**Article**

Multi-Performance Characteristics of AA5052 + 10% SiC Surface Composite by Friction Stir Processing

Rungwasun Kraiklang *, Jariyaporn Onwong and Charuayporn Santhaweesuk

Department of Industrial Engineering, Faculty of Engineering, Ubon Ratchathani University, Ubon Ratchathani 34190, Thailand; jariyaporn.s@ubu.ac.th (J.O.); charuayporn.s@ubu.ac.th (C.S.)

* Correspondence: rungwasun.kr.57@ubu.ac.th; Tel.: +66-045-353307

Received: 2 March 2020; Accepted: 7 April 2020; Published: 8 April 2020

**Abstract:** In this paper, optimization of the fabrication parameters of an aluminum surface composite with respect to tensile strength and tool wear rate is reported. The surface layer was reinforced with SiC particles to improve the tribological properties of AA-5052. The Taguchi design with orthogonal array L₈ was used for the experimental design, which included three processing parameters: the number of passes, rotational speed, and traversal speed. The experiment used optimal fabrication parameter searching to produce a multi-response prediction of both the tensile strength and tool wear rate. The experimental result was determined by grey relational analysis for multi-performance characteristics. Afterward, the prediction result of the optimal fabrication parameters was confirmed by repeated experiments to confirm the selection of optimal process parameters. The results revealed that the optimal fabrication parameters for multi-performance characteristics are two passes, rotational speed of 1000 revolutions per minute (RPM), and traversal speed of 30 mm/min (condition N1R1T2). These showed high tensile strength (229.90 MPa), low tool wear rate (0.0851), and a uniform distribution of SiC particles in the matrix. In addition, grey relational analysis showed that the parameter priority was 51.68% for rotational speed (the most significant process parameter), 36.18% for transversal speed, and 7.05% for the number of passes. Therefore, the grey-based orthogonal array Taguchi method can optimize multi-performance characteristics through the setting of process parameters for friction stir processing of an aluminum surface composite.

**Keywords:** aluminum surface composite; friction stir processing; grey relational analysis; Taguchi method

---

1. Introduction

Aluminum surface composites (ASC) have a large variety of application in the transportation, food, and aerospace industries due to their characteristics of being lightweight and having good wear resistance, hardness, and good performance at high temperatures. These properties are good mechanical and physical properties for ASC, which are formed by the reinforcement of hard ceramic particles in an aluminum matrix layer. Both the mechanical and physical properties of ASC are very important for their continued usability [1–4].

There are several techniques for composite surface fabrication including high energy laser treatment, plasma sprayed coating, cast sintering, and casting, which are techniques for improving the properties of materials. These techniques can improve the property of many materials such as composite fiber fabrication by dissolving the coating process for producing the conductive polymer composite material. The composite fiber shows good mechanical properties and high electrical conductivity [5,6]. The tool steel surface coating with the high current pulsed electron beam technique displays dense structure and corrosion resistance improvement [7]. Meanwhile, the property
improvement of aluminum by using the casting process shows the high mechanical property of composite aluminum, but the appearance of structural porosity defection [8]. However, fabrication of ASC by the casting process is liquid-state fabrication at temperatures higher than the melting point of the base material. The cooling temperature of the casting process is a difficult-to-control parameter that affects the quality of the structure in the surface matrix. Defects appear in the majority of composite surface structures, these being voids, cavities, and the formation of unstable phases. In addition, the internal structure engenders surface interfacial reactions between reinforcement particles in the aluminum matrix [9,10].

Friction stir processing (FSP) has been used to solve the problems in fabricating ASC. FSP is a solid-state processing technique for aluminum composite surface fabrication. The basic concept of FSP is a rotating tool for the creation of friction with a shoulder and pin penetrating the work piece in a downward motion until the shoulder makes contacts with the surface. The friction between the FSP tool and the substrate material generates heat and the soft substrate material of the contractive surface. The softening of the substrate material is called plastic deformational softening. Then, the FSP tool initiates a forward movement in the processing direction. The FSP tool’s role is stirring the plastic deformation by the pin and shoulder, by which the soft material and movement direction of particles changes from the front to back within the shoulder and turnaround of the pin [1,11]. The stirring of the internal material between the plastic deformation and reinforced particles causes mixing and grain refinement in the structure.

The three most important FSP process parameters are material properties, tool design parameters, and machine parameters. The material properties are more important than the tool design parameters and machine parameters. The mechanical properties of the base material play an important role in the process of parameter selection as it undergoes plastic deformation during fabrication. The tool design parameters are the tool geometry design and dimensions such as shoulder diameter, shoulder feature, pin feature, and pin size. The tool geometry influences heat generation and material flow in the processed zone. Thus, tool geometry directly controls the properties and the quality of the composite surface. The machine parameters consist of the rotation speed, traveling speed, tilt angle, and plunge depth. Both the rotational and travelling speed are important parameters in controlling the amount of heat generation at the workpiece. The tilt angle and plunge depth affect the material flow and formation of microstructural restructuring in the stir zone [12].

