

Research Article

Optimization of Process Parameters of Copper Composites Produced via Friction Stir Processing

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Abstract

Increasing demands for operating properties of fabricated elements on one hand, and a necessity of reducing mass of a structure on the other, triggers materials engineering research into producing surface layers representing required functional properties. Methods commonly used in the production of surface layers, such as surfacing, spraying or re-melting with a laser beam have been known for years. A new method is the friction stir processing (FSP) of surface layers. The FSP process is primarily used for the modification of microstructure in near-surface layers of processed metallic components this deformation is produced by forcibly inserting a non-consumable tool into the workpiece, and revolving the tool in a stirring motion as it is pushed laterally through the workpiece. This is promising process for the automotive and aerospace industries where new material will need to be developed to improve resistance to wear, creep, and fatigue. The main objective of this project is to be producing copper reinforced metal matrix composite layers using micro sized TiB₂ particles via friction stir processing (FSP) in order to enhance surface mechanical properties. Taguchi method was used to optimize these factors for maximizing the mechanical properties of surface composites. The fabricated surface composites were examined by optical microscope (OM) and scanning electron microscopy (SEM) for dispersion reinforcement particles. It was found that TiB₂ particles are uniformly dispersed in the stir zone. Mechanical properties like tensile, Impact, hardness, were also evaluated. The results showed that functional characteristics of surface composites increased with the increase in vol. % of the micro sized TiB₂ reinforcement particles. The observed mechanical properties are correlated with microstructure and fracture features.

Keywords: Friction Stir Processing, Copper, TiB₂ Micro reinforcement particles, Micro Structure, Stir Zone, Orthogonal Array, Signal –to-Noise ratio, Grey Relational Analysis, ANOVA.

1. Introduction

Friction stir processing (FSP), a solid state technique based on the principle of Friction stir welding (FSW) is used for material processing in order to modify the microstructure and mechanical properties of surface composites and fabricate the surface composites. Over the last several decades they has been considering interest in the use of copper based metal matrix composites (CMMCs). For many applications pure Cu cannot be used because of its low strength therefore improvement in the properties of copper has become essential for its applications in cutting edge technological applications. The improvement of the mechanical properties of copper is important for its use in larger number of fields. Composite materials offer several applications in the aerospace, electronics, automotive, and ship building industries as they have certain advantages over other conventional metals.

The major benefit of copper based composites is their low density, improved fatigue, superior corrosion resistance and higher specific strength in many engineering applications, the surface properties decides life of the component rather than their bulk properties. The surface layer reinforced with ceramic particles (TiB₂) is normally called surface composites. Through several techniques are available to fabricate surface composites, friction stir processing(FSP) is simple, green and low energy consumption route based on the principle of FSW to fabricate the surface composites with superior results.

Titanium Di-boride (TiB₂) particles are of great technological importance because of their applications as reinforcement for metal matrix composites. High mechanical strength, as well as high stiffness and hardness, are the main TiB₂ characteristics. Furthermore, in contrast to most ceramic materials, TiB₂ is electrically and thermally conductive. Metal transfer, generated between the tool shoulder and the work piece plate, place an important role in influencing the mechanical properties during FSP.

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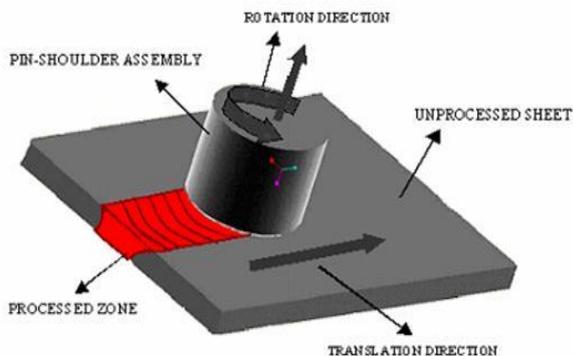


Fig 1 Friction Stir Processing

An FSP tool consists of a shoulder and a pin. Firstly the tool without pin is used and moves traverse along the groove consisting of reinforcement particles thus forging it latter the tool with pin is used moves along the desire line to cover the region underneath the shoulder. Friction between the tool and work piece results in localized heating that softens and plasticizes the work piece during this process, the material undergoes plastic deformation, thus resulting in grain refinement to improve its mechanical properties.

