IJESRT
INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

EXPERIMENTAL INVESTIGATION TO PREDICT DENSITY OF P/M ALUMINIUM-COPPER COMPOSITES IN COLD COMPRESSION USING SURFACE RESPONSE METHODOLOGY

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DOI: 10.5281/zenodo.154226

ABSTRACT

In this study, response surface methodology (RSM) has been applied for the prediction of the influence of process parameters, namely, deformation load, aspect ratio, initial relative density and copper content (%) on the densification behaviour of powder metallurgical aluminium-copper preforms. The process has been successfully modeled using RSM, Box-Behnken design technique, and tested for its adequacy. The significant process parameters have been identified and verified. The results revealed that all the process parameters are significant in which deformation load and initial relative density are positively affected the final relative density while aspect ratio and copper content (%) are negatively affected the final relative density of the preforms. The experimental and predicted values are in a good agreement. The model sufficiency is very satisfactory as the coefficient of determination (R^2) is found to be 99.38% and adjusted R^2 (adj R^2) is 98.77%.

Keywords: Powder metallurgy; Response surface methodology; Box-Behnken design; ANOVA

INTRODUCTION

The engineering application of powder metallurgy (P/M) aluminium alloys or composites has been increased due to its light weight and high performance material system. The P/M route provides an advantage of producing near net shape or net shaped products and excellent materials utilization. However, components produced by P/M have relatively poor mechanical properties due to the presence of inherent porosity left after sintering. Thus, bulk forming processes such as forging, extrusion and hot deformation are convenient methods for reducing or eliminating the porosity [1]. Understanding of the final density of the porous metals after bulk deformation processes is essential because it determines the performance of service and life of the components. Therefore, manufacturers of P/M components are interested in predicting the final density and optimizing the involved process parameters. The deformation and densification behaviour of P/M components are considerably different from that of the corresponding wrought or cast metals due to the presence of inherent porosity. The flow stress of P/M components is greater to that of corresponding conventional metals due to densification phenomena [2, 3]. The flow of metals is restricted in the transverse direction in the P/M components as compared to the conventional metals. Moreover, the presence of porosity in the P/M components leads to severe interface frictional conditions [4]. Therefore, the deformation and densification behaviour information of conventional metals are not applicable for the corresponding P/M components with the same composition.

The important process parameters controlling the material and pore closure phenomena during plastic deformation of P/M components are deformation load, initial relative density, aspect ratio and material composition. The rate of densification during cold or hot deformation of sintered components can be considerably controlled by the proper control of the input process parameters. Nowadays, modeling techniques are being used in prediction and optimization of process variables in different manufacturing areas. In the current work, surface response methodology (RSM) was adopted to investigate the influence of deformation load, aspect ratio, initial relative density and copper content in the aluminum-copper composites on final relative density of the preform. The prime advantage of using RSM is reduced number of experimental runs required to generate sufficient information for developing the desired relation between variables and response. Many researchers [5-12] have adopted RSM successfully to investigate the interaction between several process variables and one or more response variables in different manufacturing applications. Tiernan et al [13] employed RSM to determine the influence of process parameters; namely, the conical die angle, die land height and die exit diameter on the extrusion force, and concluded that all the process variables have a strong influence on the extrusion force behaviour. Srinivasulu et al [14] conducted experimental investigation to study the influence of process variables on the mean diameter of AA6082 tube in flow forming process by employing RSM. They found that axial feed of the roller and roller radius are quite significant parameters influencing the mean diameter of flow forming AA6082 flow formed tube. The influence of other process parameter i.e. the speed of mandrel on the mean diameter is not significant. Optimization of electric discharge machining (EDM) parameters using RSM for EN31 tool steel machining was carried out successfully by Lakshmanan et al [15]. They reported that the parameters pulse off and current are most significant but pulse on and voltage are non-significant to the material removal rate during EDM of EN31 tool steel.

Literature studies related to modeling of densification behaviour of P/M preforms during plastic deformation are scarce. In this investigation, the influence of process parameters such as deformation load, aspect ratio, initial relative density and percentage of copper content on the final relative density of P/M aluminium-copper composites when they are subjected to cold axial forming using RSM is presented. Further, a model to successfully predict the final relative density of the P/M preforms is discussed.