In the FSP fabrication of surface composites, the distribution of reinforced particles mainly depends on the number of process passes. Multiple passes lead to a decrease in the particle size of clusters and grain size in the matrix, which significantly influences the mechanical properties of the composite material [9]. Additionally, several studies have reported that increasing the rotational speed and the number of passes leads to an ultimate increase in tensile strength. On the other hand, increasing the travelling speed leads to a decrease in the ultimate tensile strength [13–17]. However, an important consideration in surface composite fabrication, aside from the process parameters, is tool wear, which is an undesirable feature and obstacle for the quality of the workpiece. Tool wear in FSP can potentially increase defects and degradation in the product’s quality. Tool wear is directly proportional to tool rotational speed and traversal distance [18–21]. The process parameters can help decrease tool wear and reduce the interaction between the workpiece and the moving contact area of the tool. ASC are reinforced with ceramic particles, producing a very hard reinforced material. Therefore, the tool material for ASC fabrication was heated to harden it before use. In addition, the combination of high temperatures and flow stresses in the FSP of hard ceramic particles causes significant wear of the tool [22]. The hard reinforcing particles erode the probe features and probe length, which inhibits the material flow and increases the likelihood of defect formation. Understanding the material flow control in FSP is important to decreasing the number of defects and for good mechanical properties. Moreover, a reduction in tool wear for extended tool life is necessary for good-quality composite surface formation. Therefore, the highest ultimate tensile strength and lowest tool wear in FSP represent the optimal level of process parameters [9,23].
Normally, the quality of products is directly influenced by the input parameters during the fabrication processes. Various factors of fabrication influencing on the composite quality have to be considered to achieve the required specifications. For each factor, input parameters are considered and level adjusting takes place until the optimal parameter levels are found. This method has a high cost due to the cost of materials and time consumption for trial and error. Various design of experiment (DOE) techniques and optimization methods have been used for optimal parameter and relational specification between the input parameters and the desired outputs. In particular, the Taguchi method is one of the most powerful optimization techniques. Several DOE methods such as full factorial and response surface have more effectiveness over the Taguchi method. However, these methods are complex and difficult in practice as the number of the experiments will be increased due to the number of factors, while the Taguchi method requires a minimum number of experiments, which results in a reduction in terms of time and budget, but also provides robust design solutions. The Taguchi technique calculates the signal-to-noise (SN) ratio for each parameter combination. The maximum SN ratio combination is defined as the optimal setting of a Taguchi experiment. However, the optimal solution by the Taguchi technique is to find one performance characteristic; its purpose is not to cover varied performance characteristics. Therefore, the multi-objective optimization condition finding is calculated based on the grey relational analysis theory [24]. Grey relational analysis (GRA) is used for complicated interrelationship problem solving of multiple objective optimization. The GRA is a data transformation of normalized experimental data, which becomes the grey relational coefficient and grey relational grade. The grey relation grade performance index employs an evaluation of the weighted value response of various performance characteristics. Therefore, their comparative significance can be quantitatively described with the optimal combination of process parameters. Even if the use of the Taguchi method in collaboration with grey relational analysis in identifying multi-objective optimization has a limitation in terms of the accuracy of the prediction, it provides advantages in terms of a reduction in the sample size, time, and budget. Moreover, this combination of methods is suitable for practical applications in the general manufacturing industry, which has been successful in optimizing the process in many prior studies such as Ramu et al. [25], Wojciechowski et al. [26], and Reddy and Jawahar [27].

The challenge in the fabrication of ASC using FSP is achieving the maximum ultimate tensile strength and minimum tool wear simultaneously. Therefore, this research aims to achieve the optimal parameters of the composite surface fabrication in FSP. The Taguchi Ls orthogonal array was applied to the experimental planning of FSP parameters. The parameters of study were the number of passes, rotational speed, and traverse speed. The two responses were ultimate tensile strength and tool wear rate. The experimental result of the Taguchi method was transformed by the grey relation analysis method, which is used for the prediction of the optimum combination of the friction stir process parameter for both the maximum ultimate tensile strength and minimum tool wear rate during the AA5052 + 10% SiCp surface composite process fabrication.

2. Experimental Procedure

2.1. Preparation of Tool and Workpiece

In this study, the FSP tool was machined by a CNC lathe for tool geometry fabrication. The tool material was DC53 cold working tool steel that was hardened to 62-64 HRC. The physical properties of the tool material are shown in Table 1. The FSP tool was a cylindrically threaded pin profile with 6 mm diameter, 1 mm pitch distance, 4.5 mm pin height, and 18 mm shoulder diameter, as shown in Figure 1a.

The experimental materials used were AA5052-O aluminum plates with a 6 mm thickness. The chemical components and mechanical properties are shown in Table 2. The workpiece of the surface composite plate was prepared by drilling holes with a 2 mm diameter, 4.5 mm depth, and a distance of 4.19 mm. The drilled hole had a rolling direction on a medium plate with dimensions of 200 mm × 100 mm × 6 mm, as shown in Figure 2. Afterward, SiC particles with an average diameter of 18 μm were used as reinforcements on the workpiece surface. The SiC particles were filled with the drilled
hole and pressed tightly before the FSP. The drilled hole on the workpiece was closed by using a stirring tool without a pin for protective scattering of the particles (Figure 1b).

