Studies on friction stir processing parameters on microstructure and micro hardness of Silicon carbide (SiC) particulate reinforced Magnesium(Mg) surface composites

T Rajmohan¹, K Gokul Prasad¹, S Jeyavignesh¹, K Kamesh¹, S Karthick¹, S Duraimurugan¹.

¹ Department of Mechanical Engineering, Sri Chandrasekharendra Viswa Mahavidyalaya, Kanchipuram -631561, Tamilnadu, India.

Abstract. Friction stir processing (FSP) is promising as a capable method for producing surface composites in aluminum and magnesium-based alloys. In FSP a specifically intended rotating cylindrical tool that includes of a pin and shoulder that have dimensions relative to the plate thickness. The pin of the rotating tool is forced into the material and the shoulder comes into contact with the surface of the sheet, and then pass through in the desired direction. The present work focused to fabricate Silicon carbide (SiC) particulate reinforced Magnesium (Mg) surface composites. The effect of FSP parameters such as tool rotational speed, processing speed and wt % SiC on microstructure and micro hardness was investigated. A groove was contrived on the 6mm thick Mg plates and packed with SiC particles. FSP was carried out using Taguchi’s L9 orthogonal array. Scanning electron microscopy and XRD were employed to study the microstructure of the fabricated surface composites. The results indicated that the selected FSP parameters significantly influenced the area of surface composite, distribution of SiC particles and microhardness of the surface composites.

1. Introduction

The study of magnesium and its alloys as one of the capable research topics for their load bearing capacity and good mechanical properties [1]. Surface composites are apposite materials for engineering applications bumping into surface contacts [2]. Friction stir processing (FSP) is one of the procedure for making surface composites and varying microstructural characteristics [3]. Many research have established that rigorous plastic deformation (SPD) is a useful method of producing ultrafine-grained materials [4-5]. Mishra et al. [6] effectively made Al matrix SiC reinforced surface composite using FSP. The majority of the accounted work has been on cramming the microstructural evolution, hardness, corrosion and wear properties [7–8].

In FSP process variables are divided into three categories of machine variables, tool design variables and material properties. Main machine variables are tool rotating rate and tool traverse speed. Tool rotational and transverse speeds decide the quantity of heat generated in the work piece [9]. Tool geometry mostly comprises shoulder diameter, shoulder feature, probe shape, probe size and probe feature. Stream of plasticized material in processed zone is influenced by tool geometry as well as traverse and rotational motion of the tool [10]. Tool geometry is an important aspect of FSP as it affects heat generation, material flow and ensuing microstructure [11]. Nano-sized grains can be accomplished by proper cooling arrangements. Cooling during FSP also provides the extra function of
plummeting tool wear [12]. Su et al. [13] examined the ensuing microstructure of friction stir processed 7075 Al using TEM. Mahoney et al. [14] proved that the FSP microstructures of NiAl Bronze alloy have considerably greater mechanical properties compared to the as-cast microstructure. Ma et al. [15] investigated the super plasticity in friction stir processed cast A356 at different temperature and strain rate.

There have been limited published studies concerned with the effect of FSP parameters on microstructure and micro hardness SiC reinforced Mg matrix composites. Experiments were conducted using Taguchi L9 orthogonal array. Microstructure of prepared composites were examined by SEM.

2. Experimental

2.1 Materials and Methods
The principal resources used for synthesis were Mg billet acquired from Micro Fine chemicals, India, and SiC particles from US Research Nanomaterials Inc, USA. Mg is used as the matrix material. The Mg matrix was reinforced with 5-15 wt% of SiC particles with the particle size varying from 100 µm to 150 µm.

2.2 Experimental design
The FSP are intended using a Taguchi method which involves sinking the deviation in a process through strong design of experiments. The experimental design proposed by Taguchi involves using orthogonal arrays to organize the parameters affecting the FSP and the levels at which they should be varied. From the literature and trials done by the authors in the field, the independently controllable predominant FSP parameters, which influences the performance of MMC is identified and presented Table 1. This experimentation has 4 variables at 3 diverse settings. Taguchi experiment with a L9 (34) orthogonal array was implemented to FSP of MMCs.

| Control parameters | Symbol | Level 1 | Level 2 | Level 3 |
|--------------------|--------|---------|---------|---------|
| Speed rpm          | S      | 600     | 700     | 800     |
| Transverse speed mm/min | f | 30      | 40      | 50      |
| Axial depth mm     | d      | 1.5     | 2       | 2.5     |
| wt % SiC           | w      | 5       | 10      | 15      |

