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Fabrication process optimization for improved mechanical properties of Al 7075/SiC$_p$ metal matrix composites

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A B S T R A C T

Two sets of nine different silicon carbide particulate (SiC$_P$) reinforced Al 7075 Metal Matrix Composites (MMCs) were fabricated using liquid metallurgy stir casting process. Mean particle size and weight percentage of the reinforcement were varied according to Taguchi L$_9$ Design of Experiments (DOE). One set of the cast composites were then heat treated to T6 condition. Optical micrographs of the MMCs reveal consistent dispersion of reinforcements in the matrix phase. Mechanical properties were determined for both as-cast and heat treated MMCs for comparison of the experimental results. Linear regression models were developed for mechanical properties of the heat treated MMCs using list square method of regression analysis. The fabrication process parameters were then optimized using Taguchi based grey relational analysis for the multiple mechanical properties of the heat treated MMCs. The largest value of mean grey relational grade was obtained for the composite with mean particle size 6.18 µm and 25 weight % of reinforcement. The optimal combination of process parameters were then verified through confirmation experiments, which resulted 42% of improvement in the grey relational grade. Finally, the percentage of contribution of each process parameter on the multiple performance characteristics was calculated through Analysis of Variance (ANOVA).

1. Introduction

MMCs are broadly utilized as a part of airplane innovation, automobiles and electronics engineering due to their excellent physical, mechanical and development properties (Kaczmar et al. 2000). High specific stiffness and strength, better high temperature properties and thermal capacity; and low thermal expansion coefficient make the Al-based MMCs suitable for application in aerospace, marine, automotive drive shaft fins, explosion engine components, heat sinks, solar panels and antenna reflectors etc. (Alaneme & Aluko 2012; Das et al., 2014; Das et al., 2015; Mishra et al., 2015).

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Hardness of SiC_p reinforced Aluminium Matrix Composites (AMCs) is improved by heat treatment (Rao et al., 2010). T6 condition of heat treatment or aging treatment improves the ultimate tensile strength, flexural strength and fracture toughness of SiC_p reinforced AMCs (Kalkanli & Yilmaz 2008; Alaneme & Aluko 2012). Hardness of AMCs is increased with an increase in reinforcement percentage (Sahin & Murphy, 1996; Rao et al., 2010; Veeresh Kumar et al., 2012), but it is reduced with an increase in reinforcement particle size (Deshmanya & Purohit, 2012). Ultimate tensile strength is improved with an increase in reinforcement content in the Al-based MMCs (Srivatsan & Prakash, 1995; Manoharan & Gupta, 1999; Bhushan & Kumar, 2011; Alaneme & Aluko, 2012; Veeresh Kumar et al., 2012). A continuous reduction of ultimate tensile strength with increasing volume fraction of SiC in solution annealed, peak-aged and over-aged conditions of Al-Zn-Mg alloy matrix composites was observed by Kumar and Dwarakadasa (2000). Open literatures have reveled the significant influence of reinforcement percentage on compression strength and flexural strength of Al-based MMCs (Kumar & Dwarakadasa, 2000; Demir & Altkinkok, 2004; Kalkanli & Yilmaz, 2008). Impact strength of Al 6061/SiC/fly ash hybrid MMC is increased with an increase in weight % of SiC, which is due to proper dispersion of reinforcements into the matrix; and strong interfacial bonding between the matrix and reinforcement interfaces (Ravesh & Garg, 2012).

Mechanical properties of MMCs are highly influenced by the effective fabrication process, controlled processes parameters, heat treatment conditions, type of metallic phases, particle size, and percentage & type of ceramic reinforcement. Therefore, optimization of fabrication process parameters, conditions of heat treatment and selection of reinforcement and matrix phases are highly essential for strength-based applications of a composite material. Moreover, we observed almost no literature on multiple response optimization of fabrication process parameters of Al-based MMCs considering their mechanical properties as performance criteria. Accordingly, this paper presents a comparative study of mechanical properties of SiC_p reinforced Al 7075 (Al 7075/SiC_p) MMCs, both in as-cast and heat treated conditions, fabricated through low cost liquid metallurgy stir casting method. Keeping a view on the industrial demand for the materials with optimal combination of strength-based properties, multiple response optimization of processing parameters is conducted and linear regression models are developed for different mechanical properties of the heat treated MMCs.

