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Aerospace Research Letters (ASREL)
DOI: 10.56753/ASREL.2022.2.3

Cilt: 1 Sayı:2 Yıl: 2022 Araştırma Makelesi / Research Article E-ISSN: 2980-0064

Optimization of Ni-Cr Alloy Coatings Produced Using the Electrodeposition Method

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Article Info

Article History
Received: 23/11/2022
Accepted: 19/12/2022
Published: 31/12/2022

Keywords:
Electrodeposition,
Corrosion,
Taguchi,
ANOVA

ABSTRACT

Electrodeposition method is an important technique for obtaining metal, alloy or composite coatings. Ni-Cr alloy coatings obtained by electrodeposition method are preferred especially in corrosive environments. This study is about obtaining Ni-Cr alloy coating by electrodeposition method and determining optimization parameters using Taguchi method, which is one of the experimental design methods. Taguchi method has been used to determine the experimental parameters due to the excess of parameters in the electrodeposition method. For this purpose, Taguchi L9 orthogonal array was chosen. As a parameter; three different levels of pH, temperature, current density and stirring rate were used. Since Ni-Cr coatings are preferred in corrosive environments, the effects of the factors affecting the coating on the corrosion results obtained by the tafel extrapolation method were investigated by signal-to-noise ratio, average effects and variance analysis. Corrosion rates (mm / year) of the curves obtained using the Tafel extrapolation method were determined. Among the experiments, the best result was given by pH 2.5, temperature 40 °C, current density 200 mA/cm2 and the sample without stirring. In addition, it has been confirmed by scientific and statistical studies that current density has dominant effects on Ni-Cr corrosion results.

Elektrodepolama Yöntemi Kullanılarak Üretilen Ni-Cr Alaşım Kaplamaların Optimizasyonu

Makale Bilgileri

Makale Geçmişi
Geliş: 23/11/2022
Kabul: 19/12/2022
Yayın: 31/12/2022

Anahtar Kelimeler:
Elektrodepolama
Korozyon,
Taguchi,
ANOVA

ÖZ

Elektrodepolama yöntemi, metal, alaşım veya kompozit kaplamaların elde edilmesinde önemli bir tekniktir. Elektrodepolama yöntemi ile elde edilen Ni-Cr alaşımlı kaplamalar özellikle korozif ortamlarda tercih edilmektedir. Bu çalışma, elektrodepolama yöntemi ile Ni-Cr alaşım kaplama elde edilmesi ve deneysel tasarım yöntemlerinden biri olan Taguchi yöntemi kullanılarak optimizasyon parametrelerinin belirlenmesi ile ilgilidir. Elektrodepolama yönteminde parametre fazlalığı nedeniyle deneysel parametreleri belirlemek için bu yöntem tercih edilmiştir. Bu amaçla Taguchi L9 ortogonal dizisi seçilmiştir. Parametre olarak; pH, sıcaklık, akım yoğunluğu ve karıştırma hızının üç farklı seviyesi kullanılmıştır. Korozif ortamlarda Ni-Cr kaplamalar tercih edildiğinden, kaplamayı etkileyen faktörlerin tafel ekstrapolasyon yöntemi ile elde edilen korozyon sonuçlarına etkisi sinyal-gürültü oranı, ortalama etkiler ve varyans analizi ile incelenmiştir. Tafel ekstrapolasyon yöntemi kullanılarak elde edilen eğrilerin korozyon oranları (mm/yıl) belirlenmiştir. Deneyler arasında en iyi sonucu pH 2.5, sıcaklık 40 °C, akım yoğunluğu 200 mA/cm2 ve numunenin karıştırılmaması vermiştir. Ayrıca akım yoğunluğunun Ni-Cr korozyon sonuçları üzerinde baskın etkilere sahip olduğu bilimsel ve istatistiksel çalışmalarla doğrulanmıştır.

INTRODUCTION

Hard chrome coatings reduce the surface roughness and wear of the materials while increasing their corrosion resistance. Because of these superior corrosion and wear properties, hard chrome coatings are used quite frequently in decorative, aerospace, automotive and many other industries. However, due to the toxic nature of +6 valent Cr ions and the increased sensitivity of people to harmful wastes, their use is either banned or restricted in many countries. Due to these limitations, studies in recent years have focused on trivalent Cr coatings, which are environmentally friendly, convenient and low-priced. However, it is known that trivalent chrome coatings alone do not show good corrosion and wear properties like hard chrome coatings. The properties of these coatings can be improved by the addition of metals from the iron family (Ni, Co, etc.) (He et al., 2013; Sheibani Aghdam, Allahkaram, & Mahdavi, 2015). Chromium with nickel; They are preferred for their good corrosion and high oxidation resistance and high temperature resistance (Demir, 2021; Etminanfar & Heydarzadeh Sohi, 2012; Peng, Zhou, Wang, & Zhang, 2004; Sheibani Aghdam et al., 2015; Zhang, Liu, Bai, & Liu, 2015).

It is one of the most widely used methods to protect the materials from external factors by coating them and to gain the desired properties. One of the coating methods is the coating of materials by electrodeposition method. It is relatively simple among various coating methods, allows coating materials with mixed geometries, does not require high cost vacuum systems, many test parameters can be controlled, offers coating thickness from a few nanometers to microns, has the possibility to store at room temperature and low installation cost electrodeposition method stands out compared to other coating methods. The coatings obtained by this method can be used for a wide variety of purposes such as protection, decorative, corrosion resistance, magnetic property, heat resistance, abrasion resistance, lubricity, electrical permeability and jewelry coatings (Demir, Kanca, & Karahan, 2020; Dini, 1993; Gamburg & Zangari, 2011).

The electrodeposition process depends on experimental parameters such as a strong bath composition, pH, deposition potential or current, temperature, stirring speed, time, etc. (Firouzi-Nerbin, Nasirpouri, & Moslehifard, 2020; Kunyarong & Fakpan, 2018). Considering the multitude of parameters in the electrodeposition method, the application of experimental design methods is an extremely efficient approach in order to both perform the experiments in the most efficient way by considering economic conditions and time constraints, and to interpret the results correctly (to be able to detect the relationship between controllable or uncontrollable factors and their outputs and to optimize them) (Demir, 2015; Gupta, Pandey, Garg, Khanna, & Batra, 2014). With the experimental design method, the necessity of doing experiments repeatedly is eliminated. However, it is seen that experimental design methods are not used much in the studies encountered.

Jeyaraj et al. carried out a study for the deposition of micron-sized chromium particles in a nickel matrix with electrodeposition principles. The bathroom content detection experiments were designed with the Taguchi experimental design approach, with the L27 orthogonal array. They used 5 different variables and 3 different levels in their designs. These were determined as current density, pH, temperature, bath particle density and stirring rate. The effects of the aforementioned electrodeposition parameters on the coating hardness were investigated with mean effects, signal-to-noise ratio and analysis of variance (Jeyaraj, Arulshri, Ramesh, & Muthukumaran, 2018).

