Optimizing the effect of reinforcement, particle size and aging on impact strength for Al 6061-red mud composite using Taguchi technique

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Abstract. Aluminium 6061-red mud composite has been successfully casted by using stir casting. Fairly uniform distribution of red mud reinforcement has been observed by SEM micrographs. The presence of red mud particles inside the matrix has been confirmed by energy dispersive spectroscopy. Impact testing has been done on impact testing machine with a range of 1–300 J (Charpy), 1–175 (Izod) and least count of 0.5 J. Impact strength shows a decreasing trend with increase in percentage reinforcement, aging time and is increased with increase in particle size. ANOVA analysis found that the effect of particle size and aging time are significant but the effect of percentage reinforcement has not been found significant. From the analysis, it can be observed that the particle size influences the impact strength most followed by aging time and percentage reinforcement. Optimum value of impact strength has been predicted by using Taguchi technique and confirmed experimentally by confirmation experiment results.

Keywords. Metal matrix composite; red-mud; aluminium 6061; impact strength; ANOVA.

1. Introduction

Aluminium and its alloys reinforced with hard ceramic particulates, called as aluminium matrix composites (AMCs). Normal aluminium alloys failed to meet the increasing demand for modern heavy performance applications. AMCs have improved properties like stiffness, strength, resistance to wear, higher strength-to-weight ratio, low coefficient of thermal expansion, tensile strength, fatigue strength, makes them preferable over aluminium alloys. Application of advanced composite materials in structural components is attractive due to their good stiffness/strength-to-weight ratios. Reinforcement type, fabrication method affects the properties of AMCs. AMCs emerged as one of the most useful materials for aeronautical, automobile, defence, and some other areas of application [1–10]. Mostly hard ceramic particles have been used as reinforcements and commonly used reinforcements in AMCs are SiC, TiB₂, B₄C, BN, AlN, Al₂O₃, TiO₂, CNT, SiO₂ etc. Waste materials such as rice husk ash, fly ash, bamboo leaf ash, bagasse, etc. are also used as reinforcement in AMCs. Reinforcement materials used may be of a single material or a combination of two or more materials [11–15]. There have been

many fabrication methods for producing AMCs. The methods used for production of AMCs can be classified as solid state, liquid state, and gas state. Powder metallurgy and diffusion bonding has been most used solid state methods. Liquid state methods include, squeeze casting, compo casting, in situ, spray co-deposition, stir casting, etc. Gaseous state method used physical vapour deposition. Out of these available methods, stir casting has been most simple economical and flexible. In this method, particles of reinforcement have been added to matrix by mechanical stirring [16–22]. Design of experiment (DOE) techniques such as Taguchi, response surface methodology, etc. has gained importance in recent times for finding influence of parameters, their combined effects and order of significance of their effect [23]. Taguchi method uses orthogonal array for reducing counting of experimental runs for optimizing considered parameters and their interactions. In Taguchi technique, signal-to noise (S/N) ratio represents performance characteristic and highest S/N ratio value has been considered. By considering objective of the experiment, anyone of the S/N ratios, called lower-the-better, larger-the-better and nominal-the-better has been chosen for further analysis [24].

It has been observed from the literature that the impact strength of aluminum matrix composite has not been much

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investigated for ceramic reinforcements. Authors have also not found much on the effect of particle size on the impact strength of composite under study in literature. Therefore, the present study has been aimed to the investigation of effect of parameters on impact strength of Al 6061/red-mud composite. The composite has been fabricated by stir casting method. A statistical analysis of parameters affecting impact strength has been carried out using Taguchi technique. Analysis of variance (ANOVA) has been applied to find out contribution of effect of each parameter on impact strength of the developed composite.

2. Experimental procedure

2.1 Material preparation

Aluminium 6061 has been selected as matrix material. Red-mud a waste produced during extraction of aluminium from bauxite by Bayer's process has been taken as reinforcement material and is procured from HINDALCO, Renukut, Uttar Pradesh, India. XRF analysis of red mud used for experimentation has been given in table 1.

Table 1. XRF result of red mud used in this study

Sr. no.	Constituent	Percentage
1	Fe ₂ O ₃	35.26
2	Al ₂ O ₃	21.89
3	TiO ₂	15.11
4	SiO ₂	12.46
5	Na ₂ O	11.82
6	CaO	1.83
7	P ₂ O ₅	0.40
8	V ₂ O ₅	0.38
9	SO ₂	0.19
10	ZrO ₂	0.15
11	K ₂ O	0.13
12	MgO	0.12
13	Cr ₂ O ₃	0.11
14	Cl	0.04
15	MnO	0.04
16	CuO	0.02
17	Sc ₂ O ₃	0.01
18	Ga ₂ O ₃	0.01
19	Nb ₂ O ₅	0.01
20	ZnO	65PPM
21	Ag	56PPM
22	PbO	47PPM
23	As ₂ O ₃	45PPM
24	NiO	44PPM
25	SrO	43PPM
26	Y ₂ O ₃	22PPM

Experiments have been planned using Taguchi design of experiments. According to chosen variable parameters and their variation levels L25 Orthogonal Array (OA) has been selected for experimentation. The variable parameters with their variation levels have been given in table 2.

