Investigation of microstructural and wear behavior of Al6061 surface composites fabricated by friction stir process using Taguchi approach

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Keywords: surface composites, dry sliding wear, microstructure, friction stir process, Taguchi

Abstract

The surface composites of aluminum alloys have a higher scope of applications encountering surface interactions in the aerospace, automobile, and other industries compared to the base aluminum alloys. The friction stir process (FSP) is recently the preferred method to prepare aluminum-based surface composites due to its capability to produce improved physical properties and refined microstructure at the surface. The study examines the Al6061 alloy-based surface composite fabricated by FSP for their wear behavior and microstructure. In this study, the Al6061 alloy-based hybrid surface composites are prepared with varying weight% of copper and graphite microparticles mixture as reinforcement by FSP with two tools having unique pin profiles, i.e., threaded cylindrical and plain cylindrical. These prepared composites are investigated for the dry sliding wear test on a pin-on-disc test set-up. The experiments are designed using the L9 orthogonal array and analyzed by the Taguchi approach to obtain the influence of disc speed, load, and reinforcement weight% on wear rate. The significant parameters influencing the wear rate of the samples tested are obtained using ANOVA. Later the effects of the friction stir process and the wear tests on the microstructure of the workpieces are investigated using FE-SEM/EDS tests. It is concluded that the decrease in wear rate with the rise in reinforcement weight% (Cu + graphite) from 2% to 6%. The load has the maximum effect on the wear rate for the samples prepared by threaded cylindrical FSP tool pin profile, while reinforcement weight% affects significantly the wear rate of the samples prepared by FSP with plain cylindrical pin profile tool.

1. Introduction

Aluminum alloys are extensively utilized in various industries like aerospace, automotive, shipbuilding, maritime, railways, and manufacturing due to their superior characteristics like high strength-to-weight ratio, good resistance to corrosion, and high conductivity electrical and thermal conditions. Although, aluminum alloys have a lower surface wear-resistance which can be overcome by preparing surface composites of aluminum alloys with ceramic reinforcements like—SiC, Al₂O₃, B₄C, TiC, AlN, fly ash, TiB₂, TiO₂, etc [1]. These surface composites are excellent in surface hardness and wear behavior while retaining the lightweight characteristics in the bulk material. Friction stir processing (FSP) manufactures aluminum alloy-based surface composites with single or multiple reinforcements for enhanced surface properties. FSP is capable of changing the material properties through localized plastic deformation [2]. It is a derived process from friction stir welding (FSW). The specific non-consumable tool used in FSP/FSW is comprised of a cylindrical shoulder with a pin integrated at the end so that shoulder to pin diameter is generally 3:1. The FSP tool is advanced towards the workpiece so that the pin inserts into the workpiece surface and the shoulder surface rubs over it laterally along a path. The tool pin stirs the material around it, leading to a homogeneous refinement of grains and improved

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mechanical properties [3]. The surface composites are fabricated when the aluminum alloy matrix plates with holes or grooves on their surfaces are filled with suitable reinforcements. Then FSP is conducted over the reinforced surfaces [4]. With this improvisation, the application areas of aluminum alloys are intensified and widened.

Many researchers have used the analysis of parameters during the fabrication of surface composites using different techniques. Some authors [3] investigated the manufacturing of Al6061 alloy during FSP. They found the influences of the number of passes in FSP, type of reinforcements, and post-treatments of the material, upon the microstructural and physical characteristics of the fabricated surface composites. The authors used the Taguchi method to optimize maximum hardness, tensile strength, and elongation percentage. Also, ANOVA was applied to get the parameter with maximum effect on the output responses. It is concluded that surface composites’ surface hardness and tensile strength increase when Al₂O₃ is added as the reinforcement. Also, the hardness of the stir zone increased by the post-heat treated Al6061 (T6) and by third pass FSP. Another group of researchers optimized the friction stir processing parameters—rotation speed of tool, feed, and number of FSP passes on the hardness of the composite fabricated using the Taguchi method. In another work, the authors investigated the effects of wear and microstructure of the aluminum alloy-based composites with varying amounts of addition of reinforcements (by % volume) [6]. The fabricated composites were characterized by the SEM, XRD, hardness, and wear tests. It was found that the higher amount of reinforcement dispersed in aluminum alloy matrices increases the hardness, density, and resistance to wear of the fabricated composites.

Another work to find the influence of process parameters—tool pin profile, depth of penetration, the rotation speed of tool, and tool feed on the ultimate tensile strength of the Al6061/SiC composite manufactured by FSP, was attempted using Taguchi method of analysis [7].

Some authors studied the effects of process factors on the formability of processed (by FSP) Mg AZ31B sheets during biaxial stretching. The parameters were optimized using Taguchi’s ANOVA statistical technique [8]. Also, they developed an empirical model to estimate the relationship between the parameters with formability. Similar work was attempted for input parameters optimization of friction stir processed Al6061/TiB₂. The effects of the input parameters—reinforcement%, traverse speed, rotational speed, and axial load on FESEM image and physical surface properties are examined. The Taguchi (one variable) and Grey Relational Analysis (multi-variable) are used for the optimization of process parameters [9].

