Friction stir processing of ZE41 Mg alloy: Optimizing the process parameters

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Abstract. Friction stir processing is a novel processing route used to develop fine grained structures. Processing by FSP is influenced by tool rotational speed, tool travel speed, workpiece temperature, tool tilt angle, etc. Processing at optimum parameters is important to avoid defects and to develop grain refined materials by FSP. In the current research work, ZE41 magnesium alloy was processed by FSP to develop fine grained structure. FSP was done at different tool rotational and travel speeds. Decreased grain size and increased hardness was observed for all FSPed samples. However, variations were clearly observed within the measured grain size, hardness and nugget zone width of the FSPed samples. From Taguchi optimization studies using gray relation analysis; optimum set of parameters which yield smaller grain size and higher hardness were identified. From the results, 1400 rpm and 25 mm/min were observed as optimum. The design model presented in the current study helps to select optimized parameters in producing fine grained ZE41 for upcoming energy efficient applications.

Keywords: Magnesium alloys, FSP, optimization, fine grains, optimization.

1. Introduction

Developing fine grained structures at the surface of metals by friction stir processing (FSP) is novel in the surface engineering [1]. FSP completely eliminates the complex problems elevated during materials processing which involve melting and solidification. However, there are several process parameters which influence the success of FSP which include tool tilt angle, rotational speed and tool travel speed, etc. [2]. Optimizing these parameters is crucial to achieve higher level of grain refinement without defects in the stir zone. There are several optimization techniques available to select the most appropriate process parameters which give better outcome. Gray relation analysis is such as simple optimization technique which helps to identify the optimized process parameters to achieve highest grain refinement [3].
Due to their excellent properties such as low weight and high strength, damping properties and good machinability; Magnesium (Mg) based materials gained popularity in the materials Engineering [4, 5]. Developing grain refined Mg alloys helps to produce structures made of Mg alloys with better properties as the grain refined materials exhibit better properties. Producing fine grained Mg based alloys by liquid state processing methods is complex due to the nature of Mg. On the other hand, processing Mg within the solid state is difficult due to the low formability at the ambient conditions. In this context, FSP can be a viable solution to process Mg alloys to develop grain refined structures. Several other reports demonstrated using FSP to develop grain refined Mg alloys [6, 7]. Among the available Mg alloys, ZE41 alloy that has zinc (Zn), rare earths (RE) and zirconium (Zr) as the main alloying elements is used in automobile and aerospace applications [8, 9, 10]. Developing grain refined ZE41 Mg alloy helps to achieve improved performance in the structures produced by ZE41 Mg alloy. Hence, in the present research work, FSP of ZE41 Mg alloy was done at three different tool rotational and tool travel speeds and optimized process parameters were obtained to achieve smaller grains without defect in the stir zone.

2. Materials and methods

ZE41 Mg alloy billet was commercially procured (Exclusive Magnesium, India) and workpieces of size 100 X 50 X 5 mm³ were machined form the billet. The ZE41 Mg alloy contains 3.9 % Zn, 0.56% Zr, 1.1 % RE, 0.003% Fe and balance being Mg (by wt.%). FSP was done on a vertical milling machine. The experiments were carried out by using a tool manufactured with H13 tool steel. 1120, 1400 and 1800 rpm rotational speeds and 16, 25 and 50 mm/min tool travel speeds were adopted. Since the spindle of the milling machine does not have tilt facility, tilt angle during FSP was considered as zero. FSP illustration and photograph of the process are shown in Fig 1. In the present research work, the process parameters were selected to form an L₉ full factorial orthogonal array (3²). Table 1 lists the process parameters adopted to complete the experiments. Taguchi model using orthogonal array was adopted and from the grey relation analysis optimum set of parameters which yield smaller grain size and higher hardness were identified. In grey relation analysis; grey relation coefficients are calculated by measuring performance characteristics and normalizing them to a value between zero to one. The grey relation grades are obtained from the coefficients and the overall performance response is obtained. Details on carrying grey relation analysis can be referred elsewhere [3].