Table 1. The physical properties of DC53 cold working tool steel.

| Specific Gravity (g/cm³) | Young's Modulus (kgf/mm²) | Modulus of Rigidity (kgf/mm²) | Poisson's Ratio | Heat treated Hardness (HRC) |
|-------------------------|---------------------------|-------------------------------|----------------|-----------------------------|
| 7.87                    | 21,700                    | 8480                          | 0.28           | 62–64                       |

Table 2. Chemical and mechanical properties of the as received AA5052-O.

| Chemical Composition w/% | Si  | Fe  | Cu  | Mn  | Mg  | Cr  | Zn  |
|--------------------------|-----|-----|-----|-----|-----|-----|-----|
|                          | 0.08| 0.23| 0.002| 0.005| 2.24| 0.20| 0.007|

| Mechanical Properties    | Yield Strength (MPa) | Tensile Strength (MPa) | Elongation (%) | Brinell Hardness (HB) |
|--------------------------|----------------------|------------------------|----------------|----------------------|
|                          | 148.36               | 201.10                 | 23.51          | 57.07                |

Figure 1. Friction stir processing (FSP) tool: (a) stirring tool, and (b) pinless tool.

Figure 2. A schematic diagram of preparing AA5052 composites by FSP.

2.2. Taguchi Experimental Design Methods

The experimental design was based on the Taguchi method, as the Taguchi orthogonal array would markedly reduce the number of experiments required. In this investigation, FSP parameters included rotational speed, traverse speed, and number of passes. The rotational direction of each pass was altered in the opposite direction. The number of process parameters and each response level are shown in Table 3.
Table 3. Process parameters and levels.

| Parameters                  | Code | Levels  |               |       |
|-----------------------------|------|---------|---------------|-------|
| Number of pass (passes)     | N    | 1       | 2             | 3     | 4     |
| Rotational speed (RPM)      | R    | 1000    | 1500          |       |
| Traverse speed (mm/min)     | T    | 10      | 30            |       |

According to Taguchi’s design of experiments, for three parameters, L₈(4^1*2^2) was selected with the number of replications of two for each condition. All eight conditions in this study generated using Minitab 17. The six parameters were fixed in this study including a threaded cylindrical pin profiled tool (Figure 1a), shoulder diameter to pin diameter (D/d) ratio (3.0), tilted angle (3°), shoulder plunged depth (0.3 mm), dwell time (30 seconds), traverse distance (150 mm), and stirring area (6 mm × 150 mm). The fixed parameter references are shown in Table 4. All eight conditions are shown in Table 5. Finally, a CNC milling machine (HAAS, TM2, Haas Automation, Inc, Oxnard, CA, USA) was used to conduct the FSP experiments in this research (see Figure 3).

Table 4. Fix parameters and references.

| Parameters                                      | Values               | References |
|------------------------------------------------|----------------------|------------|
| Tool shape                                      | cylindrical threaded pin profile | [28,29]    |
| Shoulder diameter to pin diameter ratio         | 3:1                  | [30]       |
| Tilted angle                                    | 3°                   | [31,32]    |
| Shoulder plunged depth                          | 0.3 millimeters      | [33]       |
| Dwell time                                      | 30 seconds           | [30]       |
| Traverse distance per time                      | 150 millimeters      | [20]       |

2.3. Measurement Method and Test Results

The FSP performance was evaluated based on the response variables, namely, tensile strength and tool wear rate. The tensile specimens were prepared by a waterjet cutting machine with an ASTM E8M-04 standard (Figure 3) for each experiment. The tensile tests were tested by a LLOYD LS100-Plus (AMETEK Sensors, Test & Calibration, Largo, FL, USA) universal testing machine at room temperature and at a crosshead speed of 0.5 mm/min.

![Figure 3](image-url) Layout of the tensile specimens and the microstructure specimens.

After friction stir processing, the aluminum accumulated on the tool pin. This aluminum was eradicated post-process by immersing the tool in NaOH solution and water [34]. Tool pin wear was measured by mass loss techniques. The tool wear of each experiment was compared using the original tool pin and comparing it with the mass loss of the tool after wear [35,36]. The percentage tool wear calculation was carried out using Equation (1):
\[ \% \text{Tool wear} = \frac{W_e - W_o}{W_o} \times 100\% \]

where \( W_e \) denotes the initial mass of the pin and \( W_o \) is the change in mass of the pin.

2.4. Grey Relational Analysis of the Experimental Data

The grey relational analysis started with all of the experimental data, which was normalized to obtain the grey relational generation range from zero to one. In this study, the normalized results, \( x^*_i(k) \), for the larger-the-better characteristics (e.g., tensile strength) were calculated using Equation (2):

\[ x^*_i(k) = \frac{x_i^{(0)}(k) \cdot \min_{all(i)} x_i^{(0)}(k)}{\max_{all(i)} x_i^{(0)}(k) - \min_{all(i)} x_i^{(0)}(k)} \]  

The tool wear rate, which is a smaller-the-better characteristic, was calculated using Equation (3):

\[ x^*_i(k) = \frac{\max_{all(i)} x_i^{(0)}(k) - x_i^{(0)}(k)}{\max_{all(i)} x_i^{(0)}(k) - \min_{all(i)} x_i^{(0)}(k)} \]

where \( x^*_i(k) \) is the sequence after the data preprocessing; \( x_i^{(0)}(k) \) is the reference sequence; \( \min_{all(i)} x_i^{(0)}(k) \) is the smallest value in the reference sequence; \( \max_{all(i)} x_i^{(0)}(k) \) is the largest value in the reference sequence; the reference sequence is \( i = 1, 2, \ldots, m \), and \( k = 1, 2, \ldots, n \); \( m \) is the number of experiments; and \( n \) is the amount of experimental data [37].

