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Fabrication of surface nano composites of Al/B4C at selected regions by Friction stir processing

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Abstract: *Friction stir Processing is an important surface modifying technique to produce composite surface layer. This paper evaluates the effect of tool rotational speed, traverse speed and shoulder diameter on hardness and wear behavior of Al-B4C surface nano composite produced by FSP method. A Five level rotatable central composite design is used to predict the optimum input process parameters to fabricate the sound composite layer. Response surface methodology (RSM) Technique was used for analyzing the relationship between responses and process parameters. The results revealed that the shoulder diameter has more influence on achieving maximum hardness and wear resistance. To study the wear mechanisms, the selected wear worn out samples are analyzed through SEM studies.*

Keywords: *Friction Stir Processing; Boron Carbide (B4C); RSM; Wear; Hardness*

I INTRODUCTION

Aluminum based Metal Matrix Composites (MMC) have found in variety of applications in the aerospace, automotive, structural and defense industries due to it has excellent wear resistance, high dimensional stability, high stiffness to density ratio, low thermal expansion, etc. Aluminum MMC are fabricated by several conventional techniques over decades such as stir casting, liquid metal infiltration, powder metallurgy, diffusion bonding, mechanical alloying and compo casting. Particulate reinforced composite materials are easy to process with conventional fabrication methods due to lower cost, easy fabrication and isotropic in nature [1]. Recently great attention has been paid for fabricating the nano composite layer, the nano particles have greater property advantage over coarse grain particles due to balance in physical and mechanical properties. It is very difficult to control the uniform dispersion of nano-sized reinforcement on the matrix by conventional methods. In most of the engineering applications based upon only surface layer [2]. Nano particles can be selectively reinforced on the required zones, so that it is easier to retain the bulk properties with enhanced surface properties in order to modify the surface layer with improved properties.

Recently much attention is given to fabricating thick composite layer using Friction Stir Processing (FSP) Technique. This technique has several advantages such as low-cost operation, easy to adapt and simplicity. In addition, it produces fine-grained microstructure, uniform dispersion of reinforcement particles and it also eliminates the casting defects [3-4]. Several researchers have reported on the fabrication of Aluminum based surface nano composite layer by FSP Method [5]. Narimani et al. [6] studied the effect of AA6063/B4C/TiB2 hybrid composite produced by FSP. The hardness of the hybrid composite is reported to be increased due to finer refinement of the Aluminum matrix and uniform dispersion of reinforcement particles in the matrix. The hardness of the composite processed with nano particles was higher than the sample processed with micro particle composite [7]. Bauri et al. [8] filled the tungsten particles in the Al5083 matrix groove and processed with threaded pin tool length of 4mm and found that the particles had reached the entire length of the tool pin. The composite exhibited higher tensile strength with retained ductility due to dynamic recrystallization process. In FSP, the material flows in the complex pattern due to stirring. Designing of suitable tool shoulder diameter can produce the defect free processed region and tool material should be sufficiently strong, less wear and low heat conductivity. The shoulder diameter, tool rotational speed and traverse speed are important critical parameters for heat generation during FSP. These tool parameters are greatly influencing the material flow behavior, grain refinement and homogeneous particle distribution on the matrix. Extensive studies have been reported for FSP of composites using different process parameters [9]. However, work on combined effect of tool shoulder, tool rotational speed and traverse speed parameters for producing Al/B4C nano composites is limited. B4C is an excellent reinforcement particle due to its lower density and higher hardness. Al-B4C composites have high stiffness and hardness, and it uses in neutron absorber, structural applications and ballistic applications [10]. The present work is to optimize the FSP process parameters to attain the maximum hardness and minimum wear rate of FSPed Al-B4C surface composite using Response Surface Methodology (RSM).

II. METHODOLOGY

2.1 Response surface methodology

Response surface methodology (RSM) technique is mostly used for the modeling and analysis of several independent variables influence a dependent variable and is to optimize the response [10]. In the response surface design analysis, the independentvariables like x_1, x_2, $x_3 \cdots x_n$ influence a dependent variable Y or response, and the aim is to optimize the response. The mechanical and wear properties of the FSPed composites are depending upon various factors like tool rotational speed, traverse speed, shoulder diameter, tool pin profile, tool material, tool tilt angle, plunge depth, axial force, etc. These factors can have following type of relationship:

$$Y = \{ x_1, \ x_2, \ x_3 \cdots x_n \} \pm E_r \ldots \ldots \ldots (1)$$

The relationship between output variable of response Y and controllable input variables (x_1, x_2, $x_3 \cdots x_n$) of n quantitativefactors, the function is called response surface or function. The term represents error occurs during experimentation. To optimize the response 'y' the first step is to find an appropriate approximation for the true functional relationship between independent variable and response surface. In this present study, the second order polynomial equation is used for finding the quality characteristic of the composite. The quadratic equation used to represent the response surface Y is given by:

$$= B_0 + \Sigma B_i x_i + \Sigma B_{ii} x_i^2 + \Sigma B_{ij} x_i x_j + E_r \ldots \ldots \ldots (2)$$

