Application of an experimental design to study AISI 4340 and 300M steels electropolishing in a concentrated perchloric/acetic acid solution

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Abstract

The objective of this study was to assess AISI 4340 and 300 M steels electropolishing performance in a concentrated perchloric/acetic acid electrolyte. The statistical analysis on a two-level fractional design (FFD) 2^{4-1} was proposed to define an adequate tool to describe the dissolved thickness and the final surface via arithmetic roughness Ra. A compromise zone was defined for each steel by considering all the requirements for both responses of each steel: dissolved thickness between 15–17 μm and arithmetic roughness criteria less than 0.06 μm.

1. Introduction

Electropolishing (EP) is an electrochemical surface cleaning-finishing process allowing metal to be electrolytically removed under specific conditions [1–19]. The first work dealing with this process was published in 1910 [1] and its development is due to Jacquet [2]. Nowadays, EP is traditionally used for esthetic applications resulting in an attractive mirror finish. This process can also be used for deburring, brightening and passivating —ferrous and nonferrous alloys. EP has become a common treatment for stainless steels [5, 13–18], copper [8, 10, 11], titanium [7, 12, 19] and niobium [20] in several high-technology applications such as cardiovascular and orthopedic body implants, pharmaceutical and semiconductor installations and so on.

The main objective of EP is to drastically minimize the surface micro roughness thus reducing the risk of dirt or product residues adherence and improving the clean ability of the surfaces [13–18, 21]. Another benefit of EP is that, contrary to classical cleaning processes (acid pickling, etc), this technique produces a surface free of hydrogen. Furthermore, this electrolytic process permits to obtain undisturbed and metallurgical clean surfaces [21, 22] contrary to the mechanical surface treatments which provide mechanical and thermal stresses.

Most of the published works related to the fundamental understanding of EP comprised the study of copper electropolishing in phosphoric acid [23–25] although some works on stainless steels and passive metals (Ti, Ni, Cr, Nb, Al) have also been reported [9, 14–20, 26, 27]. Readers can find numerous theories developed to understand the electrochemical fundamental mechanisms in recent reviews [14–17]. If little information is available on the mechanisms involved regarding ferrous alloys, the technological aspects of electropolishing, including electrolyte composition and operating conditions, have been described in literature [13, 28–30]. EP is carried out in electrolytes with high viscosity and/or low conductivity such as concentrated acids (e.g. sulfuric, phosphoric) or non-aqueous solutions (ethylene glycol, methanol-sulfuric acid). Tegart [4] and Shigolev [31] have reported overviews of typical formulas of EP electrolytes for different metals and alloys. It is well known

Table 1. Composition (max) of AISI 4340 and 300 M steel sheets.

	Amount of the following elements (wt.%)									
Element	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	V
4340	0.43	0.85	0.35	0.015	0.008	0.90	2.00	0.30	0.35	/
300 M	0.45	0.90	1.80	0.01	0.01	0.95	2.00	0.50	0.35	0.1

that, for a given material, the surface property depends on numerous EP operating parameters such as applied current density, voltage, temperature, concentrations of the chemicals used, etc.

The aim of this paper is to determine a set of EP process experimental conditions for AISI 4340 and 300M steels in a perchloric/acetic solution allowing (i) the dissolution of a sufficient thickness to eliminate the layer affected by residual compressive stresses due to the mechanical polishing and (ii) a low roughness. Most of the previous works investigated the influence of each EP parameter one at a time while keeping the others constant. This conventional step by step approach for optimization purposes involves a large number of independent runs and does not take into account the possible interactions between factors. In order to overcome this problem, an experimental design is used. This approach has the dual advantage of taking into account the combined effects of several input variables and requiring only a moderate number of experiments [32–36]. In this respect, a two-level fractional factorial design (FFD), noted 2^{4-1}, is conducted. Based on the literature review, the following EP variables were investigated in the FFD study: (i) electrical charge amount, (ii) anodic current density, (iii) iron concentration of solution perchloric/acetic solution and (iv) temperature of EP solution. It is worth noting that in our knowledge any paper is devoted to study the AISI 4340 and 300 M steels electropolishing performance in a concentrated perchloric/acetic acid electrolyte.