EXPERIMENTAL PROCEDURES AND METHODS

1.1. Experimental procedure

Sintered aluminium-copper preforms were prepared from atomized pure aluminium and copper powders of each -325μm mesh size. The purity level of aluminium powder is 99% with a maximum of 0.53% insoluble impurity limit. Copper powder is 99% pure with 0.5% and 0.03% of iron (Fe) and heavy metals (Pb) of impurities, respectively. Figs. 1(a) and (b) represent the SEM photographs of aluminium powder and copper powder, respectively. Pure Al, Al-3%Cu and Al-6%Cu compacts of different initial relative densities, 80%, 85% and 90% and aspect ratio of 0.5, 0.75 and 1, were prepared by applying recommended uniaxial compaction pressures. Zinc stearate was used to lubricate the die, punch and butt to reduce the interface friction. The powder compacts were sintered in a muffle furnace at 550 ±10$^{o}{}_{C}$ for a holding time of 45 minutes in argon gas flowing atmosphere. Immediately after the completion of sintering schedule, the preforms were made to cool to room temperature inside the furnace by switching off the power source of furnace. The Archimedes' principle was employed to measure the density of the preforms throughout the investigation. The compression test was conducted in between two parallel flat platens on a hydraulic press of 50 ton capacity. Each specimen was subjected to experimentally designed deformation load of 20 KN, 45 KN and 70 KN. The density after compression test was measured for each test specimens.

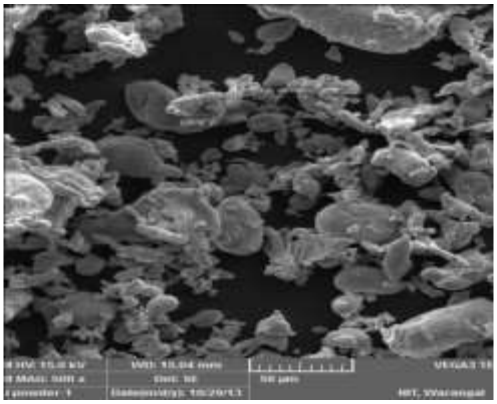

Fig. 1a The SEM Photograph of Al powder

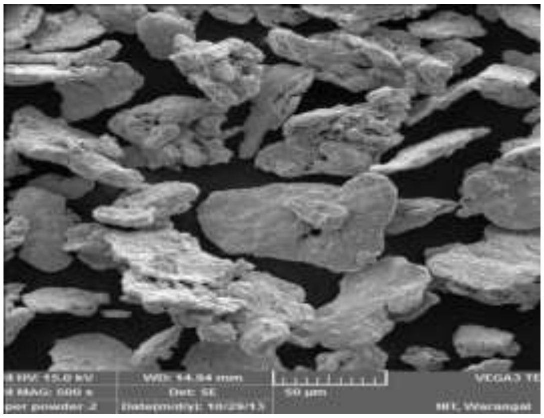

Fig. 1b The SEM Photograph of Cu powder

1.2. Response Surface Methodology

Response surface methodology was employed to investigate the effect of four independent variables; namely, deformation load, aspect ratio, initial relative density and copper content on the response function. The design procedure of RSM employed was reported elsewhere [16, 17]. Box-Behnken experimental design consisting of 29 experiments was conducted for developing the mathematical model for the determination of final relative density of the P/M preforms. The number of experiments required for developing Box-Behnken design matrix is defined as N = 2k(k-1) + r, where (k) is the number of independent variables and (r) is the replicate number of the central point. The input variables and their levels chosen for investigation are given in Table 1.

Table 1 Input process parameters and their levels used for Box–Behnken design

Variables, Unit	**Low (-1)**	**Medium (0)**	**High (1)**
Deformation load (KN)	20	45	70
Aspect ratio	0.5	0.75	1
Initial relative density (%)	80	85	90
Copper content (%)	0	3	6

The relationship between the response and the input parameters is described by

$Y= f (x_1, x_2 , x_3 ,................x_k) + \epsilon$, where f is the response function, y is the response, x_1, x_2 , x_3 ,...and x_k are the input process parameters and ϵ is the residual factor associated with the experiment.