2.2 Identifying important process parameters

From the literature and preliminary trials work it has been found that many factors affect the mechanical and wear properties of the composite. The various factors are tool rotational speed, transverse speed, tool shoulder diameter, pin diameter, tool pin profile, tool tilt angle, axial force, number of passes, etc. The major important process parameters such as Rotational speed (N), Transverse speed (S), Shoulder diameter (D) were selected for this study. These parameters influence greatly by the heat generation.

2.3 Finding the limits of the process parameter and design matrix

A large number of preliminary trial runs were conducted to find the upper and lower limits of FSP process parameters, by varying one of the parameters and keeping the other parameters at constant. Typical defects such as tunnel defect, crack, ribbon flash, and surface galling were observed during trial periods. Feasible upper and lower limits of each factor were chosen in such a way that the processed composite should be free from any kind of visible defects. The upper limit and lower limit each factor was coded as 1.682 and -1.682 respectively. The intermediate coded values were estimated from the following equation [12]

$$= 1.682[2X - (X_{max} + X_{min})]/(X_{max} - X_{min}) \ldots \ldots \ldots (3)$$

Where Xi is the required coded value of a variable X; and X isany value of the variable from Xmin to Xmax. Xmin is the lower limit of the variable and Xmax is the upper limit of the variable. The three factor five level central composite rotatable design consisting of 20 sets of coded conditions composed of a full factorial 2^3=8, plus 6 center points and 6 star points [13]. The selected design matrix of the parameters and levels are shown in Table 1. Fig. 1 shows the fabricated FSP tools as per design matrix and Fig. 2 shows the dimensions of the tool.

2.4 Fabrication of composite as per design matrix

Al 5083-O rolled plates of 8mm thickness with a chemical composition of 4.1%Mg, 0.06%Cr, 0.5%Mn, and rest aluminum was used. The substrates were cut into the rectangular size of 200mm x 80mm and a longitudinal groove size of 1mm width and depth 3mm were cut in the middle of the substrate. The nano sized B4C particles size of 30-60nm average and purity of 99.9% were used as reinforcements. The SEM micrograph of B4C nano particles is depicted in Fig. 3. Before filling of reinforcement particles, the grooves were cleaned with acetone. The reinforcement particles were mixed with acetone and filled inside the groove in the form of the slurry. The Aluminum alloy plates were dried inside the oven at 50°C for 30 minutes.

The specimens were clamped on the hydraulic fixture of the FSW machine capacity of 11 kW, and 40kN. Fig. 4 shows the FSP experimental setup. To prevent the scattering of the particles during FSP, the groove was closed with a pinless tool having shoulder only and then processed with the tool having a pin. For the fabrication of composites, the hardened H13 tool steel with a cylindrical threaded pin diameter of M6 x1.0mm and pin length of 5mm was used. Tool tilt angle of 2^{o} and plunge depth of 0.15mm was considered after the number of preliminary trials.

Fig. 1 Fabricated FSP tools as per design matrix

The number of FSP passes limited to three only and in the second pass tool rotational direction was changed for uniform mixing of the reinforcement particles in the matrix. The cross section of the stir zone was analyzed for its microstructural changes through optical microscopy (OM) and scanning electron microscope (SEM).

2.5 Recording of response

The specimens for hardness testing were extracted from the middle of the stir zone of the cross section of the processed region. The hardness was determined by using microhardness tester at the load of 100g and the dwell period of 10s. For experimental purpose, the average of five hardness values was taken. The wear samples were extracted in the middle of the stir zone of the composite by wire EDM.

Dry sliding wear tests of the composites were carried out using pin and disc tribometer under the following conditions: diameter of wear test specimens = 8mm, counter disc material = EN-24 steel of 55-58HRC, surface roughness of the disc = 0.2µm (Ra), Normal load = 30N, Sliding speed = 2 m/s, wear test distance = 2000m. The wear samples were cleaned with acetone before and after the test dried and weighed by electronic weighing balance at anaccuracy of 0.01mg.

