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THE INFLUENCE OF PROCESS PARAMETERS ON THE FORMATION OF AL COATING ON STEEL BY PLANETARY BALL-MILLING

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ABSTRACT

The effects of milling time, ball/powder ratio, and milling speed on coating thickness are investigated using response surface methodology (i.e., central composite design) in detail. The individual and interaction effects of the factors are determined by the statistical analysis of the data. A Scanning Electron Microscope (SEM) is used for measuring the coating thickness. The results show that the ball/powder ratio is the most significant parameter on the coating thickness. The maximum coating thickness of 164 µm is achieved at a milling speed of 400 rpm, ball-to-powder ratio of 15:1 and milling time of 8 h.

Keywords: Milling, Coating, Response surface methodology

1. INTRODUCTION

Ball milling is a novel investigation area with possible engineering applications for fabricating coatings on the low carbon steel. The coatings produced by high energy ball milling method are obtained by both the surface mechanical attrition treatment and mechanical alloying processes. The method is suitable for coating. Chemical bonding occurs at the interfaces between particle and substrate material under suitable milling conditions. The method is a solid state powder treatment process which is proved to be capable of synthesizing equilibrium alloys including solid solutions and intermetallic, non-equilibrium phases (such as supersaturated solid solutions, nanostructures and amorphous alloys) and composite materials. The method includes cold welding, fracturing and re-welding. The morphological changes of the starting particles are observed due to hard ball collisions throughout high energy ball milling. The ductile particles become smooth due to more ball collisions. Work hardening occurs because of severe plastic deformation. Moreover, the cold welded particles are available in the system [1, 2]. The passive oxide layer on metallic substrates are certainly broken and removed by severe ball impacts during ball milling. So, atomic diffusion between the milled powder and active substrate surface is improved. A heavy duty metallurgical bonding between the powder and substrate is definitely achieved at room temperature and atmospheric pressure [3, 4]. The ball milling is a multipart method that contains the optimization of many variables to produce best coatings. The process optimization is the most important fragment that reveals the efficiency of the production methods and then its profitable feasibility. The using of optimization softwares, such as Response Surface Methodology (RSM) has built reputation due to capability in optimizing the process parameters by decrasing the working time and production cost. Mostly, RSM is the process of gathering the statistical and mathematical methods that are beneficial for analysis and modeling in the applications where the concerned outcome is effected by some parameter [5-9].

Several researchers fabricated coating on different substrate materials using ball milling method. Shahzad et al. [10] prepared Ni-Al coating on hypo-eutectoid steel by high energy ball milling using pre-activated Ni-Al composite granules as coating material. They concluded that the mechanical alloying of 1 h produces the composite granules entirely in the ball mill and heat was formed during the milling process which facilitates the fabrication of denser and robust adhesive coating of Ni-Al intermetallic on the hypo-eutectoid steel substrate.

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Bafandeh et al. [11] studied coating on low carbon steel with Ni, Al and Fe powder mixture by mechanical alloying for the milling time of 2, 4, 8 and 12 h. According to their study, coating thickness of 65 µm was obtained after the milling time of 8 h and no crack on the coating was observed after milling time of 12 h. They reported that the increase in micro-hardness from 200 HV to 930 HV decreased corrosion current density for low carbon steel from 5.49 × 10− 6 A/cm2 to 3.16 × 10− 8 A/cm2 at the end of 8 h for milled and annealed sample, respectively.

Although, some studies [10, 11] reported in the literature on investigation of effect of milling parameters on the coating thickness, there has been no study that used RSM for a detailed systematic investigation. In the current study, both individual and interaction effects of several parameters on production of a thick and strong adhesive coating of Al powder on the low-carbon steel substrate were investigated.

2. EXPERIMENTAL PROCEDURE

The as-atomized Al powders (d_{50}: 128 µm) were used as the coating materials. Low-carbon steel plates (12 × 12 × 3 mm) were used as substrates. Central composite design (CCD) was adopted for the study. The effects of process parameters (milling speed, ball/powder ratio and milling time) in three levels were investigated. Table 1 shows the three parameters with corresponding coded and uncoded (actual) levels.

Table 1. Ball milling parameters investigated with corresponding levels.