2.2 Fabrication of composite

The composite under study has been fabricated by stir casting. Aluminium 6061 has been melted inside graphite crucible to a temperature of 800°C using muffle furnace. Vortex of molten metal has been obtained by stirring. Stirring has been done by mechanical stirrer (figure 1) at 400 RPM. Figure 1 explains the design of mechanical stirrer used for mixing of reinforcement during the fabrication process.

Reinforcement pre-heated at 400°C has been poured to vortex ensuring homogeneous distribution of red-mud. Magnesium has been added to improve the wettability of red-mud particles with molten aluminium 6061. Magnesium has been added in the form of magnesium turning by wrapping it into aluminum foil before the addition of red-mud particles. The melt has been poured into pre-heated metallic moulds. Solutionizing of fabricated composite has been done for homogenizing the structure and properties. In this process, the composite has been heated at 525°C for eight hours in a furnace. After heating, the composite has been quenched in water. Aging of the prepared samples has been done in electric muffle furnace. Aging temperature of the samples has been 175 ± 5°C. The SEM images of casted, homogenised and aged samples have been shown in figure 2a–c. It can be observed from the figures that homogenizing and aging improves surface of composite and reduce cracks.

Aging time durations taken are: 0.5, 6, 12, 18 and 24 h. Energy dispersive spectroscopy (EDS) of fabricated samples confirms the presence of constituents of red mud inside the aluminium 6061 matrix. Spectrum of EDS has been presented in figure 3 and is given below.

Table 2. Selected parameters and corresponding levels

Sr. no.	Parameters	Level-1	Level-2	Level-3	Level-4	Level-5
1	Percentage reinforcement (by wt)	4	8	12	16	20
2	Particle size (µm)	250	177	149	125	74
3	Ageing time (h)	½ h	6	12	18	24

Figure 1. Stirrer used for casting of aluminium 6061 red-mud composite and stirrer blades.

2.3 Impact test

Impact testing has been done on impact testing machine with a range of 1–300 J (Charpy), 1–175 (Izod) and least count of 0.5 J. Testing has been carried out at atmospheric condition of $25 \pm 3^\circ\text{C}$ temperature and relative humidity of 40–60%. A repetition of two has been done for each kind to reduce errors.

2.4 Scanning electron microscopy (SEM)

SEM images of fabricated composites have been taken after casting and after impact test. Casted samples have been machined and polished before SEM. Surface of SEM samples prepared by rubbing with emery papers of grit sizes 60, 150, 220, 400, 600, 1000, and after that it is polished with high concentration diamond paste with $1\ \mu\text{m}$ particle size on polishing cloth. SEM images of fractured surface after impact test has also been taken at different magnifications shown in each image. Sample thickness of 5 mm has been taken for each sample.

3. Results and discussion

3.1 Microstructure

From SEM images for the samples after casting shown in figure 4a–e, clearly visible particles inside the aluminium

6061 matrix are seen. It has also been observed from the images that the distribution of particles inside the matrix is fairly uniform.

SEM images in figure 5 reveal that fracture is of brittle nature in the region of reinforcement and is of ductile in rest of matrix phase. Figure 5a and d show the low magnification image of impact fracture and observed that cracks are propagated near the red-mud particles. The $100\times$ magnification images (figure 5b and e) show the red-mud particles on the fractured surface. Higher magnification images of fractured surface at $200\times$ i.e., figure 5c and f indicate that fracture is partially ductile and partially brittle in nature. The granular surface indicates brittle nature of fracture and the rough surface indicates ductile nature of the fracture. It has also been observed from figure 5c and f that higher particle size (figure 5f) shows more ductile nature than the smaller particle size (figure 5c) and the same is also reflected from table 3 experiment number 21 (impact strength = 8.50 J) has more impact strength than experiment number 18 (impact strength = 8.00 J).

3.2 Impact strength

Effects of variables on impact strength have been studied at five levels. Orthogonal array L_{25} has been selected for conducting the experiments. Total number of fifty experiments have been conducted with a repetition of twice. The

Figure 2. SEM image of fabricated samples in conditions: as cast, homogenized and after aging. (a) SEM image of casting without heat treatment. (b) SEM image of homogenized heat treated sample. (c) SEM image of sample after aging.

experimentally obtained results have been reported in table 3. There are three types of quality characteristics in Taguchi method which are as follows: smaller is better, nominal is

Figure 3. EDS spectra taken from red-mud particle.

better and larger is better. Larger is better quality characteristics have been used as higher impact strength is desired.