Then, the grey relational coefficient \( (\xi(k)) \) was calculated to identify the relationship between the ideal and actual experimental results using Equation (4):

\[ \xi(k) = \frac{\Delta_{\min} + \xi \Delta_{\max}}{\Delta_{\max} - \Delta_{\min}} \]  

where \( \Delta_{\min} = |x^*_i(k) - x^*_{j_i}(k)| \) is the deviation sequence of the reference sequence \( x^*_i(k) \) and the comparability sequence \( x^*_{j_i}(k) \); \( \Delta_{\min} = \min_{j_i} |x^*_i(k) - x^*_{j_i}(k)| \) is the lowest value of \( \Delta_{\min} \); \( \Delta_{\max} = \max_{j_i} |x^*_i(k) - x^*_{j_i}(k)| \) is the largest value of \( \Delta_{\min} \); and \( \xi \) is the distinguishing coefficient [37].

The grey relational grades symbolize the overall performance characteristic of efficiency fabrication of the aluminum surface composite. Consequently, the multi-objective optimization problem was distorted into a single equivalent objective optimization problem using the grey relational grades. Based on the fabrication efficiency of the aluminum surface composite using friction stir processing, this shows significant promise, as tool wear is a more important property than tensile strength. In this study, the distinguishing coefficient for the tensile strength and the tool wear were assumed to be 0.30 and 0.70, respectively. After obtaining the grey relational coefficient, the grey relational grade was computed by averaging the grey relational coefficient corresponding to each performance characteristic. The grey relational grade was calculated using Equation (5):

\[ \gamma_i = \frac{1}{n} \sum_{k=1}^{n} \xi(k) \]  

where \( \gamma_i \) is the grey relational grade for the \( i^{th} \) experiment and \( n \) is the number of performance characteristics [37]. The grey relational grades were tabulated for overall performance characteristics (tensile strength and tool wear). After that, all values of the grey relational grade were calculated as the grey relational grade average value to find the optimal level of each parameter. Optimal level selection was determined as the highest grey relational grade for each parameter, with the highest grey relational grade implying the optimal level for the parameter. However, if the highest level grey relational grade exhibition was not shown in the Taguchi design L8 orthogonal array, then the
optimal level parameter did not appear. Then, the optimal level parameter was found by the grey relation grade estimation method following Equation (6). The grey relation grade estimated value was compared with the grey relation grade value of the best on the L8 orthogonal array experiment. If the grey relation grade estimated value was higher than the grey relation grade value of the best on the L8 orthogonal array experiment, then that was the optimum condition.

\[
\hat{\gamma} = \gamma_m + \sum_{i=1}^{q} (\bar{\gamma}_i - \gamma_m)
\]

(6)

where \(\hat{\gamma}\) is grey relation grade estimation; \(\gamma_m\) is the total mean of the grey relational grade; \(\bar{\gamma}_i\) is the mean of the grey relational grade at the optimal level; and \(q\) is the number of the process parameters that significantly affects the multiple performance characteristics [37].

After that, optimal process parameters were confirmed by repeated experiments, for which the repeated experiment result was compared with the predictable response for statistical error checking. Statistical analysis with analysis of variance (ANOVA) was used for error analysis of the response between the experimental result and predicted response and to check the importance of each parameter. The microstructure of a cross-section of the specimen was analyzed using scanning electron microscopy (SEM) for support of the optimal condition.

3. Results and Discussion

3.1. Tensile Strength and Tool Wear

The obtained values of tensile strength and tool wear corresponding to each experiment are shown in Table 5 and Figure 4. Thereafter, the effects of parameters were plotted as shown in Figure 5.

Table 5. Experimental design using the L8 orthogonal array with tensile strength and percentage tool wear values for experiments.

| Condition | A (passes) | B (RPM) | C (mm/min) | Tensile Strength (MPa) | Tool Wear Rate (%) |
|-----------|------------|---------|------------|------------------------|--------------------|
|           |            |         |            | \(\bar{x}\)         | S.D.               | \(\bar{x}\)      | S.D.               |
| N1R1T1    | 1          | 1000    | 10         | 187.92                | 2.5314             | 0.0695            | 0.0088             |
| N1R2T1    | 1          | 1500    | 30         | 154.59                | 10.2389            | 0.0561            | 0.0023             |
| N2R1T1    | 2          | 1000    | 10         | 233.32                | 8.1600             | 0.0926            | 0.0073             |
| N2R2T1    | 2          | 1500    | 30         | 225.86                | 1.4354             | 0.0911            | 0.0053             |
| N3R1T1    | 3          | 1000    | 30         | 233.24                | 3.8325             | 0.0926            | 0.0045             |
| N3R1T1    | 3          | 1500    | 10         | 211.16                | 6.0387             | 0.1013            | 0.0044             |
| N4R1T1    | 4          | 1000    | 30         | 234.73                | 5.6993             | 0.1085            | 0.0064             |
| N4R1T1    | 4          | 1500    | 10         | 218.07                | 6.0245             | 0.1209            | 0.0090             |
Figure 4. Tensile strength and percentage tool wear values for the experiments.

