Optimization in tribological behaviour of Al-nanoTiO2 powder metallurgy composites using response surface method

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Abstract. The Al- nano TiO2 composite processed powder metallurgy and wear behaviour was experimentally investigated on aluminium with combined nano TiO2 weight percent reinforcement of 4%,7% and 10% at different sliding velocity of 1.5 m/s,2.0 m/s,2.5 m/s and different normal loads of 10N,30N,50N. The effect of specific wear rate and co efficient of friction optimised by response surface method. The results predicted by the developed model are concurring well within the measured values. The correlation co-efficient of model, regarding the specific wear rate and co efficient of friction, were about 0.96 and 0.94 respective with conform the degree of accuracy of the mathematical model. The optimal condition attain the minimum specific wear rate and co-efficient of friction at sliding velocity 2.04 m/s and Normal load 13.5 N with Al- TiO2 10% weight. The predicted value of specific wear rate 1.08e-7 and co efficient of friction 0.31 respectively.

1. Introduction
Powder Metallurgy (PM) techniques are used in manufacture of new composite material component in aerospace and automobile industry due to its superior characteristics. Aluminium metal matrix composites (AMCs) are quite attractive and various studies were reported on wear behavior of Aluminium composite reinforced with different materials [1-3]. Powder Metallurgy routed technique in manufacture of metal matrix nano composite will produce uniformity in the reinforced distribution[4,5].

Aluminium based hybrid AMCs along with TiO2 reinforced with various particle of SiC,SiO2 and Gr will improve the mechanical and wear resistance[6,7]. Addition of metal oxide TiO2 with Aluminium alloy possesses better wear resistant property [8-11]. The porosity and tensile strength of Al with nano TiO2 (size 50nm) will provide superior wear resistance[12].

1.1. Response Surface Methodology
As mentioned, RSM can be an excellent approach to study a process response and to figure out the best correlation among the parameters of a process. This is done via developed models based on the statistical methods to investigate the relation between the inputs and outputs of any process. The mathematical model has been developed based on the experimental results, with an objective to determine the specific wear rate and coefficient of friction; also to study the inter relationships between the test variables of Al – nano TiO2 composite.

The basic concept of response method [13] is to construct an approximation of this implicit limit state by an approximate simple and explicit polynomial function. A response surface equation is usually formulated as

$$Y = \beta_0 + \sum_{i=1}^{m} \beta_i x_i + \sum_{i=1}^{m} \beta_{ii} x_i^2 + \sum_{i=1}^{m} \sum_{j=i+1}^{m} \beta_{ij} x_i x_j + \epsilon \quad (1)$$

Where $\beta_0$, $\beta_i$, $\beta_{ii}$, and $\beta_{ij}$ represent regression coefficients; $x_i (i = 1,2,\ldots,m)$ are design variables, $\epsilon$ is the random error, $Y$ is the response and $m$ is the total number of design variables.
Design Expert V8.0.6.0 software was used for the regression graphical analysis of the results and also to obtain the coefficient regression equations. To analyses statistically the model, the Analysis of the Variance (ANOVA) was used. Further the results have been used for the prediction of the outputs for new conditions. The Fisher’s F-test (overall model significance), its probability p(F), determination coefficient $R^2$ (used to measure the fitness of regression model) were also studied by ANOVA [9].

1.2. Multiple response optimizations
In most of the modern technological situations, more than one response variable is pertinent to the success of an industrial process or system. In this study, the influence of sliding velocity, sliding velocity and weight percent reinforcement of nano TiO$_2$ on the specific wear rate and Co efficient of friction is investigated and further the optimal input parameter is investigated and further the optimal minimizing specific wear rate and minimizing co-efficient of friction. A desirability function based simultaneous optimization technique is used in this work [14].

2. Materials and Experimental Details
2.1. Materials
The materials used in present work are Aluminium fine powder with size range 130 – 180 micrometer, the purity 98% as base material bought from Loba Chemi, India. The reinforced material TiO$_2$ powder of size 5 nm, the purity 99.89 % bought from US Research Nano Materials Inc, USA.

2.2. Pin Specimen preparation
The Blending process is done by high energy planetary ball milling machine using Al powder with 4 wt%, 7 wt%, 10 wt% of TiO$_2$ nano powder. The Blended powder is compacted in a Compacting machine to attain 30mm height with applied pressure of 8.5MPa. The compacted specimens are sintered using sintering furnace for 90min at 580°C.