Tables 4 and 5 report the mean values of S/N ratios and mean of mean for impact strength of the fabricated composite at each level. The difference between the highest and lowest value for each variable has been called delta value. The delta analysis observed that the particle size affects the impact strength most (rank 1) followed by aging time (rank 2) and percentage reinforcement (rank 3). The plot of mean values of impact strength at each level has also been shown in figure 6.

3.2a ANOVA analysis: Analysis of variance (ANOVA) of Impact strength has been done using Minitab 17 software. In this analysis sum of squares, variance, F-ratio and *P* value has been calculated as shown in following table 6. Percentage contribution of effect of percentage reinforcement on impact strength is least (2.29%), for aging time it is (10.77%) and for particle size (79.12%) is highest. For a parameter to called significant *P* value should be less than 0.05. According to *P* value effect of particle size and aging time has been found to be significant while the effect of percentage reinforcement has not found to be significant.

Figure 4. (a) Secondary electron (SEC) and backscattered (BEC) image of casted samples of aluminium red-mud composite at 4% red-mud: (i) red-mud 4% SED. (ii) red-mud 4% BEC. (b) Secondary electron (SEC) and backscattered (BEC) image of casted samples of aluminium red-mud composite at 8% red-mud: (i) red-mud 8% SED. (ii) red-mud 8% BEC. (c) Secondary electron (SEC) and backscattered (BEC) image of casted samples of aluminium red-mud composite at 12% red-mud: (i) red-mud 12% SED. (ii) red-mud 12% BEC. (d) Secondary electron (SEC) and backscattered (BEC) image of casted samples of aluminium red-mud composite at 16% red-mud: (i) red-mud 16% SED. (ii) red-mud 16% BEC. (e) Secondary electron (SEC) and backscattered (BEC) image of casted samples of aluminium red-mud composite at 20% red-mud: (i) red-mud 20% SED. (ii) red-mud 20% BEC.

Figure 4. continued

The delta analysis of mean values of experimental results in table 5 also found the same results as obtained by ANOVA analysis i.e., particle size (79.12%, rank-1) has been most influencing parameter followed by aging time (10.77%, rank-2) and percentage reinforcement of red-mud (2.29%, rank-3).

3.2b Estimation of optimum impact strength: Under this part, optimum value of impact strength and its confidence interval is predicted theoretically using Taguchi's approach. By recognising the effect of factors, the optimum value of impact strength has been predicted. The significant process parameters for impact strength are B_5 and C_1 . The mean value of calculated impact strength is [25]:

$$\mu_J = B_5 + C_1 - T_J \quad (1)$$

where T_J is average of all values of impact strength 7.40 J; B_5 average value of impact strength at fifth level of particle size = 10.60 J; C_1 average value of impact strength at first level of aging time: 8.60 J; $\mu_J = 11.8$ J

Predicted value of confidence interval

$$CI = \sqrt{F_{\alpha}(1, f_e) V_e \left[\frac{1}{n_{eff}} + \frac{1}{R} \right]} \quad (2)$$

where CI, confidence interval; $F_{\alpha}(1, f_e)$, the F-ratio at a confidence level of $(1 - \alpha)$ against DOF = and error DOF (f_e); V_e , variance of error; R , sample size for confirmation experiment; n_{eff} , effective number of replications = $N / [1 + \text{total DOF in the estimation of mean}]$; N , total number of results = 50

Using the following values (table 6):

$$V_e = 0.6641$$

Total degree of freedom in estimation of mean = 8

$$n_{eff} = 8.33$$

$$F_{0.05}(1, 8) = 5.32$$

The confidence interval = 2.19; The CI of predicted impact strength at 95% confidence interval is = 11.8 ± 2.19 J

The optimum value of process parameters for the predicted range of impact strength are:

Reinforcement: 4%

Particle size: 250 μ

Aging time: 0.5 h

3.2c Confirmation experiments: On the basis of conclusions drawn based on Taguchi's method, the predicted results are verified. As the predicted optimum parameters of experiment number 1 of table 3 there has been no need to do confirmation experiments. The results of experiment number 1 are near to the predicted value.

