# Powder Metallurgy & Mining

# The Application of Response Surface Methodology for the Optimization of Compaction Parameters in $Cu-Al_2O_3$ Composite

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### Abstract

In the present work copper-alumina composite has been successfully produced by mechanical milling. Response Surface Methodology (RSM) was employed to study and optimize the effect of compaction parameters, namely; compaction pressure (Mpa), lubricant content (%) and compaction temperature (°C). The response of the system was green density (GD). Results demonstrated that all the above mentioned factors had meaningful effect on the GD. It has been shown that prominent parameters can be impacted to the final production during compaction process. Meanwhile statistically significant interactions were found between compaction pressure (Mpa), lubricant content and compaction temperature (°C). A suitable model equation was also predicted.

**Keywords:** Copper-alumina composite; Compaction; Response surface methodology (RSM); Optimization

# Introduction

For many applications such as electronic packaging or manufacturing of electrodes and contact materials [1], that the materials must have a good combination of electrical conductivity, and resistance to erosion and welding [2]. Mechanical attrition of copper powders with ceramic particles has allowed the uniform introduction of small strengthening phases and also promotes a microstructural grain refinement [3] Copper-Alumina metal matrix composites (MMCs) combine the high electrical and thermal conductivity of the copper phase and high strength and high thermal and chemical stability of the Alumina phase. Thus Copper-Alumina MMCs have the potential to offer both high strength and high electrical conductivity. In order to achieve high fracture toughness and low processing cost, the Alumina phase in the microstructure should be in particulate form, and the particle size should be small. Several techniques can be used to synthesize Copper-Alumina MMCs. Casting [4] is not suitable since it produces very coarse grains that would degrade the strength significantly. This limitation can be avoided using the powder metallurgy (PM) process. As a way to improve the mechanical properties at low temperatures, the matrix must be strengthened with very low solubility particles [5]. Mechanical alloying (MA) is a solid-state powder processing technique involving repeated welding, fracturing, and rewelding of powder particles in a high-energy ball mill. Mechanical alloying is a complex process and hence involves optimization of a number of variables to achieve the desired product phase and/or microstructure [6-8]. Distribution of reinforcing particles during ball milling is not only dependent on ball milling parameters but also on initial particle sizes [9]. The optimum milling time depends on the type of mill, size of the grinding medium, temperature of milling, ball-to-powder ratio and desired phase formation [10]. Identification of the correct optimum value of the effective parameters in mechanical alloying system is a prerequisite for their successful exploitation. Different parameters identified in previous research as significantly influencing the powder compaction system are compaction pressure (Mpa), lubricant content and compaction temperature (°C) [6-9].

There are some literatures on powder compaction systems but an answer to the question of what is the optimum level of these parameters cannot be reliably found by reference to these reported studies. A survey of previous literature on powder compaction systems provides no clues as to whether such interaction between the important process parameters exists or not. This is because in previous studies one-factorat-a-time methodology has been used to optimize the abovementioned parameters. This methodology is very inefficient and furthermore gives absolutely no information about interactions between parameters in a process. The only methodology capable of providing an answer to this question is factorial design of experiments (DOE), which - through the use of techniques such as Response Surface Methodology (RSM) - is able to simultaneously consider several factors at different levels, and give a suitable model for the relationship between the various factors and the response [10]. There are "full" as well as "fractional" factorial DOEs; the former gives a complete information regarding interaction between parameters but the number of experiments becomes excessive when the number of factors or their levels becomes relatively large. Additionally, higher order interactions are usually statistically insignificant and, consequently, information about them is not very useful. Fractional factorial designs (FFD)-such as central composite design (CCD) or Box-Behnken can give information regarding parameter interactions with the use of less experimentation. However, reliable information about first order interactions can only be obtained from the results of DOEs which are not highly fractionated. The aim of the present work was to evaluate and quantify interaction between important parameters in a powder compaction system by using appropriate methodology, namely RSM. A half fractional factorial CCD was chosen as the design matrix since it allows reliable identification of first order interaction between factors and provides a second order polynomial model which can be used to predict optimum level of these parameters [11].

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# **Experimental Procedure**

## Materials

The optimized copper-alumina in our previous study was used to optimize the compaction parameters. Depend on Table 1 which is lists of values of 18 combinations of factor levels, two Stages were designed. Samples (18 samples) were taken out from the mould and amount of green density of them were studied.

## **Experimental design for RSM**

A central composite design (CCD) was adopted to study three factors at three levels. Eighteen experimental runs by the principle of RSM using MINITAB Release 15. The levels employed for the different factors, according to CCD design, are listed in table 1. The statistical significance of the full quadratic models predicted was evaluated by the analysis of variance (ANOVA). The significance and the magnitude of the effects estimates for each variable and all their possible linear and quadratic interactions were also determined. Unless otherwise stated, the significance level employed in the analysis was 5% (p<0.05). Finally, the model was used to predict the optimum value of the factors which results in maximum or fairly high lattice parameter (a). All the analyses were carried out using MINITAB Release 15.

