

Experimental Analysis of Density of Sintered SiCp Reinforced AMMCS Using the Response Surface Method

Sujit Das, R. Behera, P.K.Bardhan, S. Patra, B. Oraon, G. Sutradhar

Abstract- The continuous development of technology in automotive manufacturing process demands new solutions which is largely dependent on the development of lightweight, non-pollution for the environment materials of improved mechanical properties and also with a low cost production. According with these required characteristics of materials, the aims of this paper were to manufacturing Al-SiCp composites by powder metallurgy (P/M) processing route. Since density is a pre-dominant factor in the performance of powder metallurgy components, it has been primarily considered for the present investigation. An experimental investigation have been undertaken in order to understand the variation of density with respect to the variation of process parameters viz., variation of silicon carbide proportion, compacting pressure and sintering time. The relation among the various process parameters with density has been studied. A mathematical model has been developed using second order response surface model (RSM) with central composite design (CCD) considering the above mentioned process parameters. The mathematical model which developed in this investigation would help in predicting the variation in density with the change in the level of different parameters influencing the density variation. This mathematical model also can be useful for setting of optimum value of the parameters for achieving the target density.

Keywords: Powder Metallurgy, Density, Sintering, Response Surface Model, Central Composite Design.

I. INTRODUCTION

The continuous development of technology in automotive manufacturing process has required new solutions adapted to the growing requirements of lightweight, non-pollution for the environment materials with a low cost production. According with these required characteristics of materials, the aims of this paper were to manufacturing Al-SiCp composites by powder metallurgy (P/M) processing route and characterization of the powders and compacted/sintered mixture powders [1, 2]. Development of powder metallurgy (P/M) technology is providing itself as an alternate lower process cost to machining, casting, stamping, forging and other similar metal working technologies [3,4].

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Apart from these it also provides some outstanding advantages such as high material utilization, more refined microstructure that provides superior material properties as well as greater microstructure homogeneity. Among others, however, the powder metallurgy (P/M) method has known as a very promising route, which is most attractive due to several reasons. Firstly, in P/M technique micro structural control of the phases is possible. Secondly, the lower temperatures employed during the process accounts for the strict control of interphase kinetics. Poor distribution of reinforcement degrades the composites in terms of its physical and mechanical properties and negates the attractiveness of reinforcement additions [5-9]. Composites combine the characteristics of aluminium and aluminium alloys matrix (low density in comparison with ferrous materials, good corrosion resistance and machinability) with the characteristics of ceramic particles (e.g. SiCp, TiCp, B₄Cp, Al₂O₃, SiO₂, etc.) which improve in special mechanical, tribological and thermal expansion characteristics [10-12]. As sintering is a predominant factor for controlling the density of the P/M products, variation of wt% of reinforcing materials, compacting pressure, sintering time, temperature largely affects the density of the P/M components [13-17]. The sintered parts of high density can be steam treated to close the surface pores. It is also observed that the green density and sintered density is a function of powder type and compacting pressure [15]. Present study examines the variation of density (R1) as a function of process parameters (weight percentage of SiCp x_1 , compacting pressure x_2 , and sintering time x_3) of sintered iron P/M components. The samples were produced by changing the process parameters as per the design of experiment (DOE) and the response surface methodology (RSM) has been used to plan and analyze the density. The experimental plan adopts the face-centered central composite design (CCD). A second order response surface model (RSM) has been used to develop a predicting equation of density based on the data collected by a statistical design of experiments [18-20]. The analysis of variation (ANOVA) shows that the observed data fits well into the assumed second order RSM model. It is worth mentioning that this model is one of the most widely used methods to solve the optimization problem in manufacturing technology [16]. In the experiment, porosity of the samples, compacted and sintered under different conditions were investigated by the optical microscope [13]. It is found that porosity of the samples decreases with the increase of compacting pressure, sintering temperature and sintering time.