2.3 Experimental work
A specifically intended tool with a straight cylindrical pin of 6-mm diameter, 5.7mm length, and a shoulder of 18-mm diameter was used. FSP was carried out using a tool which was mounting in a vertical CNC milling machine. Figure 1 shows the tool and the complete experimental setup. Before the beginning of the process, the samples were fastened firmly using ridged backing and holding plates. By using a drilling tool equipped with a CNC machine, a small hole with a diameter of that of the pin was drilled. The pin of the tool going ahead revolving and at the same time it was strained into the sample. The shoulder was then in get in touch with the surface of the specimen. Finally, the tool transmitted along the required length.

Nine FSP tests were conducted using different combinations as per Taguchi’s experimental design. Table 2 shows an example of effectively friction stir processed composites. Vickers micro-hardness was measured using a QV-1000DM digital micro-hardness tester with a load of 1,000 gram and dwell time of ten seconds. Samples were taken from the crossways sections at the middle of the pass. Then, the micro-hardness was measured at three different positions within the thickness of the composites. Three to five readings were taken and averaged for each position and presented in Table 2.
**Figure 1.** Experimental setup

**Table 2.** Experimental results

| Sample no | Speed rpm | Transverse feed | Axial depth mm | % SiC | Micro hardness VHN | Prepared FSP composites |
|-----------|-----------|-----------------|----------------|-------|--------------------|-------------------------|
| 1         | 600       | 30              | 1.5            | 5     | 65.2               |                         |
| 2         | 600       | 40              | 2              | 10    | 70                 |                         |
| 3         | 600       | 50              | 2.5            | 15    | 71.2               |                         |
| 4         | 700       | 30              | 2              | 15    | 66.2               |                         |
3. Results and Discussion

3.1 SEM analysis

The micro structural and surface morphology of Mg metal matrix and SiC particles reinforced composites were done using scanning electron microscope with EDX. EDX was used to evaluate the dispersion of SiC in magnesium matrix composites. Figure 2 shows the SEM micrograph of Mg metal matrix reinforced with SiC and attachment of silicon carbide particles can be established over the surface of Mg. In order to confirm the presence of SiC particle, EDX analysis was done over a particular region. EDX analysis of the 10% SiC reinforced Mg matrix composite is presented in Figure 3. It is clear that the peaks for magnesium, silicon and carbon were attained.

FSP is a novel procedure for fabrication of surface composites and it is beneficial in ease of dispersion and elimination of clusters of reinforcement particles. The uniform distribution of reinforcement particles in Mg matrix is shown in Figure 2. Dispersion and distribution of reinforcement particles in the surface composite can be successfully achieved after multi-pass FSP. Also, a consistent particle distribution is attained. This can afford outstanding combinations of strength and ductility in surface composites.
3.2 XRD analysis
Diffraction pattern was composed using BRUKER D8 FOCUS (9 kW) at approximately 2 min intervals and all XRD analysis were matched with the JCPDS database available in the operating software to determine all phases present. The XRD pattern of the Friction stir processed Mg-SiC composites are shown in Figure 4. As shown in figure the diffraction peaks at $2\theta = 33^\circ, 34^\circ, 65, 80^\circ$ can be attributed to the reflection of Mg (JCPDS card no 36-1451), $2\theta = 69^\circ, 72^\circ, 81^\circ$ correspond to those of SiC (JCPDS file 29-1129).
3.3 Optimization of FSP parameters

The analysis of variance was used to set up statistically considerable FSP parameters and the percent contribution of these parameters on the micro hardness. The intention of Taguchi’s quality loss function is quantitative assessment of quality loss due to practical variation. A quality characteristic is the objective of interest of a product or process. It is called as fundamental characteristic. The difference between the functional value and objective value is emphasized and identified as the loss function. The loss function is given in equation 1

\[ L(Y) = \frac{l(m)(y-m)^2}{2} = K(y - m)^2 = K(MSD) \]  

(1)

where, \(L(y)\) is loss function, \(y\) is value of the quantity characteristics, \(m\) is target value of \(Y\), \(K\) is proportionality constant and \(MSD\) is mean square deviation.