![Fig 1a) Schematic diagram of FSP and b) photograph showing surface of FSPed ZE41 Mg alloy](image)

For microstructural studies, the samples were metallographically polished by following a standard procedure which includes first polishing by using coarse grained sheets followed by fine grained sheets. Finishing of polishing is done by using alumina paste with the help of disc polishing machines.
The cleaned samples were then dried. Chemical etching (picric acid reagent) was done on the polished surfaces to reveal the microstructure. Microstructures were recorded by using optical microscope (Leica, Germany). Stir zone width was also measured and analyzed from the cross section of the samples. By conducting Vicker’s indentation tests, hardness was measured by applying 100 g load was applied on the surface by a diamond indenter for 10 s dwell time. Measurements were done across the FSPed region of all the samples. From the grey relation analysis, optimum process parameters to obtain higher hardness, higher stir zone width and smaller grains size were obtained.

Table 1. FSP process parameters (tool rotational and travel speeds) selected in the current research work.

| Factors      | Rotational speed (rpm) | Travel speed (mm/min) |
|--------------|------------------------|-----------------------|
| Level 1      | 1120                   | 16                    |
| Level 2      | 1400                   | 25                    |
| Level 3      | 1800                   | 50                    |

3. Results and discussion

Friction stir processing results in grain refinement in metals. During FSP, the heat that is generated in the stir zone helps to plastically deform the material and causes dynamic recrystallization (DRX) and produce fine grains [2]. The stirring action of the pin during FSP helps to deform the material and the heat that is generated during FSP dissolves the intermetallics into the solid solution grains. Therefore, several alloy which exhibit intermetallics at the grain boundary demonstrate decreased amount of intermetallics. In ZE41 Mg alloy, base microstructure reveals solid solution grains and intermetallics of Mg-Zn at the grain boundaries (Fig 2). The average grain size was measured as 107 ± 6.7 µm for base alloy Fig 2 (a). After FSP, a clear observation was made that confirms the grain refinement in all the samples. Typical microstructure of stir zone of FSPed alloy (1400 rpm & 25 mm/min) is shown in Fig 2 (b) which is evident that FSP leads to grain refinement.

Fig 2 Microstructure of the samples: a) base alloy and b) FSPed alloy (1400 rpm & 25 mm/min).

The normalized values for grain size, width of the stir zone and hardness were obtained for the grey relation analysis as demonstrate elsewhere [11]. From the values, it is noted that for grain size, lower is better characteristic is considered. For hardness and stir zone width, higher is better characteristic is considered. Table 2 lists the average grain size, width of the stir zone width and average microhardness values of all the samples obtained at different FSP process parameters. Table 3 shows the corresponding grey relation normalized values, Table 4 lists grey relation coefficients and Table 5 shows the grey relation grades and their relative ranks.
Table 2: Average grain size, width of the stir zone and microhardness of the samples processed at different process parameters.

| S. No. | Speed (rpm) | Feed Rate (mm/min) | Grain Size (µm) | Width (µm) | Micro-hardness (Hv) |
|--------|-------------|-------------------|-----------------|------------|---------------------|
| 1      | 1120        | 16                | 4.122           | 2357.599   | 96.26               |
| 2      | 1120        | 25                | 2.8             | 2140.067   | 90.4                |
| 3      | 1120        | 50                | 2.727           | 2643.196   | 89.3                |
| 4      | 1400        | 16                | 4.337           | 2519.256   | 112.5               |
| 5      | 1400        | 25                | 2.633           | 2316.477   | 128.2               |
| 6      | 1400        | 50                | 3.351           | 2175.144   | 121.7               |
| 7      | 1800        | 16                | 4.474           | 2054.16    | 97.3                |
| 8      | 1800        | 25                | 2.683           | 2272.132   | 95.8                |
| 9      | 1800        | 50                | 3.271           | 2006.216   | 102.1               |

Table 3 Grey relation normalized values

| S.No. | Speed (rpm) | Feed Rate (mm/min) | Grain Size (µm) | Width (µm) | Micro-Hardness (Hv) |
|-------|-------------|-------------------|-----------------|------------|---------------------|
| 1     | 1120        | 16                | 0.1912          | 0.5516     | 0.1789              |
| 2     | 1120        | 25                | 0.9092          | 0.2101     | 0.0282              |
| 3     | 1120        | 50                | 0.9489          | 1          | 0                   |
| 4     | 1400        | 16                | 0.0744          | 0.8054     | 0.5964              |
| 5     | 1400        | 25                | 1               | 0.4870     | 1                   |
| 6     | 1400        | 50                | 0.6099          | 0.2652     | 0.8329              |
| 7     | 1800        | 16                | 0               | 0.0752     | 0.2056              |
| 8     | 1800        | 25                | 0.9728          | 0.4174     | 0.1670              |
| 9     | 1800        | 50                | 0.6534          | 0          | 0.3290              |