2.3. Wear test methodology
Wear test conducted using Pin on Disc tribometer on Al- TiO$_2$ P/M pin specimen as per ASTM G99 standard and recorded the output value of wear rates, frictional force, coefficient of friction based on the design variable mention in Table 1.

Table 1. Experimental factor and their levels

| Symbol | Parameter          | Unit | Coded values and the corresponding values of parameters |
|--------|--------------------|------|--------------------------------------------------------|
| A      | Load               | N    | 10 30 50                                               |
| B      | Sliding Velocity   | m/s  | 1.5 2.0 2.5                                            |
| C      | TiO$_2$ Weight     | %    | 4 7 10                                                  |

Sliding distance 1000 m

Specific wear rate (SWR) are calculated by wear volume per unit distance and load and Co efficient of friction (COF).

3. Results and Discussion
3.1. RSM Modeling
The experimental design and corresponding results are presented in Table 2. The following model in Eq.(2) and (3), were respectively developed model for the Specific wear rate and Co efficient of friction using regression coefficient summarized in Table 2. Following the calculation of the
regression coefficient, the model for prediction of Specific wear rate and Coefficient of friction was determined as follows:

\[
\text{SWR} = 2.11 \times 10^{-6} + 4.64 \times 10^{-7} A + 2.31 \times 10^{-6} B - 1.04 \times 10^{-6} C + 2.37 \times 10^{-7} A^2 + 9.68 \times 10^{-3} B^2 \\
- 4.94 \times 10^{-7} C^2 + 4.3 \times 10^{-3} AB - 3.39 \times 10^{-3} AC - 9.06 \times 10^{-3} BC \tag{2}
\]

\[
\text{COF} = 0.4989 + 0.0390 A + 0.1192 B - 0.0683 C + 0.0119 A^2 + 0.0257 B^2 \\
- 0.0199 C^2 + 0.0137 AB - 0.0055 AC - 0.0159 BC \tag{3}
\]

Where A, B and C are coded values pertaining to the temperature, strain rate and solid lubricant respectively.

The significance of each coefficient in the regression in Equation (2) and (3) are determined by t-values and p-values listed in Table 2. Higher t-value and lower p-value regarding a coefficient indicate that the coefficient is more significant. In general, any term having the value of p < 0.05 is significant.

Table 2
Analysis of the design results for Specific wear rate and Coefficient of friction

| Specific wear rate (SWR) | Coefficient of friction (COF) |
|--------------------------|------------------------------|
| Regression Coefficient  | t-value | p-value | Regression Coefficient  | t-value | p-value |
| Constant                 | 2.11E-06 | 8.091   | <0.0001 | 0.49898 | 24.775 | <0.0001 |
| A                        | 4.64E-07 | 3.822   | 0.0014 | 0.03903 | 4.165  | 0.0006 |
| B                        | 2.31E-06 | 19.923  | <0.0001 | 0.11924 | 13.344 | <0.0001 |
| C                        | -1.04E-06 | -8.589  | <0.0001 | -0.06837 | -7.295 | <0.0001 |
| A^2                      | 4.13E-07 | 2.998   | 0.0081 | 0.01372 | 1.290  | 0.2144 |
| B^2                      | -3.39E-07 | -2.285  | 0.0354 | -0.00550 | -0.481 | 0.6367 |
| C^2                      | -9.06E-07 | -6.572  | <0.0001 | -0.01597 | -1.501 | 0.1517 |
| AB                       | 2.37E-07 | 1.132   | 0.2734 | 0.01189 | 0.735  | 0.4723 |
| AC                       | 9.68E-07 | 5.339   | <0.0001 | 0.02571 | 1.838  | 0.0837 |
| BC                       | -4.94E-08 | -0.236  | 0.8165 | -0.01994 | -1.233 | 0.2343 |

