MINISTRY OF EDUCATION AND TRAINING MINISTRY OF INDUSTRY AND TRADE

NATIONAL RESEARCH INSTITUTE OF MECHANICAL ENGINEERING

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RESEARCH ON THE PLASMA SPRAY TECHNOLOGY CREATE SILICON CARBIDE COATING ON STEEL SUBSTRATE TO PROTECT AGAINST CORROSION IN THE FLUORINE-CONTAINING ACIDIC MEDIUM

Field of study: Mechanical engineering

Code: 9520103

SUMMARY OF DOCTORAL THESIS

Hanoi - 2022

The thesis is completed at

National Research Institute of Mechanical Engineering, Ministry of Industry and Trade

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INTRODUCTION

1. Reason for choosing the topic

Metal wear and corrosion is a state of destruction of equipment and works that takes place continuously under the mechanism of abrasion and corrosion due to the influence of the working environment, causing economic and labor losses.

In chemical plants, equipment that always works in harsh conditions is quickly destroyed by mechanical and chemical wear. Through survey and evaluation at a number of chemical plants, equipment in the production line is often damaged very quickly due to chemical corrosion, especially equipment working in harsh conditions containing hazardous elements. strong oxidizing agents, acids and high temperatures. At Lam Thao Super Phosphate and Chemical Plant, equipment is often damaged due to corrosion, causing imbalance, deformation... Some details can be listed such as: Turbine, propeller, solution pump, Reactor tank, storage tank.

Anti-corrosion is a very important and necessary task in industry, in economy, in cultural and social works... in order to improve operational efficiency and prolong the life of products before the destruction of environment, especially harsh chemical environment. It is one of the research tasks of the past, present and future.

One of the methods of protection against wear and corrosion is the separation coating. Many works around the world focus on creating better thermal spray coatings to protect against mechanical wear and chemical corrosion.

In Vietnam, spray coating is still a new field. New studies have focused only on coatings that restore size due to mechanical wear, and very few studies on thermal coatings that resist wear in chemical environments.

Silicon carbide (SiC) is known as a special material used in coatings for equipment working in corrosive environments because it has very good wear resistance and corrosion resistance.

Thesis: "*Research on the plasma spray technology for fabrication of SiC/Cu plasma coating on steel substrate to protect against corrosion in the fluorine-containing acidic medium* " is a new domestic research work in this field, which is of great scientific significance. , respond positively to the urgent domestic demand.

2. Research purpose

- Create SiC-Cu plasma coating on steel surface.

- Research on some important properties of SiC-Cu plasma coating technology on steel surface to apply anti-corrosion protection for machine parts working in HF-containing environments.

3. Objects and scope of the thesis

3.1. Objects of the thesis

Effect of 3 parameters of current I, injection distance L, powder feed flow M on the quality of SiC-based plasma coating on steel surface. The wear resistance of the resulting coating.

3.2. Scope of the thesis

Research on some typical properties on the influence of plasma spraying technology parameters on the quality of SiC - Cu coating on steel substrates, including:

- Technical specs:	The basic criteria of the coating that need to be surveyed and evaluated:
+ Spray particle size - W(µm).	+ Thickness
+ Mixing content -S (%).	+ Adhesion strength.
+ Amperage - I (A).	+ Porosity.
+ Spray distance - L (mm).	+ Micro hardness.
+ Powder feeding speed - M (g/min).	+ Corrosion resistance of steel in fluorine-containing acidic medium

4. Scientific significances of the thesis

- The study gives the particle size and SiC-Cu mixing content to ensure the coating formation process meets the requirements for further research.

- Analyze the plasma coating process, improve the spray gun in Ar shielding gas, design and manufacture a mixed wear test device compatible with actual conditions, build a test procedure and evaluate the corrosion-wear resistance of coating.

- Establishing a suitable set of technological parameters for creating SiC/Cu plasma coating on steel surface

- Data processing by Taguchi experimental planning plan, using ANOVA to evaluate the relationship between technological parameters and achieved quality criteria..

5. New findings of the thesis

- This is the first project in Vietnam to study the technology of creating SiC/Cu plasma coating on steel surface.

- Improved method of plasma spraying technology in the air by plasma spraying in Ar shielding gas.

- Finishing SiC/Cu coating on steel surface by PTFE penetration.

- Design and manufacture of composite wear equipment to provide a method of synthetic wear assessment.

6. Pactical significances of the thesis

- The research results of the thesis contribute to creating a solution to protect against wear for equipment and works working in chemically corrosive environments, namely creating protective coatings on impellers and pump impellers. working in a fluorine-containing environment at Lam Thao Superphosphate and Chemical Factory.

- Contributing to improving the level and supplementing technical documents for the domestic water spray technology industry.

7. Content of the thesis

Chapter 1: Overview of anti-corrosion thermal coatings

Chapter 2: Technology to make SiC plasma coating on steel base

Chapter 3: Materials and research methods

Chapter 4: Fabrication, analysis and evaluation of SiC plasma coatings on steel substrates

Chapter 5: Determination of technological parameters for SiC-50Cu plasma spraying on C45 steel substrates

Chapter 1. OVERVIEW OF ANTI-CORROSION THERMAL SPRAYING COATING

1.1. Research situation on thermal spray coating in Vietnam

In Vietnam, the thermal spray coating method is in the research and application stage of the world's results. There have been a number of researches and applications of thermal spray coating technology such as: Vung Tau petroleum joint venture; MAR 60 Mechanical Company - Irrigation, Mechanical Research Institute; Institute of Energy - Mines; The Institute of Technology of the Ministry of Defense, etc. has applied electric arc spraying technology with spray wire… to create an Al, Zn anti-rust surface layer. Facilities such as Irrigation repair company. Traffic Engineering Institute; Mechanical Quang Trung; Hanoi Polytechnic University...

1.2. Research situation on thermal spray coating in the international contries

1.2.1. Research situation on thermal spray coating in the international contries

Spray coating technology has been developing strongly in advanced countries such as the US, Japan, Russia, UK, France, the German Federation, Switzerland, etc. work in creating coatings with special properties from materials such as ceramics, carbides, alloys, etc., which are used for wear resistance and corrosion protection purposes.

1.2.2. SiC coating for corrosion protection of steel in corrosive environments

Silicon carbide (SiC) is a ceramic material with an excellent combination of mechanical and chemical factors: SiC has a very high microscopic hardness, and is not reactive to strong acids and bases. SiC has very good wear resistance in corrosive environments (Table 1.1). SiC material has a melting point of 2730 °C, so it can work at high temperatures of about 1000-1500ºC. Research on fabrication of SiC coating is the direction of many research groups around the world.

Table 1.1. Corrosion resistance of ceramic materials in chemical environments [2, 3].

Testing media		Corrosion rate (mg/cm^2.year)			
Chemicals	**Temp.(°C)**	**Si/SiC composite (12% Si)**	**WC (6% Co)**	**Al_2O_3 (99%)**	**SiC (0% Si)**
98% H_2SO_4	100	55,0	> 1000	65,0	1,8
50% NaOH	100	> 1000	5.0	75,0	2.5
53% HF	25	7,9	8,0	20,0	<0.2
85% H_3PO_4	100	8,8	55,0	> 100	<0,2
70% HNO_3	100	0,5	> 1000	7,0	<0,2
45% KOH	100	> 1000	3,0	60,0	<0,2
25% HCI	70	0.9	85.0	72,0	<0,2
10% HF + 57% HNO_3	25	> 1000	> 1000	16,0	<0,2

- Bartuli and colleagues fabricated a protective coating of ZrB_2–SiC by plasma spray technique and studied the properties of this coating at high temperature. The authors of Alosime studied the oxidation properties of ZrB_2-SiC composite during plasma spraying.

- Doctoral thesis of Fahmi Mubarok (2014) of the University of Oslo, Norway, on thermal spray of silicon bits in the coating process and research on plasma spray coating of suspended micro-SiC particles.

- Samarin Peter and colleagues at Bauman Maxcova University [30] use radiant laser energy on the SiC coating on the aluminum alloy surface to form a SiC - Al composite coating to protect against corrosion.

Many attempts to create coatings containing 100% SiC have been unsuccessful, because SiC is prone to decomposition at high temperatures.With the other approach, Tului et al. successfully fabricated a coating containing 66% SiC (by volume) by plasma spraying with a mixture of SiC and ZrB_2 powders. The authors reported that these two compounds formed a eutecticphase, at a temperature lower than the decomposition point of SiC.

1.2.3. Study on fabrication of plasma sprayed SiC composite coating

The studies are aimed at 2 goals: reducing the spray temperature and increasing the rate of coating formation. Ibrahim said that when spraying SiC - B_4C mixture using B_2O_3 as an intermediate layer, the coating will have higher adhesion and lower porosity compared to SiC separately.

Kang et al fabricated metal matrix composite (MMC) containing SiC particles by spray method. These powders have different mixing ratios such as (Cu–27SiC, Cu–50SiC, Cu–60SiC) between SiC powders (<45 µm) and Cu powders (<45 µm). The authors show that SiC decomposes to Si and C, and then copper silicide (Si_3Cu) is formed upon plasma injection.

Comments:

- *SiC is strongly decomposed when plasma spraying at high temperature (temperature range 900°-1100oC). On the other hand, the melting point of SiC is very high (2730°C). This is the challenge for SiC plasma coatings.*

- *It is not possible to create a single SiC Plasma coating, but it can only be done with a combination of SiC composite powders such as: SiC/Ni60; SiC - B4C; SiC-Cu SiC- ZrB2…*

- *Protective coating in fluorine-containing environment will focus on SiC-Cu plasma coating research.*

1.3. SiC and SiC composite coatings provide corrosion protection for steel in fluorinated environments

In the production of pesticides, Na_2SiF_6 is a fluorine-containing environment: This environment will produce hydrofluoric acid HF, a causative agent for equipment. Especially if in this environment mixed with other acids: H_2SO_4, HCL, H_3PO_4... will create super acids with very strong destructive power to all metals, stainless steel including glass. The protective coating must demonstrate two possibilities:

- Resistance to abrasion is characterized by: Micro hardness, Adhesion strength

- Resistance to separation corrosion is characterized by: electrochemical properties, porosity, thickness and the spray material itself must be resistant to corrosion. Therefore, SiC coating is selected for study.

Because SiC thermal coating is difficult to apply due to decomposition and difficult to melt, this coating can only be achieved with composite materials (SiC composites) and the component materials that play the role of bonding in coating formation. (1.4)

If sprayed without a protective inert gas, Qin et al. [85] suggest that Cu_3Si is formed below 900°C at the border after SiC decomposition as follows: $SiC + 3Cu \rightarrow C + Cu_3Si$ (1.5)

- Spraying in the shielding gas: to isolate the SiC–Cu spray stream from oxidation because both Cu and SiC are easily oxidized in the air.

- Penetrating to seal the pores: to completely isolate the substrate from the outside environment through the coating.

Conclusion of chapter 1

- Analyze and learn about the world's anti-corrosion thermal coatings. SiC is a material with high microscopic hardness and inert in chemical environments, SiC is the research target of anti-corrosion coatings in acidic fluorine-containing environments.

- In Vietnam, it is not possible to fabricate or apply SiC coating to protect against corrosion/abrasion for machine and equipment parts.

- Determination of plasma method and finishing technology measures is a research method to create SiC/Cu coating on steel surface because the main feature is plasma arc heat source without using combustible gas, so the degree of oxidation is reduced. for powder spray and well applicable to SiC/Cu coating.

Chapter 2. THEORETICAL BASIS OF PLASMA SPRAYING TECHNOLOGY

Plasma spraying is a form of thermal spraying, the main feature is that it does not use combustible gas, but uses a plasma arc as a heat source to heat the powder. Therefore, the general theory of thermal coating is suitable for plasma spraying.

2.1. Theory of mantle formation

Many scientists have developed theories of mantle formation, in which the theories play an important role, including: The theory of Pospisil-Sehyl, Schoop, Karg, Katsch, Reininger and Schenk. Scientists around the world can describe the formation of the coating as: the melting phase of the spray material, the dispersion phase to form liquid metal droplets, the flying phase of the metal droplets, the phase of the metal droplets. impact of metal droplets on the substrate surface to form the coating (Figure 2.1).

Figure 2.1. Heat injection process stages.

2.2. Plasma spraying technology

2.2.1. Structure of plasma spraying system

Plasma is the fourth state of matter that has been sprayed with this energy source.

The schematic diagram of plasma injection process is shown in Figure 2.5 and Figure 2.6.

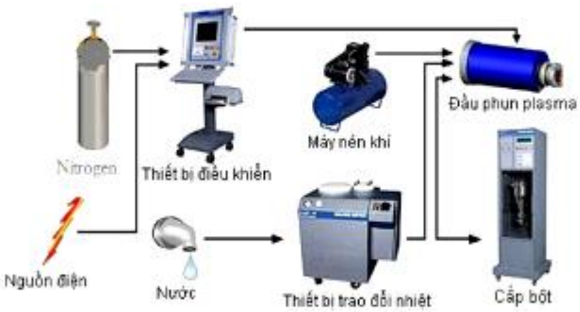


Fig. 2.5. atmospheric plasma spraying APS

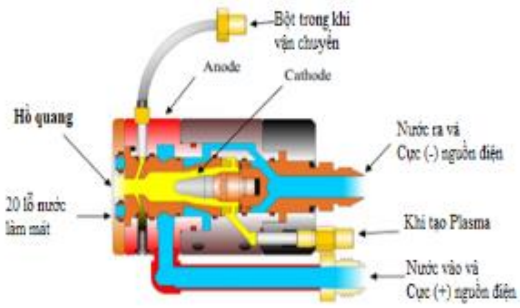


Fig. 2.6. Structure of plasma spraying gun

The plasma stream has a very high temperature of up to 10,000°C, up to 30,000°C at the center of the flame, so plasma spraying to create coatings from all kinds of difficult-to-melt materials is one of the outstanding advantages of this method. this. Plasma spraying equipment uses electrical energy in combination with Hydrogen, Helium, Argon and Nitrogen gases.