Figure 5. Effect of parameters on (a) tensile strength, and (b) percentage tool wear rate.

As shown in Figure 5, the results showed an increase in tensile strength and tool wear with an increasing number of passes. The number of passes is possibly a more important parameter than tensile strength and tool wear rate in FSP. The condition of N4R1T2 exhibited a higher tensile strength than the other experiments, with a tensile strength of 234.73 MPa (Figure 4). Due to the high number of passes and high traverse speed, there was a high level of dislocation to the grain. On the other hand, this condition showed high tool wear rate at 0.1085% of pin mass, which affected the microstructure and uniformity of SiC particles on the surface. In fact, FSP requires high strength, but low tool wear rate and a uniformity of SiC particles in the microstructure; therefore, the N4R1T2 is not exactly optimal. For this reason, the experimental results in Table 5 were analyzed by the grey relational method to find the optimal parameters for the optimal performance characteristic of high tensile strength and low tool wear rate.

The results of the experiment were converted into normalized data by the grey relational method following Equations (2) and (3). The normalized data of both tensile strength and tool wear rate are exhibited in Table 6.

Table 6. Normalized data $x(k)$ of each performance characteristic.

| Condition   | Normalized data, $x(k)$ |
|-------------|-------------------------|
|             | Tensile Strength | Tool Wear Rate |
| Ideal sequence | 1.0000          | 1.0000         |
| N4R1T2      | 0.4159           | 0.7931         |
Afterward, the normalized data were translated to the relational coefficient and grey relational grade by using Equations (4) and (5) for ranking, before finding the optimal combination of process parameters. The results are shown in Table 7.

| Condition | Grey Relational Coefficient | Grey Relational Grade | Order |
|-----------|------------------------------|-----------------------|-------|
| NiR:T1    | Tensile Strength: 0.3393    | Tool Wear: 0.7719     | 6     |
| NiR:T2    | 0.2308                      | 1.0000                | 5     |
| NiR:T1    | 0.7465                      | 0.5545                | 2     |
| NiR:T2    | 0.7305                      | 0.5644                | 3     |
| NiR:T1    | 0.5050                      | 0.5010                | 4     |
| NiR:T2    | 1.0000                      | 0.4642                | 5     |
| NiR:T1    | 0.5907                      | 0.4118                | 6     |

From the grey relational grade, it was found that the condition of NiR:T1 (two passes, rotational speed of 1000 RPM and traverse speed of 10 mm/min) was the best condition among eight experimental conditions for the multi-response of high tensile strength and low tool wear rate. However, it may not always be the optimal condition, as the Taguchi L8 experimental design had a lower experimental number. Therefore, the grey relational grade of the eight conditions was used to find the optimal multi-response conditions for each parameter.

The mean of the grey relational grade of the eight conditions was calculated for each parameter, and the selection of the optimal level from the calculation result is exhibited in Table 8 and Figure 6. The response table displayed the highest level value (NiR:T2) with the number of passes set to two, rotation speed at level 1, and transverse speed at level 2 (two passes, rotational speed of 1000 RPM, and traverse speed of 30 mm/min). These parameter levels were the optimal combination, or the best conditions for both high tensile strength and low tool wear.

| Parameters | Grey Relational Grade |
|------------|----------------------|
| N          | 0.5855               |
| R          | 0.6963 *             |
| T          | 0.5773               |

Total mean grey relational grade = 0.6315.

* Levels for optimum grey relational grade.
Finally, the Analysis of Variance (ANOVA) results of the grey relational grade summarize the contribution of each process parameter; these are shown as a contribution percentage in Table 9.

Table 9. ANOVA of the grey relational grade.

| Parameters | DF | SS    | MS    | % Contribution |
|------------|----|-------|-------|----------------|
| N          | 3  | 0.0137 | 0.0046 | 7.05           |
| R          | 1  | 0.0336 | 0.0335 | 51.68          |
| T          | 1  | 0.0235 | 0.0235 | 36.18          |
| Error      | 2  | 0.0066 | 0.0033 | 5.08           |
| Total      | 7  | 0.0774 | -     | -              |

The results of the ANOVA test showed that the most important parameter is rotational speed (51.68% contribution), the second is traverse speed (36.18% contribution), and the third is the number of passes (7.05% contribution). The rotation speed and transverse speed were the parameters that influenced the improvement of tensile strength and tool wear rate. Therefore, both rotation speed and transverse speed were very important parameters in aluminum surface composite fabrication by friction stir processing.

3.2. Confirmation Experiments

After evaluation of the optimal parameter settings (N:R:T), the next step was to predict the total mean grey relational grade by Equation (6). The optimal parametric combination of the predicted results was verified by repeatable experiments for the enhancement of quality characteristics.

3.2.1. Microstructural Characterization

After the confirmatory test, the cross-section specimen microstructure analysis of N:R:T condition by scanning electron microscope (SEM) showed that SiC particle separation and insertion in the flow direction of stirring affects dislocation, uniform dispersion, and homogeneity in the matrix (Figure 7).
Figure 7. Scanning electron microscopy (SEM) images of the stir zone of the N2R1T2 condition.

From the dislocation in the structure and uniform distribution of SiC particles, tensile strength improvement was induced.

