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Wear behavior and Microstructural Characterization of AA7075/MWCNT Surface Composites fabricated through Friction Stir Processing

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Abstract : *There is huge demand for Aluminum Alloy 7075 in the field of aerospace and automobile industry because of its high strength to weight ratio. However, its surface properties such as hardness and wear resistance are relatively poor and hence constraining its usage as a structural material. In the present work, an attempt is made to synthesize AA7075 Matrix Surface Composites (AMSC) through Friction Stir Processing (FSP). The Multiwall Carbon Nanotubes (MWCNT) are used as reinforcement particles. The FSP process parameters such as Rotational Speed (RS), Traverse Speed (TS) and No. of Passes (NP) are varied in order to obtain a variety of AMSC billets with enhanced mechanical and micro-structural properties. Subsequently, few experiments are carried out to analyze and access wear behavior and micro-structure of the AMSC billets. Taguchi technique is adopted for the design of experiments and ANOVA is applied to identify the most influencing FSP process parameter with respect to the response variables. It has been observed that average grain size of AMSCs is refined to a great extent and the wear resistance of the AMSCs is significantly influenced by the number of passes of FSP.*

Keywords: *Surface Composites, FSP, MWCNT, Grain size, Wear Resistance.*

I. Introduction

Aluminum and its alloys are the most popular materials in the aerospace and automobile industries. The 7xxx series of Aluminum alloys mainly consist of Zn, Mg and Cu as the major alloying elements. Aluminum Alloy 7075 (AA7075) has got good specific strength, toughness and fatigue resistance. However, its usage is constrained by the poor surface properties such as hardness and wear resistance. Researchers are trying to enhance the surface properties of AA7075 by various techniques such as Excimer laser treatment, Plasma treatment and surface coatings etc. The surface properties of the aluminum alloys may also be enhanced by dispersing reinforcement particles during server plastic deformation. This can be achieved by Friction Stir Processing (FSP).

FSP is basically a clone of Friction Stir Welding (FSW), used as a technique for grain refinement and surface modifications of metals, specifically non-ferrous alloys. Thus, FSP is surface modification technique, widely carried out below the melting point of metal/alloy being processed. The FSP is carried out by a cylindrical tool that consists of a shoulder and project pin of required geometry. Very similar to FSW, in FSP also the tool is rotated and plunged into the work surface until the shoulder had an abrasive action. Heat is generated due to the friction between the tool and base metal. The heat thus developed along with the axial force exerted, deforms the base metal surface by the combined stirring action of the rotating tool. Subsequently, the tool is traversed over surface to be modified. Three different zones are formed during Friction Stir processing are Stir Zone (SZ) or Nugget Zone, Thermo-mechanically affected zone (TMAZ), Heat Affected Zone (HAZ) along with the Unaffected or Parental Material. The side of the processing tool where surface motion is in the same direction as the travel direction is referred to as the advancing side. The opposite side, where surface motion opposes the travel direction, is referred to as the retreating side. When the processed zone reaches to normal temperature, it forms a defects free dynamically recrystallized surface composite with fine grained microstructure. Thus, the mechanical properties such as microhardness, wear resistance, corrosion resistance, fatigue and creep resistance of the surface composites are improved to a great extent.

In the present work, an attempt is made to reinforce the Multi Wall Carbon Nanotubes (MWCNT) into the surface layers of AA7075 by FSP which in fact forms the surface composite layer atop the alloy. MWCNT are very popular as the reinforcements for fabricating composites. The special properties they afford high elastic modulus, specific strength and good electrical and thermal conductivity. The MWCNT can be embedded into the composites by many methods viz., spark plasma sintering [1], Plasma spraying [2] and high energy ball milling[3] etc. Mishra *et al.* [4] reported to achieve the hardness of AA5083 to 173 HV, almost double the hardness of the base alloy by using the FSP technique. Hossein Bisadi *et al.* [5] investigated the effects of processing parameters on particle dispersion and hardness in AA7075 reinforced with TiB_2 micro particles. They reported to have enhanced the hardness up to 50% compared to that of parent metal.

