

Effect of agro waste ash as reinforcement on microstructure and mechanical properties of aluminium surface composite fabricated via FSP technique

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Abstract:

The life span of AA6061 components in many applications depends on surface properties. Therefore, it is advantageous to reinforce the surface layer while the bulk material retains its ductility and toughness. Non-deformable nature of ceramic reinforcement particles in AMCs poses loss in ductility and toughness of the composite. Rice husk ash (RHA) is an agro-industrial waste and by product of rice husk. Friction stir processing FSP is an evolving novel process used to fabricate surface metal matrix composites. AA6061/RHA surface composite was produced using friction stir processing (FSP). Optical and scanning electron microscopy, micro hardness and tensile tests were used to characterize the specimens. The results indicated that uniform distribution of RHA particles in the stir zone SZ by FSP process can improve the micro hardness of specimens. The micro hardness of the AA6061 aluminium matrix beside the FSP zone, the stir zone of specimen processed by the FSP without RHA particles, and the stir zone of specimens processed with RHA using (one, two, and four FSP passes), were about: 66HV, 46.6HV, 106HV, 99HV, and 86HV, respectively. Higher hardness value of 106HV was achieved with the Sample processed using one pass FSP. RHA shows encouraging positive potential for further study as reinforcement for the fabrication of low cost high performance surface composite.

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1. Introduction

The ceramic particles incorporated into aluminium metal to form aluminium matrix composites (AMCs) contribute to high strength, improved resistance to wear, creep and fatigue which make AMCs promising structural materials for aerospace and automobile industries. However, non-deformable nature of the ceramic reinforcement particles in AMCs poses loss in ductility and toughness of the composite which limits their wide applications to some extent. The life of components in many applications depends on surface properties [1]. Therefore, to reinforce the surface layer of metals while the bulk material retains its ductility and toughness is important. Such modified surface layer is known as surface metal matrix composites SMMCs [2]. Number of methods such as stir casting, and powder metallurgy etc. are used to produce MMCs. But there are some drawbacks related to the methods such as; difficulty to achieve uniform dispersion of reinforcement, and issue of interfacial reaction between reinforcement and metal matrix [3].

Friction stir processing (FSP) is a new technique developed for modification of microstructures in materials

based on the principle of Friction stir welding (FSW). In the process a non-consumable rotating tool consisting of pin and shoulder is inserted into a substrate and the interfacial friction between the rotating tool shoulder and the substrate generate local and sufficient heat to produces a highly plastically deformed zone (stir zone) [3]. Surface composite fabrication using FSP is normally made, first by making a groove on the surface of a work piece, and reinforcement particles are compacted into the groove; then follow by single or more FSP pass along the groove. Material flow induced by the tool pin mixes the two materials together and a composite layer is produced. Apart from the forcible mixing involved with this process, another advantage of friction stir processing is that no bulk melting occurs during the process Nandan, *et al.* [4] and Alidokht, *et al.* [5], therefore deterioration of mechanical properties or dissolution of the reinforcing material may be reduce, and these are typical problems in casting and powder metallurgy based metal matrix composite fabrication techniques.

Number of researchers reported investigations studying upper surface modification of light metals via

friction stir processing (FSP). Don-Huyun, *et al.* [6], fabricated AA6061-T4/SiC surface composite by FSP, they indicated that SiC particles were uniformly dispersed into an AA6061-T4 matrix and microhardness of the material was significantly improved due to the grain refinement and the distribution of the SiC particles. Dolatkah, *et al.* [7], produced Al5052/SiC surface metal matrix composite and they showed that decrease of SiC particles size enhance hardness and wear properties. Mehdi, *et al.* [8], carried out FSP to produce AA5083 aluminium alloy with copper particles reinforced layer. Result showed that the reinforced specimens present fine grains and higher level of hardness. Sathiskumar, *et al.* [9] reinforced copper surface with B4C via FSP and they reported homogeneous distribution of B4C particles and well bonded with the matrix and hardness of the processed zone was increased by 26%. Sharifitabar, *et al.* [10] incorporates Al₂O₃ particles on 5052Al alloys using FSP and reported improvement in the tensile and yield strength of the metal after FSP.