II. EXPERIMENTAL PROCEDURES

A. Production of metal matrix composite

Despite of the advantages of processing by P/M of powders, in aluminium matrix composites, the powder mixtures are more difficult to compact and sinter than other composites the presence of hard ceramic particles in aluminium ductile matrix increases this processing difficulty. Air atomized aluminium powder (average particle size of 400 mesh) reinforced with SiC particulates (Fig. 1) (average size of 400 mesh) are used as the test material along with commercially pure aluminium. In this paper, we have developed new materials in terms of composition (Al-SiCp) and manufacturing process and were determined the optimal technological parameters of densification of composites.

The above composites and aluminium has fabricated by powder metallurgy technique (steps of this technique shown in Figure 2). Al-SiCp were blended on a pot mill (diameter 40 mm height 35 mm), at a constant speed of 1500 rpm for 1hour to obtain a homogeneous powder blend. Blending is one of the crucial processes in P/M where the metallic powders have mixed with the ceramic reinforced particles and the binder (Zinc Stearate). Several parameters such as particle size, blending speed and duration should be taken into consideration to ensure the SiCp particles distributing homogeneously in the matrix powders.

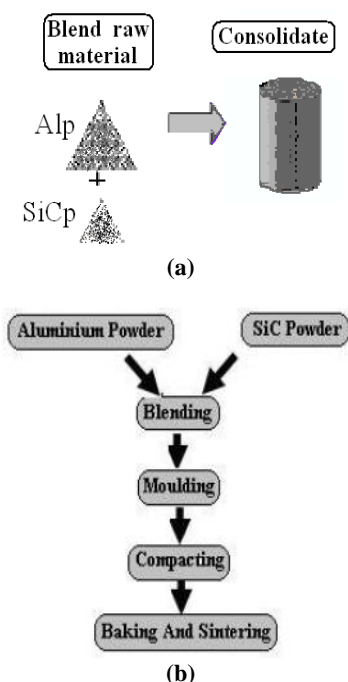


Figure 1 (a) & (b): Various steps involved in synthesis of Al-SiCp composites in P/M technique.



Figure 2: Tubular Vacuum Furnace.

One of the major objectives of present investigations is to shade light on the density of the compacted sintered samples. In this context 60 different P/M components (diameter 25 mm) were produced according to design of experiment (DOE). Related density (R_1) of these samples were measured by hydrostatic weighing method against the variation of controllable process variables like weight percentage of SiCp (x_1), compacting pressure (x_2), and sintering time (x_3). After pouring the Al-SiCp powder mixture the Green compacts of the powder blend were prepared in a closed cylindrical die Capacity in a closed 120-Ton hydraulic press (Make-Lawrence & Mayo). The compacting pressure applied and maintained for 5min to obtain green compacts for all composition of SiCp composites. During compacting, the die was lubricated with Zinc stearate. The sintering process was carried out in a tubular vacuum furnace (diameter of hot zone 75 mm lengths of hot zone 150 mm and maximum temp 1450°C) using argon as an inert atmosphere (Fig.2).

B. Density Measurement

The density of the composites was obtained by the Archimedian principle of weighing the sample first in air and then in water. Then, theoretical density of composite and its alloy has calculated from the chemical analysis data. The measured relative density of the compacts was about 81.2%. The gain refinement of metal matrix-based composites reinforced by tough particles can interpret by the increased effective extrusion ratio with increasing volume fraction of incompressible reinforcements

The P/M samples sintered at fixed temperature (530°C) for fixed sintering time (40 mins.) under different compacting pressure, have been prepared and the microstructures Fig. 3 (a-e) examined by using microscope Olympus, CK40M). The white portion of the figure indicates Al Matrix and the black portions indicate SiCp in the specimen. From the figure it is quite evident that with gradual increase of compacting pressure the porosity of the samples gradually decreases. Similar behavior is also observed with the variation of sintering time and sinter temperature, the porosity changes (not shown in figure). Decrease in porosity would increase the density. The plastic deformation is beneficial to improve the homogeneity of the reinforcement. Particle matrix debonding and particle agglomerate decohesion are the two mechanisms are of secondary importance when the particles are well distributed and strongly bonded. Particles enhance the relative density of the materials and refine the metal matrix grains, which consequentially result in the improvement of mechanical properties of the composites.