2. Materials and methodology

2.1. Electropolishing apparatus and electrolyte

A typical electropolishing installation is used, comprising a direct current (DC) power source (Fontaine S30050 (300 V-50 A)), a cell fitted with a lead sheet (50 × 100 mm^2) as cathode (−) and a steel part as anode (+) with an inter-electrode distance of 20 mm. The amount of metal removal depends on the electrolyte composition, the temperature, the current density and the metal being electropolished.

The electrolytes used are mixtures of concentrated analytic grade perchloric acid/acetic acid (5/95 Vol.%) [31]. To study the effect of the iron enrichment of EP electrolytes during electropolishing, an iron ion concentrate solution is preliminary prepared by dissolving iron sheet in a perchloric/acetic acid mixture. The working solution is obtained by diluting the iron -ion concentrate solution into adequate concentration. The temperature is maintained at a given level by thermostated water circulating through the jacketed electrochemical cell (250 ml).

2.2. Sample preparation and characterization

AISI 4340 and 300M steel sheets (electrode area 3 cm^2) are used in this study. Their chemical compositions (wt.%) are given in table 1. 300 alloy is similar to 4340 with the addition of vanadium and higher silicon content. 300 M steel offers a combination of toughness and ductility at high strength levels without increasing carbon content.

Before EP, the specimens are (i) degreased in an alkaline solution, (ii) mechanically polished with 600-grit SiC paper, (iii) rinsed with water then with alcohol, (iv) air-dried and then (v) weighted. After EP, the specimens are (i) rinsed with tap water, distilled water and alcohol, (ii) air-dried and (iii) weighted again. The dissolved thickness is computed using Faraday's law. Each test is repeated twice under the same conditions.

Surface morphology is observed by Contour GT-I 3D Optical Microscope and surface roughness measurements are made using a Mahr-Penthen perthometer S6R profilometer. Measurements are repeated three times for each specimen and the criteria roughness (R_a) is obtained thereby.

2.3. Methodology - experimental design [32–36]

As the literature indicates [1–15], the factors that affect the electropolishing process are numerous including pre-treatment of the metal surface, orientation from the workpiece in the electropolishing bath, choice of cathode material, electrode spacing, bath age, electropolishing time, temperature, composition of the bath, voltage or current density imposed, …

As many factors are involved and must be optimized in an electropolishing process, there is no one-fit-all parameter set for all electropolishing setups. So, the following four variables are selected and investigated in the

Table 2. Experimental domain of the EP process.

Factors	Unit	Associated variable	Lower level (−1)	Upper level (+1)
Electrical charge	A min dm^{-2}	X_1	48	108
Current density	A dm^{-2}	X_2	12	18
Iron ions concentration	mg l^{-1}	X_3	0	500
Temperature	°C	X_4	14	26

Table 3. Effects and aliases.

$l_0 = b_0$	$l_4 = b_4$
$l_1 = b_1$	$l_{12} = b_{12} + b_{34}$
$l_2 = b_2$	$l_{13} = b_{13} + b_{24}$
$l_3 = b_3$	$l_{23} = b_{23} + b_{14}$

FFD study: U_1: electrical charge amount Q (A min dm^{-2}), U_2: anodic current density (A dm^{-2}), U_3: Iron ions concentration of the EP solution, U_4: temperature of the EP solution.

In addition to the current density and the electrical charge, the choice of factors is strongly linked to industrial practices. The electropolishing bath age i.e. the iron ions concentration in the EP bath is also a big concern during the process. Indeed, it is a standard practice in industry to reuse the electrolyte to keep a low profile of cost and minimize the detrimental effect to the environment [4]. Moreover, the temperature impacts the surface brightness which decreases with the decrease of the temperature. The reaction rate in the limiting current region becomes mass transport controlled as the temperature is increased [5, 14, 15].

A two-level fractional factorial design (FFD), noted 2^{4-1}, is implemented to identify the most influential variables affecting the studied responses and to carry out a low number of experiments, ensuring that the results are as precise as possible and to focus on the main effects and low-order interactions. A non-dimensional coded variable X_i is associated with each natural variable U_i. The limits of the experimental domain in terms of coded variables are identical for all variables and the extreme values are equal to ± 1 (table 2). To check the validity of the empirical model, three central experiments need to be added to the experimental design.

