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CHARACTERIZATION OF EN-AW-6061-O–GRAPHITE NANOFLAKE COMPOSITES FOR ENHANCED MECHANICAL PROPERTIES USING MULTI-PASS FRICTION STIR PROCESSING

ABSTRACT

In this study, graphite (Gr) nano flakes dispersion with increasing number of passes (1, 2, and 3) inside the resultant aluminium ENAW-6061-O-Gr composites using Friction Stir Processing (FSP) has been accomplished successfully. The objectives were to embed the Gr nanoflakes inside ENAW-6061-O-Gr composites, investigate the effect of number of FSP passes on the mechanical properties of aluminium 6061/graphite composites. The ENAW-6061-O-Gr composite samples were evaluated with tensile tests and elemental analysis through SEM with EDX and mapping. The dispersion and presence of graphite particles is confirmed. Multi-pass FSP improved the tensile strength of the ENAW-6061-O-Gr composites. The UTS of C3-Composite processed with three passes is 153.65 MPa, that is 24% of improvement compared to the unreinforced aluminium ENAW-6061-O base metal alloy.

Keywords: *Friction Stir Processing; composites; aluminum; graphite*

INTRODUCTION

In the recent years, aluminium has been increasingly used for weight reduction because of their low density. The density of aluminium is reported about 2.7 g/cm³ which makes it ideal for light weight applications especially in automotive industry [1]. It has been increasingly important to develop lightweight and high-strength materials for automotive components in reducing the fuel usage. Friction stir processing (FSP) is used to alter the microstructural composition of a material, which is an efficient technique for refining grain structures. In FSP, a tool composed of a pin and a shoulder rotated horizontally and plunged the surface materials on the processed zone. Friction generated by the rotating process would soften the material at temperatures below melting [2]. The pin induces severe plastic deformation and static grain growth in the processed zone, allowing for grain refinement on the microstructure. FSP provide advantages toward the base material likes produce grain refinement, fine and homogenous microstructure and densification of participates in the composites. FSP is designed to enhance the surface properties of metal alloys, improve ductility, toughness, hardness and strength, and increase fatigue life without altering the properties of bulk metal [3].

Aluminium matrix composites (AMCs) are new features that have recently been used and are capable of meeting the need for innovation in manufacturing applications. There are several types of reinforcement that have recently been used in FSP studies. The most widely used reinforcement materials are inorganic metallic powders such as silicon carbide, titanium oxide, graphene, iron, and stainless steel, as well as less organic powders such as rice husk, fly ash, palm kernel shell ash, and coconut shell ash [3].

ENAW-6061 is frequently used in the automotive industry because it has good properties for industry use, particularly high strength with low density and high corrosion resistance. However, the extensive use of materials results in the subservience of tribological properties. By employing the proper processing technique, it is possible to refine the surface of the aluminum microstructure by adding reinforcement with homogeneous dispersion. The use of conventional methods to incorporate reinforcement for the production of AMCs is hard to fabricate, lengthy and energy consuming. Liquid metallurgy and powder metallurgy are some of the methods used in manufacturing AMCs. The problem with the powder metallurgy method, which involves pressing and sintering, is that the nanoparticle reinforcements would inhibit densification in matrix composites. In some cases, the nanoparticles aid in the formation of coarse clusters through agglomeration [4].

Production of AMC using FSP show the advantages on reducing these issues. In comparison to powder and liquid metallurgy, FSP promotes good grain refinement of nano particles reinforcement with intense plastic deformation in a solid-state process [5]. However, the uniform distribution of particles is slightly quite difficult to achieve in a single pass, and it was suggested that a multi-pass approach be implemented in FSP to improve the distribution of reinforcement inside composites [6].

FSP is a new method developed by The Welding Institute (TWI) in 1991, adapted from FSW. FSP it is solely used for microstructure modification which the reinforcement is packed into the deposition holes or groove on the matrix material. This method can be expanded to be used in the production of metal matrix composites (MMCs) with superior properties to the base material [7]. Without altering the properties of bulk metals, FSP improves the surface properties, ductility, formability, hardness and strength of metal alloys [8].

FSP is done using the similar method like FSW, in which a rotating tool is passed along the processed zone on the metal plate and this modify the microstructural of the composite. Thus, R. S. Mishra developed aluminium surface MMCs that integrate SiC particles through the use of FSP method to improve the material's properties on the tensile strength and hardness by the adjusting the particles distribution of reinforcement particles in the composites in the pursuit of better material processing [9]. FSP aids in the elimination of casting defects, the prevention of custody failure, and the reduction of material porosity [10]. The use of aluminium alloys, which benefit from its lightweight application in the automotive industry, has been developed with a view to reducing fuel consumption, especially in automotive components such as bearings, automotive pistons, cylinder blocks and drive shafts [11]. Some of the properties shown in aluminium alloys include high specific strength, high corrosion resistance, low density and higher stiffness. By consolidating ceramic particles such as silicon carbide, titanium carbide and carbonous materials into aluminium alloys, this results the composites to have a desirable combination of physical and mechanical properties. The strength and hardness of aluminium alloys has improved dramatically through the synthesis of aluminium composites. Through adding reinforcement particles and altering the volume fraction of aluminium alloys, the proper-ties of the materials that will be used in the manufacture of automotive components needing certain promising mechanical properties would be improved.