2.3. Factors affecting the quality of SiC plasma sprayed coating

When plasma coating is sprayed, there are many technological parameters that affect coating quality, but it can be seen that the main parameters affecting coating morphology and quality are as follows:

Powder parameters:	Spraying parameters:	- Survey targets:
+ Grain size	+ Current I, + Spray distance L	+ Adhesion strength, + Porosity,
+ Ratio of powder ingredients	+ Powder supply flow, M	+ Micro hardness, + Coating composition

2.4. SiC plasma sprayed coating

SiC plasma coating is the biggest challenge because SiC decomposes strongly in the plasma environment when spraying. The SiC spray modes are applied by many studies on the basis of ceramic materials (for single SiC plasma spraying) or according to the bonding agent's spray mode (for SiC composite injection materials).

Conclusion of chapter 2

Presented on plsma spray coating technology method, coating forming process. The analysis highlights the influencing technological factors, giving key survey specifications and quality criteria to be achieved.

- Spray specifications: Plasma intensity is characterized by amperage I (A); Spray distance L(mm); Powder feeding flow M(g/min)

- Powder parameters: Granularity (powder size); Mixing powder ingredient ratio

- Indicators: Adhesion strength; Porosity; Micro hardness; Coating composition

Chapter 3. MATERIALS AND METHODS

3.1. Plasma spraying materials

3.1.1. Spraying powder

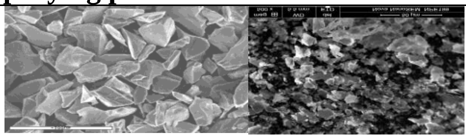


Fig.3.1.SEM images of SiC (left) and Cu powder (right)

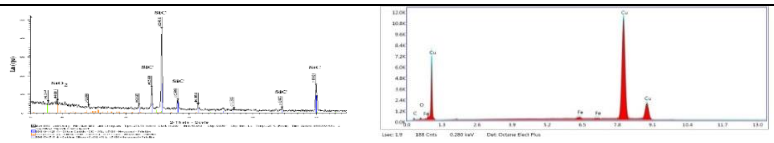


Fig. 3.2. XRD patterns of SiC (left) and Cu powders (right)

3.1.1.1. Characterization of SiC powder during plasma spraying

Figure 3.1 SiC powder (SiC > 80%, 50 µm, density = 3.2 g.cm-3. For silicon carbide when exposed to high temperature in an air plasma environment, the surface oxidation of SiC is at high temperature form SiO2. Hahnel et al. [82, 83] detailed a 4-stage model of the formation of the junction between SiC and oxygen:

$SiC + 3O_2 = 2SiO_2 + 2CO$ (1.1)

$SiC + O_2 = SiO + CO$

$2SiO + O = 2SiO_2$ (1.2)

$2SiC + O_2 = 2SiO + 2C$

$SiC + O_2 = SiO_2 + C$

$2C + O_2 = 2CO$ (1.3)

$2CO = C + CO_2$

3.1.1.2. Characteristics of Cu powder during plasma spraying

When considering the calculation and mixing of two types of Cu and SiC powders for the input materials of the plasma spraying device, it is found that the difference in their density is quite far from Cu (ρ = 8.96g/cm3) compared with SiC. (ρ = 3.2 g/cm3). Therefore, it is necessary to choose powder sizes: Cu (30 µm) and SiC (50 µm) to enhance the uniformity in the spraying process.

With the melting point of Cu (1085°C) much lower than that of SiC (2730°C), in the plasma spray flame, copper easily forms coatings, quickly achieving higher thickness and adhesion. When the Cu content is increased, the coating formation rate will be increased, but the coating quality will decrease.

3.1.2. Substrate material

C45 steel base sample size 50×50×4 mm.

Figure 3.3. C45 steel sample before and after spray cleaning to create roughness

Before applying coating, the surface of C45 steel needs to be cleaned and roughened.

3.2. Technological parameters and experimental equipment

3.2.1. Selection of technological parameters and key technical criteria

-Characteristic parameters of powder materials *+ SiC particle size; Cu: W (µm)* *+Ratio of powder ingredients:S (%)*	*- Specifications of injection technology* *+ Amperage: I (A)* *+ Spray distance: L (mm)* *+ Powder feeding speed: M_{cb} (g/min)*	*- The quality indicators of SiC/Cu grade* *+ Adhesion strength; + Porosity;* *+ Micro-hardness* *+ SiC content in the coating* *+ Wear resistance*

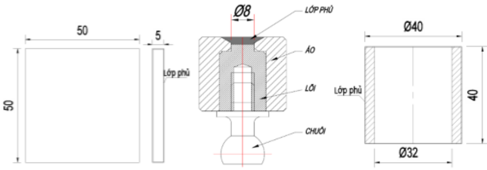
3.2.2. Instrumentation

- Coating Microhardness Tester HH 401 (Mitutoyo - Japan) (Figure 3.4 a)
- Coating thickness gauge (Figure 3.4 b): DIGI – DERM 745 (Mitutoyo – Japan)
- Measurement of adhesion strength (POSITEST - AT - M, Defelsko - USA) (Figure 3.4 c).
- Optical microscope AXIOKOP 2 MAT MOT (Figure 3.4 d) (Germany) uses AxioVision software to analyze the phase composition of the coating to determine the coating porosity by image analysis

a)Micro-hardness device b) Coating thickness device c) Adhesion device d) Optical microscope

Fig. 3.4. Coating quality testing equipment

3.2.3. Sample preparation



a) b) c)

Hình 3.5. Samples

1) Microscopic test specimen, composition, porosity measurement, Microscopic hardness test, corrosion and abrasion test (Figure 3.5 a)

2) Adhesion test specimen (Figure 3.5 b)

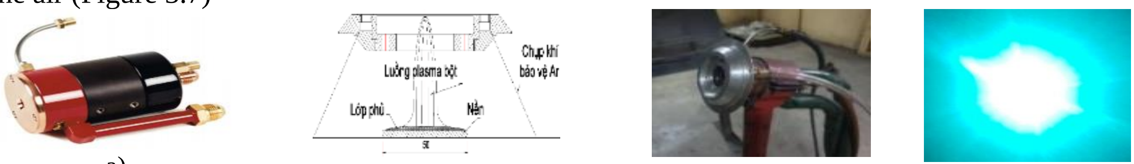
Material: SUS 304; C45. Used in testing single SiC and SiC – Cu coatings

3.2.4. APS device

Hình 3.6. PRAXAIR plasma spraying

3.2.5. Modified spraying gun in shielding Argon gas

The improvement by the method of plasma spray technology in the Ar shielding gas to solve the oxidation problem of the spray powder in the air (Figure 3.7)



a) b) c) d)

a) SG100 gun; b) Model of shielding Argon gas; c) Cover on gun; d) Plasma flame in cover of gun under Argon gas .

Figure 3.7 Modified spraying gun in shielding gas

Principle: when working, the supplied argon 5Psi (0.04 MPa) gas will create a cone-shaped gas cover that encloses the plasma stream and the substrate surface during the spraying process, separating the powder flow and the spray object from the outside air. Figure 3.7d is a shot of a spray gun with a shielded Ar gas. White plasma flame, blue separating protective gas ring.

3.3. Methods of analysis and evaluation

3.3.1. Measure the thickness and porosity of the coating

The coating thickness was measured by a Digi-Derm instrument (Model DGE-745, Mituyo, Japan).

The visible porosity of the coatings was determined by optical microscope AXIOKOP 2 MAT MOT (Germany): using the software AxioVision phase composition analysis to determine the coating porosity by analytical method. Photo. Standard applicable ASTM B276.

3.3.2. Morphological analysis of the coating surface by scanning electron microscopy

Surface morphology of the samples was analyzed by FEI Nova Nano SEM 450 Scanning Electron Microscope (Japan), Energy-dispersive X-ray spectroscopy EDX (Energy-dispersive X-ray spectroscopy EDX).

3.3.3. Phase composition analysis by X-ray diffraction (XRD)

D8 Advance phase component analyzer of Brucker (Germany). Measurement parameters include temperature 25°C, 2θ angle scanning from 15˚ to 65˚. After collecting the XRD diagram, the data were analyzed using specialized software (using Eva software) and semi-quantitative evaluation (using Dquant software with an error of ± 3%).

3.3.4. Experimental planning method to evaluate the influence of technology regime on coating mechanical properties

The thesis uses experimental design developed by Genichi Taguchi on the basis of the theory of sustainable design (Robust Design). The aim is to adjust the parameters to the optimum level so that the process/product is stable at the best quality level.

3.3.5. Method for assessing the ability to protect against corrosion

To evaluate the anti-corrosion protection of the edema layer, the thesis uses electrochemical analysis and mass loss measurement methods. The contents of the methods are as follows:

1) Electrochemical analysis method

a. Prepare sample for electrochemical measurement:

The samples after being sprayed with SiC coating on C45 steel were welded with wire at the corner of the sample (Fig. 3.8).

Fig. 3.8. Electrochemical samples

Fig. 3.9. Electrochemical test

b. Electrochemical measuring device

Corrosion properties of the coating were investigated by electrochemical measurement methods: measuring Tafel polarization curve and total electrochemical resistance. (Figure 3.9).

c. Tafel polarization curve measurement method

Compared with linear biased resistance measurement, this method provides more information over a wide range of potentials. The data obtained are more and more useful such as: providing information about the corrosion rate, explaining the corrosion mechanism and predicting the corrosion behavior when the voltage changes...

d. Electrochemical impedance measurement (EIS) method

The corrosion process of steel in a solution of 3.5% NaCl can be evaluated by measuring the total electrochemical resistance at the open circuit potential (E_{oc}) of the base steel. The impedance spectrum is the set of all measured Z values at different frequencies.

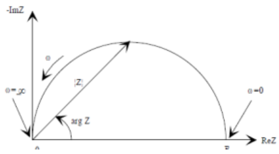


Figure 3.10. Nyquist diagram with total impedance vector of Z/electrolyte (C45/NaCl steel)

Equivalent circuit used in EIS analysis for samples in 3.5% NaCl solution

Figure 3.11 equivalent circuit diagram for the SiC-Cu/steel sample in NaCl, where Rs is the electrolyte resistance (which is the resistance between the C45 steel-working electrode, and the reference electrode. Cdc is the layer capacitance. double charges form on the boundary between the steel surface and the electrolyte.Rct is the charge transfer resistance at the junction of the steel surface and the solution.Rp is the sum of the resistances on the shielding surface.

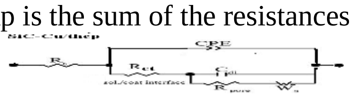


Fig. 3.11 Equivalent circuit diagram for the SiC-Cu/steel sample in NaCl

When PTFE is present, the equivalent circuit diagram of PTFE/SiC-Cu/steel coating is shown in Figure 3.12.

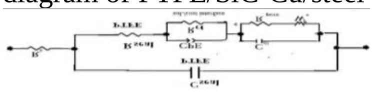


Figure 3.12. Equivalent circuit for PTFE/SiC-Cu/steel sample in NaCl.

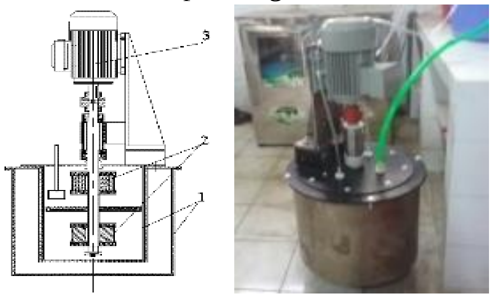
Where R_{seal} and C_{seal} the resistance and capacitance of the PTFE coating/shield respectively. Cseal depends on dielectric constant, thickness, area of PTFE layer. Reseal resistance is related to electrolyte penetration

2) Method of measuring mass loss by composite wear tester

This device is a product of the author's research, design and manufacture (Figure 3.13) used to evaluate the corrosion and abrasion resistance of coatings in an environment compatible with actual conditions (corrosive environment). typical corrosion at Lam Thao Superphosphate and Chemical Company).

The experimental equipment creates a similar working state with the impeller and impeller details. Two specimen holders. The lower chamber is a chemical solution of 10% HF(30%); 20% H2SO4 (98%) and contains 0.2 - 0.25mm SiC abrasive particles, the upper chamber is the chemical vapor that rises when heated. Test temperature 70ºC; Stirring speed 1000 r/min; Test cycle: 124h.

Samples: SUS304, C45, SiC/C45, PTFE/SiC/C45 steel samples; SiC-30Cu/C45, PTFE/SiC-30Cu/C45; SiC-50Cu/C45, PTFE/SiC-50Cu/C45. Measurement method: sample weight over time.



1. Chemical tank;
2. Samples;
3. Centrifugation

Figure 3.13. Mixed wear tester

3.4. Experimental process

- Prepare samples, clean and roughen the steel substrate surface, Spray according to the setting mode.

- Perform the experiment in two steps:

+ Survey and probe SiC and SiC - Cu plasma coatings with the aim of creating a SiC coating of sufficient thickness and containing a high percentage of SiC in the coating to establish and fix powder parameters: mixing ratio (S) and grain size (H).