The design is constructed by using the independent generator 1234. This type of design is classified as —'resolution IV' design [33, 34] i.e. all the main effects are confounded with three-factor interactions. From the principle of effect sparsity, a system is likely to be driven primarily by main factor and low-order interaction effects. So, the effects of the high-order interactions (three or greater) are assumed to be negligible, and therefore enabling all the main effects to be determined. Two-factor interactions are confounded with each other thus making it impossible to determine all of them for all responses. The eight selected effects and their aliases are listed in table 3. Eight experiments are replicated to determine the influence of the variables on the four responses noted:

1. Y_1, dissolved thickness (μm) and Y_2, mean roughness (arithmetic average of the absolute values (Ra)) for AISI 4340 steel,
2. Y_3, dissolved thickness (μm) and Y_4, mean roughness (arithmetic average of the absolute values (Ra)) for 300 M steel.

Nemrod-W software is used for regression, statistical analysis and graphical analysis of the obtained data [37].

3. Results and discussion

The requirements for each run and the measured response are summarised in table 4, considering the two levels defined for each of the four retained factors. The evaluation of the model quality of the four responses is done by means of the analysis of variance the results of which are reported in table 5. As can be seen, for each response the regression sum of squares is statistically significant at 99.9% confidence level (***). The main effect of a factor X_i (noted l_i) and the aliases of two-factor confounded variables interaction effects (noted l_{ik}) are estimated by least squares regression. The value and the significance of each coefficient, l_i or l_{ik}, determined by the p-values, are listed in table 6. It is important to mention that the p-value is the probability of getting the displayed value for the coefficient if its true value is zero. In other words, the 'null hypothesis' (H_0 hypothesis) is tested for each l_i or l_{ik}.

Table 4. Experimental matrix in coded variables and measured responses obtained for AISI 4340 and 300 M steels.

Run	X_1	X_2	X_3	X_4	Y_1	Y_2	Y_3	Y_4
1	−1	−1	−1	−1	10.5	0.09	10.6	0.05
1bis	−1	−1	−1	−1	10.7	0.10	10.2	0.07
2	1	−1	−1	1	16.4	0.06	15.8	0.07
2bis	1	−1	−1	1	15.9	0.08	16.0	0.08
3	−1	1	−1	1	16.1	0.11	16.7	0.08
3bis	−1	1	−1	1	15.8	0.09	16.9	0.09
4	1	1	−1	−1	24.0	0.22	24.4	0.19
4bis	1	1	−1	−1	24.3	0.20	24.7	0.16
5	−1	−1	1	1	10.4	0.12	10.1	0.07
5bis	−1	−1	1	1	10.2	0.09	10.4	0.11
6	1	−1	1	−1	15.4	0.07	16.1	0.08
6bis	1	−1	1	−1	15.6	0.08	16.3	0.07
7	−1	1	1	−1	15.8	0.06	16.2	0.04
7bis	−1	1	1	−1	15.6	0.08	16.0	0.05
8	1	1	1	1	24.0	0.11	24.2	0.10
8bis	1	1	1	1	24.2	0.12	24.0	0.11

Table 5. ANOVA of the responses Y_1 to Y_4 for AISI 4340 and 300 M steels.

	Source of variation	Sum of square	Freedom degree	Mean square	F ratio	p-value
Response Y_1	Regression	383.1444	7	54.7349	1390.0930	<0.01 ***
	Experimental error	0.3150	8	0.0394		
	Total	383.4594	15			
Response Y_2	Regression	0.0289	7	0.0041	17.7547	0.0277 ***
	Experimental error	0.0019	8	0.0002		
	Total	0.0307	15			
Response Y_3	Regression	397.7475	7	56.8211	1683.5873	<0.01 ***
	Experimental error	0.2700	8	0.0337		
	Total	398.0175	15			
Response Y_4	Regression	0.0231	7	0.0033	15.1035	0.0497 ***
	Experimental error	0.0017	8	0.0002		
	Total	0.0248	15			

The statistical significances of the model equations are evaluated by the F-test for analysis of variance (ANOVA), which show that the regression is statistically highly significant at a 99.9% ($p < 0.001$) confidence level (***).

Table 6. Values and statistical analysis of the effects for AISI 4230 and 300 M steels.