2. Materials and methods

2.1 Fabrication of MMCs

Al 7075 MMCs reinforced with SiC particulates were fabricated through stir casting method or liquid metallurgy route. Chemical composition test results of the alloy are presented in Table 1. After heating the matrix alloy up to 820 ± 10^0C in an electrical resistance furnace, SiC particulates (preheated to 900 ± 1^0C for two hours) were added at about 10 grams per minute, into the vortex of molten alloy, created by stirring at 160 rpm. The immersion depth of the stirrer was two-third of the height of the molten alloy in the crucible. After SiC_p addition the stirring was continued for 10 minutes more at a speed 220 rpm. About 10 grams of solid hexachloroethane tablet was then inserted into bottom of the crucible containing molten composite slurry for degassing. The composite slurry was then poured into a steel mold at pouring temperature of 800 ± 10^0C.

Table 1
Chemical composition test result of the aluminium alloy

Element	Si	Fe	Cu	Mn	Mg	Zn	Ti	Cr	Al	Others
Weight %	0.143	0.313	1.39	0.137	2.46	5.60	0.044	0.198	88.9	Rest

2.2 Experimental parameters and design of experiments

For fabrication of the MMCs, mean particle size (*s*) and weight % (*w*) of SiC_p reinforcement were considered as process parameters. Three levels for each parameter are presented in Table 2. MMCs were fabricated as per Taguchi L_9 DOE (Table 3). Two sets of nine different MMC samples were produced and numbered accordingly as A1, A2, A3, B1, B2, B3, C1, C2 and C3, where the alphabets (A, B and C) represent levels of mean particle size and the Arabic numerals (1, 2 and 3) represent those of weight % of SiC_p.

Table 2

Fabrication process parameters and their levels

Process parameters	Levels of parameters		
	Level 1	Level 2	Level 3
Mean particle size of SiC, *s* (μm)	30.65	8.18	6.18
Weight % of SiC, *w*	5	15	25

Table 3

Taguchi L_9 DOE for MMC fabrication

Sample no.	*s*	*w*
A1	30.65	5
A2	30.65	15
A3	30.65	25
B1	8.18	5
B2	8.18	15
B3	8.18	25
C1	6.18	5
C2	6.18	15
C3	6.18	25

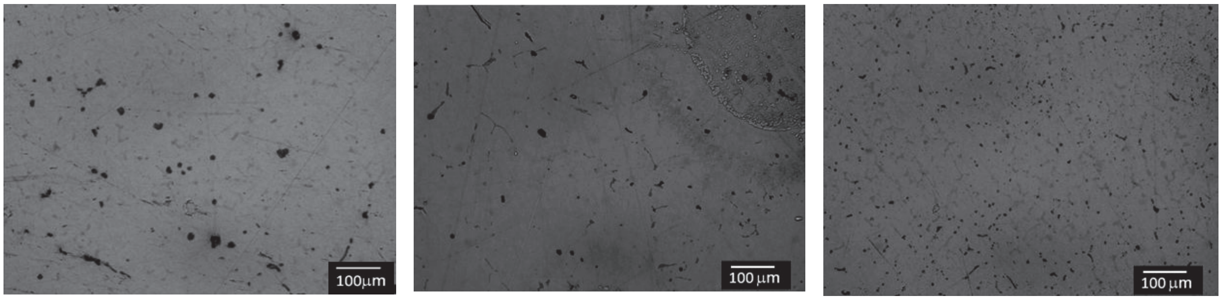
2.3 Heat treatment

One set of the fabricated MMC samples was heat treated to T6 condition using a Labotech-BDI 73 muffle furnace. The heat treatment process are involved solution annealing at 483 ± 3^0C for 2 hours followed by water quenching; and then precipitation hardening (aging) at 122^0C for 24 hours followed by air cooling (Kalkanli & Yilmaz, 2008; Kumar & Dhiman, 2013; Web link 1). Fig. 1 shows the image of heat treated MMC samples.