Many researchers have optimized parameters using the Taguchi approach for various manufacturing operations. A group of authors, in their work, used Taguchi analysis and Grey Relational Analysis for the optimization of different parameters after the Wire-Electrical Discharge Machining process of a Copper and Boron Nitride composite fabricated by FSP. An orthogonal L27 array was designed in this analysis, and the most significant factor was obtained using ANOVA [10]. Other manufacturing operations like the turning of hardened steel were analyzed by parametric optimization by the Taguchi method. ANOVA and regression analyses were performed to study the surface roughness and coefficient of friction for the influence of process parameters [11]. Other techniques for the design of experiments like Factorial methods are also used for ANOVA analysis for wear of metal matrix composites. The influence of reinforcement weight%, load, disc speed, and sliding distance is investigated on wear rate and friction coefficient of the aluminum-based composite with varying amounts of reinforcements SiC and graphite manufactured by powder metallurgy [12].

The optimization of parameters is also performed for the wear of the composites. A group of authors attempted a dry sliding-type wear test for the composite fabricated by epoxy resin composite with fly ash as reinforcement using Taguchi design of experiments [13]. An orthogonal array was analyzed to inspect the effects of the parameters for minimum wear and frictional force using ANOVA and TOPSIS methods. The influential parameters were selected as - tool rotation speed, tool feed, the tilt angle of the tool, and penetration depth on the surface hardness of Magnesium based alloy using Taguchi optimization method by signal to noise (S/N) ratio analysis [14]. Another work includes designing a mathematical model for the wear test of aluminum-based composite reinforced with AlB₂ analyzing the wear rate for the effects of parameters using the Taguchi approach. ANOVA obtained the S/N ratio analysis of the orthogonal array. The process parameters were optimized and validated conditions [15]. Another work was performed to inspect the physical and wear characteristics of Al6061 alloy surface composites manufactured by reinforcing Al₂O₃ and SiC particles by friction stir processing [16]. Taguchi method was utilized for S/N ratio analysis using Minitab software to inspect the most influential process parameters on the output responses and the optimum parameters’ optimum conditions. High strength materials, i.e., AA7075 reinforced with varying amounts of TiC by casting technique [17] and surface-modified 60/40 brass plate [18], were tested for wear behavior and analyzed by the Taguchi method. The optimum conditions of reinforcement %, load speed, and sliding distance were evaluated using ANOVA and were validated by confirmation tests. Rajesh Siriyala et al [19] investigated the aluminum-based composite’s dry sliding wear behavior reinforced by the graphite. The optimization of the parameters and their influence on wear rate and the friction coefficient was attempted using PCA. Al6061 alloy-based matrix composite reinforced with SiC and graphite coated with copper particles was investigated for its wear behavior with the Taguchi design.
S/N ratio analysis was used to obtain the influence of process parameters—disc speed, sliding distance, and load on the specific wear rate and coefficient of friction [20]. Taguchi’s approach was implemented in analyzing the hardness and wear for the wear test of Al6061/SiC composite fabricated by powder metallurgy. The effects of process parameters on the wear rate were tested. Later SEM of the worn surfaces was performed to analyze the wear mechanism of the samples [21].

Jacob John et al [22] attempted the analysis of Taguchi design to investigate the friction stir processing parameters. They used Response surface methodology to develop a mathematical model capable of predicting the output parameters—ultimate tensile strength, yield strength, and elongation as an influence of FSP process parameters. Another group of researchers [23] welded Al2024 alloy by FSW utilizing the X-ray radiographic image using image processing techniques. The Taguchi-based array was designed, analyzed the experiments, and obtained the optimum minimum defect area and maximum tensile strength parameters. Also, the erosion performance of the Al7075 alloy-based composite manufactured by FSP was analyzed by optimizing input parameters using the Taguchi approach [24]. Optimization of the wear rate has been performed by a group of authors [25] for Al/SiCnp/E-glass fiber composite using the Taguchi approach analyzed by Artificial Neural
Networks. Some authors [26, 27] have examined the turning tool wear to examine the influence of deep cryogenic cooling on WC coated inserts using Taguchi’s Method. Another work of same group of authors [28] included a new approach of combining powder metallurgy and angular pressing to prepare Al6061 based composites with varying reinforcements and inspecting their wear behavior.

