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Ni/Al7475 Surface Composites Fabricated by Friction Stir Processing

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Abstract: The major object of this study is to fabrication surface Aluminum Matrix Composites (AlMCs) with optimum hardness and the adjusted microstructural as addition to high erosion resistance. Friction Stir Processing (FSP) is used to production the surface composite-Al7475 with nickel particulates as the reinforcements under several parameters. Furthermore, FSP with a rotational speed of 2500 rpm and a traveling speed of 35 mm/min was carried out on the Al7475 matrix composite. The FSPed composites samples were homogenized at 400°C/90 min+430°C/12 h +490°C/10 min and then ageing at 130°C/20 h as well as the retrogression and reaging treatment at 130°C/20 h+190°C/15 min+130°C/20 h were done. Microstructures observations were performed through Optical Microscopy-(OM) and Scanning Electron Microscopy-(SEM) as well as with energy-dispersive X-Ray Spectroscopy-(EDS) and X-Ray Diffraction-(XRD) to identify the intermetallics and precipitations phases of the Al-7475 composites specimens. Results gave that the Al7075 with the nickel reinforces underwent FSP have optimum mechanical properties and uniform microstructural compared with base Al7475-alloy. The grain refiner and uniform dispersions of the Ni-reinforcements within the Al-7475 matrix were acquired due to the optimized parameters of FSP. The more of Ni-intermetallics with the precipitations phase having after heat treatments. The roles of Ni-intercompounds and grain refinements with the hardening of precipitation led to a significant increase in Vickers hardness of the FSPed Al-composites.

Key words: Surface composite, AA7475-aluminium, nickel particles, friction stir processing, retrogression, intermetallics

INTRODUCTION

Aluminium Matrix Composites (AlMCs) is one of the widely kinds of composites materials which are consists of a mixture or a combination two or more materials (Chen *et al*., 2010; Kumar *et al*., 2014). AlMCs is exhibited significantly unusual combination of properties such as high specific strength, damping capacity, low densities, high impact resistance. AlMCs are that cannot be meeting by the conventional metallic alloys and un-reinforced alloys. Heat-treatable Al 7000-Alloys are widely used in engineering applications that especially for airframe and airfoil structures in the aircraft and aerospace as well defence armour and military vehicles (Naeem *et al*., 2014a, b). Hence, the productions of Al-alloys had been designed based on the supreme properties establishing to the specific needs of the operation conditions. However, the trial of aluminium alloys are possess low resistance to erosion-wear and to abrasive-corrosion forms which are abortive to any tribological application (Leon-Patino *et al*., 2013; Alshmri *et al*., 2014; Kala *et al*., 2014). Aluminium-matrix composites embedding particulates as reinforcements are considered as the likely solution for improving the surface of Al-alloys to the best resistances of solid particle erosion. Several techniques have been adduced for the production of alloys surface composite but Friction Stir Processing (FSP) has been recognized as one of the most promising techniques. FSP is as a solid-state processing technique which considered a fairly simple and economical process. FSP has originally developed by Ma *et al*. (2003) and Wang *et al*. (2011) based on the basic principles of friction stir welding. FSP comprises a rotating tool which has inserted in a monolithic work-piece to modify microstructure for specific property enhancement rather than joining the metals as shown in Fig. 1. The rotating tool consists of shoulder and pin. Pin is responsible for plastic deformation of the work-piece material while shoulder is responsible for frictional deformation. The

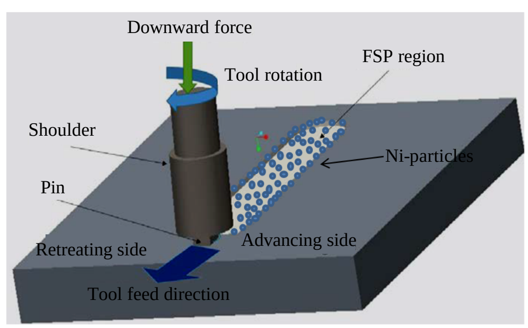


Fig. 1: Schematic for the friction stir process

stirring action of the pin generates fine grain microstructure zone which is called the Stir Zone (SZ). Therefore, FSP is essentially a thermo-mechanical process that takes material temperatures to a range (0.48 temperature melting) for plastic deformation (Naeem *et al*., 2015; Ma and Mishra, 2005). There are many reports on the fabrication of surface composites for the different Al-alloys using friction stir processing were available (Mishra *et al*., 2003; Dixit *et al*., 2007; Raaft *et al*., 2011; Devaraju *et al*., 2013).