The worn-out surfaces of the specimenswere examined by SEM with Energy Dispersive Spectrometer(EDS) to determine the wear mechanism.Fig. 5 shows the typical wear test specimens extracted from the composite region.The design matrix and experimental results of hardness and wear rate are presented in Table 2.

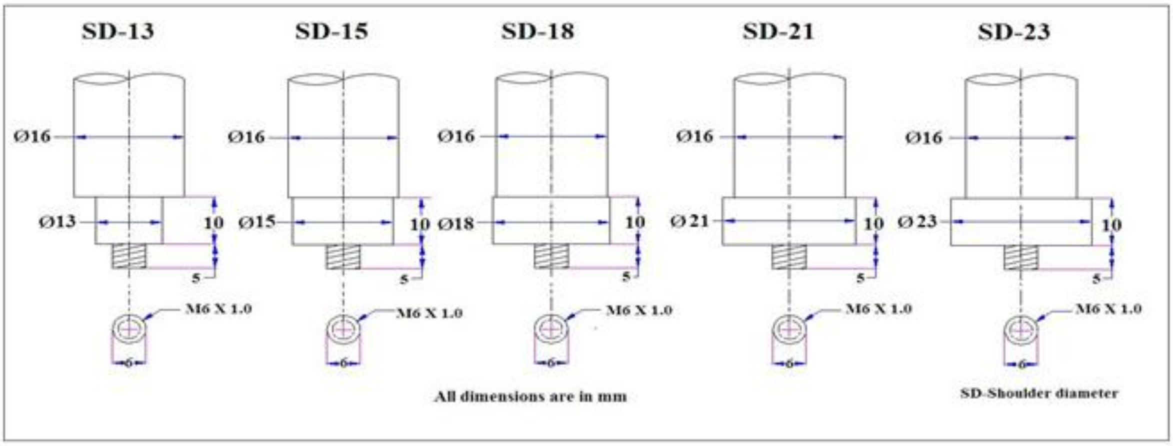

Fig. 2 Dimensions of the FSP tool

Table 1 Process parameters and the levels used in the experiments

S.No.	Parameter	Notation	Unit	Levels				
				-1.682	-1	0	1	1.682
1	Rotational speed	N	rpm	579.55	750	1000	1250	1420.45
2	Traverse speed	S	mm/min	14.77	25	40	55	65.23
3	Shoulder diameter	D	mm	12.95	15	18	21	23.05

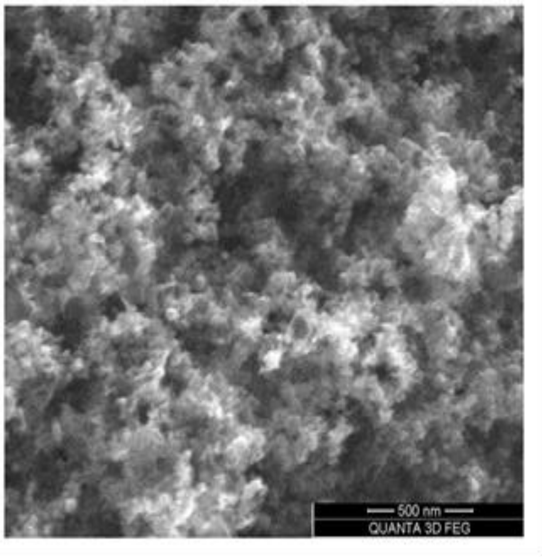

Fig. 3 SEM micrograph of B_4C nano particles

Table 2 Design matrix and experimental results

S.No.	FSP process parameters			Hardness (Hv)	Wear rate (mg/m)
	N	S	D		
1	-1	-1	-1	102.6	0.00281
2	1	-1	-1	102.8	0.00279
3	-1	1	-1	105.8	0.00264
4	1	1	-1	108.5	0.00256
5	-1	-1	1	106.4	0.00262
6	1	-1	1	112.8	0.00241
7	-1	1	1	103.6	0.00271
8	1	1	1	112.4	0.00244
9	-1.682	0	0	102.8	0.00278
10	1.682	0	0	109.2	0.00252
11	0	-1.682	0	112.6	0.00244
12	0	1.682	0	113.8	0.00232
13	0	0	-1.682	101.2	0.00288
14	0	0	1.682	109.8	0.00253
15	0	0	0	124.8	0.00198
16	0	0	0	124.6	0.00198
17	0	0	0	125.2	0.00196
18	0	0	0	124.5	0.00195
19	0	0	0	123.8	0.00197
20	0	0	0	125.2	0.00196