Almost all the reported surface composite fabricated via FSP uses ceramic particles, silicon carbide, alumina Al₂O₃ etc. as reinforcement. Particulate ceramic materials such as silicon carbide (3.18 g/cm³) and alumina (3.9 g/cm³) have been widely utilized as reinforcement in AMCs [2] are however, denser than Aluminium (2.7 g/cm³) and thus result in increase in the weight of Aluminium based composites depending on the weight percent of the reinforcing phase [11] New trend currently explored by composite materials researchers is the consideration of ashes obtained from the controlled burning of agro-wastes

such as rice husk, bagasse, bamboo leaf, coconut shell and ground nut shell as particulates reinforcement for the development of AMCs [12, 13]. These agro-waste ashes often contain a high percentage of silica (SiO₂) with a distribution of other refractory oxides such as Al₂O₃ and hematite (Fe₂O₃) [14]. The agro-waste ashes are characterized with densities far lower than that of SiC (3.18 g/cm³) and Al₂O₃ (3.9 g/cm³). Saravanana and Senthil [15] study the possibilities of reinforcing aluminium alloy (AlSi10Mg) with locally available inexpensive rice husk ash for developing a new material by stir casting process. They reveal that the percentage reinforcement of RHA will increase ultimate tensile strength, compressive strength and hardness of the composite. As the processing of the surface composite during FSP is carried out at temperatures below the melting point of the substrate, problems seen in conventional techniques based on liquid phase processing at high temperatures may be avoided.

The objective of this study is to evaluate the feasibility of incorporating rice husk ash particles RHA as reinforcement particulate into the surface of aluminium alloy AA6061 via friction stir processing FSP and to evaluate the effect of the RHA on microstructure and mechanical properties of the alloy AA6061. The use of RHA in this study is motivated by its very low density (0.3-1.6 g/cm³) in comparison to alumina Al₂O₃ (3.9 g/cm³), its large availability and even distribution in most part of the world, and it's cheap and simple processing requirement [16].

Table 1- Chemical composition of AA6061

Element	Al	Mg	Si	Cu	Zn	Ti	Mn	Cr	Fe	others
wt %	Bal	1.0	0.8	0.15	0.25	0.15	0.15	0.35	0.7	0.05

Table 2- Chemical composition of rice husk ash

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	Others
wt%	94.04	0.249	0.136	0.622	0.442	0.023	2.49	3.52

2. Experimental procedure

Commercial available aluminium alloy AA6061 rolled plate thickness: 5mm, and rice husk ash particles (RHA: average size 32µm) were used to fabricate surface composite via FSP, the temper condition of the as received AA6061 was unknown to the authors. The chemical composition of the plate and the RHA are shown in Table 1 and Table 2 respectively, the scanning electron (SEM) micrograph of the as received Rice husk ash particles is shown in Figure 1. For producing the composite layer, a groove of 1.2mm in width, and 1.2mm in depth was made in the middle of the plate using power saw then compacted with RHA, this FSP fabrication procedure has been documented elsewhere [8]. A pinless tool was initially employed to cover the top of the groove after filling with the powder particles to prevent the particles from spattering during FSP. Finally, a tool with pin was plunged into the plate until the shoulder's head face reached 0.20mm under the upper surface in order to produce the composite. The tool used in this study was made from tool steel (H13), with cylindrical shoulder diameter of 12mm, threaded pin diameter 4mm, and pin length of 3.5mm. Friction stir

processing (FSP) in Figure 2 was carried out on a vertical milling machine at tool rotational speed of 1400rpm and traverse speed of 26mm/min, and tool tilt angle of 2.5°. Single/one FSP pass was first made on the specimen without RHA and one to four passes were made on the specimen with RHA particles in order to evaluate the effect of the FSP passes on the RHA particles homogeneity/distribution. In order to hold the work-piece they were clamped onto thick mild steel (backing plate) by using bolts.

After the FSP transverse section of the processed specimens with and without RHA particles were cut, mechanically polished, and etched using Keller's reagent (1mL hydrofluoric acid, 1.5mL hydrochloric acid, and 2.5mL nitric acid in 95mL distilled water). Microstructures changes were observed by employing optical microscope (OM) at the cross section perpendicular to the FSP direction Figure 5. Vickers hardness test was carried out using 780.9mN load for 15 sec. The micro hardness was measured along the cross section of the processed samples surface.

Tensile specimens were taken from the surface composites normal to the FSP direction and made as per

ASTM: E8/E8M-11 standard by using CNC milling, machining to the required dimensions. The schematic sketch of the tensile specimen is shown in Figure 3. The tensile test was carried out on a computer controlled universal testing machine at a cross head speed of 0.5 mm/min.

