On the multi-parametric optimization of quality characteristics of the hybrid Al-6061 composites fabricated through powder metallurgy

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Abstract. Aluminium matrix composites (AMCs) have gained enormous attention of almost all types of manufacturing sectors owing to their excellent strength to weight ratio, ease of processing, good thermal conductivity, corrosion resistance, and desirable mechanical properties. Amongst the various processing routes available for AMCs, mechanical alloying assisted powder metallurgy (MA2PM) is particularly efficient in achieving the required physical, mechanical, and metallurgical characteristics. Therefore, in this present work, an effort has been made to develop the hybrid Al-6061 composites via MA2PM route under the influence of controllable and significant process variables. A specified proportion of graphite, silicon carbide (SiC), magnesium (Mg), zinc (Zn), and fly ash were selected and the process parameters (such as compaction pressure, sintering temperature, and sintering time) were varied as per Taguchi L9 orthogonal array. It has been found that the hardness and wear responses of the developed samples were significantly influenced by the processing parameters at 95% confidence level. Overall, the study conducted indicated that for hardness and wear rate only the compaction pressure found as significant parameter with 89.40 and 72.52% contribution.

1. Introduction

Nowadays, the requirement of high strength and wear resistant materials is increasing enormously in various manufacturing sectors for outstanding performances. Particularly in the last three decades, the development of technologically advanced materials has benefitted the aerospace, automobile, entertainment, process tools and biomedical industry [1, 2]. Various types of composites materials and functionally graded materials have taken all the loads of the essential requirements of the material systems as these offer good flexibilities enabling the materialistic modifications aiming at their end applications. The word “composite” generally defines a material system consisted of one or multiple discrete constituent(s) embedded in a continuous phase, either uniformly or non-uniformly, through physical interaction [3]. Amongst different, aluminium matrix composites (AMCs) have attracted the most attention especially in aircraft, aerospace, automobiles industries because of their low weight [4, 5]. AMCs present a category of metallic composites which possess low density, high stiffness and strength, excellent tribological properties, low thermal expansion, higher fatigue strength, and better geometrical stability at elevated temperature [6]. In automotive and aerospace industry, the use of AMCs has resulted in the reduction of overall weight, fuel consumption and pollution in the automobiles and aircrafts [7]. Silicon carbide (SiC) and aluminium oxide (Al2O3) particles are attractive materials as reinforcements for these applications [8]. However, on the down side, the use of reinforcements make the end part more heaving since the density of reinforcements are higher than the most of aluminum alloys [9]. Also, the presence of the reinforcements in higher concentration can turn the material system more harder which causes problems in the post processing of the products, for instance machining [6]. Therefore, it has been advised by numerous researchers to include multiple reinforcements in the matrix to produce a hybrid composite system. For this, the combination of the multiple reinforcements can be decided on the basis of their roles in the end product. For example, ceramic reinforcements (primary) can be used in combination with the others (secondary) to achieve a trade-off of hardness and ductility. The secondary reinforcements can be considered as
natural or waste materials such as fly ash, seal coal, coal ash, graphite, rice husk, etc. [10]. Moreover, the application of hybrid reinforcements enhances the quality characteristics of final composite parts and introduces novel features [10-12].

AMCs can be processed through various routes, majorly include liquid state, solid state, and vapour state processing [13], offering their own merits and demerits in terms of the processing easiness, involved expenditures, end properties, dimensional stability, and compatibility with selected material ingredients. In solid state processing, powder metallurgy (PM) route offers benefits in terms of control over the strong interfacial reaction, minimizing the undesired reactions between the matrix and the reinforcement, when compared with the other processes in the same class. Further, PM ensures the uniformity of the reinforcement which not only improves the structural properties but also the reproducibility level in the properties [14]. It is majorly used for the production of produce small spherical components, bolts, cams, gears, pistons, valves, surgical equipment, and high-strength/heat resistant materials [15]. The PM process involves the mixing of reinforcement and matrix powders, through mechanical alloying, followed by mechanical compaction and sintering [16]. In [17], authors produced fly ash reinforced AMCs through PM route and found that this production method is highly suitable for fabricating composites with high degree of dispersion and can overcome the problems existing in liquid based processing methods. Moreover, it has also highlighted in the literature that the level of mechanical properties, porosity, metallurgical and chemical properties of the resulting composites can be easily tailored through the process variable [18-20].

In the present research work, an attempt has been made to develop hybrid AMCs through mechanical alloying assisted powder metallurgy (MA2PM) route and to investigate the effect of input process variable on the obtained mechanical and metallurgical properties of the same. Taguchi L9 based design of experimentation (DoE) method has been employed for planning the experimental runs and further to optimize the response variable. Moreover, multi-parametric optimization method has been used to find out a unique setting of input variables at which the selected responses can be enhanced.