On the other words, a fewer articles achieved about the production of surface composite of Al-7000 Alloys, such Naeem (2017) found that the possibility to creating the surface composite of Al7075-alloy reinforced with the milled Al-Ni particulates using Friction Stir Processing. They were investigated the improving mechanical properties and microstructures of the FSPed Al7075 reinforced surface composite. Rana *et al*. (2016) found that manufacturing surface composite B4C/Al7075 could be possible. They conducted to the reinforced surface Al7075 possess improved hardness as well the wear resistance 100% best than the Al7075-base metal. Bisadi and Abasi (2011) reported that fabrication of Al7075-TiB_2 surface composite using FSP as result into develop mechanical properties of composite as compare with base aluminium.

However, available many research did not covered all categories of 7xxx series of aluminium alloys that used various engineering applications. In the present research, AA 7475-aluminium alloy had been used as metal-matrix. Whereas, Nickel particles are used as reinforcement phases. The novelty of this present study is forming Al7475/Ni surface composite using several combination of tool traverse and rotation speed of FSP. Investigation of microstructural, microhardness and erosion properties had been done (Fig.1).

MATERIALS AND METHODS

Experimental procedures: The 120 mm length, 13 mm thick and 20 mm width of AA 7475 aluminium plates as the base metal (matrix). Chemical composition of aluminium plates is mentioned in the Table 1. Nickel particulates powder (particulate size D50 of 11 µm and purity, 99.50%) has been used as the reinforcements.

Fabrication of surface composite: The longitudinal V-groove with (width) 2.5 mm and (depth) 2 mm in the centerline of plate of Al7475-alloy. The reinforcements of nickel particles were filled and plugged with squeezed into the groove as much as possible and closed by means of a pin-less tool to prevent splashing them out during friction stir process. The experiments of FSP are

Table 1: The chemical composition of Al 7475-Alloys (wt.%)

Elements	Amount (wt.%)
Si	0.1
Fe	0.14
Cu	1.87
Mn	0.054
Mg	2.81
Cr	0.23
Zn	6.38
Ti	0.06
Al	Balance

Table 2: The homogenizing and heat treatments for the FSPed Al 7475 alloy

No.	Type	Treatment specifics
A	Homogenization	400°C/90 min+430°C/12 h +490°C/10 min
B	Ageing treatment (T6-temper)	130°C/20 h (Peak ageing)
C	Retrogression and Re-ageing process	130°C/20 h+190°C/15 min +130°C/20 h

conducted using the heavy-duty automatic feed CNC milling machine. FSP tool was made of stainless steel that had a threaded tapered pin profile with a flat shoulder was used. The working parameters are the rotating speed (ω) 2000-3000 rpm, 20-50 mm/min of traverse speed (υ) and 2-5 of tilt angle. In this study, parameters were selected of 2500 rpm the rotational speed, traverse speed of 35 mm/min and tilt angle of 3. FSP samples were subjected to homogenization then and artificial treatments as detailed in the steps in Table 2. Each step of the treatment was followed the quenching in cool water.

To examining different the FSPed Al-samples that are, cutting with the desired dimensions, thus, ground and polished with a 0.5-0.1 µm diamond paste at the final stage. The microstructural characterizations were carried out for the FSPed Al-composite samples by using Optical Macro and Micro-Scopay (OM) with a Scanining Elctron Micrascopy (SEM) and followed via a coupled Enrgy-Dispersve X-ray Spectrscopy (EDS). The X-Ray Diffraction (XRD) have been used for detection the different phases during processing of friction stir and various of the heat treatments. Microhardness tests were conducted for the FSPed Al-sample, using Vickers digital microhardness tester. Solid Particle Erosion (SPE) testing has been carried out at two impact angels 90 and 45° for each the FSPed composite specimens a system which was designed according to the standards in ASTM G 76 (Naeem, 2015).