II. Experimental Procedure

2. Materials and Methods:

The details of materials, machine and methods applied in the fabrication process of AA7075/MWCNT Surface Composites are described below:

2.1 Matrix Material

The matrix material i.e., AA7075 billets for the fabrication of surface composites are cut to a standard dimension of 100X75X6 mm and made ready for the processing. The chemical composition of the substrate material AA7075 is as follows:

Table 1: Chemical Composition of AA7075 (by wt%)

Si	Fe	Cu	Mn	Mg	Cr	Zn	Al
0.4	0.5	1.5	0.3	2.4	0.23	5.6	Balance

2.2 Reinforcement Particles:

Multiwall Carbon Nanotubes (MWCNTs) have attracted much attention as the reinforcement particles for fabricating composites. MWCNTs possess excellent modulus of elasticity, Tensile strength, good thermal and electrical properties etc. MWCNT dispersion into the matrix materials is a typical task, as there is a possible formation of bundled clusters of the reinforcement. This is usually happens because of their large aspect ratios and the strong Vander forces [6]. The clustering of MWCNTs in the composites results into poor mechanical properties. Therefore, FSP not only eliminates the clustering formation difficulty but also provides unique opportunity to embed 'wrought' nano-structures in to 'cast' components by a localized modification [7].

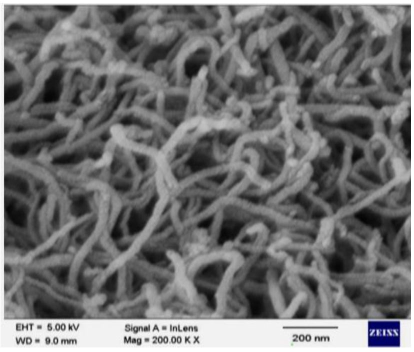

Fig1 SEM image of MWCNT

The MWCNT used for this work were procured from M/s United Nanotech Pvt. Ltd., Bangalore, India. The technical data sheet of MWCNT is shown in table below.

Table 2. Date sheet of MWCNT

MWCNT	Description
Production method	Chemical Vapor Composition
Available form	Black Powder
Diameter	Avg. outer diameter: 5-20 nm
Length	Avg. Length 1-10 microns
Nanotubes purity	>98%
Metal particles	<1%
Amorphous carbon	<1%
Specific Surface Area	330 Sq.m/g
Bulk density	0.2 – 0.35 g/cc

The quantity of MWCNT in the present work was taken as 5% by weight.

2.3 Fabrication through Friction Stir processing

The fabrication process was carried out on Vertical Milling Machine, HMT make FN-2, 10HP, 3000 rpm. The FSP tool was made of H13 tool steel with shoulder diameter of 24 mm, pin dimensions are 6 mm length and 4 mm diameter. The FSP tool was heat treated to about 58 HRC. A maximum axial force of 5 KN is given during the machining process. The FSP tool rotational speed ranges from 900 to 1200 rpm and the traversing speed 32 to 50 mm/min. The number of passes of the FSP was varied as single double and triple consecutively as per the design table. A special fixture was used to keep the work piece intact on the machine table during FSP. The samples were processed to a depth of 5 mm at different process conditions by varying the processing speeds, traverse speeds with single, double and triple number of passes as required by the design matrix.

2.3.1 The Design of Experiments for the fabrication process

The Design on experiments was framed by using Taguchi technique. The number of process parameters and their levels are shown in following table.