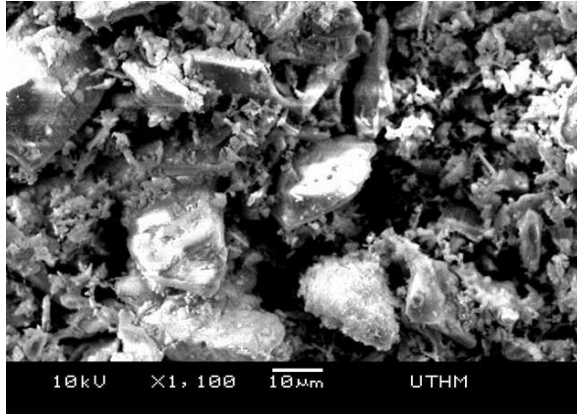


Figure 1: SEM micrograph showing RHA particles

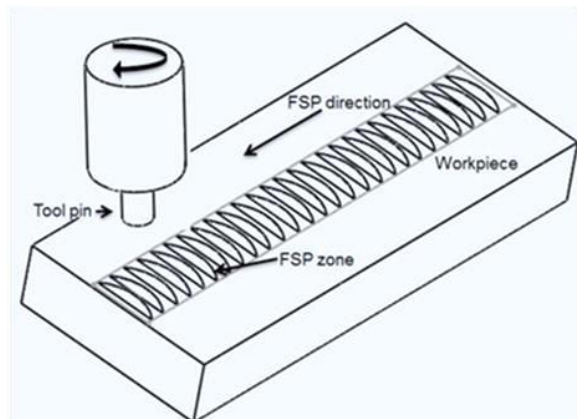


Figure 2: Schematic illustration of FSP process used for AA6061 with RHA particles

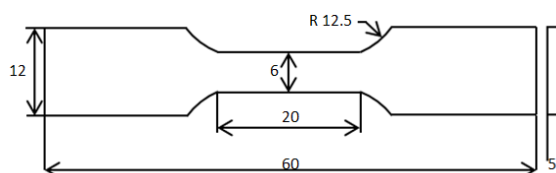


Figure 3: The schematic of tensile specimen (Dimensions in mm)

3. Result and discussion

3.1. Microstructure

Figure 6a presents the microstructure of the as received material. Second phase particles and elongated grains can be seen within the entire microstructure. Formation of precipitates on grain boundaries is common in aluminium alloys with magnesium content [17], and could increase possibility of micro voids formation and lead to reduced ductility [18]. FSP runs were carried out perpendicular to the rolling direction of the as received material Figure 4.

Figure 5 depicts macrograph of the stir zone of Al 6061 sample friction stir processed at rotational speed of 1400rpm and traverse speed of 28 mm/min. Micro image from the friction stir zone SZ was presented in Figure 6b. It was observed that the elongated grains and precipitations seen in Plate 6a have modified after FSP, to finer dispersed particle which is an indication of particle refinement via FSP. Such change in particles size is very important in terms of the mechanical properties. It is evident that the grain structure in the SZ, deduced from the microscopy, attains the equiaxed morphology that has developed through dynamic recrystallization. Within this zone, severe plastic deformation and frictional heating occur during FSP resulting in the formation of fine-grained microstructure due to DRX. This result confirms with that reported earlier by Magdy and Ehab [19].

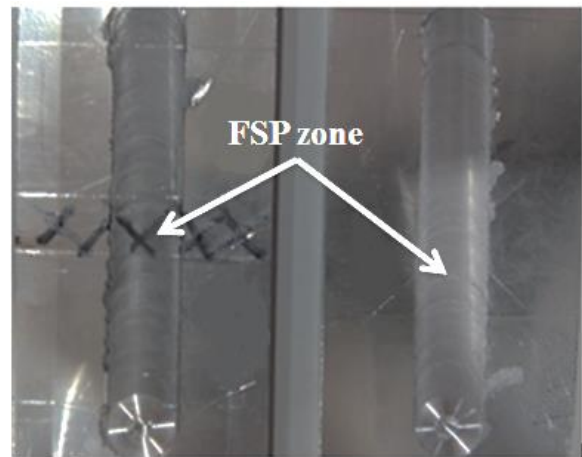


Figure 4: Friction stir processed specimens

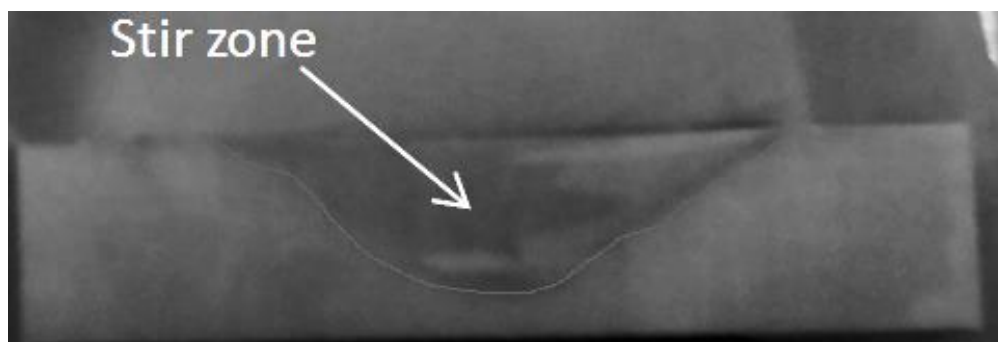


Figure 5: Optical macrographs: cross section of the FSP specimen showing the stir zone shape of samples friction stir processed at: 1400 rpm, and 26 mm/min

After the FSP process, AA6061/RHA surface dispersion strengthen composites was produced. A micrograph taken from the cross section of the AA6061/RHA composites specimen showing bonding interface between the metal and the RHA particles is

presented in Figure 7. It shows from the figure that the composite layer was well bonded to the aluminium substrate and no defect was observed along the interface region.