3.2.2. Comparison of Process Parameters Condition

The best and worst conditions of grey relation grade in Table 7 and confirmed experiment conditions for the optimal process parameters were compared with the different results for multi-response confirmation; a summary of the results is shown in Table 10. The comparison of three process parameter conditions found that the confirmed experimental condition exhibited slightly lower tensile strength (229.90 MPa) than the best L8 experimental condition (233.32 MPa), but the confirmed experimental condition showed a lower tool wear rate than the best L8 experiment condition (0.0075). The confirmed experiment condition displayed both higher tensile strength and lower tool wear rate than the worst L8 experiment condition.

Table 10. Comparison of the process parameter conditions.

| Factor Level          | Process Parameter Conditions |
|-----------------------|------------------------------|
|                       | Best L8 Experiment | Worst L8 Experiment | Confirmed Experiment |
| Tensile strength (MPa) | N2R1T1       | N2R1T1       | N2R1T2       |
| Tool wear rate (%)    | 233.32        | 218.07       | 229.90       |
| Grey relational grade | 0.0926        | 0.1209       | 0.0851       |
|                       | 0.7496        | 0.5012       | 0.8175       |

Improvement in the grey relational grade = 0.0679.

The microstructure of the three conditions is shown in Figure 8, which shows the different SiC particle distribution and uniform particle microstructure. The microstructure of the best L8 experiment condition (N2R1T1) showed a good small split SiC particle dispersion, as shown in Figure 8a. This has high tensile strength, and has an effect on the tool wear rate, as shown in Figure 9a. Figure 9a shows the post-processed pin for N2R1, and the pin shows it has slightly lost its addendum part of the thread. Condition N2R1T1 exhibited a few breaks of SiC particles, as shown in Figure 8b. The few breaks of SiC particles resulted in low tensile strength and high tool wear rate, with the thread of the pin degenerating extremely quickly. For confirmation, the experimental condition had a small split of SiC particles, similar to the N2R1T1 condition, as shown in Figure 8c. This resulted in nearly the same tensile strength as N2R1T1, but a lower tool wear rate than the N2R1T1 condition. The small SiC particles diffused into the matrix structures in the direction of the movement of the pin, so these small SiC particles were not agglomerated in the matrix. Furthermore, it can be seen that there were many broken SiC particles and particles in the stirring flow direction. Moreover, an improvement of
mechanical properties is due to a reduction in the size of SiC particles (broken SiC particles), which was similar to the findings in prior research [38]. The broken SiC particles can also reduce the tool wear [39].

![Figure 8. SEM images of stir zone of (a) N0R1T1; (b) N0R2T1; (c) N2R1T2.](image)

![Figure 9. Tool wear images of (a) N0R1T1; (b) N0R2T1; (c) N2R1T2.](image)

As a consequence, the confirmed experimental condition N2R1T2 represents the optimal process parameters for FSP in terms of both tensile strength and tool wear rate.

5. Conclusions

This research found the optimal conditions for the fabrication of AA5052 + 10% SiCp by FSP. The Taguchi method, based on grey relational analysis on multiple responses, namely, tensile strength and tool wear rate, was used to determine the optimal conditions. The Taguchi design was used for the primary experiment; after that, the primary experiment result was analyzed by grey relational analysis for the optimal conditions of FSP. The primary experimental results showed increasing tensile strength and tool wear rate with an increasing number of passes. The N2R1T2 condition of the primary experiment showed the highest tensile strength (234.73 MPa) and high tool wear rate (0.1085). However, FSP required the highest tensile strength and the lowest tool wear rate to achieve good mechanical properties and effectiveness for the fabrication of AA5052 + 10% SiCp. Therefore, the next step was to search for the optimal condition of FSP by grey relational analysis. The grey relational analysis showed the optimum number of passes as two, the rotational speed of 1000 RPM, and traverse speed of 30 mm/min (condition N2R1T3). The optimal condition for the multiple response analysis exhibited high tensile strength (229.90 MPa) and low tool wear rate (0.0851). The results of ANOVA indicated that the most important parameter was rotational speed (R), followed by traverse speed (T), and number of passes (N).

Microstructure analysis (condition N2R1T1) by scanning electron microscope (SEM) showed that the SiC reinforcement particles’ separation and insertion in the direction of stirring flow affected the dislocation, uniform dispersion, and homogeneity of the matrix. The small SiC particles had good
diffusion in the matrix direction in the direction of the movement of the pin; these small SiC particles were not agglomerated in the matrix. Furthermore, it could be seen clearly that there were many breaks of SiC particles and that particles flowed in the direction of stirring.

Therefore, the conditions of Na:R:T: are optimal for the fabrication of AA5052 + 10% SiCp, providing a balance between the mechanical properties and low rate of tool wear. Reducing the tool wear rate leads to an increased tool life, which decreases the overall process time for FSP.

The experimental results confirmed that the application of the Taguchi method in conjunction with grey relational analysis in multi-objective optimization in this research was satisfactory. Thus, it can effectively be applied to solve decision-making problems in other industrial processes. However, the disadvantage of this method is the issue of accuracy. For future works, other methods for identifying multi-objective optimization using other methods such as Topsis, Fuzzy, GA, etc. should be applied and compared with the current methods.