In automotive industries, among the most popular alloy used is AA6xxx aluminium alloy series which the main alloying elements include silicon and magnesium. This aluminium alloy series has a high strength to weight ratio and good formability. It is also a low-cost material that is likely to be used in the production of lightweight parts for vehicles. ENAW-6061-O-Gr material is commonly used in the production of AMCs, with the option modifying the composite strength. The addition of reinforcements in AMCs provides the opportunity to improve mechanical properties like tensile strength and hardness achieved through composite production. Aluminum alloys of various types are now commercially available and provide significant benefits due to their exceptional properties.

The presence of graphite with high strength properties and a low-density material that can be easily deformed due to its soft nature graphite provides a lubricant layer that decreases shear stress during manufacturing, which passes to the matrix composites, improving the plastic deformation just below the surface [12]. It is also intended to improve the strength and ductility of aluminium alloys by using graphite as a reinforcement. Moreover, graphite has anti-friction properties for AMCs due to the soft nature of the reinforcement material that ultimately extends its use in automotive parts such as bearings and cylinder blocks. It is possible to enhance the tensile behavior of the composites if the distribution and morphology of graphite is altered to a uniform distribution of fine particles. Due to their exceptional mechanical, thermal, electrical and tribological behavior, graphitic structured materials such as carbon nanotubes (CNTs), graphite and graphene have been among the most widely researched materials [13]. For certain challenging applications especially in automotive industry, the need for high performance and lightweight materials has resulted in comprehensive efforts to develop AMCs. By these advantages, multipass FSP is used for AMC production with graphite as reinforcement.

Plastic deformation is an important factor in producing fine grain microstructures within AMCs. ECAP and FSP are the most popular methods for plastic deformation studied by the researchers in their project. A number of passes, with at least six passes required in ECAP to achieve ultra-fine grain refinement in composites [14]. The thermo-mechanical stirring process done through the tool with heat dissipation which caused intense plastic deformation alters the properties of the composites in the FSP method. As a result of the occurrence of dynamic recrystallization, grain refinement occurred. Nakata et. al. reported on an increasing trend in the mechanical properties of aluminum alloys when multi-pass FSP was used [15]. Johannes and Mishra investigated the AA7075 microstructure properties using a single pass and four passes with a constant FSP parameter in another study. When compared to the four passes, the single pass on AA7075 exhibits less superplasticity [16]. As a result, the focus of this research is on the impact of using multiple-pass FSP to improve the mechanical properties and microstructure characterization of ENAW-6061-Gr composites. The various reinforcement strategies like boron nitride, vanadium carbide [17], titanium oxide, silicon carbide, zirconium oxide [18], Ti_3AlC_2 [19], ZrB_2 [20] for the improvement of the tribological, mechanical properties of the aluminium 6061.

For this research, ENAW-6061-O plates are used as a base material along with graphite as the reinforcement for the development of AMC using multi-pass FSP method. When compared to unreinforced metals, AMC has demonstrated the ability to increase mechanical properties such as tensile strength and microhardness, and eventually provide ways to provide better material properties. Graphite is known to be one of the commonly used reinforced materials. Multi-pass FSP method is implemented to further improve the dispersion of the reinforcement inside the surface composites. The microstructure and tensile strength of FSP samples will be observed and evaluated by changing the number of passes.

MATERIALS AND METHODS

The purpose of this study is to conduct the experimentation and testing of specimens taken from FSP composites within the scope of the study. This research focuses on the effect of number of passes on the mechanical properties and the microstructure characterization focusing on distribution of graphite particles of ENAW-6061-O-Gr metal matrix compo-sites produced through FSP method. The base material to be used is ENAW-6061-O in the annealed condition and under-goes FSP process on its surface with the addition of graphite as reinforcement. The base alloy has been procured from Nova scientific Sdn. Bhd. The percentage volume of the graphite powder, the tool tilt angle and plunge depth are kept constant. The independent variable is only the number of passes. The tensile test will be performed to assess the tensile strength. Microstructural investigations of ENAW-6061-O-Gr composite samples will be performed using the SEM and FESEM with EDX together with mapping analysis to examine the distribution of graphite particle.