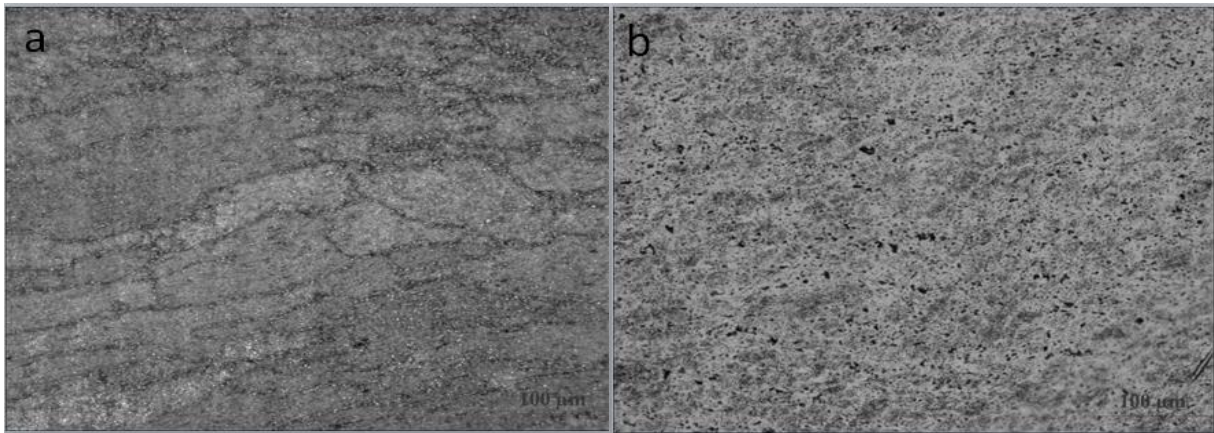


Figure 6: Optical micrographs: (a) as received material (b) After 1 FSP pass at 1400rpm

Optical micrographs of the stir zone (SZ) of AA6061/RHA composite layer are shown in Figure 8a, b, and c. It is observed that RHA particles are distributed and bonded well with the AA6061 matrix as the rotating tool gives sufficient heat generation and a circumferential force to distribute the reinforcement particles to flow in a wider area [20]. The distributions of the RHA particles in the matrix keep improving after every FSP pass. A more homogeneous distribution was achieved after four FSP passes. It means more dislocation sources will be available in additional FSP passes and finally higher extend of grain refinement will occur.

Bauri, *et al.* [21] and Dolatkah, *et al.* [7], in their investigation on Al/TiC and Al 5052/SiC mention similar case. It is also observed the size of the reinforcement particles (i.e. RHA) was reduced in size (to 2µm) than the as received particles Figure 9, and this is attributed to the FSP tool which provides a shear force substantial to breaking of reinforcement particles in stir zone. The representative SEM micrograph, and EDS profiles of the AA6061/RHA surface composites are presented in Figure 10 a, and b, which confirms that the reinforcement particles are dispersed in the aluminium matrix. The EDS, profile

show peaks of aluminium (Al), oxygen (O), carbon (C), iron (Fe), and silicon (Si). The presence of oxygen confirms the SiO₂, Al₂O₃, potassium oxide and ferric acid Fe₂O₃, which are the main constituent derived from the RHA.