C. Mathematical Modeling

From the results of ANOVA a mathematical model has been proposed for the evaluation of density, RCCD (Density) of the powder metallurgy components. The proposed model is expressed as

$$\begin{aligned} \text{RCCD (Density)} = & - 0.820967 + 0.218738 x_1 \\ & + 0.008407x_2 - 0.571286 x_3 \\ & + 0.007148x_{12} - 0.000002x_{22} \\ & + 0.064705x_{32} - 0.000333x_{1x2} \\ & - 0.020574x_{1x3} + \\ & 0.000767x_{2x3} \end{aligned}$$



Where, RCCD: response, i.e., density in central composite design

The results obtained through the experiments are given in Tables 1 and 2 and the available data have been analyzed by response surface method using Minitab software (version 14).

III. RESULTS AND DISCUSSION

Table 1
Symbols, levels and values of process parameters

Process parameters (Independent variables)	Symbols		Levels					
	Actual	Coded	Actual			Coded		
weight percentage of SiCp	Z1	X1	2	5	8	-1	0	+1
Compacting pressure(Ton)	Z2	X2	40	60	80	-1	0	+1
Sintering time (Mins)	Z3	X3	30	40	50	-1	0	+1

TABLE 2
Observed Density values for different settings of process parameters based on 23 full factorial design

Std Order	Run Order	Pt Type	Blocks	Wt.% SiCp	Comp. Pr. (Ton)	Sint Time (Mins)	Result	Density
1	55	1	1	2	40	30	2.712	2.712
2	59	1	1	8	40	30	2.892	2.892
3	1	1	1	2	80	30	2.702	2.702
4	3	1	1	8	80	30	3.02	3.02
5	35	1	1		40	50	2.7144	2.7144
6	9	1	1	8	40	50	2.9304	2.9304
7	4	1	1	2	80	50	2.724	2.724
8	19	1	1	8	80	50	3.084	3.084
9	27	-1	1	-0.04538	60	40	2.70027	2.70027
10	44	-1	1	10.04538	60	40	3.132	3.132
11	13	-1	1	5	26.36414	40	2.7405	2.7405
12	2	-1	1	5	93.63586	40	2.862	2.862
13	32	-1	1	5	60	23.18207	2.76	2.76
14	52	-1	1	5	60	56.81793	2.834	2.834
15	15	0	1	5	60	40	2.808	2.808
16	54	0	1	5	60	40	2.809	2.809
17	58	0	1	5	60	40	2.807	2.807
18	29	0	1	5	60	40	2.808	2.808
19	47	0	1	5	60	40	2.808	2.808
20	21	0	1	5	60	40	2.809	2.809
21	14	1	1	2	40	30	2.713	2.713
22	38	1	1	8	40	30	2.893	2.893
23	25	1	1	2	80	30	2.71	2.71
24	10	1	1	8	80	30	3.01	3.01
25	41	1	1	2	40	50	2.7145	2.7145
26	17	1	1	8	40	50	2.9303	2.9303
27	18	1	1	2	80	50	2.724	2.724
28	45	1	1	8	80	50	3.086	3.086
29	37	-1	1	-0.04538	60	40	2.70026	2.70026
30	23	-1	1	10.04538	60	40	3.131	3.131
31	49	-1	1	5	26.36414	40	2.74054	2.74054
32	42	-1	1	5	93.63586	40	2.863	2.863
33	48	-1	1	5	60	23.18207	2.75	2.75
34	6	-1	1	5	60	56.81793	2.833	2.833
35	50	0	1	5	60	40	2.807	2.807
36	36	0	1	5	60	40	2.809	2.809
37	20	0	1	5	60	40	2.808	2.808
38	39	0	1	5	60	40	2.809	2.809
39	22	0	1	5	60	40	2.809	2.809
40	46	0	1	5	60	40	2.806	2.806
41	33	1	1	2	40	30	2.714	2.714
42	31	1	1	8	40	30	2.8921	2.8921
43	12	1	1	2	80	30	2.73	2.73
44	16	1	1	8	80	30	3.04	3.04
45	40	1	1	2	40	50	2.7115	2.7115
46	26	1	1	8	40	50	2.9306	2.9306
47	5	1	1	2	80	50	2.7245	2.7245
48	7	1	1	8	80	50	3.0845	3.0845
49	8	-1	1	-0.04538	60	40	2.70025	2.70025