From the literature, it is clear that several kinds of research have been undergone for the wear test of composites and their microstructural analysis. However, the hybrid surface composites manufactured using the FSP technique with \textit{in situ} and \textit{ex situ} reinforcements and their wear analysis with a microstructural characterization of the reinforcements is rarely studied. In this proposed manuscript, the Al6061 based hybrid surface composites reinforced with a varying weight\% mixture of copper and graphite microparticles (1:1 by weight) are fabricated using FSP. These composites are mounted on a pin-on-disc tester to inspect their tribological behavior. The experiments are designed and analyzed by the Taguchi approach using Minitab software. The influence of the input factors, i.e., disc speed, reinforcement weight\%, and load, on the wear rate for all the specimens is investigated using ANOVA. A regression model is predicted to depict the relation between process parameters with wear rate. Later, these specimens are tested on the Field Emission Scanning Electron Microscopy - Energy Dispersive Spectroscopy (FESEM-EDS) for studying the effects of the wear test on the microstructure.

2. Materials and methods

The matrix material for the surface composite fabrication is Al6061 alloy (Mn = 0.0\%–0.15\%, Fe = 0.0\%–0.70\%, Mg = 0.80\%–1.20\%, Si = 0.40\%–0.80\%, Cu = 0.15\%–0.40\%, Zn = 0.0\%–0.25\%, Ti = 0.0\%–0.15\%, Cr = 0.04\%–0.35\%, Others = 0.00\%–0.15\%, Al = balance). Al6061-T6 alloys are precipitation hardened and good in strength to weight ratio. So, they have high formability and weldability properties and act as excellent matrix base materials. For this purpose, Al6061-T6 alloy plates of size 100 mm \times 30 mm X 20 mm are purchased from Shree Balaji Steel House, Bhosari, Pune, India. The reinforcement for hybrid composite fabrication is prepared by mixing copper and graphite microparticles. On the basis of the literature, it is found that copper forms a strong intermetallic compound with Al6061 alloy. Hence it is selected as \textit{in situ} reinforcement for Al6061 based composite. However, graphite acts as a solid lubricant which imparts its lubricative properties to the composite reducing wear and hence is selected as \textit{ex situ} reinforcement for the surface composite fabrication. For this purpose, copper and graphite particles (50 to 40 microns size) are purchased from Bhoomi Metals and Alloys, Mumbai, India. The copper and graphite microparticles are mixed in equal weights inside a planetary ball mill, with the drum rotating at 300 rpm for half an hour [29]. A slurry is prepared by adding a measured amount of ethanol to the milled copper and graphite particles mixture. This slurry is filled in the holes drilled on the Al6061 matrix plates in three different weights \%, i.e., 2\%, 4\%, and 6\%. The tool material selected for friction stir processing (FSP) is H13 tool steel. The FSP tool shoulder to pin diameter selected is 3:1, i.e., 15 mm and 5 mm, respectively. Two different pin profiles are used in this fabrication, threaded cylindrical (Tool A) and plain cylindrical (Tool B). These customized tools are purchased from Vishwakarma Steel Works, Roorkee, Uttarakhand, India. FSP is conducted on a CNC milling machine (Jyoti CNC Automation Ltd), available at Advanced Manufacturing Laboratory, Symbiosis Institute of Technology, Pune, India (as shown in figure 1 (a)). Fabrication of the composites is accomplished using FSP tools (A and B) mounted on the tool holder one at a time. Hence the composites are prepared for each of the reinforcement wt.\% (2\%, 4\%, 6\%) and tool (Tools A and B) on the milling machine with selected parameters, i.e., 1400 rpm, 40 mm s^{-1} traverse speed, 5 mm depth of cut (equal to pin length), travel path = 80 mm. Six composites are prepared (three different reinforcement weights\% (2\%, 4\%, 6\%) and processed by each tool, i.e., Tool A and Tool B). These composites fabricated are tested for their wear behavior on a Ducom dry sliding-type pin-on-disc tester, integrated with two sensors—an LVDT and a load cell sensor, available at the Advanced Manufacturing Laboratory, Symbiosis Institute of Technology, Pune, India. Blocks of specific sizes (12 mm \times 12 mm \times 20 mm) are cut from the fabricated composites using a Wire EDM cutter at a commercial industry in Bhosari, Pune, India, according to the sample holder configuration on the wear tester. Eighteen samples are prepared for the wear tests (nine each for the composites fabricated by Tool A and B, respectively). After the wear tests, six selected samples (with maximum and minimum wear rates) are tested for their microstructure on a Field Emission Scanning Electron Microscope equipped with Energy Dispersive Spectroscopy (FESEM-EDS) setup, available at Central Instrumentation Facility, Pune University, Pune, India. One of these samples is examined on FESEM before the wear test too, in order to study the effect of wear test alone on the microstructure of surface composites manufactured by FSP.
**Table 1.** Set of hole dimensions for specimens numbered 1 to 6 for H13 tools of two-pin profiles (1—Threaded cylindrical, 2—plain cylindrical).

| Hole diameters | Hole diameters |
|----------------|----------------|
| HOLEs (pin 1—Th. Cyl.) | 3 mm | 4 mm | 5 mm |
| HOLEs (pin 2—Plain Cyl.) | 3 mm | 4 mm | 5 mm |
| Hole Depth | 3 mm | Sample 1 (2% reinforcement) | Sample 2 (4%) | Sample 3 (6%) |
| Hole Depth | 3 mm | Sample 4 (2%) | Sample 5 (4%) | Sample 6 (6%) |
3. Experimental procedure