3.2d Effect of selected parameters on impact strength: The effect on impact strength by affecting variables has been shown in figure 6. The impact strength shows a decreasing trend with increase in amount of reinforcement as in case with Ozden *et al* [26]. This effect may be due to the presence of hard reinforcement particles reduced toughness. With increase in particle size of the reinforcement, value of impact strength increases [26]. It may be due to larger particle size improves ductility by reducing the surface area of reinforcement. Also the larger size particles increases void space which results in improvement ability to allow more dislocation hence increasing shock absorbing capacity. This trend also visible from fracture surfaces shown in figure 5c and f of experiment no. 18 having impact strength = 8 J, and experiment no. 21 having impact strength of 8.5 J. Figure 5f shows more ductile zone than the figure 5c i.e., experiment number 21 has more ductile fracture area than experiment no. 18. Also, it can be observed from experiment number 21 and experiment number 18 that despite an increase in percentage reinforcement from 16 to 20% and aging time 24 h (which have a negative effect on impact strength), impact strength still increases. This shows that the effect of particle size has been more significant than percentage reinforcement and aging time. When the aging time of the fabricated composite increased it gives a negative effect

Figure 5. SEM image of fractured surface after impact testing at different heat treatment, percentage and particle size of reinforcement, and magnification. (a) Homogenized, 16%, 149 μm, ×50. (b) Homogenized, 16%, 149 μm, ×100. (c) Homogenized, 16%, 149 μm, ×200. (d) Aged 24 h, 20%, 250 μm, ×50. (e) Aged 24 h, 20%, 250 μm, ×100. (f) Aged 24 h, 20%, 250 μm, ×200.

Table 3. Impact strength results with S/N ratios

Experiment no.	Percentage reinforcement (by wt)	Particle size (μm)	Aging time (h)	Impact strength (J)	S/N ratio
1	4	250	0.5	14.0	22.9226
2	4	177	6	7.00	16.9020
3	4	149	12	7.25	17.2068
4	4	74	18	6.00	15.5630
5	4	125	24	5.50	14.8073
6	8	250	6	10.50	20.4238
7	8	177	12	7.50	17.5012
8	8	149	18	6.75	16.5861
9	8	74	24	6.25	15.9176
10	8	125	0.5	6.50	16.2583
11	12	250	12	10.00	20.0000
12	12	177	18	7.25	17.2068
13	12	149	24	6.00	15.5630
14	12	74	0.5	6.50	16.2583
15	12	125	6	7.00	16.9020
16	16	250	18	10.0	20.0000
17	16	177	24	7.00	16.9020
18	16	149	0.5	8.00	18.0618
19	16	74	6	5.50	14.8073
20	16	125	12	6.00	15.5630
21	20	250	24	8.50	18.5884
22	20	177	0.5	8.00	18.0618
23	20	149	6	7.50	17.5012
24	20	74	12	5.00	13.9794
25	20	125	18	5.50	14.8073

Table 4. Response table for signal to noise ratios

Level	Percentage reinforcement	Particle size	Aging time
1	17.48	15.31	18.31
2	17.34	15.67	17.31
3	17.19	16.98	16.85
4	17.07	17.31	16.83
5	16.59	20.39	16.36
Delta	0.89	5.08	1.96
Rank	3	1	2

Table 5. Response table for means

Level	Percentage reinforcement	Particle size	Aging time
1	7.95	5.85	8.60
2	7.50	6.10	7.50
3	7.35	7.10	7.15
4	7.30	7.35	7.10
5	6.90	10.6	6.65
Delta	1.05	4.75	1.95
Rank	3	1	2

on impact strength [15]. This reduction in impact strength may be due to hardening of matrix because of aging. This increase in hardness reduces toughness that results in

Figure 6. Mean values of impact strength.

reduction of impact strength. ANOVA analysis reports that the effect of particle size and aging time has been significant whereas percentage reinforcement does not have a significant effect. The significance of particle size has been highest and of percentage reinforcement has been least.

Table 6. ANOVA analysis for impact strength

Source	Degree of freedom	Sum of squares	Variance	F ratio	P value	Percentage contribution
Percentage reinforcement	4	2.335	0.5838	0.88	0.505	02.29
Particle size	4	80.661	20.1652	30.36	0.000	79.12
Aging time	4	10.981	2.7453	4.13	0.025	10.77
Residual error	12	7.970	0.6641			07.81
Total	24	101.947				

4. Conclusions

On the basis of experimental work and analysis carried out, the following conclusions can be drawn.

1. Composite has been successfully casted by using stir casting. Fairly uniform distribution of red mud reinforcement has been observed by SEM micrographs.
2. Impact strength shows a decreasing trend with increase in percentage reinforcement, aging time and is increased with increase in particle size.
3. ANOVA analysis found that the effect of particle size (79.12%) and aging time (10.77%) are significant but the effect of percentage reinforcement (2.29%) has not been found significant. Particle size has been influencing the impact strength mostly followed by aging time and percentage reinforcement.
4. Optimum value of impact strength (11.8 ± 2.19) has been predicted by using Taguchi technique and the same has been confirmed experimentally.

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