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ORIGINAL ARTICLE

Optimization of Friction Stir Process Parameters for Micro-Hardness and Wear Characteristics of Silicon Carbide-Reinforced Al-7075 Surface Composite

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Abstract Friction stir processing is a newly emerged manufacturing method for modifying the surface of materials by localized plastic deformation for enhancing their surface characteristics. This research focuses on the optimization of process parameters for silicon carbide-reinforced 7075 aluminum alloy surface composite produced by friction stir processing. For placing silicon carbide particles, an array of 2 × 2 mm blind holes of size 2 mm with a spacing of 4 mm was machined on the AA7075 base plate. A tool with a square pin of 6 mm size having a shoulder diameter of 25 mm was utilized for the fabrication of composites. Process parameters such as tool rotational speed, travel speed and tilt angle were selected in this study and the effects of these parameters on micro-hardness and wear properties were analyzed. These parameters were varied in four levels and surface composites were manufactured according to the Taguchi's L16 experiment. Optimization of process parameters for enhancing the micro-hardness and wear resistance was performed with grey relational analysis. Experimental results showed that the micro-hardness and wear rate of the composites were most significantly affected by tool travel speed followed by the tool rotation and tilt angle, respectively. Both responses were optimized when the AA7075/SiC surface composite was manufactured with 1120 rpm as tool rotational speed, 50 mm/min as tool travel speed and 3° as tilt angle. The surface composite developed in this research work can be used as a viable alternative material for applications that require superior surface characteristics.

Keywords Friction stir processing · 7075 Aluminum alloy · Silicon carbide · Surface composite · Micro-hardness · Wear resistance · Grey relational analysis

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1 Introduction

Because of the lightweight, high strength and relatively low density, aluminum alloys find major applications in automobile and aerospace industries [1–3]. In spite of this, the application of aluminum is limited owing to its poor surface properties [4]. The surface characteristics of aluminum alloy can be significantly improved by modifying its surface. Traditionally, it is done by heat treatment process such as surface hardening [5]. In recent years, several surface modification processes are proposed in the literature, and among them, friction stir processing (FSP) has been proved to be a green and energy efficient method [6]. FSP is one of the solid state processes in which a rotating tool having a pin and shoulder is inserted into a base material until the shoulder come in contact to the surface of base material. The frictional heat generated between the tool shoulder and the surface of the base material is enough to stir the base material plastically using the pin [7]. FSP considerably influences the mechanical and tribological properties of the stir zone by producing a unique

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microstructure having fine and equiaxed grains with the high angle grain boundary through a dynamic recrystallization mechanism [8, 9].

FSP process has been successfully used to enhance the surface characteristics of the aluminum alloy by introducing the reinforcing material onto the surface either in nano- and microsize [10–12]. Surface composites demonstrate superior characteristics on the surface without modifying the base material properties. Many researchers have attempted to manufacture aluminum alloy surface composites with various reinforcements such as SiC, Al2O3, B4C, TiC, etc. [13]. Inclusion of these reinforcements has been found to increase the hardness and wear resistance of the surface composites [14–16]. The increase in hardness can be attributed to the grain strengthening mechanism of reinforcement. Increasing the rotational speed of the tool, shoulder penetration depth and changing the tool travel direction produce a homogeneous distribution of reinforcing particles that improve the surface characteristics of the composite [17, 18].

Hashemi et al. [19] studied the influence of different tool geometries and the number of passes on the hardness and wear resistance of FSP of Al-7075-T651 reinforced with TiN nanosized particles. Experimental results showed that the substrate properties such as wear resistance and coefficient of friction got increased under high loading force.

Kumar et al. [20] studied the effect of TiC on the microstructural characteristics, hardness and corrosion behavior of Al7075/TiC surface composites fabricated using friction stir processing. Authors used different volume fractions of 3.5 μm TiC particles as reinforcement and performed FSP process at 30 mm/min travel speed and 1200 rpm rotational speed. It was observed that increasing the TiC volume fraction increased the hardness and corrosion resistance of the surface composites than the base material.

Several studies were found in which the hardness, wear and friction characteristics of FSP processed surface composites fabricated from 7075-T651 aluminum alloy reinforced with silicon carbide (SiC) were studied [21–23]. The average hardness and wear resistance at the FSP zone of the composite were found to be more than the parent metal due to the fine distribution of SiC particles. Tool wear and mechanical properties of the surface composite were greatly influenced by the rotation speed and number of passes, whereas the traverse speed had more effect on micro-hardness. [24].