Based on the experimental design, the final relative density of the P/M aluminum-copper composites were measured after compression for each experimental condition and given in Table 2.

Table 2 Box–Behnken experimental design matrix and experimental responses

Experimental run	**Deformation load (KN)**	**Aspect ratio**	**Initial relative density (%)**	**Copper content (%)**	**Final relative density (%)**
1	45	0.50	90	3	93.61
2	45	0.75	90	0	93.77
3	70	1.00	85	3	93.55
4	45	0.75	85	3	91.78
5	20	0.75	85	6	88.25
6	70	0.50	85	3	94.08
7	20	0.75	85	0	89.66
8	45	1.00	85	0	91.55
9	70	0.75	85	0	94.11
10	70	0.75	90	3	95.04
11	70	0.75	85	6	93.49
12	45	0.50	80	3	89.92
13	45	1.00	90	3	92.78
14	45	0.75	80	0	89.06
15	45	1.00	85	6	90.25

16	45	0.75	85	3	91.78
17	45	0.75	85	3	91.78
18	45	0.50	85	0	92.56
19	20	0.50	85	3	89.01
20	45	0.75	80	6	88.76
21	45	0.50	85	6	91.44
22	45	0.75	85	3	91.78
23	20	0.75	90	3	91.34
24	45	0.75	90	6	93.15
25	20	0.75	80	3	85.02
26	20	1.00	85	3	88.31
27	45	0.75	85	3	91.78
28	70	0.75	80	3	91.92
29	45	1.00	80	3	88.36

RESULTS AND DISCUSSIONS

1.3. Box-Behnken statistical analyses

Based on the experimental results, Box-Behnken technique was carried out to study the influence of deformation load, aspect ratio, initial relative density and copper content on the final relative density of the preforms. From the regression statistics, the coefficient of determination which describes the prediction capability of the model is 99.38%, and it is very close to 1, which is desirable. The difference between predicted R^2 and adjusted R^2 should be not greater than ~ 0.2% to be in a reasonable agreement. In the current work, the predicted R^2 is 96.46% and adjusted R^2 is 98.77%, and it is within permissible limit. ANOVA was used to test the significance and adequacy of the model for the prediction of response, and is given in Table 3.

In the Table 3, ASR is aspect ratio, IRD is initial relative density and Cu (%) is percentage of copper in the composite.

The results from ANOVA table shows that all the four process variables; namely, deformation load, aspect ratio, initial relative density and copper content are statistically significant because their P-values are very small (< 0.05). The results revealed that deformation load is most significant as it contributes the highest effect on densification, followed by initial relative density, aspect ratio and copper content, respectively. Fig.2a-2d depict the main effects of process variables on the response, i.e. final relative density.

It is clearly observed that deformation load and initial relative density affect the final relative density positively while the aspect ratio and copper content in the composite affect negatively. The effect of aspect ratio on the final relative density is higher in the case of preforms with lower initial relative density than higher initial relative density.

The regression equation to predict the final relative density of the P/M aluminium-copper composites is:

$$\begin{aligned} &Final\ relative\ density \\ &= -114.013 + 0.6871(load) - 10.386(ASR) + 4.084(IRD) + 0.2918\big(Cu(\%)\big) \\ &+ 0.0068(load * ASR) - 0.0064(load * IRD) - 0.0026\big(load * Cu(\%)\big) + 0.146(ASR * IRD) \\ &- 0.06\big(ASR * Cu(\%)\big) - 0.0053\big(IRD * Cu(\%)\big) - 6.0067E^{-04}(load)^2 - 2.7267(ASR)^2 \\ &- 0.0202(IRD)^2 - 0.0102(Cu)^2 \qquad (1) \end{aligned}$$

