

# A STUDY ON THE EFFECTS OF PLASMA SPRAYING PARAMETERS ON THE ADHESION STRENGTH OF Cr<sub>3</sub>C<sub>2</sub>-NiCr COATING ON 16Mn STEEL

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## Abstract

This paper experimentally studied the adhesion strength of Cr<sub>3</sub>C<sub>2</sub>-30 % NiCr coating created on 16Mn steel substrate by plasma thermal coating technique in relation to spraying parameters. Experiments were carried out according to the central composite design (CCD) experimental matrix with three parameters: current intensity, powder feeding rate, and spray distance. Samples consisting of an annular disc and a latch made of 16Mn were fabricated according to the JIS H8664-1977 standard. Cr<sub>3</sub>C<sub>2</sub>-30 % NiCr coating was then created on the top surface of the disc including end of the latch. Adhesion strength of the coating to the substrate was measured through the tensile test. ANOVA analysis of variance was performed to evaluate the influence of the spraying parameters on adhesion strength and to build an empirical regression function representing the relationship between those parameters and the adhesion. Optimization problem was solved by ANOVA method and genetic algorithm (GA) to determine the value of the spraying parameters at which the coating has the greatest adhesion strength to the substrate. The results showed that the spraying parameters greatly affected the adhesion of the Cr<sub>3</sub>C<sub>2</sub>-30 % NiCr coating to the 16Mn substrate. Among them the spray distance has the greatest influence while the powder feeding rate has the least. Secondly, the regression function was well reflected the relationship between the three parameters and adhesion strength of the coating on the substrate. Using the values of spray parameter obtained from the GA optimization to create Cr<sub>3</sub>C<sub>2</sub>-30 % NiCr coating on 16Mn steel, the adhesion strength of the coating to the substrate reached a value of 98.4 % compared to the prediction.

**Keywords:** Plasma thermal spraying, Cr<sub>3</sub>C<sub>2</sub>-30 % NiCr, 16Mn, Spraying parameters, Adhesion strength, ANOVA, Genetic Algorithm.

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## 1. Introduction

Thermal plasma spraying is well-known for producing high-quality coatings with many desirable properties such as abrasion resistance, corrosion resistance, heat resistance, and environmental protection, etc. [1–4]. In the process of plasma spray, the spray current has a direct influence on the anode and the anode affects the arc power fluctuation, which results in a strong influence on the temperature, velocity, and trajectory of the spray particles [5, 6]. Yuyan Wang and the colleague reported that if the power source is too small, horizontal cracks and gas pitting are more likely to occur, however, if the power source is too high, longitudinal cracks can occur [7]. Next, the powder feed rate is also one of the parameters that have a significant influence on the formation and quality of the coating. Some studies have shown that when the powder feed rate is small, the spraying capacity will be reduced because the dispersion causes the loss of spraying particles, which leads to the fact that one spraying can not fully cover the surface. In addition, the results of some other studies show that when the powder feed rate changes, the temperature and particle speed change accordingly. For each specific plasma spraying deposition process, there is an optimal distance that depends on the arc current, the plasma air flow rate, the type, and size of the spray powder. At a short spray distance, with high particle velocity, the porosity is often high. With a long spraying distance, some particles are re-solidified because they

are cooled down, resulting in high porosity and low deposition efficiency. Fincke et al. found an inverse relationship between porosity and deposition efficiency. The efficiency decreases if the spray distance increases [8].