Güler and Karakaya conducted a study on the deposition of Ni and MoS2 particles together. In their studies, they used L8 orthogonal arrays according to the Taguchi experimental method. As variable parameters; two different levels of MoS2 particle content, temperature and coating were used. As a result, they examined the effects of these variables on the material internal stress (Güler, 2017).

Hou and Chen used statistical Taguchi method to produce Ni-W/Al2O3 composite coatings by electrodeposition method. In their study, they produced the coatings by forming L9 orthogonal arrays from the three parameters of current density, pulse frequency, Al2O3 concentration and duty cycle. As a result; They found that the Taguchi method is useful in the selection of parameters that will determine the chemical, physical and mechanical properties of coatings (Hou & Chen, 2011).

As can be seen from the studies mentioned above, the use of experimental design methods in the optimization of the electrodeposition method is quite limited. In this study, the effects of production parameters on the corrosion behavior of Ni-Cr alloys were investigated with statistical and scientific approaches. The parameters affecting the coating are current density, pH, temperature and stirring rate, which were determined with the support of the literature. The effect of three different levels of these parameters on coating corrosion resistance was investigated.

METHOD

The bath components used in the study are given in Table 1. Watt type bath containing 200 mL of electrolyte was used for electrodeposition and 15 x 15 x 5 mm dimensions were used as a substrate.

Table 1. *Bath composition*

Electrolyte composition	Amount	Function
$NiSO_4.7H_2O$	50 g/L	Source of Ni^{2+}
$NiCl_2.6H_2O$	45 g/L	Source of Ni^{2+}
$CrCl_3$. $6H_2O$	75 g/L	Source of Cr^{3+}
H_3BO_3	50 g/L	Buffer agent
$C_6H_8O_7$	70 g/L	Buffer agent
$Na_3C_6H_5O_7.2H_2O$	105 g/L	Complexing agent
$NaC_{12}H_{25}SO_4$ (SDS)	0,2 g/L	Wetting agent

While the substrate is not cleaned well, the adhesion force between the coating and the substrate will be weak and will adversely affect the deposition of the coating. In addition, foreign materials such as unwanted rust, dirt and oil on the surface will adversely affect the bath composition. In cleaning the substrate material, it was first sanded with three different sandpapers (600-1200-2400) to clean it mechanically. After sanding, the substrate was cleaned with acetone, and the acetone was then washed away with distilled water. Then an alkaline solution, methanol, was used to clean it, and distilled water was used to rinse it. It was then cleaned with distilled water, prepared for deposition, and etched for 20 seconds in a 20% concentrated HCl acid solution.

Prior to the electrodeposition procedure, all baths were stirred using an ultrasonic stirrer of the Hielscher UP 200 S brand for 5 minutes at 0.5 cycle value and 50% amplitude values. A continuous current of 200 mA/cm2 was used to deposit the coatings on the surfaces. For electrodeposition, a Watt type bath with 200 mL of electrolyte was employed. H2SO4 or NaOH was added to the bath to bring the pH down to 2.5. The electrolyte temperature was maintained at 40±1 °C throughout the procedure. Through the use of a CHI 608 model device and the cyclic voltammetry technique (CV), electrochemical characteristics of solutions with additional sugar were studied. At a rate of 10 mV/s, scanning was done in the potential range of 1.5 V to -1.5 V.

Using a Thermo Scientific Apreo S SEM with Ultra Dry EDS and Quasor II EBSD detectors working at 30 kV acceleration voltage, metallographic examinations were performed on the sample surfaces. By performing X-ray diffraction (XRD) analyses in a computer-controlled RIGAKU SmartLab with Cu Ka radiation (= 0.154 nm) and 2 angles ranging from 5° to 90°, the phases produced on the AISI 1040 surface were identified.

Using the CHI 608E analyzer test apparatus, electrochemical corrosion tests of the coatings were performed in a 3.5 wt% NaCl solution. Using a three-electrode coupling device running at room temperature, the Tafel for the samples was obtained [15]. The corrosion cell included a working electrode that was mounted on the test sample, a counter electrode, a reference electrode, and a holder. The open circuit potential range of -250 mV to +250 mV was used for the potentiodynamic corrosion experiments, and a potential scanning rate of 0.1 mV/s was used. The results of each corrosion test were averaged after being run three times.

The electrodeposition conditions were changed by keeping the bath components constant. As a result of the studies conducted in the literature, it has been determined that four different factors play an active role in the results of the electrodeposition. Three different levels of these four factors were included in the experimental parameters to be investigated. Electrodeposition conditions and bath components were determined in accordance with the literature (Table 2, Table 3) (Bahrami Mousavi, Baghery, Peikari, & Rashed, 2012).

Table 2. *Factors and levels affecting the coating*

Experimental Factors	Units	Levels		
		I	II	III
pH	-	2,5	3,5	4,5
Heat	°C	30	40	50
Current Density	mA/cm^2	100	200	250
Stirring rate	rpm	0	100	300

If these three-level, four-parameter coatings were to be made with a full factorial experimental design, it would have to be done with 34=81 experiments. In order to recognize and verify the effects of electrodeposition parameters on the corrosion of Ni-Cr alloy coating, the Taguchi approach proposed the L9 sequence. The factor levels of the proposed L9 series are given in Table 3.

Table 3. *Factor levels*

Name	Parameters			
	pH	Heat	Current Density	Stirring Rate
T1	I	I	I	I
T2	I	II	II	II
T3	I	III	III	III
T4	II	I	II	III
T5	II	II	III	I
T6	II	III	I	II
T7	III	I	III	II
T8	III	II	I	III
T9	III	III	II	I

FINDINGS / RESULTS

The potential values of nickel and chromium sources added to the solution for the Ni-Cr bath were determined by the Cyclic Voltammetry (CV) method. First of all, the CV graph of the nickel sulfate bath added to the bath is given separately because it is different from the others. CV studies were carried out by adding only boric acid, citric acid, sodium citrate dehydrate and nickel sulfate to the deposition bath. The study was carried out at a scanning rate of 10 mV/s and a range of 1.6 V to -1.6 V. The CV graph obtained is given in

Figure 1. A sudden increase in current around -0.9 V can be observed in the figure. This indicates that nickel has started to be deposited. In the range of -0.9 V to 1.3 V, the current remained constant at 0 mA and no reduction reaction took place in this range. With the scanning direction turning from the cathodic to the anodic side, a dissolution peak around -0.4 V was detected. This indicates the dissolution range of the stored Ni metal.