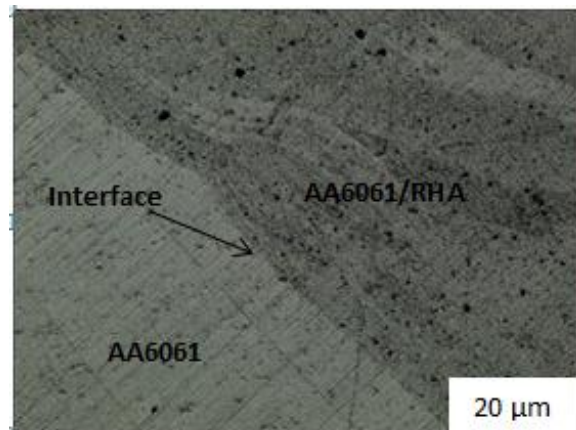
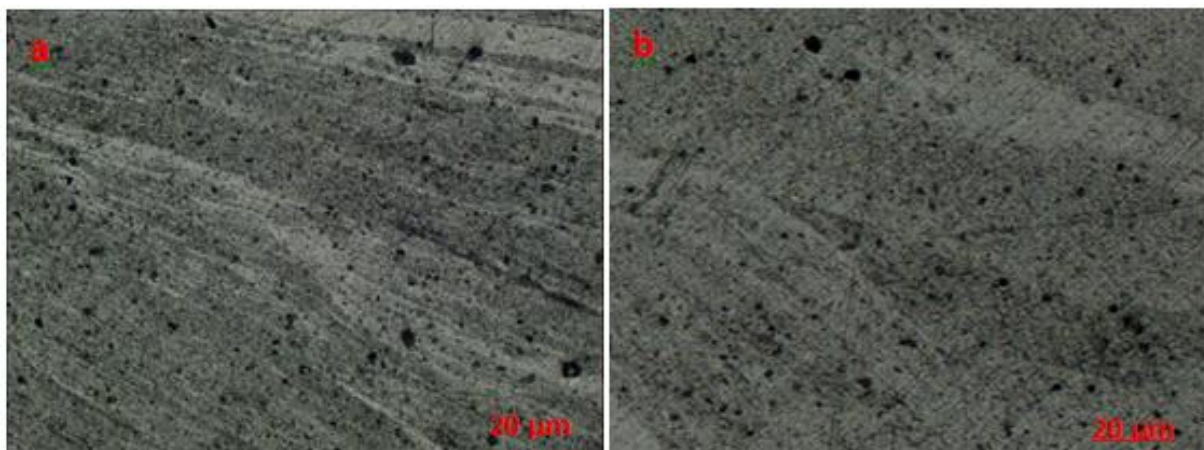


Figure 7: Cross section of the AA6061/RHA specimen showing bonding interface



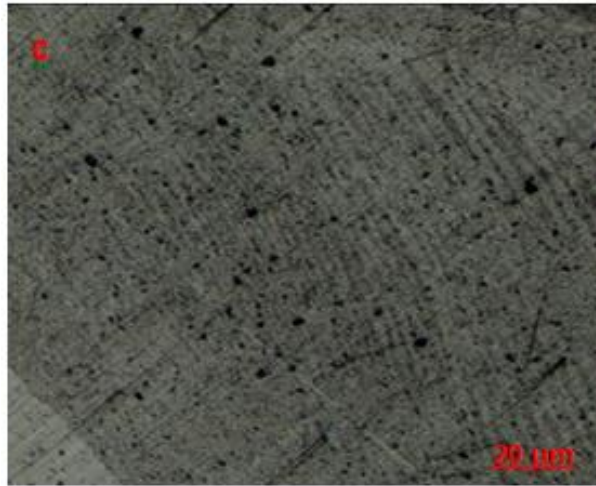


Figure 8: Optical micrographs the stir zone (SZ) of AA6061/RHA composite layer after; (a) 1- FSP pass (b) 2- FSP passes and (c) 4- FSP passes