Among plasma thermal coatings, the  $\text{Cr}_3\text{C}_2\text{-NiCr}$  is one of the most popular use. The coating is corrosion and oxidation resistant, has high hardness, good mechanical properties, and resists high-temperature wear [9]. From the results of previous works,  $\text{Cr}_3\text{C}_2\text{-NiCr}$  coatings with different carbide content have been widely used to resist abrasion at high temperatures (up to  $800\div 900\text{ }^\circ\text{C}$ ) and to resist corrosion in an environment with an erosion agent [10]. The corrosion resistance of the  $\text{Cr}_3\text{C}_2\text{-NiCr}$  coating is involved in the interactions between the carbide components and NiCr. Another study found that when heat-treated at  $980\text{ }^\circ\text{C}$ , the thermal conductivity of the  $\text{Cr}_3\text{C}_2\text{-NiCr}$  coating fabricated by plasma coating method increases significantly [11]. Simultaneously, as the amount of chromium carbide in the spray powder increases, so does the erosion resistance of the ceramic-metal coating.  $\text{Cr}_3\text{C}_2\text{-NiCr}$  powder consists of many different elements, each of which plays a specific role in the coating, namely: Ni contributes to increasing the plasticity between the particles to form a bonding layer, while  $\text{Cr}_3\text{C}_2$  contributes significantly to the hardness of the coating. This powder is ideal for spraying new fabrications or restoring wear- and temperature-resistant parts. A recent study by D. X. Thao et al showed that increasing the carbide content from 50 % to 80 % by weight-reduced coating adhesion by three times, from 43.58 MPa to 16.32 MPa. Meanwhile, coating hardness increased about 2 times from 411 HV to 701 HV [12]. 16Mn alloy steel is known as a material commonly used to manufacture machine parts in wear-resistant environments, such as coal powder loading propeller, boiler ash directional propeller and smoke impeller in coal-powered thermal power plants and in cement plants [13]. In plasma coating technique, many published experimental study and modeling study related to the spraying process have shown that the performance and microstructure of ceramic metal coating are mainly affected by spray parameters [14–16]. However, little has been published about the complex interaction of plasma spray parameters affecting the adhesion strength. Especially, there has been no publication studying the simultaneous effects of spray parameters on the rating criteria of adhesion strength ( $\sigma_{\text{AS}}$ ) of the coating  $\text{Cr}_3\text{C}_2\text{-30\% NiCr}$  on 16Mn alloy steel.

In this study, experiments were carried out to investigate the effects of 3 spray parameters, which are spray current, powder feeding rate, and spray distance on the adhesion strength of the coating  $\text{Cr}_3\text{C}_2\text{-30\% NiCr}$  on 16Mn steel substrate. The 16Mn steel is known as a material commonly used to manufacture machine parts in wear-resistant environments, such as coal powder loading propeller, boiler ash directional propeller and smoke impeller in coal-powered thermal power plants and in cement plants [13]. An empirical regression function representing the relationship between those parameters and the adhesion strength was built. Solve the optimization problem with the criterion of adhesion strength as high as possible using the ANOVA analysis and genetic algorithm. The optimal values of the spray parameters are experimentally verified to evaluate the accuracy of the predictive model.

## 2. Material and method

The sample including a disc and latch body made of 16Mn steel is fabricated according to the JIS H8664-1977 standard in **Fig. 1**. Before coating, disc surface was cleaned and roughened by abrasive blasting. The roughness of the disc surface was  $R_z \sim 1\text{ }\mu\text{m}$  for the good adhesion is given [17].

The Plasma spray equipment 3710-PRAXAIR-TAFA (USA) was used for coating. The 1264 powder feeding system that operates on a volumetric feed principle and directly controls the powder feed rate by varying the rotation speed of the disk and the spray gun SG-100 with external powder injection were used.

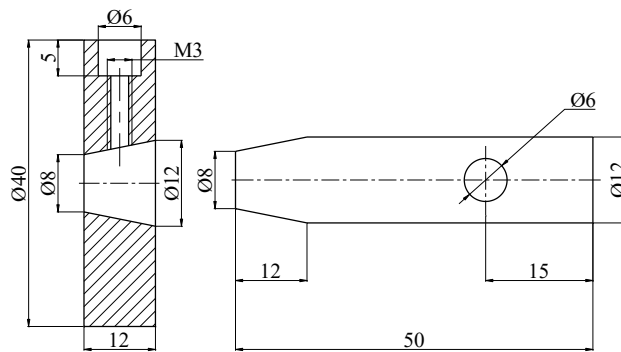
$\text{Cr}_3\text{C}_2\text{-30\%NiCr}$  powder (Sulzer Metco – Singapore) is chosen for coating in this study. The size of  $\text{Cr}_3\text{C}_2\text{-30\%NiCr}$  particles is about  $-30/+5\text{ }\mu\text{m}$  and the chemical composition is  $\text{C} \leq 0.2\text{ \%}$ ,  $\text{Si} \leq 0.5\text{ \%}$ ,  $\text{NiCr} 29.5\text{ \%}$  and  $\text{Cr}_3\text{C}_2 69.8\text{ \%}$ .