RESULTS AND DISCUSSION

Figure 2 depicts the effect of the friction stir zone pass and tool rotational rate on the dispersion of the micro-sized nickel particles and formation the stir zone. Fig. 2a, b shows the macro-optical of the Al7475 samples fabricated by FSP at the tool rotational speed of 2500 rpm. The Ni particles as fine dark regions are a good

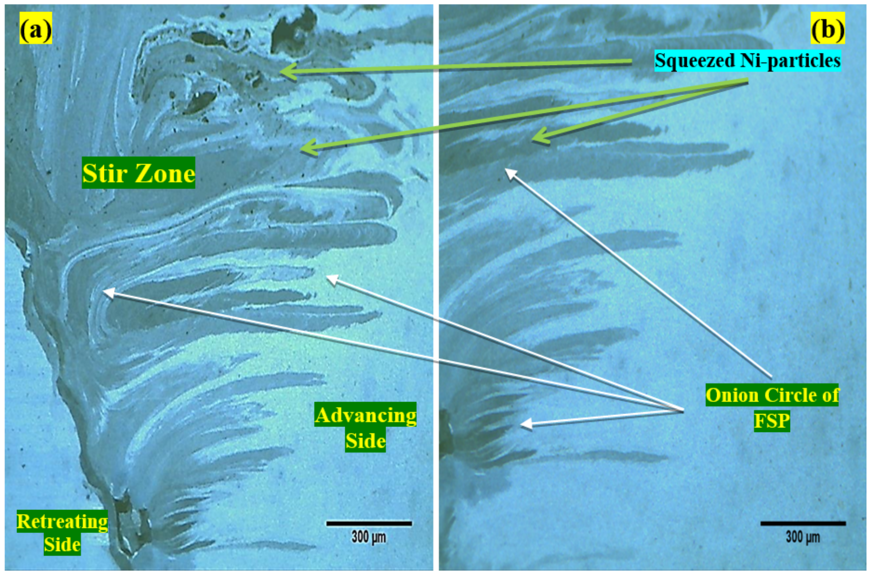


Fig. 2: a, b) Macro graphs of Al-specimens under friction stir producted at 2500 rpm, rotational speed with traverse speed of 35 mm/min

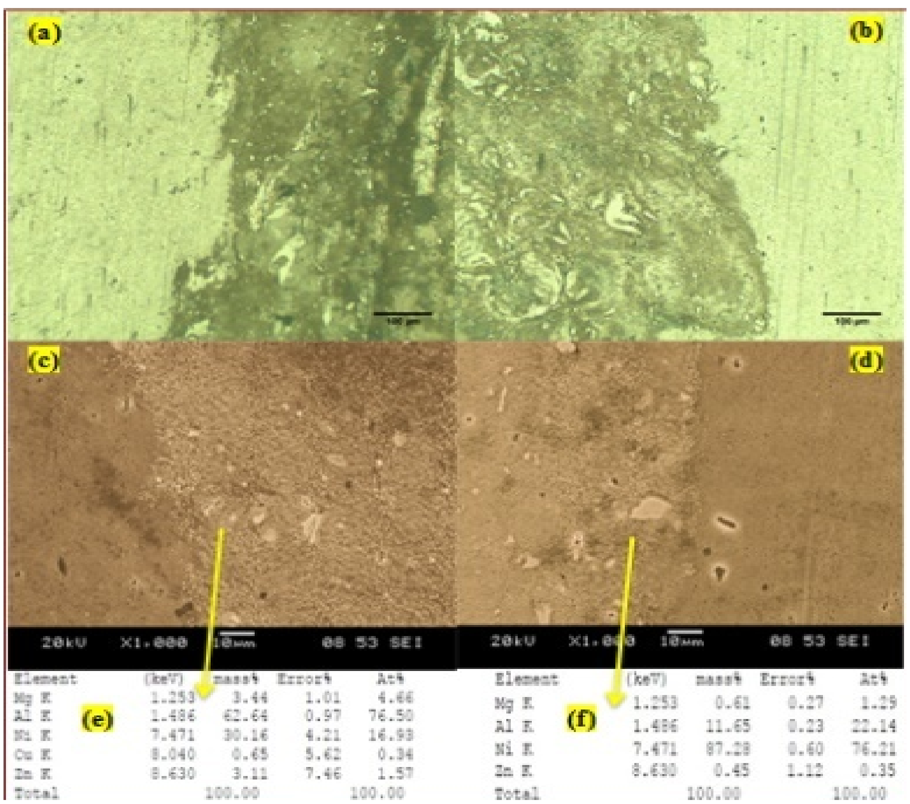


Fig. 3: a, b) OM: c, d) SEM micrograph and e, f) EDS microanalysis of the cross section of FSPed Al 7475composite layer

distributed in the Stir Zone and thus composite layer fabrication was successful. Shape of the onion circle within SZ depends on the parameters, tool geometry (threaded trapped) workpiece temperature and thermal conductivity of the material as mentioned (Naeem, 2017; Rana *et al*., 2016; Bisadi and Abasi, 2011). Figure 3a, b optical microstructure of the FSPed Al 7475 surface composite that reveal the homogenize of