Table 3. FSP process parameters and their levels

Process Parameters	Levels		
	1	2	3
Tool Rotational Speed (rpm)	900	1120	1200
Traversing Speed (mm/min)	32	40	50
No. of passes	One	Two	Three

The input FSP process parameters are arranged as per the Taguchi L9 Orthogonal Array. The Design Matrix, L9 Orthogonal Array for the above mentioned parameters is as follows:

Table 4. Process parameters as per the L9 Orthogonal Array Design Matrix

Exp. No.	Speed (rpm)	Traverse speed (mm/min)	No. of Passes
1	900	32	1
2	900	40	2
3	900	50	3
4	1120	32	2
5	1120	40	3
6	1120	50	1
7	1200	32	3
8	1200	40	1
9	1200	50	2

To start with the process, the plates were first machined for a surface groove of dimension 1mm width and 2 mm depth, at the centerline of the billet so as to hold the reinforcement particles. Subsequently, the slot was filled with measured quantity of MWCNT particles very carefully without having any spill of powder around surface slot. The initial capping pass of FSP was carried out on each billet to prevent possible scattering of reinforcement powder during the process. The pinless FSP tool was used for the initial capping pass. Subsequently, the Friction Stir processing was carried out as per the design matrix to fabricate the Aluminum Matrix Surface Composites. The nine number of AMSC billets were obtained as per the Design of Experiments. The AMSC billets were then prepared suitably to carryout wear tests and microstructure analysis. Wire EDM was used to cut the AMSC billets as per the tests required dimensions. Friction stir processing of AA707/MWCNT surface composited being fabricated on Vertical Milling Machine is shown in the fig.2 below.

Fig. 2 The FSP carried out on AA7075 billet

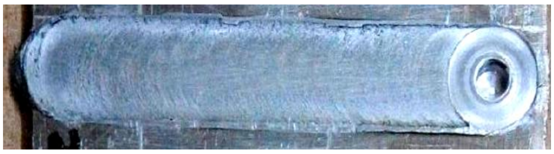

Fig. 3 Typical AA7075/MWCNT Surface composite billet fabricated through FSP

2.4 Microstructural study of the AMSC

The surface composites are formed due to severe plastic deformation and subsequent dynamic recrystallization at the stir zone. Hence, in this work the stir zone was specifically considered for the microstructural study. The distribution of the nano-particles plays a very important role in the deciding properties of the surface composites. The interface bonding resulted between the matrix material (AA7075) and the dispersed MWCNT also have a significant effect on the mechanical and tribological properties of the surface composites. The metallographic specimens were prepared as per standards practices. The specimens were prepared by mechanical grinding followed by polishing using alumina powder. The Keller's reagent (1% HF + 1.5% HCl + 2.5% HNO_3 + 95% distilled water by volume fraction) was used for etching the specimens. Microstructural study was carried out using Optical microscope OLYMPUS-BX51M make.

2.5 Wear behavior of the AMSC

The dry sliding wear behavior of AMSC billets and the base metal AA7075 was studies on a Pin-on-disc wear apparatus so as to evaluate the wear rate of samples. The specimen for wear tests were cut prepared by WEDM according to ASTM G99 standard. The wear test specimen was made of prismatic round pin with 5mm diameter and 30 mm length. The test was carried out at room temperature on Wear rest apparatus, DUCOM make. Rotating disc against the loaded pin setup was adopted to determine the wear rate. The Disc material was AISI 1045 steel, hardened and heat treated to 60 HRC. The objective of this work was to determine the effect of FSP process parameters on the wear rate of the resulting AMSC billets, hence, the wear test parameters were kept const during all the wear tests. The parameters of wear test include load (30N), speed (1.5 m/s), temperature (31ºC), roughness (Ra=0.2 µm) and sliding distance (100 m). Before the wear test, each specimen was ground down to 1000 grit abrasive paper. All wear test specimens were cleaned in acetone and weighed to an accuracy of 0.001g prior to testing. The wear rate (WR) is calculated using the following formula

$$\boldsymbol{WR}\ \mathbf{(mm^3/m)} = [\nabla \boldsymbol{h}\ \mathbf{(mm)}\ \mathbf{X}\ \boldsymbol{A}\ \mathbf{(mm^2)}]\ /\ \boldsymbol{D}\ \mathbf{(m)}$$

Where, WR = Volumetric Wear rate in mm^3/m

∇h = Variation in specimen's height in m

A = Specimen face area in mm^2

D = Sliding distance in m

III. Results and Discussion

The Friction stir processing results were obtained by carrying the experiments on consideration of the process parameters as per the L9 Orthogonal Array. The process parameters such as tool rotational speed, traverse speed and the number of passes were varied to obtain different types of AMSC billets. The micro structural study of the billets was carried out. Subsequently the wear tests were also carried out on all the nine specimens.

