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Characterizations Particulates of Crushed Particles (Al_Zn_Mg_Cu_Ni) for Fabrication of Surface Composites Al-Alloy Using Friction Stir Processing Route

Haider Tawfiq Naeem*
Chemical Engineering Department., Engineering College ,,Al-Muthanna University

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ABSTRACT

Productions of surfaces composites have high mechanical properties and altered microstructural is attracting considerable attention. One of the methods of fabricating composite surfaces of aluminium matrix alloys are the procedure of Friction Stir Process (FSP). In this investigation, the producing for the surface composites of Aluminum_Zinc_Magnesium_Copper alloy (7000 series) with the pulverizing particulates of (Al_5wt%Zn_2.5wt%Mg_1.5wt%Cu_4wt%, 6%, 8% Ni), as the reinforcement particles using FSP was discussed. The reinforcements were from Al_Zn_Mg_Cu particulates as well various nickel powder (mass percentages) synthesized using the high-energy, ball milling alloying process. Composites samples underwent for the homogenizing treatment through various temperatures. These samples have subjected the ageing treat with 120 °C for one day. Results showed a 70% decrease in the grain sizes of the surface of composites Al-alloys compared with as-received Al-alloy. The grain refinement and uniform dispersion of the reinforcements during the Al-matrix were obtained because of the optimized parameters of FSP. The intensity of precipitations intermetallic, which as reinforcements increased after heat treatments along with the phases of the precipitates. The effects of the grain reduction and intercompounds with the precipitation phases led to a valuable rise in the hardness property of surface of aluminium 7000 alloy-reinforced composities.

توصيف جسيمات الدقائق المطحونة (ألمنيوم-زنك-مغنيسيوم-نحاس-نيكل) لتصنيع سطح متراكب لسبيكة المنيوم بأستخدام عملية الأحتكاك الفائق

الخلاصة

أنتاج سطح متراكب للألمنيوم بخواص ميكانيكة عالية بالاضافة الى خواص مجهرية معدلة اجتذب اهتماما كبيرا ولذا تعتبر طريقة ألاحتكاك الفائق (Friction Stir Processing) من إحدى الطرق المتميزة لتصنيع (لافتعال) سطح مركب معدني .في هذه الدراسة، يتم إنتاج سطح متراكب من سبائك الألومنيوم-زنك-مغنيسيوم-نحاس (السلسلة ٧٠٠٠) مع دقائق من مسحوق مطحون بنسب مختلفة من التكوين التالي [المنيوم- (5% -زنك)(2.5%- مغنيسيوم) (1.5%- نحاس) 8% -(4% -نيكل)] بأستخدام طريقة الأحتكاك الفائق ، دقائق المسحوق(المتكونة من ألمنيوم-زنك-مغنيسيوم-نحاس-نسب مختلفة من النيكل) تم توليفها باستخدام عمليات الطحن السبكي بالكرات ذات الطاقة العالية. بعد ذلك تم وضع دقائق المسحوق في داخل شق صفيحة من سبيكة الالمنيوم ثم ضغطت مع تثبيت العينة على ماكنة الفارزة العمودية واجريت عملية الاحتكاك الفائق لتكوين سطح مقوى متراكب من سبيكة الألمنيوم. عمليات التجانس الحراري تمت عند درجات مختلفة وكذلك معاملات التعتيق الصناعي الحرارية في ١٢٠ درجة مئوية لمدة ٢٤ ساعة اجريت على العينات. أظهرت النتائج انخفاضا كبيرا بنسبة ٧٠٪ في أحجام الحبيبات المجهرية لسبيكة الالمنيوم المتراكبة المعززة بالجسيمات بالمقارنة مع سبيكة الالمنيوم الاصلية. النتائج اوضحت سبب تنعيم البلورات المجهرية وكذلك التجانس الحاصل في المركبات الوسطية داخل سبيكة الالمنيوم المعززة يعزى الى المقاييس الجيدة لظروف عمل طريقة ألاحتكاك الفائ . كثافة المركبات الوسطية من الاطوار المترسبة التي تعتبر كمقويات للسبيكة تزداد بعد المعاملات الحرارية.أثار تنعيم الحبيبات بألاضافة الى المترسبات قاد الى زيادة الصلادة السطحية للسبيكة المتراكبة.

الكلمات المفتاحية

التقوية بالمترسبات الوسطية، السبك الميكانيكي ، عمليات الاحتكاك الفائقة

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*Corresponding author.
E-mail addresses:dr.haider.ta@mu.edu.iq

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Introduction

The particles intermittent hardened aluminium matrix composites have enticed great attention, this is due (i) availability of different kinds of reinforcement within competitive estimates, (ii) the prosperous growth of manufacturing operations for fabricating aluminium matric composites (AlMCs) with re-producible properties, and (iii) the availability of metal working ways, which could be applied to manufacture those AlMCs [1-3]. That AlMCs were made either way by straight inserting reinforicaments into aluminium matrix or by compilation the reinforcemants in situe in aluminium matrix. The intermetellic compounids of Al-Ni displiay an catchy combination of chemical, physicel and mechanical properties [4, 5].