The adhesion strength is determined using a tensile compression tester (Model BESTUTM 500HH, Korea). The principle of the tensile test is showed in **Fig. 2**. In the test, the disc including

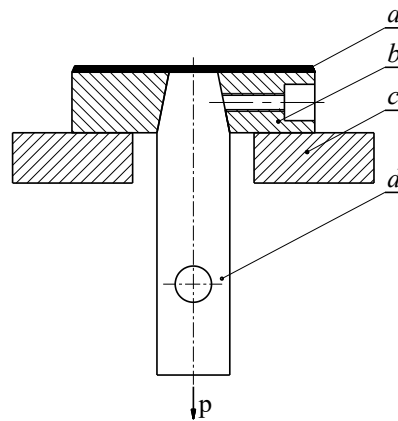
the coating on the top (with surface area  $F$ ) is separated from the latch by the force ( $P$ ) pulled in a perpendicular direction, as shown in **Fig. 2**. The adhesion strength ( $\sigma_{As}$ ) is defined as (1):

$$\sigma_{As} = \frac{P}{F}, \text{ MPa.} \quad (1)$$

In this study, the experiment was designed according to Central Composite Design (CCD) method with  $2^k$  factorial experiments (coded as  $-1$  and  $+1$ ), 6 central points (coded as 0) and  $2^k$  axial points (placed at  $-\alpha$  and  $+\alpha$ , where  $(\alpha = \sqrt[4]{2^3} = 1.682)$  [18]. The values of each input at all the levels are shown in **Table 1**. Matrix of experiment is presented in **Table 2**.



**Fig. 1.** Drawing of 16Mn sample for tensile test including the latch disc (left) and the latch body (right)



**Fig. 2.** Principle of the tensile test:

$a$  – Coating;  $b$  – Latch disc;  $c$  – Overriding plate;  $d$  – Tensile latch;  $p$  – Applied force

Spraying process was performed at a voltage of 35 V, along with the main air flow was 50 l/min, the secondary air flow was 5 l/min, and the spray angle was  $90^\circ$ . Adhesion measurement was carried out at the room conditions. The thickness of the coating is about 1000  $\mu\text{m}$ .

**Table 1**  
Input parameters value at levels

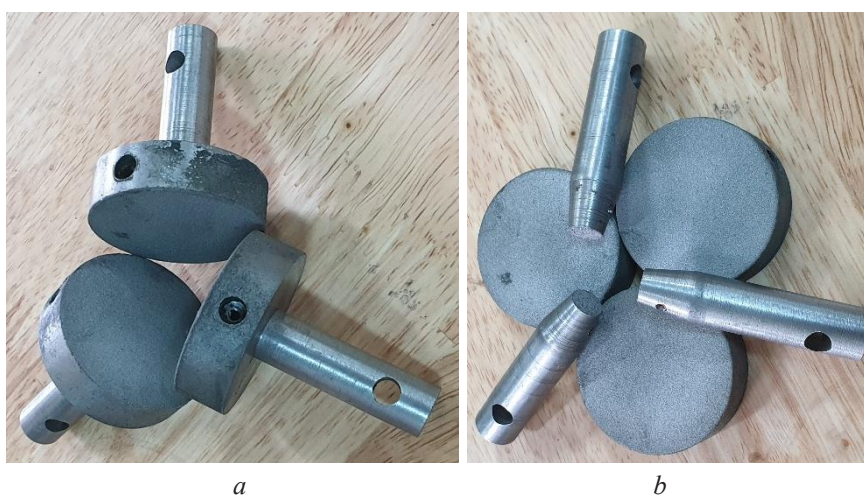
Parameter	Symbol	Unit	Values at levels				
			$-\alpha$	$-1$	0	1	$+\alpha$
Spray current	$I_s$	A	381.8	450	550	650	718.2
Powder feed rate	$m_s$	g/min	13.18	20	30	40	46.82
Spray distance	$L_s$	mm	92.72	120	160	200	227.28