It is evident from the above literature that the different process parameters, tool geometries and reinforcement particles have significant effects on the microstructural characteristics, micro-hardness, wear and mechanical properties of surface composites produced by FSP. Among the process parameters, rotational speed, traverse speed and tilt angle of the tool are found to be significantly influencing the micro-hardness and wear resistance properties of FSP processed aluminum-based surface composites [21–24]. It is essential to optimize these parameters during FSP to produce surface composites with the enhancement of these properties by inhibiting defect formation in the stir zone.

Few studies [25, 26] are found in which parameters such as rotational speed, tool traverse speed, diameter of hole and pin profile were optimized for enhancing the tensile strength of the FSP processed aluminum-based surface composite. Multi-objective optimization of process parameters for maximizing the micro-hardness and wear resistance properties of SiC-reinforced Al-7075-T651 surface composite was not done by any researchers. Hence, in this study, an attempt has been made to analyze the effect of rotational speed, traverse speed and tilt angle of the tool on the micro-hardness and wear resistance of surface composites fabricated from Al7075 reinforced with SiC particles using FSP process and to select an optimal combination of these parameters for enhancing these multiple response parameters. In addition to this, this study also identifies the most significant FSP process parameters in the fabrication of Al7075/SiC surface composites. Enhanced surface properties of the fabricated Al7075/SiC surface can enable it to be used as a viable material for producing automobile parts that are subjected to friction.

2 Materials and Methods

2.1 Materials Used

Commercially available Al7075 alloy was selected in this study because of its superior properties when compared to other grades of aluminum alloy [26]. Al7075 alloy is most commonly used in industrial applications because of high strength to weight ratio, corrosion resistance and good fracture toughness characteristics [27]. Rolled plates of Al 7075-T651 of size 150 mm × 400 mm × 6.2 mm were used as the base metal. Table 1 furnishes the chemical composition of Al7075-T651 with its components in weight %.

Silicon carbide (SiC) particles of size 37 μm (400 mesh) were used as reinforcement in Al7075 base metal. SiC particle was chosen as reinforcement because of its superior properties such as high strength, high resistance to wear and corrosion and good thermal stability.

A H13 steel tool having a flat shoulder with 25 mm diameter and 100 mm length and a square pin with 6 mm circumscribed circle diameter and 4 mm height was used in this study. A square pin is selected, because it produces more turbulence and pulsating action during plastic

Table 1 Chemical Composition of Al7075-T651

Component	Al	Zn	Mg	Cu	Fe	Si	Ti	Cr	Mn	Others
Weight %	87.1–91.4	5.1–6.1	2.1–2.9	1.2–2	0.5	0.5	0.2	0.18–0.28	0.3	0.2

deformation and results in better properties at the stirred zone [28–30]. For the capping process, a pinless tool with a diameter of 25 mm and length 100 mm made of H13 steel was used. The image of the FSP stirring tool is shown in Fig. 1.

2.2 FSP Procedure

For prepositioning the SiC particles on the base metal, an array of 2 × 2 mm blind holes as shown in Fig. 2 was drilled on the surface of the base metal using a vertical milling machine. To prevent agglomeration of reinforcing particles and for their uniform distribution [31], the spacing between the two blind holes was maintained as 4 mm.

In this study, the rotational speed, traverse speed and tilt angle of the tool were selected as FSP parameters and the effects of these parameters on the micro-hardness and wear rate of the Al7075/SiC surface composite were investigated. Based on preliminary experiments, the levels of these process parameters were selected and are presented in Table 2.

Al7075/SiC surface composites were fabricated using a three-axis automatic FN2EV model vertical milling machine (Make: HMT Ltd. Pinjore, India) which was suitably modified for the FSP process. The experimental setup is shown in Fig. 3. Surface composites were prepared using the combination of the process parameter levels specified in Taguchi L16 orthogonal design. An axial load of 8 KN and an initial period of dwelling of 15 s were maintained constant in all experimental runs. At the beginning of FSP process, the capping process was done with a pinless tool to close the blind holes so as to avoid the scattering of SiC particles from the blind holes in the following stirring pass. During the capping process, a rotational speed of 560 rpm, a 25 mm/min traverse speed and a plunging depth of 0.3 mm were used. A single pass FSP was performed on each sample after the capping step was completed. To thwart the formation of tunnels and voids in the stir zone [18], the penetration depth of the shoulder into the base metal was kept as 0.24 mm while performing FSP. The flash generated was removed at the end of the process with a diamond file to ensure proper alignment of the specimen for measuring responses.