Fig. 1. Heat treated MMC samples

2.4 Optical microscopy

Uniform distribution of SiC particles with some local agglomeration in the matrix alloy was observed through Lieca DMI3000 M inverted optical microscope. Metallographic specimens were prepared as per ASTM E3-95 standard for optical microscopy. Fig. 2 represents optical micrographs of some heat treated MMC samples, as illustration.



(a) (b) (c)

Fig. 2. Optical micrographs of (a) A2; (b) B1; and (c) C3

2.5 Mechanical properties

Vickers Hardness (v) of the MMCs was determined using Leco-LM247AT microhardness tester (Fig. 3) as per ASTM E384 standard. Tensile tests were conducted as per ASTM E8 standard using Instron-8801 fully automated servo-hydraulic testing machine (Fig. 4) to determine ultimate tensile strength (σ_{ut}) of the MMCs. Compressive strength (σ_c) was determined using Heico-HL590.15 universal testing machine (Fig. 5) as per ASTM E9 standard. Charpy impact tests were conducted as per ASTM E23 standard using Instron-600 MPX impact testing machine (Fig. 6) to determine impact strength (σ_i) of the MMCs. Flexural strength (σ_{fl}) was determined by three point bend tests using Tinius Olsen H50K-S universal testing machine (Fig. 7). All the tests were conducted in room temperature environment.

Fig. 3. Hardness test

Fig. 4. Tensile test

Fig. 5. Compression test

Fig. 6. Impact test

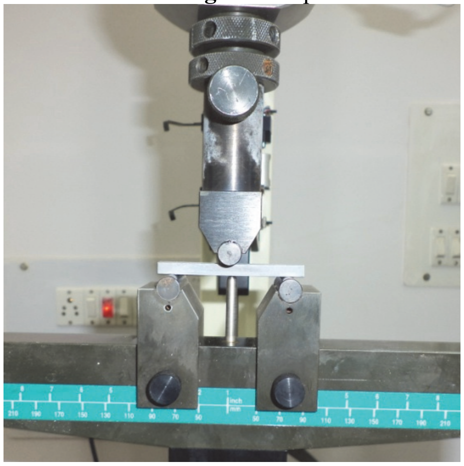


Fig. 7. Three point bend test

3. Results and discussion

Experimental results of the mechanical properties for both as-cast and heat treated Al 7075/SiC$_p$ MMCs are presented in Tables 4 and Table 5, respectively. From the results, better mechanical properties are observed for the heat treated MMCs than those of their as-cast counter parts. So, for further studies only heat treated MMCs are focused.

Table 4
Mechanical properties of as-cast Al 7075/SiC$_p$ MMCs

Sample no.	v (HV)	σ_{ut} (MPa)	σ_c (MPa)	σ_i (kJ/m^2)	σ_{fl} (MPa)
A1	136.40	249.32	550.32	59.1	477
A2	148.00	256.51	619.11	66.4	415
A3	167.30	251.35	696.82	70.6	385
B1	153.80	278.27	634.39	61.2	323
B2	168.10	285.42	695.54	72.4	268
B3	187.00	279.16	782.17	73.9	201
C1	159.60	280.86	654.78	62.4	253
C2	172.70	288.57	712.10	74.1	199
C3	191.00	282.43	811.47	76.2	122

Table 5
Mechanical properties of heat treated Al 7075/SiC$_p$ MMCs

Sample no.	v (HV)	σ_{ut} (MPa)	σ_c (MPa)	σ_i (kJ/m^2)	σ_{fl} (MPa)
A1	178.00	399.90	774.52	89.80	606
A2	189.30	349.87	817.83	96.40	489
A3	210.60	298.50	899.36	99.60	467
B1	195.70	481.82	820.38	90.10	435
B2	207.20	422.24	872.61	100.70	345
B3	229.00	360.56	933.76	102.00	232
C1	201.30	496.81	836.94	91.20	372
C2	211.00	436.21	894.27	104.00	233
C3	232.50	375.76	978.34	105.80	133