2. Experimentation

In this work aluminium (Al-6061; purity ~ 99%, mesh size = 400; source: Swastik Metals Pvt. Ltd., Kokar) has been selected as the matrix material (refer Figure 2a for microscopical view) and a total of five different reinforcements (such as fly ash, silicon carbide, magnesium, zinc and graphite) have been considered for the fabrication of AMCs through MA2PM route, refer Figure 1. The proportions of the various ingredients has been judicially selected, refer Table 1, and the process parameters (such as compaction pressure, sintering temperature, and sintering time) have been varied in accordance of DoE. The fly ash (mesh size = 200) was collected from the thermal power plant, Goindwal Sahib, Punjab (refer Figure 2b for microscopical view). The elemental composition of fly ash was investigated by X-ray diffraction (XRD) analysis and is given in Table 2. However, all the other reinforcements (mesh size ~ 250) were locally purchased (source: Shiva Chemicals, Ludhiana). The complete control log of experimentation is given in Table 2. Each experiment has been repeated three times to overcome the possible experimental errors.

Various ingredients, in powder form, were first mixed by using mechanical alloying method using ball milling (make: Rathpon Engineering) for about 2hrs at 250rpm in order to achieve a uniform dispersion of different compounds. The particular patterns used to mix the powder materials according to the behaviour of the materials to get the better results. And, the powders were heated before mixing and after mixing at 200°C for about 60 minutes to remove the moisture content and volatile impurities. After this, the prepared samples were compacted by using a hydraulic compression moulding machine (source: Lovely Professional University, Punjab).
High speed alloy steel, die steel and tool steel used to make the parts of compaction die and various samples of 50mm diameter and 7mm height were prepared. In the last, sintering of the resulting green samples was carried out in a muffle furnace by using powder silicon dioxide and flux by layers to prevent from the oxidation. The silica present around the sample also helped in the uniform distribution of heat and resulted in better quality material with more effective mechanical and elastic properties. The finally prepared samples are shown in Figure 3. The wire cutting (CNC EDM wire cut – ultra cut S2 by “ELECTRONICA”) machine at CIPET, Amritsar was used for the preparation of the test specimens.

Table 1: Proportion of different ingredients of AMCs.

| Ingredient of the composite | Al | SiC | Mg | Graphite | Zn | Fly-ash | P.F. (CNSL) |
|-----------------------------|----|-----|----|----------|----|---------|-------------|
| Proportion (%vol.)          | 74 | 5   | 5  | 5        | 5  | 6       | 3% (of total mixture) |

Table 2: XRD result for fly-ash.

| Compound           | Percentage |
|--------------------|------------|
| Alumina            | 28.98%     |
| Silicon dioxide    | 53.96%     |
| Calcium oxide      | 0.20%      |
| Carbon             | 1.06%      |

Figure 1. Schematic description of MA2PM processing for the composites.

Figure 2. Microscopic view of Al-6061 (a) and fly-ash (b).
3. Results and Discussion

In the present research work, hardness and tribological testing have been carried out in order to investigate the effect of input process parameters. Rockwell hardness testing machine used to check the hardness of samples at B scale with 981N load and 1/16" steel ball indenter (source: Dr. BR Ambedkar National Institute of Technology, Jalandhar). Furthermore, pin-on-disc type tribological apparatus (source: Dr. BR Ambedkar National Institute of Technology, Jalandhar) has been used to record the wear resistant properties, of the developed AMCs, at 98.10N load, disc speed of 500rpm and running time 10min.

Table 3: Control log of experimentation.

| Exp. No. | Compaction Pressure (KN) | Sintering Temperature (°C) | Sintering Time (hrs) |
|----------|--------------------------|-----------------------------|----------------------|
| 1        | 600                      | 630                         | 3                    |
| 2        | 600                      | 660                         | 4.5                  |
| 3        | 600                      | 690                         | 6                    |
| 4        | 700                      | 630                         | 4.5                  |
| 5        | 700                      | 660                         | 6                    |
| 6        | 700                      | 690                         | 3                    |
| 7        | 800                      | 630                         | 6                    |
| 8        | 800                      | 660                         | 3                    |
| 9        | 800                      | 690                         | 4.5                  |

Table 4: Obtained results and S/N ratios.

| Exp. No. | Brinell hardness (B Scale) | S/N Ratio (dB) | Wear rate (mm³/10min) | S/N/ Ratio (dB) |
|----------|-----------------------------|-----------------|------------------------|-----------------|
| 1        | 62                          | 35.8478         | 1.93                   | -5.7111         |
| 2        | 70                          | 36.9020         | 1.61                   | -4.1365         |
| 3        | 66                          | 36.3909         | 1.46                   | -3.2871         |
| 4        | 68                          | 36.6502         | 0.83                   | 1.6184          |
| 5        | 74                          | 37.3846         | 0.45                   | 6.9357          |
| 6        | 72                          | 37.1466         | 0.31                   | 10.1728         |
| 7        | 73                          | 38.3816         | 0.78                   | 2.1581          |
| 8        | 88                          | 38.8897         | 0.26                   | 11.7005         |
| 9        | 86                          | 38.6900         | 0.21                   | 13.5556         |

Table 4 shows the observed results of hardness and wear resistance. MINITAB-17, a commercial statistical software package, has been used for the analysis of the obtained raw data. Signal to noise (S/N) has calculated and thereafter to draw the same in graphical format in order to understand the effect of input parameters, refer Figure 4.