+ Investigate the influence of 3 parameters of plasma spraying technology: I, L, M on coating quality.

Conclusion of chapter 3

- The analysis provides a solution to create a SiC plasma coating that protects against corrosion in the HF environment. The addition of copper (Cu) to the spray powder is a possible condition for the formation of the SiC-Cu composite coating.

- Provide key technological parameters for SiC-Cu plasma coating. Building a mathematical model to simulate coating quality according to Taguchi Experimental Plan to determine the mixing ratio and particle size.

- Analyzing and giving criteria to evaluate coating quality.

- Design and manufacture 02 groups of coating samples for research;

- Provide technological solutions: design and manufacture a gas shield to protect the plasma spray from the outside air.

- Analyze and propose a method to evaluate the wear protection for coatings through electrochemical properties and mass loss. Design and manufacture of composite wear test equipment to evaluate coating wear and synthetic corrosion protection.

- Outline the experimental process in 2 steps:

+ Surveying and creating SiC - Cu coating, fixing the parameters of powder S, H to investigate the spray mode.

+ Investigate the influence of 3 parameters of plasma spraying technology: I, L, M on coating quality.

Chapter 4. PRODUCTION AND ANALYSIS AND ASSESSMENT OF PLASMA SiC Coatings ON STEEL BASIS

4.1. Fabrication of SiC single coating on steel base

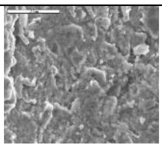
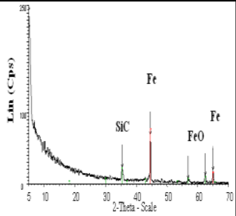
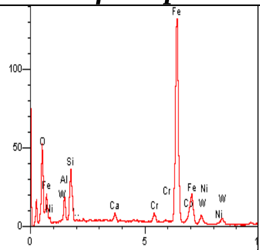
4.1.1. SiC plasma spray technology mode on steel base

Plasma injection technology mode for SiC is referenced to plasma spraying for ceramic materials:

Table 4.1. SiC/Steel plasma spray technology mode.

Parametrs	WC-20Ni	ZrB2-SiC-ZrC	Ni-Al-SiC	SiC
Current, A	600	450 - 600	600	600
Voltage, V	70	64	28	44,5
Primary air flow rate (Ar), L/min	40	35		
Secondary air flow rate (H2 gas), L/min	12	12		
Carrier gas flow rate (Ar), L/min	5	2		
Carrier gas pressure, kPa			30	30
Powder feed rate, g/min	90-100	60		30
Spacing, mm		40-60	100-125	40
Particle size, µm	22-45			20-45

4.1.2. Structure and mechanical properties of SiC plasma spray coating on C45 steel

 ***Fig. 4.2. SEM image of SiC/C45 coating magnification × 2000***	 ***Fig. 4.4. XR pattern of SiC/C45 coating***	 ***Fig 4.5. EDS spectra of SiC/C45 coating***	***Results:*** - SiC Thickness 8- 10 μm - EDS measurement shows that the elemental composition content is C (5.95 %), O(7.11 %), Al(4.50 %), Si (6.93 %), Ca (1.33 %), Cr (2.00 %), Fe (58.47 %), Co (3.33 %), Ni (4.33 %), W (6.04 %). Combined with the XRD spectrum of Figure 4.4, the SiC composition in the mantle is low (7-10%).	***Table 4.3. Microscopic Hardness (HV) value of SiC coating on C45 steel Load 10g; Measurements in order (from inside to outside)***

Point	(HV) Average
1	412,7
2	411,0
3	420,7

From the experimental results, it is shown that spraying SiC single coating by plasma is very difficult and does not preserve the SiC content in the coating. This is consistent with the results of many research works around the world on plasma SiC coating.

4.1.3. Electrochemical analysis of SiC/steel plasma plasma spray coating

1) Polarization curve measurement results

Figure 4.8 depicts the polarization curve of C45 steel sample with or without protective coating in 0.1 M K2SO4 solution (~1.7% K2SO4). Corrosion potential value, the PTFE/SiC-Cu coating system shifts the potential of the steel to a more positive direction, demonstrating better isolation than the metal (C45 steel) from the corrosive solution.

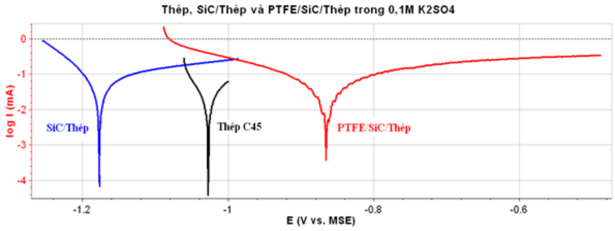


Figure 4.8. Polarization curve of C45 steel sample in 0.1 M K2SO4 solution

Table 4.5. Corrosion potential value and corrosion current density of C45 steel substrate and coating samples (sample area 7.5 cm2, MSE comparison electrode: Mercury/Mercury sulfate)

Mẫu	Điện thế ăn mòn		Dòng ăn mòn	
	E_{corr} (mV/MSE)	E_{corr} (mV/SCE)	I_{corr} (μA)	I_{corr} (μA/cm^2)
Nền thép C45	-1026	-626	63,29	8,44
SiC/thép	-1177	-777	99,93	13.32
PTFE/SiC/thép	-861	-461	59,90	7,99

Corrosion current of C45 steel from 8.44 to 7.99 μA. When comparing the corrosion currents of the two coating systems, the presence of PTFE reduces the corrosion current from 13.32 μA to 7.99 μA, which can be calculated as an inhibitory efficiency of 40%.

2) Mass loss measurement results

Table 4.6. Weight loss measurement results in acidic medium

Time (hours)	Mass loss SUS304 (mg/cm^2)	Mass loss PTFE/SiC/ C45 (mg/cm^2)
8	41,6	0
16	45,0	2,1
24	43,1	4,34
32	46,0	6,65

Weight loss measurement results of steel samples immersed in acid environment: SUS 304 steel sample lost up to 45g, while if C45 steel was coated with PTFE/SiC system, it only lost 5mg. Since the amount of SiC on the surface of the coating is very low (7-13% by mass), this corrosion protection is provided by the outer PTFE layer.

Comments

When plasma spraying with single SiC: 1) SiC is difficult to melt, difficult to form a coating; 2) SiC is easily decomposed at high temperature.

To solve these difficulties, it may be necessary to combine SiC powder with an anti-corrosion (Cu) binder to:

1) Lower the spray temperature to reduce decomposition; 2) Increase adhesion to the substrate

With SiC/Cu powder materials, the binder is Cu powder due to its ability to be difficult to oxidize in HF environment, high adhesion and bonding ability; The melting point (10500C) is much lower than the melting point of SiC. Since the densities of Cu and SiC are different, the SiC particle size should be chosen to be larger than that of Cu to ensure uniformity in order to:

+ Avoid stratification during flight due to the kinetic energy of two different types of material particles.

+ Increase the dispersion density of copper element in SiC, increase the cohesion in the coating formation process

+ Reduce plasma intensity while ensuring coating formation rate.

4.2. Fabrication of SiC-Cu composite coating on steel substrate

4.2.1. Analysis of SiC-Cu powder technology factors when plasma spraying on C45 . steel

4.2.1.1. Particle size:

Refer to some similar plasma spray powders: AL2O3 and Cr_3C_2 are 45 +15 µm. Kang also fabricated SiC/Cu plasma spray powder with the corresponding particle size 45/45 µm. To avoid separation of plasma spray powder because Cu is nearly 3 times heavier than SiC, the Cu particle size should be 2 ÷ 3 times smaller than SiC. So, it is possible to choose SiC and Cu particle sizes for the experiment: SiC: 25 ÷ 65 µm; Cu: 20 ÷ 55 µm.

4.2.1.2. Mix ratio

Mixing ratio to meet: Formation of coating thickness > 100 µm; ensure the required content of SiC in the coating. Reference for some two- or more-component spray powders for plasma spraying [107]. Can lead out with powders: Ni - 20Cr; Cr-50Co. Here Ni, Co or Cu all play the role of binder. Kang [55] also gives the ratio for powder spray plsma SiC/Cu: Cu–27SiC, Cu–50SiC, Cu–60SiC.

From there, choose the ratio of SiC and Cu powders for investigation are: SiC-20Cu; SiC-30Cu; SiC50Cu

4.2.1.3. Technology mode of coating SiC-Cu composite on steel base

The plan to create SiC-Cu plasma composite coating solution to solve 2 goals:

- Coating thickness > 100 µm.

- The SiC content in the coating is increased.

Plasma spray mode (SiC-Cu) is applied according to Cu copper powder to select SiC - Cu plasma spray technology mode:

Table 4.7. Preliminary selection of SiC-Cu injection technology mode on steel base.

Parameters	For SiC-Cu spraying coating
Current, A	400
Voltage, V	58
Primary air flow rate (Ar), L/min	35
Secondary air flow rate (H_2 gas), L/min	12
Carrier gas flow rate (Ar), L/min	2
Powder feed rate, g/min	42
Spacing, mm	50
Particle size, µm	50 (SiC); 30 (Cu)
Powder mix ratio, % Cu (by mass)	SiC–20Cu ; SiC–30Cu; SiC-50Cu)

4.2.2. Effect of plsma SiC/Cu spray in Ar shielding gas

a) Spray SiC-30Cu without shielding gas

Figure 4.11 and 4.12 are SEM images and EDS spectra of SiC-30 Cu coating when sprayed without Argon shielding gas.

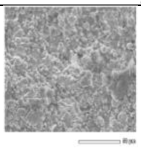


Fig. 4.11. SEM images of SiC-30Cu/C45 coating –Sprayed without Ar gas (magnification: × 750)

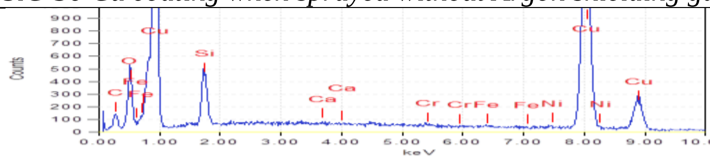


Fig 4.12. EDS spectra of SiC-30Cu/C45 coating –Sprayed without Ar gas

Table 4.8. Results of EDS analysis for SiC-30/C45 –Sprayed without Ar gas .

Elements	*C K*	*O K*	*Si K*	*Ca K*	*Cr K*	*Fe K*	*Ni K*	*Cu K*
% Mass	*6.30*	*7.09*	*3.66*	*0.09*	*0.20*	*0.26*	*1.28*	*81.13*
% Atom	*18.79*	*16.86*	*5.42*	*0.09*	*0.16*	*0.19*	*0.9*	*53.04*

b) Spray when there is a protective Ar gas

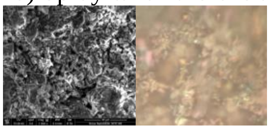


Fig. 4.13. SEM images of SiC-30Cu/C45 coating –Sprayed with Ar gas (magnification: × 1000)

Fig. 4.14. Thickness test for SiC-30Cu/C45 coating –Sprayed with Ar gas

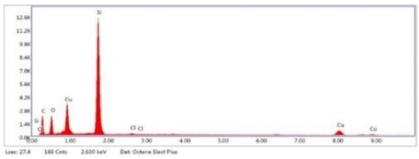


Fig. 4.15. EDS spectra of SiC-30Cu / C45 coating –Sprayed with Ar gas

Table 4.9. EDS analysis results for SiC-30Cu sample on C45 steel substrate

Nguyên tố	C K	O K	Si K	Cl K	Cu K
% Khối lượng	32,41	11,32	33,49	0,41	22,37
% Nguyên tử	54.38	14,26	24,03	0,23	7,09

Elemental analysis by EDS spectrum (Figure 4.15 and Table 4.9) showed C (32.41%); Si (33.49%); O (11.32 %); Cl (0.41%); Cu (22.37%). SiC accounts for 66% of the mantle mass.

c) Thickness results and composition content of SiC/Cu coatings (Table 4.10)

Table 4.10. Measurement results of coating thickness, SiC content in the coating.

Number of test	Cu/SiC ratio(%wt)	Particle size (µm)		Coating thickness, (µm)	SiC content in coating (%)
		S_{SiC}	S_{Cu}		
1	20	25	20	106	41,3
2	30	45	20	178	58,6
3	50	65	20	287	59,5
4	20	25	35	104	68,4
5	30	45	35	193	68,7
6	50	65	35	292	47,8
7	20	25	50	100	64,5
8	30	45	50	186	56,4
9	50	65	50	209	37,6

Spraying SiC/Cu plasma in the shielding gas reduces the degree of oxidation while improving the SiC content in the resulting coating.

4.2.3. Set reasonable powder parameters for SiC-Cu plasma spray powder.

From the obtained results, it is possible to make an experimental plan to determine the powder parameters (mixing ratio H and particle size S) for 2 criteria: coating thickness (δ) and SiC content (H) in coating. On the basis of theory and exploratory research results, the technology regime is established as in Table 4.11.

Table 4.11. Select the SiC-Cu spray experimental technology mode on the steel substrate.

Parameters	For SiC-Cu spraying coating
Current, A	400
Voltage, V	58
Primary air flow rate (Ar), L/min	35
Secondary air flow rate (H2 gas), L/min	12
Carrier gas flow rate (Ar), L/min	2
Powder feed rate, g/min	42
Spacing, mm	50
Particle size, µm	20 - 70 (SiC); 15 - 55 (Cu)
Powder mix ratio, % Cu (by mass)	SiC–20Cu ; SiC–30Cu; SiC-50Cu)

Using the Taguchi method, the influence of 3 factors (Cu particle size, SiC particle size and Cu/SiC mixing ratio by mass) was studied on two criteria: coating thickness and SiC content in the layer. government.

