EFFECT OF THE ELECTRODEPOSITION CONDITIONS ON THE EROSION CORROSION OF AUSTENITIC STAINLESS STEEL 316L USING RESPONSE SURFACE METHODOLOGY (RSM)

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ABSTRACT

This study represents an attempt to improve the erosion corrosion resistance for industrial pumping station used to produce sulfuric acid through electrodeposition coating samples of Austenitic stainless steel 316L. Minitab 19 program was used to precipitate a composite coating layer (Cr-WC) because it is used in many industrial applications that require corrosion resistance, As well as high hardness. The samples were coated using different variables as inputs: temperature(T) ,WC concentration(WC wt% ), stirring velocity (r.p.m) and deposition time (t) for four levels using central composite design for design laboratory experiments. The factors of coating thickness (C.T), surface roughness (Ra) and micro- hardness (HV) were adopted as outputs response and a complete analysis of variances (ANOVA) at constants significances levels of %5, was done to fully identify the most significant parameters.

The surface response methodology (RSM) was used to provide an optimized model with the best optimum properties such as micro-hardness, surface roughness, coating corrosion rate and oxidation corrosion rate to improve the corrosion resistance of Austenitic 316L stainless steel samples..

The results showed respectively that the wc wt.% was the most effective on all the outputs response then deposition time (min), temperature(T) and stirring velocity (r.p.m).

Key words: Austenitic stainless steel 316L, Electroplating, Cr-WC Composite Coating Layer, Hardness,Erosion corrosion resistance, Response Surface Methodology (RSM), Central Composite Design (CCD).
1. Introduction

Erosion-corrosion has many definitions. Erosion-corrosion is the strength of the corrosive assault due to the corrosion and erosion simultaneously. The attack is harsher than if only corrosion or erosion were acting alone. The erosive action extracts metal from the surface as dissolved ions, as solid corrosion products particles or as elementary metal. Corrosion from erosion may primarily be an erosive attack, or primarily a chemical attack, or somewhere between. All gases and liquids can cause an assault. Almost all volatile corrosive media can cause erosion. Erosion-corrosion has grooves, waves, rounded holes and/or shaped grooves in nature. The affected areas are typically free of deposits and corrosion products, while corrosion products can sometimes be found if erosion-corrosion occurs intermittently and/or the liquid flow rate is relatively small [1,2]. During such turbulences, erosion starts to strip the passive layer from the metal surface. When this removal is quicker than the re-passivation of the stainless steel, this component is quickly damaged by erosion-corrosion. Erosion-corrosion happens only in areas of high liquid speed [3].

Several parameters are applied to corrosion erosion[4,5]; • angle of impact; • hardness of material; • particle size; • particle shape; • type of material; • protective layer; • velocity of flow; • fluid temperature.

That material must have certain physical, mechanical and corrosion-resistant properties when selecting a construction material for a particular application. Cost is also a big consideration in the selection process. While there may be several alloys available to meet the requirement requirements, the cost of such alloys may be prohibitive. As a result, many coating and lining materials have been created that can be applied to less costly construction materials to provide the required protection against corrosion [6].

It is well known that stainless steels owe their resistance to corrosion primarily to the creation of passive films on the alloy surface. Stainless steels show good corrosion resistance where the passive films formed on the surface are stable in oxidizing corrosive mediums. In many reductive corrosion media, however, such as boiling dilute solutions of sulfuric acid or boiling acetic plus formic acids, Passivity can not be formed continuously on the surface and aggressive corrosion occurs in stainless steels. For passive-fitted alloys such as titanium alloys or stainless steels, If the corrosion potential of the alloy is increased by applied anodic current or by alloying with elements with higher oxidation / reduction potential from active potential into the passive zone, the corrosion resistance will be improved. [7]

Composite deposition is a protective coating technique that incorporates homogeneously inert particles of a different substance in the metal matrix; this two-phase coating improves mechanical resistance and corrosion. Improving these properties depends on the combination of the metal matrix and the particles. The electrodeposition of composite coatings consists of a metal or alloy matrix consisting in second phase hard particles such as Al2O3, TiO2, SiC, WC or SiO2 [8,9].

The Purposes of this study are to protect steel by The electrodeposition technique process is economically and to develop this process this work is to deposit hard chromium from a hexavalent chromium bath and Tungsten Carbide simultaneously on the surface of austenite
stainless steel 316L. The main objective of this work is to increase the resistance to corrosion, microhardness, friction coefficient and to select the best coating condition.

Response Surface Methodology (RSM): is a synthesis of mathematical and statistical (hybrid) methods,; the main purpose is design experience, modeling and analyzing of input parameters influencing on output responses and improvement of these parameters by decrease the number of experiments needed to optimize the operation and give statistical deduction on the optimum conditions and also helps in limiting the relationship between the controllable input parameters and the progressing response roof. RSM is supposed to be a technique of experimental strategy first defined in 1951 by Box and Wilson to restrict the best conditions for multivariate systems [10]-[13].

In this study, the influence of the deposition coating conditions 316L alloy samples of austenite stainless steel are investigated on the physical and mechanical properties using RSM method. Response surface methodology (RSM) mathematical models are also developed for the prediction of the surface roughness (Ra), coating thickness (C.T), micro-hardness(HV) corrosion rate (C.R) and erosion corrosion rate (E.C.R) under various coating conditions.