The importance of Aluminium_Zinc_Magnasium_Copper alloys (7000 series) is increasing in transportations, aerospaces, and military applications. These applications require higher strength, along with lower density [6]. Various techniques to fabricate these Al/Al-Ni, which as intermetallic composites are available, such as directionally solidification [7-8], and mechanical alloying [9]. Originally developed as a solid-state working technicality, friction stire processe is a simple and economical process [10, 11]. Friction Stir Welding (F.S.W) is a new technicality for retraction aluminium alloys fabricated in 1991 at the welding institute in British [10-13]. Based for the essentials of F.S.W, Mishra e.t al. [14] sophisticated friction stir processing (F.S.P) for microstructures amendment of matrices alloys. In FS.P, in friction stir processing, acylindric rotaiting device with a concentrico pins and shoulders is dived into the metal to be treated. The friction between the rotating device and the base-metal (working_pieces) generated focused heating to elevate the local temp. for the materials into the warm working zone where that could be plastical distorted simply. With the active temp. is reached, the device tool of FSP is over-passed along the line of interested. Material flux into the back of the pin tool, where it is extruded or forged happened behind the tool of FSP, combined, and cooled under hydrostatic pressurie conditioins[15-16]. Subsequently, FSP is considered an essentially a thermo-mechanism processing that occupied metals temperature into a domain (commonly more than 0.49 melting temperature) with plastically deformation [17]. A simply graphical explanation for the Friction Stir Process framework was given in Figure 1.

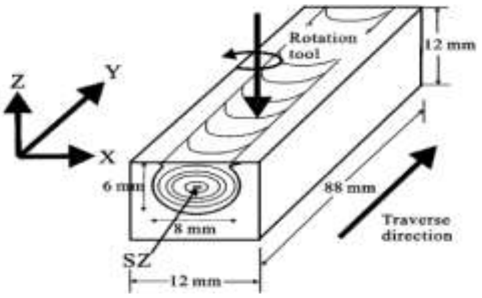


Figure 1: Schematic for the friction stir process [17].

Not enough studies information [18-20] about the possibility working of the hard intermetallic compounds reinforced the surface and matrix of aluminium composites by using friction stir processing. However, results for the synthesis of metal particulates powders as reinforcements of Al-alloy composites using FSP have not been reported. In the present investigation, milling alloying synthesizes the fabrication of dispersion and reinforcements of the powder particulates (Al-Zn-Mg-Cu-Ni). Then, FSP is used to embed blender-milled powder into an Al-alloy to produce surface composite of Al-matrix. The new advantage of the treated composite is the ability to create a manifold of intercompounds that are free of harmful matter and uniformed distribution, which can significantly improve strength.

Experimental procedures

The base material utilized in this study was an AA 7075 Al-alloy plate that was 13 mm thick with length 90mm and 20 mm wide. The measured chemical compositions was given at Tab. 1.

Table 1: The chemical compositions for the base Al-alloys (weight percentages %).

Elements	Amounts (wt. %)
Silicon	0.061
Iron	0.28
Copper	1.87
Manganese	0.054
Magnesium	2.81
Chrome	0.58
Zinc	6.28
Titanium	0.03
Aluminium	Balance

To synthesize aluminium alloy powders by use the ball milling process, the elemental powder precursors of Aluminium, Zinc, Magnesium, Copper, and Nikel were used as the raiw materialse for the elaboration of the three kinds of aluminum-milled powders as detailed in Table 2.

Table 2: Chemical compositions of the powders were the reinforced A, B, and C.

Powders	**Amounts (wt. %)**				
	Cu	Mg	Zn	Ni	Al
A	1.5	2.5	5	4	Balance
B	1.5	2.5	5	6	Balance
C	1.5	2.5	5	8	Balance

Ball milling process for the powders were applied within a high-energy planetary type of the balle mill Fritsich and with an argon atmospherie by use stianless stael balles with daimeters from ,10 mn to 20, mm. The balls into powder weight ratiio were 10;1 and the rotatoin spaed was 350, rpm. Milling process of the particulates of powder were carried out through a milliang time at about 13 hours. The V-shaped groove had been made in a suitable widith and adepth in the centerline of plate of the Al-alloy. The experiments of FSP are conducted using a milling machine. A stainless-steel tool that had a threaded tapered pin profile with a flat shoulder was used. The reinforcements A, B, and C of the milled powders were embedded in the groave. The groove opening has firstly plugged by use a pinless shoulder to eschew the flight of reinforcement particulates from the groove during processing of friction stir, as shown in Figure 2. The tool traveling speed (υ) was 39 mm./min. and the rotating speed, (ω) of the tool is 2300 rpm,. All the parameters of FSP have been used experimentally to obtain the optimal result. FSP samples were subjected to homogenization, artificial treatment, as given in the steps in Table 3. Each step of the treatment was followed with cold water quenching.

Table 3: The homogenizing and artificial ageing for the current study alloys under the FSP.

No.	**Type**	**Details of treatment**
A	Homogenization	450°C/120min. + 470°C/1440min. + 480°C/30min.
B	Ageing treat (T6-temper)	120°C/1440min. (Peak Ageing)

To carried out different tests, FSP Al-alloys samplies were cutting with the desired dimensioins, then ground and polished with a 0.5 µm diamond paste at the final stage. The samples were etched using Keller's reagent to detection the microstructure. The microstructural evaluations were conducted at the cross section of FSP sample composites that had normal FSP direction by opticale microscopay (OM) with a scanining elctron micrascopy (SEM), and followed via a coupled enrgy-dispersve X-ry spectrscopy (EDS). The X-ay dffraction anlysis (XRD) had used for detect the phases and intercompounds during FSP and subsequent heat treatments. Microhardness tests were conducted during the crosses sections of the stir zone for samples composites that had ordinary FSP direction, using Vickers digital microhardness tester.