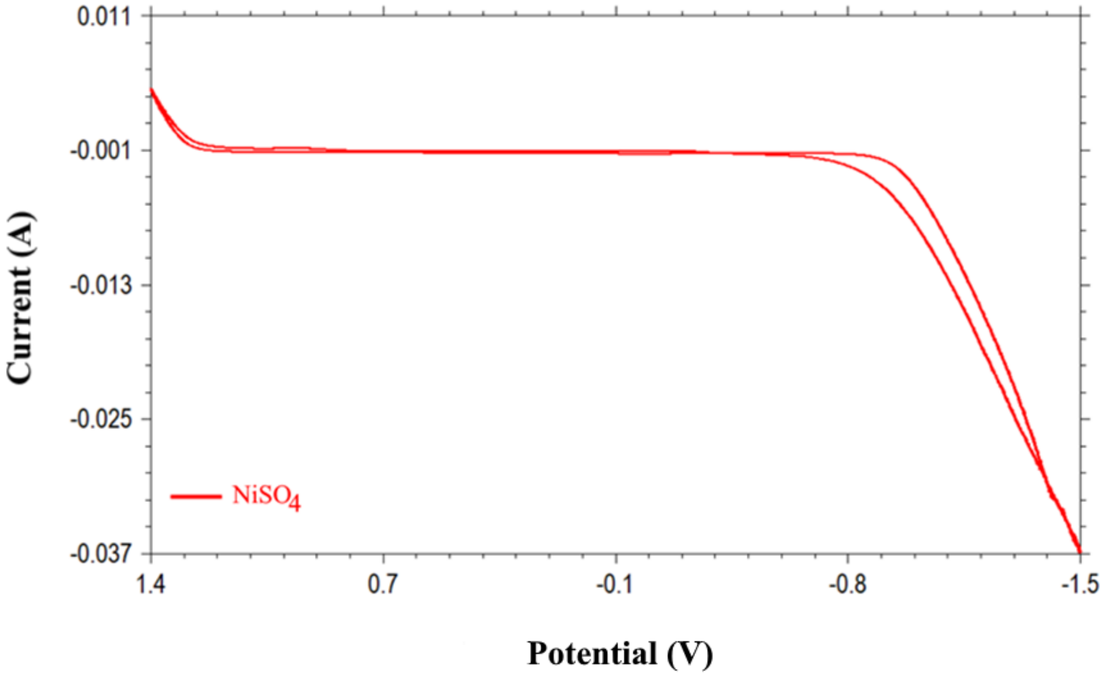

Figure 1. *Cyclic voltammetry plot of the nickel sulfate bath*

In addition to nickel sulfate, first nickel chloride and then chromium chloride were added to the storage bath to obtain CV graphs. The study was carried out at a scanning rate of 10 mV/s and a range of 1.6 V to -1.6 V. As seen in Figure 2, the reduction potential with nickel chloride added to the nickel sulfate bath was around -0.85 V. At the same time, the current value between 0 V and 0.83 V was around 0 A and no reduction occurred. When switching to the anodic side, a dissolution peak of around -0.35 V was obtained. Then, with the chromium chloride added to the bath, the reduction potential shifted to the positive side by about 0.13 V and was obtained around -0.7 V. In addition, the current range, which is constant at 0 A, where reduction does not occur, is observed between -0.7 V and -0.2 V. Although the anodic dissolution peak occurred around -0.35 V, similar to the previous bath, it was determined that the obtained peak was larger as seen in section A. This information shows that the deposition takes place in a single phase (Ersin Ünal, 2016). This is in line with the CV studies of NiSO4 alone and NiSO4+NiCl2 baths in the work of Ünal and Karahan on Ni-B coatings (Ünal & Karahan, 2018).

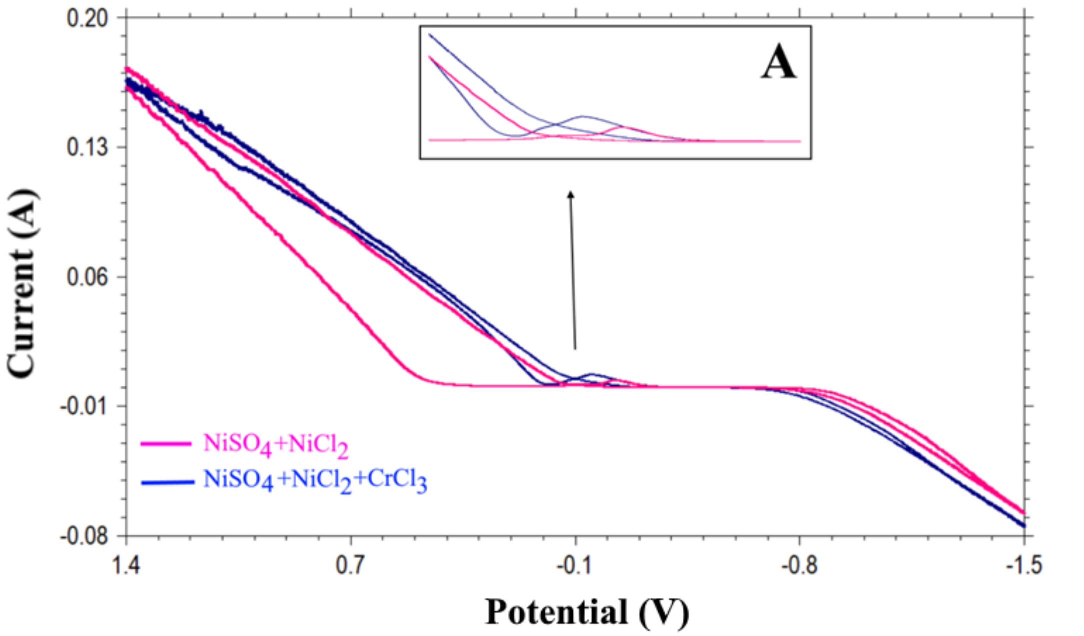

Figure 2. *Cyclic voltammetry curves from baths*

The preferred corrosion measurement technique has been the Tafel extrapolation method. As a result of the test, the corrosion rate of the sample (mm/year) was taken. Tafel graphs taken from the obtained coatings are given in Figure 3. If a preliminary assessment is made of the Tafel results, it can be said that the sample closer to the potentially positive side has better corrosion resistance. However, this alone is not sufficient for evaluation. At the same time, the fact that the current has logarithmically smaller values shows that the corrosion resistance of the sample is better (Wang et al., 2022).