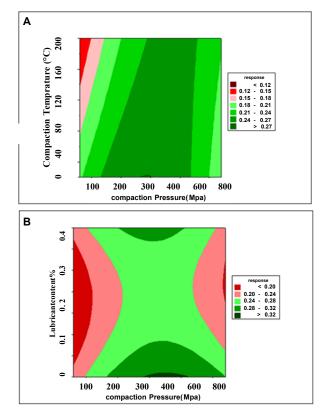
# **Results and Discussions**

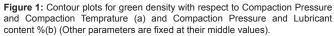
## Model fitting

Table 1 lists the values of difference of lattice parameters at each of the 18 combination of factor levels with the values ranging from 7.66 g.cm<sup>-3</sup> to 8.31 g.cm<sup>-3</sup>. The values of the regression coefficients are presented in table 2. The linear terms as well as the second order terms of the independent parameters, apart from compaction Pressure (Mpa) are significant. The statistical analysis of the interaction terms showed that, at 5% significance level, there are significant interactions between compaction Pressure (Mpa), Lubricant content% and Compaction Temprature (°C). Based on the calculated values of the regression coefficients (Table 2) a polynomial regression model equation that fitted 88.8% of the variation in the data is proposed as follows (coded values): Response: 0.235-0 + 0.0151CP- 0.0135LC +0.0192CT-0.033428LC.LC

Experiment Number	Factors	Response		
	Compaction Pressure(Mpa)	Lubricant Content%	Compaction Temprature (°C)	(g.cm <sup>-3</sup> )
1	100	0	0	7.70
2	700	0	0	7.66
3	100	0.4	0	8.23
4	700	0.4	0	8.20
5	100	0	200	7.73
6	700	0	200	7.74
7	100	0.4	200	8.31
8	700	0.4	200	8.27
9	100	0.2	120	8.13
10	700	0.2	120	8.10
11	400	0	120	7.74
12	400	0.4	120	8.10
13	400	0.2	120	8.14
14	400	0.2	0	8.10
15	400	0.2	0	8.10
16	400	0.2	120	8.14
17	400	0.2	120	8.14
18	400	0.2	120	8.14

Table 1: Central composite design arrangement and response..





	Regression coefficient	Standard error	T-value	
Constant Linear	0.230	0.005	42.140	
CP	-0.0151	0.004	-3.482	
LC	-0.0135	0.004	-3.537	
СТ	0.0192	0.004	4.606	
Quadratic				
CP. CP	-0.008572	0.011	-0.801	
LC. LC	0.033428	0.011	3.122	
CT. CT	-0.095	0.011	-8.834	
Interactive				
CP. LC	0.039	0.0043	4.420	
CP. CT	0.0255	0.0043	6.630	
LC.CT	-0.0165	0.0043	-3.373	

Table 2: Values of calculated regression coefficients (coded values).

	DF	SS	MS	F-values	P-values
Total	27	0.0713			
Regression	14	0.0675	0.0048	16.31	0.000
Residual error	13	0.0038			
Lack of fit (model error)	10	0.0023	0.0002	0.46	0.848
Pure error (Replicate error)	3	0.0015	0.0005		
R2	90.02				

Abbreviations: DF = degrees of freedom; SS = sum of squares; MS = mean squares.

#### Table 3: ANOVA Table

#### -0.095CT.CT+0.039CP.LC+0.0255 CP.CT-0.0165LC.CT (3)

The low values of P determined for the regression (P < 0.001), as well as the fact that the lack of fit of the model was not significant (P > 0.05), revealed the suitability of the model (Table 3).

# Study of interaction amongst factors in the mechanical alloying

In the cases where interaction between factors is statistically significant, surface plots give more complete information regarding the effect of a factor on the response. Examination of the surface plot presented in figure 1 has been shown.

#### Optimization of parameters in the mechanical alloying

Initially, the optimization of the levels of the four factors for achieving maximum response as carried out using the proposed second order polynomial model (Equation 3). This exercise predicted that the maximum green density as 8.35 g.cm<sup>-3</sup> under the following conditions:

Compaction Pressure=100 Mpa, Lubricant content=0.4% and Compaction Temprature 200°C.To confirm this prediction, and therefore the applicability of the proposed second order model for further optimization exercises, confirmation runs (i.e. runs at the predicted optimum level of the factors) were carried out in triplicate. The 90% confidence interval for green density under optimized conditions was obtained as 8.32 g.cm<sup>-3</sup>. Since the value predicted by the model is within this interval, this can be taken as the confirmation of the suitability of the regression model for predictive purposes [11].

#### Conclusions

Response Surface Methodology has been successfully used to optimize compaction factors and to examine the effect of four most effective parameters namely compaction pressure (Mpa), lubricant content% and compaction temprature (°C) in the compaction of Copper-Alumina powder. It was shown that compaction temprature is the most effective parameter as compared to other variable .This exercise predicted that the maximum achievable green density is 8.35. Compaction pressure (Mpa), lubricant content% and compaction temprature (°C) were 100 (Mpa), 0.4%, and 200°C respectively.

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