2. Experimental Methods

2.1 Alloy Substrate

In this study, the Austenitic stainless steel 316L was used as substrate. Table 1[14] indicates the nominal composition of Austenitic 316L stainless steel in wt. per cent. Whereas Table 2 indicates Spectrochemical analysis of 316L alloy Austenitic stainless steel in wt. percent

Table 1: Limiting chemical composition of austenitic stainless steel in wt. % according to[14]

| EL  | Fe  | C   | Mn  | Si  | Cr  | Ni  | P   | S   | Mo | V  | Cu |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|----|----|----|
| Wt.%| bal.|<0.02-|2.0  |1.0  |16-18|10-14|_    |_    |2-3 |_   |_   |
|     |     |<0.03|     |     |     |     |     |     |     |     |     |

Table 2: Spectrochemical analysis of austenitic stainless steel 316L in wt. %.

| Metal | C   | Si  | Mn  | P    | S    | Cr  | Ni  | Mo  | Cu  | Fe |
|-------|-----|-----|-----|------|------|-----|-----|-----|-----|----|
| Wt.%  | 0.0536|0.352|1.07 |0.0423|0.0021|17.28|11.51|2.62 |0.261|Bal.|

Austenitic stainless steel 316L samples have been cut by electric cutting machine (ACRA) to disk shape with diameter (10mm) and length (10mm) dimensions. All surfaces were grinding with silicon carbide papers in sequence of grit 180, 220, 320, 400, 600, 800, 1000, 1200, 1500,
2000, 2500, 3000 to obtain flat and scratch-free surface. These samples were polished using alumina solution with polishing cloth and cleaned with distilled water, Acetone degreasing and ultrasonic cleaning using ethanol as a medium for thirty minutes. The samples are placed in a glass case after drying.

2.2 The electrodeposition Coating

2.2.1. The steps of the work of Electrodeposition of Cr–WC composite coating

First step: - Preparation of coating solutions:

| Table 3: Materials used to obtain coating solutions |
|---------------------------------------------------|
| Material                                           | Amount wt. % | Supplier   | Purity % |
| Hexavalent chromium oxide                         | 250 g/l      | Canning    | 99       |
| Tungsten Carbide                                  | 10,20,30 g/l | CBC India  | 99.9     |
| Sulfuric acid                                     | 2.5 m/l      | Sigma-Aldrich Co.LLC | 99.77 |

These materials are added to distilled water to complete one liter. Initially it was added chromium oxide (CrO₃) to form a solution dichromic acid (H₂Cr₂O₇) and mixing until it dissolves. Secondly, the Sulfuric acid is adding it to the previous solution to be completed one liter and add tungsten carbide (WC) at the end of each stage after replacing the solution.

Second step: - Electroplating work: -

1- Cleaning the samples in alkaline after completing the grinding process with silicon carbide papers in sequence of grit 600 according to the American specifications meaning for 5 minutes.
2- Samples are washed with distilled water after alkaline solution and then immersed for 4 minutes in a dilute solution of HCl acid concentration before dilution 33% then washed with water 3 times and attached to the cathode wire and finally run the stirrer at the speed determined by the variables of each experiment and determine the current according to the voltage and sample area.

2.2.2. The electroplating coating process:-

This has been tested by putting the solutions in the plating bath and evaluating the positive electrodes and preferred materials for plating samples. The bath was prepared with its current intensity and necessary voltages for the coating process at the appropriate temperature to avoid
changes in concentration of the solution obtained due to loss of evaporation as shown in Table 4

Significant parameters for treating traditional electrochemical deposition coatings..

The thickness of the coating layer defines the time period for putting the samples in the plating bath and the current density. After the appropriate time of precipitation a certain thickness of the coating is made of the metal. Samples are taken from the washed plating bath with distilled water, alcohol and dried with hot air. After the weight measurement is taken and the samples are then stored in a glass desiccator to remove the wetness that causes corrosion. The electrochemical cell used in this work, coating solution and sample shape (Ideal sample after RSM testing) after coating process is shown in figure 1 below.

Table 4: Major processing parameters for a typical electrochemical deposition coating.

| Plating Bath |                     |
|--------------|---------------------|
| wc particles size | 4.531 μm |
| PH | 2 |
| Current Density | 38 A/cm² |
| Voltage | 6 V |

Figure 1: A) Shows plating device. B) The optimum sample after coating process by (Cr-WC) layer

2.3. Vickers Hardness Test :-

Hardness of the coating layers were measured by using Digital Micro Vickers Hardness Tester TH 717), at the materials lab./ Babylon University, Fig.(3.13). This process was carried out distribution at coated layer for SS316L, SS316L-Cr, SS316L-Cr-WC and base metal before and after heat treatment. A load of 25 g was applied for 30 sec. Three readings were recorded for each specimen coated and one at the substrate. Then, the average value was taken.

2.4. Coating Thickness Measurements: -

This research was performed at Materials Eng. Babylon University Labs./. Coating thickness gage was performed using a Chinese-origin digital coating thickness gage system (TT 260), the precision of the tool (0.1μm±). This test was carried out to determine the thickness of the coating layer on the surface samples by taking them in three places to give an average thickness.

2.5. Surface Roughness Test :-

This test was carried out at the Materials Eng. Labs./ University of Babylon. Surface roughness device type (TT 200) of Chinese origin has been tested Measuring the surface roughness of the sample by a sensor that records the surface roughness of the sample and takes data directly from the screen of the device, and measuring the surface roughness at all three locations to provide an average surface roughness.

2.6 Simple Immersion Test (weight loss measurement):-

Weight loss method: The easiest way to calculate a metal's corrosion rate is often called clear immersion method. In this method specimens are for a period of time immersed in sulfuric
acid. Job performed in this respect in Al Furat General Company for Petrochemical Industries and Pesticides in Al Saddah. Sulfuric acid was used which have concentrations (98%) for Immersion solution. disc sample with (10*10) mm length and Diameter respectively and sample area 471mm². The volume of the immersion solution was 94 ml agreeing to (G 31) ASTM "Specimen area solution volume" ratio is 0.20 mL / mm². the Corrosion rate was calculated every three days for a period of 30 days. and entering the results into a program in responses surfaces methodology (RSM) To find the Optimum sample. Finally a plot of the corrosion rate Vs. Time, was carried out.