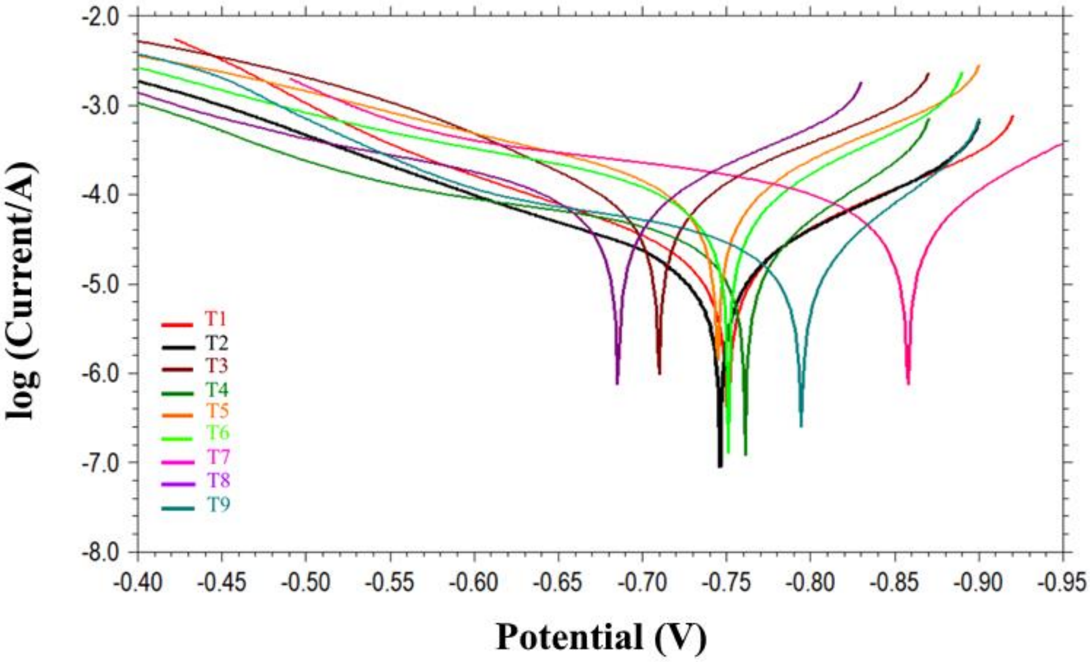

Figure 3. *Tafel curves of Ni-Cr coatings*

Corrosion values (mm/year) were determined as a result of the analysis of Tafel curves. Optimization of these values was determined by the Taguchi method. The S/N ratios of the experimental data were calculated in Table 4 to determine the variable levels that give the optimal corrosion value.

Table 4. *Assignment of level and factor values and experimental results*

Name	Parameters				Corrosion Rate (mm/year)	S/N Rate (dB)
	pH	Temperature (°C)	Current Density (mA)	Stirring Speed (rpm)		
T1	2,5	30	100	0	0,31928	9,91657
T2	2,5	40	200	100	0,22260	13,04950
T3	2,5	50	250	300	1,20421	-1,61404
T4	3,5	30	200	300	0,35941	8,88820
T5	3,5	40	250	0	0,62001	4,15203
T6	3,5	50	100	100	1,32486	-2,44340
T7	4,5	30	250	100	0,94488	0,49247
T8	4,5	40	100	300	0,72898	2,74569
T9	4,5	50	200	0	0,26492	11,53771

Table 4 shows the effects of pH, temperature, current density and stirring rate on the corrosion value. According to Table 5, the level that gives the smallest value is the best value for that parameter. If this situation is examined from the chart; 2.5 pH, which is the smallest preferred pH value among the experiments, had the lowest corrosion value. For temperature, it was obtained at the second level, 40 °C. 200 mA/cm2, which is the second level, gave the best value in current density. At the stirring rate, the first level without stirring gave the smallest value. These parameters, which affect the corrosion result, are examined below, respectively.

Table 5. *Corrosion values and order of factors*

Level	pH	Temperature (°C)	Current Density (mA)	Stirring Speed (rpm)
1	**0,47**	**0,54**	**0,79**	**0,34**
2	**0,77**	**0,43**	**0,25**	**0,83**
3	**0,65**	**0,93**	**0,92**	**0,76**
Difference	**0,29**	**0,50**	**0,67**	**0,49**
Ranking	**4**	**3**	**1**	**2**

pH Effect

The pH value was limited between 2.5 and 4.5 by the literature review in the study. The effect of pH on corrosion of Ni-Cr alloy coating is shown in Figure 4. The best pH value was found to be 2.5 in the proposed bath. Control of internal stress in coatings occurs with pH. In order to obtain acceptable internal stress values, low pH is generally preferred (Mohamed & Golden, 2016; Schlesınger & Paunovıc, 2010). However, this situation is different for each solution and is experimental. There is no linear behavior between the increase in pH value and the increase in corrosion resistance. In corrosion behavior, it is seen that when the pH value is 4.5, it gives better corrosion resistance than 3.5.

Researchers working on Ni-Cr alloy coating used different pH values in their studies. In general, the preferred pH value for the researchers was between 2 and 3 and is consistent with our study (Etminanfar & Heydarzadeh Sohi, 2012; Firouzi-Nerbin et al., 2020; Sheibani Aghdam et al., 2015; J. Sun, Du, Lv, Zhou, Wang, & Qi, 2015; Surviliene, Češuniene, Selskis, & Butkiene, 2013; Tharamani, Hoor, Begum, & Mayanna, 2006; Zhang et al., 2015). Studies on determining the pH range of the Ni-Cr alloy have used the range of 1.5 to 4 (X. Li-jian et al., 2017; Yang et al., 2006). Xu Li-jian's study also determined the pH value with the highest Cr storage amount and hardness value as 2.5 (X. Li-jian et al., 2017). Yang Yu-fang also obtained the highest hardness value at pH 2.5 (Yang et al., 2006).

In coatings obtained by electrodeposition method other than Ni-Cr, the change of pH, which is the acidity value, depends on the coating hardness (Ferkel, Müller, & Riehemann, 1997; Jeyaraj et al., 2018), the amount of particles present in composite coatings (Jeyaraj et al., 2018; Kim & Yoo, 1998; Lee, Lee, & Jeon, 2007; Low, Wills, & Walsh, 2006; Narasimman, Pushpavanam, & Periasamy, 2011; Srivastava et al., 2017; W. chang Sun, Zhang, Zhao, Tian, & Wang, 2015), zeta potential (Lee et al., 2007; Xu, Wang, Dong, Jiang, & Tu, 2005) and surface properties (NIU et al., 2007) are studies that affect the results. The fact that the results are not linear with the differentiation of pH has been confirmed not only in terms of corrosion, but also in the aforementioned results.