Table 3 ANOVA for final relative density

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value Prob > F**	**Remarks**
Model	147.55	14	10.54	161.51	< 0.0001	significant
A-Deformation load	78.03	1	78.03	1195.83	< 0.0001	Significant
B-Aspect ratio	2.82	1	2.82	43.26	< 0.0001	Significant
C-Inital relative density	59.19	1	59.19	907.03	< 0.0001	Significant
D-Copper content	2.40	1	2.40	36.83	< 0.0001	Significant
AB	7.225E-003	1	7.225E-003	0.11	0.7443	
AC	2.56	1	2.56	39.23	< 0.0001	Significant
AD	0.16	1	0.16	2.39	0.1443	
BC	0.13	1	0.13	2.04	0.1750	
BD	8.100E-003	1	8.100E-003	0.12	0.7298	
CD	0.026	1	0.026	0.39	0.5412	
A^2	0.91	1	0.91	14.01	0.0022	Significant
B^2	0.19	1	0.19	2.89	0.1114	
C^2	1.67	1	1.67	25.52	0.0002	Significant
D^2	0.055	1	0.055	0.84	0.3762	
Residual	0.91	14	0.065			
Lack of Fit	0.91	10	0.091			
Pure Error	0.000	4	0.000			
Cor Total	148.46	28				

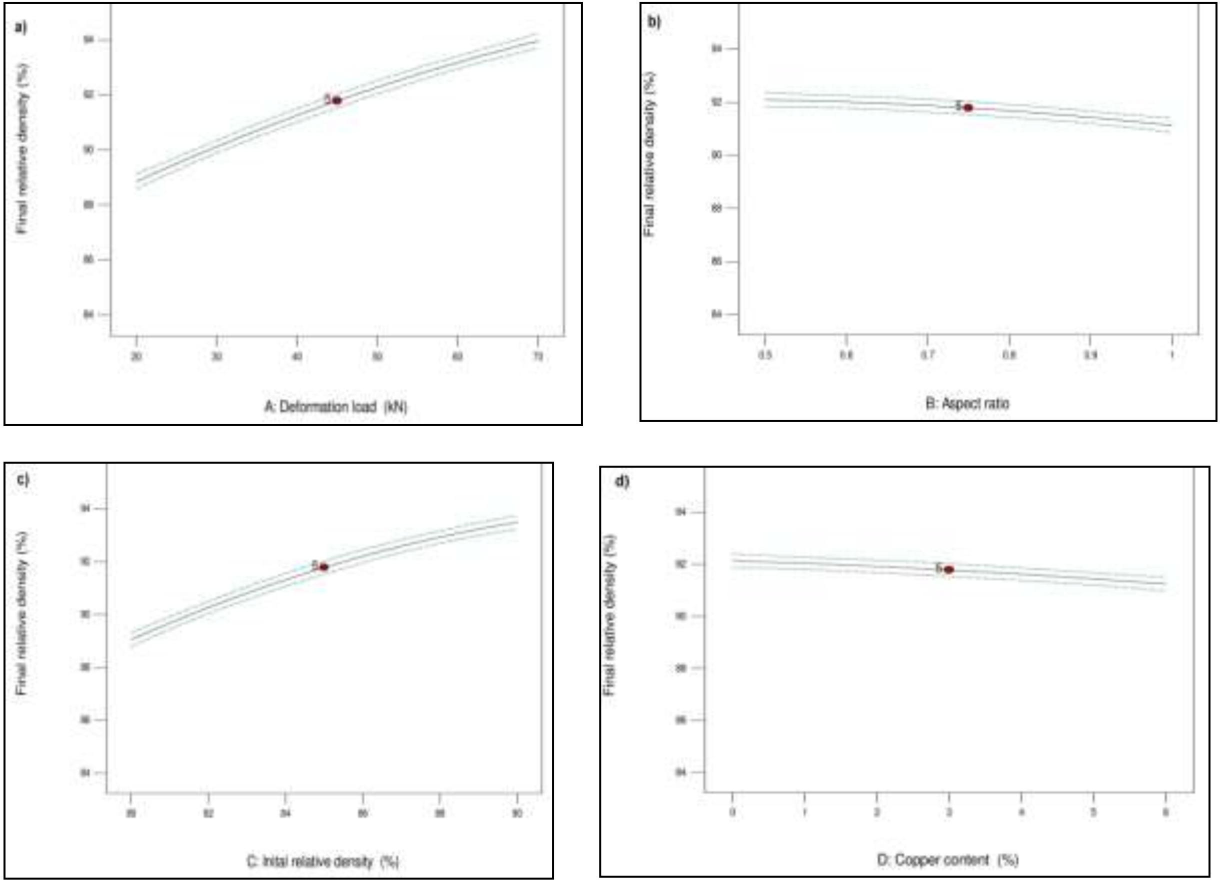

Fig. 2 Main effects of deformation load, aspect ratio, initial relative density and copper content on the final relative density