Table 3 Validity test results

FSP Process parameter			Hardness (Hv)		Error %	Wear rate (mg/m)		Error %
N	S	D	Experimental value	Predicted Value		Experimental value	Predicted Value	
-1	0	0	115.3	115.89	-0.50	0.00231	0.002288	0.96
1	0	0	120.9	120.13	0.64	0.00216	0.002134	1.22
0	-1	0	120.4	119.99	0.34	0.00215	0.002149	0.21
0	1	0	120.5	121.13	-0.52	0.00206	0.002077	-0.82
0	0	-1	114.9	115.64	-0.63	0.00230	0.002317	-0.73
0	0	1	120.8	120.02	0.64	0.00215	0.002141	0.42

2.6 Development of mathematical model

The response function is a function of rotational speed (N) rpm,traverse speed (S) mm/min, and shoulder diameter (D) mm.These independent variables are measurable, controllable in the experiments with negligible error which are expressed below$Y = f(N, S, D) \ldots \ldots \ldots \ldots \ldots (4)$ For the three factors the selected second order polynomial couldbe expressed as

$$Y = B_0 + B_1(N) + B_2(S) + B_3(D) + B_{11}(N^2) + B_{22}(S^2) + B_{33}(D^2\ ) + B_{44}(NS) + B_{13}(ND) + B_{23}(SD) \ldots \ldots \ldots \ldots \ldots \ldots (5)$$

Where B_0 is the free term of the regression equation; B_1, B_2, andB_3 are linear term coefficients B_{11}, B_{22} and B_{33} are quadraticterm coefficients; B_{12}, B_{13} and B_{23} are interaction termcoefficients. The coefficients are computed using the DesignExpert statistical software package. The mathematical modelswere developed after estimating the coefficients. Thecoefficients were checked with 95% confidence level and the significant coefficients were used for final model. The finalempirical relationship with processing factors to estimate thehardness and wear rate is given in the equation 6 and 7respectively.

$$Hardness\ (Hv) = 124.6954 + 2.113479(\mathrm{N}) + 0.565149(\mathrm{S}) + 2.194021(\mathrm{D}) - 6.68426(\mathrm{N}\square) - 4.13868(\mathrm{S}\square) - 6.86104(\mathrm{D}\square) + 0.6125(\mathrm{NS}) + 1.5375(\mathrm{ND}) - 1.5125(\mathrm{SD}) \ldots \ldots \ldots \ldots .\,.\,(6)$$

$$Wearrate = 0.001966 - 0.000074(N) - 0.000035(S) - 0.000088(D) + 0.000243(N\square) + 0.000148(S\square) + 0.000263(D\square) - 0.000015(NS) - 0.000048(ND) + 0.000065(SD) \ldots \ldots \ldots \ldots (7)$$

2.7 Conformity test

Experiments are conducted to verify the empirical relationshipof hardness and wear rate which are presented in the equation 6and 7 respectively. Six FSP experiments were conducted in thesame experimental setup. The process variables were selectedother than used in the design matrix and the experiments werecarried out to verify the model. The results of validity tests are presented in Table 3. The results obtained are close to thepredicted value. Hence the model developed is accurate.

III RESULTS AND DISCUSSIONS

3.1 Effect of shoulder diameter

During FSP maximum heat is generated between the tool and substrate. The maximum temperature produced at stir zone is a significant factor in determining the hardness of the composite due to the effect of microstructure and grain size [13]. In FSP, metals never reach their melt temperature in the processed region. The tool rotational speed, traverse speed of tool and forging force of the tool shoulder are responsible for the generation of heat in stir zone. The severe plastic deformation induced by the rotating tool results in grain refinement. The average grain size of the base material is 49.5µm. After processing with 18 mm shoulder diameter the grain size of the Al-B4C composite is reduced to approximately 3µm. Increase in grain size due to the material is not getting the appropriate heat input for grain refinement. During the FSP rotating tool moves along the line of interest and the tool, shoulder diameter provides adequate forging action during processing. The following equation shows that FSP heat input in the stir zone during processing [14].