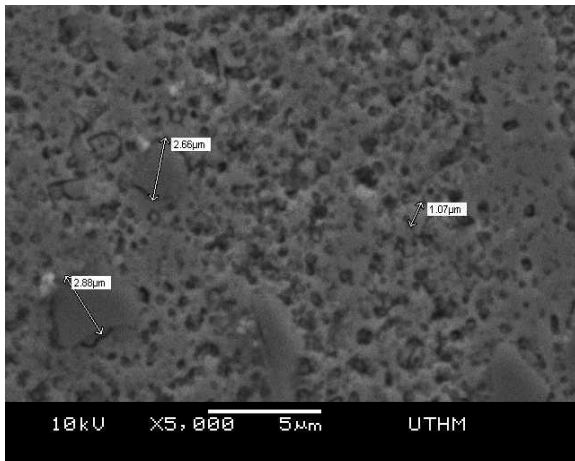


Figure 9: SEM micrographs RHA reinforcement particle in AA6061 after FSP

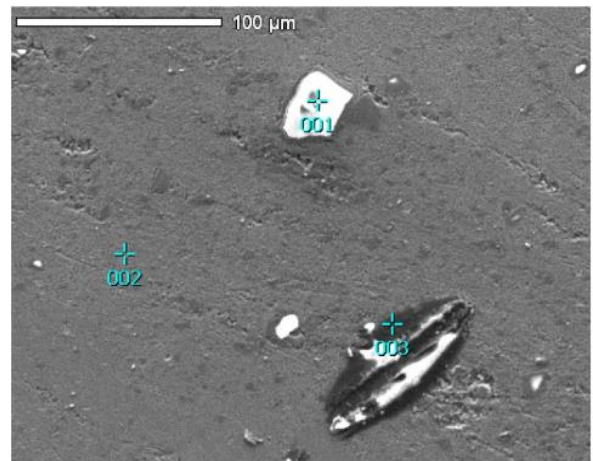


Figure 10a: The representative SEM micrograph for EDS analysis

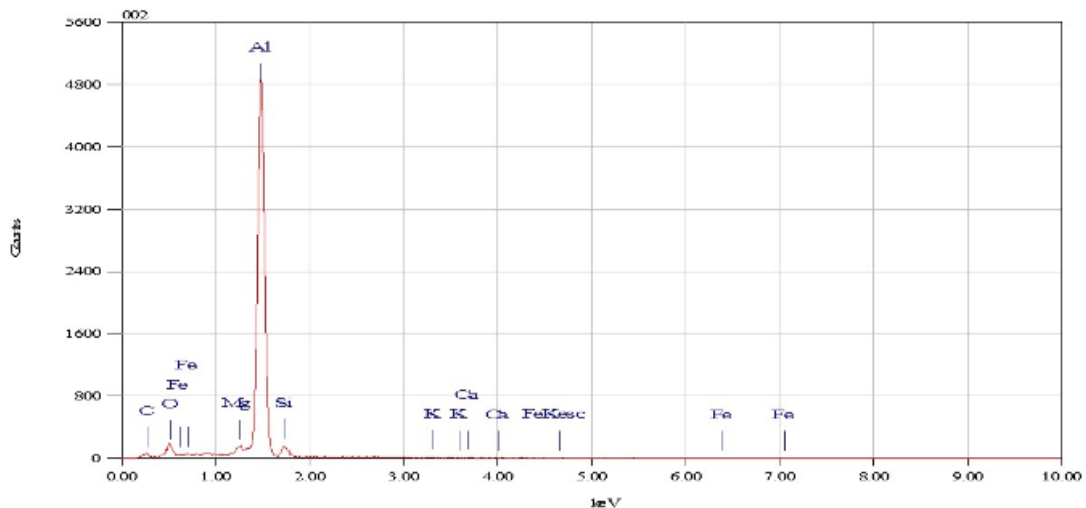


Figure 10b: EDS profiles of the AA6061/RHA surface composites

3.2. Mechanical properties

3.2.1. Microhardness

Figure 11 shows micro hardness test results of the friction stir processed (FSPed) specimens. In the specimen friction stir processed without RHA particles, the average

hardness value decreased to 46.6HV in stir zone, compared with 66 HV of the aluminium matrix beside the FSP zone. This may be because, within the FSP stir zone SZ, severe plastic deformation and friction heating during FSP result in generation of a recrystallized equiaxed

microstructure due to the occurrence of DRX, which results in low dislocation density structure. Mc Nelly, *et al.* [22], reports this rises of temperature anneals material in the stir zone. Hence annealing can cause decrease in dislocation density and residual compressive stresses of initially rolled AA6061 sheet and as a result, hardness value decreases. In other words, another probable reason is that intense deformation causes fragmentation of second phase particles and precipitates, leading them to be redistributed in new locations at the interior of grains as reported by Dadbakhsh, *et al.* [23]. This consequently leads to less strain and stress localization contributing to material softening and lower hardness. Similar drop of hardness due to softening of the SZ was also reported in the work conducted by Magdy and Ehab [19], on AA 6082, and Rodrigues, *et al.* [24], on AA 6061. In the specimens friction stir processed with RHA particles average hardness of the stir zone SZ was significantly improved from 46.6HV to 106.5HV by 1- FSP pass. Two factors may be involved in this hardness enhancement: presence of reinforcement RHA particles in the stir zone which contain high amount of hard SiO₂ that acted as resistances to slip; hence, reinforced the alloy and increases it hardness. And quench hardening effect due to difference in thermal contraction coefficient of aluminium matrix and RHA particles. A similar reason was given by Aigbodion [25] on Al-Si-Fe/Rice husk composite, Saravanan and Kumar [15] on AlSi10Mg/RHA composite. For the 2- FSP passes specimen hardness increased to 99HV, slightly less than that of 1- pass specimen. But in the 4- FSP passes specimen the hardness drop to 86HV, lower than in 1 & 2- FSP passes. The annealing effect may be attributed to the slight drop in hardness observed in the 2 & 4-passes specimen. Increased in hardness due to the presence of reinforcement particle in surface metal composites via FSP have been reported by many researchers such as Choi, *et al.* [6], Zohoor, *et al.* [8] and Sathiskumar, *et al.* [9].