Table1. Mechanical Properties of base metal

Material	UTS/Mpa	YS/Mpa	% EL	Micro hardness/HV	IT/J
Pure Copper	255	210	10	80	12

Table 2 Chemical Properties of base metal

Component	Cu	Zn	Pb	Fe	Bi
Weight %	99.99	<0.026	<0.05	<0.0025	<<0.001

2. Experimental design using Taguchi’s method

Taguchi's technique has been used widely in engineering design. It is an efficient tool which enables the up gradation of the performance of the product, process and design with significant prediction of cost and time. The main trust of Taguchi's technique is the use of parameter design, which is an engineering method for product or process design that focuses on determining the parameter settings producing the best Level of a quality characteristic with minimum variation. It is simple, efficient and systematic approach to determine optimal process parameters. Taguchi design provides a powerful and efficient method for designing processes that operate consistently and optimally over a variety of conditions. In friction stir processing the parameters used in this experimental work are tool travel speed, tool rotation speed, and vol. % age of reinforcement particles. Feasible levels of the process parameters were chosen in such a way that the surface composite should be free from defects.

2.2 Experimental Layout L₉ Orthogonal Array

Table 3 Experimental Layout

Expt. No	Process Parameters		
	Tool travel Speed (mm/min)	Tool rotational speed/rpm	TiB ₂ p. Vol%
FSP 1	20	900	8
FSP 2	20	1120	16
FSP 3	20	1400	24
FSP 4	30	900	16
FSP 5	30	1120	24
FSP 6	30	1400	8
FSP 7	40	900	24
FSP 8	40	1120	8
FSP 9	40	1400	16

In this study, the analysis was carried out using statistical software MINITAB-17.Hence orthogonal array was selected in the present work. Each condition of experiments was repeated twice in order to reduce the noise error effects. The quality characteristics in the analysis of S/N ratio. The S/N ratio for each combination of the process parameters is computed based on the Signal Noise analysis, such as ultimate tensile strength (UTS), yield strength (YS), percentage of elongation (% EL),micro hardness (HV)and impact toughness (IT) of surface composite were evaluated for all the trials and then statistical analysis of variance was carried out. Based on analysis of variance, the contribution of each element influencing the quality characteristic is evaluated. The optimum combinations of process parameters were predicted and verified.

2.3 Experimental Procedure

- Commercially available pure copper (C11000) plates of 200mm length, 100mm width, and 6mm thickness, micro sized Titanium Di-boride (TiB₂) was used as reinforcement particle.
- The average size of the reinforcement particle is 120nm, 80nm respectively and SEM microstructure of as received, in this project work to fabricate the surface composites with a vertical milling machine (Make: HMT FN-3, 10hp, 1800 Rpm).
- The copper plates of required dimensions cut from the given plate using Power hacksaw machine. After that, the sharp corners of the samples are rounded by filing and these surface plates were cleaned with grinding paper and methanol before processing.
- A groove was made in the middle of the copper plate using slitting cutter on milling machine and the groove size was varied as groove width 0.5mm, 1mm, 1.5mm, groove depth 4.5mm and groove length 200mm.The TiB₂ reinforcement particles was filled in the groove.
- The tool material used as H13 Tool Steel. It consisted of a shoulder with a diameter of 22 mm and pin with diameter 4.5 mm and length 6 mm respectively.

- This tool is fitted into the tool holder and work piece is rigidly clamped to machine table using fixtures. The TiB₂ reinforcement particles were compressed into the groove and the upper surface of the groove was closed with a FSP tool without pin to prevent the TiB₂ particles to escape from the groove. In the next stage the tool is plunged with the pin into the plate to stir the material.
- The tool rotation speed and tool travel speed was adjusted at 900, 1120 and 1400 rpm, and 20, 30, 40 mm/min along the axis of the work piece with different Vol. % age of reinforced particles as 8, 16 and 24 to produce the surface composites.