Results and discussions

The microstructures of Al-alloy that had reinforcements with and without FSP are given in Fig. 3. A comparison between the as-received Al-alloy in Fig. 3a and Fig. 3b for the stir zone region showed that FSP with reinforcements had decreased the grain size from µm 47 to 19 µm, and microstructures with coarse elongated grains changed to a structure consisting of fine equiaxed grains in the stir zone. The reinforcements dispersion easily observed the material flow. The reinforcements have crowded in a specified region of the stir zone. The reinforcements were dispersed to take shape the onion circle within the stire zane for the FSP saimple as shown in Figure 3c.

The formation mechanism of the multiple vortexes is notarized to have a rotaiting sped, addition to effects of threaded traped pin profile as reported [21, 22]. Fig. 3d shows the major regions of the microstructure after FSP, namely, the stir zone (SZ), the ThermoMechanically Affecited Zon (TMAZ), and the HeatAffected Zone (HAZ). Reinforced **C** particles were found as the uniformly distributing white particles powder which are very well-bonded with Aluminium matrix substrate and had no visible defects within the friction stirred zone as presented in Fig. 3e. This result is because of vigorous stirring during FSP.

The creations of micro-grains in the Al-alloys through FSP were generally because of various re-storation mechanisms, such dynamic recoveary, and discontinuous dynamic recrysitallization [23, 24]. The reinforcements dispersion and alloying elements influenced the definitive grain sizes of the sitr zone. The amounts and dispersions of the reinforcements particles in the second phase of alloying elements could restrict the movemente of the grain-boundaries, preventing the grain-growth mentioned by Bahrami et al. [25]. The SEM image within the stir zone underwent the T6 aging shown in Fig. 4a revealed that the reinforced **B** particles was high separated in the Al matrix. The EDS microanalysis results shown in Fig. 4b proved that the increasing volume fraction of Ni content within the matrix was. This situation was because of the percentage increase in the reinforced **B** by about 6% wt. The increase in nickel mass is a consequence of the addition of the milling alloying process addition to the process heat input, which is the main governing mechanisms because of the extensives plastic deformations relating to the elevated temperature and the increasing the creat and precipitate of the Al-Ni intercompounds at the temperature of FSP for the Al7075 alloy [26-27]. The Al-matrix/the reinforced

C composite were aged with a T6 temper. Fig. 5a depicts the SEM pic. of the microstructure for the Aluminium matrix/reinforced **C** after applying the FSP and subsequent T6 aging, and the abundance of dispersion particles in the reinforced **C** within the matrix, This situation can be supported using the EDS microanalysis in Fig. 5b, which indicates that a higher percentage of nickel.

The X-ray differaction has performed to recognize the all phases of the FSP Al-specimens with reinforcements particls and subsequent heat treatments. The XRD patterns are shown in Fig. 6b, verifying the presence of the intermetallic phases and phase transformation of the Al/the reinforced **A** FSP sample after FSP. The α-Al with AlNi was the major peaks had amounts of the Al_7Cu_4Ni, $MgZn_2$ and Mg_2Zn_{11} compounds. The FSP led to a temperature increase that caused the reactions between the alloying elements and the reinforcements. This heat generation is attributed to the ratiio of the toal rotations speed into the traverses speed addition to the higher ratio led for the higher-heat-inputs in the stir zone metal-working. The high shoulder dia./ pin dia. ratio of the FSP tool used in the present study also caused the effect, which was chosen based on previous studies [28, 29]. In addition, the milling alloying process had the main effects on the combination of the aalloying elementes with the extension limit solubility of Ni in the mixture when subjected to a long time of milling.