3.1 Regression models

In this section, linear regression models are developed using least square method of regression analysis, to predict the mechanical properties (v, σ_{ut}, σ_c, σ_i and σ_{fl}) of the heat treated Al 7075/SiC$_p$ MMCs for different values of fabrication process parameters (s and w). The regression equations for each of the responses are presented in Eqs. (1-5). For most of the models (except for σ_i) the coefficients of determination (R^2) are more that 90%, which indicate very good prediction of the responses; however, the viability of prediction for σ_i is 83%. Further, the predicted values of R^2 are in reasonable agreement with the adjusted R^2 for all the models. So, it can be concluded that the models are adequate for representing the process and fit the sample data (Reddy and Rao 2006). The normal probability plots of residuals for different responses (Figs. 8 a-e) depict that the residuals lie reasonably close to the normal probability lines, implying that residuals are distributed normally and the terms mentioned in the regression models are significant and adequate (Suresha and Sridhara 2012).

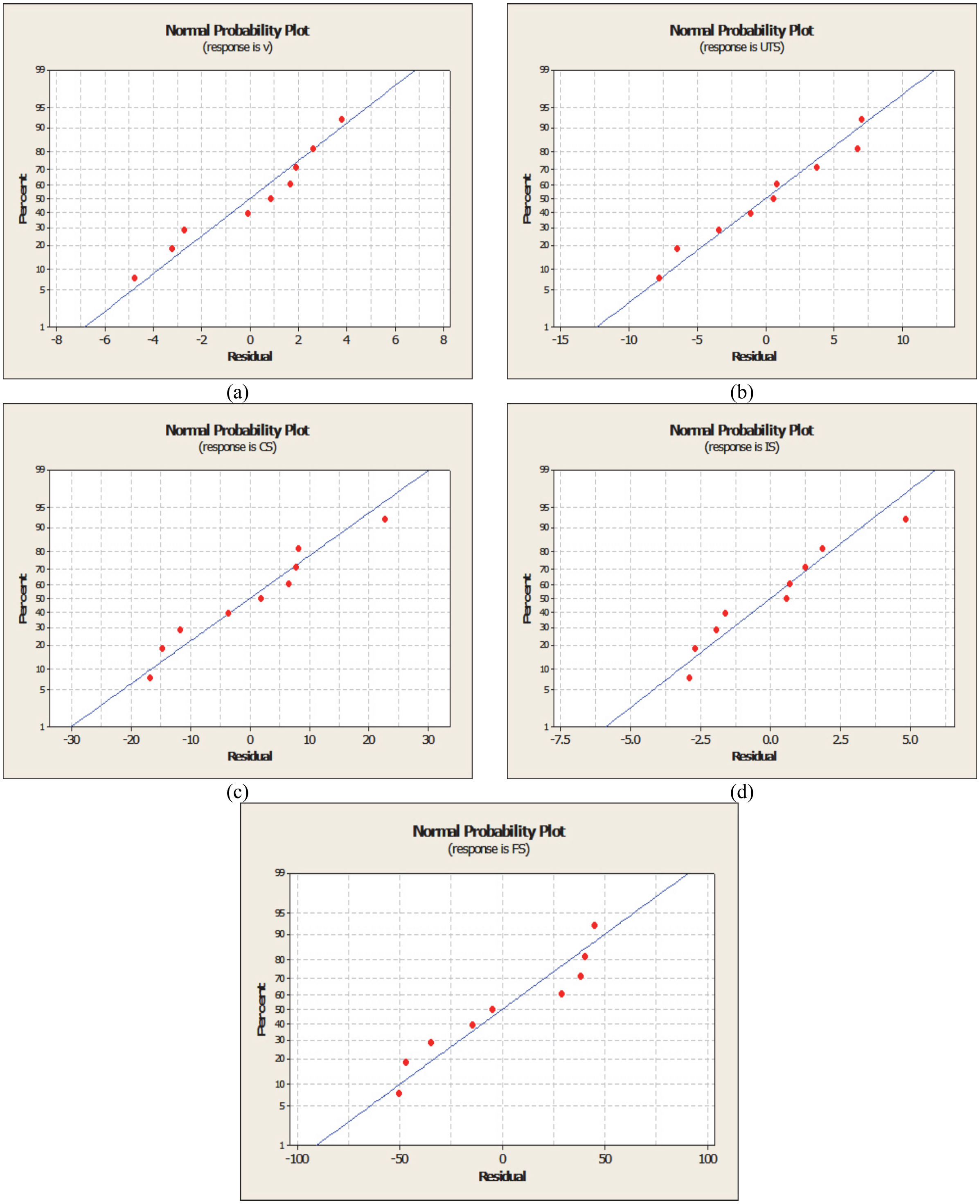
$$v = 195 - 0.866s + 1.62w \quad R^2 = 97.2\%, R^2(adj) = 96.3\% \tag{1}$$