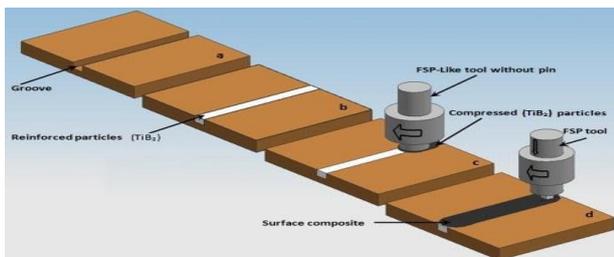


Fig.2 Friction Stir Process

After FSP, microstructure observations were carried out at the cross section of stir zone (SZ) of surface composites normal to the FSP direction mechanically polished and etched with 10% potassium dichromate in 100cc of water+2cc of Conc. HCL. Microstructure changes were observed by optical microscope (OM) in the SZ/N. The tensile specimens were prepared as per the ASTM E8 Standards by wire cut EDM to the required dimensions which is transverse to the SZ. The tensile test was conducted with the help of a computer control universal testing machine. The impact specimens were prepared as per ASTM A370 Standards. Charpy V notch impact test was carried out using pendulum type impact testing machine at room temperature. Micro hardness test was carried out using Vickers digital micro hardness tester with a 0.5kg load for 10sec duration at the cross section of SZ of surface composites normal to the FSP direction as per ASTM E92 Standard.

3. Results and Discussions

3.1 Microstructure

The material is under severe plastic deformation (SPD) that causes a fine grain structure with high angle boundaries. The taper cylindrical threaded tool pin profile made uniform distribution of TiB₂ reinforcement particles in cu based metal matrix composites. FSP can be successfully used for fabrication of micro composite layer with uniform distribution of TiB₂ particles at low heat input under condition of 2° tilt angle and combination of various process parameters such as (Tool rotational speed, Tool travel speed, % of vol. fraction). The heat input that causes annealing and grain growth in sides region

of SZ. Agglomeration of TiB₂ particles cannot be observed.

The main difference between HAZ and BM is grain boundary. Grain boundary in HAZ is not obvious as in BM, but because of annealing, grains growth in HAZ favored. No cluster of particles is seen. Further, there is no segregation of particles along the grain boundaries. The distribution is almost intergranular. The mechanical tribological properties of CMMCs are dictated by the nature of distribution. Homogeneous and intergranular distribution is preferred to obtain superior properties.

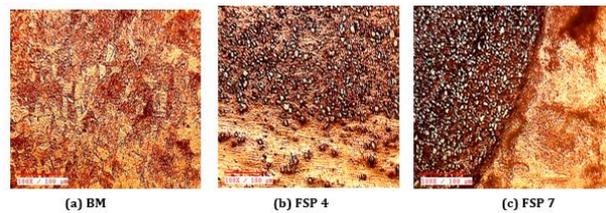


Fig.3 Optical microstructures of Base metal and FSP 4, FSP 7 Surface composites

SEM micrographs of CMCs reinforced with TiB₂ ceramic particles at higher magnification. The etchant used in this work reveals the grain boundaries. The interface between the copper matrix and ceramic particles is clear without the presence of pores on reaction products. TiB₂ ceramic particles bonded well with the copper matrix. No such pores are observed near any ceramic particle. This can be related to sufficient material flow and plasticization of copper under chosen experimental condition. Good interfacial bonding is prerequisite in spite of homogeneous distribution to enhance the properties.