$$Q=(4\pi^2\mu\omega PD^3)/3\nu \ldots\ldots\ldots\ldots(8)$$

Where Q is the heat input, µ is the coefficient of friction between tool shoulder and the work surface, ω the tool rotational speed, P is the pressure, D is the tool shoulder diameter, ν is tool traverse speed. Based on the equation (8) tool shoulder diameter is one of the key parameters for governing the heat input during FSP. With an increase in the tool shoulder diameter leads to an increase in the heat input and produces a larger volume of plasticized material around the tool pin due increase in frictional area. The heat losses also higher in the larger shoulder diameter and the volume of shoulder driven metal flow reduces marginally. In the lower shoulder diameter, the heat generation is less, and it is insufficient for uniform stirring of the material. Jafari et al. [15] have observed that the FSP peak temperature of Cu/CNT composite of 665.8°C and 449.4°C for 12mm and 16mm shoulder diameter respectively. The 12mm and 16 mm shoulder diameter processed composite grain size were 4µm and 10µm respectively. The grain size of the smaller shoulder diameter processed Cu/CNT composite was much lower than, the larger diameter processed composite. The larger shoulder diameter causes more annealing effect due to the higher heat input.

The Fig. 6 shows the effect of shoulder diameter on micro hardness and wear rate values taken for the constant rotational speed of 1000 mm/min and traverse speed of 40 mm/min. The maximum hardness value observed for 18mm shoulder diameter processed specimen. The stir zone temperature was measured by using Thermal imaging camera at different shoulder diameter is shown in Fig. 7. The optimum heat input refines the grain size of the material and uniform dispersion of particles in the matrix yields the maximum hardness. Fig. 8 shows the SEM image of Al-B4C composite processed with 18mm shoulder diameter. Similarly, 18mm shoulder processed sample has lower wear rate due to the increased hardness ofprocessed region. As per Archard's relationship, higher the hardness of the material, lower the wear rate [16]

3.2 Effect of Tool rotational speed

The Fig. 9 shows the effect of rotational speed on micro hardness and wear rate value taken for constant traverse speed of 40 mm/min and shoulder diameter of 18mm. The hardness value increases with increase in rotational speed from 580 rpm to 1000 rpm and then decreases. Similarly, the wear rate decreased with increase in rotational speed till 1000 rpm and then decreases. At 1000 rpm maximum hardness and minimum wear rate values are observed as 124.7Hv and 0.00197 mg/m respectively. Mosallaee et al. [17] suggested that the tool rotational rate and traverse speed are the main process variables for heat generation in the FSP. The following relationship shows the maximum heat generation with the process parameters during FSP.

$$T/T_m = k(w^2 / v * 10^4)^{\alpha} \cdots\cdots\cdots\cdots\cdots \quad (9)$$

Where T is the peak temperature, Tm is the melting point of the base material, ω is the tool rotational speed, v is the tool traverse speed, α is the exponent and k constant are material parameters. The increase in heat ratio (ω^2/v) leads to increasing of temperature in the stir zone. During FSP increase in temperature leads to more plastic deformation which refines the grain size of the material in the processed region. However, grain coarsening occurs with the increase in excessive peak temperature. With the increase in rotational speed decreases the grain size of the matrix material. However, after certain rotational speed, the grain size increases due to excessive heating of the material leads to the formation of grain growth and causes adverse effects on the mechanical properties. The tool rotation rate is also one of the important key input process parameter for the heat generation in FSP. According to the equation 8, the Q (heat input) has directly proportional to the tool rotation rate and inversely proportional to the tool traverse speed. This relationship is stated below [13]

$$Q \propto \omega / V \quad \cdots\cdots\cdots \; . \; . \quad (10)$$

3.3 Effect of Traverse speed

The Fig. 10 shows the effect of traverse speed on micro hardness and wear rate for the constant rotational speed of 1000 mm/min and shoulder diameter of 18mm. The hardness value increases with increase in traverse speed from 15 mm/min to 40 mm/min and then decreases. Similarly, the wear rate decreased with increase in traverse speed till 40 mm/min and then decreases. If the traverse speed is too low the residing time of the tool is high resulting high heat input which makes adverse material properties. According to the equation 8 the decrease in traverse speed increases the heat input causes grain growth which results in lesser hardness. T. Saeid et al. [18] have found that the mechanical properties of friction stir welded duplex stainless steel are improved with increased traverse speed. With the increase in traverse speed, the heat generated in welding is less and as a result, better mechanical properties are observed. The traverse speed is also an important process parameter for controlling the heat input.