Elimination of the insignificant terms to adjust the fitted quadratic model gives the adequate model to predict the final relative density in terms of the input process parameters. The associated P-value for this model is lower than 0.05; i.e. α = 0.05 or 95% confidence level. Hence, the terms having P-values less than 0.05 are considered as significant while the terms having P-values greater than 0.05 are insignificant. After eliminating the insignificant terms, the final proposed model is given as:

$$\begin{aligned} Final\ relative\ density &= -108.743 + 0.6941(load) - 1.94(ASR) + 3.8939(IRD) - 0.1491\big(Cu(\%)\big) \\ &\quad - 0.0064(load * ASR) - 5.339E^{-04}(load)^2 - 0.0186(IRD)^2 \qquad (6.20) \end{aligned}$$

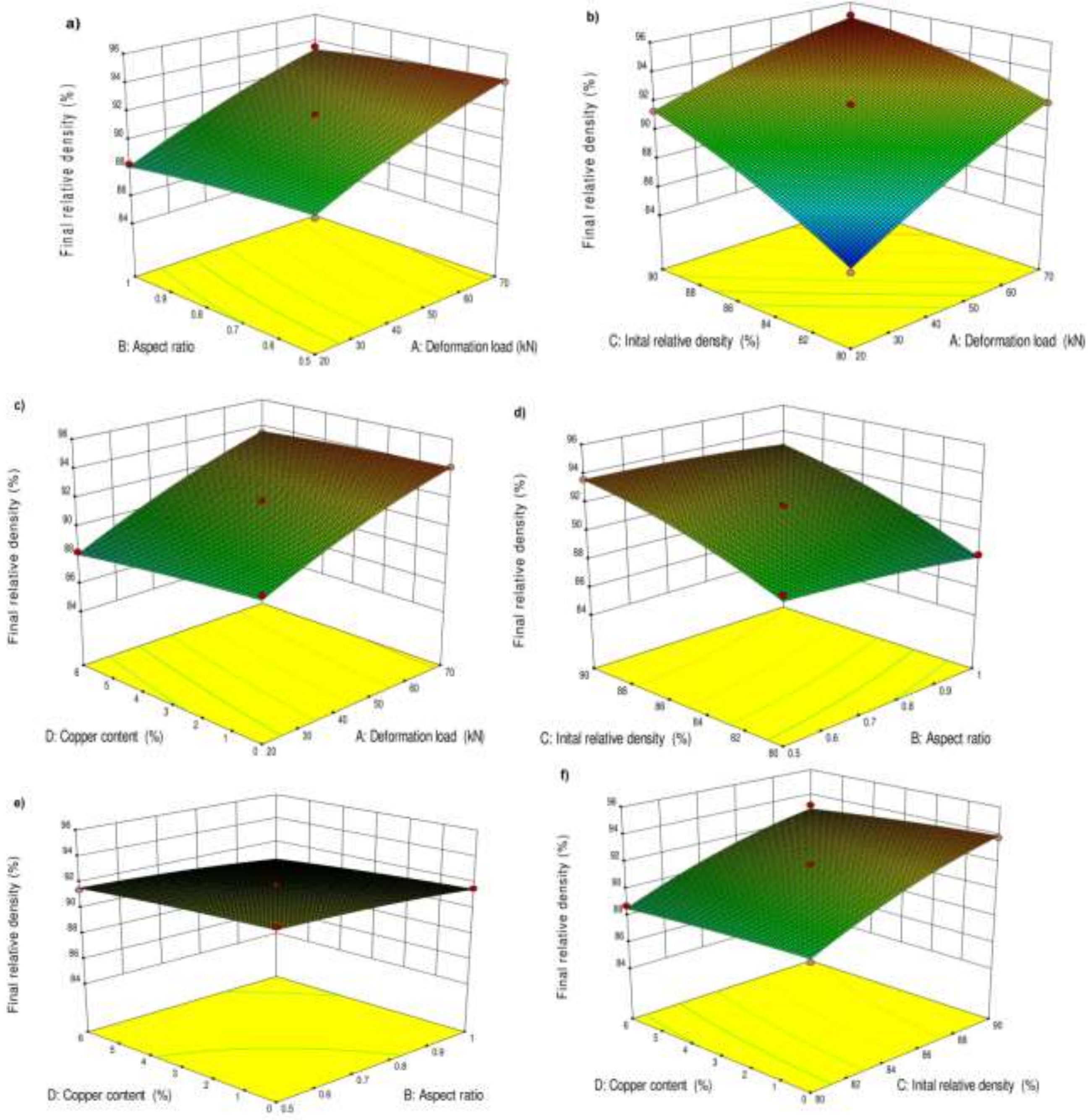

Fig. 3 The 3D response surface plots for final relative density a) effect of load and initial relative density (as aspect ratio = 0.75 and copper content = 3%) b) effect of load and aspect ratio (as initial relative density = 85% and copper content= 3%) c) effect of aspect ratio and initial relative density (as load =45KN and copper content= 3%) d) effect of aspect ratio and copper content (as load = 45KN and initial relative density = 85%) e) effect of copper content and initial relative density (as aspect ratio = 0.75 and load = 45KN) f) effect of load and copper content (as aspect ratio = 0.75 and initial relative density = 85%)