$$\sigma_{ut} = 539 - 3.41s - 5.73w \quad R^2 = 99.3\%, R^2(adj) = 99.1\% \tag{2}$$

$$\sigma_c = 813 - 2.57s + 6.33w \quad R^2 = 95.9\%, R^2(adj) = 94.5\% \tag{3}$$

$$\sigma_i = 91.1 - 0.164s + 0.605w \quad R^2 = 83.0\%, R^2(adj) = 77.4\% \tag{4}$$

$$\sigma_{fl} = 364 + 9.95s - 9.68w \quad R^2 = 93.2\%, R^2(adj) = 90.9\% \tag{5}$$



(a) (b)

(c) (d)

(e)

Fig. 8. Normal probability plots of residuals for (a) v; (b) σ_{ut}; (c) σ_c; (d) σ_i; and (e) σ_{fl}

3.2 Optimization for multiple performance characteristics

Taguchi based grey relational analysis is used for optimization of the fabrication process parameters (s and w) for the multiple performance quality characteristics (i.e. v, σ_{ut}, σ_c, σ_i *and* σ_{fl}) of the heat treated Al 7075/SiC$_p$ MMCs. Various steps involved in this method are discussed bellow.

Step I. Normalization of experimental results or grey relational generation.

It generates normalized data sequence of the experimental results within 0 and 1. If the response in the original sequence is “smaller is better”, then it is normalized using Eq. (6). However, if the response is “larger is better”, then Eq. (7) is used for its normalization (Tzeng et al., 2009; Mishra et al., 2015).

$$x_i^*(k) = \frac{\max\ x_i^0(k) - x_i^0(k)}{\max\ x_i^0(k) - \min\ x_i^0(k)} \tag{6}$$

$$x_i^*(k) = \frac{x_i^0(k) - \min x_i^0(k)}{\max\ x_i^0(k) - \min\ x_i^0(k)} \tag{7}$$

where $x_i^*(k)$ is the series after the data processing or compatibility sequence, $x_i^0(k)$ is the original series of the target value for $i = 1, 2, 3….,\ m$ and $k = 1, 2, 3….,\ n$. Here m is total number of experiments conducted and n is total number of observed data or responses. For the present analysis, $m = 9$ and $n = 5$. The normalized data of the experimental results are presented in Table 6.

Table 6

Normalized data of the experimental results

Sample no.	v	σ_{ut}	σ_c	σ_i	σ_{fl}
A1	0.0000	0.5113	0.0000	0.0000	1.0000
A2	0.2073	0.2590	0.2125	0.4125	0.7526
A3	0.5982	0.0000	0.6125	0.6125	0.7061
B1	0.3248	0.9244	0.2250	0.0187	0.6385
B2	0.5358	0.6240	0.4813	0.6813	0.4482
B3	0.9358	0.3129	0.7813	0.7625	0.2093
C1	0.4275	1.0000	0.3063	0.0875	0.5053
C2	0.6055	0.6944	0.5875	0.8875	0.2114
C3	1.0000	0.3896	1.0000	1.0000	0.0000

Step II. Determination of deviation coefficient and grey relational coefficient.

Deviation coefficient is the absolute value of the difference between reference sequence and compatibility sequence, i.e.

$$\Delta_{0i}(k) = \left| x_0^*(k) - x_i^*(k) \right| \tag{8}$$

where $\Delta_{oi}(k)$ is deviation coefficient, $x_o^*(k)$ is reference sequence or ideal sequence. Grey relational coefficient is determined using Eq. (9).

$$\gamma\left(x_0^*(k) \cdot x_i^*(k)\right) = \frac{\Delta_{\min} + \varsigma \cdot \Delta_{\max}}{\Delta_{0i}(k) + \varsigma \cdot \Delta_{\max}} \tag{9}$$

where $\gamma(x_o^*(k).x_i^*(k))$ is the grey relational coefficient and ς is distinguishing coefficient (0~1). The deviation coefficient and grey relational coefficient (with $\varsigma = 0.5$) of the responses are presented in Tables 7 and 8 respectively.