2.7. Erosion Corrosion Test:-

In this test, device used was manufactured locally in Al Furat State Company for Chemical Industries and Pesticides agreeing to (G 73) ASTM. This erosion-corrosion test involves stainless steel 316L tank, motor with discharge 53 L/min, head max 38m, and tubes to project the chemical solution on the surface of specimens from small nozzle. The nozzle with (1 mm) diameter and located at an immovable distance of (10 mm) from the end of surface specimen. The impact angle between chemical solution and surface specimen is 90°. Calculating the corrosion rate and entering the results into a program in responses surfaces methodology (RSM) to find the Optimum sample.

2.8. Design of the experiments & mathematics models using RSM method

Centrals composites designs (CCD) during responses surfaces methodology (RSM) was selected to design the experiments. Three levels for each affecting parameters were consider as shown in Table (5).

Experiments were performed in this study to determine the impacts of the coating conditions: temperature(T) deposition time (t), wc concentration (wt.%) and stirring velocity (r.p.m) on the responses represented by surface roughness (Ra), coating thickness (C.T), Micro-hardness (HV), corrosion rate (C.R) and erosion corrosion rate (E.C.R) for Austenitic stainless steel 316L. Responses surfaces methodology’s (RSM) was selected centrals composites designs (CCD) to design the present study experiments. In this study, three levels were taken for each input parameters effecting on the output parameters as shown in the Table 5.

Table 5: Input process parameters and their levels.

| Actual-factors | Parameters          | Units | Actual levels |
|----------------|---------------------|-------|---------------|
| Wc(wt.%)       | Concentration Of WC | g/L   | 10 20 30      |
| r.p.m          | Stirring velocity   | r.p.m | 800 1200 1600 |
| t              | time                | Min   | 20 40 60      |
| T              | temperature         | °C    | 40 50 60      |

3. RESULTS AND DISCUSSION
3.1. Experimental Results of coating process

Central Composite Design (CCD) is used to design the current research studies, involving eight corner points, six as axial points and six as central points, resulting in a total of twenty trials for three factorial input factors in one block. The findings of those studies have been clearly illustrated in Table 6.

### Table 6: Experimental data for input and output Parameters.

| Deposition conditions (input parameters) | output parameters |
|------------------------------------------|------------------|
| Temperature C° | Concentration of WC g/L | Deposition t (min) | Stirring velocity (r.p.m) | Erosion C.R *10^-5 gm/Cm^2.day | C.R *10^-5 gm/Cm².day | Micro Hardness HV | Coating Thickness (C.T μm) | Surface roughness (Ra μm) |
|----------------|--------------------------|------------------|---------------------|-----------------------------|-----------------------------|----------------|------------------------|------------------------|
| 40             | 10                       | 60               | 1600                | 1.57                        | 1.13                        | 686            | 56                     | 0.511                  |
| 40             | 30                       | 20               | 1600                | 1.27                        | 0.99                        | 1168           | 47                     | 0.674                  |
| 40             | 10                       | 60               | 800                 | 2.26                        | 1.98                        | 650            | 58                     | 0.739                  |
| 50             | 20                       | 40               | 1600                | 1.98                        | 1.7                         | 996            | 52                     | 0.226                  |
| 60             | 30                       | 60               | 1600                | 0.99                        | 1.27                        | 1230           | 64                     | 0.208                  |
| 50             | 20                       | 40               | 1200                | 1.41                        | 1.2                         | 1205           | 52                     | 0.596                  |
| 60             | 30                       | 20               | 800                 | 2.12                        | 2                           | 1030           | 57                     | 0.249                  |
| 40             | 10                       | 20               | 800                 | 2.19                        | 1.56                        | 710            | 42                     | 0.375                  |
| 60             | 20                       | 40               | 1200                | 1.27                        | 0.85                        | 930            | 65                     | 0.243                  |
| 50             | 20                       | 20               | 1200                | 1.34                        | 0.78                        | 1050           | 47                     | 0.29                   |
| 60             | 10                       | 60               | 800                 | 1.49                        | 1.34                        | 698            | 51                     | 0.182                  |
| 60             | 10                       | 20               | 800                 | 2.34                        | 0.92                        | 632            | 44                     | 0.229                  |
| 50             | 20                       | 1200             | 2.2                 | 1.56                        | 984            | 55                     | 0.276                  |
| 60             | 30                       | 20               | 1600                | 1.63                        | 2.55                        | 1082           | 52                     | 0.277                  |
| 40             | 30                       | 60               | 800                 | 1.99                        | 1.27                        | 1280           | 68                     | 1.192                  |
| 50             | 20                       | 1200             | 2                 | 1.34                        | 920            | 56                     | 0.574                  |
| 50             | 20                       | 1200             | 1.84                 | 1.06                        | 943            | 58                     | 0.365                  |
| 60             | 10                       | 60               | 1600                | 0.99                        | 1.1                         | 872            | 52                     | 0.125                  |
| 40             | 30                       | 20               | 800                 | 2.62                        | 1.98                        | 1098           | 54                     | 0.521                  |
| 50             | 20                       | 40               | 800                 | 2.9                         | 1.84                        | 1024           | 55                     | 0.314                  |
| 40             | 10                       | 20               | 1600                | 2.48                        | 2.83                        | 790            | 46                     | 0.349                  |
| 50             | 20                       | 1200             | 1.77                 | 1.41                        | 810            | 54                     | 0.4453                 |
| 50             | 20                       | 1200             | 1.91                 | 1.49                        | 850            | 48                     | 0.537                  |
| 30             | 40                       | 1200             | 1.7                 | 1.27                        | 1320           | 62                     | 0.215                  |
| 60             | 30                       | 60               | 800                 | 1.49                        | 1.2                         | 1298           | 67                     | 0.245                  |
| 40             | 20                       | 1200             | 2.26                 | 1.56                        | 904            | 63                     | 0.142                  |
| 50             | 20                       | 1200             | 2.48                 | 1.7                         | 1032           | 70                     | 0.304                  |
| 50             | 10                       | 40               | 1200                | 2.69                        | 2.34                        | 722            | 48                     | 0.637                  |
3.2. Experimental Data Analysis Using RSM