Table 4.13. Results of analysis of SN ratio of thickness and SiC content in the coating.

No.	Cu/SiC ratio (%wt)	Particle size (µm)		Coating thickness		SiC content in coating	
		S_{SiC}	S_{Cu}	Experimental (µm)	SN (dB)	Experimental (%)	SN (dB)
1	20	25	20	106	32,86	41,3	32,32
2	30	45	20	178	39,01	58,6	35,36
3	50	65	20	287	48,44	59,5	35,49
4	20	25	35	104	32,76	68,4	36,58
5	30	45	35	193	40,71	68,7	36,74
6	50	65	35	292	48,67	47,8	33,59
7	20	25	50	100	31,85	64,5	36,19
8	30	45	50	186	39,03	56,4	35,03
9	50	65	50	209	48,53	37,6	31,50

Table 4.14. SN and effect of 3 parameters on coating thickness

Level	S_{Cu}(mm)	S_{SiC}(mm)	H (%wt Cu/SiC)
1	44,77	46,49	44,97
2	46,71	45,37	46,43
3	45,92	45,54	46,00
Delta	1,94	1,12	1,46
Score	1	3	2

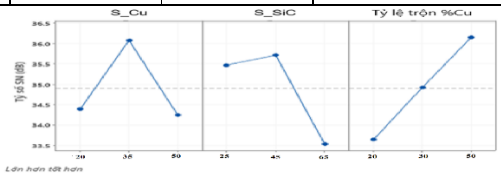


Fig. 4.16. Effect of parameters on coating thickness by SN

Table 4.15. SN and effect of 3 parameters on SiC contents

Level	S_{Cu}(mm)	S_{SiC}(mm)	H (%wt Cu/SiC)
1	34,39	35,47	33,64
2	36,07	35,71	34,92
3	34,24	33,53	36,14
Delta	1,83	2,18	2,50
Score	3	2	1

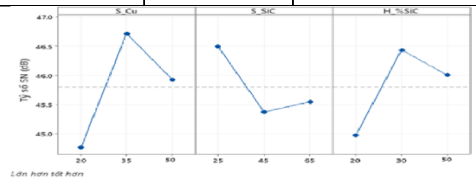


Fig. 4.17. Effect of parameters on SiC contents by SN

Comments:

a) For coating thickness

- Copper particle size (S_{Cu}) has the strongest influence on the coating thickness criterion (ranked 1). Accordingly, the average Cu particle size is from 20 to 35 µm, the coating thickness increases as the S_{Cu} particle size increases. But S_{Cu}(35 - 50 µm), the coating thickness decreases with increasing Cu particle size. Cu particles from 30 µm to 40 µm result in effective coating formation.

- The percentage Cu/SiC by mass has the second effect on the coating thickness criterion. From the graph and table of influence analysis, it is shown that the percentage of Cu/SiC affects the coating thickness mainly in the range of 30-50%, when increasing the percentage of Cu/SiC, the coating thickness increases, in the range of 20-30%, when increasing the percentage of Cu/SiC, the coating thickness is low. This result indicates: the coating thickness is high, it is necessary to choose the % Cu/SiC ratio in the range of 30-50 %.

- Silicon carbide particle size (S_{SiC}) has the weakest influence on the coating thickness criterion (ranked third). In the range of average SiC particle size from 25 to 35 µm (strong decrease graph), while in the range of average SiC particle size from 35 to 50 µm (weak increase graph) less influence. This result indicates that, in order to achieve the target of coating thickness, it is necessary to choose a more effective SiC particle size of 40 - 50 µm.

b) For SiC content in the coating

- The percentage Cu/SiC by mass has the strongest influence (ranked 1) on the SiC content indicator in the coating. The % Cu/SiC ratio affects the SiC comb function in the coating in the direction that increasing the % Cu/SiC ratio helps to increase the SiC content sharply, the % Cu/SiC ratio below 25% is not effective for coating thickness specifications. This result in order to have a high SiC content in the mantle, it is necessary to choose the percentage of Cu/SiC as high as possible in the survey area. Thus, the proportions of SiC-30Cu and SiC-50Cu powder mixtures can be applied.

- Silica carbide (SSiC) particle size has a second effect on the SiC content in the coating. The results show that, in order to have the SiC comb function in the large coating, it is necessary to choose a medium SiC particle size, the more effective range of 40 - 50 µm.

- Copper particle size (SCu) has the weakest influence on the content of SiC in the coating (ranked 3rd). The average Cu particle size is from 20 to 30 µm, the SiC comb function in the mantle increases with increasing SCu particle size, but in the average Cu particle size range from 35 to 50 µm, the SiC comb function in the mantle increases. decreased with increasing Cu particle size. This result indicates that the Cu particle size from 20 µm to 30 µm, the effective SiC content.

To simultaneously achieve the target of coating thickness and high SiC content in the coating, corresponding to:

- Powder mixing ratio: SiC-30Cu; SiC-50Cu

- Particle size: SiC from 40 - 50 µm; Cu from 20 - 30 µm.

- Technology mode: I (A): 400; L (mm): 50; M(g/min): 40.

4.3. Results on microorganism of SiC-Cu/Steel plasma coating in Argon shielding gas

Samples were sprayed in Ar shielding gas after 10 sprays. The coating thickness achieved:

- SiC-30Cu : 201.67 ± 26.5 µm

- SiC-50Cu : 220 ± 20.5 µm.

4.3.1. SiC-30Cu coating structure on C45 - steel substrate

From optical microscopy image analysis (Axiovert 40 MAT instrument), the average porosity value of the coating is 1.65% (1.38%). This value is much lower than the porosity of the plasma spray coating Cr3C2-25NiCr (3.1% - 3.4% [26]). In the publication [26], the authors used Cr3C2-25NiCr powder (size 35±5 µm), plasma current of 600 A and injection distance of 100 mm.

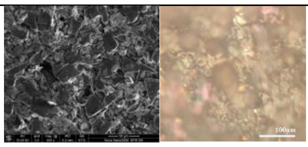


Fig. 4.22. SEM images of SiC-30Cu/C45 (magnification: ×500)

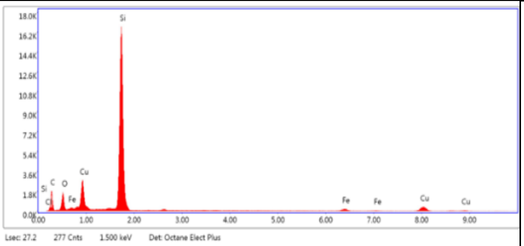


Fig. 4.23. EDS spectrum of SiC-30Cu coating

Table 4.16. EDS data for SiC-30Cu/C45

Elements	% Mass	% Atom
C K	30,48	52,27
O K	8,29	10,67
Al K	0,54	0,42
Si K	40,96	30,04
Cl K	0,49	0,28
Fe K	1,73	0,64
Cu K	17,52	5,68

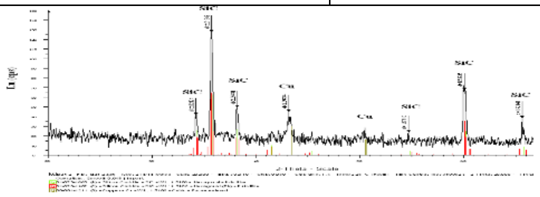
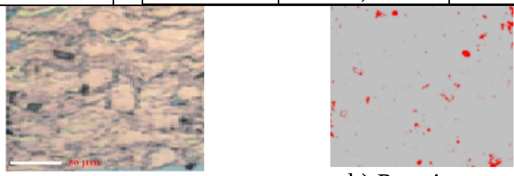


Fig. 4.24. XRD pattern of SiC-30Cu coating



a) *Optical image* b) Pore image

Fig. 4.25. Cross-sectioning of SiC-30Cu coating (magnification: ×200)

4.3.2. SiC-50Cu coating structure on C45 - steel subtrate

Figure 4.26 depicts the surface of SiC-50Cu coating. The coating surface is less rough, more uniform when increasing the Cu powder content when spraying. As a result of EDS spectroscopy (Figure 4.27), Si content is lower in the coating (~29%).

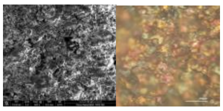


Fig. 4.26. SEM image of SiC-50Cu coating (magnification: ×500)

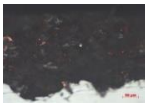


Fig 4.21. Ả Cross-sectioning of SiC-50Cu coating (magnification: ×200)

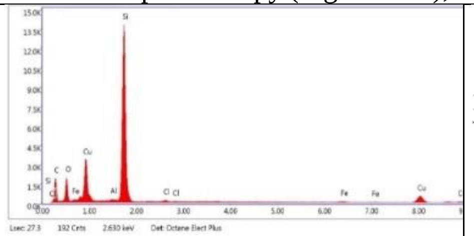


Fig 4.27. EDS spectrum of SiC-50Cu coating

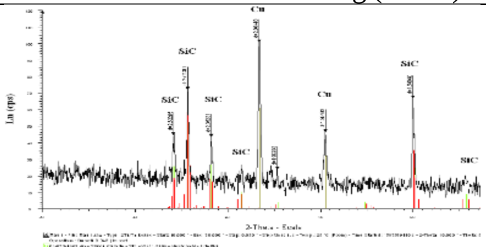


Fig. 4.28. XRD pattern of SiC-50Cu coating

Based on the results of XRD analysis (Figure 4.28), the content of SiC phases accounts for 53%, of which the crystalline phases are 42% and the amorphous phase accounts for 11%. The Cu content in the coating is about 47 % (with the error of ± 3% with Dquant software).

Table 4.17. EDS analysis results for sample SiC-50Cu/steel

Elements	% Mass	% Atom
C K	30,03	52,38
O K	9,60	12,57
Al K	0,36	0,28
Si K	35,69	26,62
Cl K	0,32	0,19
Fe K	1,08	0,40
Cu K	22,91	7,55

From the microscopic results of the coating obtained, it was found that with powder spray SiC-30Cu; SiC-50Cu achieves the criteria of thickness, coating structure better than other blend components. So the powder parameters are as follows:

- *SiC-30Cu; SiC-50Cu*
- *SiC particle size: 40 - 50 µm*
- *Cu particle size: 20 - 30 µm*

In order to choose the best coating for corrosion protection, it is necessary to experiment with electrochemical properties and aggregate mass loss.

4.4. Corrosion resistance of plsma SiC-Cu coating

4.4.1. Electrochemical properties of SiC-Cu/steel plasma spray coating in 3.5% NaCl medium

Good corrosion protection when: High bias resistance, low capacitance, and small corrosion current.

4.4.1.1. Polarization curves

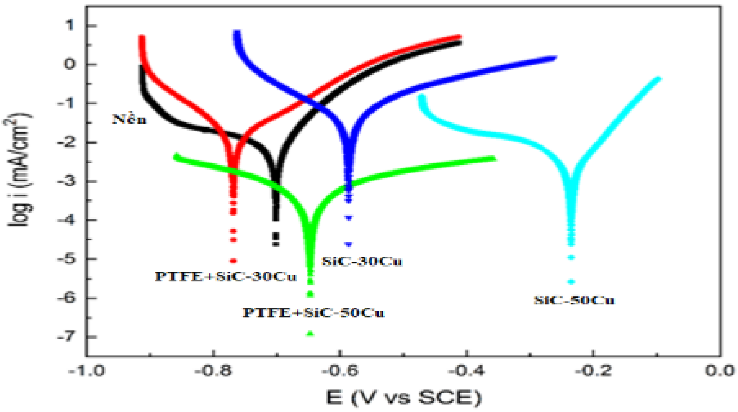


Figure 4.29. Polarization curve of C45 steel sample with and without protective coating in 3.5% NaCl solution.

Table 4.18. Corrosion potential value and corrosion current density of C45 steel substrate and coating samples

Samples	**Corrosion potential E_{corr} (mV/SCE)**	**Corosion current density I_{corr} (µA/cm^2)**
Nền thép C45	-707,242	9,44
SiC-30Cu/ C45	-576,492	55,055
SiC-50Cu/ C45	-211,828	5,597
PTFE/SiC-30Cu/ C45	-758,022	12,542
PTFE/SiC-50Cu/ C45	-647,73	0,593

Regarding the value of corrosion potential, Figure 4.30 shows the corrosion potential values of C45 steel in NaCl solution with or without protective coating. Looking at Figure 4.30, it can be seen that 3 types of test samples: C45 , SiC-Cu and PTFE/SiC-Cu all shift the potential of the steel to a more positive direction, showing better isolation than metal (C45 steel).) from the corrosive solution. The cathode shift of the corrosion potential in PTFE/SiC-50Cu coatings is related to galvanic electrochemical corrosion due to direct contact of Cu and steel when the coating does not completely cover the steel surface or at in the voids/porosity of the coating.

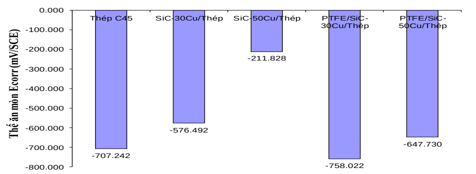


Fig. 4.30. Corrosion potential of C45 in NaCl solution

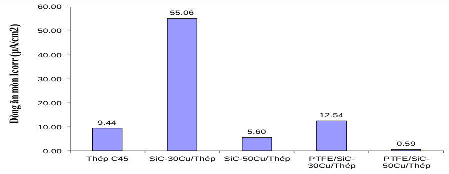


Fig. 4.31. Corrosion current of C45 in NaCl solution

Two coating systems, SiC-50Cu and PTFE/SiC-50Cu, both strongly reduced the corrosion current of C45 steel from 9.44 to 5.6 and 0.59 µA/cm2, respectively. This reduction in corrosion current translates to an inhibitory efficiency of 40.7% and 87.5%, respectively.