3. Wear Rate:

Wear rate of the base AA7075 alloy was recorded as 13.7 (10^{-3}) mm^3/m. It has been observed that there was a significant fall in the wear rate of all the nine AMSC billets when compared to that of base AA7075 alloy. This may be because of the dispersion of MWCNTs particles into the surface layers of AA7075 during FSP.

The results obtained are tabulated as follow:

Table 5 Results of Wear Rate Test on the all nine AMSC billets

Exp. No.	Rotational Speed (rpm)	Traverse speed (mm/min)	No. of passes	WR (10^{-3}) mm^3/m
1	900	32	1	8.2
2	900	40	2	9.1
3	900	50	3	7.4
4	1120	32	2	7.9
5	1120	40	3	7.0
6	1120	50	1	7.8
7	1200	32	3	7.7
8	1200	40	1	7.6
9	1200	50	2	7.8

The surface plots depicting the variation of Wear rate with respect to FSP process parameters RS, TS and NP.

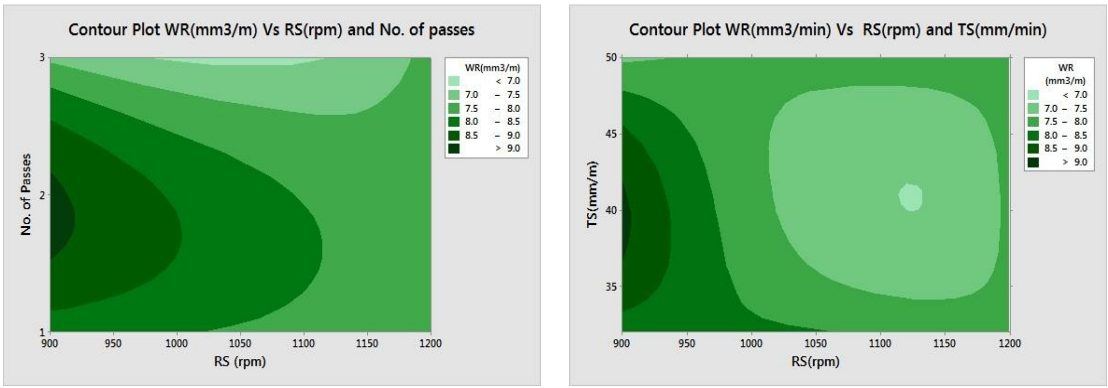

Fig. 4 Contour plots showing the effect of RS, TS and NP on the Wear Rate

3.1 Signal to noise ratios (S/N):

The Signal to Noise (S/N) ratios of the Wear rate (response variable) were obtained using the standard equations of S/N_{LB}, Lower the Better condition from MINITAB-17 software.
For the Wear Rate the Lower the better condition is suitably applied for calculating Signal to Noise (S/N) ratios using the following equation.

$$S/N_{LB} = -10\log_{10}[\Sigma \frac{Y_{ij}^2}{n}]$$

Y_{ij} is the value of the response
'j' in the i_{th} experiment condition, with i= 1,2,3,...n;
j = 1,2,3,....k and S_2 are the sample mean and variance

Table 6 Signal to Noise (S/N) ratios for the Wear Rate response variable

Exp. No.	Rotational Speed (rpm)	Traverse speed (mm/min)	No. of passes	WR (10^{-3}) mm^3/m	S/N ratio (dB)
1	900	32	1	8.2	-18.276
2	900	40	2	9.1	-19.181
3	900	50	3	7.4	-17.385
4	1120	32	2	7.9	-17.953
5	1120	40	3	7.0	-16.691
6	1120	50	1	7.8	-17.842
7	1200	32	3	7.7	-17.729
8	1200	40	1	7.6	-17.613
9	1200	50	2	7.8	-17.842