	Y_1 response		Y_2 response		Y_3 response		Y_4 response	
Effects	Estimates	p-value	Estimates	p-value	Estimates	p-value	Estimates	p-value
l_0	16.56	<0.01***	0.104	<0.01***	16.79	<0.01***	0.088	<0.01 ***
l_1	3.42	<0.01***	0.012	1.14 *	3.40	<0.01 ***	0.019	0.0763 ***
l_2	3.42	<0.01***	0.019	0.109**	3.60	<0.01 ***	0.013	0.661 **
l_3	−0.16	1.36 *	−0.013	0.855**	−0.12	2.62 *	−0.011	2.12 *
l_4	0.07	20.3	−0.007	8.7	−0.02	60.1	0.001	90.9
l_{12}	0.73	<0.01***	0.026	0.0132***	0.54	<0.01 ***	0.020	0.0675 ***
l_{13}	−0.02	71.5	−0.009	5.5	0.09	9.3	−0.008	5.7
l_{23}	0.08	14.0	−0.018	0.173 **	−0.16	0.764 **	−0.018	0.138 **

*** Highly significant at the level 99.9%, ** Significant at the level 99%, *Significant at the level 95%.

For a determined factor, if the H_0 hypothesis is verified, this factor is said to be not influent. In practice, a confidence level of 95% is considered i.e. the alpha-level is set at 5%. The alpha level corresponds to the risk of rejecting the H_0 hypothesis when this hypothesis is verified. The test of the H_0 hypothesis is thus rejected, and the factor is considered as influent when $p < 0.05$. Accordingly, the smallest value of the p-value indicates the high significance of the corresponding coefficient.

Table 7. Measured and calculated values for the confirmation experiments for AISI 4340 and 300 M steels.

Runs	Experimental conditions	Measured responses				Calculated responses			
		Y_1	Y_2	Y_3	Y_4	Y_1	Y_2	Y_3	Y_4
17	$X_1 = X_2 = X_3 = X_4 = 0$ i.e.	16.82	0.084	16.67	0.097	16.79	0.088	16.56	0.104
18	$U_1 = 78$ A min dm^{-2},	16.62	0.092	16.56	0.107				
19	$U_2 = 15$ A dm^{-2},	16.80	0.085	16.58	0.112				
	$U_3 = 250$ mg l^{-1},								
	$U_4 = 20$ °C								

3.1. FFD 2 $^{4-1}$ for AISI 4340 steel

From an examination of the results in table 4, the dissolved thickness (Y_1) and Ra (Y_2) of AISI 4340 steel vary respectively from 10.2 to 24.3 μm and from 0.06 to 0.22 μm, indicating that certain factors and/or interactions should show significant effects on the measured responses (Y_1 and Y_2).

For the Y_1 response, data from table 6 reveal that only X_1, X_2, X_3 factors and two confounded interaction effects, 'l_{12}' and 'l_{23}' are significant at a confidence level greater than or equal to 95%. If we consider that interaction effects between the extra factors (X_4) and the basic factors (X_1, X_2 and X_3) are not taken into account in the theoretical analysis due to the hypothesis on the construction of the fractional design, we are able simplify the expression of the confounded effects. For the confounded interaction effect, $l_{12} = b_{12} + b_{34}$, we can assume that the factor interaction effect b_{12} is the dominant term since electrical charge amount (X_1) and current density (X_2) have the greatest effects on the response, as emphasized in the literature. In this respect, we can conclude that the factor interaction effect b_{23} is the dominant term for the confounded interaction effect $l_{23} = b_{23} + b_{14}$.

So, according to the empirical model obtained, the dissolved layer can be represented by the following equation:

$$Y_1 = 16.79 + 3.40X_1 + 0.60X_2 - 0.12X_3 + 0.54X_1X_2 - 0.16X_2X_3 \quad (1)$$

The equation (1) indicates that the positive coefficients of X_1, X_2 and X_1X_2 have a constructive contribution to the dissolved layer response. However, the negative coefficients of X_3 and of X_2X_3 indicate antagonistic effects on the response.