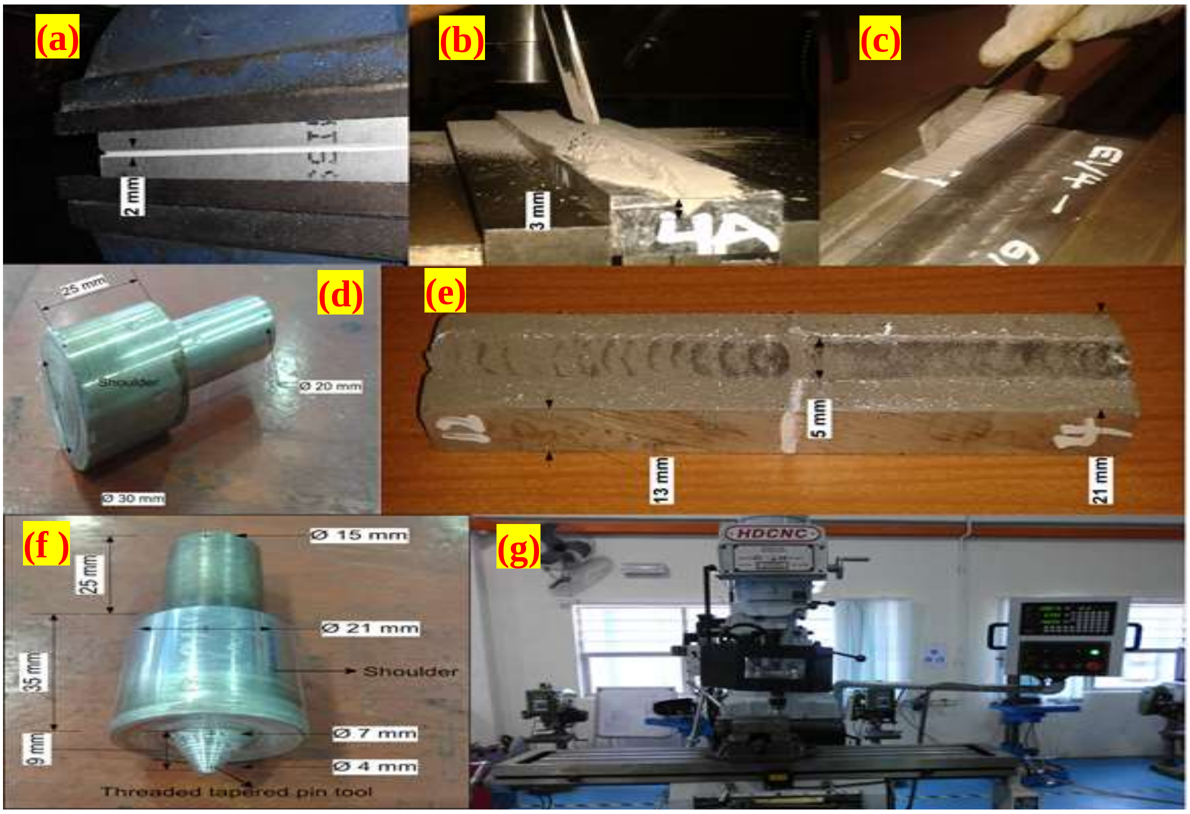


Figure 2: The FSP procedure: (a-c) cutting V-shaped groove(s) and inserting the reinforcements (A, B, C); (d-e) using a flat shoulder to undertake the sample repair; (f) applying threaded trapped pin tool to undertaken the FSP and (g) conducting a single pass of FSP machine.

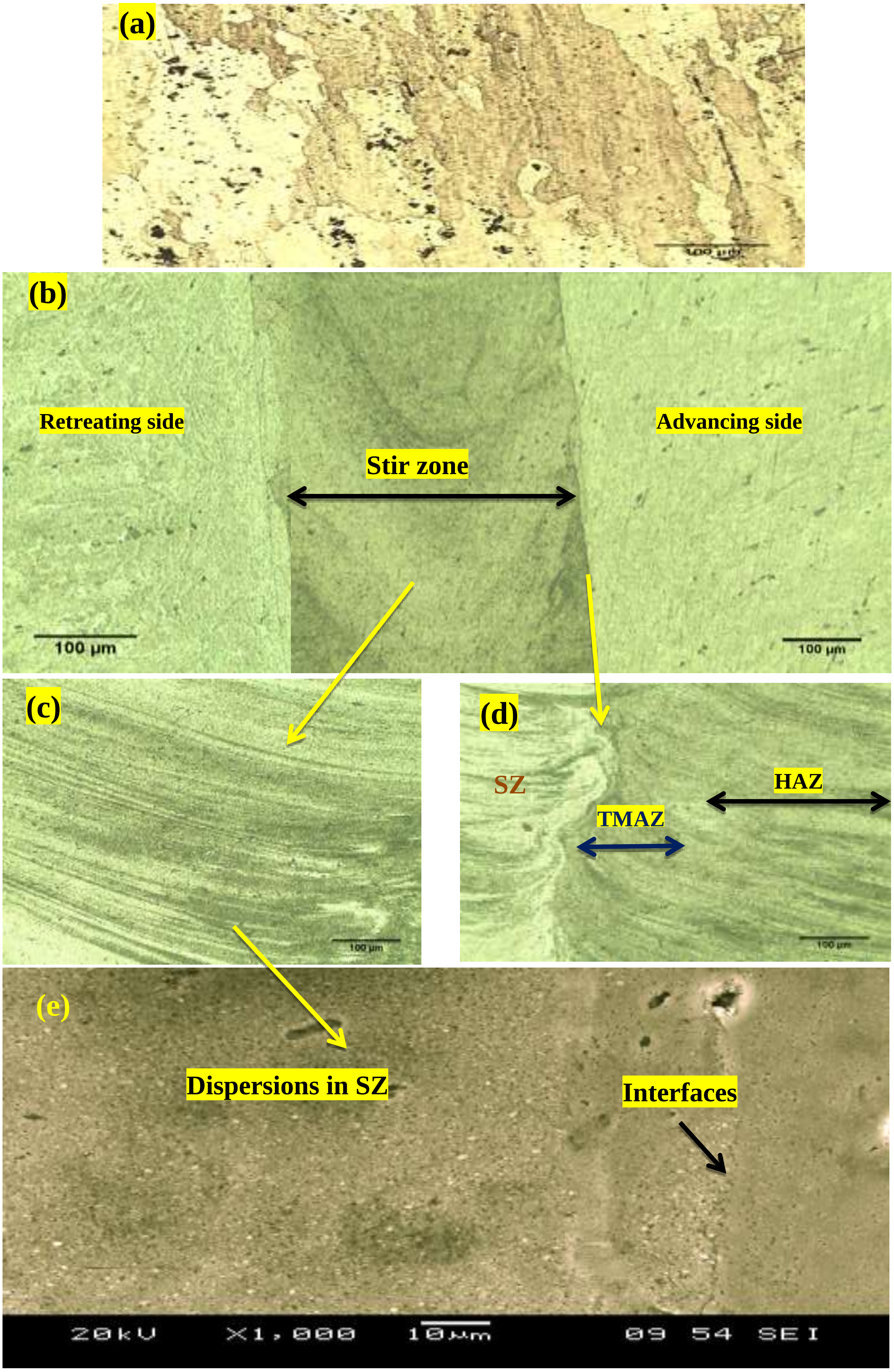


Figure 3: (a) OM of the received Al-alloy, (b) collected microstructure of surface of Al/reinforcement under FSP, (c-d) the different regions of Al/reinforcements composites and (e) SEM image interface of the Al matrix and dispersion powder-particles for reinforced C,.