IV PROCESS PARAMETER OPTIMIZATION

To analyze the suitable combination of input parameters and to achieve the maximum responses the mathematical model equation’s (6) and (7) were used. To obtain the optimized condition of process input parameters on hardness and wear rate the design expert software was used with various combinations. The maximum achievable hardness and wear rate are 125.09Hvand 0.00195 mg/m respectively. The corresponding inputprocess parameters for obtaining both the responses are 1044.33rpm tool rotational speed, 41.05 mm/min traverse speed and18.52 mm shoulder diameter.

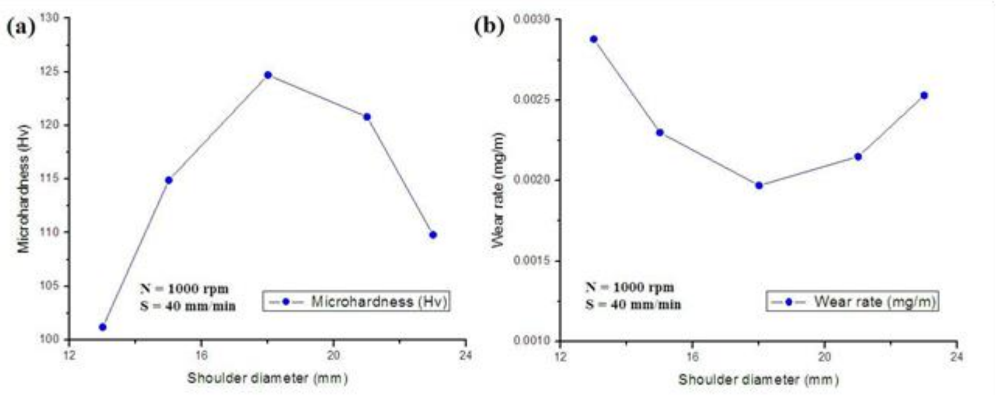

Fig. Effect of shoulder diameter on (a) Hardness (b) wear rate Al-B_4C composite

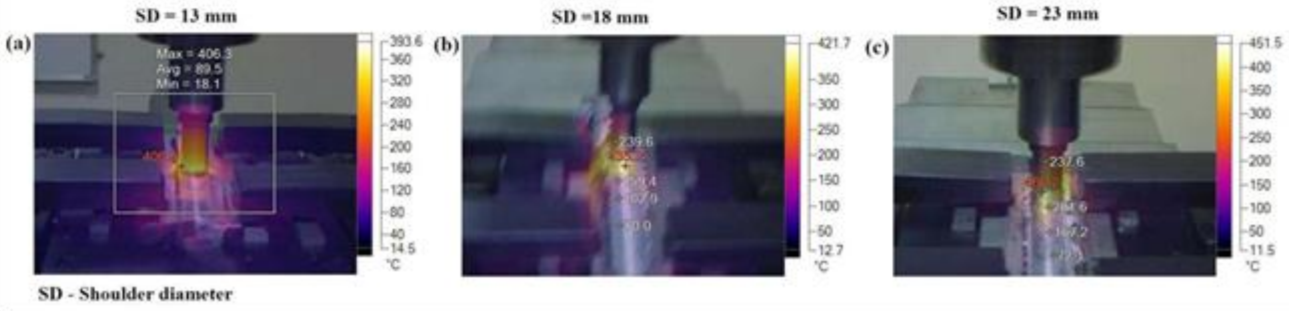

Fig. 7 The stir zone temperature of the composite samples processed at different shoulder diameters (rotational speed 1000 rpm and traverse speed 40 mm/min)