Fig. 1 Stirring tool used in FSP process

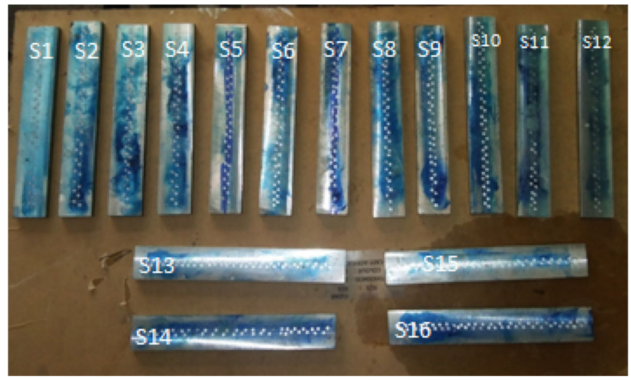


Fig. 2 Samples of Al-7075/SiC surface composite

2.3 Measurement of Micro-hardness and Wear Rate

Micro-hardness and wear rate were two response variables measured on the specimen after each run. Micro-hardness was measured according to ASTM E-384 at the stir zone using the Vickers micro-hardness tester (HV-1000B). A pyramid diamond indenter with a standard load of 10 kg and a dwelling time of 20 s were used during the test. Five readings of micro-hardness were taken across the stir zone and the average was calculated. As per ASTM-G99 standard, the wear test was conducted with the Pin-on-disk tester (Make: DUCOM). Wear test samples of size 10 mm × 6 mm × 25 mm were cut from the stir zone using a wire EDM machine. The disk of the tester was made of steel having hardness of 72 HRC. The wear test was carried out at room temperature under the normal load of 10 N. The sliding distance, sliding speed and track diameter of the disk were selected as 1000 m, 200 rpm and 80 mm, respectively. The weight difference of each sample

Table 2 Process parameters and their levels

Parameters	Level-1	Level-2	Level-3	Level-4
Tool rotational speed (rpm)	560	710	900	1120
Tool traverse speed (mm/min)	25	50	65	80
Tilt angle (°)	2	2.33	2.66	3

Fig. 3 Experimental setup for friction stir processing of Al-7075/SiC surface composite

was measured before and after the wear test with 0.1 mg precision in order to calculate the wear rate. Microstructure analysis of the stir zone after the wear test was performed with scanning electron microscopy to assess the distribution of SiC particles and the worn out surface.

3 Results and Discussion

3.1 Optimization of Process Parameters

Table 3 summarizes the experimental results for the micro-hardness and wear rate of FSP processed Al7075/SiC surface composites fabricated with the process parameters according to L16 orthogonal array. As there are two response parameters which have been considered in this study, Grey Relational Analysis (GRA) was used to optimize the FSP parameters for improving the micro-hardness and wear resistance of the fabricated surface composite. Experimental results were further processed for performing GRA analysis. The normalized responses, deviation sequences, grey relational coefficients, calculated grey relational grade (GRG) and rank for each experimental run are tabulated in Table 4. According to GRA analysis, the experimental run having the maximum grade value indicates the near optimum combination of process parameters. Therefore, from Table 4, it has been observed that experimental run 13 has the highest value of GRG, indicating the initial optimum combination of process parameters. Therefore, according to the experimental run 13, tool rotational speed of 1125 rpm, tool traverse speed = 25 mm/min and tool tilt angle = 2° are most likely the optimum parameter settings for maximizing the micro-hardness and wear resistance.

Table 5 shows the mean GRG values for each level of the process parameters and the same is graphically represented in Fig. 4. The optimal combination of FSP parameters corresponds to the parameter levels having the highest GRG value. From Fig. 4, optimal combinations of process parameters are identified as: Tool rotational speed = 1125 rpm, Tool traverse speed = 50 mm/min and tool tilt angle = 3°.

3.2 Significant Process Parameters

The significant process parameters can be identified from the values of rank and delta in the response table for GRG as presented in Table 5. It is evident that, among the process parameters considered, the most influential parameter on the properties of the surface composite is the traverse speed, followed by tool rotational speed and tilt angle, respectively.