Table 7
Deviation coefficients of the responses

Sample no.	v	σ_{ut}	σ_c	σ_i	σ_{fl}
A1	1.0000	0.4887	1.0000	1.0000	0.0000
A2	0.7927	0.7410	0.7875	0.5875	0.2474
A3	0.4018	1.0000	0.3875	0.3875	0.2939
B1	0.6752	0.0756	0.7750	0.9813	0.3615
B2	0.4642	0.3760	0.5187	0.3188	0.5518
B3	0.0642	0.6871	0.2187	0.2375	0.7907
C1	0.5725	0.0000	0.6937	0.9125	0.4947
C2	0.3945	0.3056	0.4125	0.1125	0.7886
C3	0.0000	0.6104	0.0000	0.0000	1.0000

Table 8
Grey relational coefficient of the responses with $\varsigma = 0.5$

Sample no.	v	σ_{ut}	σ_c	σ_i	σ_{fl}
A1	0.3333	0.5057	0.3333	0.3333	1.0000
A2	0.3868	0.4029	0.3884	0.4598	0.6690
A3	0.5544	0.3333	0.5634	0.5634	0.6298
B1	0.4254	0.8687	0.3922	0.3376	0.5804
B2	0.5186	0.5708	0.4908	0.6107	0.4754
B3	0.8862	0.4212	0.6957	0.6780	0.3874
C1	0.4662	1.0000	0.4189	0.3540	0.5027
C2	0.5590	0.6207	0.5480	0.8163	0.3880
C3	1.0000	0.4503	1.0000	1.0000	0.3333

Step III. Determination of grey relational grade and its order sequencing.

Grey relational grade $(\gamma(x_o^*.x_i^*))$ is the weighted sum of the grey relational coefficients and represents the level of correlation between the reference and compatibility sequence. It can be calculated using Eq. (10).

$$\gamma\left(x_0^* \cdot x_i^*\right) = \frac{1}{n}\sum_{k=1}^{n} \gamma\left(x_0^*(k) \cdot \gamma\left(x_i^*(k)\right)\right) \tag{10}$$

The grey relational grades are than sequenced in descending order. For higher values of grey relational grades, the relation between the reference sequence and compatibility sequence becomes stronger (Lin, 2004). Table 9 represents grey relational grades and their order for the multiple performance characteristics.

Table 9
Grey relational grades and their order

Sample no.	Grey relational grade	Order
A1	0.5011	8
A2	0.4614	9
A3	0.5289	6
B1	0.5208	7
B2	0.5332	5
B3	0.6137	2
C1	0.5483	4
C2	0.5864	3
C3	0.7567	1

Step IV. Analysis of experimental results using the grey relational grades.

Response table for means of grey relational grade (Table 10) is generated using Taguchi method to calculate the mean grey relational grade for each level of the process parameters.

Table 10
Response table for means of grey relational grade

Process parameters	Grey relational grade			Max-min	Rank
	Level 1	Level 2	Level 3		
s	0.4971	0.5559	0.6305	0.1333	1
w	0.5234	0.5270	0.6331	0.1097	2
Total mean grey relational grade = 0.5612					

The highest values of grey relational grade represent the optimal combination of parameters for the desired responses (Lin, 2004; Tzeng et al., 2009). In Table 10 the highest value of mean grey relational grade is obtained for the composite with combination of s_3-w_3, which indicates that the optimal combination of fabrication process parameters for the multiple performance characteristics is 6.18 μm of mean particle size and 25 weight % of SiC reinforcement. The main effects plot for the means of grey relational grade is shown in Fig. 9. The central horizontal line in the main effects plot represents the total mean grey relational grade, i.e. 0.5612.