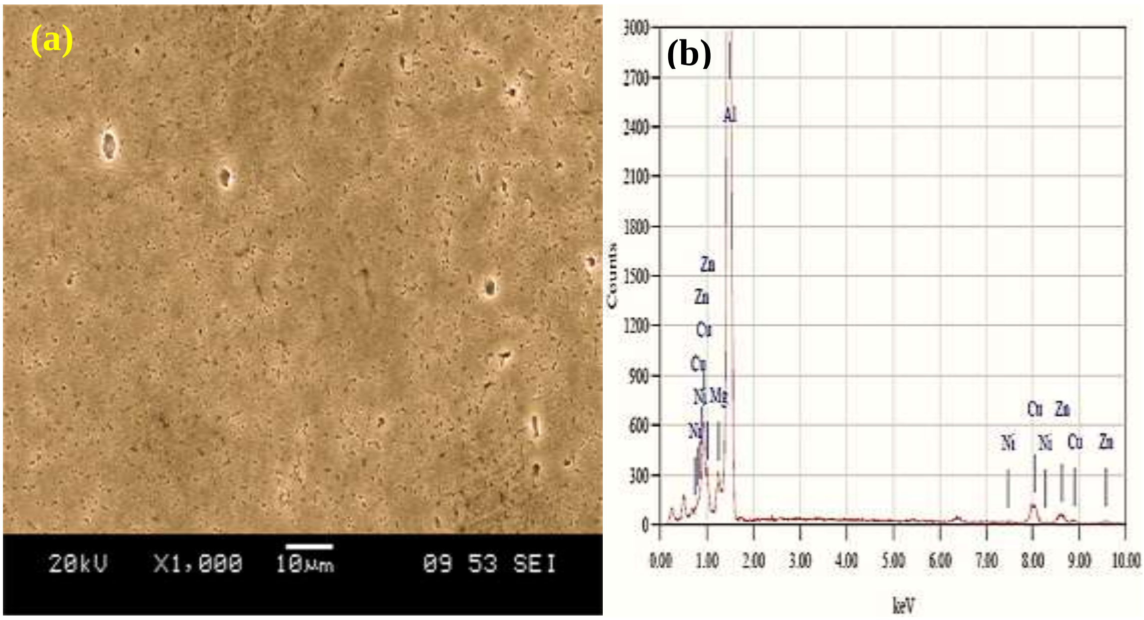
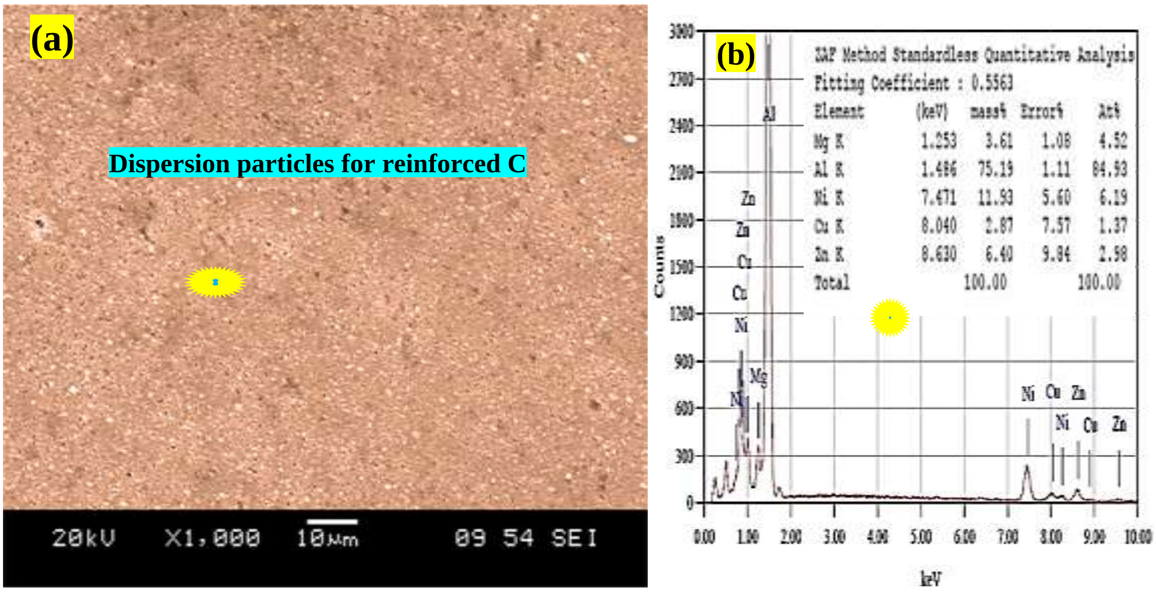


Figure 4: (a) SEM, and with (b) EDS, in sitr zone within the surface composite of Al7075/the reinforced B particulates undergoes the T6 treatment.



ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.5563

Element	(keV)	mass%	Error%	At%
Mg K	1.253	3.61	1.08	4.52
Al K	1.486	75.19	1.11	84.93
Ni K	7.471	11.93	5.60	6.19
Cu K	8.040	2.87	7.57	1.37
Zn K	8.630	6.40	9.84	2.98
Total		100.00		100.00

Figure 5: (a) The SEM-pic. & (b) EDS-analyss for Al 7075/reinforced C composite that underwent the T6 SEM image of the dispersions for the stir zone in FSP.

Fig. 6a showing X-ray diffiraction patterns for Aluminium-matrix/ reinforced **A** specimens, under applying T6 under the FSP. The α-Al, AlZn, and AlNi were considered as the essential peaks that had a new phase of Al_3Ni_2, with the Al_7Cu_4Ni and $MgZn_2$ phases. Fig. 6a shows the new appearance of the AlZn peak in T6 temper. The over dissolution of zinc in the aluminum matrix/reinforced **A** was because of heat input during the series of the FSP and heat treatments.

The XRD plots for Al7075/reinforced B composite after conducting FSP are found in Fig. 7b. The primary phases wereα-Al and AlNi, while the $MgZn_2$ was the minor phase. In Fig. 7a, XRD sample patterns with T6, detected the α-Al and AlNi phases, in addition to many $MgZn_2$ phases, Al_7Cu_4Ni, and Al_3Ni_2 compounds. Changes in the main peaks took place as result of the superabundance of severe plastic deformation in both the milling alloying and the FSP, to produce the reinforcements and the dispersion particles within matrix, respectively, as well as to influence precipitation that the heat treatments caused.

The increase in Ni% used in the reinforcement led to more appearances of intermetallic compounds with AlNi and Al_3Ni_2 as the major peaks. This result was because of the limit extension solubility of nickel into aluminum in the solid state a mentioned above.

Fig. 8b presents the XRD plots of the Al matrix/reinforced **C** composite undergoing FSP. Fig. 8a depicts the XRD sample results after FSP at the T6 temper.

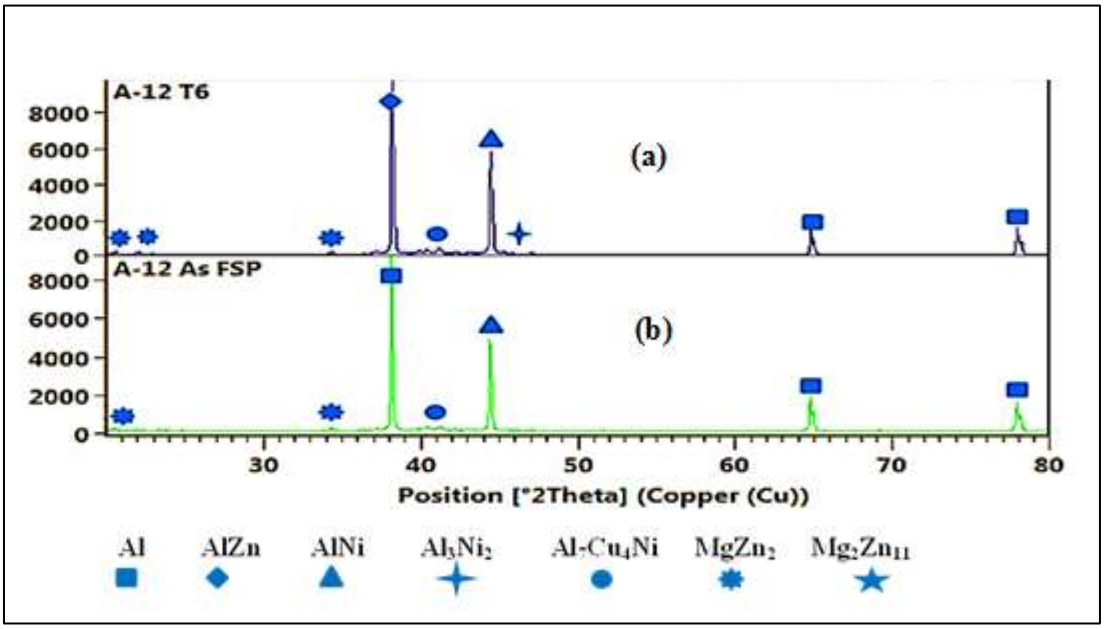


Figure 6: The XRD plots for Al matrix/reinforced A composite after FSP and T6 temper.