Materials

ENAW-6061-O alloy plates used are of dimensions- nominal thickness of 6.35 mm, width-100 mm and length 150 mm. The nominal composition of the ENAW-6061-O alloy is mentioned in Table 1. Nano graphite powder is used as the reinforcement to disperse in the compo-sites. The thickness of the graphite flakes is less than 40 nm and flakes diameters in the range of 3-6 μm .

Table 1. Chemical composition of ENAW-6061-O

Elements	Mg	Mn	Fe	Cu	Si	Al
Percentage (mass fraction, %)	0.80	0.07	0.40	0.20	0.54	Balance

The linear blind holes pattern is used for the deposition of graphite powder. Each plate is made with 10 holes along the centerline, with dimension (2 mm diameter, 4 mm depth). The 5 mm space is maintained between holes as shown in Figure 1.

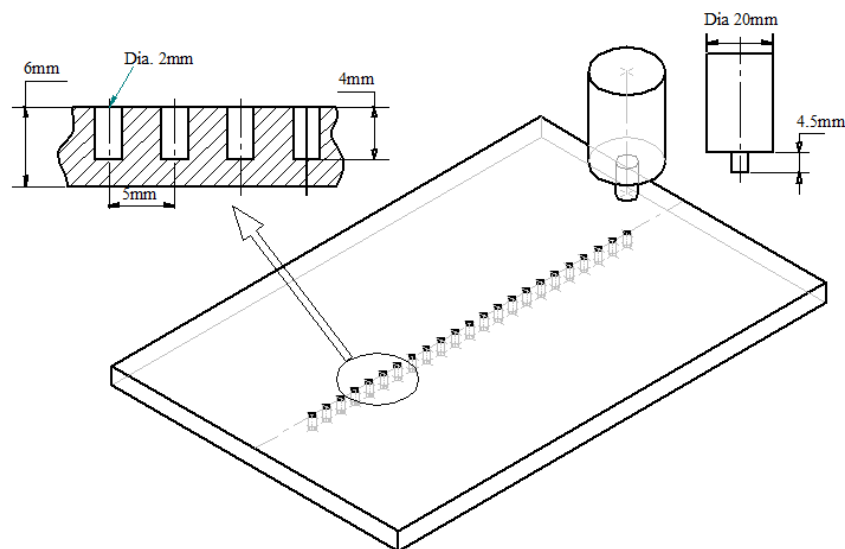


Fig. 1. Linear blind holes arrangement schematics

Graphite nano flakes are packed into the deposition holes on the ENAW-6061-O alloy plate. Graphite nano flakes are pressed using manual compaction to make it filled properly in the deposition holes. The FSW machine (Model-FSW-TS-F16, CHINA FSW CENTRE) is used to perform the FSP as shown in Figure 2. Pin-less H-13 steel tool is mounted to make the capping pass. The capping pass completely closes the surface and guarantees no loss of graphite powder during the next FSP stirring action. Before the start of FSP process, the ENAW-6061-O must be tightly clamped with the holding plates.

FSP pin made of H-13 steel tool type is used to dispersed the graphite into the work-piece surface at a given translation speed. In each sample different number of passes were used in the processing. The composite C1 is processed with one pass FSP, C2 with two and C3 with three FSP passes, respectively. The multi-pass FSP was completed without changing the direction, allowing the advancing and retreating sides of the process zone to remain in place. Figure 3 shows the successful fabrication of ENAW-6061-O-Gr Composites using multi-pass FSP. Surface defects were not observed on the surface composites.



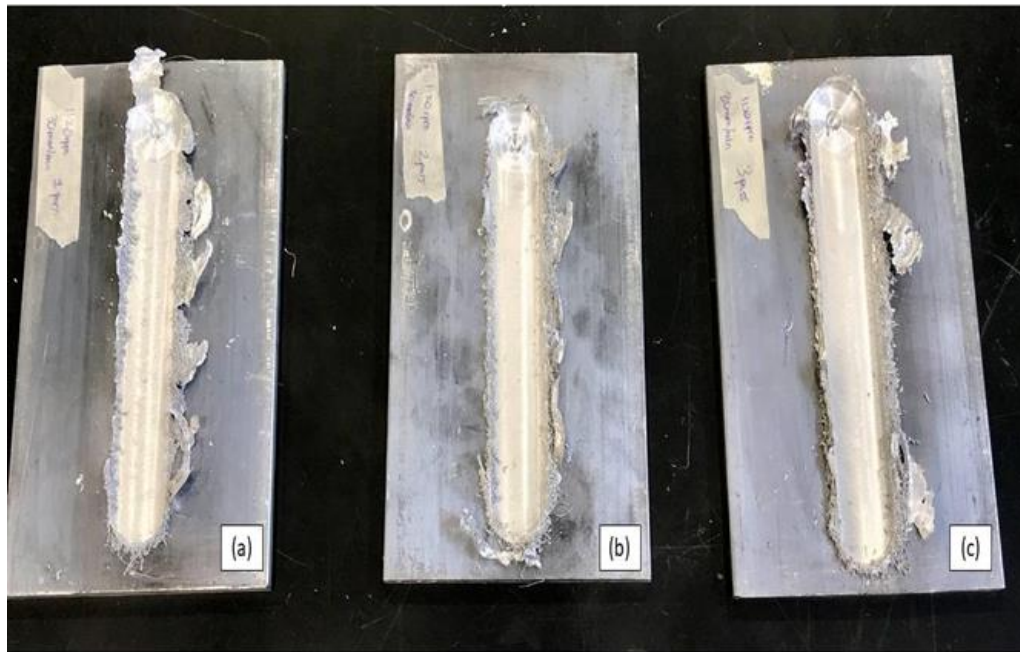
Fig. 2. Friction stir welding machine (FSW-TS-F16 model)