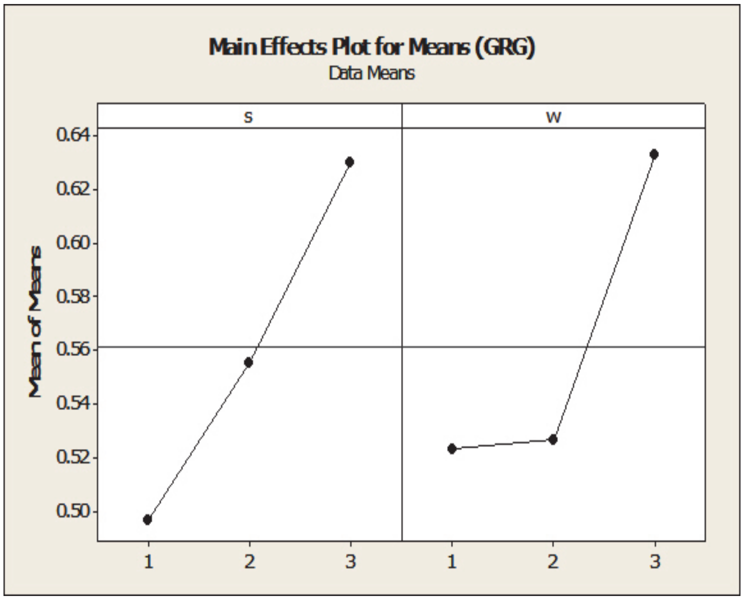


Fig. 9. Main effects plot for means of grey relational grade

Step V. Verification of optimal process parameters through confirmation experiment.

Table 11 shows the results of confirmation experiments for the mechanical properties of the MMCs with initial and optimal process parameters. The values of v, σ_c and σ_i for optimal combination of process parameters are sufficiently higher, but σ_{ut} and σ_{fl} were lower as compared to those for initial process parameters. The predicted value of grey relational grade is very close to its experimental value. The improvement in grey relational grade for optimal process parameter is 0.2235, i.e. around 42 %.

Table 11
Results of confirmation experiments

Level	Initial process parameters	Optimal process parameters	
		Prediction	Experiment
	s_2-w_2	s_3-w_3	s_3-w_3
v	207.20	232.9	232.50
σ_{ut}	422.24	378.79	375.76
σ_c	872.61	970.55	978.34
σ_i	100.70	105.06	105.80
σ_{fl}	345	155.33	133
Grey relational grade	0.5332	0.7024	0.7567

Improvement in grey relational grade = 0.2235

3.3 Analysis of Variance (ANOVA) for grey relational grade

Statistical ANOVA at 95 % confidence level is conducted to analyze the grey relational grade of the experimental results. Percentage of contribution of each fabrication process parameter on the multiple mechanical properties is determined. ANOVA data for grey relational grade are presented in Table 12. Results indicate that the mean particle size of SiC has the highest contribution (45.58 %), followed by weight % of SiC$_p$ reinforcement (39.62 %) for affecting the multiple performance characteristics under consideration.

Table 12
ANOVA for grey relational grade

Process parameters	DF	SS	MS	F-Value	% of contribution
s	2	0.0268	0.0134	6.15	45.58
w	2	0.0233	0.0116	5.35	39.62
Error	4	0.0087	0.0022		14.80
Total	8	0.0588			100

4. Conclusions

Consistent dispersion of SiC particulates in the matrix alloy was observed in the optical micrographs of Al 7075/SiC$_p$ MMCs. T6 condition of heat treatment improved all the mechanical properties under consideration. For prediction of mechanical properties for different values of fabrication process parameters, linear regression models were developed. Adequacy of the models were verified through coefficients of determination (higher values). Normal distribution of the residuals and significance of the models were observed through normal probability plots of residuals. During multiple performance optimization, the largest value of the mean grey relational grade was achieved for the MMC with mean particle size 6.18 µm and 25 weight % of SiC$_p$ reinforcement. It is the recommended combination of levels of fabrication process parameters for Al 7075/SiC$_p$ MMCs for the multiple response criteria under consideration. Around 42% of improvement in grey relational grade was achieved during confirmation experiments. ANOVA results for grey relational grade indicate that mean particle size of SiC is the more influencing process parameter than its weight % in the MMCs.

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