4.4.1.2 Electrochemical impedance spectrum

Figure 4.32 is the Nyquist impedance spectrum of the C45 base steel, which can be seen as the spectrum showing the double charge layer between the metal/solution boundary, as depicted by the equivalent circuit in Figure 3.12.

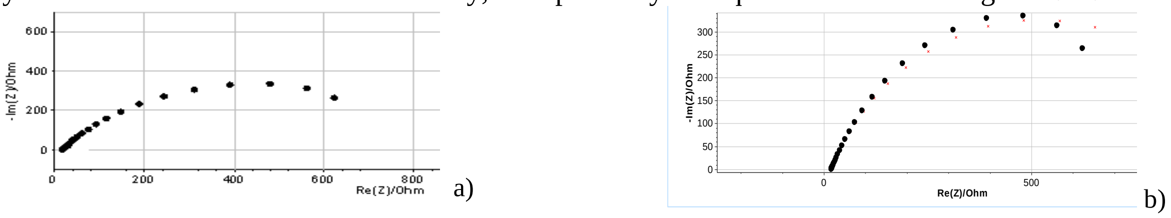
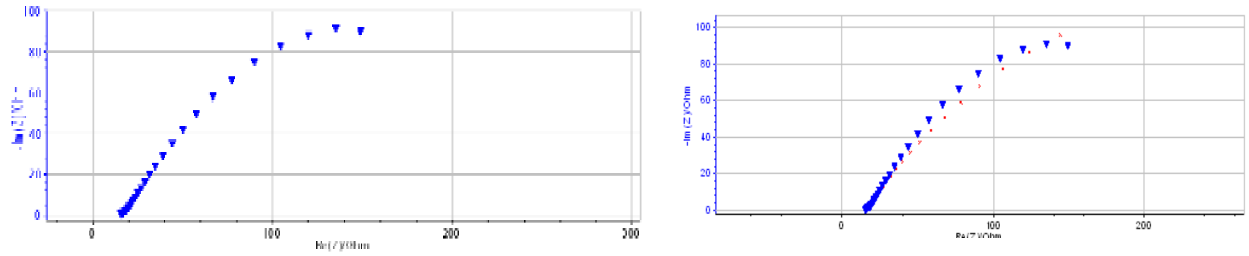


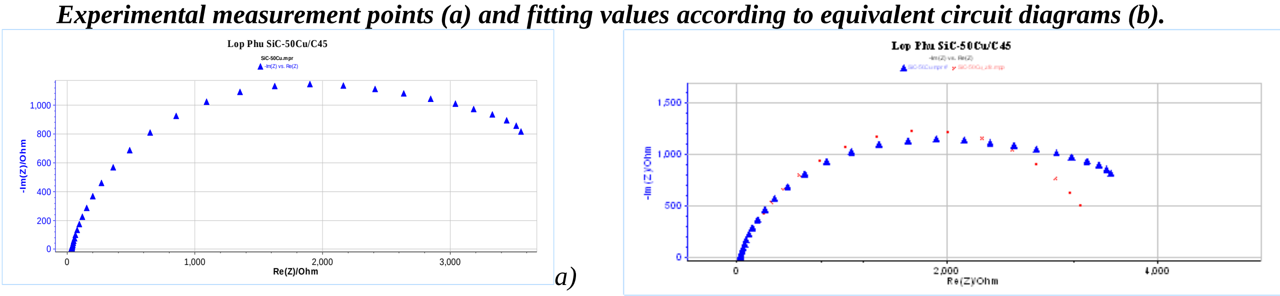
Figure 4.32. Nyquist impedance spectra of C45 base steel (without coating) in 3.5% NaCl. solution

Figures 4.33 and 4.34 are spectra of SiC-30Cu and SiC-50Cu coatings on steel substrates in NaCl solution. It can be seen that the spectrum has only 1 semitone representing 1 RC pair. The contribution of the diffusion resistance or the RC pair of Rpore and Cdl is not seen. This can be explained by the very low porosity of the coating (thanks to the presence of Cu powder when spraying).



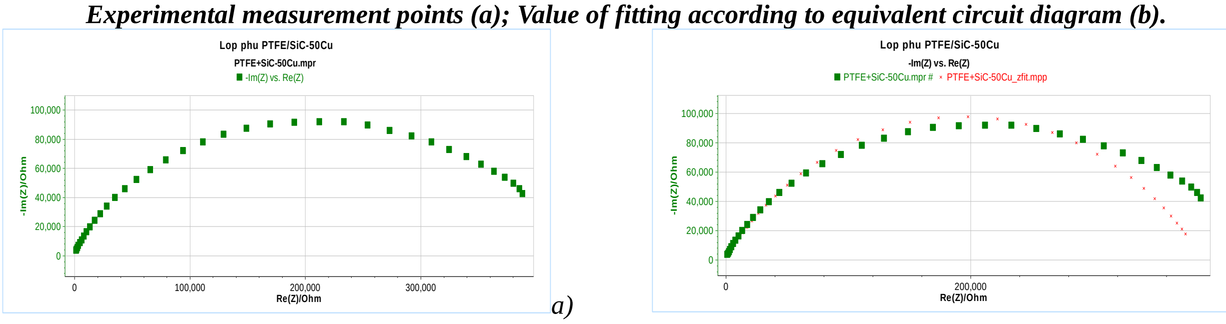
a) b)

Figure 4.33. Nyquist impedance spectra of SiC-30Cu/C45 in 3.5% NaCl solution. Experimental measurement points (a) and fitting values according to equivalent circuit diagrams (b).



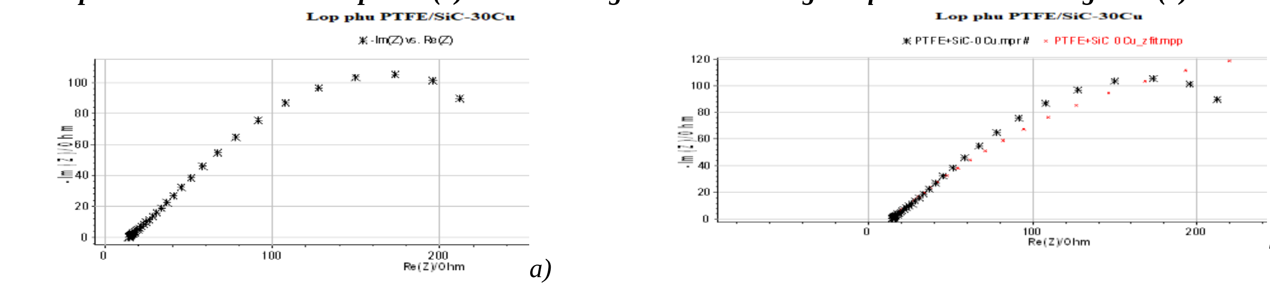
a) *b)*

Figure 4.34. Nyquist impedance spectra of SiC-50Cu/steel in 3.5% NaCl solution. Experimental measurement points (a); Value of fitting according to equivalent circuit diagram (b).



a) *b)*

Figure 4.36. Nyquist impedance spectra of PTFE/SiC-50Cu on steel in 3.5% NaCl solution. Experimental measurement points (a) and matching values according to equivalent circuit diagrams (b)



a) *b)*

Figure 4.37. Nyquist impedance spectra of PTFE/SiC-50Cu on steel in 3.5% NaCl solution. Experimental measurement points (a) and matching values according to equivalent circuit diagrams (b)

From the results of the impedance spectrum analysis, Table 4.19 describes the values of the electrochemical parameters of the SiC-Cu coating system on the C45 steel base in a 3.5% NaCl solution.

Table 4.19. Electrochemical parameters of SiC-Cu/C45 steel coating system in 3.5% NaCl solution

Samples	Electrochemical partameters (Surface area of sample: $1cm^2$)		
	$R_s(\Omega)$	Q(CPE/C_{seal}); (F.$s^{(\alpha - 1)}$)	R_p (Ω)
C45 steel	17.88	0.343E-3 (α = 0.7355)	1002
SiC-30Cu/C45	15.84	0.01903 (α = 0.5642)	514.2
SiC-50Cu/ C45	32.13	18.8E-6 (α = 0.778 9)	3517
PTFE/SiC-30Cu/ C45	14.21	0.01235 (α = 0.5306)	576
PTFE/SiC-50Cu/ C45	32.4	50.13E-9 (α = 0.6345)	369.096

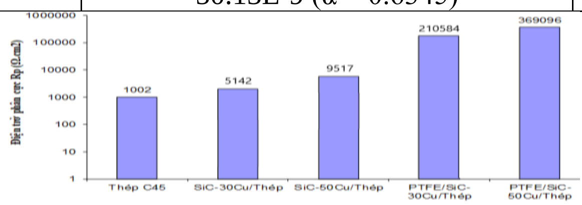


Figure 4.39. Polarization resistance of C45 steel samples with and without coating, after 1 h of immersion in 3.5% NaCl solution.

Figure 4.39 shows the corrosion resistance values of steel with or without protective coatings. When there is no coating, the C45 steel bias resistance is 1002 Ω. This value in SiC-30Cu and SiC-50Cu coatings is 5142 Ω and 9517 Ω, respectively. This bias resistance value is increased with two coating systems PTFE/SiC-30Cu and PTFE/SiC-50Cu, corresponding to values of 210,584 Ω and 369,096 Ω. The SiC-30Cu and SiC-50Cu coatings, have lower bias resistance values due to the porosity associated with inadequate shielding. Porous pores between Cu and steel, galvanic corrosion takes place strongly. The presence of PTFE in combination with SiC-50Cu coating increases the polarization resistance by 370 times compared to C45 steel, so PTFE/SiC-50Cu coating has good protection.

4.4.2. Mass loss of SiC-Cu spray coating on steel base in corrosive medium containing HF acid

Test conditions for 10% HF/20% H2SO4 acid solution + 0.2mm SiC solid particles, temperature 70ºC, rotational speed 1000 r/min

Table 4.20. Mass loss of spray coating sample and C45 steel substrate in 10% HF/20% H2SO4 . acid solution

Mass lost (mg/cm^2)						
Time (hours)	C45	SUS 304	C45 phủ SiC-30Cu	C45 phủ SiC-50Cu	C45 phủ PTFE/SiC-30Cu	C45phủ PTFE/SiC-50Cu
I2	3605	705	57	43	19	14
24	3574	670	*57*	45	18	15
36	3644	687	*56*	48	17	13
48	3700	674	58	51	17	15
60	3755	725	57	45	16	14
72	3624	736	58	44	15	15
84	3674	747	56	46	17	14
96	3756	680	57	45	19	15
108	3742	700	58	44	17	13
120	3778	740	55	46	18	14
136	3704	735	59	46	19	14
Total loss	42556	7809	628	503	192	156

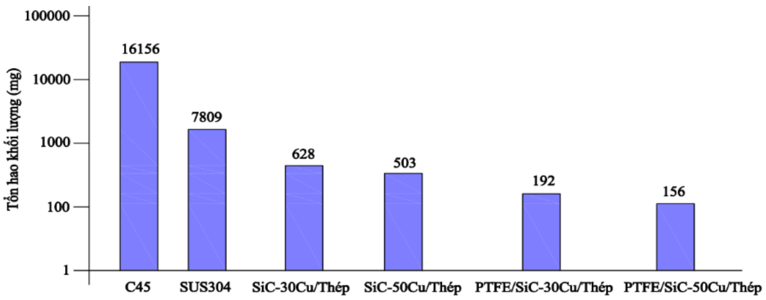


Figure 4.41. Sample mass lost after 136 h in mixed wear tester, with 10% HF/20% H_2SO_4 acidic solution

According to Figure 4.41 (drawn from the data in Table 4.20), the weight of uncoated steel decreased fastest. The following one is the SiC/Cu coated samples, PTFE sealed samples have a very low mass loss, with the lowest is PTFE/SiC-50Cu sample (145 mg after 136 h, Table 4.20). The PTFE coating itself is also reported by many authors for its resistance to corrosion/abrasion thanks to its very low coefficient of friction and very high chemical resistance. Therefore, once the plasma coating could overcome its high porosity, which limits the movement of the chemically aggressive environment, then it could improve the effectiveness of anti-corrosion protection.

The PTFE/SiC-50Cu/steel sample is the least susceptible to corrosion (156mg). This is main research finding of the thesis, i.e. to create a SiC-50Cu plasma coating to protect against corrosion for steel working in an acidic environment containing fluorine.

Conclusion of chapter 4.

- It is very difficult and impossible to spray SiC single coating with plasma.
- The role of copper in the composition of SiC-Cu powder is a binding agent that creates good conditions for the coating formation process. The presence of copper (Cu) has the following effects: Lower the injection temperature to reduce SiC decomposition; Increases adhesion to the substrate.
- With the SiC-Cu plasma coating, the powder parameters can be determined: The particle size S and the mixing ratio H can create a coating with the necessary SiC content and thickness to study the influence of the spray mode on the properties of the coating. coating material.
- The results show that with SiC/Cu composite coating (SiC-30Cu and SiC-50Cu with the corresponding SiC and Cu particle sizes: SiC about 40 - 50 µm; Cu from 20 - 30 µm) on C45 steel substrate in clear The shielding gas reached the required thickness (> 200 µm) and the SiC content in the coating was obtained from 56.4% to 68.7% (Table 4.12).