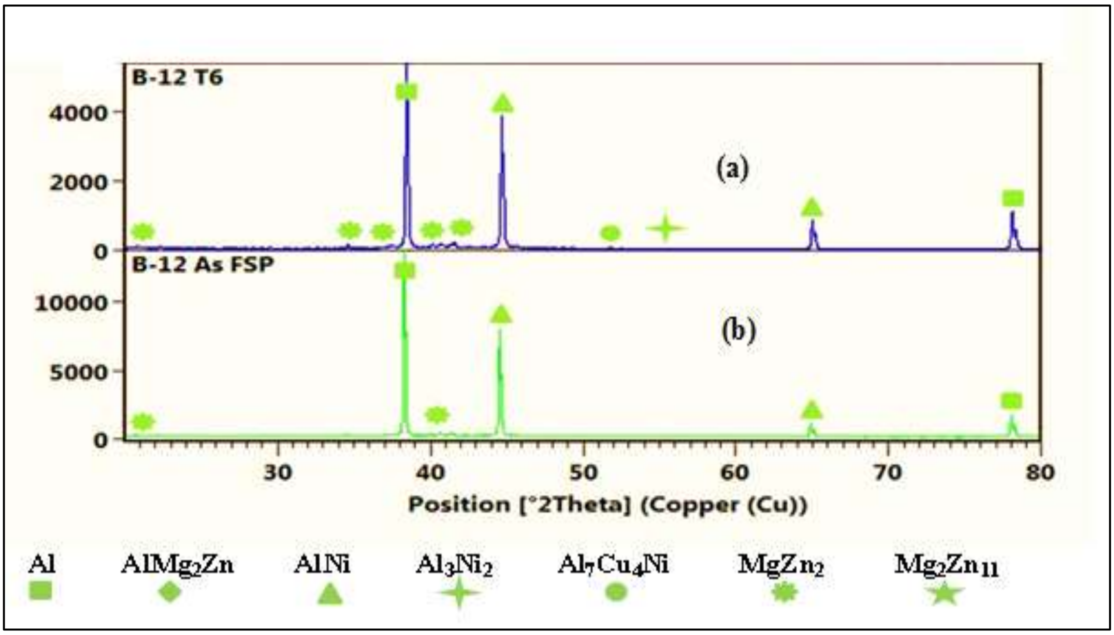


Figure 7: The XRD patterns of Al7075/reinforced B composite under the FSP and T6 temper.

The microhardness profile of the central cross sectional FSP and subsequent heat treatment specimens are shown in Fig. 9. The microhardness curves of FSP were symmetrical near the processing centerline, which were uniformly distributed at both the advancing and retreating side. A high profit was obtained from the Vickers hardness of the Al matrix/reinforced A, B, C composites that underwent FSP in the stir zone, These profits were 78, 83, and 106 HV, respectively, when compared with as-received aluminum alloy. FSP specimens with reinforced C give thehigher value of hardness than those of FSP sample underwent the reinforced B and A.

This result was attributed to the rise of % nickel in the reinforced C, which caused the formation of more fine Al-Ni compounds and uniform dispersions within the Al matrix as mentioned in the SEM, EDS, and XRD outcomes.

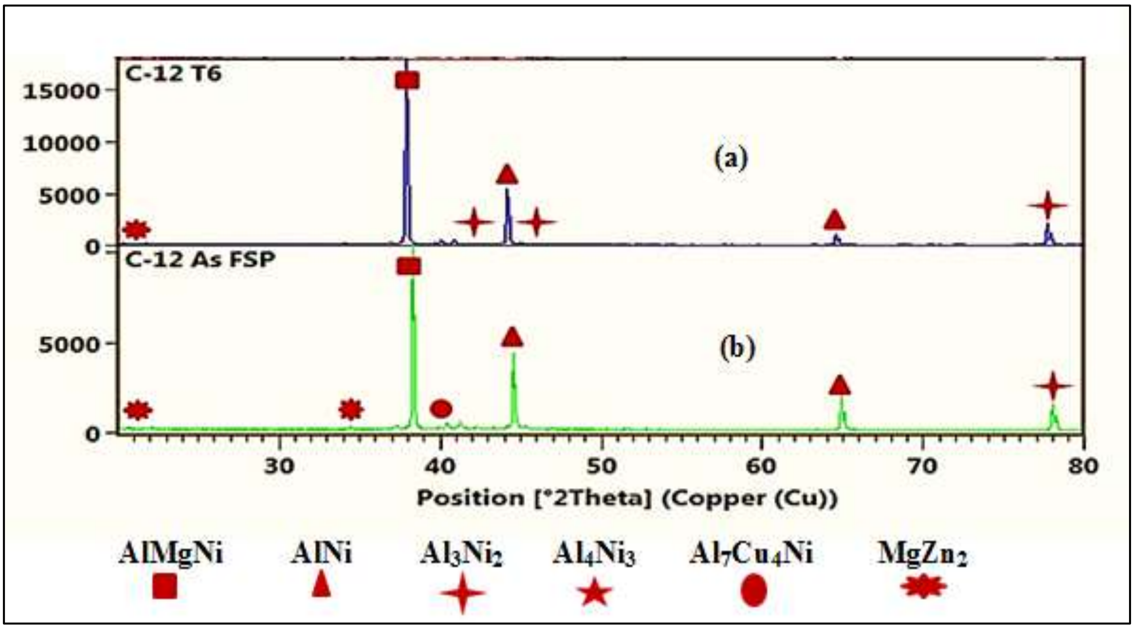
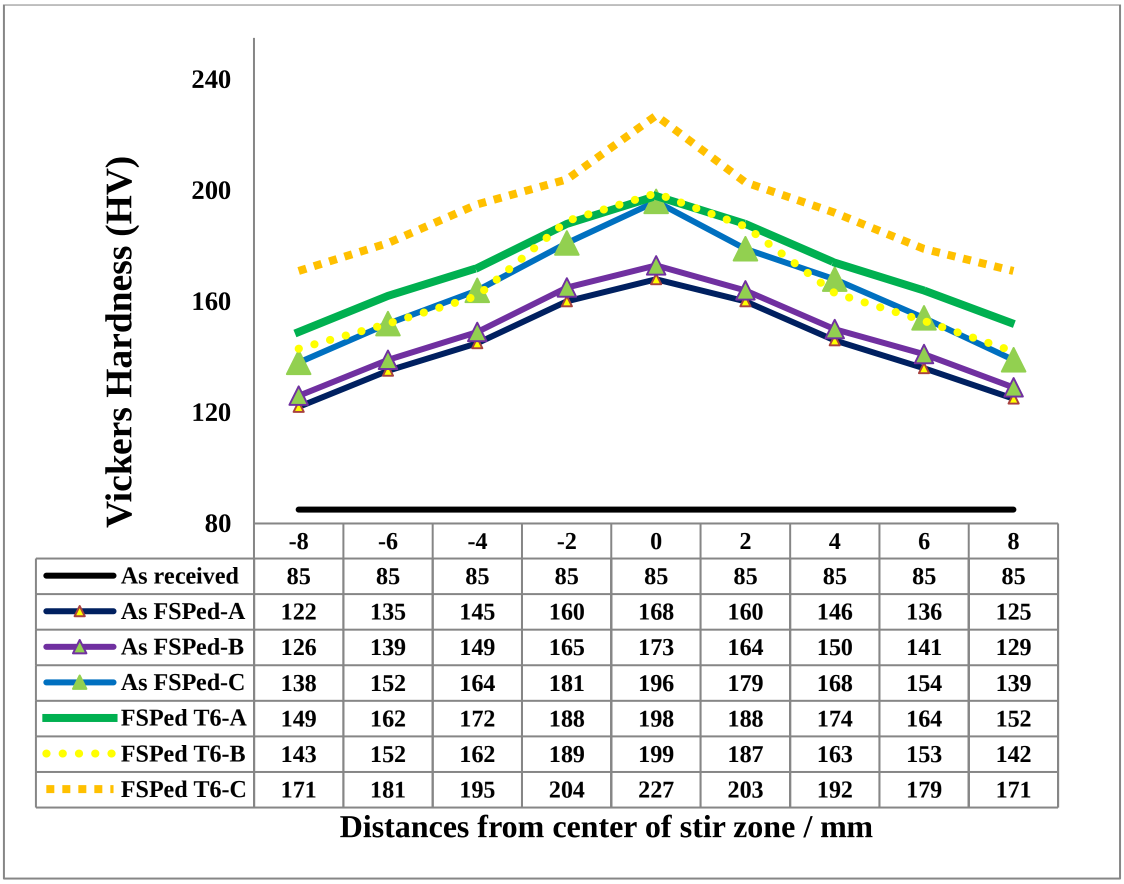