The experiments work were designed using the RSM method. These experiments were conducted using Minitab statistical program, “Ver.19” to choose the right models. Regressions equations were developed using experimental data in Table 6 and it is plotted to discover the effect of precipitation conditions (input parameters) on parameters output properties responses. Finally, the results are analyzed statistically using the ANOVA approach. Table 7 illustrates the multiple regression coefficients associated with three responses (CT, HB and Ra) parameters. As for response prediction, the response parameters can be used, which are reliable tools for developing mathematical models. The Figures 2 illustrate the differences between the measured results and predicted based on the developed models.

### Table 7: Regression coefficients for Ra, CT, HV, C.R and E.R.C.R model parameters.

| Term          | CT Model | HV Model | Ra Model | C.R  | Eroson C.R |
|---------------|----------|----------|----------|------|------------|
|               | Coef.    | T value  | P value  | Coef | T value  | P value  | Coef | T value  | P value  | Coef | T value  | P value  |
| Constant      | 56.38    | 49.59    | 0.00     | 986.2| 33.49    | 0.00     | 0.3755| 6.80     | 0.00     | 1.392| 9.16     | 0.00     |
| T             | 0.020    | 0.02     | 0.984    | -1.2 | -0.05    | 0.963    | -0.1788| -3.78    | 0.002    | -0.091| -0.70    | 0.495    |
| WC wt%        | 5.757    | 5.92     | 0.00     | 236.3| 9.38     | 0.00     | 0.0504| 1.07     | 0.303    | -0.061| -0.47    | 0.645    |
| t             | 7.035    | 7.23     | 0.00     | 38.5 | 1.53     | 0.147    | 0.0564| 1.19     | 0.251    | -0.170| -1.30    | 0.212    |
| r.p.m         | -1.368   | -1.41    | 0.180    | 29.1 | 1.16     | 0.266    | -0.0085| -0.18    | 0.860    | 0.058 | 0.45     | 0.660    |
| Tx T          | 5.84     | 2.45     | 0.027    | -86.5| -1.40    | 0.181    | -0.085 | -0.74    | 0.472    | -0.245| -0.77    | 0.454    |
| WC wt% x WC wt% | -3.16   | -1.33    | 0.204    | 17.5 | 0.28     | 0.780    | 0.148 | 1.28     | 0.219    | 0.355 | 1.12     | 0.282    |
| t x t         | 0.34     | 0.14     | 0.888    | 37.5 | 0.61     | 0.552    | 0.019 | 0.17     | 0.869    | -0.210| -0.66    | 0.520    |
| r.p.m x r.p.m | -4.66    | -1.96    | 0.069    | 6.5  | 0.11     | 0.918    | -0.008 | -0.07    | 0.948    | 0.320 | 1.01     | 0.330    |
| TxWC wt%      | 1.48     | 1.42     | 0.176    | -24.5| -0.91    | 0.377    | -0.0933| -1.85    | 0.085    | 0.209 | 1.50     | 0.154    |
| T x t         | -0.90    | -0.86    | 0.402    | 24.0 | 0.89     | 0.388    | -0.1276| -2.52    | 0.023    | -0.072| -0.52    | 0.613    |
| Tx r.p.m      | -0.48    | -0.46    | 0.653    | 7.0  | 0.26     | 0.798    | 0.0328| 0.65     | 0.526    | 0.108 | 0.78     | 0.440    |
| WC wt% x t    | 1.15     | 1.10     | 0.288    | 48.5 | 1.80     | 0.092    | 0.0527| 1.04     | 0.314    | -0.028| -0.20    | 0.843    |
| WC wt% | x r.p.m | -1.52 | -1.46 | 0.164 | -23.5 | -0.87 | 0.397 | -0.0022 | -0.04 | 0.966 | -0.083 | -0.60 | 0.559 | -0.067 | -0.55 | 0.588 |
|--------|--------|-------|-------|------|-------|------|-------|---------|-------|------|---------|-------|------|---------|-------|------|
| t x r.p.m | -0.15 | -0.14 | 0.889 | -12.5 | -0.46 | 0.649 | -0.0605 | -1.20 | 0.250 | -0.164 | -1.18 | 0.256 | 0.014 | 0.12 | 0.909 |
| R²     | 94.36% | 90.85% | 95.78% | 96.47% | 96.12% |

A. Normal Probability Plot
B. Normal Probability Plot
C. Normal Probability Plot
D. Normal Probability Plot
E. Normal Probability Plot
The equations (1, 2, 3, 4 & 5) were represented the final response models after backward elimination:

\[
C_T = 56.38 + 0.02 T + 5.757 WC \text{ wt\%} + 7.035 t - 1.368 \text{ r.p.m} + 5.84 T^2 - 3.16 (WC\text{ wt\%})^2 + 0.34 t^2 - 4.66(\text{r.p.m})^2 + 1.48 T*WC \text{ wt\%} - 0.90 T*t - 0.48 T*r.p.m + 1.15 WC\text{ wt\%}*t - 1.52 WC \text{ wt\%}*r.p.m - 0.15 t*r.p.m \ 
\]

\[
R_a = 0.375 - 0.178 T + 0.05 WC \text{ wt\%} + 0.056 t - 0.008 \text{ r.p.m} - 0.085 T^2 + 0.148 (WC\text{ wt\%})^2 + 0.019 t^2 - 0.008 (\text{r.p.m})^2 - 0.093 T*WC \text{ wt\%} - 0.127 T*t + 0.032 T*r.p.m + 0.052 WC \text{ wt\%}*t - 0.002 WC \text{ wt\%}*r.p.m - 0.06 t*r.p.m \ 
\]