Ni-particles clusters within the base metal. This improving distribution of Ni-reinforces and a change in the material flow due to the increase in heat input which result of the tool rotational speed in the Stir Zone (SZ) (Dixit *et al*., 2007; Raaft *et al*., 2011; Devaraju *et al*., 2013). The FSPed Al-sample that Ni-reinforces are dispersed easily as well the defect-free and without any great overcrowded in the Stir Zone as in Fig. 3 c-d. The EDS analysis in Fig. 3 e-f showed that evidence for the great presence of Ni-component in elements for the Al7475 matrix.

Figure 4 displayed the stir region that described by equi-axed and fine grains because of the severe plastic deformation and frictional heating leading to recrystallization in the SZ. Creation of the finer grains in the Al7475 alloys under the FSP was because of to the dynamic recovery, geometric dynamic recrystalization and dis-continous dynmic recrystalliation (Naeem *et al*., 2015). Addition of Ni micro-sized particles reduced the grain size of these Al-samples that referring to the grain boundary pinning of Ni-reinforcement and the second-phase particulates.

Figure 5 a-b shows the SEM images of the FSPed Al7475 specimens with nano-sized Ni after the series of homogenizing at 400°C/1.5 h then followed 430°C/12 h and then 490°C/10min as addition to the againg treatment at 130°C/20 h. Regarding that (Fig. 5) the stir zone of the FSPed Al7475 surface composite was plenty rich-nickel intercompounds with regular distribution particulates and very good interfacing cohesion without defects in the mixture. The EDS microanalysis results shown in Fig. 5b depicted that the increasing volume fraction of Ni content within the matrix. Nanoparticles of nickel within matrix that were exhibited a better distribution in the Stir Zone acting as numerous obstacles against the movement of grain boundaries preventing grain growth (Naeem, 2015).

On the other words, the series of heat treatments carried outing that is the main governing mechanisms for the increasing generate of intermetallics-Ni. Increasing the tool rotational speed causes high temperature exposure which increases the grain size of these specimens (Naeem (2017; Rana *et al*., 2016; Bisadi and Abasi 2011; Naeem 2015) but homogenous dispersion of particles in higher tool rotational rates improves the pinning effect in the SZ restricting, the grain growth (Naeemb *et al*., 2015; Bisadi and Abasi 2011). These two opposite factors cause small changes in the grain size of samples.

The X-ray differaction has performed to identify the all phases of the FSPed Al-7475 specimens with reinforces of Ni-particles and subsequent heat treatments. The XRD patterns are given in Fig. 6 that verified the presence Ni-intermetallics as well precipitation phases within As FSP-sample.