Parameters	**Levels and Ranges**		
	Low (-1)	Medium (0)	High (1)
A-Milling speed (rpm)	200	300	400
B-Ball/powder ratio	5	10	15
C-Milling time (h)	2	5	8

A planetary ball mill (Fritsch Pulverisette 7, Premium line) was used in the milling tests. Tungsten carbide bowl and balls (10 mm in diameter) were used for milling. No process control agent was used in the tests. Milling chamber was sealed and argon atmosphere was used to avoid atmospheric pollution.

The phase analysis of steel substrate and Al coated sample were performed by X-ray diffraction (XRD) analysis. The XRD patterns were recorded in the 2θ range of 0 –100° (step size 0.02° and time per step 1 s). The coating thickness was measured by a scanning electron microscope (SEM). The average thickness was determined by measuring ten different distances from shortest to highest thickness for each sample (Figure 1).

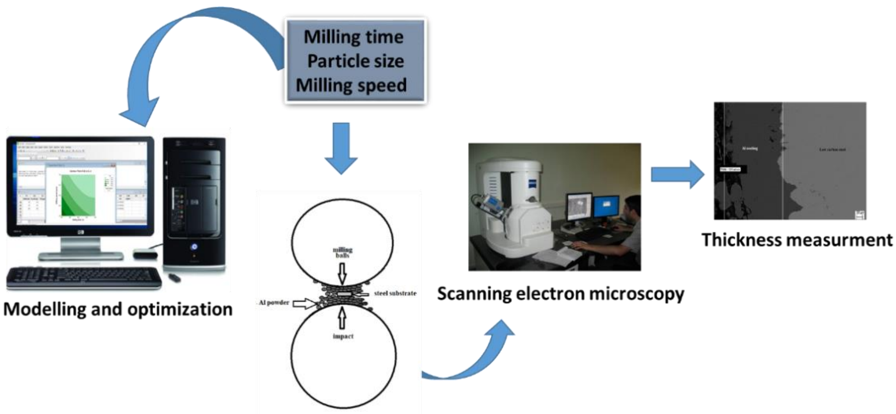

Figure 1. Demonstration of the main steps followed in the current study.

2. RESULTS AND DISCUSSION

Optimization of milling parameters for Al coating on low carbon steel substrate was performed by the CCD method. The experimental layout used in the current study with responses (i.e., coating thickness) of each test are given Table 2. The average measured thickness of Al coating varied from 7 µm to 164 µm. A second order polynomial model was built and shown in Equation (1). The regression coefficients of the model implied that ball/powder ratio had the major linear (main) effect on the ball milling process with a positive mode of effect.

$$\text{Coating thickness } (\mu m) = -412{,}8 + 2{,}115\ A + 27{,}0\ B - 4{,}9\ C - 0{,}00330\ A{*}A - 1{,}840\ B{*}B + 1{,}61\ C{*}C + 0{,}0460\ A{*}B - 0{,}0242\ A{*}C + 0{,}050\ B{*}C \quad (1)$$

Table 2. Experimental layout of the central composite desing with corresponding response of each test

Exp.No	Speed (rpm)	Ball-to-powder ratio	Time (h)	Coating thickness(µm)
1	200	5	2	7
2	400	5	2	79
3	200	15	2	10
4	400	15	2	141
5	200	5	8	56
6	400	5	8	66
7	200	15	8	29
8	400	15	8	164
9	200	10	5	24
10	400	10	5	150
11	300	5	5	36
12	300	15	5	112
13	300	10	2	106
14	300	10	8	163
15	300	10	5	138
16	300	10	5	138
17	300	10	5	138
18	300	10	5	138
19	300	10	5	138
20	300	10	5	138

The comparison between the experimental and predicted results (coating thickness) by the regression model is presented in Figure 2. It is apparent that the regression model can be suitably used for prediction of coating thickness. Table 3 shows the analysis of variance (ANOVA) indicating the statistical importance of the regression model and individual/interaction effects of parameters. The P-value for the model is <0.001 confirming that the model is statistically significant at a confidence level of 99.9%. The multiple coefficient of determinations (R2) of is the model was 0.938.