From the analysis of ANOVA (table 5), the results shown in table 6 and following the same analysis as above, a fitted polynomial model (2) can be generated for the roughness criteria, Y_2:

$$Y_2 = 0.088 + 0.019X_1 + 0.013X_2 - 0.011X_3 + 0.020X_1X_2 - 0.018X_2X_3 \quad (2)$$

This model quantitatively elucidates the effects of the EP variables with statistical significance. The equation indicates the synergetic effects of X_1, X_2 and X_1X_2 and antagonistic effects of X_3 and X_2X_3 on the Ra response. Note that for two models (1) and (2), the temperature (X_4) shows no significant effect without any significant interactions.

All these results are confirmed by the high values of the multiple correlation coefficient squares (R^2): 99%, 93% for Y_1 and Y_2 respectively. It should be noted that R^2 values represent the percentage variation in the responses explained by the deliberate variation of the factors in the course of the experiments.

To validate the statistical models (1)–(2), three additional central experiments are conducted. The predicted values are in fair agreement with those measured suggesting that the first order models chosen are suitable and can be used as a prediction equation (table 7).

3.2. FFD 2 $^{4-1}$ for 300 M steel

As previously mentioned, the fractional design allows the calculation of the estimates for factors and two confounded interaction effects for the dissolved thickness Y_3 and the roughness criteria, Y_4 of the 300M steel (table 6).

Following the procedure described above, the equations of the fitted models are:

$$Y_3 = 16.56 + 3.42X_1 + 3.42X_2 - 0.16X_3 + 0.73X_1X_2 \quad (3)$$

$$Y_4 = 0.104 + 0.012X_1 + 0.019X_2 - 0.013X_3 + 0.026X_1X_2 - 0.018X_2X_3 \quad (4)$$

The equation (3) indicates that the positive coefficients of X_1, X_2 and X_1X_2 have a constructive contribution to the dissolved layer response. The negative coefficient of X_3 indicates an antagonistic effect on the response whilst factor X_4 has null effect. Accordingly, the dissolution is enhanced when factors X_1 and X_2 are respectively set as the highest level to obtain a synergistic effect and X_3, as the lowest level. For Y_4 response, X_1, X_2, X_3 factors show significant effect with significant interactions (X_1X_2 and X_2X_3) whilst factor X_4 has null effect.

The values of multiple correlation coefficients, R^2, equal to 99% and 94% for Y_3 and Y_4 respectively show a good fit to the experiment data.