3.1. Friction stir processing (FSP)

The FSP experimentation is conducted on the CNC Vertical milling machine. The machine parameters are selected on the basis of several trial FSP experiments conducted for surface composite fabrication based on achieving good visibility of alloy preparation, i.e., with minimum machine vibration and uniform tool-work contact. These are; tool rotation speed = 1400 rpm, tool feed = 40 mm min⁻¹, depth of cut = 5 mm (pin length), path length of FSP = 80 mm, number of FSP passes = 2 (opposite order). The FSP is conducted clockwise, followed by counter-clockwise direction for each composite being fabricated so that the reinforcements are dispersed inside the matrix surface uniformly. Initially, four holes of specific dimensions are drilled at equal distances in the 80 mm pathlength on each of the six specimens (on 100 mm × 30 mm face) with the help of HSS drill bits for varying reinforcement weight% deposition, as shown in table 1. The prepared reinforcement slurry is then filled manually into the holes. Three different reinforcement weight% addition is obtained due to varying hole dimensions. Later, the FSP is carried out with the rotating tool given a vertically downward motion towards the specimen (Al6061 matrix reinforced with reinforcement slurry) clamped to the machine table. The FSP set-up photograph is shown in figure 1(a) and an AutoCAD drawing of the set-up is shown in 1(b). The tool is advanced towards the work specimen so that the pin is inserted into the specimen surface and the shoulder rubs on the matrix surface. Then the tool is moved across the path (length = 80 mm) over the reinforced surface to form surface composites. As shown in table 1, for sample 1, the hole volume is given (Π 3²/3 = 84.78 mm³). So, the weight% of reinforcement added in the matrix in sample 1 can be calculated as (Volume of each hole X density of reinforcement slurry Cu + Gr)/ (Volume of tool travel in matrix X density of Al6061). The weight% of copper and graphite mixture added in samples 1 and 4 is calculated at 2%. Similarly, for samples 2 and 5, weight% added is 4%, and for samples 3 and 6, weight% added is 6%. Accordingly, all the six specimens reinforced with the slurry are processed. The photograph of the friction stir processed samples is shown in figure 1(c) and the macrostructure of the processed (stir) zone as taken for one of the samples by FESEM with minimal magnification is shown in figure 1(d) that clearly shows the uniformly...
dispersed particles of copper and graphite particles in reduced sizes into the matrix alloy surface. In these set of experiments, each specimen is prepared two times separately, and the better one (as per visibility) is selected as the final to minimize the errors. Hence, twelve samples are prepared, and six are selected for investigations.

### 3.2. Wear test

Surface composites as-fabricated by FSP are investigated for their wear behavior by the set of experiments on a dry sliding-type pin-on-disc set-up as per the ASTM G99-05 standard. According to the machine configuration, the specimens to be tested are prepared in a specific size, i.e., 12 mm × 12 mm × 20 mm using Wire EDM cutter. The three factors to be studied selected are Disc speed, reinforcement weight %, and load applied with three levels each, as shown in table 2.

In this study, Ducom pin on disc Tribometer is used for accurate tribological characterization of the samples. The set-up is integrated with two sensors—an LVDT and a load cell to measure real-time measurement and analysis of the wear and frictional behavior in terms of wear measurement (µm) and frictional force (N), respectively with integrated friction and wear controller. The set-up is shown as an image and a line diagram in figure 2(a) and (b). A monitor connected to the controller is installed with Winducom software, an integrated machine control software with data acquisition and post-processing features.

The samples are one by one fixed on the specimen holder so that the composite surface is in contact with the disc. The disc is made of tungsten carbide-coated steel, with a surface hardness of 7.0 GPa and a surface roughness of 0.5 µm [27]. The disc speed and time are set using the controller. Then, the load is placed according to the plan (as shown in table 3(a) and (b)). The surface, which is to be tested for wear, is kept stationary and is pressed on the rotating steel disc following the track diameter of 85 mm. Before the wear tests, the specimen face (to be tested) and the disc face are polished with the help of abrasive papers (80–120 grit size) to ensure uniform surface contact of the specimen face with the disc. A wear test is performed for the samples one by one for a specific duration selected (ten minutes in this study). The wear measurement (µm) and frictional force (N)

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**Table 2. Input parameters and levels selected.**

| Process parameters | Unit | 1   | 2   | 3   |
|-------------------|------|-----|-----|-----|
| Disc Speed        | rpm  | 200 | 400 | 600 |
| Reinforcement wt.%| %    | 2   | 4   | 6   |
| Load              | N    | 30  | 40  | 50  |

**Table 3.** (a) Design of Experiments for the wear test of samples fabricated by FSP tool A. (b) Design of Experiments for the wear test of samples fabricated by FSP tool B.