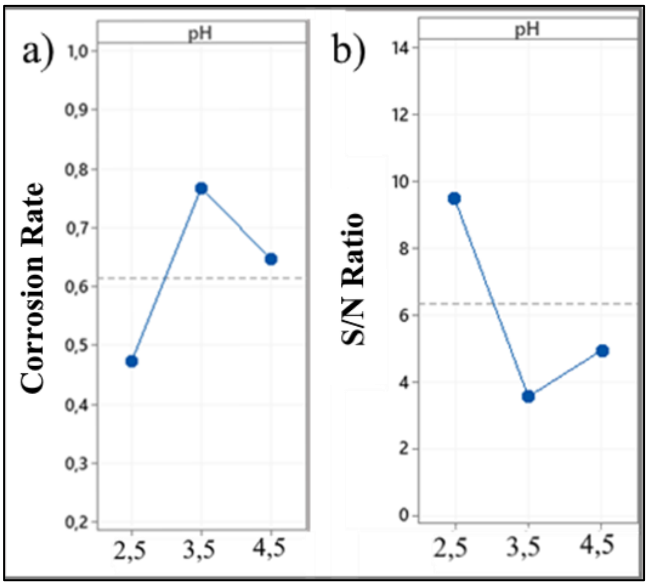

Figure 4. *The effect of pH value on a) corrosion b) S/N ratio*

Effect of Temperature

Nickel baths are usually deposited at temperatures between 30 °C and 60 °C (Bathini, Prasad, & Wasekar, 2022; You et al., 2022; Zhou et al., 2022). Therefore, the range of 30-50 °C was chosen to detect the effect of temperature on the coating. Figure 5 shows the effects of bath temperature on the corrosion behavior of the Ni-Cr coating. It has been observed that the room temperature level is not compatible for corrosion. With the increase in temperature up to 40 °C, samples with better corrosion values were obtained. With the increase in temperature more than this value, the decrease in the negative direction was quite high. Due to the two opposing effects of temperature, it is thought that when starting from room temperature and increasing the temperature, the deposition becomes more homogeneous and fine-grained as diffusion accelerates. However, it is thought that there is a decrease in the corrosion resistance of the coating due to the effect on the cathode polarization with the continuation of the temperature increase (Morana, 2006). These reciprocal effects make it difficult or impossible to determine the operating temperature for any given bath, so the temperature must be determined experimentally.

Ni-Cr coatings are generally studied at room temperature and 30 °C temperature due to the difficulty of heating the coating bath and keeping it at a constant temperature (Etminanfar & Heydarzadeh Sohi, 2012; Huang, Chang, Chen, Liao, & Mayer, 2013; Kunyarong & Fakpan, 2018). Articles examining the temperature range in Ni-Cr coating studies have chosen the range of 30 to 50 °C (Surviliene, Češuniene, et al., 2013; Yang et al., 2006). Jeyaraj, in his study on optimizing Ni-Cr composite coating, made an examination between 30 and 60 °C temperatures and determined that the ideal value is 45 °C. He stated that the hardness of the coatings obtained below or above this temperature value is adversely affected (Jeyaraj et al., 2018). Gamburg and Zangari reported that the ideal temperature for Cr deposition is 40 °C in the Cr related chapter of their book (Gamburg & Zangari, 2011).

Apart from Ni-Cr coatings, similar temperature ranges have been used in studies on the deposition of different alloy or composite coatings by electrodeposition method. Kerimzadeh et al. In their review studies, they stated that the Ni-Co temperature range was inconsistent in terms of temperature among the researchers. However, they reported the temperature range as 40-60 °C in their bath recommendations (Karimzadeh, Aliofkhazraei, & Walsh, 2019). Narasimman et al. They examined the range of 30-60 °C in Ni/SiC electrodeposition studies (Narasimman et al., 2011). Accurate control of the operating temperature in the electrodeposition process is a vital factor for the proper performance of the electrolyte (Karimzadeh et al., 2019).

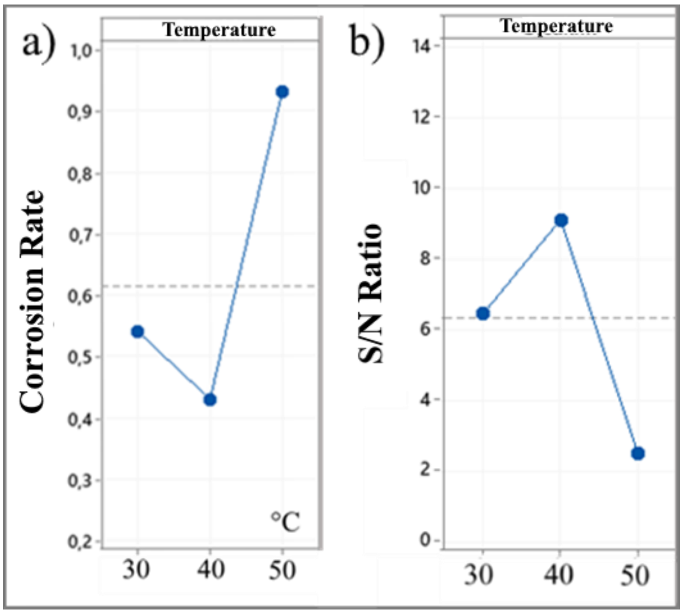

Figure 5. *The effect of temperature value a) corrosion b) S/N ratio*

Effect of current density

The effect of current density on corrosion of Ni-Cr alloy coating is shown in Figure 6. According to Figure 6, the current density value that gives the best corrosion resistance is 200 mA/cm2. It is seen that the corrosion resistance value has increased slightly compared to 100 mA/cm2. The main reason for this is the increase in the amount of Cr accumulated with increasing current density values (Huang, Chen, Chen, Kelly, & Lin, 2014; Huang, Lin, & Chen, 2009). It is known that the increase in the presence of Cr in the coating improves the corrosion resistance (Etminanfar & Heydarzadeh Sohi, 2012; Kunyarong & Fakpan, 2018). At higher current density levels, increasing the presence of Cr metal increases cracks in the coating. The characteristic feature of Cr coatings is that they contain cracks (Surviliene, Češūnienė, Selskis, & Butkienė, 2013; Tavoosi & Barahimi, 2017). In addition, increased current density can cause irregularities in the coating. Many researchers have investigated in their research that higher current density levels are not suitable for improved deposition and final properties (Jeyaraj et al., 2018).

In studies investigating the effect of current density, it was similarly supported that the change in corrosion values with the change of current density is not linear. Aghdam took it in the range of 170, 190, 210 mA/cm2 and obtained the best corrosion value at 190 mA/cm2 (Sheibani Aghdam et al., 2015). Sun et al. The microhardness values of the coatings obtained at different current density values (150-350 mA/cm2) for Ni-Cr coatings were examined. In their study examining the effect of current density on microhardness, they determined the current density, which gives the best hardness value, as 250 mA/cm2 (J. Sun, Du, Lv, Zhou, Wang, Qi, et al., 2015). Similarly, for this case, it shows that there is no continuous increase in the hardness value with the increase in current density. Razaghi et al. They examined the current density in the range of 100 to 400 mA/cm2 and its effect on the amount of Cr. They reported that the amount of Cr increased with the current density, but the corrosion resistance first increased and decreased with the increase of the current density value (Razaghi, Rezaei, & Tabaian, 2020).