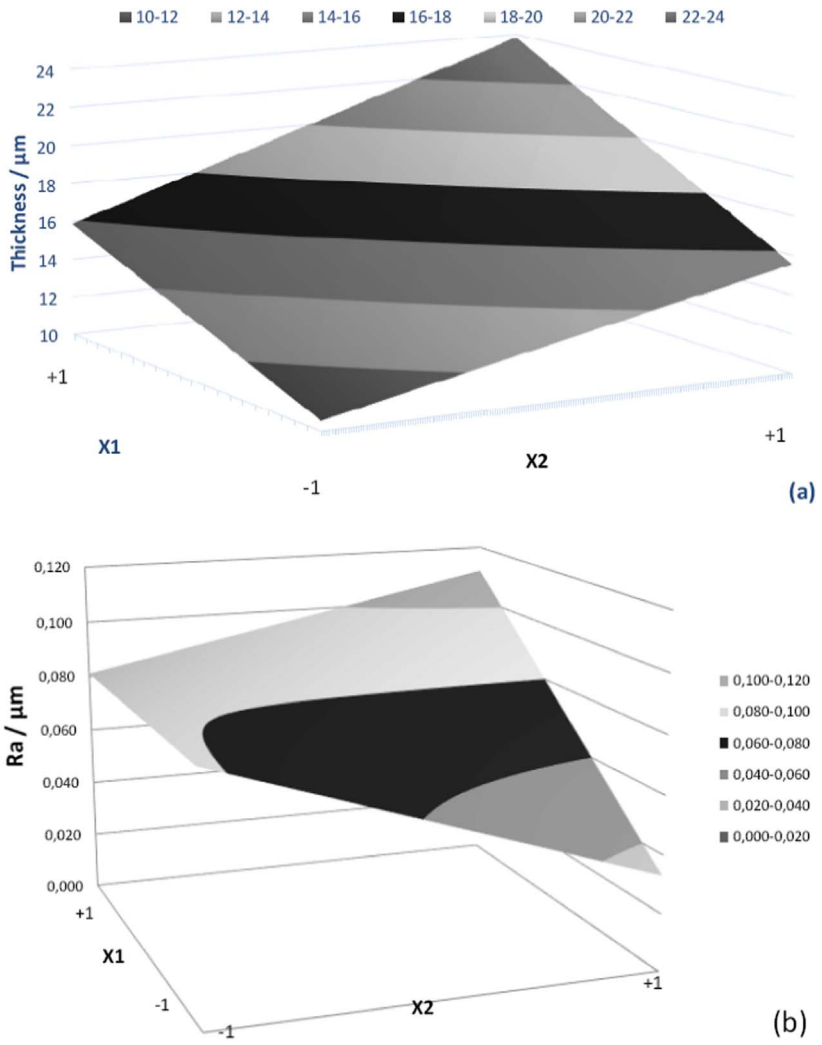

Figure 1. Surface responses: (a) dissolved thickness, (b) Ra for AISI 4340 Steel- with X3 $= +1$.

Three additional central experiments are conducted to validate the statistical models. As shown in table 7, the measured and the predicted values are in close-agreement. For overall results, we can conclude that the first order models chosen are considered suitable for this study and can be used as a prediction equation.

At this point, it is worth noting that the influence of the factors cannot be discussed separately due to the importance of their interactions [32–36]. Indeed, data from table 6 reveal that three factors, X_1, X_2 and X_3, and their interactions are significant according the literature. However, the factor X_4 is not significant which is at odds with literature [5, 14, 15]. Several authors have shown the importance of the temperature for electropolishing on the plateau current density and so on the diffusion coefficient of the rate limiting species in the electropolishing bath. The insignificance of the temperature in the FFD is likely due to the fact that the interval of variation is small.

3.3. Graphical exploitation of validated models—optimum' choice of the EP operating conditions for the two substrates

As indicated previously, the focus of this study is to satisfy one principal objective which is finding the EP operating conditions allowing (i) the dissolution of a sufficient thickness to eliminate the layer affected by residual compressive stresses due to the mechanical polishing and (ii) low roughness. For this purpose, we define an acceptable range for each response: (i) a dissolved thickness comprised between 15–17 μm and (ii) an arithmetic roughness criterium less than 0.06 μm. The evolution of the four considered responses Y_1 to Y_4 are plotted (figures 1–2). Note that, according to the expression of these responses, the X_3 factor (iron ion-concentration) has been kept at its high level (+1). By mere inspection of these diagrams, it is easy to accurately choose each part of the domain that is acceptable according to the criteria above. A satisfactory zone is the part of the domain for which the value of each one of the calculated responses is acceptable (table 8). Taking into account all the requirements for the two responses of each steel, looking for a compromise where all the