| S.No. | Disc Speed (rpm) | Reinforcement wt% | Load (N) | Wear Rate (g/min) | S/N Ratio |
|-------|------------------|-------------------|----------|-------------------|-----------|
| (a)   |                  |                   |          |                   |           |
| 1     | 200              | 2                 | 30       | 1.41              | −2.984 38 |
| 2     | 200              | 4                 | 40       | 1.45              | −3.227 36 |
| 3     | 200              | 6                 | 50       | 1.46              | −3.287 06 |
| 4     | 400              | 2                 | 40       | 1.61              | −4.136 52 |
| 5     | 400              | 4                 | 50       | 1.65              | −4.349 68 |
| 6     | 400              | 6                 | 30       | 1.33              | −2.477 03 |
| 7     | 600              | 2                 | 50       | 1.75              | −4.860 76 |
| 8     | 600              | 4                 | 30       | 1.57              | −3.917 99 |
| 9     | 600              | 6                 | 40       | 1.51              | −3.579 54 |

| (b)   |                  |                   |          |                   |           |
| 1     | 200              | 2                 | 30       | 1.07              | −0.587 68 |
| 2     | 200              | 4                 | 40       | 1.1               | −0.827 85 |
| 3     | 200              | 6                 | 50       | 1.08              | −0.668 48 |
| 4     | 400              | 2                 | 40       | 1.3               | −2.278 87 |
| 5     | 400              | 4                 | 50       | 1.1               | −0.827 85 |
| 6     | 400              | 6                 | 30       | 1.01              | −0.086 43 |
| 7     | 600              | 2                 | 50       | 1.4               | −2.922 56 |
| 8     | 600              | 4                 | 30       | 1.2               | −1.583 62 |
| 9     | 600              | 6                 | 40       | 1.1               | −0.827 85 |
values are displayed on the controller, and the individual graphs are generated in the Winducom software. The photograph of the samples after the wear test is shown in figure 2 (c).

Taguchi approach is used to design the experiments, identify the controlling factors and to obtain the most influential factor in the process. The reason for selecting this method is its practicality in designing systems with a variety of factor level combinations and reduced variance. The experiments are conducted according to the three-level design using Minitab software. The L9 orthogonal array design is selected to obtain the effects of input factors on the wear rate with minimum experimentation cost and effort. Two sets of experiments (nine samples each) are planned, one for the samples cut from the composites fabricated by FSP Tool A, another for pieces of the composites fabricated by FSP Tool B. All the samples are inspected for their weights before and after the wear tests. After the experiments, the wear rate and S/N ratios are calculated as shown in equations (1) and (2).
$W_R = \left( \frac{w_1 - w_2}{\text{Time}} \right) \times 100$ \hspace{1cm} (1)

\[ S/N = -10 \times \log \left( \frac{\sum(R^2)}{n} \right) \] \hspace{1cm} (2)

where $W_R$ = Wear Rate (in g/min),

$w_1$ = mass of the sample before wear test,

$w_2$ = mass of the sample after wear test,

$S/N = \text{signal to noise ratio},$

$R = \text{Wear rate response for each sample test, and}$

$n = \text{Number of responses.}$

The $S/N$ ratios represent the mean to standard deviation ratio. In this analysis, smaller-the-better quality is selected for the wear rate for enhanced material machinability.

The experimental designs for the two sets of experiments for the wear tests of composites fabricated by Tool A and Tool B are shown in table 3 (a) and (b). The wear rate obtained as the result of wear tests and the $S/N$ ratios calculated for all the cases are shown as response columns.

The coefficient of friction graphs are auto-generated from the Winducom 2010 software during the pin-on-disc wear tests. The graphs for the samples with minimum and maximum wear rates, selected from serial numbers—1, 6, and 7 from both the sets of experiments (table 3(a) and (b)) processed by Tool A and Tool B, respectively, numbered as Sample numbers 1 to 6, are shown in figure 3.

3.3. Microstructural examination
The FSP fabricated surface composites before (one sample), and after (six samples) the wear tests, are investigated to study effects of FSP and the wear test parameters (disc speed, load and reinforcement weight%) on their microstructure. Out of eighteen samples tested for wear tests, six samples with maximum and minimum wear rates are selected (as discussed in section 4 b) for microstructural examination by FESEM/EDS. One of these six specimens is examined on FESEM before the wear test too in order to identify the effects of wear tests alone on the microstructure of FSP fabricated composites. These samples were cut in specific cube sizes (i.e., 4 mm $\times$ 4 mm $\times$ 4 mm) with a Wire EDM machine according to the machine configuration for microscopic examinations. All the samples are marked at the top faces to be inspected. FESEM is used to study the effect of FSP and wear tests on the microstructure of the samples. The energy dispersive spectroscopy (EDS) characterizes the reinforcements dispersed on the surface.