**Table 2**  
Experimental matrix and results

No.	Spray parameters			Measurement results
	$I_s$ (A)	$m_s$ (g/min)	$L_s$ (mm)	$\sigma_{As}$ (MPa)
1	450	20	120	21.9
2	650	20	120	33.5
3	450	20	200	29.8
4	650	20	200	34.5
5	450	40	120	30.6
6	650	40	120	31.8
7	450	40	200	35.1
8	650	40	200	31.9
9	550	30	160	38.5
10	550	30	160	38.4
11	550	30	160	39.2
12	550	30	160	38.6
13	550	30	160	38.3
14	550	30	160	38.0
15	381.82	30	160	28.3
16	718.18	30	160	32.5
17	550	13.18	160	29.6
18	550	46.82	160	32.1
19	550	30	92.73	25.5
20	550	30	227.27	31.7

### 3. Results and discussion

**Fig. 3** shows the sample after fabrication and being coated (**Fig. 3, a**) and after the tensile test (**Fig. 3, b**). It is clearly seen that the disc was not damaged and the coating was remained on the top surface of disc after tensile tests.



**Fig. 3.** Sample test: *a* – Samples before the tensile test; *b* – Samples after the tensile test

The results of analysis of variance (ANOVA) for adhesion strength of coatings are shown in **Table 3**. The statistical results in this table show that: for the linear model, the spray distance has

the greatest effect on adhesion strength (9.89%/21.12%), followed by the current (7.89%/21.12%), and the powder feeding rate has the least effect compared to the two parameters mentioned above (3.34%/21.12%). The interaction of the parameters on adhesion strength has a greater impact, the results are  $L_s * L_s = 33.54\%$ ,  $m_s * m_s = 14.19\%$ ,  $I_s * I_s = 13.97\%$ ,  $I_s * m_s = 9.88\%$ ,  $I_s * L_s = 3.77\%$ , and finally  $m_s * L_s = 0.55\%$ , respectively.

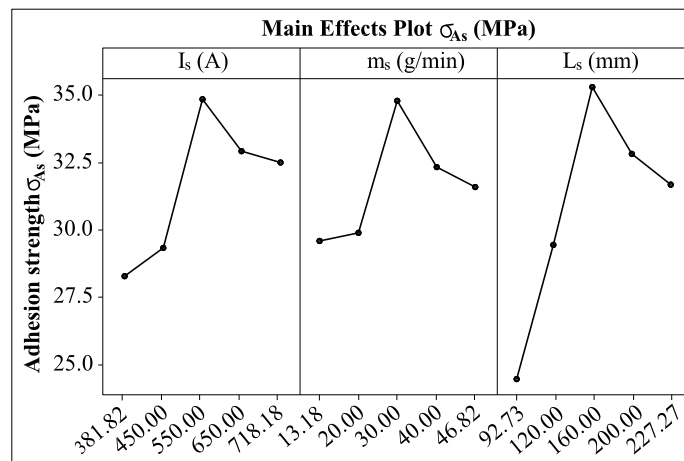
**Table 3**  
ANOVA analysis method for the coating adhesion strength

Source	DF	Seq SS	Contribution	Adj MS	F-Value	P-Value
Model	9	411.072	97.02 %	45.675	36.12	0.000
Linear	3	89.496	21.12 %	29.832	23.59	0.000
$I_s$	1	33.419	7.89 %	33.419	26.43	0.000
$m_s$	1	14.156	3.34 %	14.156	11.19	0.007
$L_s$	1	41.921	9.89 %	41.921	33.15	0.000
Square	3	261.442	61.70 %	87.147	68.91	0.000
$I_s * I_s$	1	59.188	13.97 %	90.347	71.44	0.000
$m_s * m_s$	1	60.143	14.19 %	79.228	62.65	0.000
$L_s * L_s$	1	142.111	33.54 %	142.111	112.38	0.000
Interaction	3	60.134	14.19 %	20.045	15.85	0.000
$I_s * m_s$	1	41.861	9.88 %	41.861	33.10	0.000
$I_s * L_s$	1	15.961	3.77 %	15.961	12.62	0.005
$m_s * L_s$	1	2.311	0.55 %	2.311	1.83	0.206
Error	10	12.646	2.98 %	1.265	–	–
Lack-of-Fit	5	11.846	2.80 %	2.369	14.81	0.005