Table 2. Process parameters

Parameter	Value
Rotational Speed	1120 rpm
Transverse Speed	30 mm/min
Pass	C1-1 pass, C2- 2 passes, and C3- 3 passes
Tool tilt Angle	2.5°
Tool plunge depth	5.3 mm

Table 3. Tool pin profile

Parameter	Value
Tool Pin Profile	Straight cylinder
Pin Length	4.5 mm
Pin Diameter	6 mm
Shoulder Diameter	20 mm

**Fig. 3.** Composites after multi-pass FSP for (a) one pass (b) two passes (c) three passes

The FSP samples are then cut using the wire electric discharge machine (EDM) to obtain a tensile and microstructural characterization sample. The ASTM E8-M standard is used in the preparation of sub size samples for the tensile test as represented in Figure 4 (a) and (b). The tensile specimen was cut across the processed zone with gauge length of 25 mm and width of 6 mm. The rectangular shape samples that were cut with 20 mm x 10 mm dimension for microstructural analysis as shown in Figure 4 (a) and (b). Tensile tests are performed for the prepared tensile samples by using 50 KN universal testing machine (UTM). The tests are conducted at room temperature. The cross-head speed is kept constant of 1 mm/min for each sample's tensile test.

The ENAW-6061-O-Gr composite samples are mounted and then surfaces are grinded, polished and etched for microscopic imaging and elemental analysis. The grinding process conducted over the samples using the SiC grinding papers with different grit sizes to achieve a smooth and flat surface. These steps are required to remove damage during the cutting process which allows the surface to be free of scratches. After that, the polishing is conducted with polycrystalline abrasive diamond solution applied on polishing disks. Finally, the samples will be etched by dipping the samples using the chemical Keller reagent solution (50 ml of water, 10 ml HF, 15 ml HCl and 25 ml of HNO₃). The samples are rinsed with running water and dried for microstructural investigation.

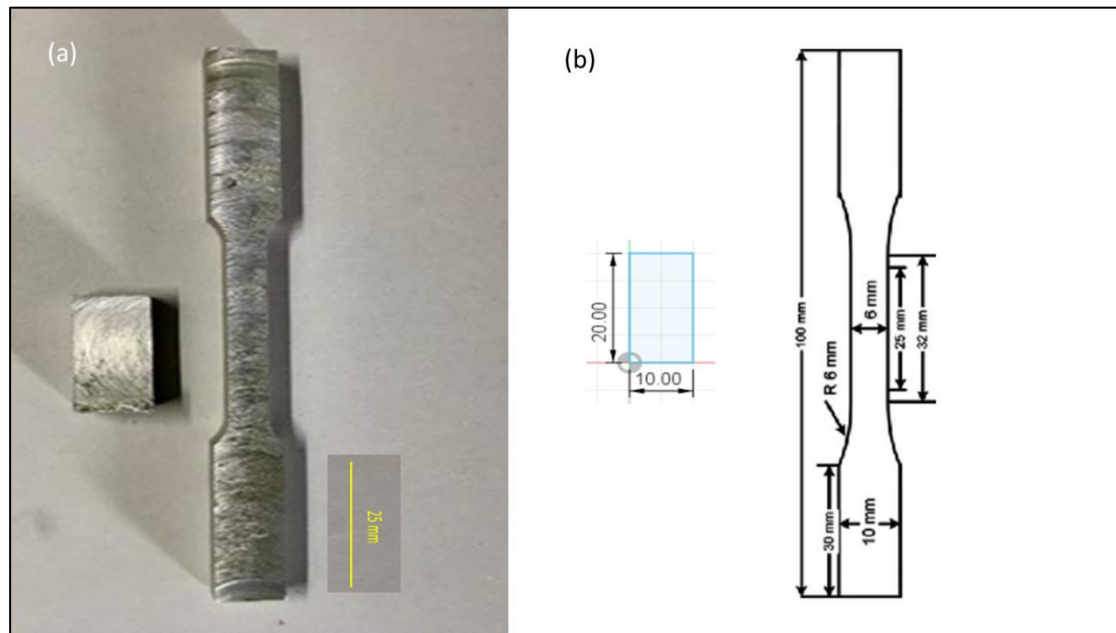


Fig. 4. (a) Specimen cut for microstructural analysis and tensile test as per ASTM E8-M
(b) sketch of samples with dimensions

Microstructure investigation across the processed zone on each FSP specimens were observed using different characterization microscopes, including SEM in Phenom Pro-X model as well as the FESEM-EDX elemental mapping analysis. The magnification used for the characterization on SEM are 10000x and were taken at random spots within the processed zone.