4. Result and discussion

4.1. Wear test analysis
The analysis of the Taguchi design is performed in terms of obtaining the influence of three factors, i.e., disc speed, reinforcement wt.%, and load, for three levels each, on the wear rate are investigated (table 3(a) and (b)) using Minitab software. This analysis includes - i) Effects of Disc Speed, Reinforcement wt.% and Load on Wear, ii) Estimation of influential parameters, iii) Multiple regression analysis, as discussed in the following subsections.
4.1.1. Effects of disc speed, reinforcement wt.% and load on wear

Figure 4(a) and (b) depict the Main Effects Plots for Means (showing the mean values of wear rate) and for S/N ratios (showing S/N ratios for wear rate) respectively for the samples processed by FSP tool A. Similarly, figure 5(a) and (b) depict the Main Effects Plots for Means and S/N ratios, respectively, for the samples processed by the FSP tool B. These graphs show the effects of disc speed, reinforcement wt.%, and load on the wear rate.

The disc speeds selected are 200 rpm, 400 rpm, and 600 rpm for varying reinforcement wt.% and load. The mean wear rate appears to be linearly increasing with rise in disc speed, as shown in figure 4(a) and 5(a) for both sets of experiments. However, figure 4(b) and 5(b) show the S/N ratios with smaller-the-better quality characteristics so that for minimum wear rate, 6% is the optimum reinforcement wt.% for minimum wear.

The load is varied as 30 N, 40 N, and 50 N for different reinforcement wt.% and speeds. It is clear that with a rise in load, the wear rate increases almost linearly and non-linearly respectively for both the sets of experiments, as shown in figure 4(a) and 5(a), respectively. Figure 4(b) and 5(b) show the signal-to-noise ratios with smaller-the-better quality characteristics so that for minimum wear rate, 30 N is the optimum load for minimum wear.

Table 4. (a) Analysis of Variance for S/N Ratio for samples processed by FSP tool A. (b) Analysis of Variance for S/N Ratio for samples processed by FSP tool B.

| Source                  | DF | Seq SS  | Adj SS  | Adj MS  | F     | P    | %P   |
|-------------------------|----|---------|---------|---------|-------|------|------|
| Disc Speed              | 2  | 1.3631  | 1.3631  | 0.681   | 15.32 | 0.069| 30.99293 |
| Reinforcement wt%       | 2  | 1.3138  | 1.3138  | 0.656   | 13.03 | 0.071| 29.87199 |
| Load                    | 2  | 1.6204  | 1.6204  | 0.810   | 16.07 | 0.059| 36.84318 |
| Residual Error          | 2  | 0.1008  | 0.1008  | 0.050   |       |      |      |
| Total                   | 8  | 4.3981  |         |         |       |      |      |
| R²                      |    | 95.46   |         |         |       |      |      |

| Source                  | DF | Seq SS  | Adj SS  | Adj MS  | F     | P    | %P   |
|-------------------------|----|---------|---------|---------|-------|------|------|
| Disc speed              | 2  | 1.8196  | 1.8196  | 0.9098  | 1.98  | 0.335| 27.62243 |
| Reinforcement wt%       | 2  | 2.9932  | 2.9932  | 1.4966  | 3.26  | 0.235| 45.43826 |
| Load                    | 2  | 0.8574  | 0.8574  | 0.4287  | 0.93  | 0.517| 13.01576 |
| Residual Error          | 2  | 0.9171  | 0.9171  | 0.4586  |       |      |      |
| Total                   | 8  | 6.5874  |         |         |       |      |      |
| R²                      |    | 83.26   |         |         |       |      |      |
4.1.2. Estimation of influential parameters

The significance of the factors has been evaluated using ANOVA (Analysis of Variance) for S/N ratios by Minitab software, shown in table 4(a) and (b). This analysis helps to identify the most influential parameters among disc speed, reinforcement wt.%, and load on the wear rate of the friction stir processed samples. The contribution factor (%P) of the parameters are calculated by finding out the percentage of the corresponding Sequential Sum of Squares (Seq SS) values with the Total Seq SS value in the table. From the values of %P for both the tables, it can be said that for the samples processed by FSP tool A, the load (highest contribution factor) has the highest significance, followed by disc speed and reinforcement wt.% respectively. Similarly, for the samples processed by the FSP tool B, the most influential parameter is the reinforcement wt.% followed by disc speed and load, respectively, for the S/N ratio analyses.

Table 5(a) and (b) show the response tables for the S/N ratio analysis for the samples processed by the FSP Tools A and B, respectively. Then, the delta values for all three factors are calculated using the subtraction of minimum from the maximum values for both cases. Since the load has the highest value of delta in table 5(a), it has the highest significant effect on the wear rate, followed by disc speed and reinforcement wt.%, respectively, for the samples processed by FSP tool A. Similarly, table 5(b) shows that reinforcement wt.% has the most significant influence on the wear rate, followed by disc speed and load, respectively, for the samples processed by FSP tool B.