Referring to the value of specific wear rate presented in Table 3, the sliding velocity and the weight percentage of reinforcement that have p-value less than 0.05 indicates that they are significance with respect to specific wear rate, the interaction among the load and weight percentage of reinforcement parameters was significant. In terms of coefficient of friction, sliding velocity and the weight percentage of reinforcement parameters affects considerably the coefficient of friction values. Regarding the variables interaction, no interaction among the parameters was significant for coefficient of friction as well.
Table 3. Response for Specific wear rate and Co efficient of friction observed experimental and predicted

| Exp. No | Load N | Sliding Velocity m/s | TiO2 Weight Fraction % | Experimental result | Predicted value |
|---------|--------|----------------------|------------------------|---------------------|-----------------|
|         |        |                      |                        | Specific wear rate m³/Nm | Coefficient of friction | Specific wear rate m³/Nm | Coefficient of friction |
| 1       | 10     | 2.0                  | 4                      | 2.30E-06            | 0.495            | 2.33E-06            | 0.534                |
| 2       | 30     | 2.5                  | 10                     | 3.47E-06            | 0.519            | 3.39E-06            | 0.561                |
| 3       | 30     | 2.5                  | 7                      | 4.97E-06            | 0.613            | 5.39E-06            | 0.629                |
| 4       | 30     | 1.5                  | 4                      | 1.01E-06            | 0.440            | 6.90E-07            | 0.448                |
| 5       | 50     | 1.5                  | 10                     | 7.25E-07            | 0.361            | 5.75E-07            | 0.351                |
| 6       | 50     | 2.5                  | 7                      | 6.91E-06            | 0.722            | 6.50E-06            | 0.667                |
| 7       | 30     | 2.5                  | 4                      | 6.90E-06            | 0.721            | 7.29E-06            | 0.696                |
| 8       | 50     | 2.0                  | 10                     | 1.46E-06            | 0.474            | 1.25E-06            | 0.475                |
| 9       | 30     | 2.0                  | 7                      | 2.10E-06            | 0.491            | 1.94E-06            | 0.505                |
| 10      | 30     | 2.0                  | 10                     | 1.55E-06            | 0.450            | 9.19E-07            | 0.437                |
| 11      | 10     | 1.5                  | 7                      | 8.74E-07            | 0.391            | 9.87E-07            | 0.343                |
| 12      | 50     | 1.5                  | 4                      | 1.16E-06            | 0.473            | 1.25E-06            | 0.486                |
| 13      | 50     | 2.5                  | 4                      | 9.77E-06            | 0.848            | 8.74E-06            | 0.734                |
| 14      | 50     | 2.0                  | 4                      | 3.12E-06            | 0.529            | 3.88E-06            | 0.610                |
| 15      | 50     | 2.5                  | 10                     | 3.39E-06            | 0.569            | 4.16E-06            | 0.599                |
| 16      | 10     | 2.5                  | 4                      | 6.37E-06            | 0.651            | 6.31E-06            | 0.658                |
| 17      | 10     | 2.0                  | 7                      | 1.94E-06            | 0.486            | 1.75E-06            | 0.467                |
| 18      | 50     | 1.5                  | 7                      | 1.01E-06            | 0.440            | 9.65E-07            | 0.419                |
| 19      | 30     | 2.0                  | 4                      | 2.40E-06            | 0.503            | 2.87E-06            | 0.572                |
| 20      | 10     | 1.5                  | 4                      | 9.32E-07            | 0.426            | 6.00E-07            | 0.410                |
| 21      | 10     | 2.0                  | 10                     | 1.35E-06            | 0.432            | 1.06E-06            | 0.399                |
| 22      | 10     | 1.5                  | 10                     | 5.52E-07            | 0.250            | 1.28E-06            | 0.275                |
| 23      | 10     | 2.5                  | 10                     | 3.29E-06            | 0.510            | 3.09E-06            | 0.523                |
| 24      | 30     | 1.5                  | 10                     | 6.24E-07            | 0.312            | 6.88E-07            | 0.313                |
| 25      | 30     | 1.5                  | 7                      | 8.87E-07            | 0.421            | 7.39E-07            | 0.381                |
| 26      | 50     | 2.0                  | 7                      | 2.40E-06            | 0.503            | 2.61E-06            | 0.543                |
| 27      | 10     | 2.5                  | 7                      | 4.54E-06            | 0.594            | 4.75E-06            | 0.591                |