3.2 Mechanical properties

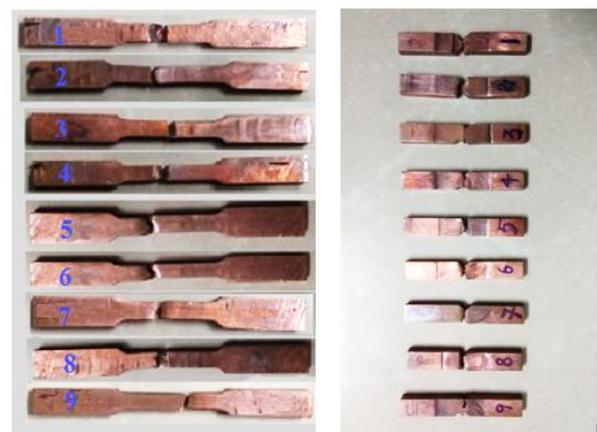


Fig.4 Micro Friction Stir Processed TENSILE & IMPACT& Tested Specimens (FSP1-FSP9)

The mechanical properties of CMCs are generally influenced by the type, size, and shape, % of vol. fraction and spatial distribution of ceramic particles.

Increased in the UTS, YS of the surface composites over the pure copper matrix is due to the grain refinement of copper in the surface composite which can be related to the interaction between the powder particles and dislocations with the matrix. It is also found that with increase in the addition of TiB₂ reinforcement particles and is uniformly distributed on copper matrix and thus improving strength at elevated temperature.

The elongation of the FSPed specimens with addition of TiB₂ particles were reduced as compared to the pure copper due to the increase in %vol. of reinforcement particles which increases the effective slip distance of dislocations during the deformation. The impact toughness of the surface composites values were observed at various process parameters and higher impact toughness values were observed at higher vol. Fraction of the reinforced particles with copper matrix. These characteristics to the addition of reinforced particles which causes softening of the matrix due to frictional heat of the tool and shoulder pin.

3.3 Micro hardness

The micro hardness profile shows that hardness value in the SZ is much higher than that of the BM. The micro hardness of as received copper was 80Hv. The reinforcement of ceramic particles increase the micro hardness above 110Hv. Cu-TiB₂ CMCs recorded a max hardness of 119.5Hv. The rise in micro hardness of CMCs indicates that the ceramic particles contributed remarkably to the strengthening of the copper matrix. The hindrance to dislocation movement by higher dislocation density enhances hardness. Higher hardness/strengthening can be attributed in the stir zone due to following factors;

- Distribution of TiB₂ particles in the pure Cu matrix as a hard phase.
- The hardness of the ceramic particles is extremely higher to that of copper matrix.
- The grain size of CMCs is smaller to that of the copper matrix due to grain refinement of ceramic particle. The fine grains improve the hardness.

4. Determination of optimal machining parameters

Grey Relational Analysis (GRA): Taguchi's method is focused on the effective application of engineering strategies rather than advanced statistical techniques. The primary goals of Taguchi method are:

- A reduction in the variation of a product design to improve quality and lower the loss imparted to society.
- A proper product or process implementation strategy, which can further reduce the level of variation.

The steps involved in Taguchi's Grey Relational Analysis are:

STEP 1- : The transformation of S-N Ratio values from the original response values was the initial step. For that the equations of "larger the better", "smaller the better" and "nominal the best" were used. Subsequent analysis was carried out on the basis of these S/N ratio values. This is shown in table 5.

$$\text{Type1: } S/N_{HB} = -10 \log_{10} \left[\left(\frac{1}{n} \right) \left(\sum \frac{1}{Y_{ij}^2} \right) \right]$$

$$\text{Type1: } S/N_{LB} = -10 \log_{10} \left[\sum \frac{Y_{ij}^2}{n} \right]$$

$$\text{Type1: } S/N_{NB} = -10 \log_{10} \left[\frac{1}{S^2} \right]$$

Where

Y_{ij} is the value of the response 'j' in the ith experiment condition, with i=1, 2, 3...n; j = 1,2...k and S² are the sample mean and variance.

STEP 2: In the 2nd step of the grey relational analysis, pre-processing of the data was first performed for normalizing the raw data for analysis. This is shown in Table 6. The following formula to avoid the effect of adopting different units and to reduce the variability. The normalized output parameter corresponding to the larger-the-better criterion can be expressed as,

$$x_i^* = \frac{x_i^0(k) - \min x_i^0(k)}{\max x_i^0(k) - \min x_i^0(k)}$$

STEP 3: The grey relational coefficient, is calculated to express the relationship between the larger (best) and actual normalized experimental results. Before that the deviation sequence for the reference and comparability sequence were found out. These are given in Table 6 and the grey relational coefficient can be expressed as,

$$\xi_i(k) = \frac{\Delta_{\min} + \zeta \Delta_{\max}}{\Delta_{0i}(k) + \zeta \Delta_{\max}}$$

$$\Delta_{0i} = x_0(k) = x_i(k)$$

X_i (k) denotes the comparability sequence. 'ξ' is distinguishing or identified coefficient. The Value of ξ is the smaller and the distinguished ability is the larger. ξ= 1 is generally used.