RESULTS AND DISCUSSION

Tensile Test Results

All the specimens failed within the gauge lengths as represented in Figure 5. The engineering stress-strain curves corresponding to the ENAW-6061-O-Gr specimens is shown in Figure 6. The comparative graph of stress vs strain indicates that the C3 ENAW-6061-O-Gr showed highest tensile strength amongst three combinations of the composites.

The tensile tests were conducted for each composite three times and average value is mentioned in the Table 4. C1 composite specimen resulted in ultimate tensile strength of 113.27 MPa. The specimen elongated 1.21 mm before it fractured and having 4.25 KN peak load. The ultimate tensile strength (UTS) of 137.71 MPa is observed for C2 composite sample. The specimen elongated 1.55 mm before it fractured and having 5.15 KN peak load. This second passes FSP specimen showed improvement in tensile strength than the single pass. For C3 composite processed with three FSP passes, the average ultimate tensile strength of 153.65 MPa is obtained. The specimen elongated 2.02 mm before it fractured and having 5.72 KN peak load. The highest tensile strength is shown in three passes processed composite than other ENAW-6061-O-Gr specimens.

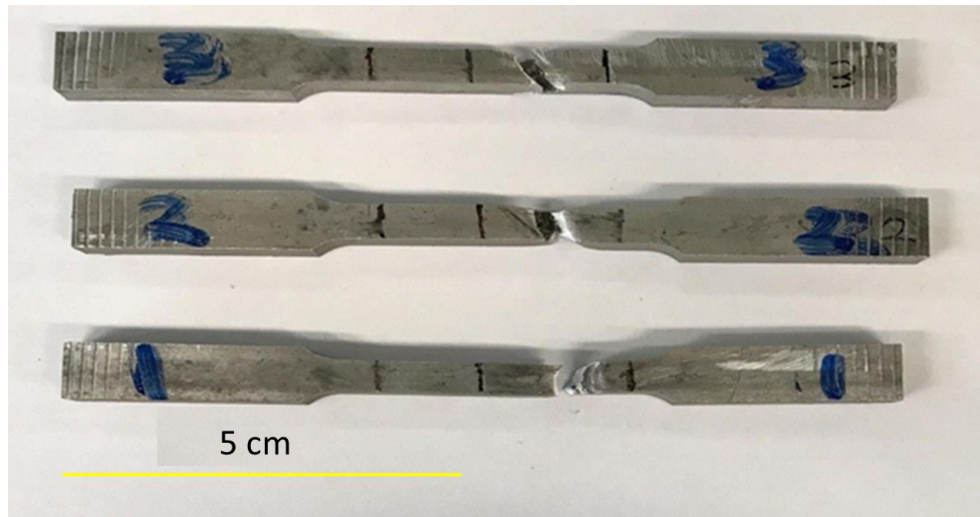


Fig. 5. Fractured tensile test specimens

The results obtained in the investigation are in line with the reported studies [21-23]. The graphite nanoflakes dispersion extent is important factor in deciding the microstructure and tensile properties of the composites. The ultimate strength has enhanced with the use of nano-graphite powder. Increases in ductility and hardness values show that nano graphite flakes can be successfully used as a reinforcement for the aluminum 6061 matrix [21]. The graphene nanoplates were evenly distributed throughout the 6061-aluminum matrix alloy, as demonstrated by the FESEM micrographs, and the yield strength, hardness, and tensile strength significantly increased in comparison to the unreinforced 6061 aluminum alloy [22].