The Signal to Noise ratios were calculated using the Minitab-17 software. The analysis is carried out for three factors i.e., the Rotational Speed (RS), Traverse Speed (TS) and the Number of Passes of FSP (NP) at three different levels. Negative sign for the S/N ratio means Noise Power is greater than Signal Power. It just means a lot of noise dominating the signal in the present condition. There are situations in which the receiver copes very well even with this noisy environment, in Spread Spectrum Transmission, where the signal is "buried" deep into the noise. .
The Main effects plot for S/N ratio of Wear rate Vs the three parameters of FSP are described as follows:

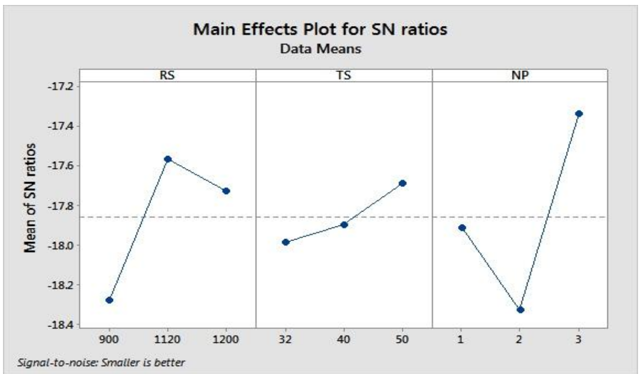

Fig. 5 The Main effects plot for S/N ratios of Wear Rate Vs RS, TS and NP

The effect of Rotational speed on the wear rate of the AMSC billets has a considerable effect, the effect of Rotational speed from 900 rpm to 1120 rpm showed an increased wear rate; as the RS increased to 1200 rpm the severe plastic flow of the base metal increased and so the bond strength between the matrix (AA 7075) and MWCNT particles of the surface composites. Also there will be uniform distribution of the reinforcement particles. Hence the wear rate is decreased, which can be observed in the above graph. The Traverse Speed has a low significance on the wear rate of the AMSC billets. The wear rate is highly influenced by the number of passes during FSP. From the graph it can be learnt that, the wear rate does not have any interaction from single pass to the double pass. When the number of passes of FSP increased from double to triple, there is a considerable increase in the wear rate, which may be because of the softening of the matrix material.

3.2 Analysis of Variance (ANOVA) for S/N ratios:

The ANOVA is adopted for the Signal to Noise ratio using Minitab-17 software, to obtain the highly influencing factor among the three process parameters of the Friction Stir Processing. Following are the details obtained:

Table 7 Analysis of variance for S/N ratios

Source	DF	Seq SS	Adj SS	Adj MS	F	P
RS	2	0.8421	0.8421	0.42106	1.21	0.452
TS	2	0.1397	0.1397	0.06986	0.20	0.832
NP	2	1.4718	1.4718	0.73589	2.12	0.320
Residual Error	2	0.6936	0.6936	0.34680		
Total	8	3.1472				

Table 8 Response Table for Signal to Noise Ratios for Wear Rate VS RS, TS and NP Smaller is better

Level	RS	TS	NP
1	-18.28	-17.99	-17.91
2	-17.57	-17.90	-18.33
3	-17.73	-17.69	-17.34
Delta	0.72	0.30	0.99
Rank	2	3	1

From the above tables, it can be observed that Number of passes of FSP during the fabrication of AMSC has got the Rank 1, and proved to be giving significant effect on the wear rate followed by the Rotational speed. The Traverse speed has a bleak impact on the wear rate of the AMSC billets.