Analysis of Variance (ANOVA) was also performed using Minitab 18.1 statistical software, to determine the relative significance of process parameters and their percentage contribution to the properties of Al7075/SiC surface composites. The result of ANOVA analysis conducted on grey relational grade values is shown in Table 6. ANOVA analysis has been carried out at 0.05 level of significance. It is observed that except the tilt angle, both tool rotational speed and tool traverse speed are statistically significant. The result of ANOVA analysis also reveals that tool traverse speed has the highest influence with 46.19% contribution on the properties of the surface composites, followed by tool rotational speed (32.63%) and tool tilt angle (7.44%), respectively.

3.3 Effect of Process Parameters

An increase in tool rotational speed increases the relative velocity between the tool and the base metal. This increases the plastic flow of the base metal and helps with the homogeneous mixing of the reinforcing material as

Table 3 Micro-hardness and wear rate of Al7075-SiC metal matrix composites

Runs	Tool's rotating speed (rpm)	Tool's traversing speed (mm/min)	Tilt angle	Micro-hardness(HV)	Wear rate(mg/m)
1	560	25	2.00	135.89	0.03245
2	560	50	2.33	98.78	0.0212
3	560	65	2.66	96.70	0.06765
4	560	80	3.00	157.78	0.08509
5	710	25	2.33	124.76	0.03347
6	710	50	2.00	163.57	0.02987
7	710	65	3.00	173.65	0.08786
8	710	80	2.66	125.78	0.08571
9	900	25	2.66	165.89	0.03987
10	900	50	3.00	213.89	0.01893
11	900	65	2.00	203.98	0.05956
12	900	80	2.33	164.56	0.06385
13	1120	25	3.00	243.87	0.0323
14	1120	50	2.66	194.33	0.01344
15	1120	65	2.33	195.34	0.05491
16	1120	80	2.00	232.00	0.0774

Table 4 Grey relational coefficients, grey relational grade and rank for each run

Exp. run	Normalized value		Deviation sequences		Grey relational coefficients		Grey relational grade (GRG)	Rank
	Micro-hardness	Wear rate	Micro-hardness	Wear rate	Micro-hardness	Wear rate		
1	0.266	0.745	0.734	0.255	0.405	0.662	0.534	10
2	0.014	0.896	0.986	0.104	0.337	0.827	0.582	6
3	0.000	0.272	1.000	0.728	0.333	0.407	0.370	15
4	0.415	0.037	0.585	0.963	0.461	0.342	0.401	14
5	0.191	0.731	0.809	0.269	0.382	0.650	0.516	11
6	0.454	0.779	0.546	0.221	0.478	0.694	0.586	5
7	0.523	0.000	0.477	1.000	0.512	0.333	0.423	13
8	0.198	0.029	0.802	0.971	0.384	0.340	0.362	16
9	0.470	0.645	0.530	0.355	0.486	0.585	0.535	9
10	0.796	0.926	0.204	0.074	0.711	0.871	0.791	3
11	0.729	0.380	0.271	0.620	0.648	0.447	0.548	7
12	0.461	0.323	0.539	0.677	0.481	0.425	0.453	12
13	1.000	0.747	0.000	0.253	1.000	0.664	0.832	1
14	0.663	1.000	0.337	0.000	0.598	1.000	0.799	2
15	0.670	0.443	0.330	0.557	0.603	0.473	0.538	8
16	0.919	0.141	0.081	0.859	0.861	0.368	0.614	4

well as the heat input at the stir zone. The grain growth that generally accompanies with the heat input to the stir zone is retarded by the presence of reinforcing material. In addition to this, high frictional heating and intense plastic deformation caused by higher tool rotational speed, result in dynamic recrystallization at the stirred zone (SZ), which is also responsible for grain refinement [32]. Due to the combined effect of grain refinement and SiC reinforcement particles in the matrix, there is an increase in the micro-hardness and a reduction in wear rate. Hamdollahzadeh et al. [33] and Barmouz et al. [34] also observed the similar results.