50	43	-1	1	10.04538	60	40	3.132	3.132
51	11	-1	1	5	26.36414	40	2.7405	2.7405
52	28	-1	1	5	93.63586	40	2.863	2.863
53	56	-1	1	5	60	23.18207	2.77	2.77
54	53	-1	1	5	60	56.81793	2.834	2.834
55	34	0	1	5	60	40	2.809	2.809
56	30	0	1	5	60	40	2.808	2.808
57	57	0	1	5	60	40	2.807	2.807
58	24	0	1	5	60	40	2.807	2.807
59	51	0	1	5	60	40	2.806	2.806
60	60	0	1	5	60	40	2.808	2.808

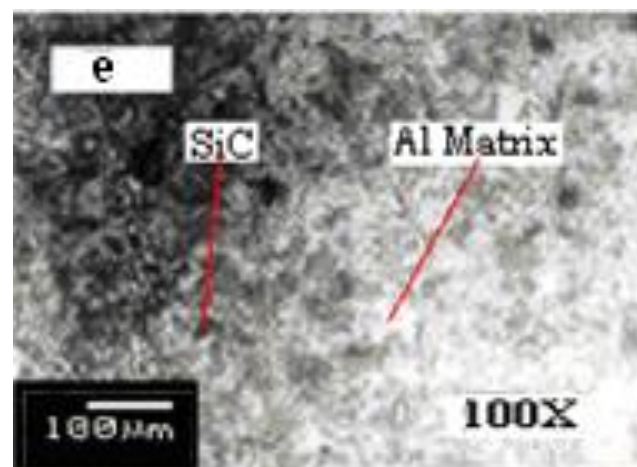
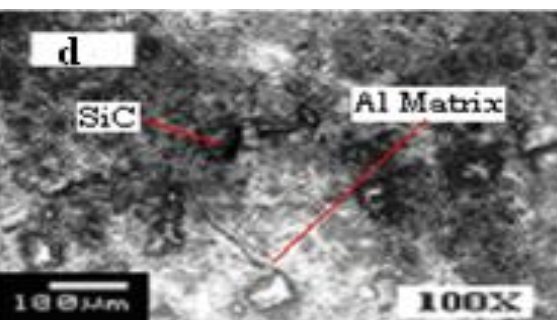
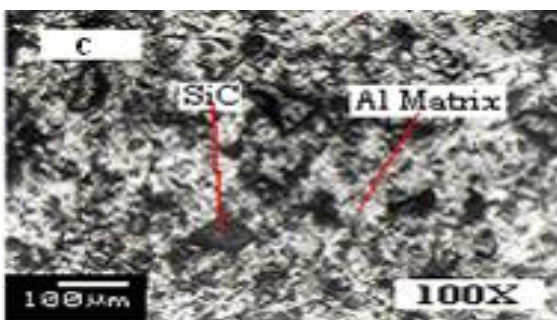
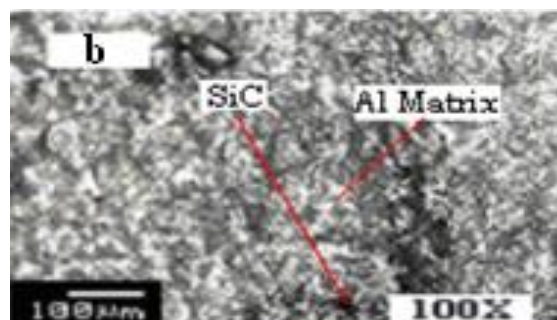
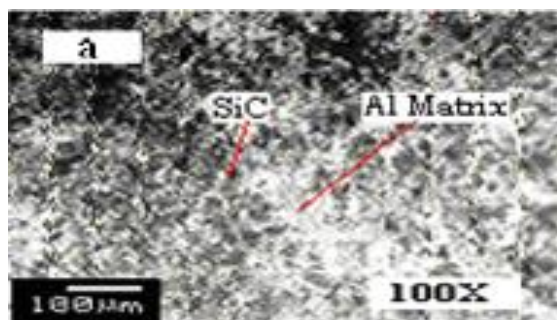