The model F-value for the model is 161.51 which is very large as compared to the F value obtained from standard distribution curve that implies the adequacy of the model. According to Davidson et al [18], there is only 0.01% chance that a “model F-value” could occur due to noise. To test the validity of model, “Lack of Fit Test” is used to compare the residual error to the pure error from replicated design points. The lack of fit F-value is 0.914 which is not significant as the P-value is > 0.05. Fig. 4 shows the plot of actual relative density versus the predicted relative density of the preforms. The coefficient of determination (R^2) is 0.99, which reveals an excellent fit between predicted values and the experimental data points. Further, the R^2 value explains the extent of variation that the independent variables affect the response and it also describes that the model does not explain for only 1% variations.

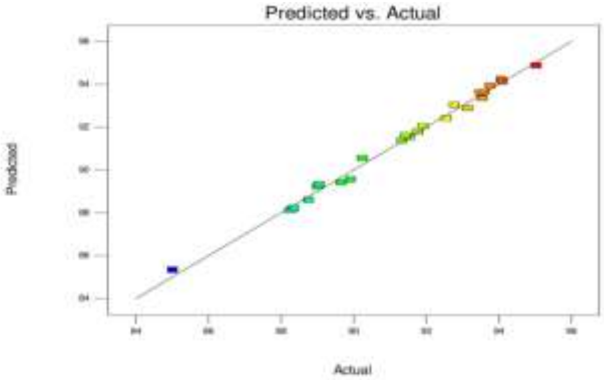

Fig. 4 Plot of actual final relative density versus predicted final relative density

The model adequacy was further analyzed by the examination of the residuals which is the difference between the observed response and predicted response. Figs. 5(a)-(b) show the normal probability plot and residuals versus fits for final relative density. It is clearly observed from Fig. 5(a) that all the data points are distributed close to the best fit line which proves the adequacy of the model.