**Author Contributions:** Conceptualization, J.O.; Methodology, R.K and J.O.; Validation, R.K., J.O. and Ch.S.; Formal analysis, R.K.; Investigation, R.K. and J.O.; Resources, R.K., J.O., and Ch.S.; Data curation, R.K. and J.O.; Writing—original draft preparation, R.K.; Writing—review and editing, J.O. and Ch.S.; Visualization, R.K.; Supervision, J.O.; Project administration, J.O. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Acknowledgments:** This research was supported by the Department of Industrial Engineering, Ubon Ratchathani University, and Rajamangala University of Technology Isan, Nakhon Ratchasima, Thailand.

**Conflicts of Interest:** The authors declare no conflicts of interest.

**References**

1. Sharma, V.; Gupta, Y.; Kumar, B.V.M.; Prakash, U. Friction Stir Processing Strategies for Uniform Distribution of Reinforcement in a Surface Composite. Mater. Manuf. Process. 2016, 31, 1384–1392.

2. Thankachan, T.; Prakash, K.S. Microstructural, Mechanical and Tribological Behavior of Aluminum Nitride Reinforced Copper Surface Composites Fabricated Through Friction Stir Processing Route. Mater. Sci. Eng., A 2017, 688, 301–308.

3. Eskandari, H.; Taheri, R. A Novel Technique for Development of Aluminum Alloy Matrix/TiB/TiO2 Hybrid Surface Nanocomposite by Friction Stir Processing. Procselia Mater. Sci. 2015, 11, 503–508.

4. Bahrami, M.; Dehghani, K.; Givi, M.K.B. Novel Approach to Develop Aluminum Matrix Nano-composite Employing Friction Stir Welding Technique. Mater. Des. 2014, 53, 217–225.

5. Qin, Y.; Qu, M.; Pan, Y.; Zhang, C.; Schubert, D.W. Fabrication, characterization and modelling of triple hierarchical PET/PC/TPU composite fibres for strain sensing. Compos. Part A Appl. Sci. Manuf. 2020, 129, 105724.

6. Qu, M.; Nilsson, F.; Qin, Y.; Yang, G.; Pan, Y.; Liu, X.; Hernandez Rodriguez, G.; Chen, J.; Zhang, C.; Schubert, D.W. Electrical conductivity and mechanical properties of melt-spun ternary composites comprising PMMA, carbon fibers and carbon black. Compos. Sci. Technol. 2017, 150, 24–31.

7. Wang, H.; Li, L.; Qiu, S.; Zhai, W.; Li, Q.; Hao, S. Evolution of Microstructure at the Surface of 40CrNiMo7 Steel Treated by High-Current Pulsed Electron Beam. Coatings 2020, 10, 311.

8. Karbalaei Akbari, M.; Baharvandi, H.R.; Shirvanimoghaddam, K. Tensile and fracture behavior of nano/micro TiB particle reinforced castings A356 aluminum alloy composites. Mater. Des. 2015, 66, 150–161.

9. Sharifitabar, M.; Sarani, A.; Khorseshian, S.; Afarani, M.S. Fabrication of 5052Al/Al2O3 Nanoceramic Particle Reinforced Composite via Friction Stir Processing Route. Mater. Des. 2011, 32, 4164–4172.

10. Yang, M.; Xu, C.; Wu, C.; Lin, K.-C.; Chao Y.J.; An, L. Fabrication of AA6061/Al2O3 Nano Ceramic Particle Reinforced Composite Coating by using Friction Stir Processing. J. Mater. Sci. 2010, 45, 4431–4438.

11. Gandra, J.; Miranda, R.; Vilac, P.; Velhinho, A.; Teixeira, J. P. Functionally Graded Materials Produced by Friction Stir Processing. J. Mater. Process. Technol. 2011, 211, 1659–1668.

12. Sharma, V.; Prakash, U.; Kumar, B.M. Surface Composites by Friction Stir Processing: A Review. J. Mater. Process. Technol. 2015, 224, 117–134.

13. Salehi, M.; Saadatmand, M.; Aghazadeh Mohandes, J. Optimization of Process Parameters for Producing AA6061/SiC Nanocomposites by Friction Stir Processing. Trans. Nonferrous Met. Soc. China 2012, 22, 1055–1063.

14. Kurt, H.I. Influence of Hybrid Ratio and Friction Stir Processing Parameters on Ultimate Tensile Strength of 5083 Aluminium Matrix Hybrid Composites. Composites Part B 2016, 93, 26–34.