Figure 3. Microstructure of Al-SiCp P/M composite specimen at different pressure (a) Compacting pressure 93.63586Ton, Sintering temperature 530°C Sintering time 40 mins; (b) Compacting pressure 80Ton, Sintering temperature 530°C, Sintering time 40 mins; (c) Compacting pressure 60Ton, Sintering temperature 530°C, Sintering time 40 mins; (d) Compacting pressure 40Ton, Sintering temperature 530°C, Sintering time 40 mins. (e) Compacting pressure 26.36414Ton, Sintering temperature 530°C, and Sintering time 40 mins.

In the present study Al-SiCp powder mixtures of different composition are compacted, sintered at a inert atmosphere, at a fixed temperature for different time duration. The samples are compacted under different pressure range (40-93.63586Ton).The total experiment is performed according to the design of experiment (DOE). The variation of density against wt% of SiCp (x1) and compaction load for a fixed value of sintering time (40 minutes) is presented in Fig. 4. This figure exhibits an increasing tendency in density due to change in wt% of SiCp (x1) and compaction load from 40-93.63586 Ton at a fixed sintering time of 40 minutes. Identical nature of variation is noted in simultaneous increase of sintering time (x3) and wt% of SiCp (x1) for a fixed value of compacting pressure (x2).. This observation is illustrated in Figure 5.



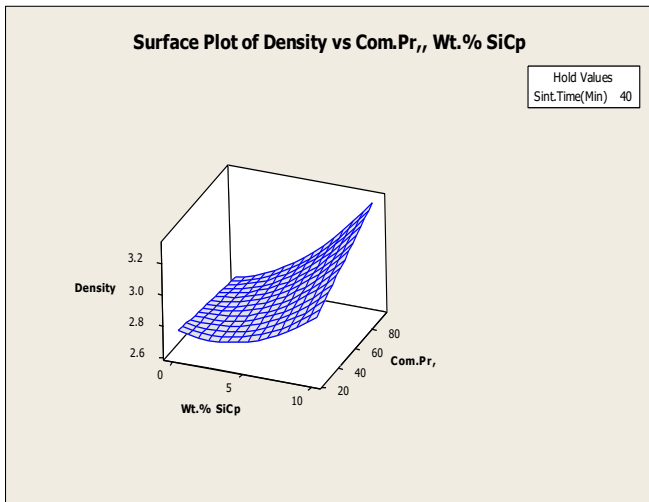


Figure 4: Surface Plot of density (R_1) vs. compacting pressure (x_2) and wt% of SiCp (x_1) for a fixed value of sintering time (x_3).

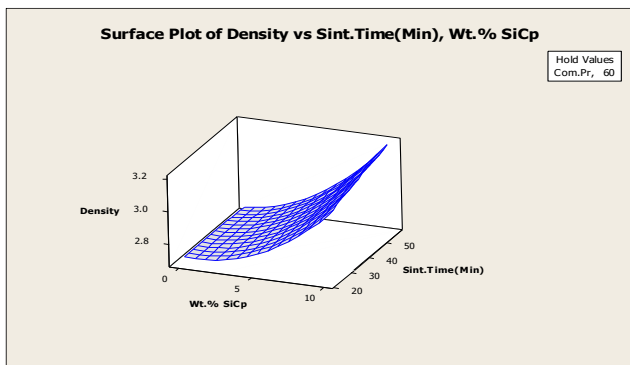


Figure 5: Surface Plot of density (R_1) vs. sintering time (x_3) and wt% of SiCp (x_1) for a fixed value of compacting pressure (x_2).