\[
H.V = 986.2 - 1.2 T + 236.3 WC \text{ wt\%} + 38.5 t + 29.1 \text{ r.p.m} - 86.5 T^2 + 17.5 (WC\text{ wt\%})^2 + 37.5 t^2 + 6.5 (\text{r.p.m})^2 - 24.5 T*WC\text{ wt\%} + 24 T*t + 7 T*r.p.m + 48.5 WC\text{ wt\%}*t - 12.5 T^*r.p.m \ 
\]

\[
C.R *10^{-5} = 1.392 - 0.091 T - 0.061 WC \text{ wt\%} - 0.17 t + 0.058 \text{ r.p.m} - 0.245 T^2 + 0.355 (WC\text{ wt\%})^2 - 0.210 t + 0.32 (\text{r.p.m})^2 + 0.209 T*WC \text{ wt\%} - 0.072 T*t + 0.108 T*r.p.m - 0.028 WC \text{ wt\%}*t - 0.083 WC \text{ wt\%}*r.p.m - 0.164 t*r.p.m \ 
\]

\[
\text{Erosion C.R *10^{-5} = 1.985 - 0.241 T - 0.121 WC \text{ wt\%} - 0.151 t - 0.268 \text{ r.p.m} - 0.35 T^2 + 0.08 (WC\text{ wt\%})^2 - 0.2 t^2 + 0.325 (\text{r.p.m})^2 + 0.023 T*WC \text{ wt\%} - 0.128 T*t - 0.007 T*r.p.m + 0.075 WC \text{ wt\%}*t - 0.067 WC \text{ wt\%}*r.p.m + 0.014 t*r.p.m} \ 
\]

3.3. Parametric Analysis of the outputs Response

The effect of the input parameters [temperature (T), wc wt\%, deposition time (t) and stirring velocity (r.p.m)] on the response of the output parameters [coating thickness (C.T), surface roughness (R.a), Vickers Hardness (H.V), corrosion rate (C.R) and erosion corrosion rate (E.C.R)] were found out using the parametric analyses process and get relationships between the input and output parameters from these plots.

3.3.1. Coating thickness Analysis

Figure 3 shows the main influence plot of four studied input parameters on the thickness of coating (C.T).
Figure 3: Main Effect Plot for Thickness Coating.

We note that when the temperatures increases, we notice the thickness of the coating layer increasing, and this indicates an increase in the activity of the movement of ions, thus a higher precipitation rate and uniformity in the thickness of the coating layer. For temperatures above 60°C, desorption forces become prominent in the initial stages resulting in reduced surface concentration of coating ions thereby reducing the coated layer. The Coating thickness (C.T) increase from (42 μm to 73 μm) with increase factor deposition t (t) from 20 min to 60 min. The reason for increasing the coating thickness with increasing deposition t is to give sufficient t for precipitation of the largest possible The number of wc particles on the Chrome layer also has a large size of wc particles, and it takes t to gravitate and stick to the Chrome layer. We note that the thickness of the coating increases with increasing sedimentation. The layers with the lowest thickness are porous, while the higher layers contain cracks, and their adhesion strength is weak [15]

Tungsten Carbide particles are large in size and they leave pores between the WC particles when deposited on the Chrome layer, but when more t is available, this t it settles more particles on the Chrome layer. These particles precipitate between the precipitated particles and fill all the pores that form by the initial deposition also it reduces the porosity and roughness of the previous deposition and increase coating thickness.

The main effect of parameter stirring velocity (r.p.m) shows reverse tendency. Increases in this parameter result in a decrease in the coating thickness (C.T) . Around the same time, WC particles move inside the electrolyte to the cathode under an electrical field and electrophoresis. The electronic affinity of WC particles to 316L SS is however low, so, due to the activity of high activity chromium ions that attract WC particles and by the movement of the mechanical stirr, the sedimentation of tungsten carbide particles within the hard chromium layer deposited on the surface of austenitic steel 316 L [16]

When the medium stirring velocity is given, the best thickness of the coating layer is given, and this indicates the uniformity of the thickness of the coating layer at a deposition speed of 1200 to allow the appropriate t and speed to precipitate the tungsten carbide particles between the deposited hard chromium layers and ensure that they are not dispersed.

3.3.2. Parametric Analysis of Roughness surface

In general, this work had a smooth surface and little roughness in the electroplating process, the composite coating on the 316L SS substratum is smooth and compact with good adhesion.
Figure (4) displays the main impact plot for the surface roughness (Ra) of four studied input parameters.

![Main Effect Plot for Means of Ra.](image)

The surface roughness (Ra) is decrease with an increase of factor temperature. With increasing temperature, the sedimentation process becomes more uniform and this is due to the increased effectiveness of the movement of chromium ions in the solution and thus attracts with them WC particles thus reducing the surface roughness of sample. This corresponds to the research the temperature was rough under the composite coating. [17]

The analysis shows the largest effects of WC concentration (wt.%) on the surface roughness. It shown increase of the surface roughness with the increase of WC concentration (wt.%) from (10 to 30 wt.%). But a little roughness for all results, and this is due to the fact that the chrome plating process is characterized by high resistance and a long coefficient of friction.

The main effect of parameter deposition time (t) on the surface roughness. It shown increases of the surface roughness from (0.59 to 1.68 μm) when increase deposition time from (20 to 40 min) because the size of tungsten carbide is large. At this time, its distribution over the surface of the sample is irregular and with sufficient time given to the precipitation process at (60 min) the surface roughness is as little as possible and this indicates the distribution of tungsten carbide particles in a well-deposited hard chromium layer.