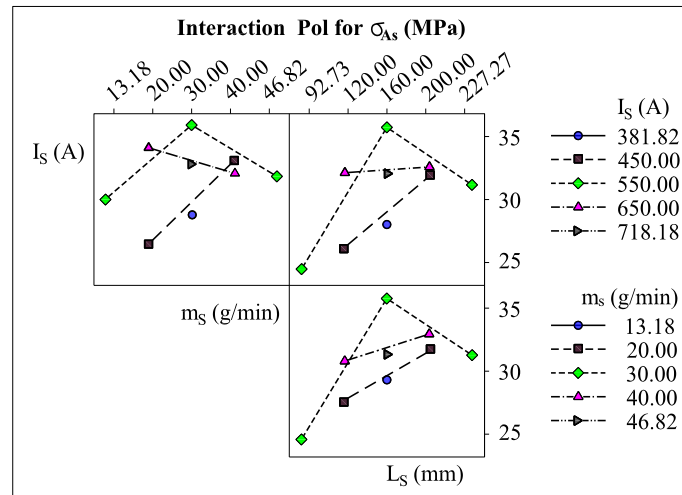
$$R-S_q = 97.02\%; R-S_q(\text{adj}) = 94.33\%$$

When considering the influence of single factors and the interaction between them, the analysis results show that all factors have an influence on the adhesion strength of the coating to the steel substrate. Among them, the  $L_s * L_s$  was the most influential (33.54%).

It is suggested to refer to **Fig. 4** to gain a better understanding of each element's effect, whereas the reciprocal interaction of each pair of elements is depicted in the surface response diagram of the **Fig. 5**.



**Fig. 4.** Effect of spraying parameters on the coating adhesion strength



**Fig. 5.** Effect of the interaction between spraying parameters on the coating adhesion strength

It could be seen from the figures that when the spray current increases from (381.82÷550) A, the powder feed rate increases from (13.18÷30) g/min, and the spray distance increases from (92.73÷160) mm, the adhesion strength is increased significantly. However, as the values of those parameters continue to increase, the adhesion tends to decrease.

This can be explained as follows:

- continuously increasing the spray current from (381.82÷550) A also cause the arc capacity to gradually raise to the melting point for more spray particles, which aids in the deposition and bonding of the coating to the substrate or another coating. If continuing to increase spray current from (550÷718.18) A, a very large arc capacity will be generated, causing some of the burnt particles to form oxides in the coating, resulting in decreased adhesion, coating hardness and increased porosity;

- due to the decreased flow of powder into the arc area, a high temperature concentration occurs, increasing the porosity of the burnt particles and reducing the bonding of the coatings. If the increases gradually powder feed rate from (13.18÷30) g/min, the temperature of the particles decreases gradually due to the thermal dispersion and when the amount of spray particles supplied to the arc area is just enough to melt the particles, resulting in the best coating deposition. The powder feed rate into the spray area is too high, resulting in a decrease in temperature, particle velocity and the quality of the coating deposition;

- spraying distance is the parameter that has the greatest impact on the adhesion strength between the coating and the steel substrate. When the spraying distance is short, the particle velocity and the porosity is high because when the air pressure is high, particles in liquid status collide with the surface and bounce out. At this time, there are mostly large size particles in solid status stacking with each other creating empty holes. At a long spraying distance, some particles are re-solidified because they are cooled down, resulting in a coating with high porosity and low deposition.

The experiment process is performed according to the sequence in **Table 2**. The adhesion strength value at each experiment is also included in this table. Use ANOVA method to build a quadratic polynomial regression model to predict coating adhesion strength as follows:

$$\begin{aligned} \sigma_{As} = & -202.6 + 0.4162I_s + 2.981m_s + 0.9064L_s - 0.000250I_s * I_s - 0.02344m_s * m_s - \\ & - 0.001963L_s * L_s - 0.002288I_s * m_s - 0.000353I_s * L_s - 0.00134m_s * L_s. \end{aligned} \quad (2)$$

ANOVA results for criteria of adhesion strength are presented in **Table 3**. First of all, it is found that in testing the appropriateness of the regression model (Lack-of-Fit), the value of  $P$  is much greater ( $\gg$ ) than the significance level. This means that the model is consistent with the data. Considering the values of each individual component of the regression model (Linear, Square, and Interaction), it is possible to see that the values of  $P$  of these components are all very small (less than  $<0.05$ , statistically significant). That is, the presence of each of these components