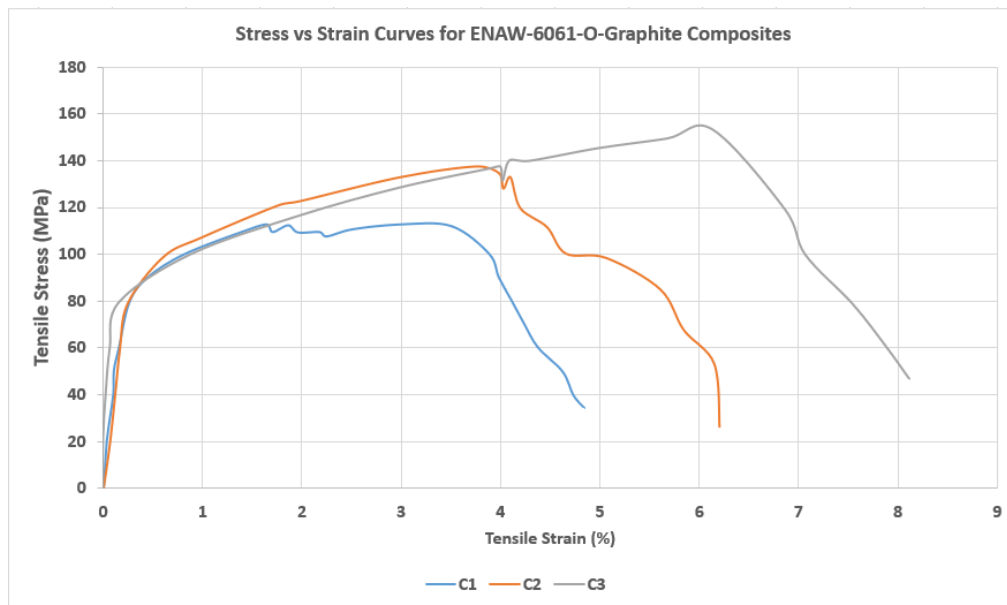


Fig. 6. Stress vs strain curve for C1 composite (One pass)

From Table 4, the tensile test values presented the increase in number of Gr-particle passes as reinforcement resulted to increase of the tensile strength of the FSP specimens. The unreinforced Al base metal i.e. ENAW-6061-O observed to have UTS of 124 MPa and Young's modulus of 90 GPa [23]. The UTS on C3 three passes sample was 153.65 MPa, that is 24% of improvement compared to the unreinforced ENAW-6061-O.

Table 4. Tensile test results

No. of passes	One	Two	Three
Ultimate Tensile Strength (MPa)	113.27	137.71	153.65
Peak Load (kN)	4.257	5.154	5.723
Elongation (mm)	1.21	1.55	2.02

M. F. Soustani et al., [23] discovered a similar trend of improved tensile strength of Al-7Fe-5Ni alloy composite, with the 4-pass FSP sample being higher than the 1-pass FSP sample alloy due to multi-pass. The tensile strength of the multi-pass FSP samples was increased in this study due to a more homogeneous grain structure inside the AMCs and the presence of graphite as reinforcement. The finer grains are formed due to intense plastic deformation occurred in multi-pass FSP process. Referring to Hall-Petch relationship, high strength of metallic materials could be achieved by decreasing the grain size [24].

Microstructure

FESEM electron-dispersive x-ray (EDX) in addition to mapping analysis was done on the specimen. The presence of Gr reinforcements nanoparticles was ensured at the aluminium matrix composite by analyzing the elements through the FESEM-EDX and mapping analysis as presented in Figure 7-9.

The high elemental peak in the EDX spectrums is found to be carbon, magnesium, and silicon. The elemental peaks and the atomic percentage confirm the presence of graphite nano flakes (Carbon-C) in the aluminum matrix composites. The very thin graphite nano flakes layers have spread over the matrix and quite covered the surface area inside the ENAW-6061-O-Gr composites with multi-pass Gr particles. Cluster size decreases as the number of passes increases due to the distribution of the nanosized particles inside the matrix over a larger area [6]. The presence of reinforcement particles in the AMCs facilitated in the microstructure refining that resulted in dynamic recrystallization from the stirring action. The graphite nano flakes reduce the anisotropy of the microstructure when dispersed uniformly inside the resultant composite material [26]. The nanoflakes dispersion extent in case of three pass FSP composite is more improved compared to the single pass and two pass composites. The graphite nano flakes have high thermal conductivity and it makes the composite surface consolidation during the friction stir processing much more challenging. Thus, in this study the major focus was on the well dispersion of content of graphite nanoflakes with increasing number of FSP passes. The exfoliation of graphite layers inside the composites due to superplastic deformation during FSP is reported in other studies [27]. Thus, the extent of graphite nano flakes exfoliation in the C3 sample is more than other two composites due to more processing FSP passes. The plasticized material of the base alloy is taken from the advancing to retreating side while each pass of FSP.

The applied tensile load is distributed to Gr particles across the passed in the AMCs. Further, the homogenous dispersion of ceramic nanoparticles throughout the aluminum matrix provides orowan strengthening [28]. During the fabrication process, severe plastic strain is generated on the matrix and reinforcement. In contrast to the conventional method of producing AMCs, FSP promotes high plastic strain development [29].