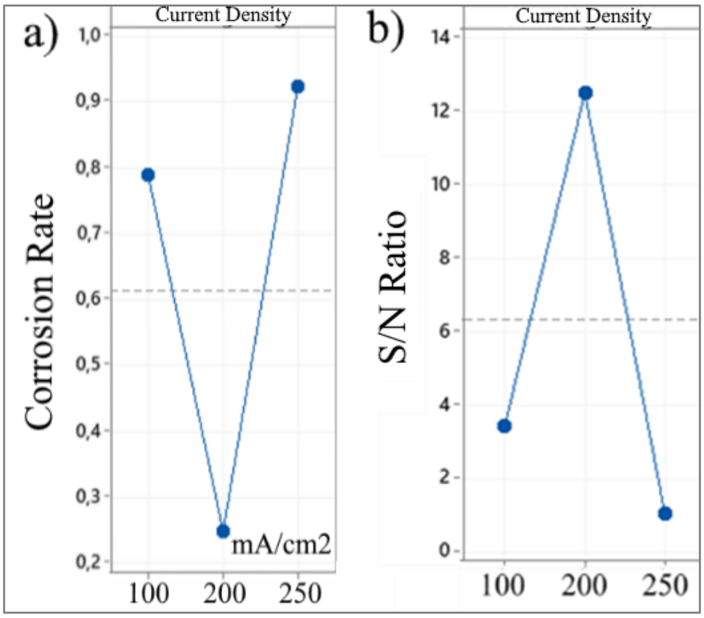

Figure 6. *Effect of current density value on a) corrosion b) S/N ratio*

Effect of stirring rate

The effect of stirring rate on corrosion of Ni-Cr alloy coating is shown in Figure 7. As can be seen from Figure 7, the condition where the bath is not stirred in the production of the Ni-Cr alloy coating has the best corrosion resistance. In this case, it can be concluded that if there are no particles in the coating bath, there is no need for stirring. A rapid decrease in corrosion resistance was observed when the bath was stirred rapidly. It can be shown that the main reason for this is that the ions in the bath had a problem in adhering to the substrate by hitting the substrate quickly when the bath was stirred.

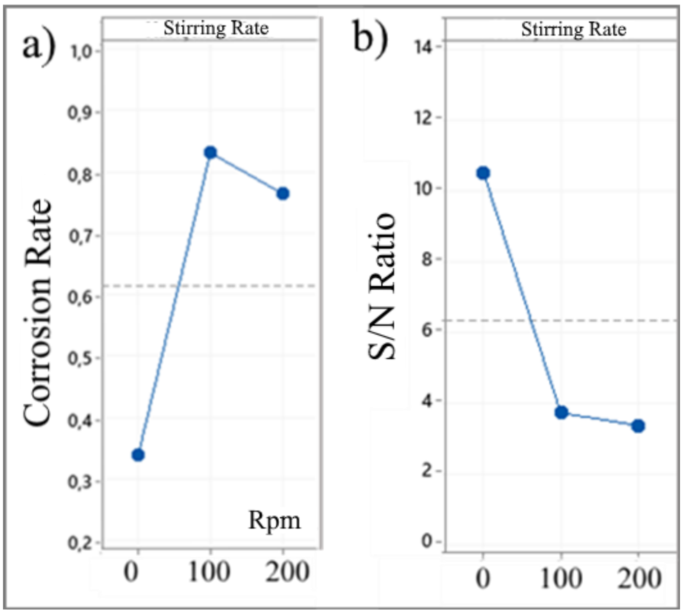

Figure 7. *Effect of stirring rate value on a) corrosion b) S/N ratio*

Stirring is generally not required to obtain alloy coatings. In such coatings, stirring is needed when particles are added to the coating bath. Maintaining a homogeneous temperature in the plating bath, keeping both ions and particles suspended in the electrolyte, and aiding their transport through the cell are the main reasons for using stirring. At the same time, a correct stirring is important in the uniformity of the coating thickness (García-Lecina, García-Urrutia, Díez, Morgiel, & Indyka, 2012; Mohamed & Golden, 2016).

In this study, the effect of stirring rate on the Ni-Cr alloy coating was investigated. Researchers working on Ni-Cr alloy coating generally did not need stirring (Adelkhani & Arshadi, 2009; Kunyarong & Fakpan, 2018; X. U. Li-jian, Zhu-qing, & Jian-xin, 2007; Peng et al., 2004; Razaghi et al., 2020; Saravanan & Mohan, 2011; Sheibani Aghdam et al., 2015; Surviliene, Češuniene, et al., 2013; Zhang et al., 2015). This study supports the result that no stirring gives the best corrosion resistance. In addition, there are studies that obtain coating by stirring. The values of 300 rpm (Firouzi-Nerbin et al., 2020; Huang et al., 2013; Huang, Chen, Hsu, & Lin, 2007; YOUSEFI, Irannejad, & SHARAFI, 2019), 250 rpm (Etminanfar & Heydarzadeh Sohi, 2012), 180 rpm (Zhong et al., 2013) were used in the studies and these values are suitable for the ranges selected in the study.

In the studies performed outside the Ni-Cr alloy coating; In the study on Ni/SiC coating, the stirring range of the coating bath was selected in the range of 50-400 rpm, and it was not observed that the storage rate first increased and then decreased (Lee et al., 2007). Bahadormanesh and Dolati changed the stirring rate between 100 and 800 rpm in Ni-Co/SiC composite coating studies and measured the amount of SiC in the coating. They stated that with the increase of stirring rate, the accumulation rate first increased and then decreased (Bahadormanesh & Dolati, 2010). Jeyaraj used 250-300-350 rpm stirring rate in their studies in which they embedded Cr particles in Ni matrix. At this stirring rate, the highest hardness was obtained at 250 rpm (Jeyaraj et al., 2018). As seen in these studies, the increase in stirring rate shows the necessity of optimizing even in the case of particles in the coating bath. The stirring rate was not investigated in non-particle alloy coatings. In this study, the effect of stirring rate on corrosion was investigated.

ANOVA Analysis

It is useful to perform statistical analyzes to confirm the reliability of the results. The Taguchi method is used to optimize a single answer. In one case, the smaller is better, while in the other case, the large is better. Multivariate analysis of variance (ANOVA) can be used in the optimization of multiple outputs. In this way, the effect percentages of the parameters on the results can be determined. The analysis of variance method was applied for Taguchi (L9) orthogonal array and the effects of input parameters on corrosion were investigated (Table 6).