- The results obtained from the corrosion test methods show that the SiC-50Cu or PTFE/SiC-50Cu coating has the best kinetic corrosion protection in fluorinated acid environments (HF-containing environments).

Chapter 5. DETERMINATION OF PLASMA SiC-50Cu SPRAY TECHNOLOGY SPECIFICATIONS ON C45 STEEL BACKGROUND

5.1. Establish an experimental plan for plasma spraying of SiC-50Cu on C45 steel to evaluate the influence of technological parameters on coating quality.

In order to establish a reasonable regime for SiC-50Cu plasma coating, it is necessary to set up an experiment according to Taguchi's experimental plan [96] on the influence of 3 main technological parameters, which are amperage (I), approx. spray method (L), powder feeding speed (M) to the coating quality criteria are: Adhesion strength: σ (MPa); porosity: γ (%); Microscopic hardness: D(HV) and SiC content in the coating: H(%). Other technological factors are taken as table 5.1

Table 5.1. Fixed selection parameters in experimental study of SiC-Cu plasma on steel substrate.

Paramaters	For SiC-Cu paraying
Voltage, V	58
Primary air flow rate (Ar), L/min	35
Secondary air flow rate (H2 gas), L/min	12
Carrier gas flow rate (Ar), L/min	2
Particle size, m	50 (SiC); 30 (Cu)
Powder mix ratio, % Cu (by mass)	(SiC – 50%Cu)

On the basis of theory and exploratory research results, select survey levels for 3 factors as shown in Table 5.2

Table 5.2. Levels of technological parameters to evaluate the mechanical properties of SiC-Cu plasma coating on C45 steel.

STT	Paramaters	Levels		
		Level 1	Level 2	Level 3
1	Current I (A)	350	400	450
2	Spray distance L (mm)	40	50	60
3	Powder feeding speed M(g/min)	30	40	50

Table 5.3. Measurement results in the fully orthogonal experimental array 3^3

TT	Technical paramaters			Evaluated criteria			
	I (A)	L(mm)	M (g/min)	σ (MPa)	γ (%)	D (HV)	H(%)
1	350	40	30	37,98	1,38	269	45
2	350	40	40	39,20	1,40	257	47
3	350	40	50	38,60	3,92	243	50
4	350	50	30	36,10	1,46	358	65
5	350	50	40	35,40	1,54	350	79
6	350	50	50	34,80	1,98	341	78
7	350	60	30	31,50	3,44	443	61
8	350	60	40	29,80	3,88	434	68
9	350	60	50	30,70	3,96	420	54
10	400	40	30	29,40	2,10	440	73
11	400	40	40	29,20	2,00	438	61
12	400	40	50	29,20	2,20	442	58
13	400	50	30	36,10	1,28	302	58
14	400	50	40	36,00	1,29	307	65
15	400	50	50	32,30	2,10	298	56
16	400	60	30	39,00	2,40	340	59
17	400	60	40	38,20	2,56	338	67
18	400	60	50	34,00	3,82	308	62
19	450	40	30	40,16	4,10	348	61
20	450	40	40	40,00	4,15	334	60
21	450	40	50	38,23	4,48	311	58
22	450	50	30	39,00	4,02	318	61
23	450	50	40	38,40	4,27	322	58
24	450	50	50	34,80	4,37	305	56
25	450	60	30	29,90	4,54	298	56
26	450	60	40	29,50	4,72	291	52

TT	Technical paramaters			Evaluated criteria			
	I (A)	**L(mm)**	**M (g/min)**	**σ (MPa)**	**γ (%)**	**D (HV)**	**H(%)**
27	450	60	50	28,30	4,71	280	49

5.2. Effect of parameters on coating adhesion strength

Table 5.4. Results of ANOVA analysis of the influence of parameters on adhesion strength

Nguồn	DF	SS	MS	F	P
I (A)	2	13,09	6,546	0,42	0,665
L (mm)	2	73,71	36,856	2,35	0,121
M (g/min)	2	20,80	10,400	0,66	0,527

Where: DF (Degree of Freedom) - number of degrees of freedom; SS (Sum of Squares): sum of squares; MS (Mean of Squares)- mean of squares; F- limit value when testing; P - probability value.

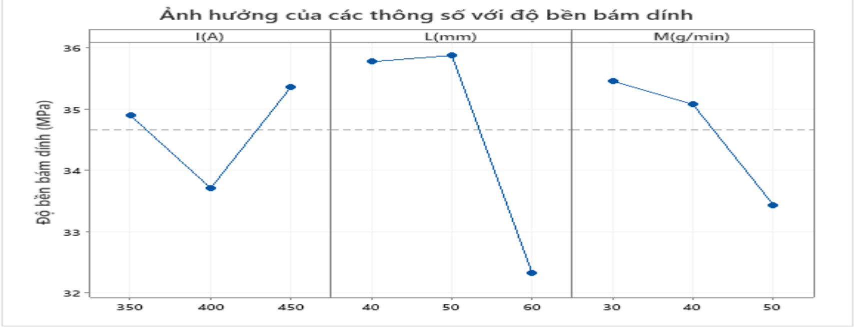


Figure 5.1. Effect of parameters on adhesion strength

he spray distance L has the greatest influence on the adhesion strength (F=2.35), followed by the powder feed rate M (F=0,66) and the least influence is the amperage I (F=0 ,42). This result is also similar to the analysis above.

Table 5.5. The results of solving the optimal problem of finding technological parameters according to the adhesion strength criterion

Values of technical parametrs			Raisonnable value of σ (MPa)	Expectation
I (A)	L(mm)	M (g/min.)		
450	50	30	37.387	0.766

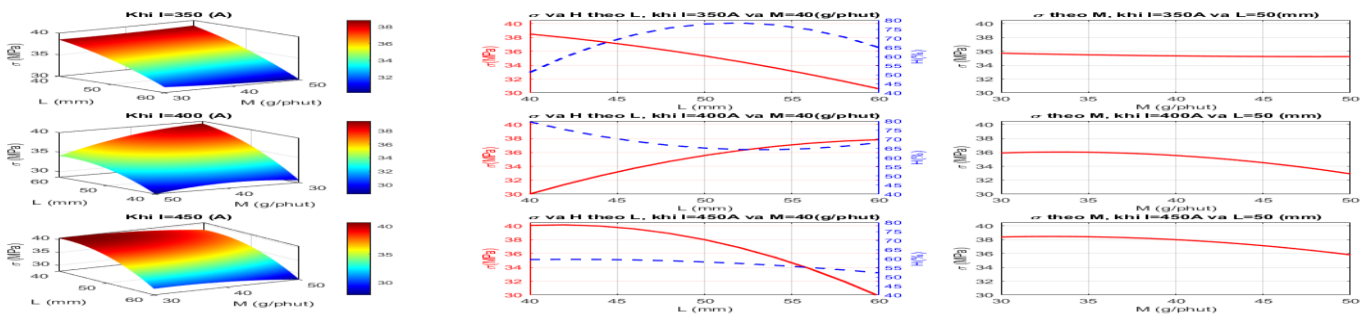


Figure 5.2. Graph of adhesion stress relationship according to L and M.

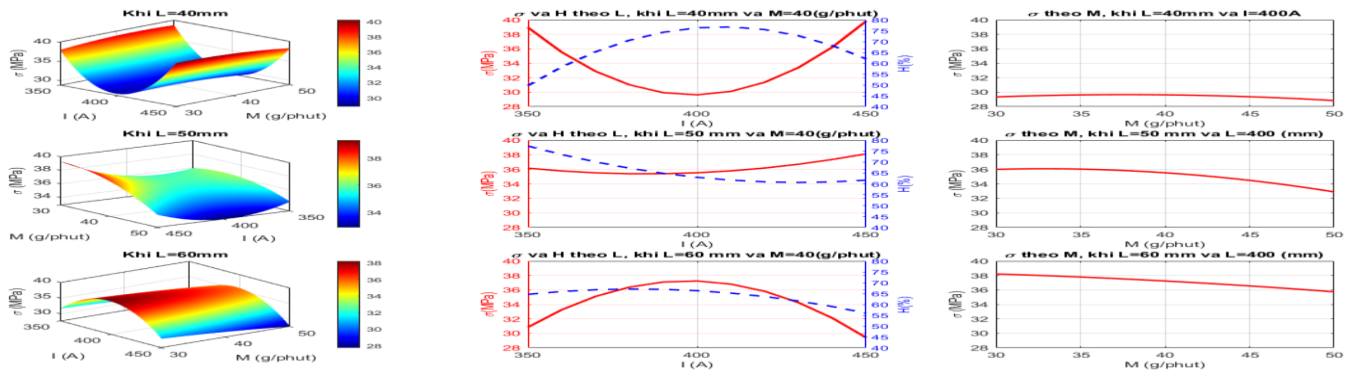


Figure 5.3. Graph of adhesion stress relationship according to I and M

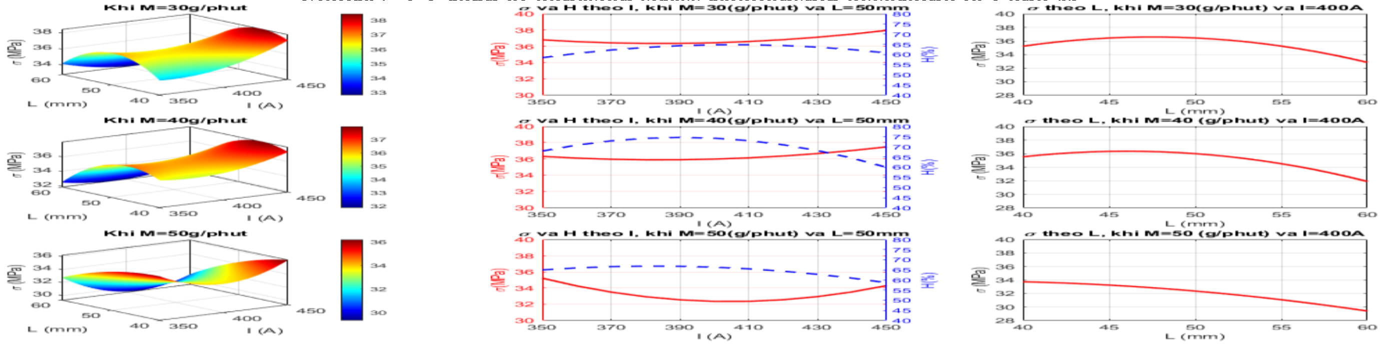


***Figure 5.4. Graph of adhesion stress relationship according to I* and L**

Comment on the effect of plasma spraying on coating adhesion strength:

- Effect of current I:

The effect of current I on the adhesion strength is ranked 3rd out of 3 parameters. When I= increases from 350 A to 400 A, the adhesion strength tends to decrease, and when I = increases from 400 A to 450 A, the adhesion strength tends to increase. Thus, the amperage for better adhesion strength, the reasonable value of amperage to achieve high adhesion strength is I=450 A (table 5.5).

- Effect of spray distance L:

The effect of spray distance L on the adhesion strength is ranked 1st out of 3 parameters. When L increases from 40 mm to 50 mm, the adhesion strength increases, and when L increases from 50 mm to 60 mm, the adhesion strength decreases rapidly. Spray distance L from 40 ÷ 50 mm gives better results for adhesion strength. The reasonable value of L to achieve high adhesion strength is L=50 mm (table 5.2), then the percentage of SiC in the coating is also high above 60%.

- Effect of powder feeding speed M:

The influence of powder feed rate M on adhesion strength is ranked 2nd. When M increases from 30 g/min to 50 g/min, the adhesion strength tends to decrease more and more strongly. Thus, the average powder feeding speed for better adhesion strength, the powder feeding speed to achieve high adhesion strength is M=30 g/min. The values of 3 technological parameters for high adhesion strength and good SiC content in coating are: I = 450A; L = 50mm; M = 30g/min

5.3. Effect of parameters on coating porosity

Using MATLAB software for porosity, the regression function is obtained:

$$\gamma = 70,3 + 0,00064I + 0,03L + 0,72M - 0,002I.L - 0,015L.M - 0,0021I.M + 0,00064I^2 + 0,008L^2 + 0,003M^2 + 0,0000345I.L.M \quad (5.4)$$

Table 5.6. Results of ANOVA analysis of the influence of parameters on porosity

Source	DF	Seq SS	Adj SS	Adj MS	F	p-value
I (A)	2	13,09	6,546	0,42	0,665	13,09
L (mm)	2	73,71	36,856	2,35	0,121	73,71
M (g/min)	2	20,80	10,400	0,66	0,527	20,80

Table 5.7. Table of results of optimal problem solving to find reasonable technological parameters according to porosity criteria

Source	**DF**	**SS**	**MS**	**F**	**P**
I (A)	2	95,21	95,21	47,607	1,14
L (mm)	2	23,42	23,42	11,708	0,28
M (g/min)	2	11,25	11,25	5,625	0,13

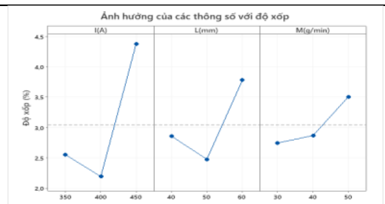


Figure 5.5. Effect of parameters on porosity

The results of ANOVA analysis showed that the current intensity I had the greatest influence on the porosity (F=47,607), followed by the injection distance L (F=11,708) and the least influence was the powder feeding rate M (F=5,625).

Solving the optimization problem to find the reasonable values of the technological parameters to the porosity index with the goal of adhesion strength as small as possible is the result as shown in Table 5.8.