3.3 Microstructural Study of AMSC samples:

As received base Aluminum alloy AA7075 has elongated matrix grain morphology along the rolling direction [9] which can be observed in the optical micrograph shown in Fig. 6. The Optical micrograph of the AA7075/MWCNT Surface Composite is shown in the Fig.7 which revealed that there exists a fine scale micro-structural region in which the second phase MWCNT particles seen distributed in a uniform pattern in the AA7075 matrix material. After subsequent passes of FSP, the MWCNT particles got further homogenously distributed within the Stir Zone forming a defect free Surface Composite. Severe plastic deformation subsequently caused dynamic recrystallization yielded into high level refinement of the grains with the stir zone. Thus, the mechanical and tribological properties of the Surface composites are highly influenced by the bond interface between the reinforcement particles (MWCNTs) and the matrix (AA7075) material apart from the presence of reinforcement particles. The analysis of microstructure of the base material (AA7075) and AMSCs, the grain size in the stir zone and as well of the base material needs to be observed optical microscopy. A typical Optical micrograph of the base alloy and FSPed billet are shown below in the Fig. 6 and Fig.7 respectively

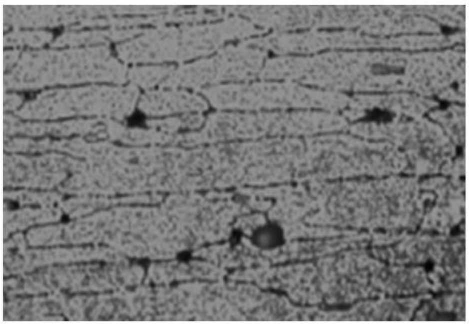

Fig. 6 Optical micro graph of the base AA7075

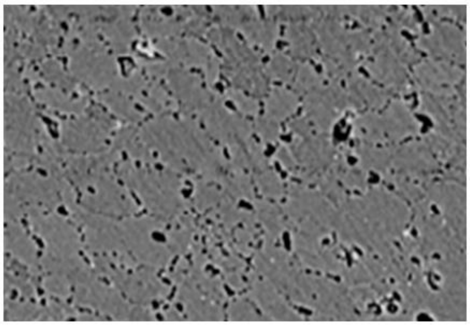

Fig. 7 Optical mircograph of a typical FSPed Billet

The grain size of the primary base metal (AA7075) is observed to in the range of 50-60 µm. Whereas the after FSP, the billets are prepared and observed under high resolution optical microscope so as to measure the grain sizes as low as 500 – 700 nm. Hence, it can be emphasized that the grain size of the FSPed billets is significantly decreased by 10 – 15 times as compared to that of the base AA7075 alloy.

Further, the microstructure of the AMSC billets exhibits a uniformly distributed equiaxed grains within the stir zone. This refinement of the grains may be because of the dynamic recrystallization during the FSP. This refinement of the grain in the AMSC billets not only influences the mechanical properties but tribological properties also for the resulting surface composites to a great extent.

IV. Conclusions

Following are the conclusions drawn for the work:

1. Friction Stir processing (FSP) can be used to fabricate Surface Composites.
2. AA7075 was used as the matrix material along with MWCNT as the reinforcement particles to fabricate AMSCs through Friction Stir Processing.
3. The process parameters of the FSP such as Rotational Speed, Traverse Speed and No. of passes are varied so as get a variety of Surface composite billets.
4. Taguchi L9 Orthogonal Array was used for Design of Experiments and the FSP is carried out accordingly.
5. The resulting FSPed Surface Composite billets were made ready for Wear Tests and as well for microstructural analysis.
6. Wear test was carried out on FSP billets so as to determine the influence of FSP process parameters on the Wear rate.
7. S/N ratios were obtained using Minitab-17 software for the resulting variable, i.e., wear rate.
8. ANOVA is adopted to find the most influencing factor among the three process parameters of the FSP with respect to the Wear Rate.
9. Rotational Speed and No. of passes found to be influencing process parameters during FSP with respect to the Wear rate.
10. The surface plots and graphs are plotted accordingly.
11. The high resolution optical micrography of the base metal and AMSC billets as well was carried out.
12. It is observed that the grain size of the AMSC billets is significantly decreased because of the dynamic recrystallization at the stir zone during FSP.

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