4.1.3. Multiple linear regression analysis

This analysis is performed using the Minitab 19 software to develop the empirical model for predicting the wear rate as the function of the factors - disc speed, reinforcement wt.%, and load. The predictive model equations obtained for wear rate for the samples processed by the threaded cylindrical FSP tool pin profile and plane cylindrical pin profile, respectively, are shown below (equations (3) and (4)),

\[
W_R = 1.1467 + 0.000425 D_S - 0.03917 R_F + 0.00917 L
\]

(3)

\[
W_R = 0.994 + 0.000375 D_S + 0.00500 L - 0.0483 R_F
\]

(4)

where, \(W_R\) = Wear Rate,
\(D_S\) = Disc Speed,
\(R_F\) = Reinforcement wt.%, and
\(L\) = Load.

The coefficient of determination (i.e., \(R^2\)) implies a statistical measure representing the proportion of the variance for the wear rate explained by the factors, i.e., disc speed, reinforcement wt.%, and load in the predicted regression model. This analysis shows the capability of the regression model, i.e., how well the model predicts the wear rate for new observations. The value of the coefficient of determination (in%) is always between 0 to 100. It should be close to 100 for a good fit of the model. For the samples of both sets of experiments, the value of \(R^2\) is 95.46% and 83.26%, respectively, which shows wear rates are estimated with some residual error or variability. This model helps us determine that the predicted equation fits with the data but is less able to provide the correct predictions for further observed wear rates. The regression model for the first set of samples (threaded cylindrical pin profile) fits better than the model for the second case. The significance of the coefficients is inspected from the residual plots.
4.2. Microstructural analysis

FESEM/EDS examines the microstructure to study the mechanism of wear and the effect of factors and wear rate on the microstructure of the samples. These samples are prepared in specific sizes (4 mm × 4 mm × 4 mm) according to the FESEM machine configuration with the help of a Wire EDM cutter. The microstructural inspection is conducted on the FESEM/EDS set-up available at Central Instrumentation Facility, Pune University, Pune.

Figure 6 shows the optical microscopic and FESEM images of a friction stir processed sample before and after the wear tests. In the microstructure of the sample processed by FSP (figure 6(a), (b)), the reinforcement particles (copper and graphite) can be seen as uniformly distributed into the matrix (Al6061 alloy) surface by FSP with reduced particle sizes. It is found that, after FSP, the reinforcements, i.e., copper and graphite particles were reduced in sizes from the range of 50 to 40 micrometers to approximately the range of 0.5 to 5 micrometers. The reason for this massive reduction in particles size is the excessive loads caused by FSP. However, in the microstructure of the processed sample after the dry sliding wear test (figure 6(c), (d)), the scratches caused by specimen wear on its track on the disc are visible. Also, the delamination is visible which occurs due to the rubbing of excess graphite particles sticking on the specimen surface. The particle sizes of the reinforcements are found to be further reduced after the wear tests due to high loads during the wear tests.

Samples 1 to 6 (as explained in section 3 (iii)) with minimum and maximum wear rates processed by both the FSP Tools (A and B) are examined for their microstructure by FESEM/EDS on the worn surfaces. The microstructures of the worn surfaces of the six specimens (with displayed factor specifications) are shown in figure 7. The deformation or scratch marks are visible on the surfaces, which verifies the significant wear of the material. These scratch marks are quite parallel since the relative motion between the disc and workpiece is the circular motion in the same direction on the same track. However, these marks are at non-uniform distances from each other. The reason may be the irregularities at the surface being worn out. During the wear test, since the workpiece surface is rubbed over the same track on the rotating disc, some particles get separated from the workpiece surface and accumulate as foreign particles. When get rubbed again on the surface with the disc, these particles lead to higher abrasion and result in chipping or delamination of material at a few places on the surfaces.

As explained earlier in the text, it is verified by from figure 7, that the wear test samples with higher reinforcement wt.% (6% copper and graphite) show minimum coefficient of friction and hence minimum wear rate. The reason is the releasing and smudging of graphite particles on the wear surface of the composite acting as...
Figure 7. (a) Sample 1%–2%Cu-Gr, 30 N, 200 rpm; (b) Sample 2%–6%Cu-Gr, 30 N, 400 rpm; (c) Sample 3%–2%Cu-Gr, 50 N, 600 rpm; processed by Tool A; (d) Sample 4%–2%Cu-Gr, 30 N, 200 rpm; (e) Sample 5%–6%Cu-Gr, 30 N, 400 rpm; (f) Sample 6%–2% Cu-Gr, 50 N, 600 rpm; processed by Tool B.