3.2.2 Tensile properties

The tensile properties of a material are measured as yield strength (YS) or ultimate tensile strength (UTS) and uniform elongation. In most engineering applications both

high strength and high ductility are desired. Unfortunately, it seems that materials may be strong or ductile, but rarely both at once. Comparison of tensile properties of all specimens processed with and without RHA particles at 1400 rpm and as-received Al alloy is shown in Figure 12. As can be seen, tensile strength of FSP samples produced without RHA was slightly lower than base material it is also observed that yield strength of the SZ is always lower than that of base material. Almost similar results were observed by Sharifitabar, *et al.* [10]. But, the tensile and yield strength of the surface composites is slightly greater than samples produced without RHA. In FSP the heat input increases with increase of rotational speed [26] which resulted in matrix softening. Actually, the softening of the material would be expected to improve the %EL. similar to this, the %EL in Figure 12 of the sample processed without RHA increases slightly from 5.0 for the as received material to 6.4 due to softening of the matrix. Very small change in UTS was seen between the as received material and the specimens processed with and without RHA powder (166, 162, and 163Mpa). It was reported that the presence of reinforcement particles such as SiC, Gr and Al₂O₃ could restrict the grain boundary sliding and dislocation motion. Also, together with weak interfacial bond between the reinforcement particles and the matrix, finally leads to deterioration of the tensile properties [27]. In other words, composite has incompatible deformation between the plastically deformed matrix and the rigid reinforcement particles causing the generation of geometrically necessary dislocation. Besides, the presence of reinforcement particles increases the effective slip distance of dislocations during deformation, which leads to reduced elongation. However contrary to that, in this study the presence of rice husk ash RHA in the matrix is found to not deteriorate the elongation of the as received/base metal (%EL of BM: 5.0, %EL of AA6061/RHA: 5.3).

The SEM fracture surface image of AA6061 FSPed at 1400rpm and that of AA6061/RHA at 1400 rpm surface composites are shown in Figure 13. It was observed large number of uniform dimples in the fracture surface of the specimen processed without RHA particles which indicate ductile fracture Figure 13a.