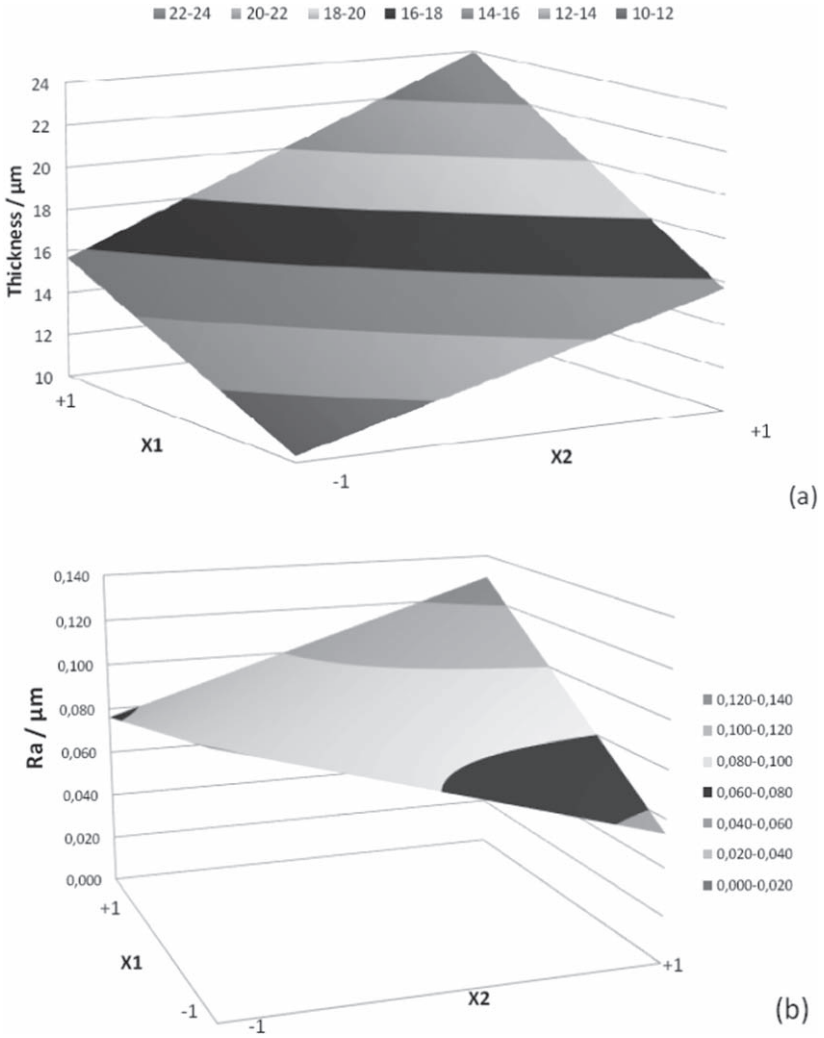

Figure 2. Surface responses: (a) dissolved thickness, (b) Ra for 300M Steel- with X3 $= +1$.

Table 8. Compromise domain fulfilling the requirements for AISI 4340 and 300 M steels.

	AISI 4340		300 M	
Requirements	$Y_1 \epsilon [15; 17]/\mu m$	$Y_2 \leqslant 0.06/\mu m$	$Y_3 \epsilon [15; 17]/\mu m$	$Y_4 \leqslant 0.06/\mu m$
X_i combinations fulfilling each Y_i requirement (i $=$ 1 to 4)	$X_1 \epsilon [-1.00; -0.60]$, $X_2 \epsilon [0.74; 1.00]$, $X_3 = 1.00$	$X_1 \epsilon [-1.00; -0.40]$, $X_2 \epsilon [0.60; 1.00]$, $X_3 = 1.00$	$X_1 \epsilon [-1.00; -0.65]$, $X_2 \epsilon [0.75; 1.00]$, $X_3 = 1.00$	$X_1 \epsilon [-1.00; -0.9]$, $X_2 \epsilon [0.84; 1.00]$, $X_3 = 1.00$
Compromise domain fulfilling $Y_i \epsilon [15; 7]$ with i $=$ 1;3 and $Y_j \leqslant 0.06$ with j $=$ i+1	$X_1 \epsilon [-1.00; -0.60]$, $X_2 \epsilon [0.74; 1.00]$, $X_3 = 1.00$		$X_1 \epsilon [-1.00; -0.9]$, $X_2 \epsilon [0.84; 1.00]$, $X_3 = 1.00$	

experimental responses fulfill the specifications imposed by the researchers to achieve the aims proposed is required (table 8).

3.4. Surface morphology of steels

Figure 3 depicts typical 2 and 3-dimensional micrographies of 300M steel samples after electropolishing according to experimental design runs (runs 1, 4 and 8).

The topography of the surface has been considerably modified and surface roughness is reduced or increased according to the operating conditions. As observed in figure 3(c), peaks and valleys are clearly observed. On the contrary, for figure 3(a) with a lower Ra value, the surface is flat due to a uniform dissolution. Similar profiles are observed for 4340 steel samples.

