301.
- [2] Kumar S., Kumar A., Vanitha C., Corrosion behaviour of Al 7075/TiC composites processed through friction stir processing, *Materials Today: Proceedings* 2019, 15, 21-29, DOI: 10.1016/j.matpr.2019.05.019.
- [3] Kumar D., Angra S., Singh S., High-temperature dry sliding wear behavior of hybrid aluminum composite reinforced with ceria and graphene nanoparticles, *Engineering Failure Analysis* 2023, 151, May, 107426, DOI: 10.1016/j.engfailanal.2023.107426.
- [4] Khodabakhshi F., Nosko M., Gerlich A.P., Effects of graphene nano-platelets (GNPs) on the microstructural characteristics and textural development of an Al-Mg alloy during friction-stir processing, *Surface and Coatings Technology* 2018, 335, 288-305, DOI: 10.1016/j.surfcoat.2017.12.045.
- [5] Jeon Ch-H. et al., Material properties of graphene / aluminum metal matrix composites fabricated by friction stir processing, *International Journal of Precision Engineering and Manufacturing* 2014, 15, 6, 1235-1239, DOI: 10.1007/s12541-014-0462-2.
- [6] Bourkhani R.D., Eivani A.R., Nateghi H.R., Through-thickness inhomogeneity in microstructure and tensile properties and tribological performance of friction stir processed AA1050-Al<sub>2</sub>O<sub>3</sub> nanocomposite, *Composites Part B* 2019, 174, June, 107061, DOI: 10.1016/j.compositesb.2019.107061.
- [7] Rathee S., Maheshwari S., Sidoliquee A.N., Srivastava M., Investigating the effects of SiC particle sizes on microstructural and mechanical properties of AA5059 / SiC surface composites during multi-pass FSP, *Silicon* 2019, 11, 797-805.
- [8] Khodabakhshi F., Gerlich A.P., Švec P., Fabrication of a high strength ultra-fine grained Al-Mg-SiC nanocomposite by multi-step friction-stir processing, *Materials Science and Engineering A* 2017, 698, 313-325, DOI: 10.1016/j.msea.2017.05.065.
- [9] Patil V., Janawade S., Kulkarni S.N., Biradar A., Studies on mechanical behavior and morphology of alumina fibers reinforced with aluminium-4.5% copper alloy metal matrix composites, *Materials Today: Proceedings* 2021, 46, 1, 99-106, DOI: 10.1016/j.matpr.2020.06.176.
- [10] Abushanab W.S., Moustafa E.B., Effects of friction stir processing parameters on the wear resistance and mechanical properties of fabricated metal matrix nanocomposites (MMNCs) surface, *Integrative Medicine Research* 2020, 9, 4, 7460-7471, DOI: 10.1016/j.jmrt.2020.04.073.
- [11] Kandasamy S., Rathinasamy P., Nagarajan N., Arumugam K., Rathansamy R., Kaliyannan G.V., Corrosion behavioral studies on AA7075 surface hybrid composites tailored through friction stir processing, *Anti-Corrosion Methods and Materials* 2020, 4, March, 345-355, DOI: 10.1108/ACMM-11-2019-2215.
- [12] Aravindan M., Balamurugan K., Tribological and corrosion behaviour of Al 6063 / SiC metal matrix composites, *Materials Science, Engineering* 2016, 2-7.
- [13] Vignesh R.V., Padmanaban R., Govindaraju M., Suganya G., Investigations on the corrosion behaviour and biocompatibility of magnesium alloy surface composites AZ91D-ZrO<sub>2</sub> fabricated by friction stir processing, *Transactions of the IMF – The International Journal of Surface Engineering and Coatings* 2019, 97, 5, 2967, DOI: 10.1080/00202967.2019.1648005.
- [14] Khan M.M., Dey A., Hajam M.I., Experimental investigation and optimization of dry sliding wear test parameters of aluminum based composites, *Silicon* 2022, 4009-4026.
- [15] Mohammed M.H., Subhi A.D., Exploring the influence of process parameters on the properties of SiC/A380 Al alloy surface composite fabricated by friction stir processing, *Engineering Science and Technology, an International Journal* 2021, 24, 5, 1272-1280, DOI: 10.1016/j.jestch.2021.02.013.
- [16] Sharma V.K., Rana S.K., Lal R., Rana R., Wear and residual stress analysis of waste sea shell and b4c particles reinforced green hybrid aluminium metal composite, *Journal of Engineering Research* 2021, 215-224, DOI: 10.36909/jer.ICARI.15317.
- [17] Samal P., Kumar R., Pandu M., Dry sliding wear behavior of Al 6082 metal matrix composites reinforced with red mud particles, *SN Applied Sciences* 2020, 2, 2, 1-11, DOI: 10.1007/s42452-020-2136-2.
- [18] Jesudoss N.R., Sankaranarayanan H.R., Catalin R.T., Pruncu I., Dispinar D., A comparative study of the mechanical and tribological behaviours of different aluminium matrix – ceramic composites, *Journal of the Brazilian Society of Mechanical Sciences and Engineering* 2019, 41, 8, 1-12, DOI: 10.1007/s40430-019-1831-7.
- [19] Sathish T., Chandramohan D., Vijayan V., Sebastian P.J., Investigation on microstructural and mechanical properties of Cu reinforced with SiC composites prepared by microwave sintering process, *Materials Science, Engineering* 2019, 009, 5-9.
- [20] Kumar H., Kumar V., Kumar D., Singh S., Wear behavior of friction stir processed copper-cerium oxide surface composites, *Evergreen – Joint Journal of Novel Carbon Resource Sciences & Green Asia Strategy* 2023, 10, 01, 78-84.
- [21] Kumar D., Singh S., Angra S., Dry sliding wear and microstructural behavior of stir-cast Al6061-based composite reinforced with cerium oxide and graphene nanoplatelets, *Wear* 2023, 516-517, 204615, DOI: 10.1016/j.wear.2022.204615.
- [22] Sun N., Jones W.J., Apelian D., Friction stir processing of aluminum alloy A206: Part II – Tensile and Fatigue Properties, *International Journal of Metalcasting* 2019, 13, 2, 244, DOI: 10.1007/s40962-018-0268-6.