Table 5.8. Table of results to solve the optimal problem of finding technological parameters according to the adhesion strength criterion

Technical values			Raisonnable value of γ (%)	Expectation
I (A)	L(mm)	M (g/phút)		
400	50	30	1.7	36.92

From the experimental results in Table 5.3 and the experimental planning equation (5.4), the porosity relationship graphs with each pair of technological parameters are shown in Figure 5.6 ÷ 5.8.

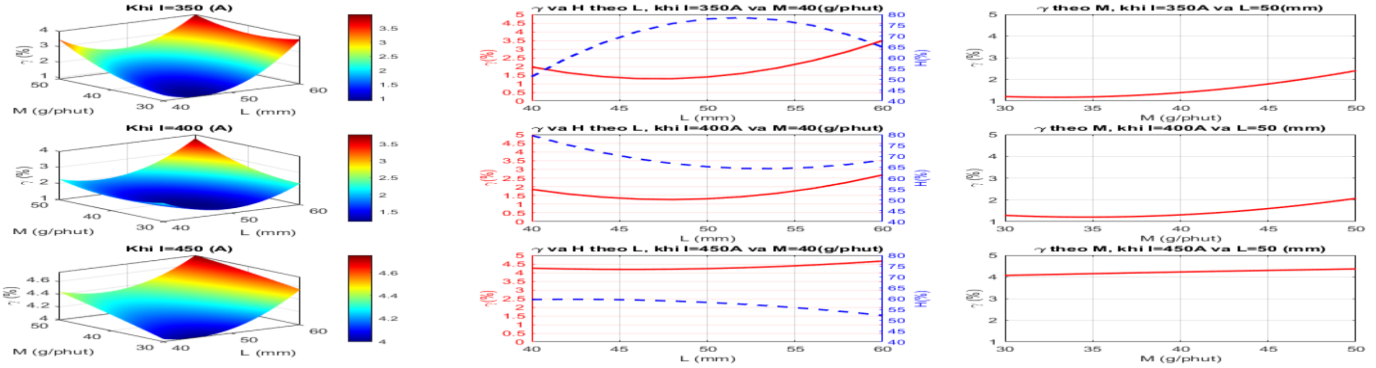


Figure 5.6. Graph of porosity relationship according to L and M.

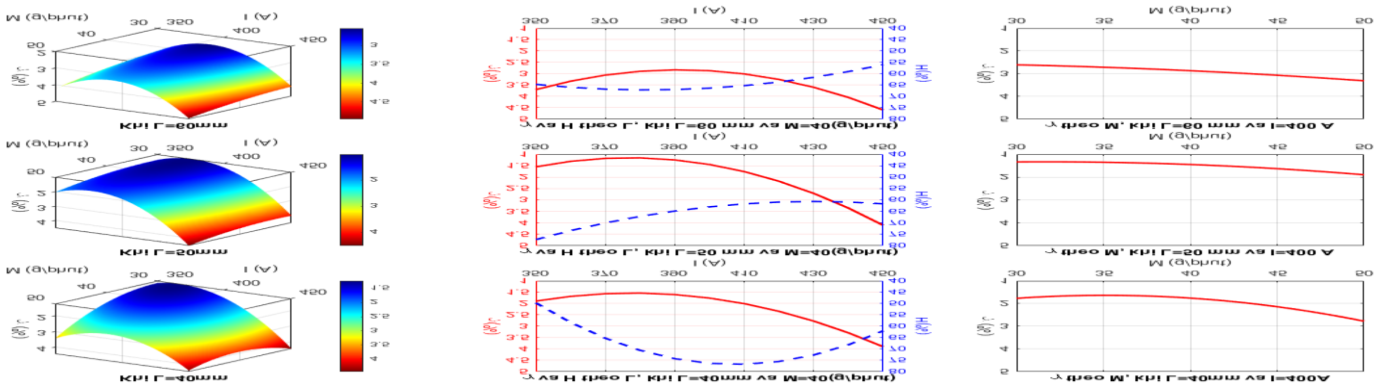


Figure 5.7. Graph of porosity relationship according to I and M.

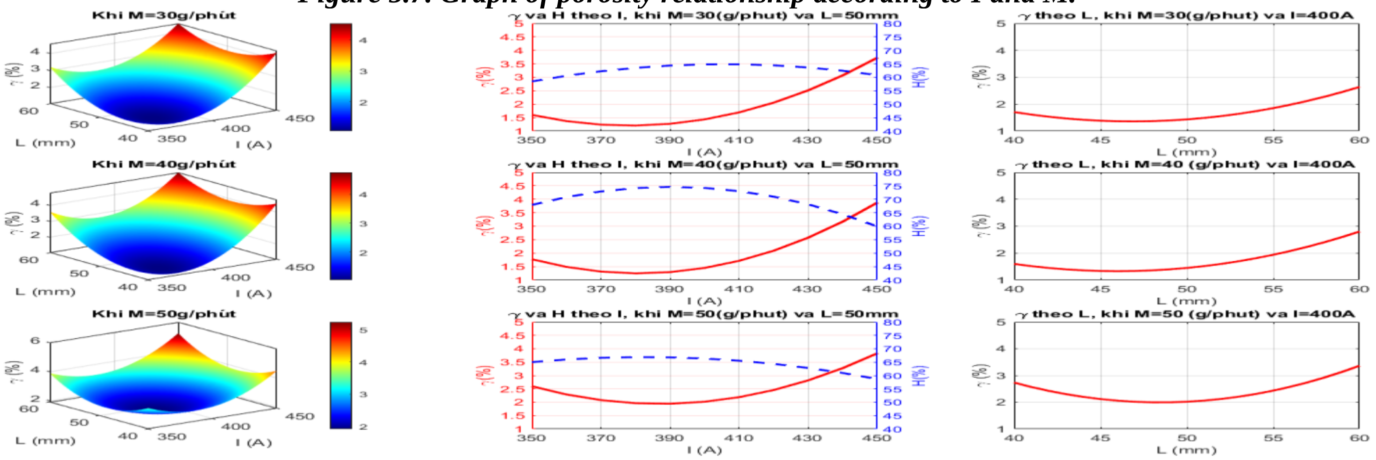


Figure 5.8. Graph of porosity relationship according to I and L.

Comment on the effect of plasma spraying on coating porosity:

- Effect of current I:

The effect of current I on porosity is ranked 1st out of 3 parameters. When increasing the spray distance L from 40-60 mm, porosity tends to increase then decrease slowly; when I=400 A, when increasing the spray distance from 40 to 60 mm, the porosity decreases then increases slowly. M increased from 30-50 g/min, porosity decreased then increased. When: I = 400A, L= 50 mm and M= 40g/min for smaller porosity. When I= increases from 350 A to 400 A, porosity decreases slowly, and when I= increases from 400 A to 450 A, porosity increases rapidly. A reasonable value of amperage for good porosity is I=400 A

- Effect of spray distance L:

The effect of injection distance L on porosity is ranked 2nd out of 3 parameters. When L increases from 40 mm to 50 mm, porosity decreases, and when L increases from 50 mm to 60 mm, porosity increases rapidly. This shows that the spray distance L from 40-50 mm gives better results for porosity. The reasonable value of the spray distance to achieve high porosity is L=50 mm (table 5.8), then the SiC content in the coating is also high above 60%.

- Effect of powder feeding speed M:

The effect of powder feed rate M on porosity is ranked 2nd, the smaller the porosity the better. When increasing the current I from 350-450A, the porosity tends to increase then decrease; Porosity is smaller when M=30g/min. When M increases from 30 g/min to 50 g/min, porosity tends to increase. Reasonable value of 3 technological parameters for high porosity and good SiC content in coating. I = 400A; L = 50mm; M = 30g/min

5.4. Effect of parameters on Coating Microscopic Hardness

Using MATLAB software for the Microscopic Hardness criterion, the regression function is obtained:

$$D = -3340 + 16,1I + 18,7L + 1,34M - 0,105I.L + 0,027L.M + 0,0043I.M - 0,014I^2 + 0,241L^2 + 0,044M^2 - 0,0000937I.L.M \quad (5.5)$$

Table 5.9. Results of ANOVA analysis of the influence of parameters on Microscopic Hardness

Source	DF	SS	MS	F	P
I (A)	2	9974	4987,1	1,28	0,301
L (mm)	2	3728	1864,1	0,48	0,628
M (g/min)	2	1681	840,3	0,21	0,808

Table 5.10. Table of results of optimal problem solving to find technological parameters according to the criterion of Microscopic Hardness

Tech. values			Raisonnable σ (HV)	Ecxpected
I (A)	L(mm)	M (g/phút)		
400	50	30	376,78	0,69

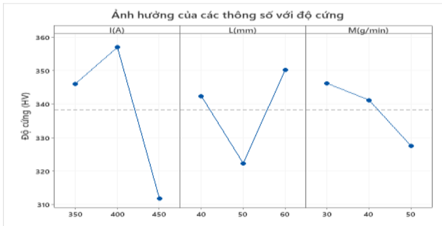


Figure 5.9 Effect of parameters on Microhardness

From this result, it is shown that the current intensity I has the greatest influence on the Microhardness (F=1,28), followed by the injection distance L (F=0.48) and the least influence on the speed of the micro-hardness. flour grade M (F=0,21).

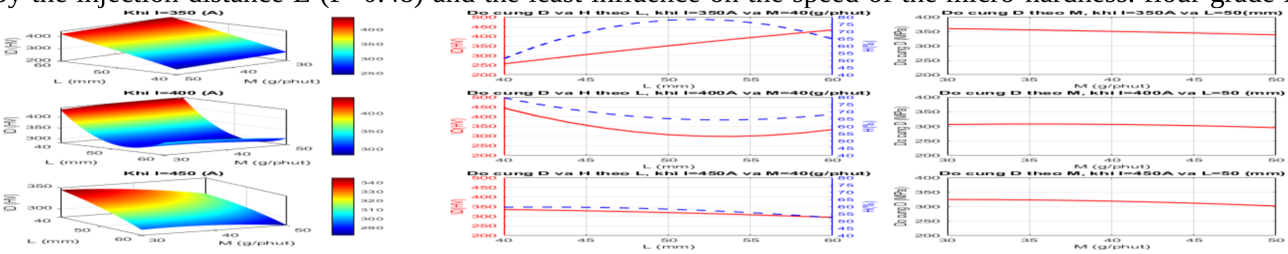


Figure 5.10. Graph of the relationship of Microhardness according to L and M.

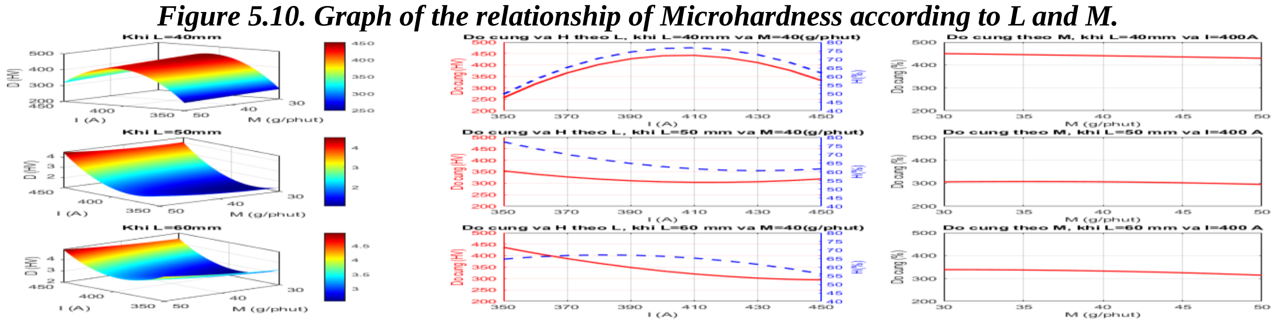


Figure 5.11. Relationship graph of Microhardness according to I and M.

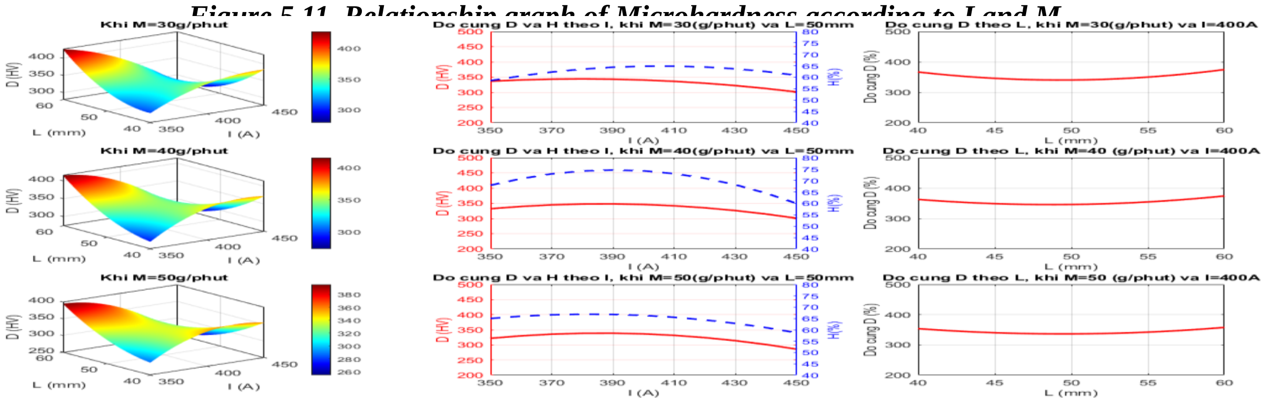


Figure 5.12. Relationship graph of Microhardness according to I and L.