Step 4: The grey relational grade was determined by averaging the grey relational coefficient corresponding to each performance characteristic. The overall performance characteristic of the multiple response process depends on the calculated grey relational grade. The grey relational grade can be expressed as,

$$\gamma_{i=\frac{1}{n}} = \sum_{k=1}^n \xi(k)$$

Where, Y_i is the grey relational grade for the Jth experiment and k is the number of performance characteristics.

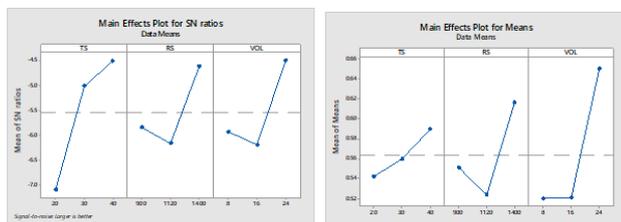
Step 5: Determination of the Optimal Factor and its Level Combination. Since the experimental design is orthogonal, it is possible to separate out the effect of each machining parameter on the grey relational grade at different levels.

The larger the grey relational grade, the better is the multiple performance characteristics. However, the relative importance among the machining parameters for the multiple performance characteristics still needs to be known, so that the optimal combinations of the machining parameter levels can be determined more accurately.

5. Taguchi Analysis for Grey Relational Grade

Table 4 For Grey Relational Grade (Larger is Better)

Level	Grey Relational Grade		
	TS	RS	Vol.%
1	0.5420	0.5512	0.5199
2	0.5598	0.5236	0.5211
3	0.5896	0.6167	0.6503
Delta	0.0476	0.0931	0.1304
Rank	3	2	1



Tool travel speed (mm/min) 40, Tool Rotational speed/rpm 1400, Vol. %age 24 is the optimal parameter level for the higher grey relational Grade. The prediction of Optimum value for Grey Relational Grade:

Predicted Response = Average of A3 + Average of B3 + Average of C3 - 2 x Mean of response (Y_{ij}).

5.1 Conformation test at Optimal Parameter Level obtained from Grey Relational Analysis:

The confirmation experiment was conducted at the Optimal Process Parameters namely Tool travel speed at level 3 (40 mm/min), Tool rotational speed at level 3 (1400 rpm), and Vol. % reinforced particles at level 3 (24%) to evaluate Response quality characteristics were represented.

Table: 6 Conformation Test Results at Optimal level of Parameters

Optimization	Predicted Grey Relational Grade	Optimal level of parameters	Conformation Test Results				
			UTS	YS	%EL	HV	IT
Grey Relational Analysis	0.7289	TS = 40 mm/min RS = 1400 rpm Vol. % = 24	270.18	202.1 4	6.8	125.4	14

The comparison again shows the good agreement between the Optimal predicted (A3B3C3) and the experimental (A3B1C3) Values

Table 5 Confirmation Results

Level	Optimal Process Parameters	
	Predicted	Experiment
	A3B3C3	A3B1C3
UTS	269.46	269.77
YS	189.2	184.51
%EL	6.08	5.48
HV	122.3	119.5
IT	16	14

6. Analysis of Variance & Percentage of contribution

The main purpose of the ANOVA is the application of a statistical method to identify the effect of individual factors on the process responses. The Taguchi experiment method could not judge the effect of individual parameters on the entire process. The percentage of contribution using ANOVA is used to compensate for this effect. The percentage of contribution is the portion of the total variation observed in the experiment attributed to each significant factors and interaction which is reflected. In order to find the effect of process parameters on various responses. The calculated F-values of the ANOVA for various responses determine the relative significance of different process parameters. Results of ANOVA revealed that the process parameters have significant effect on all the quality Characteristics.