Figure 4 also indicates that increasing the tool traverse speed decreases the properties of the fabricated surface

Table 5 Response table for grey relational grade

Level	Tool rotational speed	Tool traverse speed	Tilt angle
1	0.4718	0.6041	0.5704
2	0.4716	0.6894	0.5222
3	0.5816	0.4695	0.5165
4	0.6957	0.4577	0.6117
Delta	0.2241	0.2318	0.0952
Rank	2	1	3

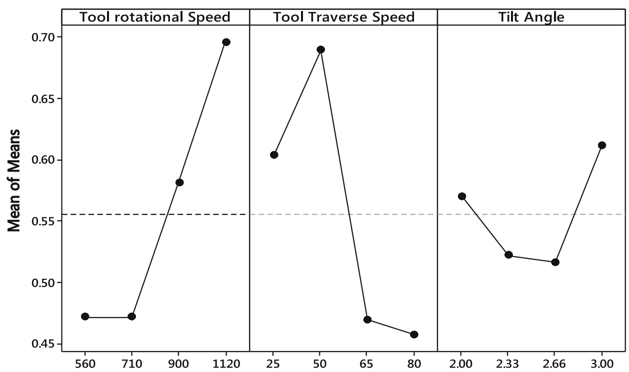


Fig. 4 Main effect plot for mean GRG

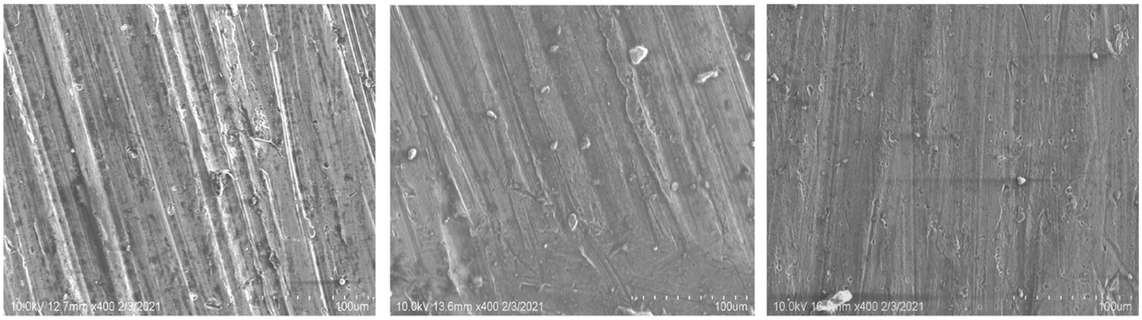
composite. High traverse speed of the tool decreases the contact time of the tool with the base metal, which reduces the frictional heat generation and the amount of stirring at the stir zone. Insufficient heat input retards the grain refinement process, thereby increasing the grain size. There is a reduction in hardness of the material with the increase in its grain size as explained by the Hall–Petch equation [33]. As reported by Kurt et al. [35], uniformly distributed SiC particles can also limit the grain growth. However, the poor mixing of material at the stir zone caused by increased traverse speed lowers the possibility of uniform distribution of SiC particles. All these reasons attribute to the reduction in hardness and increase in the wear rate of the surface composite [6].

The tool tilt angle is also one of the process parameters studied in this work, which is the angle between the spindle and the surface normal to the workpiece. Tilt angle helps in the extrusion of the processed material at the trailing edge of the tool shoulder. It is also responsible for the vertical flow of material and high dispersion of SiC particles at the stir zone. It was reported in the literature [36] that, at the lower tile angle, there was a possibility of void formation due to poor mixing of the material which contributed to the reduction in the mechanical properties of the surface composites. At the larger tilt angle, there was an increase in the flow of the deformed material and resulted in more heat generation due to the higher extrusion force excreted by the tool. More heat generation could have enhanced the grain growth, which led to the formation of the coarse grain structure. [37]. However, highly dispersed SiC particles limited the grain growth through the pinning effect as reported by Sharma et al. [8] and Zarghani et al. [38]. Hence, there was an improvement in the micro-hardness and wear resistance of the Al7075/SiC surface composite when the tool with a tilt angle of 3° was used.

The observations concerning the wear rate of the surface composite show that the homogeneous distribution of SiC particles in the Al7075 affects the wear characteristics. SEM images of the worn out surface of Al7075/SiC surface composite specimens with high, medium and low wear rates obtained from experimental runs 7, 15 and 14 are shown in Figs. 5(a), (b) and (c), respectively. A pattern of parallel scratch marks in the sliding direction is observed in all specimens. More damage and deep scratch marks are observed in the worn out surface of the specimen from run

Table 6 ANOVA for GRG

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	*F*-value	*P* value
Tool rotational speed	3	0.13759	42.63%	0.13759	0.045862	22.84	0.001
Tool Traverse speed	3	0.14908	46.19%	0.14908	0.049693	24.75	0.001
Tilt angle	3	0.02403	7.44%	0.02403	0.008009	3.99	0.070
Error	6	0.01205	3.73%	0.01205	0.002008		
Total	15	0.32274	100.00%				