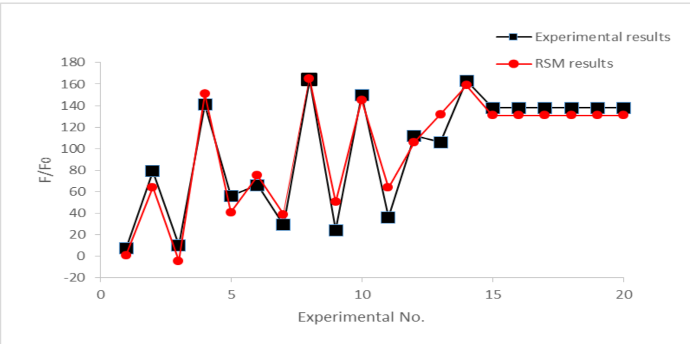

Figure 2. Residual values for coating thickness.

All the linear (main) effects of the parameters were found to be statistically important at a confidence level of 95% (Table 3) The quadratic effects of milling speed (A) and ball/powder ratio (B) as well as their interaction effect (AxB) was statistically significant at the same confidence level of 95%. While the quadratic effect of milling time (C) and its interaction effects (AxC and BxC) were found to be statistically insignificant.

Table 3. ANOVA results for coating thickness

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	52752,7	5861,4	16,79	0,000
A	1	22467,6	22467,6	64,37	0,000
B	1	4494,4	4494,4	12,88	0,005
C	1	1822,5	1822,5	5,22	0,045
A x A	1	2994,7	2994,7	8,58	0,015
B x B	1	5819,0	5819,0	16,67	0,002
C x C	1	578,2	578,2	1,66	0,227
A x B	1	4232,0	4232,0	12,13	0,006
A x C	1	420,25	420,25	1,20	0,298
B x C	1	4,5	4,5	0,01	0,912
Error	10	3490,2	349,00		
Total	19	56242,9			
R^2 =	0,938				

First stage in the coating process by ball milling is constant impacts by balls with high energy on the substrate surface which is squeeze in powders between ball and substrate material. Second stage is cold welding of particles. Final stage is delamination of the coated deposit [12]. As can be seen in Figure 3, the effect of the dual factors on the coating thickness can be clearly observed from surface and contour plots. The value of the coating thickness is maximized while the medium value of ball-powder ratio at high milling speeds (Figure 3a). The contour and surface plots show that the optimum level of ball-powder ratio for the maximum coating thickness is the medium level of 10:1. At the lowest level of ball-powder ratio (5:1), the steel substrate is coated with thin Al coating (Figure 3a). At low ball/powder ratios the steel substrate suffers from few plastic deformations under ball-substrate-ball collisions and it becomes faintly deformed. Energy is insufficient to deposit more Al powders on the steel substrate. The increase in the number of ball-substrate-ball collision occurs at the higher ball-powder ratio. In addition, it also causes the cold welding of more Al particles to the steel surface. Hence, the thickness of Al coatings increases. The highest milling speed at medium level of ball-to-powder ratio increases the energy response into the powder per part time. As ball-powder ratio varied from 5:1 (-1) to 10:1 (0), increase in the number of the balls results in decrease of free alleyway of the balls. However, the number of collisions per unit time increases, thus extra energy is transmitted to the powder particles from balls and ball milling process is more effective which results in maximum coating thickness [13]. Further increasing in ball/powder ratio from 10:1 (0) to 15:1 (+1) results in a decrease in coating thickness which is more apparent at low milling speed (Figure 3a).