Table 4 Grey relation coefficients

| S.No. | Speed (rpm) | Feed Rate (mm/min) | Grain Size (µm) | Width (µm) | Micro-Hardness (Hv) |
|-------|-------------|-------------------|-----------------|------------|---------------------|
| 1     | 1120        | 16                | 0.3820          | 0.5272     | 0.3784              |
| 2     | 1120        | 25                | 0.8464          | 0.3876     | 0.3397              |
| 3     | 1120        | 50                | 0.9073          | 1          | 0.3333              |
| 4     | 1400        | 16                | 0.3507          | 0.7198     | 0.5533              |
| 5     | 1400        | 25                | 1               | 0.4936     | 1                   |
| 6     | 1400        | 50                | 0.5617          | 0.4049     | 0.7495              |
| 7     | 1800        | 16                | 0.3333          | 0.3509     | 0.3862              |
| 8     | 1800        | 25                | 0.9484          | 0.4618     | 0.3751              |
| 9     | 1800        | 50                | 0.5906          | 0.3333     | 0.4270              |

Table 5 Grey relation grades (GRG) and their rankings
| S. No. | Speed (rpm) | Feed Rate (mm/min) | GRG     | Rank |
|--------|-------------|--------------------|---------|------|
| 1      | 1120        | 16                 | 0.42924 | 8    |
| 2      | 1120        | 25                 | 0.5246  | 6    |
| 3      | 1120        | 50                 | 0.74689 | 2    |
| 4      | 1400        | 16                 | 0.54131 | 5    |
| 5      | 1400        | 25                 | 0.83121 | 1    |
| 6      | 1400        | 50                 | 0.57208 | 4    |
| 7      | 1800        | 16                 | 0.35686 | 9    |
| 8      | 1800        | 25                 | 0.59516 | 3    |
| 9      | 1800        | 50                 | 0.45032 | 7    |

Fig 3 Main effects plot for SN ratios: Mean of SN ratios.

Fig 4 Main effects plot for means: Mean of means.

Fig 3 and Fig 4 present the SN plots. From the GRG rankings, it can be understood that 1400 rpm & 25 mm/min parameters as the optimum set of process parameters to achieve smaller grain size. In
terms of larger stir zone width, several set of process parameters were identified as prominent form the measured values as listed in Table 2. However, for higher hardness values, 1400 rpm & 25 mm/min were observed as optimum as also studied from GRG rankings. Other combination of process parameters (1120 rpm with 50 mm/min and 1800 rpm with 50 mm/min) were also observed as close to the optimized parameters (1400 rpm & 25 mm/min) to give relatively smaller grain size and higher hardness. This was also confirmed form the GRG rankings as listed in Table 5.

Achieving defect free stir zone and better grain refinement are the prime objectives behind optimizing the process parameters in FSP. Even though the FSP is a most convenient processin route to develop surface grain refined regions on metallic plates and sheets, it is very essential to optimize the process parameters for every new tool design and material systems. This essentially demands more optimization studies to know the best suited parameters to process. In addition to grain refinement, obtaining higher stir zone widths also helps to reduce the number of FSP passes carried out side by side to produce larger FSPed surface area. The grain refined region exhibits improved hardness compared with the base alloy. Therefore, the current analysis helps to understand the effect of tool rotational speeds, travel speeds on achieving grain refined ZE41 Mg alloy with better stir zone width and higher hardness.

4. Conclusions

In the present research work, FSP was done to produce grain refined surfaces on ZE41 alloy at different process parameters. Taguchi optimization analysis was used and the gray relation analysis was carried out to optimize the parameters. Defect free stir zones with smaller grain size and higher hardness with wider stir zone width was obtained at different process parameters. From the analysis, 1400 rpm & 25 mm/min parameters were found to be optimum. Hence, the present study helps to identify the best process parameters to develop fine grained ZE41 alloy by FSP.

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