Comments on the effect of plasma spraying on the coating microhardness:

- Effect of current I:

Amperage I mainly affects the microhardness of the coating. Research value range I=[350,400,450] A. ANOVA analysis, when I increases from 350A to 400A, Microhardness increases slowly, and when I increases from 400A to 450A, Microhardness decreases rapidly. The reasonable value of amperage to achieve high Microhardness is I=400A (table 5.10).

- Effect of spray distance L:

Effect of injection distance L on Microhardness is ranked 2nd out of 3 parameters. The results of ANOVA analysis showed that, L increased from 40 mm to 50 mm, Microhardness decreased, and when L increased from 50 mm to 60 mm, Microhardness increased rapidly. This shows that the injection distance L>50 mm gives better results for Microhardness. Thus, the spray distance for better Microhardness is L=50 mm (table 5.10), then the SiC content in the coating is also at a high level of over 60%.

- Effect of powder feeding speed M:

When M increases from 30 g/min to 50 g/min, the Microhardness tends to decrease. Thus, low M gives better Microhardness, the value of feed rate to achieve high Microhardness is M=30 g/min (table 5.10). Thus, the spray distance has the greatest influence on coating mechanical properties, followed by I and M. The reasonable values of I, L, M are: I=400A, L=50mm and M=30g/min.

5.5. Effect of parameters on the proportion of SiC in the coating

Using MATLAB software to solve the system of regression equations for the content of SiC:

$$H = -1060 + 3,65I + 11,8L + 9,18M - 0,0175I.L - 0,077L.M - 0,013I.M - 0,0033I^2 - 0,046L^2 - 0,0462M^2 + 0,000176I.L.M \quad (5.6)$$

Table 5.11. Results of ANOVA analysis of the influence of parameters on the proportion of SiC in the coating

Source	DF	SS	MS	F	P
I (A)	2	338,7	169,33	2,03	0,158
L (mm)	2	134,0	67,00	0,80	0,462
M (g/min)	2	134,2	67,11	0,80	0,462

Table 5.12. Table of optimal results to find technological parameters according to the content of SiC in the coating

Values			Raisonnable H (%)	Expected
I (A)	L (mm)	M (g/phút)		
400	50	40	71.55	0.676

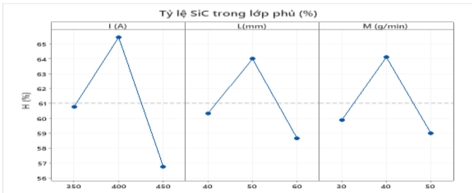


Figure 5.13. Effect of parameters on the proportion of SiC in the coating.

The current intensity I has the greatest influence on the content of SiC in the coating (F=2.03), while the injection distance L (F=0.67) and the powder feed rate M (F=0.67) have same effect on the SiC content in the coating.

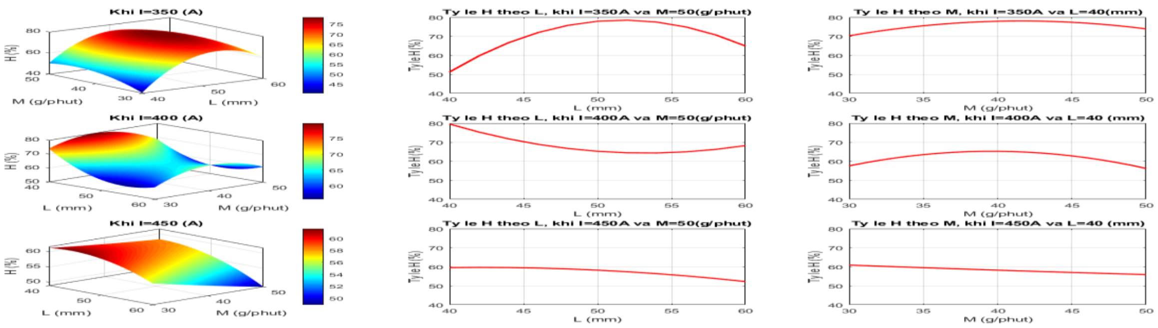


Figure 5.14. The graph of the content of SiC in the coating according to I and M

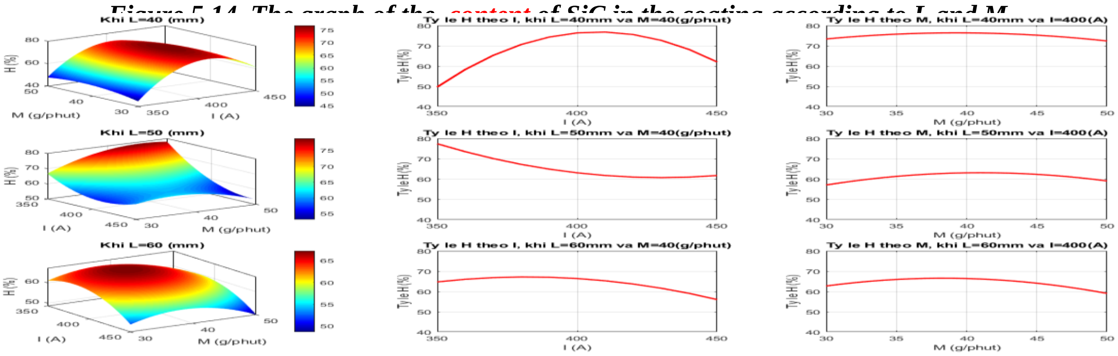


Figure 5.15. Graph of SiC content in the coating according to I and M.

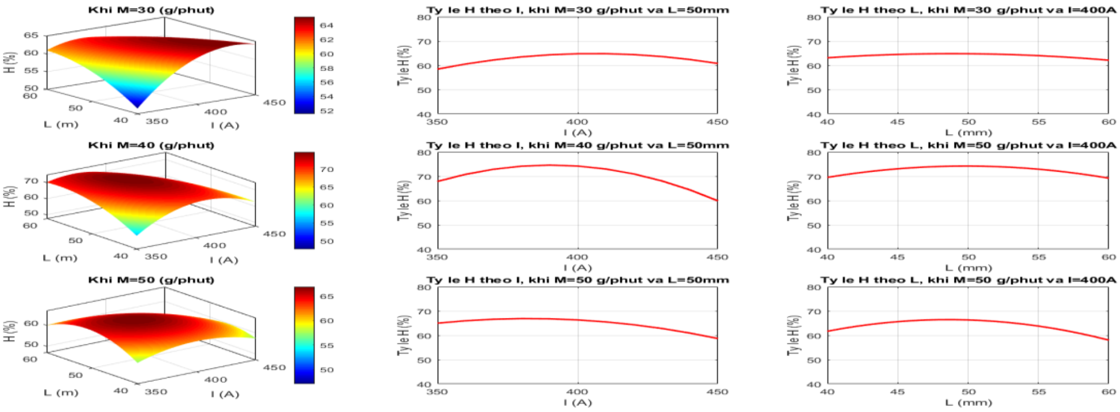


Figure 5.16. Relationship of SiC content in the coating according to I and L.

Comments on the effect of plasma spraying on the SiC content in the coating:

The influence of current I on the proportion of SiC in the coating is ranked 1st, followed by L and M. The injection distance L and powder feeding rate M have relatively strong influence on the SiC content in the coating. Graph 5.14 ÷ 5.16 shows that, when I increases from 350 A to 450 A, when increasing the spray distance L from 40-60 mm and M from 30 to 50 g/min, the SiC content in the basic coating follows the rule. increase then decrease. ANOVA analysis, in the range I increased from 350A to 400A, L increased from 40 to 50 mm, M increased from 30 to 40 g/min, the percentage of SiC in the coating tended to increase, and when I increased from 400A to 450A , L increased from 50 to 60 mm, M increased from 40 to 50 g/min, the percentage of SiC in the coating decreased. Amperage values to achieve a high SiC content in the coating are: I=400A, L=50 mm and M = 40 g/min'

5.6. General assessment of the influence of technological parameters on the quality of SiC-50Cu coating for wear resistance in experimental environment

In order to determine the technological parameters while achieving the adhesion strength as high as possible, the porosity as small as possible, the hardness as high as possible, and the SiC content in the coating ≥ 60%, using MINITAB software to solve the problem. The problem of analysis and optimization of 4 criteria at the same time for 3 technological parameters in the survey area obtained reasonable results of technological parameters as shown in Table 5.13.

Table 5.13. Table of results to solve the optimization problem of finding reasonable technological parameters with four criteria ***(σ, γ, D và H)*** at the same time

Technical values			Reasonable values of the criteria				Correlation
I (A)	L(mm)	M (g/phút)	σ (MPa)	γ (%)	D (HV)	H(%)	
416,484	46,514	32,625	37,472	2,153	346,3	61,73	0,898

This result means that, when using plasma spraying technology to create SiC-50Cu silicon carbide coating on C45 steel to protect against corrosion in acidic fluorine-containing environment, reasonable values of the technological parameters I, L, M for both mechanical properties (3 criteria) and wear resistance criteria (SiC content in the coating ≥60%) are:

I=416.484 A, L=46,514mm and M=32.625g/min.

Rounding to determine the set of technological parameters used for plasma coating SiC-50Cu

I=416A, L=45mm and M=32 g/min.

Conclusion of chapter 5

- Select a fully orthogonal planning method for experimental design to determine and optimize spray parameters affecting coating properties. Determine the influence of spray technology parameters (I, L, M) on coating properties.

- For the index of adhesion strength: The spray distance L has the greatest influence on the adhesion strength, followed by the powder feeding speed M and the least effect is the amperage I. At the value of 3 Technological parameters, Medium spray distance, low powder feed rate and high amperage for higher adhesion strength. Technological parameters for high adhesion strength are: I = 450A; L = 50mm; M = 30g/min.

- For porosity index: the current intensity I has the greatest influence on porosity, followed by the injection distance L and less influence on the powder feed rate M. With the goal of coating porosity as small as possible, good, the average injection distance for small porosity, lower amperage and feed rate for smaller porosity. The technological parameters to simultaneously achieve the lowest porosity and the highest adhesion strength are: I = 356,878A; L = 52,711 mm; M = 30g/min.

- For hardness criteria: the amperage has the greatest influence on the mechanical properties of the coating's hardness, the spray distance is less affected and the speed of the level has the least influence. The average injection distance in the study area gives high hardness, while lower amperage and feed rate give better hardness. The technological parameters to simultaneously achieve the lowest porosity, adhesion strength and highest hardness are: I=382A, L=53,884mm and M=33.182g/min.

- Regarding the content of SiC in the coating: the amperage I has the greatest influence, while the injection distance L and the powder feeding speed M have the same influence. The average value of the 3 parameters gives the highest percentage of SiC in the coating. The technological parameters for high adhesion strength are: I = 400A, L = 50mm and M = 40g/min.

- The influence of technological parameters I, L, M simultaneously on the group of mechanical properties (3 indicators) and wear resistance (SiC content in the coating ≥60%) in SiC-50Cu plasma spraying create a silicon carbide coating on C45 steel, the set of technological parameters are: I=416A, L=45mm and M=32 g/min.

CONCLUSIONS

1. This is the first project in Vietnam to successfully research the technology of creating SiC/Cu plasma coating on steel surface.

2. Determining reasonable powder parameters (mixing ratio, SiC, Cu particle size) and successfully fabricating SiC-30Cu coating; SiC-50Cu on C45 steel by plasma spraying method with required thickness (200 µm) and SiC composition as high as 71%.

3. Improved method of plasma spraying technology: Design and manufacture of Ar gas shield to protect the plasma spray stream, leading to when spraying on C45 steel with SiC-30Cu and SiC-50Cu, achieving coating quality criteria:

- Coating thickness: 204.67 ± 26.5 µm and 220 ± 20.5 µm.
- Low porosity: 1.4%.
- SiC composition in the coating achieved: 53%.

Finishing SiC/Cu coating on steel surface with PTFE penetrant to overcome porosity improving corrosion protection.

4. Design and manufacture a set of mixed wear test equipment according to actual conditions (sampling speed in abrasive grains 1000 v/min; acid mixture 10% HF; 20% H2SO4 at a temperature of 70ºC) to provides an integrated wear assessment method for weight loss over time.

5. The results of measuring corrosion protection for C45 steel when using 3.5% NaCl solution of SiC-Cu composite coating systems with or without PTFE penetration show that the two coating systems are SiC. -50Cu and PTFE/SiC-50Cu both strongly reduced the corrosion current of C45 steel from 9.44 to 5.6 and 0.59 µA/cm2. With PTFE/SiC-50Cu permeable coating increases the polarization resistance of C45 steel by nearly 370 times, which increases the effectiveness of corrosion protection.

6. The mass loss measurement results after 136 hours of testing in mixed wear test equipment show that the SiC-50Cu/steel or PTFE/SiC-50Cu/steel test pieces have very low loss mass, respectively. are 503 mg and 156mg, much lower than that of stainless steel SUS304 (7809mg).

7. Experimental research has been carried out using the 3^3 fully orthogonal planning method to evaluate the influence of the spray technology parameters I, L, M on the coating quality parameters and the ratio. SiC content in the coating. Analyzing and identifying experimental regression functions that allow to evaluate the influence of technological parameters I, L, M on each indicator and group of criteria. The set of technological parameters for plasma spraying to create SiC-50Cu coating while achieving high adhesion strength, low porosity, hardness and high SiC content in the coating are:

I=416A, L=45mm và M=32 g/min.

8. PTFE/SiC-50Cu plasma coating is a coating that can be applied for corrosion protection of C45 steel in acidic fluorine environments.

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