15. Shahraki, S.; Khorasani, S.; Behnag, R.; Fotouhi, Y.; Bisadi, H. Producing of AA5083/ZrO2 Nanocomposite by Friction Stir Processing (FSP). Metall. Mater. Trans. B 2013, 44, 1546–1553.
16. Narimani, M.; Lotfi, B.; Sadeghian, Z. Investigating the Microstructure and Mechanical Properties of Al-TiB2 Composite Fabricated by Friction Stir Processing (FSP). Mater. Sci. Eng. A 2016, 673, 436–442.
17. Sabry, I.; El-Kassas, A.M.; Mourad, A-H.I.; Thekkudan, D.T.; Qudeir, J.A. Friction Stir Welding of T-Joints: Experimental and Statistical Analysis. J. Manuf. Mater. Process. 2019, 3, 38.
18. Prado, R.; Prado, R. Effect of Tool Wear in the Friction-Stir Welding of Aluminum Alloy 6061+20%Al2O3: A Preliminary Study. Scr. Mater. 2001, 45, 743–746.
19. Sabry, I.; Huang, M. Application of grey relation analysis to minimize forces and vibrations during precise ball end milling. Precis. Eng. 2018, 51, 582–596.
20. Reddy, V.; Jawahar, M. Multi-objective optimization of parameters during EDM of aluminum alloy 6082 using grey relation analysis. Int. J. Latest Trends Eng. Technol. 2016, 6, 570–576.
21. Prado, R.; Murr, L.E.; Soto, K.F.; McClure, J.C. Self-Optimization in Tool Wear for Friction-Stir Welding of Al 6061/20% Al2O3 MMC. Mater. Sci. Eng. A 2003, 349, 156–165.
22. Prado, R.; Murr, L.E. Characterization of Tool Wear and Weld Optimization in the Friction-Stir Welding of Cast Aluminum 359+20%SiC Metal-Matrix Composite. Mater. Charact. 2004, 52, 65–75.
23. Thompson, B.; Babu, S.S. Tool Degradation Characterization in the Friction Stir Welding of Hard Metals. Weld. J. 2010, 89, 256–261.
24. Rahsepar, M.; Jarahimoghadam, H. The Influence of Multipass Friction Stir Processing on the Corrosion Behavior and Mechanical Properties of Zircon-reinforced Al Metal Matrix Composites. Mater. Sci. Eng. A 2016, 671, 214–220.
25. Deng, J.L. Introduction to Grey System. J. Grey Syst.-UK 1989, 1, 1–24.
26. Ramu, I.; Srinivas, P.; Vekatesh K. Taguchi based grey relational analysis for optimization of machining parameters of CNC turning steel 316. IOP Conf. Ser.: Mater. Sci. Eng. 2018, 377, 012078.
27. Wojciechowski, S.; Maruda, R.W.; Kroczynk, G. M., Nieslon, P. Application of signal to noise ratio and grey relational analysis to minimize forces and vibrations during precise ball end milling. Precis. Eng. 2018, 51, 582–596.
28. Reddy, V.; Jawahar, M. Multi-objective optimization of parameters during EDM of aluminium alloy 6082 using grey relational analysis. Int. J. Latest Trends Eng. Technol. 2016, 6, 570–576.
29. Zohoor, M.; Givi, M.B.; Salami, F. Effect of Processing Parameters on Fabrication of Al–Mg/Cu Composites via Friction Stir Processing. Mater. Des. 2012, 39, 358–365.
30. Dhayalan, R.; Kalaiselvan, K.; Sathiskumar, R. Characterization of AA6063/SiC-Gr Surface Composites Produced by FSP Technique. Proced. Eng. 2014, 97, 625–631.
31. Vijayavel, P.; Balasubramanian, V.; Sundaram, S. Effect of Shoulder Diameter to Pin Diameter (D/d) Ratio on Tensile Strength and Ductility of Friction Stir Processed LM25AA-5%SiCp Metal Matrix Composites. Mater. Des. 2014, 57, 1–9.
32. Zahmatkesh, B.; Enayati, M.H. A Novel Approach for Development of Surface Nanocomposite by Friction Stir Processing. Mater. Sci. Eng. A 2010, 527, 6734–6740.
33. Ke, L.; Huang, C.; Xing, L.; Huang, K. Al–Ni Intermetallic Composites Produced in Situ by Friction Stir Processing. J. Alloys Compd. 2010, 503, 494–499.
34. Qian, J.; Li, J.; Xiong, J.; Zhang, F.; Lin, X. In Situ Synthesizing AlNi for Fabrication of Intermetallic-Reinforced Aluminum Alloy Composites by Friction Stir Processing. Mater. Sci. Eng. A 2012, 550, 279–285.
35. Prater, T.J. Predictive Process Modeling of Tool Wear in Friction Stir Welding of Metal Matrix Composites. Ph.D. Dissertation, Graduate School of Vanderbilt University, Nashville, TN, USA, 2012.
36. Prater, T.; Strauss, A.; Cook, G.; Gibson, B.; Cox, C. A Comparative Evaluation of the Wear Resistance of Various Tool Materials in Friction Stir Welding of Metal Matrix Composites. J. Mater. Eng. Perform. 2013, 22, 1807–1813.
37. Surekha, K.; Els-Botes, A. Effect of Cryotreatment on Tool Wear Behaviour of Bohler K390 and AISI H13 Tool Steel During Friction Stir Welding of Copper. Trans. Indian Inst. Met. 2012, 65, 259–264.
38. Ghetiya, N.D.; Patel, K.M.; Kavar, A.J. Multi-objective Optimization of FSW Process Parameters of Aluminium Alloy Using Taguchi-Based Grey Relational Analysis. Trans. Indian Inst. Met. 2016, 69, 917–923.
39. Rathee, S.; Maheshwari, S.; Siddiquee, A.N.; Srivastava, M.; Investigating the Effects of SiC Particle Sizes on Microstructural and Mechanical Properties of AA5059/SiC Surface Composites During Multi-Pass FSP. Silicon 2019, 11, 797–805.
40. Prater, T.; Gibson, B.; Cox, C.; Cook, G.E. Strauss, A.M. Effect of particle size on tool wear in friction stir welding of Al 6061 with silicon carbide reinforcement. J. Manuf. Technol. Res. 2014, 6, 125–142.