In general, the roughness of the surface is low in this type of coating, whether it is at low stirring velocity or high stirring velocity up to (0.6 μm) Most results, but the roughness was less and a slight percentage at the speed of the mixer 1200 This is due to the regularity of the distribution of tungsten carbide particles between the hard chromium ions deposited because the carbide particles are characterized by being Heavy in weight and the movement of the mixer helps it easily to settle on the alloy surface.

### 3.3.3. Parametric Analysis of Vickers Hardness (HV)

Hardness is some of the most important properties to be studied due to its critical impact on the properties and the adhesion of the surface deduct coating sheet. The Cr – WC layer of coating shows higher hardness than both the stainless steel substratum and the pure Cr plate. Figure 5 shows the main effect plot for the Vickers hardness of each input parameter (HV)
Figure 5: Main Effect Plot for Means of HV.

The temperature of the coating solution plays an important role in raising the hardness, and this is evident through the salinity values of the samples covered with the composite coating, where a temperature rise from 40 °C to 60 °C increases the ion movement and thus increases the sedimentation efficiency. The temperature has a big role in the coating process, and this is clear through the results, as the best temperature condition ranges between 50 to 60 and this corresponds to the source.[18]

Increasing deposition from (20 min to 60 min) leads to increase the Micro Vickers hardness values. Because the coating layer contains WC particles is responsible for the high hardness of the coating layer, in order to give sufficient deposition time for the tungsten carbide particles to settle in the hard chromium layer which in turn becomes more coherent and thicker by increasing the Deposition time.

The stirring speed of the electrolyte plays an important role in any electrodeposition cycle as it facilitates the transfer of metallic ions to the electrode, increasing the deposition rate. The analysis shows increase in the micro hardness values with increasing stirring velocity (r.p.m) from (800 to 1200 and 1600 r.p.m). With increasing velocity that lead to the movement of the coating solution, this increase has the ability of ions to attract tungsten carbide particles and deposition on the hard chromium layer that contains the tungsten carbide particles that have a very high hardness. Therefore we note the increase of hardness value. Hard chromium containing tungsten carbide particles compared with hard chromium hardness without tungsten carbide particles.

Finally, the plot analysis obvious the main effect of a factor WC (wt%) that HV increase with increase in WC (wt%). The higher the amount of tungsten carbide causes the higher the hardness of the composite coating layer. This is due to the fact that the tungsten carbide particles will have very high hardness. Durability of composite coatings is determined by the amount of hard particles added, which increases the hardness of composite coatings.

The most important factors regarding the hardness of the composite coatings are WC concentration (wt.%), deposition time (t), temperature after then stirring velocity (r.p.m).

3.3.4 Parametric Analysis of Corrosion Rate

Specimens of austenitic stainless steel 316L and austenitic stainless steel 316L coated (Cr+WC) were immersed in sulfuric acid at room temperature with concentrations (98 percent)
for a period of time Over a month and then the weight loss corrosion rate. Figure (6) shows the main effect plot of four measured input parameters at the corrosion rate (C.R).

![Figure 6: Main Effect Plot for Means of C.R.](image)

Hard chrome plating is commonly used for ceramic materials for applications that require severe stress, shocks or high resistance to corrosion.

The coating solution temperature plays an important role in the the resistance to corrosion This is evident from the above illustration, and this is evident from the salinity values of the samples coated with the composite coating, Where temperature increase from 40 °C to 60 °C gives an increase in the movement of ions and thus increases the sedimentation efficiency. The temperature gives a high efficiency to the coating process and hence a high corrosion resistance, and this is clear through the results, as the best temperature condition ranges between 50 to 60 and this corresponds to the source.

Increasing deposition t from (20 min to 60 min) leads to decrease the corrosion resistance values in sulfuric acid. The sufficient time for precipitation gives more uniformity to the coating layer for Chromium ion deposition on the surface of the sample, as well as filling the pores between the precipitated tungsten carbide particles and hence a homogeneous compound coating resistant to corrosion in the concentrated sulfuric acid solution 98%.

Because the coating layer contains WC particles is responsible for the high hardness of the coating layer We notice an increase in tungsten carbide concentration which results in increased cohesion of the coating layer against general corrosion, and thus reducing the rate of corrosion. This indicates that there are no pores in the coating layer that give adequate protection to the stainless steel 316L in the concentrated sulfuric acid solution and this is consistent with the research.

The analysis shows decrease in the corrosion rate values with increasing stirring velocity (r.p.m). With increasing velocity that lead to the movement of the coating solution, this increase has the ability of ions to attract tungsten carbide particles and deposition on the hard chromium layer that contains the tungsten carbide particles that have a very high hardness therefor we note the increase of corrosion rate value.

### 3.3.5 Parametric Analysis of Erosion Corrosion Rate

The chromium in the Cr – WC film also plays an important role in the film's corrosion resistance, because higher Cr content on the surface is beneficial to a passive film formation. The introduction of Cr in Fe – Cr alloys significantly affects the active to passive transition of Fe And greatly stabilizes the passive state of the alloys, and both the passivation potential and
current decreases with increasing chromium content. It is also shown by the erosion corrosion rate of austenitic steel samples 316L coated with hard chromium and tungsten carbide. The main impact plot of each input parameter on the erosion corrosion rate (E.C.R) is shown by Figure (7).

![Figure 7: Main Effect Plot for Means of Erosion C.R.](image)

When the coating solution temperature increases, the erosion corrosion rate decreases, which means that the temperature plays an important role in the coating layer's homogeneity and adhesion. When the temperature rises from 50 °C to 60 °C, the rate of corrosion is erosion is reduced since the coating layer is non-porous and has high adhesion and due to the temperature that accelerated the sedimentation process and its uniformity.