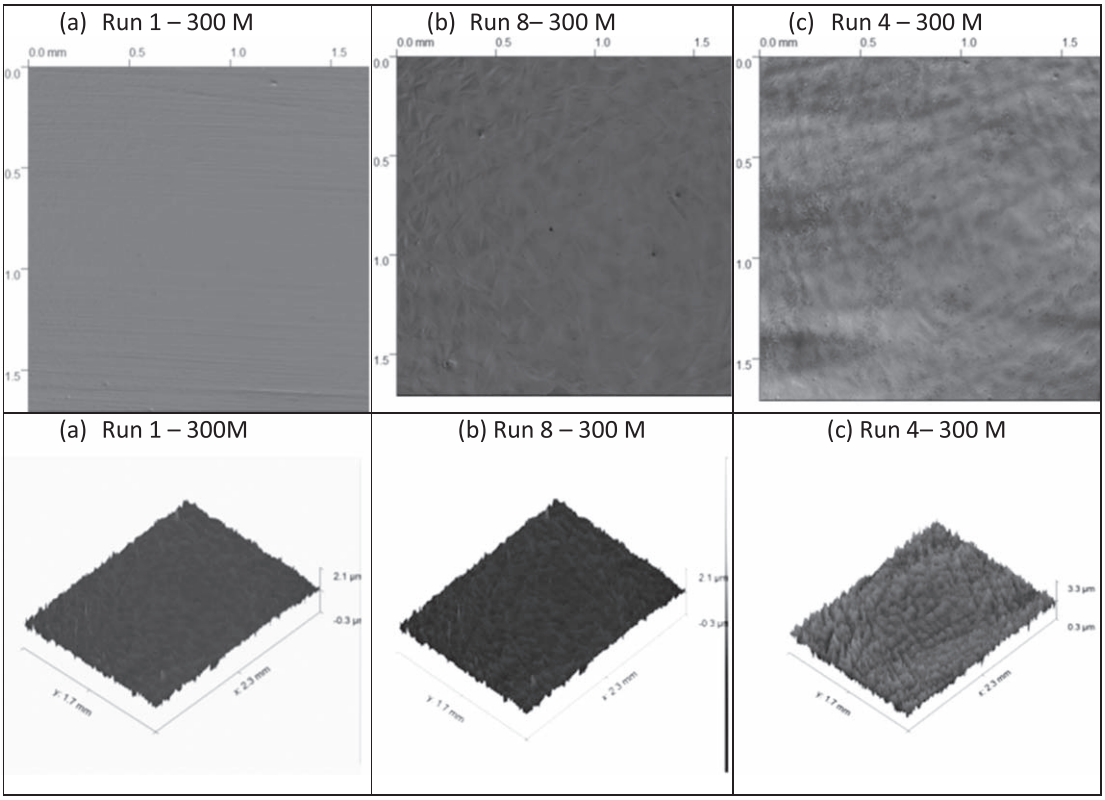

Figure 3. Typical 2D and 3D micrographies −300 M Steel.

4. Conclusion

The electropolishing technique is a surface treatment that used to remove the metal surface rough irregularities without creation of internal stresses. The present study shows that AISI 4340 and 300M steel electropolishing can be achieved in a perchloric/acetic acid electrolyte using statistical methods. The effects of the operating conditions (current density, electrical charge, temperature) and electrolyte composition (Iron ions concentration) are studied using two-level fractional factorial design (FFD). Using the sequential experiment strategies (i.e., the fractional factorial design), the factors and interactions for the EP process with the dissolved thickness and the roughness criterium (Ra) varying respectively from ca. 10.5 to 24.3 μm and 0.06 to 0.22 μm for AISI 4340 and from ca. 10.1 to 24.7 μm and 0.04 to 0.19 μm for 300M steels are clearly demonstrated. The dissolved thickness and roughness criterium (arithmetic average of the absolute values (Ra)) are described using fitted models. Model validation using ANOVA analyses, check point-tests confirmed that the results are reliable and accurate. The predicted values obtained with models are in close agreement with the experimental data. As expected, these valuable results show that, dissolved thickness and Ra strongly depend on the polarization conditions (I and Q) effects for the two ferrous metals even if Iron ions concentration also affects the EP behaviors of steels. For all responses, the positive coefficients of X_1 (Q), X_2 (I), and X_1X_2 indicate a constructive contribution and the negative coefficient of X_3 (Iron ions concentration), an antagonistic effect. Temperature (X_4) has no effect in the domain studied.

The contour plots for the dependence of thickness and Ra on factors X_1 (electrical charge) and X_2 (current density) while keeping X_3 (Iron ions concentration) constant were constructed by using the regression model for each substrate. This allowed us to define a compromise zone where all the experimental responses fulfill the specifications imposed by the researchers to achieve the aims proposed, i.e. a dissolved thickness comprised between 15–17 μm and arithmetic roughness criteria less than 0.06 μm.

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