Figure 8. FESEM images of sample 1 with higher magnifications: (a) After FSP before wear test; (b) After FSP and wear test.
a solid lubricant film between the sliding wear couple. The lower delamination visible in figure 7(b) and (e) clearly justifies the lubrication property of graphite. Hence the surface composites with 6% reinforcement addition are more wear-resistant than those with 2% and 4% addition. As the load and the disc speed during the wear test are high (50 N and 600 rpm), the resultant friction is higher, and the temperature at the rubbing surface of the samples increases. This high temperature causes local blackening at some parts of the surfaces. Also, the matrix material and the reinforcement particles may get delaminated in powder form from the composite surface due to high temperatures. The ex situ reinforced graphite particles have a higher tendency to get delaminated in comparison to copper particles which have a stronger intermetallic bonding with the matrix Al6061, which prevents them from escaping. The delamination occurs due to the faults in the reinforcement addition technique during the friction stir process and hence is undesirable. As a result, larger wear delamination is visible for figure 7 (c) and (f), which shows a more significant amount of material removed. These samples also show higher values of friction coefficients.

Some other information can be drawn from the figure 8(a) and (b), which shows FESEM images of Sample 1 at higher magnifications before and after wear tests. Figure 8(a) shows the sizes of the particles (0.4 to 1.5 micro meters approx.) in the sample after FSP. Viewing the images of all the samples, it is concluded that the reinforcements, i.e., copper and graphite particles have undergone an appreciable reduction in size from the range of 50 to 40 micro meters to the range of 0.5 to 5 micro meters. The reason for this massive reduction in particles size is the excessive loads and stresses caused by the FSP. As shown in figure 8(b), the chamfering of the crystal-shaped metal particles occurs so that the copper particles appear to be almost spherical in shapes after the
wear tests. The reason is due to increased, dynamic and repeated loadings on the composite surface during the FSP and wear tests. The higher the amount of reinforcement % added in the surface composite, the larger the clusters of reinforcement particles are visible.

In figure 9, the EDS results are shown for one of the samples (microstructure figure 7 (b)). The mapping of the elements on the microstructure is done to characterize all the elements on the surface. It can be seen that there is the formation of oxide layers of aluminum and copper particles in a large amount due to friction caused by high heat produced between the rotating disc and the worn surface during the wear test. This oxide layers formation protects further wear on the surface. Hence oxidation affects the friction coefficient and the wear rate of the surface composites.

5. Conclusion

In this study, the Al6061 based hybrid surface composites are manufactured by FSP using two FSP tool geometries—threaded cylindrical pin profile and plain cylindrical pin profile. In the proposed study, the Al6061 matrix is reinforced with varying copper and graphite microparticles mixture. A wear test is performed on the dry sliding-type wear tester to examine the fabricated composites' wear behavior. The Taguchi approach analyzes the influence of various parameters, i.e., tool pin profile, reinforcement wt.%, disc speed, and load affecting the wear rate in the tested samples. An L9 orthogonal array is designed and analyzed on Minitab software. ANOVA is implemented for obtaining influential parameters for the smallest wear rate. The following inferences are made from the experimentations:

- It is verified that the wear of Al6061 alloy-based Cu and graphite-reinforced surface composites fabricated by the FSP tools with threaded cylindrical pin profile and plane cylindrical pin profile shows almost similar wear rate trend. With the rise in disc speed from 200 rpm to 600 rpm and the increase in the load (30 N to 50 N), the wear rate increases for samples fabricated by the tool pin profiles.
- The wear rate decreases with a rise in reinforcement wt.% (Cu + graphite) from 2% to 6%. Hence the composites undergo lesser wear for higher reinforcement wt.% for the samples fabricated by both the FSP tool pin profiles.
- Using ANOVA analysis, it is found that for the composites prepared by threaded cylindrical FSP tool pin profile, after wear test, the most influential parameter affecting wear rate is the load, and least significant is reinforcement wt. %.
- Similarly, in the samples prepared by plane cylindrical FSP tool pin profile, after wear test, the most influential parameter affecting wear rate is reinforcement wt.%, followed by disc speed and load, respectively.
- In regression analysis, it is found that the predictive model estimates the wear rate with some residual error or variability. By the determined contribution factor, the predictive model for the samples processed by the FSP tool with threaded cylindrical pin profile is a better fit (i.e., predicts wear rate with higher accuracy) than that for the samples processed by FSP tool with plane cylindrical pin profile.
- For higher speeds and loads (600 rpm and 50 N) in the wear tests, the delamination of the reinforcement particles takes place from the composite surface. This delamination occurs due to weak intermetallic bonding, leading to more material removal.
- Due to high loads and stresses applied on the samples during the friction stir process and during the wear tests, the reinforcement particle sizes are reduced from 40 micrometers approximately to 400 nm. The higher the weight% of reinforcement, the greater is the amount of size reduction.
- Also, the shape of the copper particles is chamfered at the corners, and they appear to be almost spherical due to high loads and stresses applied.
- In EDS analysis, it is found that an oxide layer is formed on the worn surfaces due to the formation of aluminum and copper oxides due to high heat produced between the surface and the disc. This oxide layer on the surface leads to improved resistance to wear on the sample surface.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available with the corresponding author on reasonable request.
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