Increasing deposition time from (20 min to 60 min) leads to decrease the erosion corrosion resistance values in sulfuric acid. The sufficient time for precipitation gives more uniformity to the coating layer for precipitation of chromium ions on sample surface, as well as filling the pores between the precipitated tungsten carbide particles and hence a homogeneous compound coating resistant to corrosion in the concentrated sulfuric acid solution 98%.

The lower rate of corrosion with the increase in the percentage of tungsten carbide in the coating solution is due to the reason the strength and resistance of the coating layer to erosion corrosion in concentrated sulfuric acid. Because of the lack of pores and cracks within it. This is due to the strong bonding strength between tungsten carbide which gives the alloy higher resistance.

Finally, the most influential factors on the corrosion rate of the composite coatings (Cr-WC), temperature (T), deposition time (t), WC concentration (wt.%) after then stirring velocity (r.p.m).

### 3.4 Optimization of multiple response using the Desirability Function:

Optimum manufacturing parameters have been determined using desirability function to electroplating of stainless steel 316L for best of output parameters [C.T (μm), Ra (μm), HV, C.R , E.C.R]. In this study, it is desirable to has lower C.R , E.C.R and Ra and the higher HV and target C.T. Desirable function is represented in the mathematical formula as follows:-

Min: F1 (x) = C.T (μm)
Min: F2 (x) = Ra (μm)
Max: $F_3(x) = HV$
Min: $F_4(x) = C.R \times 10^{-5}$
Min: $F_5(x) = E.C.R \times 10^{-5}$
Matrix as :-
\[
\begin{align*}
40 \leq x_1 \leq 60 \\
10 \leq x_2 \leq 30 \\
20 \leq x_3 \leq 60 \\
800 \leq x_4 \leq 1600
\end{align*}
\]

Where, $x_1$, $x_2$, $x_3$ and $x_4$ reflect electroplating process input parameters: temperature (T), tungsten carbide concentration (WC wt percent), deposition t (t) and velocity of stirring (r.p.m). The function of desirability using objective $D(X)$ functions. These functions convert a predicted value into free-scale values ($d_i$). The desirability values are different from (0-1), and a value close to 1 is desired. If answer (Y) is to target or target, $d_i = 1$. External response The satisfactory regions are $d_i = 0$, which summarizes the key parameters set to find the optimal global setting in this study (8). Table (8): Summarize the set of key parameters required to find the optimum global environment.

### Table (8): Key Parameters

| Response          | Goal | Target | Upper | Lower |
|-------------------|------|--------|-------|-------|
| HV                | Max. | 1320   | 1320  | 632   |
| Ra ($\mu m$)      | Min. | 0.13   | 1.192 | 0.13  |
| CT ($\mu m$)      | Target. | 50  | 73   | 42    |
| $C.R \times 10^{-5}$ ($gm/cm^2*day$) | Min | 0.78 | 2.830 | 0.78 |
| $E.C.R \times 10^{-5}$ ($gm/cm^2*day$) | Min | 0.99 | 2.900 | 0.99 |

The table (9) illustrates multiple response prediction for maximum of Micro Vickers Hardness (HV), minimum each of surface roughness (Ra $\mu m$) and coating thickness (CT $\mu m$), corrosion rate (C.R) and erosion corrosion rate (E.C.R)

### Table (9): Multiple Response Prediction

| Variables          | Temperature | Deposition time (min) | Stirring velocity r.p.m | SiC concentration (wt.%)
|--------------------|-------------|-----------------------|-------------------------|--------------------------|
| Setting            | 57.7467     | 60                    | 1600                    | 22.1997                  |

The individual desirability of micro-hardness (HV), surface roughness (Ra $\mu m$), coating thickness (CT $\mu m$), corrosion rate (C.R), erosion corrosion rate (E.C.R) are 0.69574, 1.0000, 0.50780, 0.86705 and 0.899980 respectively as shown in figure (4.22), the composites desirability is 0.7728, responses inside the satisfactory regions.

#### 3.5. Confirmation Test :-

Confirmation Testing is a very important test that is used for verify the optimal conditions create from the surface response methodology As indicated in table (10). The percentage error of all response variables between the expected value and the experimental value is determined by formula (7) [19].

\[
\text{Percentage error} \% = \frac{\text{Experimental value} - \text{Predicted value}}{\text{Experimental value}} \times 100 \quad \ldots \ldots \quad (7)
\]
Table 10: Confirmation results and percentage errors

| Response | HV      | Ra    | C.T   | C.R*10^-5 | E. C.R*10^-5 |
|----------|---------|-------|-------|------------|--------------|
| Exp.     | Pred.   | Exp.  | Pred. | Exp.       | Pred.        |
| 1117     | 1110.6678 | 0.132 | 0.1250 | 59         | 61.3206      |
|          |         |       |       | 1.10       | 1.0525       |
|          |         |       |       | 1.22       | 1.1814       |

Error %  
0.56      5.30    3.93    4.31    3.16

The table (10) shows the errors percentages is within the ranges of (0.56 to 5.30) % Which were extracted by comparison of predicted models with laboratory experimental models of the optimal sample. Minitab program was used to develop predicted models can be successfully utilized to predict the optimal conditions for coating a laboratory model according to the optimum conditions (temperature, wc wt.%, deposition t (t), stirring velocity (r.p.m)) for the response of the output parameters (Micro Vickers Hardness(HV), surface roughness (Ra), coating thickness (C.T),corrosion rate,erosion corrosion rate values within the ranges of the conducted experiments

3.6. Scanning Electron Microscopy Analysis of optimum Sample:-

Scanning Electron Microscope (SEM) examined surface morphology and micro particles distribution of Tungsten Carbidecarbide particles and hard Chrome on austenitic stainless steel 316L substrate coated of optimum sample. Figure (8) show morphology for Surface coating at different magnification, Cr-WC coating layers. While figures (9), represents EDS analysis results to illustrate the chemical composition for coating layers Cr-WC and Amount of elements for EDS analysis for coating layers Cr-WC.