Figure 8: The XRD plots for Al/the reinforced C after FSP and T6 treatments.



	-8	-6	-4	-2	0	2	4	6	8
As received	85	85	85	85	85	85	85	85	85
As FSPed-A	122	135	145	160	168	160	146	136	125
As FSPed-B	126	139	149	165	173	164	150	141	129
As FSPed-C	138	152	164	181	196	179	168	154	139
FSPed T6-A	149	162	172	188	198	188	174	164	152
FSPed T6-B	143	152	162	189	199	187	163	153	142
FSPed T6-C	171	181	195	204	227	203	192	179	171

Figure 9: Mention the comparisons of the Vickers hardness values of Aluminum matrix/reinforcements composites after FSP and heat treatment.

In the FSP samples, a decreasing grains sizes as result to an improvement within the microhardness property, based on HallPetch foundations. The Hall–Petch equation explained the refinement of the grain with the uniformly distributed reinforcements within the matrix of alloy and especially in the stir zone [31, 32]. Therefore, several mechanisims that take place at the FSP were accountable of the hardness increase in the reinforcied composite. First, this effect was attributed to the microstructure variation (dislocation density)[33]. Second, this effect was due to the status of the FSP that had the reinforcing milled powders, which changed the circumstance. The results concluded that the higher rotational to traverse speed (ω/υ) ratio caused formation more intermetallics, such as AlNi, Al_3Ni_2,and Al_7Cu_4Ni and created alloying element phases, such as Mg_2Zn and Mg_2Zn_{11}. The homogenous distributions of these intermetallic particulates in the stiring zone play as pins the dislocations and enhances the microhardness values [34]. Meanwhile, the Al-Ni intercompounds that had precipitation hardening of alloying elements led to the Orowan mechanism. The average microhardness continuously increased when the heat treatment was applied on the FSP samples. The enhancement in property ofhardness could be refered to the precipitations hardening and uniform particle dispersion of the reinforcements during the stir zone.

Conclusions

Through the current research, the results showed which the 70% decrease in grain size compared with the as-received alloy and grain variations was attributed to the actions, profile tool, and rotation to traverse speed of the friction stir processing. SEM images indicated that intermetallics were spread out and had a good bond with the Al matrix that underwent FSP. EDS scan analysis also revealed the nickel percentage increase in the matrix of aluminum samples that underwent FSP and subsequent heat treatments. Observation of the XRD analysis presented the creation of many Al-Ni intermetallics, which became the major peaks. More significantly, the nickel mass percentage increased in the reinforced **C**, in addition to having main phases of alloying elements after heat treatments.

In summary, the mechanisms of hardness enhancement during the FSP with milled powder for aluminum alloy, a subsequent homogenization and the T6 temper were represented as the increasing dislocation density that the severe plastic deformation of FSP actions caused. The Hall–Petch equation explained the refinement of the grain with the uniformly distributed reinforcements within the stir zone. Meanwhile, the Al-Ni intercompounds that had the hardening ofprecipitation of thealloying elementsi led to Orowan mechanism.

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