Based on the results, Vol. %age of reinforcement particles (TiB2) is found to be the most influencing process parameter followed by tool tilt angle and concave angle at the tool shoulder. The percentage of contribution is the portion of the total variation observed in the experiment attributed to each significant factor. The percentage of contribution is the function of the sum of the squares for each significant item and it indicates the relative power of a factor to reduce the variation. If the factor levels are controlled precisely then the total variation could be reduced by an amount which is indicated by percentage of contribution. The percentage of the vol. %age of reinforcement Particles are higher because these particles play an important role in material strength through dislocation and grain boundary pinning mechanism. The presence of hard reinforcement particles thus enhances the hardness.

7. Analysis of Variance & Percentage of contribution

The main purpose of the ANOVA is the application of a statistical method to identify the effect of individual factors on the process responses

Table: 7 Anova for Grey Relational Grade

Process Parameters	Degree of Freedom (DF)	Sum of Squares (SS)	Adj-mean Sum of Squares (MS)	F-ratio (F)	% of Contribution
TS	2	0.027378	0.013689	4.99	49.77
RS	2	0.004011	0.0020055	0.73	7.29
Vol. %	2	0.018134	0.009067	3.30	32.97
Error	2	0.005485	0.0027425		9.97
Total	8	0.055009			100

8. Results & Discussions

After performing the experiment for all 9 runs of Cu-TiB₂ Surface composites and measuring the output parameters like Ultimate tensile strength, Yield Strength, %of Elongation, Micro Hardness and Impact Strength of Copper metal matrix composite material.

Main Effect Plots for Friction Stir Processed Input Parameters V/S Output Parameters

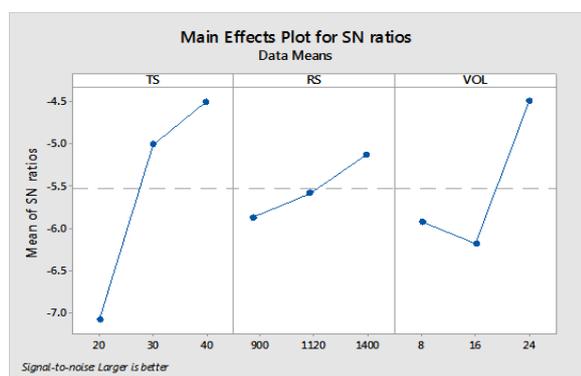


Fig.5 Grey Relational Grade

Tool travel speed (mm/min) 40, Tool Rotational speed/rpm 1400, Vol. %age 24 is the optimal parameter level for the higher grey relational Grade.

8.1 Confirmation test at Optimal Parameter Level obtained from Grey Relational Analysis

The confirmation experiment was conducted at the Optimal Process Parameters namely Tool travel speed at level 3 (40 mm/min), Tool rotational speed at level 3 (1400 rpm), and Vol. % reinforced particles at level 3 (24%) to evaluate Response quality characteristics.

Table: 8 Confirmation Test Results at Optimal level of Parameters

Optimization	Predicted Grey Relational Grade	Optimal level of parameters	Conformation Test Results				
			UTS (Mpa)	YS (Mpa)	%EL	H (Hv)	IT (J)
Grey Relational Analysis	0.7215	TS = 40 mm/min RS = 1400 rpm Vol. % = 24	270.18	202.14	6.8	125.4	14

The following table shows the corresponding improvements in Ultimate Tensile Strength (UTS), Hardness (Hv), and Impact Strength (IT) is 5.95%, 49.2%, 16.6% respectively. It is clearly shown that by applying Multi variable Optimization for process parameters optimization in FSP, greatly improved the output Response Variables.

8.2 ANOVA Analysis

From ANOVA for based Friction Stir Processed Grey Relational Grade:

The ANOVA analysis is conducted to know the percentage contribution of the input parameters on output parameters. The percentage of contribution is the portion of the total variation observed in the experiment attributed to each significant factors and interaction which is reflected. Tool Travel speed is 49.77%, Tool rotation speed is 7.29% and Vol. % of Reinforced particles is 32.97%.