Figure 8: SEM-Image Morphology for surface of the optimum sample coating (Cr-WC).

Figure 9: EDS analysis of optimum sample (57.7467 T,22.1997wt.% wc, 1600 r.p.m, 60 min).
4. CONCLUSIONS

According to these results of this study, the following can be concluded:-

1. The corrosion resistance of chromium-tungsten carbide can be related to the formation of a passive layer (protective layer) from WC towards a corrosive environment. A 22 wt.% of WC in the deposit layer (chromium matrix) is enough to form a good protective and passive layer.

2. Chromium-tungsten carbide alloy films with good adhesive strength and uniform were deposited on 316L stainless steel by electrodeposition.

3. The optimization using RSM method helps to reduce the number of attempts needed to reach the optimum solution with minimum mean absolute relative errors of the predict values as compared to experimental values.

4. The optimal settings of process parameters that can be used in coating process of responses surfaces methodology analysis results are: WC concentration (22.1997wt.% WC), deposition time (60min), temperature (57.7467) and stirring velocity (1600 r.p.m) to get maximum of micro hardness (1117 Hv), low surface roughness (0.132 μm) and low coating thickness (59μm), low corrosion rate (1.10 *10^{-5}) and low erosion corrosion rate (1.22 *10^{-5}).

5. Confirmation test had verified that, multiples regressions analysis gives a mathematical models for predictions of the responses parameters with errors percentages within the ranges of (0.56–5.30) %. So, the developed predicted equations can be used successfully to predict surface roughness (Ra), coating thickness (C.T), Micro Vickers Hardness (HV), corrosion rate (C.R) and erosion corrosion rate (E.C.R) values for any collections of the deposition time, stirring velocity, WC wt.% , temperature within the ranges of the conducted experiments.

REFERENCES

[1] Stack, M. M., and Abdelrahman, S. M. (2011) “A CFD Model of Particle Concentration Effects on Erosion-Corrosion of Fe in Aqueous Conditions”, Wear, Vol 273, No.1, pp.38-42.

[2] Adler, W. F. (Ed.). (1979) “Erosion: Prevention and Useful Application”, ASTM International, Printed in Blatimore.MD.

[3] Pracht, G., & Perschnick, N. (2016)," A material challenge–Pumps in sulphuric acid application", Procedia Engineering, 138, 421-426.

[4] Levy, A. V. (1995) “Erosion and Erosion-Corrosion of Metals”, Corrosion, Vol. 53, No.11, pp.872-883.

[5] Haydar Hasan Jabir Jamaluddin (1998) “Improving the erosion resistance of alpha-alpha alloys by few additions of some elements (Ge, Te, Al )", Master Thesis, University of Technology.

[6] Craig, B. D., Lane, R. A., & Rose, D. H. (2006)," Corrosion prevention and control: A program management guide for selecting materials", Advanced Materials, Manufacturing, and Testing Information Analysis Center (AMMTIAC).

[7] Tang, J., & Zuo, Y. (2008)," Study on corrosion resistance of palladium films on 316L stainless steel by electroplating and electroless plating", Corrosion science, 50(10), 2873-2878.

[8] Spyrellis, N., Pavlatou, E. A., Spanou, S., & Zoikis-Karathanasis, A. (2009),"Nickel and nickel-phosphorous matrix composite electrocoatings",Transactions of Nonferrous Metals society of china, 19(4), 800-804.
[9] Latif, S. (2010), "Carbide Reinforced Metal-Matrix Composite Coatings by Carburizing of Electrodeposited Amorphous and Nanocrystalline Alloys", (Doctoral dissertation, Pakistan Institute of Engineering and Applied Sciences Islamabad, Pakistan).

[10] V. Sridevi (2013) “Biosorption of methylene blue by spent biomass of aspergillus terreus using response surface methodology”, International Journal of Pharm and Bio Sciences, Issue 4, Vol. 3, pp. 317-325.

[11] Demirel M, Kayan B. (2012) “Application of response surface methodology and central composite design for the optimization of textile dye degradation by wet air oxidation”. International Journal of Industrial Chemistry.;3(24):1-10.

[12] Salam KK, Arinkoola AO, Oke EO, Adeleye JO. (2014) “Optimization of Operating Parameters Using Response Surface Methodology for Paraffin-Wax Deposition in Pipeline”. Petroleum & Coal.;56 (1):19-28.

[13] Q. Kong, G.Q., Q. H. Chen, F. Chen, (2004) “Optimization of medium composition for cultivating clostridium butyricum with response surface methodology”, J. Food Sci. 69: M163-M168.

[14] https://www.jindalstainless.com/pdfs/brochures/austenitic_stainless_steel.pdf.

[15] Muhammad Abdul-Mahdi, (2000) "Electroplating for Steel Protection", Master Thesis, College of Engineering, University of Babylon.

[16] Zhang, Z., Tang, J., Wang, Y., Wang, H., Normand, B., & Zuo, Y. (2018), "Electrodeposition of a Pd-Ni/TiO2 composite coating on 316L SS and its corrosion behavior in hot sulfuric acid solution", Coatings, 8(5), 182.

[17] Rezende, T. G., Cesar, D. V., Lago, D. C. B., & Senna, L. F. (2016). "A review of corrosion resistance nanocomposite coatings", Electrodeposition of Composite Materials. Kijeka: InTech, 147-185.

[18] Khani, H., & Brennecke, J. F. (2019), "Hard chromium composite electroplating on high-strength stainless steel from a Cr (III)-ionic liquid solution", Electrochemistry Communications, 107, 106537.

[19] Muqdad J. Dakhil (2016) „Optimization of Electro Discharge Machining of Porous Shape Memory Alloys”. MS.C thesis, college of Materials Engineering, university of Babylon, Iraq.