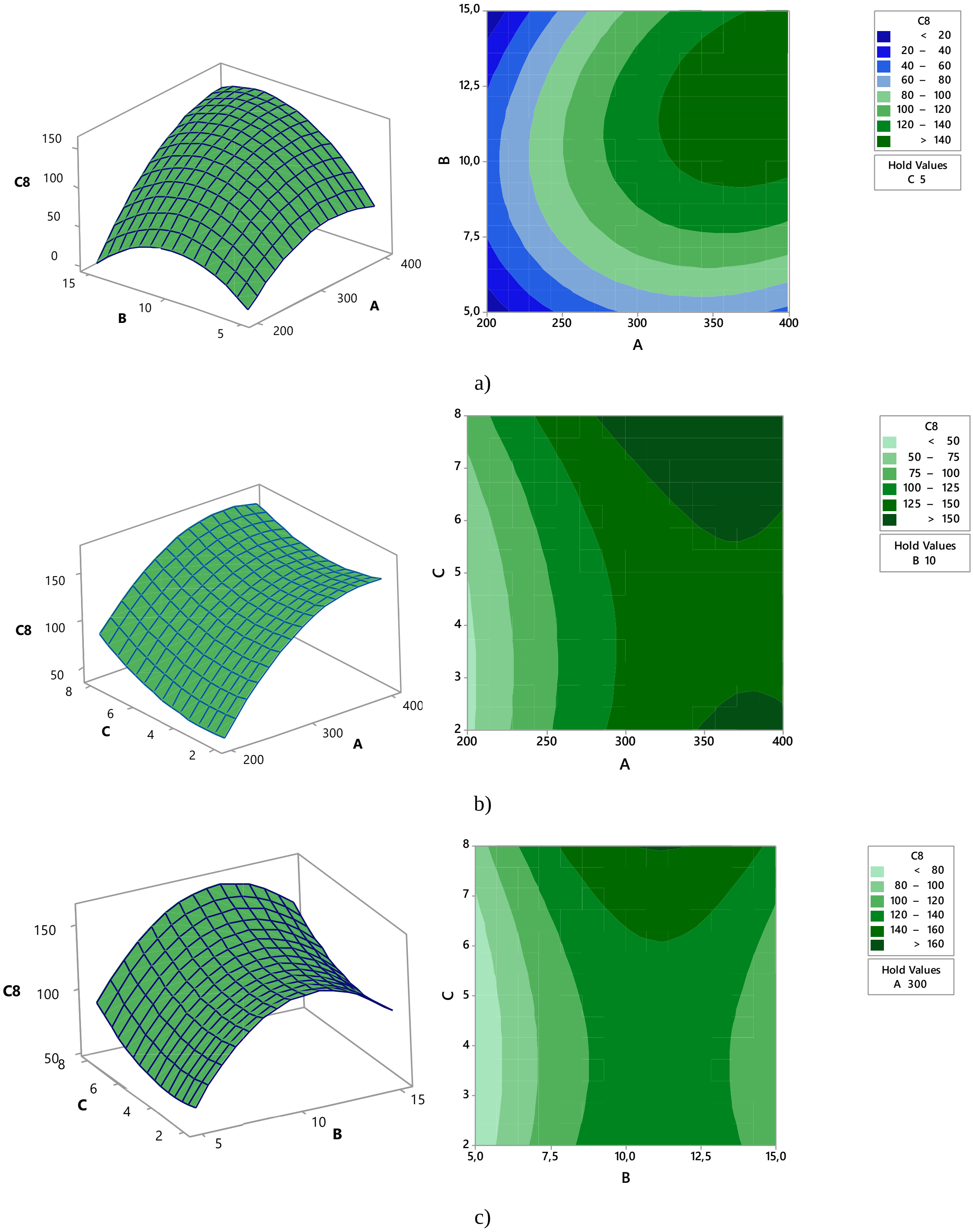

Figure 3. Surface and contour plots showing the effects of; a) ball/powder ratio and milling time, b) ball/powder ratio and milling speed, c) milling speed and milling time on the coating thickness.

The cross-sectional images of Al coatings with respect to the changes in milling parameters is presented in Figure 4. Dark region represents the Al coatings while light area refers to low carbon steel substrate. At a milling time of 5 h, the coating surface presented to be smooth. However, uneven coating surface was observed at milling time of 2 h. This can be attributed to fact that Al particles are suffered from more impacts from the extra ball-powder-ball collision with suitable milling parameter which results in more adhesive coating on the low carbon steel substrate.