Figure 3. Finally prepared AMC sample.

Overall Average S/N ratio, m = 37.36, 3.66
From Figure 4a, it has been found that the hardness of the test specimens was increased by increasing the compaction pressure and is found to be maximum at 800KN. This trend is mainly because of the fact that when the compaction pressure increased the density of the developed AMCs was also increased and helped in attaining higher levels of the hardness. Apart from this, it has also noticed that the higher levels of the compaction pressure resulted in the better dimensional accuracy of the prepared specimens. Further, in case of sintering temperature, it has been found that the maximum hardness value was obtained at 660°C and is found to be the exact temperature for the solid state processing of the Al-6061 composites. Other than this level are comparatively ineffective. Similarly, the third parameter, sintering time, is found to be insignificant for the obtained hardness of the AMCs. It can be clearly seen from Figure 4a that at all three levels of sintering time, the steepness of the curve is almost flat indicating its negligible contribution in the obtained composites' hardness. In case of wear rate, from Figure 4b, it has been found that with an increase in the compaction pressure the wear rate of the developed composites has been reduced. The reason behind this trend is similar to the hardness, as aforementioned. However for better illustration, schematic (Figure 5) has been drawn sowing the effect of density over the wear resistant property of the developed AMC.

Further in case of sintering temperature, it has been found that the wear resistance of the composites was reduced at elevated sintering temperatures. This is because of the fact that at higher sintering temperatures, the wettability of the composite matrix and its reinforcing elements was enhanced and resulted in stronger interfaces. In case of sintering time, it has been found that lesser sintering durations produced good wear resistive properties of the composites. This might be due to the fact that prolonged sintering time starts to decompose material system and thereby the tribological properties. The optimized process parametric levels for hardness are compaction pressure – 800KN, sintering temperature - 660°C and sintering time – 4.5hrs. Similarly, in case of wear resistance, optimized process parameters are compaction pressure – 800KN, sintering temperature - 690°C and sintering time – 3hrs.
3.1. Analysis of Variance

Analysis of Variance (ANOVA) has been performed in order to find out the statistical significance of the selected process variable on the output responses. All the obtained tabular values presented below are obtained by processing the raw experimental data through Minitab software. Table 5 shows the ANOVA results for the hardness and wear rate of the composites. It has been found that in case of hardness, only compaction pressure has contributed, significantly, at 95% confidence level (error = 0.05). Similarly, for wear rate too, compaction pressure has been found as statistically significant parameter at 95% confidence level (error = 0.05). The same parameter has also observed as the highly contributing variable i.e. 89.40% and 72.52% respectively. The residual error in both the cases has been recorded as 0.62% and 0.91%, respectively, therefore justifying that the experimental error involved is minimum and the obtained raw values are highly reliable.

Table 5: ANOVA results for hardness and wear resistance.

| Source                  | Degree of freedom (DoF) | Sum of square (V) | Frisher’s (F) value | Contribution (%) | Probability (P) |
|-------------------------|-------------------------|-------------------|---------------------|-----------------|-----------------|
| **Hardness**            |                         |                   |                     |                 |                 |
| Compaction pressure (KN)| 2                       | 8.1699            | 4.0850              | 144.94          | 89.40           | 0.007*          |
| Sintering temperature (°C) | 2                       | 0.8879            | 0.4439              | 15.75           | 9.71            | 0.060           |
| Sintering time (hrs)     | 2                       | 0.02332           | 0.0116              | 041             | 0.25            | 0.707           |
| Errors                  | 2                       | 0.05637           | 0.0281              |                 | 0.62            |                 |
| Total                   | 8                       | 9.1376            |                     |                 | 100             |                 |
| **Wear Rate**           |                         |                   |                     |                 |                 |
| Compaction pressure (KN)| 2                       | 303.873           | 151.936             | 39.47           | 72.52           | 0.025*          |
| Sintering temperature (°C) | 2                       | 89.564            | 44.782              | 11.63           | 21.37           | 0.079           |
| Sintering time (hrs)     | 2                       | 17.873            | 8.936               | 2.32            | 4.26            | 0.301           |
| Errors                  | 2                       | 7.700             | 3.850               |                 | 0.91            |                 |
| Total                   | 8                       | 419.009           |                     |                 | 100             |                 |