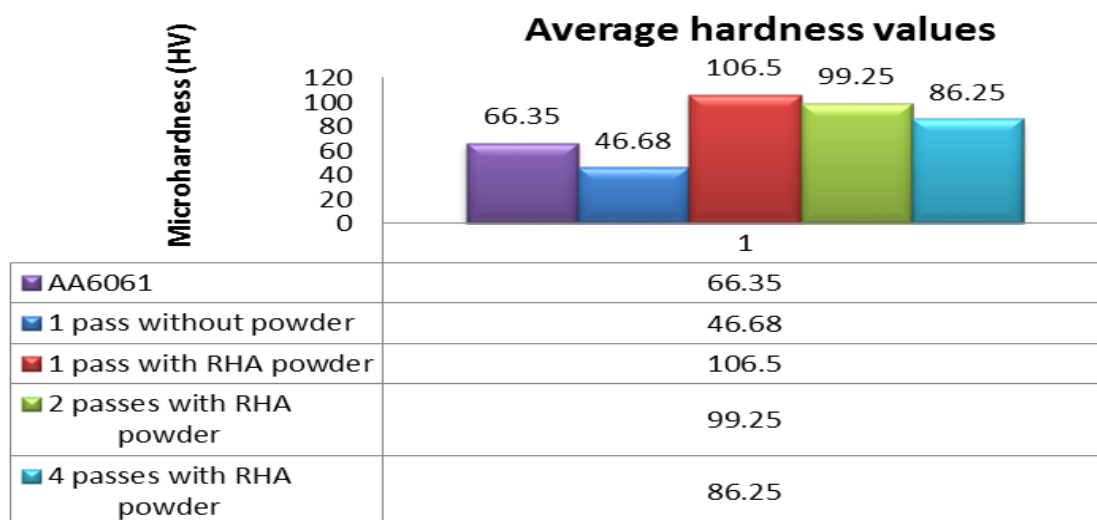


Figure 11: Microhardness test result

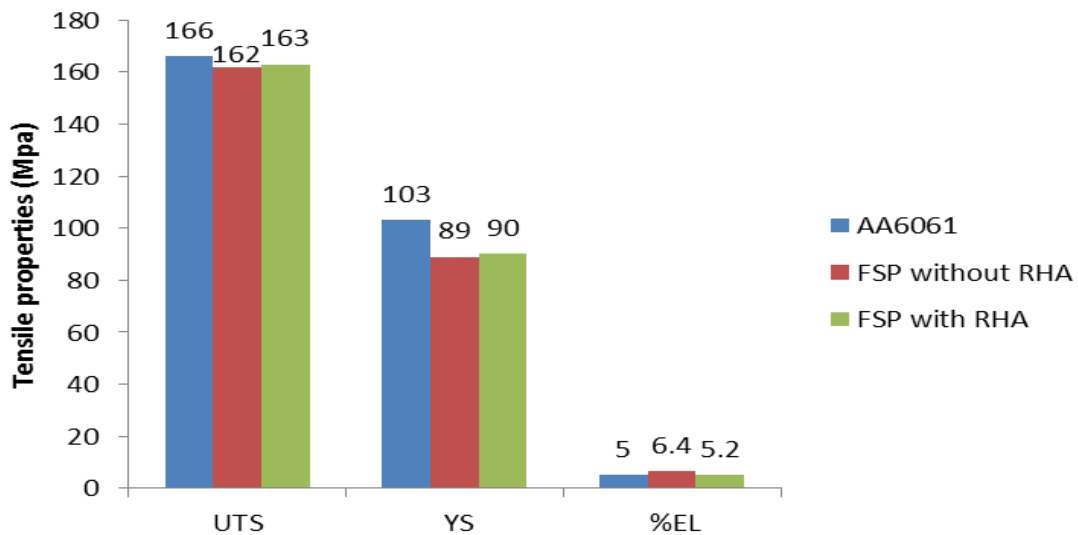


Figure 12: Comparison of tensile properties

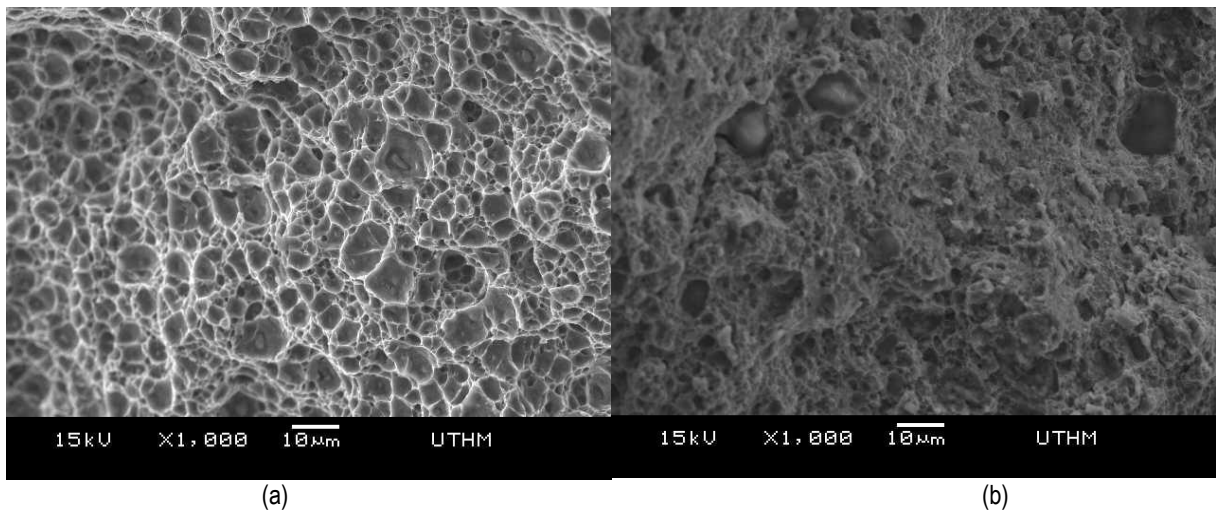


Figure 13: Fracture surface

The fracture surface of AA6061/RHA surface composite despite the present of RHA shows ductile fracture because its elongation is somewhat the same as the base metal. Dimple can be seen in the surface filled up with RHA particles. It can be concluded that there is good interfacial bond between the reinforcement particles RHA and the AA6061 matrix. This may be because of the lower density of RHA (0.3 – 1.6 g/cm³), lower than that of aluminium (2.7 g/cm³).

4. Conclusion

Fabricated composite material with rice husk ash particles in AA6061 alloy was produced via FSP. The effect of RHA on microstructure, microhardness, and tensile behavior of the surface composite was evaluated. The obtained results can be summarized as follows:

- In the stir zone SZ, the original microstructure of the BM was changed to fine recrystallized grain structure.
- The RHA particles are well distributed in the stir zone in both first, second, and fourth- FSP passes with defect free.

- Microhardness of the stir zone processed without RHA particles decreases to 46.6HV with respect to that of the aluminium matrix beside the FSP zone 66HV.
- The microhardness of the stir zone with RHA particles (AA6061/RHA surface composites) after 1-FSP pass was recovered and increased by about 40%.
- Tensile strength of the composite was slightly lower compared with the as received material due to the presence of RHA particles.
- RHA shows some great promise to be further investigated as reinforcement for the fabrication of low cost high performance surface composite.

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friction stir processing: investigating microstructure,
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