This shows that the Tool Travel speed and Vol. % of Reinforced particles are the most influence parameters in increases of Response characteristics.

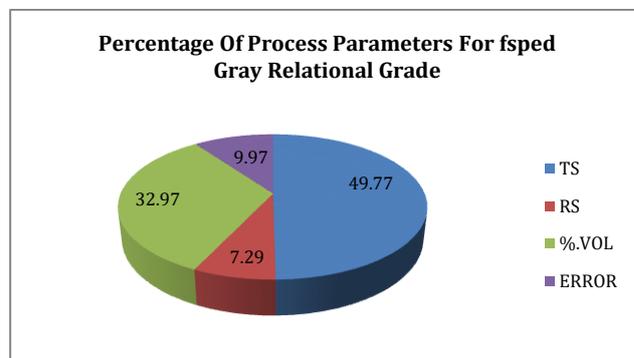


Fig.6 % of process parameters for Friction Stir Processed grey relational grade

Conclusion

The experiments were conducted to optimize the influence of process parameters such as Tool travel speed (mm/min), Tool rotational speed (rpm), and Vol. % (TiB₂ Particles) on microstructure and mechanical properties of Cu-TiB₂ surface composites fabricated via Friction stir processing. The following conclusions were drawn from the present study.

- Designing of grooves was very effective to achieve agglomeration free surface composite where the Micro TiB₂ (Titanium Di-boride) reinforcement particles were well bonded with copper matrix. The micro FSPed on the stir zone exhibited better mechanical properties than the as received pure copper
- Good interfacial bonding was observed in between Copper and Titanium Diboride (TiB₂) reinforcement particles by micro structural analysis. Stir zone has fine and equiaxed grains and distribution of TiB₂ particles

in the matrix is uniform. No intermetallic compound was formed after FSP and observed no defects after processing.

- Micro Composites tangibly showed better mechanical properties compared with non-reinforced Sample, and the improvements in ultimate tensile strength (273.02/Mpa) by (7.06%), hardness (119.5Hv) by (42.26%), and impact strength (14/J) by (16.67%) were attained in the 24 Vol. % TiB₂ FSPed Copper Composites.
- Confirmation test at Optimal Parameter levels obtained from grey relational analysis (Micro Composites), significantly improvements in ultimate tensile strength (275.82/Mpa) by (8.16%), hardness (125.4/Hv) by (49.2%), and impact strength (14/J) by (16.67%) were attained in the 24 Vol. % TiB₂ sped Copper Composites.
- Mechanical properties of Cu-TiB₂ Surface composites increased with increase in the vol.% age of reinforcement particles, whereas the specimens of micro FSPed with TiB₂ particles exhibits a poor elongation and Yield strength. The hardness (125.4/Hv) of the speed stir zone increased by 49.2% higher than that of the pure copper (84/Hv). Use of MICRO TiB₂ (Titanium Di-boride) reinforcement particles in Friction Stir Processing as composite material increases hardness at greater extent.

From Grey Relational analysis

Grey Relational analysis was applied in this work to improve the multi-response characteristics such as Ultimate Tensile strength (UTS), Yield Strength (YS), Percentage of Elongation (%EL), Hardness (H), and Impact Strength (IT). The conclusions of this work are summarized as follows:

The optimal parameters combination was determined as A₃B₃ C₃ i.e. Tool travel speed at level 3 (40 mm/min), Tool rotational speed at level 3 (1400 rpm), and Vol. % reinforced particles at level 3 (24%) were checked with experimental results and a good agreement was found, by applying Multi variable Optimization for process parameters optimization in FSP, greatly improved the output Response Variables

This work demonstrates the method of using Taguchi Grey Analysis methods for optimizing the FSP parameters for multiple response characteristics From ANOVA for Based Friction Stir Processed Grey Relational Grade:

Tool Travel speed and Vol. % of Reinforcement particles are the most influence parameters in increases of Response characteristics

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