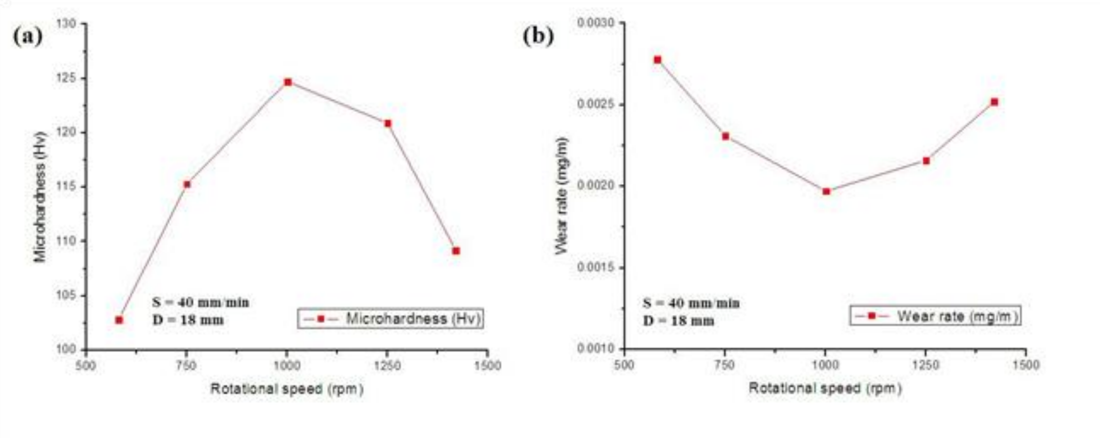

Fig. 9 Effect of Rotational speed on (a) Hardness (b) wear rate of Al-B_4C composite

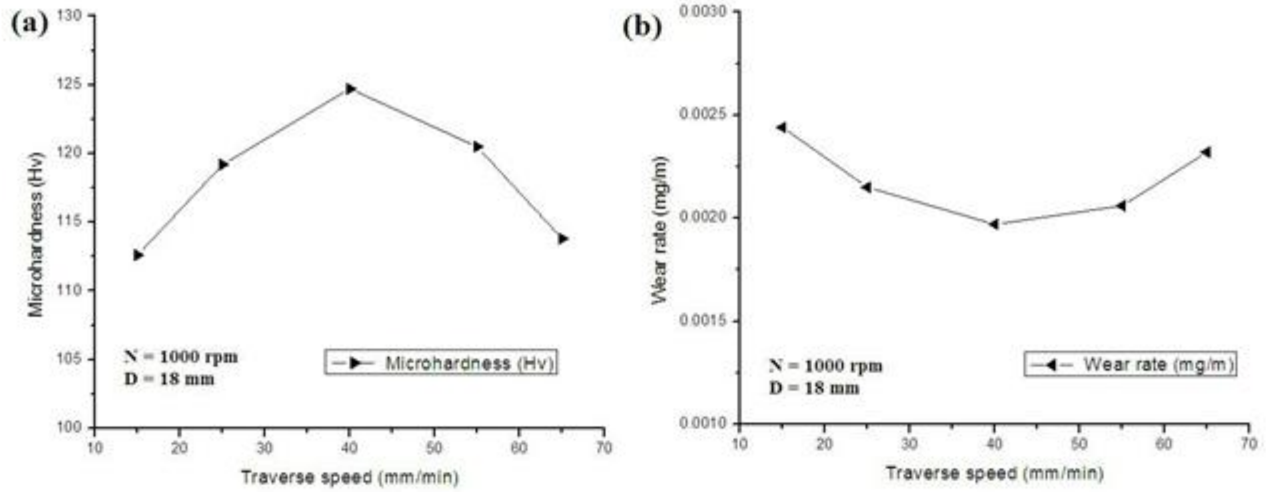

Fig. 10 Effect of Traverse speed on (a) Hardness (b) wear rate of Al-B4C composite