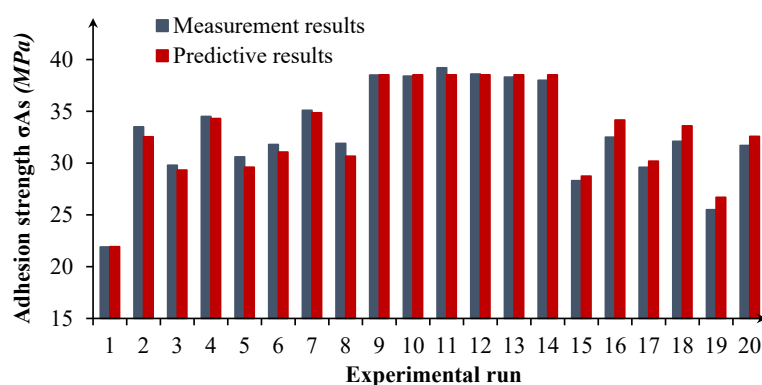
is of high significance in the regression model [18]. The regression coefficient ( $R^2$ ) is calculated to have 97.02 % of experimental data compatible with the predicted data according to the model.  $R^2(\text{adj}) = 94.33$  % match.

The regression model was used to predict adhesion strength. The comparison results between predicted and experimental adhesion strength are listed in **Table 4** and described in **Fig. 6**.

**Table 4**

Comparison of the experimental and predicted values of adhesion strength

No.	Measurement results	Predictive results	Deviation (%)
1	21.9	21.9	0.1
2	33.5	32.5	2.8
3	29.8	29.3	1.6
4	34.5	34.3	0.6
5	30.6	29.6	3.3
6	31.8	31.1	2.3
7	35.1	34.9	0.7
8	31.9	30.7	3.9
9	38.5	38.5	0.1
10	38.4	38.5	0.3
11	39.2	38.5	1.7
12	38.6	38.5	0.2
13	38.3	38.5	0.6
14	38.0	38.5	1.4
15	28.3	28.7	1.6
16	32.5	34.2	5.1
17	29.6	30.2	2.0
18	32.1	33.6	4.7
19	25.5	26.7	4.7
20	31.7	32.6	2.8
Average deviation			2.0



**Fig. 6.** Comparison between the experimental adhesion strength and the predicted adhesion strength

The comparison results show that the predicted adhesion strength is extremely close to the measured adhesion resistance, the maximum deviation is 5.1 %, the average deviation is 2.0 %. Thus, the identified regression model has been successfully tested and can be applied to predict adhesion strength as well as to optimize the criteria.

Solve the problem of optimal adhesion strength criteria with the desired size as much as possible with two different methods, ANOVA analysis method and application of genetic algorithm. Based on experimental results and regression model to predict adhesion strength with spray parameters are spray current, powder feeding rate and spray distance as formula (2). This is the basis for determining the value of parameters when spraying plasma with Cr<sub>3</sub>C<sub>2</sub>-30 % NiCr powder to achieve coating adhesion with 16Mn substrate metal steel. In addition, this model is also used to predict the adhesion of the coating to the substrate metal for different spray parameters (in the survey area) when spraying by plasma injectors. Applying Minitab 19™ software, optimization problem solving results of the model with the adhesion strength goal as large as possible and the binding conditions for technological parameters in the survey area, obtained the results as shown in Fig. 7.

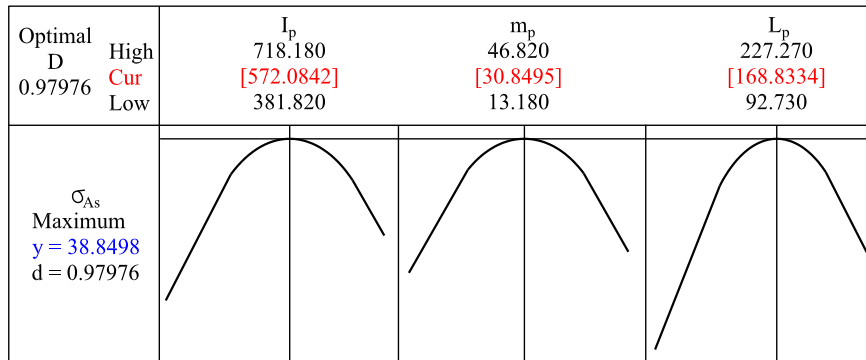


Fig. 7. The graph shows the values of the spraying parameters for maximum adhesion strength

In addition, use for genetic algorithm (GA) with the following parameters: The population is 100, the hybrid probability is 0.25, the mutation probability is 0.05, the mutation parameter is 4, the result of the optimization problem is described in Fig. 8.