(a) (b) (c)

Fig. 5 SEM images of the worn out surface of Al7075/SiC composite specimens in experimental runs **a** 7 (high wear rate), **b** 15 (medium wear rate) and **c** 14 (low wear rate)

7. Lower rotational speed and higher traverse speed of the tool in experiment run 7 cause improper blending of matrix and reinforcement and results in the poor distribution of SiC particles, whereas shallow grooves in the direction of sliding are observed in Fig. 5(b). However, the higher rotational speed and the lower traverse speed of the tool in experimental run 14 helps in the uniform distribution of SiC particles in Al7075 matrix resulting in less scratch marks and lower matrix pull out as evident in Fig. 5(c). Presence of SiC particles and their homogenous distribution increases the hardness, thereby improving the wear resistance of the FSP processed surface composites [39].

3.4 Confirmation Experiment

After optimizing the process parameters, a confirmation experiment has to be carried out to analyze the accuracy of the obtained optimal process parameters. During the confirmation test, the experiments were performed with the optimal combination of process parameters. The GRG value of the confirmation experiment was calculated with GRA analysis. For validating the confirmation experiment, the result of the confirmation experiment was compared with the predicted result. The GRG is predicted using the optimal combination of process parameters as:

$$G_p = G_m + \sum_{i=1}^{N} (G_i - G_m) \quad (1)$$

where G_m is the average of grey relational grades of all experimental runs, G_i is the mean grey relational grade at the optimum level of the ith process parameter and N is the number of input parameters that affect the multi-objective characteristics. The value of predicted GRG was calculated using Eq. (1) as:

$$\begin{aligned} G_p &= 0.5552 + (0.6957 - 0.5552) + (0.6894 - 0.5552) \\ &\quad + (0.6117 - 0.5552) \\ &= 0.8864 \end{aligned}$$

The comparisons of the confirmation experiment results and the predicted values are shown in Table 7. It is seen that the experimental values of micro-hardness and wear rate of the surface composite are almost the same as their predicted values. Table 7 also indicates the percentage improvements in micro-hardness and wear rate obtained at the optimal process parameters. When compared with the micro-hardness and wear rate values obtained with the initial best condition, at the optimal combination of process parameters, micro-hardness increases by 1.44% and wear rate decreases by 29.1%. Also, the GRG value at the optimal process parameters improves by 6% with respect to GRG of the initial best condition. Furthermore, the calculated prediction error with the experimental result is found to be 0.43%.

4 Conclusion

In this research work, friction stir processing was used to fabricate Al7075/SiC surface composites. Effects of process parameters such as tool rotating speed, tool traverse speed, and tilt angle on micro-hardness and wear rate were evaluated. These process parameters were optimized and analyzed by using Taguchi-based Grey Relational analysis and ANOVA analysis. From the investigation, the following important conclusions could be deduced:

- Increase in tool rotational speed increased the flow of the base metal and helped with the homogeneous mixing of the reinforcing material as well as the increased heat input at the stir zone. The grain growth was retarded by the presence of reinforcing material which increased micro-hardness and reduced wear rate.

Table 7 Results of confirmation experiment

Properties	Initial best process parameter (Exp. No. 13)	At optimal process parameters		Percentage improvement
		Prediction	Confirmation experiment	
Micro-hardness (HV)	243.87	244.98	247.38	1.44%
Wear rate (mg/m)	0.0323	0.0209	0.0229	29.10%
GRG value	0.832	0.886	0.882	6.00%

- Higher traverse speed of the tool reduced the stirring time and heat generation that led to the reduction in the grain refinement. This caused a reduction in hardness and increased the wear rate of the surface composite.
- Larger tilt angle increased the heat generation due to the higher extrusion force excreted by the tool. Coarsening of the grain structure due to the increased heat generation was obviated by uniformly dispersed SiC particles in the matrix.
- For the optimal micro-hardness and wear resistance, Al7075/SiC surface composite should be fabricated with a tool rotational speed of 1125 rpm, tool traverse speed of 50 mm/min and tool tilt angle of 3°.
- Tool traverse speed was found to be the most influential parameter with 46.19% contribution to the properties of the surface composites, followed by tool rotational speed (32.63%) and tool tilt angle (7.44%), respectively.
- Finally, the confirmation experimental results obtained at the process parameters were found to be close to the predicted values with a prediction error of 0.43%.

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