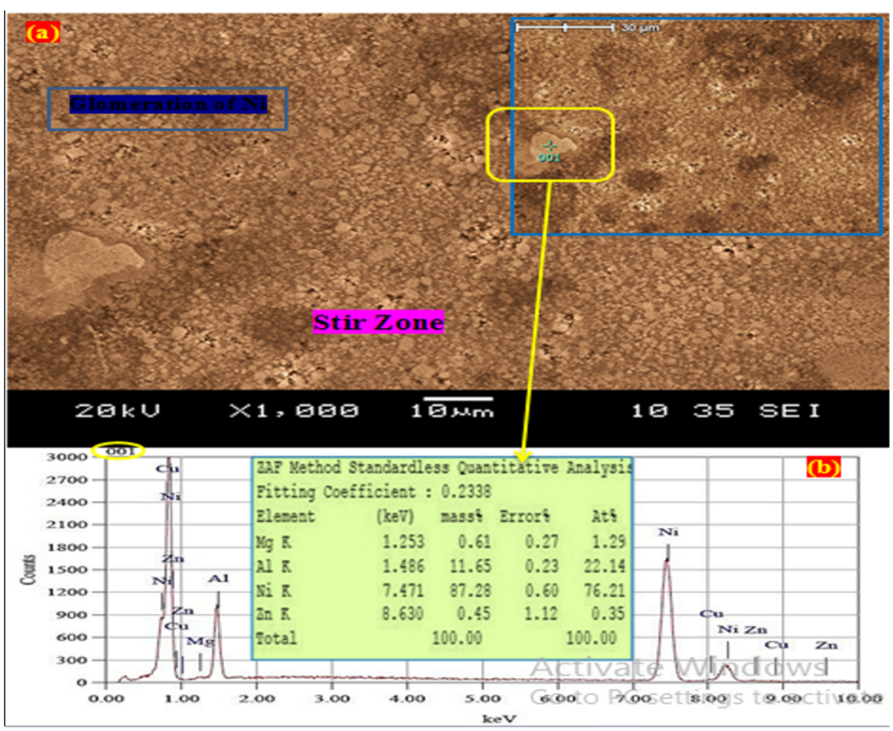


Fig. 4: a) SEM micro graph and b) EDS analysis of the FSPed Al7475 composite in the stir zone

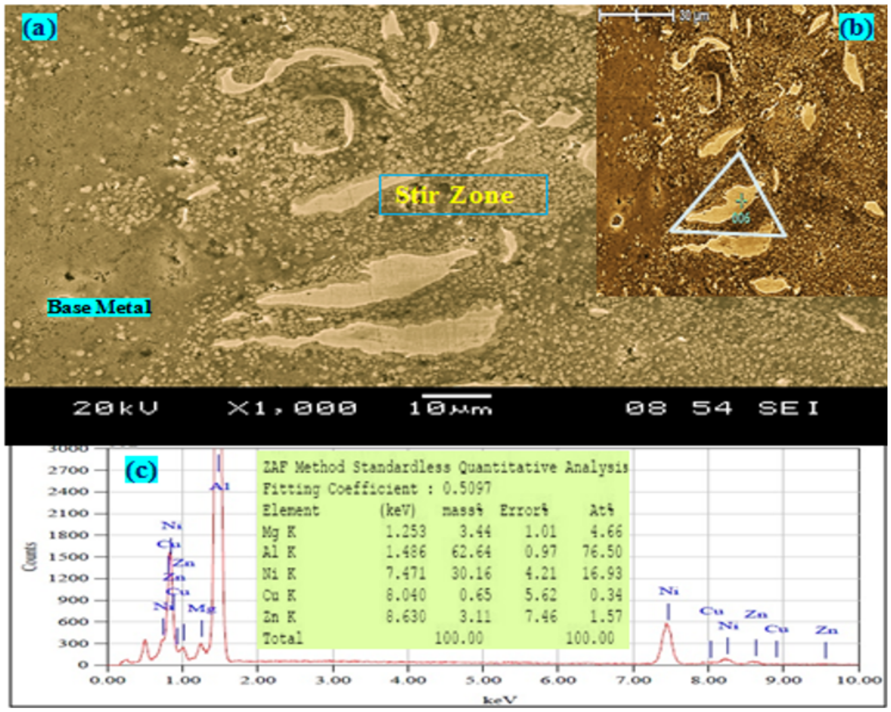


Fig. 5: a) SEM micrograph and b) EDS of the stir zone in FSPed Al 7475 surface composite