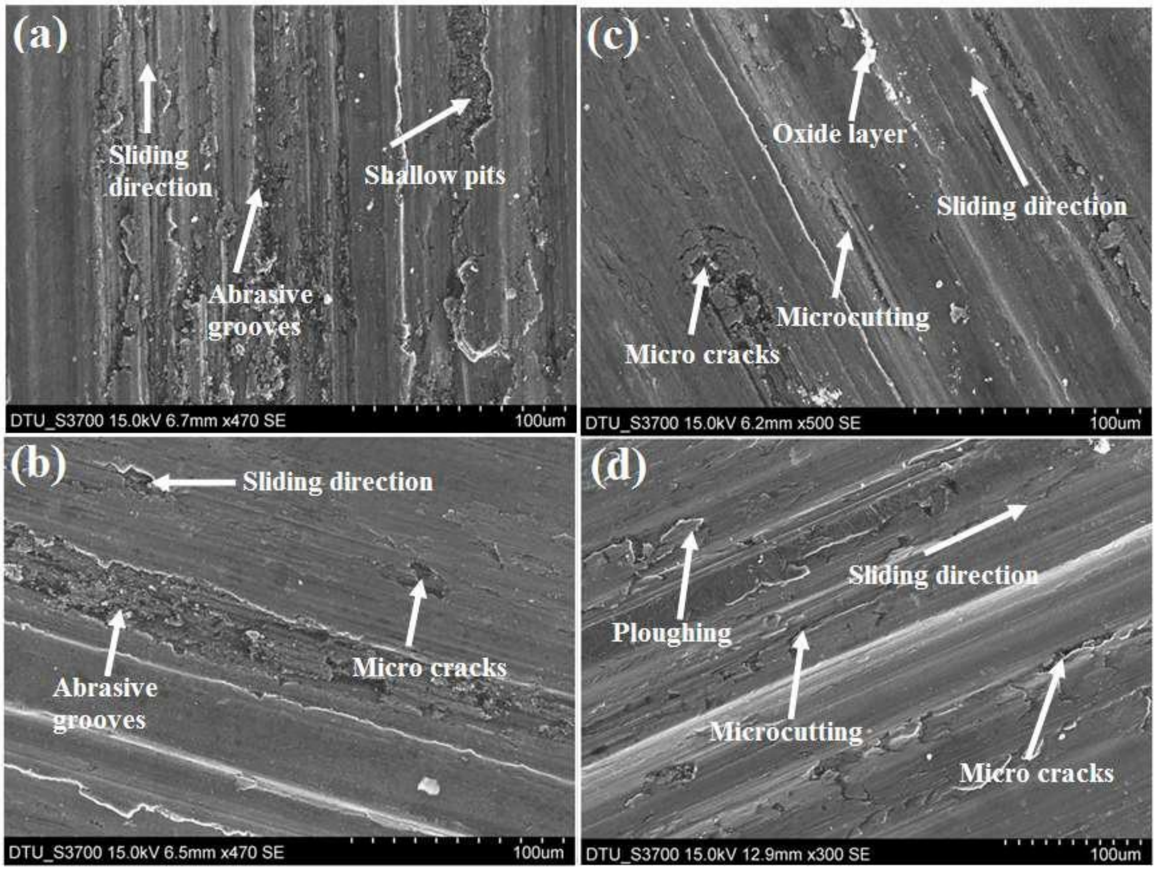

Fig. 11 SEM Micrograph of worn surface of (a) Base material (b) 13 mm shoulder diameter sample (c) 23 mm shoulder diameter sample (d) 18 mm shoulder diameter sample [composite sample processed at rotational speed of 1000rpm and traverse speed of 40mm/min]

V WORN SURFACE

The wear rate of the composite is the lower rate than matrix alloy. The hardness of the composite is higher than matrix alloy.

Under the same frictional conditions hard material has lower wear rate. The enhancement of the hardness of the composite is due to the refinement of grain size, uniform distribution of reinforcement particles in the matrix and good bonding between the particles and the matrix. The difference between the coefficient of thermal expansion between the matrix and reinforcement are also responsible for the increase in hardness of the composite. The hard nano reinforcement particles act as load bearing element during wear test. During the wear test of the composite specimen the pull-out reinforcement forms oxide layer. This layer forms three body Mechanically Mixed Layer between (MML) the sliding surfaces. This MML act as an effective insulation between the sliding surfaces, as a result, wear rate decreases. Fig. 11 shows the SEM of the worn surface of the base material and composite respectively. The worn surface of base material exhibits oxide layer, abrasion marks, micro cracks, and surface damaged pits formed along the sliding direction. In the composite sample, no excessivedamage is seen. The worn surface exhibits oxide layer, micro grooves, ploughing and micro cutting.

VI CONCLUSIONS

FSP is an attractive method to develop the surface composite layer. The FSP process parameters tool rotational speed, traverse speed and shoulder diameters are substantially influenced for fabricating the surface composite layer. The following conclusions can be drawn from this work.

- The highest hardness of 125.46Hv and minimum wear rate of 0.000196mg/m are achieved in the stir zone of the composite for the optimized input process FSP parameters of tool rotational speed of 1046 rpm, traverse speed of 41mm/min and shoulder diameter of 18mm.
- Shoulder diameter has significant influence on mechanical and wear properties of composite followed by tool rotational speed and traverse speed. The appropriate heat generation is mainly responsible for homogeneous grain refinement and uniform dispersion of reinforcement particles in the matrix.
- The grain refinement of the matrix and uniform dispersion of reinforcement particles in the matrix are responsible for the enhancement of hardness and wear resistance.
- The wear behaviour of the composite is improved due to the formation of mechanically mixed layer. The formation of oxide layer and pull out particles act as an efficient insulation between pin and the disc.

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