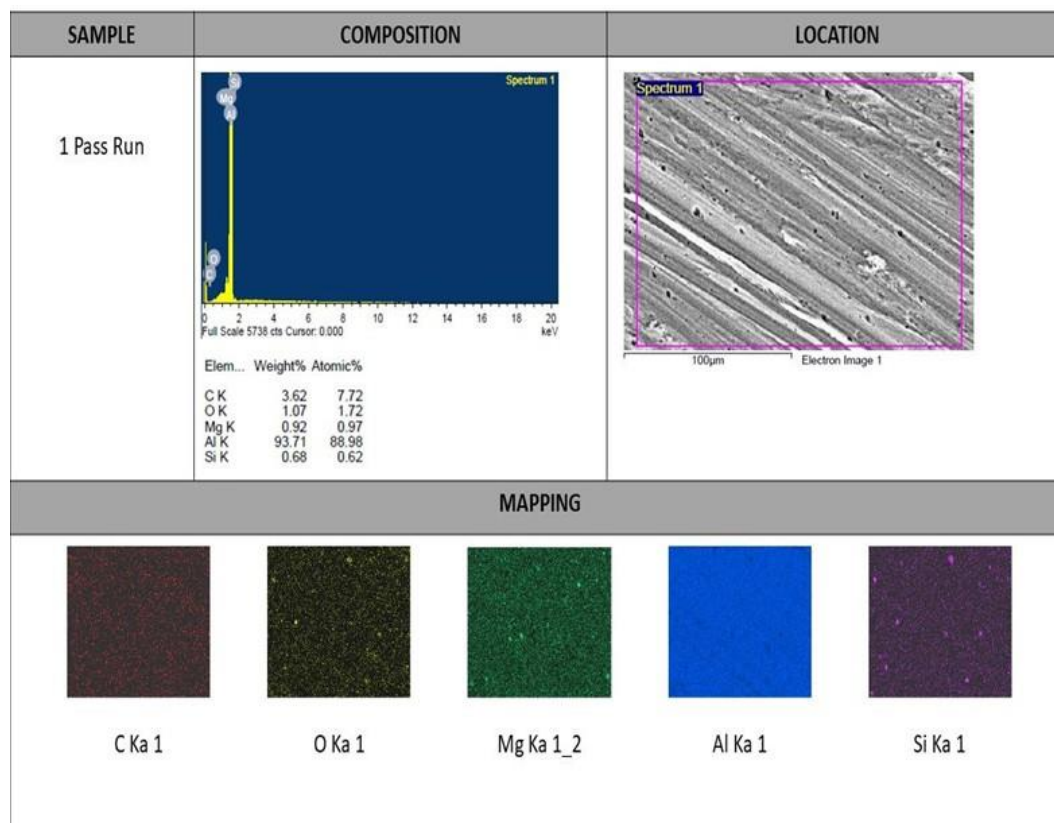


Fig. 7. FESEM-EDX and elemental mapping analysis of one pass

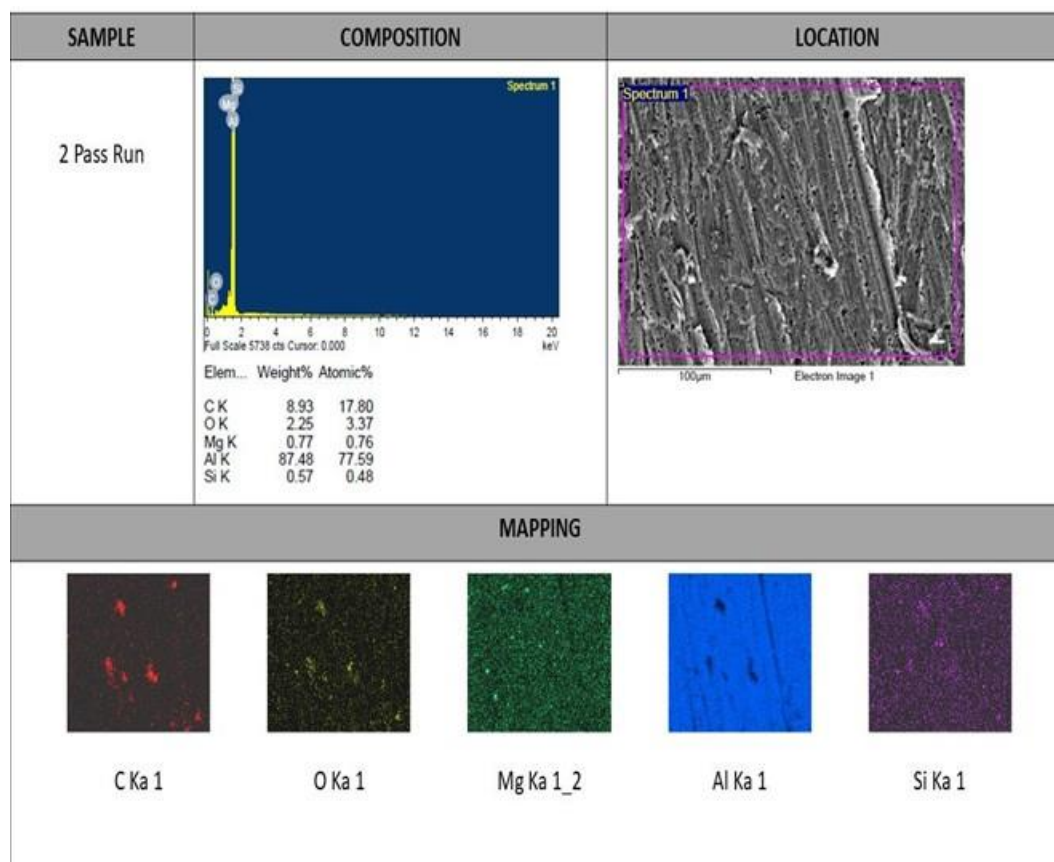


Fig. 8. FESEM-EDX and elemental mapping analysis of two passes

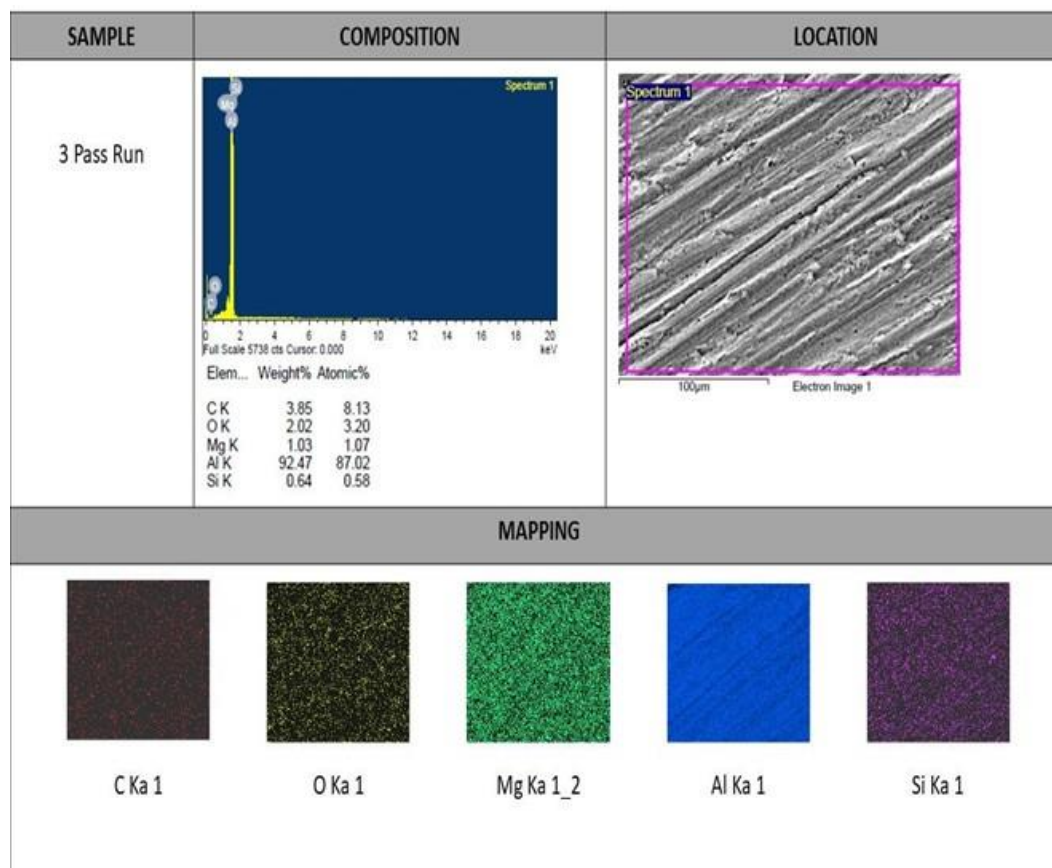


Fig. 9. FESEM-EDX and elemental mapping analysis of three passes

CONCLUSIONS

The multi-pass FSP method is used to successfully fabricate ENAW-6061-O-Gr composites. The graphite nano flakes dispersion is confirmed through EDX analysis. The graphite particles exfoliation as a single reinforcement inside aluminium matrix is achieved with no macro level defects inside the composites. Multi-pass FSP improved the tensile strength of the composites. The UTS of C3- ENAW-6061-O-Gr Composite processed with three passes is 153.65 MPa, that is 24% of improvement compare to the unreinforced ENAW-6061-O base metal alloy. The UTS, peak load, elongation have been improved with increase in number of passes. Thus, tensile properties were proportional to the number of passes and extent of graphite exfoliation inside the resultant ENAW-6061-O-Gr composites. The passes were restricted to three as the FSP band thinning because of flash formation during processing.

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Conflicts of Interest

The authors declare that they have no competing financial interests or personal relationships that could have seem to influence the study reported in this paper.

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