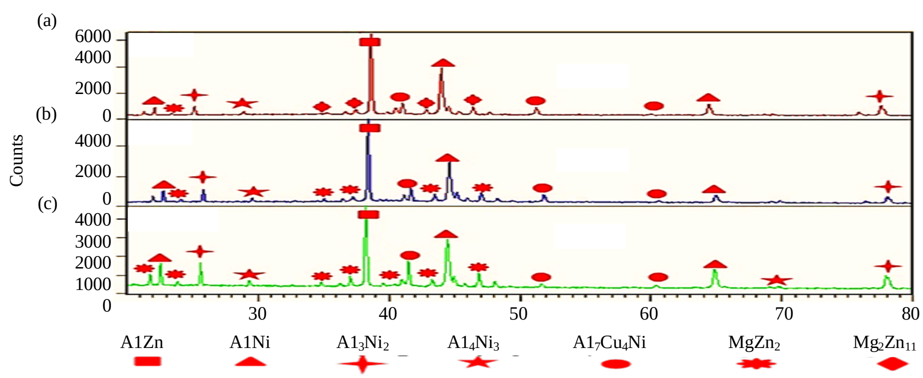


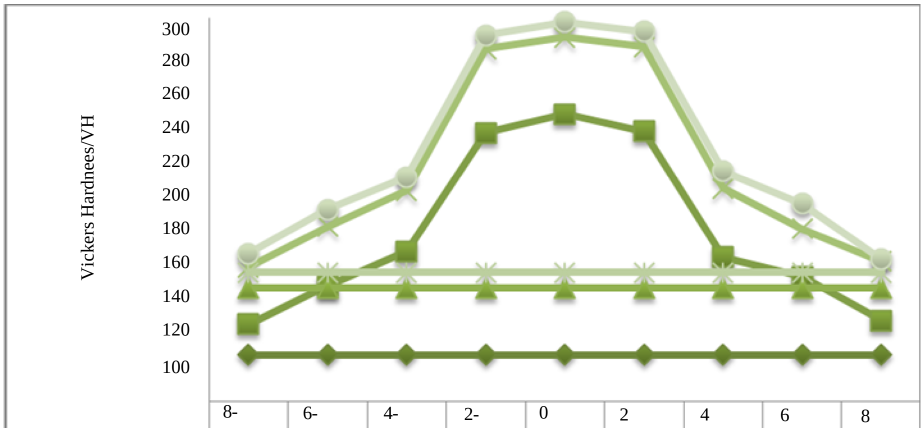
Fig. 6: XRD pattern of Al 7475 surface composite reinforced Ni-particulates underwent the friction stir processing after of heat treatments with the aging at T6 as well RRA retrogression and reaging

The primary eutectic system of the As FSP sample mainly consisted of major peaks which were the AlZn-peak, followed the phase AlNi, then the Al_7Cu_4Ni, Al_3Ni_2 intermetallics addition to $MgZn_2$ and Al_4Ni_3 compounds.

Figure 7a, b shows the XRD analysis of the FSPed Al-7475 alloy after age tempering at T6. Figure shows that the product was composed of AlZn and AlNi with Al_3Ni_2 as the major phases and a small amount of Al_7Cu_4Ni and Al_4Ni_3 as the residual phases. $MgZn_2$ as a major phase was present because of the subsequent homogenization in the aging treatment at T6 as was mentioned previously (Naeem *et al.*, 2014; Naeem, 2015).

Figure 8c shows the XRD patterns of the FSPed Al -7475 alloy after Retrogression and Re-ageing process130°C/20 h+190°C/15 min+130°C/20 h. Figure 6 reveals that the principal peaks of a Al and the Mg_2Zn_{11} originate from $MgZn_2$ and Al_4Ni_3 phases. In RRA, most of the $MgZn_2$ phase was transformed into other forms such as Mg_2Zn_{11} (Naeem *et al.*, 2014; Naeem, 2015).

The figure shows the main peaks of the AlZn and AlNi phases and a small peak for Al_3Ni_2 in addition to a minute Al_7Cu_4Ni phase. The significantly increasing reaction between the Ni-rich particles and Al matrix evidently led to the appearance of numerous AlNi phases. This phenomenon is caused by the high temperature



	8-	6-	4-	2-	0	2	4	6	8
B.M	124	124	124	124	124	124	124	124	124
As FSPed	140	160	178	240	250	241	175	165	142
B.M-T6	159	159	159	159	159	159	159	159	159
FSPed-T6	170	191	210	284	290	285	211	190	173
B.M-RRP	167	167	167	167	167	167	167	167	167
FSPed-RRP	177	200	217	291	298	293	200	203	174

Fig. 7: Mention the comparisons of the Vickers hardness values of Al 7475 before and underwent the friction stir processing with the aging at T6 as well RRA retrogression and reaging

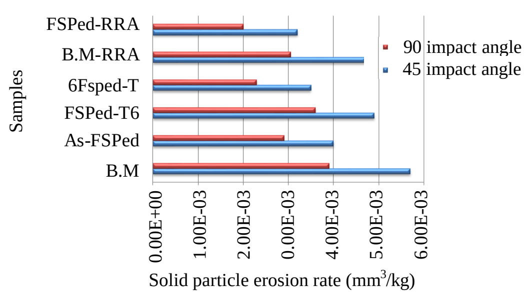


Fig. 8: Erosion rates of the treated Al7475-alloy and FSPed samples at impact angles of 90 and 45°

generated during FSP. The temperature of the SZ ranged from 400-500°C during the FSP of the Al-Zn-Mg-Cu alloy (Ma and Mishra, 2005). In summary, several AlNi intermetallic phases of were generated within a range of temperature, based on the AlNi phase diagram (Zolotorevsky etal., 2007).