The category the-higher -the-better is always preferred to calculate the S/N ratio for quality characteristics of micro hardness. The equation for calculation S/N ratio for smaller the better characteristic is given in equation 3 (in decibels)

\[ S/N = -10\log (\Sigma (1/Y^2)/n) \]

(2)

Where \(Y\) = responses for the given factor level combination and \(n\) = number of responses in the factor level combination.

Despite of type of the performance characteristics, a greater S/N ratio value communicates to improved performance. Therefore, the optimal level of the machining parameters is the level with the greatest S/N ratio value. By applying the Eqs. 1 and 2, the S/N ratio values for each experiment of \(L_9\) (Table 3) were calculated. Figure 5 shows mean S/n ratio for micro hardness. Based on the analysis of S/N ratio, the optimal machining performance for the Micro hardness was obtained at 800 rpm speed (level 3), 50 mm/min transverse feed (level 3), 5 mm of axial depth (level 3) and 10% of SiC (level 2).

The comparative significance among the FSP parameters for the micro hardness was investigated by using the ANOVA method so that optimal combinations of the cutting parameter levels can be determined more accurately. The results of ANOVA are shown in Table 4. Larger sum of square indicates that the variation of the process parameter makes a big change on the performance characteristics. Percent contribution indicates the relative power of a factor to reduce variation. For a factor with a high percent contribution, a small variation will have a great influence on the micro hardness. It is clear that the most effect on the micro hardness is wt % of SiC followed by Speed and axial depth.

![Figure 4. XRD pattern of friction stir processed Mg matrix reinforced with 10% SiC](image)
Table 3. S/N ratio for experimental data

| Ex no | S/N ratio |
|-------|-----------|
| 1     | 36.28495  |
| 2     | 36.90196  |
| 3     | 37.0496   |
| 4     | 36.41716  |
| 5     | 36.33808  |
| 6     | 37.07396  |
| 7     | 38.0618   |
| 8     | 37.33756  |
| 9     | 36.82719  |

Figure 5. Mean S/N ratio for micro hardness

Table 4. ANOVA for S/N ratio

| Term            | DOF | Sum Square | Mean Square | % Contribution |
|-----------------|-----|------------|-------------|----------------|
| Speed           | 2   | 1.09705    | 0.55        | 43.49482776    |
| Transverse speed| 2   | 0.023207   | 0.012       | 0.920083474    |
| Axial depth     | 2   | 0.285328   | 0.14        | 11.31242496    |
| wt % of SiC     | 2   | 1.116669   | 0.56        | 44.2726638     |

3.4 Confirmation experiments

The estimated/predicted S/N ratio, using the optimal level of the FSP parameters, can be calculated from following equation

\[ \eta_{predicted} = \eta_m + (\eta_0 - \eta_m) \]

where \( \eta_m \) is the total mean of the S/N ratio, \( \eta_0 \) is the mean S/N ratio at optimal level and \( N \) is the number of main design parameters that affect the performance characteristics. Table 5 shows the comparisons of predicted and actual FSP performance for micro hardness using their optimal cutting
parameters. The analysis of result (mean, from Fig. 3) indicates that the optimal solution is obtained at $S_3f_3d_3w_2$ (Micro hardness=37.01VHN).

**Table 5** Comparison between FSP performance using the initial and optimal level

|                  | Initial parameter | Optimal FSP parameter |
|------------------|-------------------|-----------------------|
| Setting level    | $S_1d_1w_1$       | $S_3f_3d_3w_2$        |
| Micro hardness VHN | 65.2              | 71.2                  |
| S/n ratio        | 36.28             | 37.01                 | 37.25 |

4. Conclusions

The results from the experimental investigation of FSP of SiC reinforced Mg matrix surface composites can be summarized as follows

- FSP of magnesium-SiC composites was accomplished using different combinations of tool rotational, transverse feed, axial depth and wt% SiC.
- Taguchi model was used to optimize FSP parameters by considering micro-hardness as performance indicator.
- An optimum parameter combination for the MAXIMUM micro hardness is obtained by using the analysis of S/N ratio. Based on the analysis of S/N ratio, the optimal FSP performance for the micro hardness is obtained between 800 rpm speed (level 3), 50 mm/min transverse feed (level 3), 5 mm of axial depth (level 3) and 10% of SiC (level 2).
- Maximum Micro hardness was obtained at ANOVA analysis was carried out to find importance of FSP parameters. wt % of SiC followed by Speed and axial depth on micro hardness.

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