Table 6. *Variance analysis of corrosion values according to S/N ratios*

	Degrees of Freedom	Sum of Squares	Mean Squares	F	P
pH	2	18,64	9,32	8,67	0,23
Temperature	2	31,76	15,88	14,76	0,18
Current Density	2	179,14	89,57	83,28	0,08
Stirring Speed	2	51,42	25,71	23,90	0,14
Error	1	1,08	1,08		
Total	9	282,03			

The parameter with a P value close to zero has a greater effect on the result (Güvenç, Çakır, & Mıstıkoğlu, 2019). In the ANOVA charts, the ratios of the parameters affecting the corrosion rate were calculated as percentages. When Table 6 is examined, it is seen that the parameter that most affects the corrosion resistance of the coatings is the current density with 63.76%. It is seen that the following parameters are stirring rate with 18.3%, temperature with 11.3% and pH value with 6.64%, respectively. Statistically, the approximation of R2 value to 1 indicates the closeness of the prediction model to the real relationship. Since R2 is 97.49% in the predictive equation obtained in this study, its acceptability is quite high. In addition, in Figure 8, the percentages of the factors are shown as pie charts.

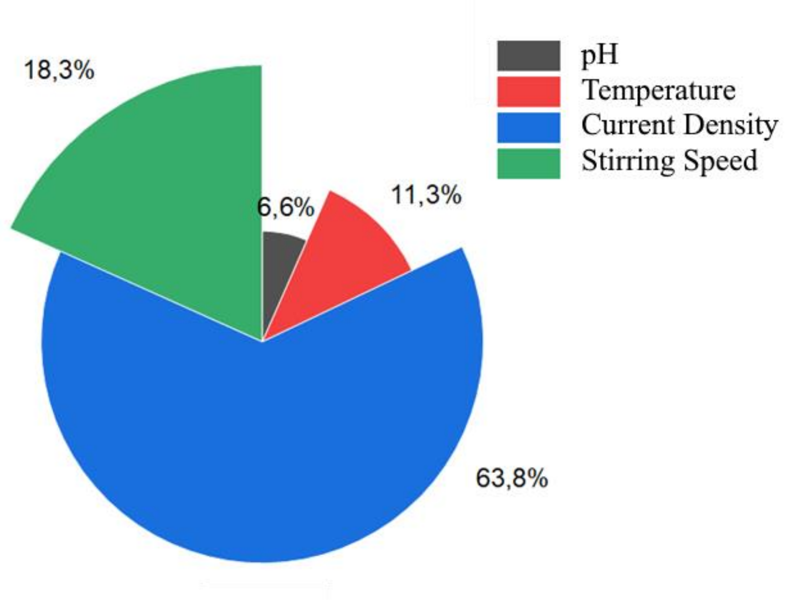

Figure 8. *Pie chart of the effects of factors on corrosion rate*

Validation Experiment

Taguchi experimental design method was used in this study on the coating of Ni-Cr alloy coating on AISI 1040 steel substrate by electrodeposition method. Three different levels of four different factors affecting the coating method were determined and experimental parameters were obtained according to Taguchi L9 orthogonal. Experiments were carried out according to the obtained experimental parameters and the experiments were subjected to corrosion in 3.5% NaCl. Corrosion values were measured by Tafel extrapolation method and corrosion rates were determined in mm/year. According to the Taguchi analysis; It shows that the best values are 2.5 for pH, 40 °C for temperature, 200 mA/cm2 for current density, and no stirring gives the best results, respectively. However, among the nine experiments, there was no level set that gave this best result. Therefore, a confirmation experiment is required (Mohamed & Golden, 2016; Pattanaik, Satpathy, & Mishra, 2016; Siddhartha, Patnaik, & Bhatt, 2011). The graph of the validation experiment obtained by the Tafel extrapolation method is given in Figure 9.

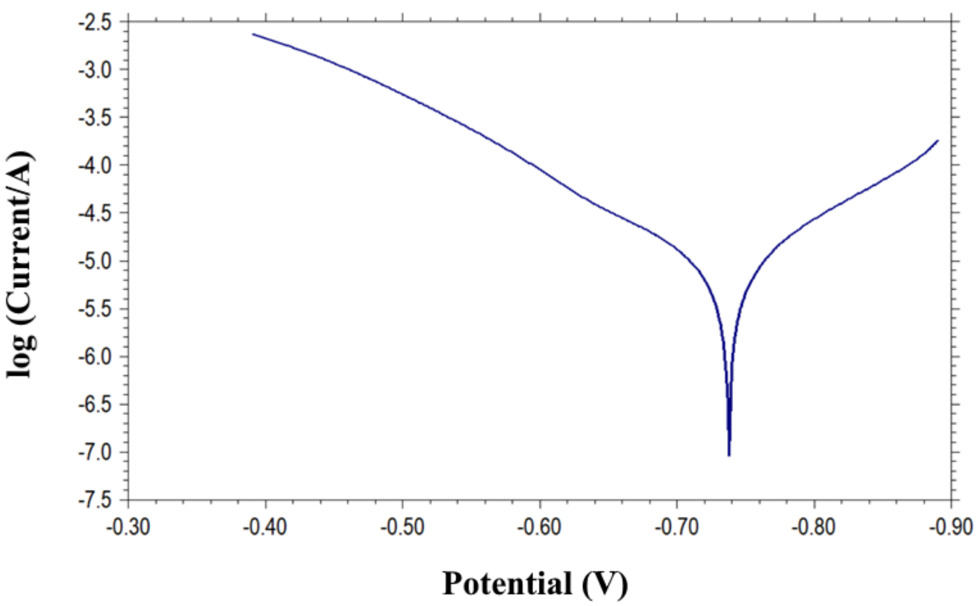

Figure 9. *Confirmation test tafel curve*

The corrosion rate and test parameters calculated according to the obtained Tafel curve are shown in Table 7. According to the table, it is seen that the T2 coating, which gives the best results among the L9 orthogonal, is subject to approximately 0.07 mm less corrosion per year. In addition, -738 mV was measured for the corrosion voltage verification sample and it was determined that it shifted to the positive side of 8 mV compared to the T2 sample.

Table 7. *Confirmation test parameters and corrosion rate*

Name	Experiment Parameters				Corrosion Rate (mm/yıl)
	pH	Temperature (°C)	Current Density (mA)	Stirring Speed (rpm)	
Validation	2,5	40	200	0	0,15100

SEM picture of the coating obtained as a result of the validation test at 5000x magnification are given in Figure 10. As seen in Figure 10, cracks were observed in the Ni-Cr coating as seen in previous studies (Surviliene, Češūnienė, et al., 2013; Tavoosi & Barahimi, 2017). The presence of cracks is characteristic of Ni-Cr coatings. To eliminate this situation, particles or additives should be added to the bath. Also, the morphology of the Ni-Cr coating exhibited a typical spherical nodular structure (Demir et al., 2020; Sheibani Aghdam et al., 2015).