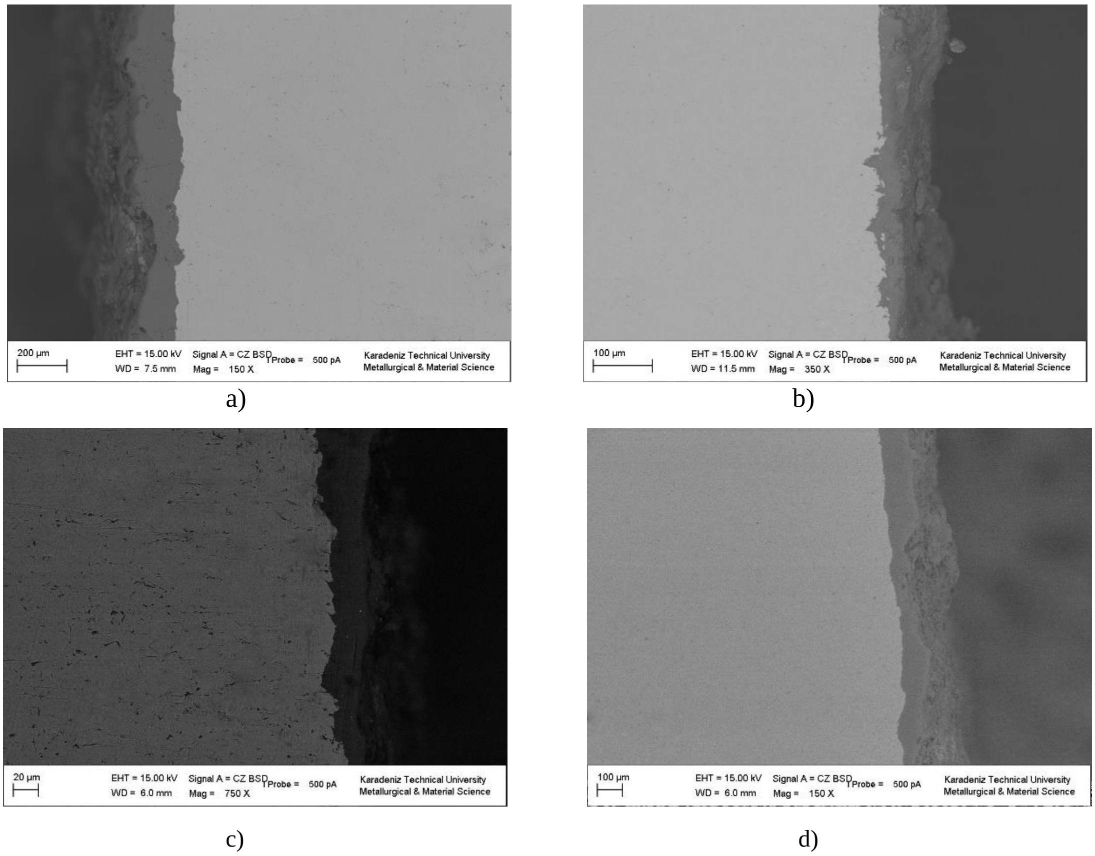

Figure 4. SEM images of coatings formed from different experiment number: (a) 4, (b) 5, (c) 11 and (d) 12.

XRD patterns of low carbon steel and Al coated samples are presented in Figure 5. XRD analysis confirmed the presence of Al and Fe in coated sample.

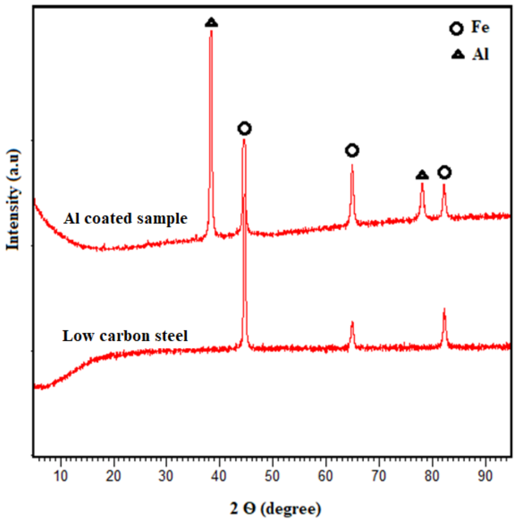

Figure 5. XRD patterns of steel substrate and Al coated sample of number 4 experiment.

4. CONCLUSIONS

Following conclusions can be drawn from the experimental results:

1. Al coating on low carbon steel substrate was obtained using ball milling method. Response surface methodology, a robust statistical design of experimental method, was adopted to reveal the individual and interaction effects of the parameters on coating thickness.

2. A second-order regression model with a multiple determination of correlations of 93.8% was established. Regression coefficients indicated that the most effective parameter was determined to be the ball/powder ratio. The statistical analysis of the data demonstrated that all linear effects of the parameters, quadratic effects of milling speed and ball/powder ratio and its interaction effect were found to be statistically significant at a confidence level of 95%.

3. Maximum coating thickness of 164 μm was achieved under the conditions of ball/powder ratio of 15:1 at a milling speed of 400 rpm.

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