Microhardness profile of the Al 7475/Ni-particulates specimens produced FSP (at the rotational speed of 2500 rpm and traverse speed of 35 mm/min) and the subsequent heat treatment are shown in Fig. 7. Vickers hardness values of the FSPed Al-specimens are increased as compared with hardness values of the base Al-alloy. A high Vickers profit was obtained from the FSPed Al7475 composite reinforced nickel in after RRA treatment. In the FSP samples, a decreasing grains sizes as result to improvement hardness, based on Hall-Petch foundations. FSPed specimens with Ni particles show higher microhardness than those of the heat-treated base Al-alloy specimens. 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 (Devaraju *et al*., 2013; Naeem, 2015, 2017). There are many mechanisms which occurred during FSP are responsible for the improving of hardness in the reinforced composites.

When the rotational speed increases, more intermetallic phases are formed. The homogenous distributions of these intermetallic particulates in the stiring zone play as pins the dislocations and enhances the microhardness values (Naeem *et al*.,2015).

Meanwhile, the Al-Ni intercompounds as well as the precipitation hardening of alloying elements of Aluminum 7475 alloys led to the Orowan mechanism (Naeem, 2015, 2017) differences between the coefficients of thermal expansion of the reinforcements and the matrix increase the dislocation density due to the formation of residual stresses during cooling time causing an increase in the hardness of the SZ (Devaraju *et al*., 2013; Naeem, 2017;

Rana *et al*., 2016; Bisadi and Abasi 2011). The enhancement in property of hardness could be refered to the precipitations hardening and uniform particle dispersion of the reinforcements during the Stir Zone. Decreasing the particles size causes a higher level of distribution of nanoparticles and therefore, the intense grain boundary pinning enhances the hardness values of nanocomposites (Naeem *et al*., 2015; Naeem, 2015, 2017).

The erosion rates of the FSPed samples and BM Al-7475 alloy that underwent heat treatments at 90° angle is given in Fig. 8. There are reductions in erosion rates for the As FSPed, FSPed-T6 and FSPed-RRA respectively as opposed to these values for sintered samples of B.M Al-alloy, B.M-T6 and B.M-RRA which underwent similar heat treatment conditions. The improvement in erosion resistance for samples of FSPed can be ascribed to the role of friction stir processing and nickel-particulates reinforcement in producing homogenized and enhanced hardness surface composite.

CONCLUSION

In the current investigation, the surface composite layer of the FSPed Al 7475 was successful manufactured and the optimum reinforcing Ni-particulates uniformed distributions within the matrix of the SZ were accomplished. Through the research, the results showed that FSPed Al 7475 with Ni-particles improved the grain refining as compared to base Al-alloy.

Observations microstructural indicated that having plenty of Ni-intermetallics were dispersed and excellent interfacing without defects within the Al 7475 matrix under FSP subsequent heat treatments. XRD analysis presented the creations of Ni-Al intercompounds that became the main peaks after heat treatments. The FSPed Al 7475 underwent RRA treatment have higher vickers hardness as well as the best resistance to solid particle erosion compared with non-FSPed samples under the same heat treatments. In summary, improving of Vickers hardness of the FSPed samples subsequent heat treatments represented as the significantly increase densities of dislocation due to the severe plastic deformation of FSP. As addition to Ni-particles which acts as pin to dislocation movements. Meanwhile, the Ni-compounds as well as the precipitations phases of the alloying elements led to strengthening.

REFERENCES

Alshmri, F., H.V. Atkinson, S.V. Hainsworth, C. Haidon and S.D.A. Lawes, 2014. Dry sliding wear of Aluminium-high silicon hypereutectic alloys. Wear, 313: 106-116.

Bisadi, H. and A. Abasi, 2011. Fabrication of Al7075/TiB2 surface composite via friction stir processing. Am. J. Mater. Sci., 1: 67-70.

Chen, C.F., P.W. Kao, L. Chang and N.J. Ho, 2010. Mechanical properties of nanometric Al_2O_3 particulate-reinforced A_l-$Al_{11}Ce_3$ composites produced by friction stir processing. Mater. Trans., 51: 933-938.