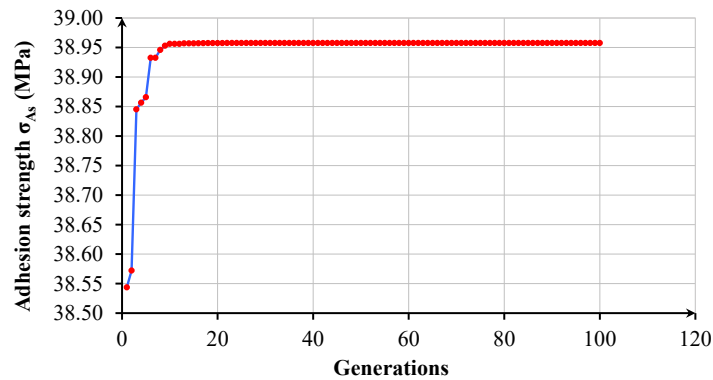


Fig. 8. Optimal results using Genetic Algorithm

The results of solving optimal problems with adhesion strength criteria by two methods are listed in Table 5. The results of comparing the two methods show that the optimal value of the technological parameters found by these methods is very similar (the largest difference is 0.28 %). Thus, this set of optimal value parameters is the best technology parameter with the criteria for the greatest adhesion strength.

Verification of optimal values on sample was performed with spray parameters according to GA:  $I_s = 572$  A,  $m_s = 30.8$  g/min and  $L_s = 169.0$  mm. The controlled experiment in this study was performed on 5 samples. The adhesion strength of five measurements is shown in Table 6. Compared with the calculated value of the adhesion in Table 5 (by GA) it is seen that the difference between two values is about 1.6 %, completely acceptable. Thus, it could be affirmed that these optimal spray parameters are the best parameters with the criterion of maximum adhesion strength and high reliability.



**Table 5**  
Comparison of the ANOVA and GA methods

Solution	$I_s$	$m_s$	$L_s$	$\sigma_{As}$ (Fit)
ANOVA	572.0842	30.8495	168.8334	38.8498
GA	572.0126	30.8424	168.9125	38.9576
Difference (%)	0.01	0.02	0.05	0.28

**Table 6**  
Adhesion strength measured on samples to verify optimal values

Sample	1	2	3	4	5	Average results
Measurement results Adhesion strength	37.6	39.1	38.5	37.8	38.7	38.34 (MPa)

The spraying process has many parameters that affect the adhesion of the coating to the substrate. In order to understand the nature of the influence of coating parameters on adhesion, especially the influence of the interaction between coating parameters, it is necessary to conduct more in-depth theoretical studies along with practical experiments for verification. That is the direction of future research.

#### 4. Conclusion

The spray distance, spray current and powder feeding rate strongly affected the adhesion strength of the coating to the steel substrate, in which the spray distance exhibited the highest influence and the powder feeding rate showed the smallest one. However, the interaction of parameters showed greater influence on the adhesion strength compared to the single one.

ANOVA analysis and Genetic Algorithm were successfully applied to solve the optimal problem with the goal objective of achieving the spray parameters for the adhesion strength index as large as possible. The results obtained by two methods showed remarkably similar (the largest difference is 0.28 %). Thus, the values of these parameters could be used to give the highest adhesion strength of the  $\text{Cr}_3\text{C}_2$ -30 % CrN coating on 16Mn steel substrate.

Applying the values of spray parameters obtained from optimization to create a  $\text{Cr}_3\text{C}_2$ -30 % CrN coating on the 16Mn substrate, the adhesion strength of the coating to the substrate was about 38.34 MPa, accounting for 98.4 % compared to the prediction. This proves that our prediction model has high accuracy and is close to the reality.

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