Note : *indicating the statistically significant parameters

Table 6 shows the response of S/N ratio, for hardness and wear rate, which were used for the prediction of the output responses at the optimized settings by using standardized mathematical equations such as 3.1 for calculating $\eta_{opt}$, 3.2 for optimized hardness, and 3.3 for optimized wear rate:

$$\eta_{opt} = x+(xA_2-x)+(xB_1-x)+(xC_3-x)$$  \hspace{1cm} (3.1)

$$y_{opt}^2 = 10^{\eta_{opt}/10}$$  \hspace{1cm} (3.2)

$$y_{opt}^2 = 10^{\eta_{opt}/10}$$  \hspace{1cm} (3.3)
Table 6: Response table for hardness and wear rate.

| Level | Compaction Pressure (KN) | Sintering Temperature (°C) | Sintering Time (hrs) |
|-------|--------------------------|---------------------------|---------------------|
|       |                          |                           |                     |
| 1     | 36.38                    | 36.96                     | 37.29               |
| 2     | 37.06                    | 37.73*                    | 37.41*              |
| 3     | 38.65*                   | 37.41                     | 37.39               |
| Delta | 2.27                     | 0.77                      | 0.12                |
| Rank  | 1                        | 2                         | 3                   |

Wear rate

| Level | S/N ratio: wear rate (dB) |
|-------|---------------------------|
| 1     | -4.3782                   |
| 2     | 6.2423                    |
| 3     | 9.1381*                   |
| Delta | 13.5163                   |
| Rank  | 1                        |

Note: *indicating maximum levels of S/N ratio for selected input variables.

The optimized values of the hardness and wear rate are calculated by using the maximum responses of Table 6 in equation 3.1 and 3.2, respectively.

For hardness, optimized predicted S/N ratio = 37.36 + (38.65 - 37.36) + (37.73 - 37.36) + (37.41 - 37.36) = 39.07

\[ y_{opt}^2 = 10^{39.07/10} \]

\[ y_{opt} = 89.84 \text{BHN} \]

For wear rate, optimized predicted S/N ratio = 3.66 + (9.1381 - 3.66) + (6.138 - 3.66) + (5.3874 - 3.66) = 13.34

\[ y_{opt}^2 = 10^{13.34/10} \]

\[ y_{opt} = 0.046 \text{mm}^3/10 \text{min} \]

In order to confirm the optimized setting, experiment has been conducted to verify the prediction. Therefore, three runs have been conducted which resulted into average value of hardness and wear rate as 89.50BHN and 0.048mm\(^3/10\text{min}\), respectively. The obtained results validated the accuracy of the predicted optimized results.

3.2. Multi-parametric optimization

Further multi-parametric optimization has also been performed to find out a unique setting of the input parameters at which both output responses can be trade-off. Here also, Minitab 17 software package has been used to calculate the composite desirability index. S/N responses for the hardness and wear rate were employed in this and weightage of „1‟ was allocated during their maximization. Table 7 shows the outcomes of the statistical run indicating composite desirability as 0.9754. This indicated the goodness of the multi-parametric optimization as both output responses consisted on minimum interferences while statistical evaluation. Further, Figure 6 showed the parametric responses during multi-optimization.

Table 7: Statistical results of multi-parametric optimization.

| Compaction pressure (KN) | Sintering temperature (°C) | Sintering time (hrs) | S/N ratio: wear rate (dB) | Wear rate (mm\(^3/10\text{mm}\)) | S/N ratio: hardness (dB) | Hardness (BHN) | Composite desirability |
|--------------------------|---------------------------|---------------------|--------------------------|-------------------------------|--------------------------|-----------------|------------------------|
| 800                      | 690                       | 5.0208              | 13.55                    | 0.043                         | 38.74                    | 88.45           | 0.9754                 |
4. Conclusions

In the present research work, hybrid AMCs has been successfully developed through MA2PM route. The fabricated components were tested for the hardness and wear resistance properties in order to establish their relationships with process variables through the use of DoE method and statistical analysis. It has been found that the hardness as well as wear resistance properties of the developed AMCs have been strongly affected by the compaction pressure and are increased by increasing the level of compaction. The observed optimized process parameters for the hardness and wear resistance are also recorded by using S/N ratio plots. ANOVA indicated that the percentage contribution of the compaction pressure for hardness and wear rate is 89.40 and 72.52%. Multi-parametric optimization has also showed negligible interferences for the enhancements of the results. Further, the predicted responses are verified through the confirmatory experiments which indicated a negligible magnitude of the error. The upcoming efforts are aimed at the microscopical study of the developed specimens for the better understanding of the metallurgical behaviours of the specimens.

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