The comparison of the results predicted by the model with those measured by experimental testing is presented in Fig.1 for SWR and COF. From the figure, it has been seen that most of the points are close to the center line and hence this empirical model with excellent agreement between this predicted and measured values implies the high accuracy of the model.
The results of the analysis of variance (ANOVA) have been shown in Table 4. ANOVA is applied to analyze statistically the performance of the models developed by RSM. The ANOVA, performed on the RSM regression, indicates that \( p \) is greater than \( F \) (\( p > F \)) demonstrating that the models are significant and there is only a 0.01\% chance that a model \( F \)-value this large could occur due to noise. Another way to check the accuracy of a model is normally by calculating the \( R^2 \) coefficient. The \( R^2 \) values were 0.9617 and 0.9374 for SWR and COF, respectively. This implies that about experimental results regarding the SWR and COF measurements are compatible with the ones predicted by the developed models. This again confirms the accuracy of models.
3.2. Contour plots

After verification of the model, the contour plots can be significant approaches to study the effect of interaction among the variables on SWR and COF of investigated AMCs. The 2D contour plots represent graphically for the response surface of SWR and COF the different interaction among load (A), sliding velocity (B) at different TiO$_2$ wt % reinforcement (C) are shown in Figure 2 and Figure 3.

From the Figure 2, it is clear that as specific wear rate increases with increase in sliding velocity and percentage of reinforcement. It is more prominent that the combination of load and the percentage of reinforcement increases, specific wear rate decreases.
Figure 2. Contour plots of the SWR with different interaction among load (A), sliding velocity (B) at different TiO₂ wt % reinforcement (C).

From the Figure 3, it is clear that as coefficient of friction increases with increase in sliding velocity and load. It is more significant that as the percentage of reinforcement increases, coefficient of friction decreases.

Figure 3. Contour plots of COF with different interaction among load (A), sliding velocity (B) at different TiO₂ wt % reinforcement (C)
3.3. Multiple response optimizations

The optimization analysis was carried out using Design-Expert software. The goal set, lower limits used, upper limits used, weights used, and importance of the factors given are presented in Table 5. In desirability-based approach, different best solutions were obtained.

| Name                              | Goal                  | Lower Limit | Upper Limit | Lower Weight | Upper Weight | Importance |
|-----------------------------------|-----------------------|-------------|-------------|--------------|--------------|------------|
| Load (N)                          | is in range           | 10          | 50          | 1            | 1            | 3          |
| Sliding Velocity (m/s)            | is in range           | 1.5         | 2.51.0      | 1            | 1            | 3          |
| TiO$_2$ Weight Fraction (wt %)    | is in range           | 4           | 10          | 1            | 1            | 3          |
| Specific wear rate (mm$^3$/Nm)    | minimize              | 5.52e$^{-7}$| 9.77e$^{-6}$| 1            | 1            | 3          |
| Coefficient of friction           | minimize              | 0.250       | 0.848       | 1            | 1            | 3          |

The solutions with high desirability are preferred. The best three solutions obtained for the multi-response optimization is presented in Table 6.

| Number | Load N | Sliding velocity m/s | TiO$_2$ weight percentage | Specific wear rate mm$^3$/Nm | Coefficient of friction | Desirability |
|--------|--------|----------------------|----------------------------|-----------------------------|-------------------------|--------------|
| 1      | 13.49  | 2.04                 | 10                         | 1.08E-06                    | 0.391                   | 0.849        |
| 2      | 13.75  | 2.04                 | 10                         | 1.07E-06                    | 0.391                   | 0.849        |
| 3      | 13.20  | 2.04                 | 10                         | 1.08E-06                    | 0.391                   | 0.849        |

From the Table 6, the developed models were used for multiple response optimizations by desirability function approach to obtain maximum strength coefficient and minimum strain hardening exponent. The desirability is found to be 0.849 as acceptable and excellent significant based on Harrington’s rating system[14]. The optimized Specific wear rate 1.08e$^{-6}$ mm$^3$/Nm and Coefficient of friction 0.3910 for load 13.49 N and sliding velocity 2.04 m/s , when the TiO$_2$ weight percentage 10(wt %) are Selected from Table 6

4. Conclusion

Response surface model and experimental design were used to model the tribological behaviour of Al-nano TiO$_2$ powder metallurgy composite response of AMCs and predict the optimum Wear behaviour, which is characterized by a significant effect of load, sliding velocity and reinforcement weight percentage condition. According to the analysis of variance (ANOVA), the accuracy of developed model conformed by R2 and p-value. The predicted and measured values have excellent agreement. Further, optimum combination of process parameters to attain the minimizing the specific wear rate and minimizing coefficient of friction can be predicted by the statistically developed models.
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