Devaraju, A., A. Kumar, A. Kumaraswamy and B. Kotiveerachari, 2013. Influence of reinforcements (SiC and Al_2O_3) and rotational speed on wear and mechanical properties of Aluminum alloy 6061-T6 based surface hybrid composites produced via., friction stir processing. Mater. Des., 51: 331-341.

Dixit, M., J.W. Newkirk and R.S. Mishra, 2007. Properties of friction stir-processed Al 1100-NiTi composite. Scr. Mater., 56: 541-544.

Kala, H., K.K.S. Mer and S. Kumar, 2014. A review on mechanical and tribological behaviors of stir cast Aluminum matrix composites. Procedia Mater. Sci., 6: 1951-1960.

Kumar, A., M.M. Mahapatra, P.K. Jha, N.R. Mandal and V. Devuri, 2014. Influence of tool geometries and process variables on friction stir butt welding of Al-4.5% Cu/TiC in situ metal matrix composites. Mater. Des., 59: 406-414.

Leon-Patino, C.A., E.A. Aguilar-Reyes, E. Bedolla-Becerril, A. Bedolla-Jacuinde and S. Mendez-Diaz, 2013. Dry sliding wear of gradient AlNi/SiC composites. Wear, 301: 688-694.

Ma, Z.Y. and R.S. Mishra, 2005. Development of ultrafine-grained microstructure and low temperature (0.48 Tm) super plasticity in friction stir processed Al-Mg-Zr. Scr. Mater., 53: 75-80.

Ma, Z.Y., R.S. Mishra, M.W. Mahoney and R. Grimes, 2003. High strain rate super plasticity in friction stir processed Al-Mg-Zr alloy. Mater. Sci. Eng. A., 351: 148-153.

Mishra, R.S., Z.Y. Ma and I. Charit, 2003. Friction stir processing: A novel technique for fabrication of surface composite. Mater. Sci. Eng. A., 341: 307-310.

Naeem, H.T., 2015. The effect of Nickel, Cobalt and Tin on the evolution of microstructural and mechanical properties of Al-Zn-Mg-Cu alloys (7000 series) via., casting, powder metallurgy and friction stir processing techniques. Ph.D Thesis, Al Muthanna University, Samawah, Iraq.

Naeem, H.T., 2017. Characterizations particulates of crushed particles (Al-Zn-Mg-Cu-Ni) for fabrication of surface composites Al-Alloy using friction stir processing route. Muthanna J. Eng. Technol., 1: 56-65.

Naeem, H.T., K.S. Mohammed and K.R. Ahmad, 2014b. The role of cobalt and nickel intermetallic phases on the mechanical properties and microstructure evolution of Al-Zn-Mg-Cu alloys. Mater. Res., 17: 1663-1676.

Naeem, H.T., K.S. Mohammed and K.R. Ahmad, 2015. Effect of friction stir processing on the microstructure and hardness of an Aluminum-Zinc-Magnesium-Copper alloy with nickel additives. Phys. Met. Metall., 116: 1035-1046.

Naeem, H.T., K.S. Mohammed, K.R. Ahmad and A. Rahmat, 2014a. The influence of nickel and tin additives on the microstructural and mechanical properties of Al-Zn-Mg-Cu Alloys. Adv. Mater. Sci. Eng., 2014: 1-10.

Raaft, M., T.S. Mahmoud, H.M. Zakaria and T.A. Khalifa, 2011. Microstructural, mechanical and wear behavior of A390/graphite and A390/Al2O3 surface composites fabricated using FSP. Mater. Sci. Eng. A., 528: 5741-5746.

Rana, H.G., V.J., Badheka and A. Kumar, 2016. Fabrication of Al7075/B4C surface composite by novel Friction Stir Processing (FSP) and investigation on wear properties. Procedia Technol., 23: 519-528.

Wang, K., F.C. Liu, Z.Y. Ma and F.C. Zhang, 2011. Realization of exceptionally high elongation at high strain rate in a friction stir processed Al-Zn-Mg-Cu alloy with the presence of liquid phase. Scr. Mater., 64: 572-575.

Zolotorevsky, V.S., N.A. Belov and M.V. Glazoff, 2007. Casting Aluminum Alloys. Vol. 12, Elsevier, Amsterdam, Netherlands, ISBN:9780080453705, Pages: 530.