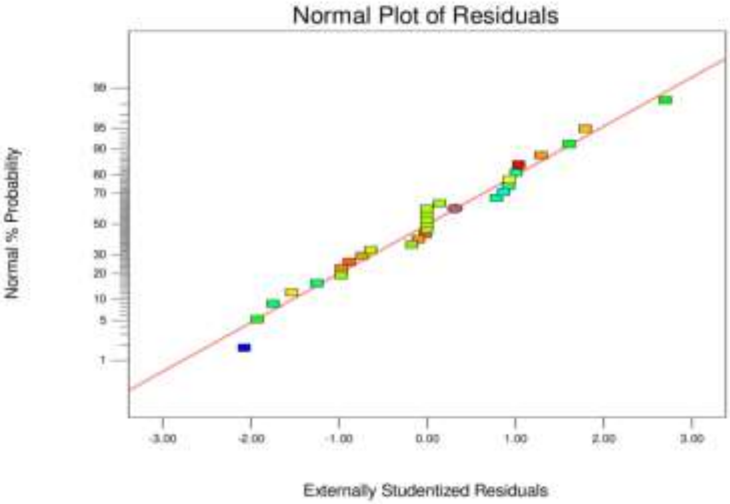

Fig. 5a Normal probability plot of residuals for final relative density

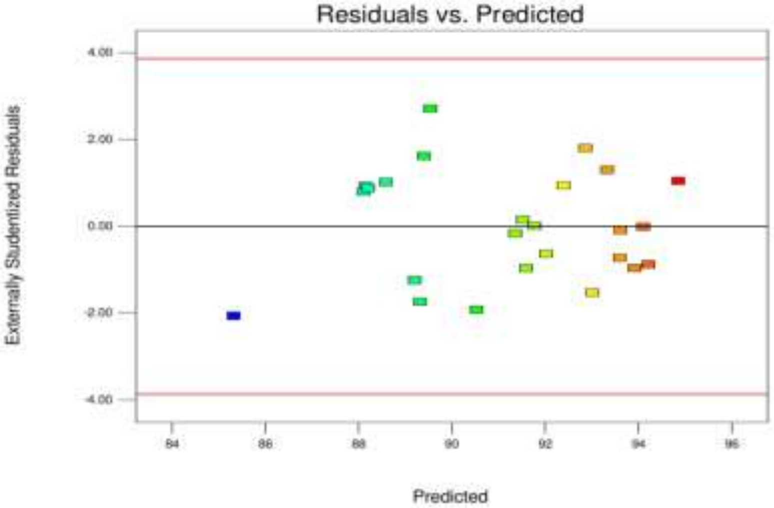

Fig. 5b Plots of residuals versus fits for final relative density

In addition, the plots of residuals versus fits, as shown in Fig. 5(b), present no obvious pattern in the distribution of the data. According to Noordi et al [9], there is no reason to suspect any violation of the independence or constant variance assumption if no usual pattern in the plots of residual versus fits for the predicted response is observed and thus, the model is adequate to predict the final relative density. The results showed that the data follows nearly normal distribution.

Confirmation tests were carried out to check the prediction capability of the model and results are presented in Table 4. The percentage absolute relative error between the experimental and predicted values was determined. The maximum absolute relative error is 0.08% which is small and thus, it confirms the ability of the model to predict the final relative density of aluminium-copper composites successfully for any combinations of the input parameters; namely, deformation load, aspect ratio, initial relative density and copper content.

Table 4 Partial experimental and predicted data and their associated relative percentage error

Deformation load (KN)	**Aspect ratio**	**Initial relative density (%)**	**Copper content (%)**	**Final relative density (%)**		**Error (%)**
				Experimental	**Predicted**	
32.5	0.65	82.5	1.5	89.22	89.26	0.04
57.5	0.8	87.5	1.5	93.71	93.74	0.03
32.5	0.65	87.5	4.5	91.39	91.43	0.04
57.5	0.65	82.5	1.5	92.28	92.21	0.08
57.5	0.9	82.5	4.5	91.34	91.28	0.07
32.5	0.9	82.5	4.5	88.34	88.32	0.02
57.5	0.9	82.5	4.5	91.27	91.28	0.01
32.5	0.65	87.5	1.5	91.92	91.88	0.05
57.5	0.9	87.5	4.5	93.02	93.10	0.08

CONCLUSION

In this study, experimental investigation on densification behaviour of P/M aluminium-copper composites is carried out with a view to correlate the process parameters; namely, deformation load, aspect ratio, initial relative density and copper content (%) with the final relative density of the preforms. The process has been successfully modeled using statistical methodology, Box-Behnken design surface response methodology, to predict the final relative density for different input process parameters. The model is validated using ANOVA, and the experimental and predicted values are in a reasonable agreement ($R^2 = 0.99$). The results revealed that all the process parameters have a significant influence on the final relative density in which the effect of deformation load and initial relative density is most imminent. Model confirmation has been performed and the maximum percentage absolute relative error is 0.08% which is a reasonable. The research findings can help researchers and industries for developing a robust, reliable knowledge base and early prediction of the final relative density of P/M aluminum-copper preforms during deformation.

ACKNOWLEDGEMENT

The authors would like to thank M.J. Davidson and A.K. Khanra for their guidance, and the technical staffs of National Institute of Technology, Warangal, India for their help.

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