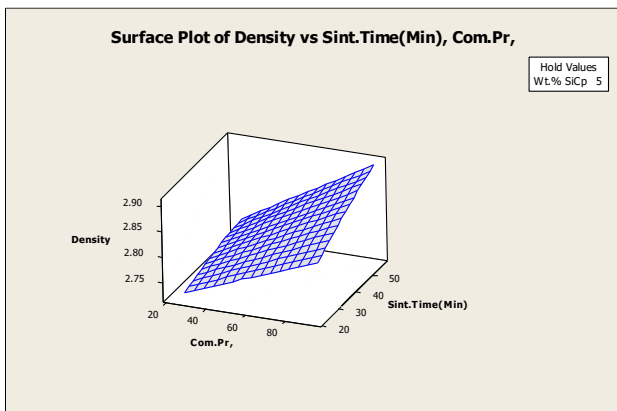


Figure 6: Surface Plot of density (R_1) vs. sintering time (x_3) and compacting pressure (x_2) for a fixed value of wt% of SiCp (x_1).

In Fig.6, the response variable, density (R_1) shows linear increase when it is plotted against sintering time (x_3) and compacting pressure (x_2) for a fixed value of wt% of SiCp (x_1). In this case, the range of variation of the parameters is similar to that of previous two cases. It is worth mentioning that in all the cases the hold values are mean value of the range of variation corresponding to each variable. Average values are preferred because of the inherent nature of the RSM model.

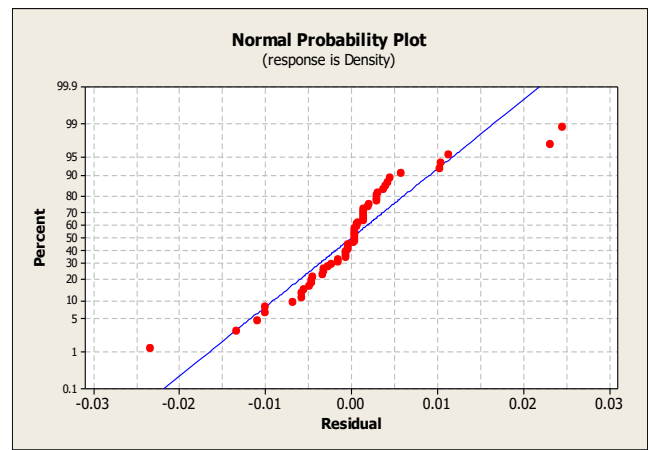


Figure 7: Plot between observed density data and predicted density for RSM model.

This is also evident from the findings that co-efficient of determination (R-Square) value is 89.8 %. Hence, it may be concluded that the prediction made by this developed model corroborates well with the experimental observations.

IV. CONCLUSIONS

In the present study Al-SiCp powder mixtures of different composition are compacted, sintered at a inert atmosphere, at a fixed temperature for different time duration. The samples are compacted under different pressure range (40-93.63586 Ton). The total experiment is performed according to the design of experiment (DOE). Using the experimental data a mathematical model has been developed to predict the density variations of the using response surface method (RSM).

The model shows increase in density due to change in wt% of SiCp (x_1) and sintering time for compaction load from 40-93.63586 Ton at a fixed sintering time of 40 minutes and for a fixed value of compacting pressure (x_2). The response variable, density (R_1) shows linear increase when it is plotted against sintering time (x_3) and compacting pressure (x_2) for a fixed value of wt% of SiCp (x_1) and the prediction of density variation from the mathematical model developed in this study matches closely with the observed data ($R^2 = 89.8 \%$).

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