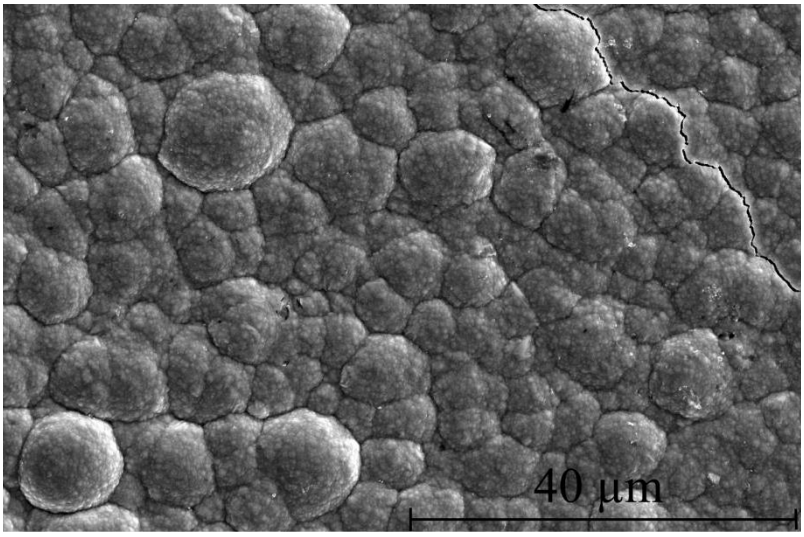

Figure 10. *SEM image of the sample obtained by the validation experiment*

Figure 11 shows the XRD analysis of the Ni-Cr alloy coating obtained as a result of the verification test. Angle values and DB card numbers of the obtained peaks are given in Table 8. The most striking point in the graph is the presence of nickel-specific diffraction peaks. The most intense peak in the sample (111) was the peak. An alloy of 0.16 (Ni_2Cr_{23}) was found at approximately 44.5, 51.8 and 76.3 degrees, which corresponds to the 2θ value at which Ni peaks occur. The presence of this peak is consistent with the literature (Suswanto, Muhammad Suchaimi, Hariyati Purwaningsih, Rochman Rochiem, 2017).

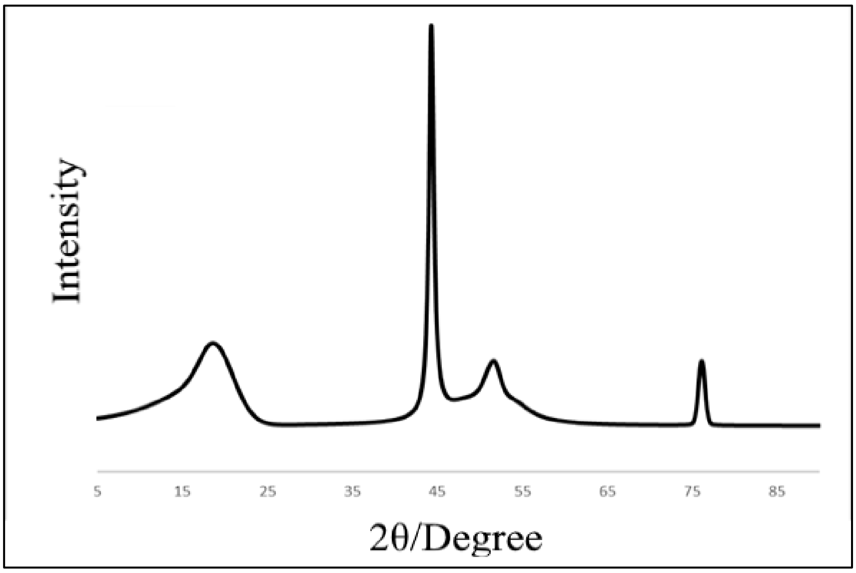

Figure 11. *X-ray diffraction patterns of Ni – Cr alloy coating*

Table 8. *Angle values and DB Card numbers*

Chemical Formula	2-theta (degree)	Phase Name	DB Kart Numbers
Ni	44,4 51,8 76,3	111 200 220	00-004-0850
Cr	18,6 44,4	100 110	01-073-9565
$(Cr_2Ni_{23})_{0.16}$	44,4 51,8 76,3	111 200 220	01-077-7616

CONCLUSION

Ni-Cr coatings were produced on AISI 1040 steel using the Taguchi experimental design method with the electrodeposition method, and the result giving the best corrosion value was determined. The results obtained are briefly summarized below.

• Deposition voltages of metal source salts added to the bath were determined by cyclic voltammetry technique (CV). Baths containing nickel sulfate, nickel sulfate + nickel chloride and nickel sulfate + nickel chloride + chromium chloride were analyzed, respectively. As a result, metal sources added to the bath changed the reduction peaks. It was determined that the bath containing three metal salts gave the highest dissolution peak and had single-phase peaks.

• The effects of four different parameters on the corrosion rate were investigated in the deposition of Ni-Cr alloy coating. The investigated parameters are pH, temperature, current density and stirring speed, respectively. Taguchi method suggested L9 orthogonal index for three different levels of four parameters. Experiments were performed according to this index.

• Coatings were obtained according to the L9 orthogonal index and the corrosion results in 3.5% NaCl were examined. Corrosion rates (mm/year) of the curves obtained using the Tafel extrapolation method were determined. Among the experiments performed, the sample with pH 2.5, temperature 40 °C, current density 200 mA/cm2 and no stirring gave the best results.

• As a result of the analysis of variance obtained according to the S/N ratio, the effects of the parameters on the corrosion in the coating were determined. Current density had the greatest effect on the corrosion resistance of the coating with 63.8%. Stirring rate 18.3%, temperature 11.3% and pH value 6.6% affected the result.

• Since R2 is 97.49% in the estimation equation obtained according to the analysis of variance, its acceptability is quite high.

• According to the results obtained, the parameter levels suggested by the Taguchi experiment are not available in the L9 orthogonals. Therefore, a validation experiment was carried out. According to the validation test, the corrosion rate was found to be 0.151 and Taguchi confirmed the proposal of the experimental design method.

• The SEM picture of the verification experiment shows that the Ni-Cr coating with characteristic cracks was obtained. In addition, it was observed that the obtained picture had a typical spherical nodular structure.

Acknowledgements

The authors would like to thank The Scientific and Technological Research Council of Turkey (TUBITAK-BIDEB) for Ph. D scholarship (Mehmet DEMİR) 2211-C (the encouragement scholarship for priority areas).

This publication was produced from Mehmet Demir's doctoral thesis.

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