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THE EFFECTS OF PASSIVATION PARAMETERS ON PITTING POTENTIAL OF BIOMEDICAL STAINLESS STEEL

Article Highlights

- Corrosion resistance of 316LVM stainless steel was increased by passivation
- Multiple regression analysis and artificial neural network (ANN) were employed
- Only the ANN model provided a statistically accurate mathematical model
- Pitting potential is highly non-linearly dependent on the passivation parameters
- Nitric acid concentration has the strongest influence on the pitting potential

Abstract

Passivation is a chemical process in which the electrochemical condition of passivity is gained on the surface of metal alloys. Biomedical AISI 316LVM stainless steel (SS) can be passivized by means of nitric acid immersion in order to improve a protective oxide layer on the surface and consequently increase corrosion resistance of the SS in the physiological solutions. In this study, multiple regression analysis and artificial neural network (ANN) were employed for mathematical modeling of the AISI 316LVM SS passivation process after immersion in the nitric acid solution. The pitting potential, which represents the measure of pitting corrosion resistance, was chosen as the response, while the passivation parameters were nitric acid concentration, temperature and passivation time. The comparison between experimental results and models predictions showed that only the ANN model provided statistically accurate predictions with a high coefficient of determination and a low mean relative error. Finally, based on the derived ANN equation, the effects of the passivation parameters on pitting potential were examined.

Keywords: stainless steel, nitric acid, passivation, multiple regression analysis, artificial neural networks.

AISI 316LVM is a vacuum melted stainless steel (SS) widely used for biomedical applications. It has high tensile strength and fatigue resistance, good deformability, and relatively low price. Examples of its biomedical applications include bone plates and screws, hip and knee prosthesis, nails and pins, dental prostheses as well as vascular and urological stents [1]. The main disadvantages of this steel are local corrosion susceptibility during prolonged contact with human tissue, and release of metal ions [2]. Additionally, nickel is known as a strong immunological reaction medium and may cause various health problems [3]. Despite the above listed weaknesses, SS has the ability to spontaneously form a stable self-protecting oxide layer (passive film) on its surface in the reaction with air or most aqueous environments. This film consists mostly of chromium oxide (Cr_2O_3) and typically shows thicknesses of few nanometers [4]. The presence of nonmetallic inclusions on the material's surface, such as sulfide inclusions, represents a discontinuity of the passive film and therefore a potential place of pitting corrosion initiation [5].

Localized corrosion may cause an accidental deterioration of the whole system with disastrous consequences, while the total mass loss is insignificant [6]. Corrosion of SS implants have two effects [7]: first, the implant may become weak and the premature failure of the implant may happen; and sec-

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ond, the release of corrosion products from the implant can cause the tissue reaction.

Corrosion pits commonly start because of chemical or physical heterogeneities at the surface, which include dislocations, mechanical damage, inclusions, or second phase particles [8]. The resistance of SS to a pitting attack depends largely on the type of SS used, and on the subsequent physicochemical properties of the protective passive oxide layer formed on its surface [9]. There has been a constant attempt by engineers and scientists to improve the surface-related properties of biomedical materials to reduce the failure of implants and leaching of ions due to wear and corrosion. A number of research groups have done extensive research on the improvement of both general and pitting corrosion resistance of SS by developing techniques for the modification of the material's surface and passive film. Further, pitting attack resistance directly depends on the physicochemical properties of the protective passive oxide layer formed on the surface [10].

A beneficial effect of nitric acid solution on chromium enrichment in the modified passive layer of SS was reported in the literature [11-14]. Immersion in nitric acid solutions is particularly effective in improving the pitting resistance of austenitic SS [15]. Also, immersion in nitric acid removes sulphide inclusions, eliminating the preferential sites for attack [16].

Mathematical modeling of the passivation process based on the scientific principles allows one to study and better understand this complex process. Multiple regression analysis (MRA) and ANNs are two important competitive data mining techniques widely used for the development of predictive mathematical models [17]. MRA is a conceptually simple method for development of the functional relationships between several independent (input) variables and one dependent (output). ANNs are a computational tool, based on the properties of biological neural systems, which have been used successfully where conventional computer systems have traditionally been slow and inefficient. Both methodologies can be successfully applied for different process modeling. However, when compared to one another, different conclusions can be drawn in certain cases [18].

In the literature there are few studies which are aimed at modeling the passivation process of biomedical material in nitric acid as well as in other fluids in general. Masmoudi *et al.* [19] studied the passivation process of CP Ti (commercially pure titanium) and Ti6Al4V alloy by immersing in HNO_3 solution. Their main aim was to improve corrosion resistance of tested materials after acid treatment. Mathematical models were obtained by employing MRA, while optimization was performed by applying the least square method. Jiménez-Come *et al.* [20] presented an automatic model based on artificial intelligence techniques to predict pitting potential values. Their model was aimed to compare pitting corrosion resistance of AISI 316L austenitic SS in different environmental conditions without requiring the use of electrochemical tests. They showed that the presented model provides an automatic way to compare the pitting corrosion resistance of austenitic stainless steel in different environmental conditions. Petković *et al.* [13] analyzed the possibilities for enhancing corrosion resistance of biomedical AISI 316LVM SS by immersing in nitric acid solutions under different passivation conditions. Namely, the effects of nitric acid solution concentration, temperature and passivation time on the pitting potential, which was selected as a parameter for corrosion resistance assessment, were investigated. A total of 27 experimental trials were carried out according to 3^3 full experimental design. A mathematical model was determined by using MRA, while optimal values for passivation parameters were found by means of genetic algorithm.

We decided to broaden the previous research in order to obtain more precise and accurate experimental results by repeating the experiment twice more and by applying ANNs for the purpose of mathematical modeling. Thus, in this paper, the pitting potential value, as the response, was calculated as a mean of the three pitting potential values measured for all 27 experiment trials (test). Moreover, three additional measurements were carried out in order to determine the pitting potential for non-passivized sample (control). Hence, there were a total of 84 experiment trials. Compared to our previous study, the application of the experiment designs with replications increases its reliability significantly.

The aim of this study was to develop a mathematical model relating passivation parameters with pitting potential as the response. To evaluate the best possible mathematical model, a statistical analysis of the results was performed. Based on the conducted statistical analysis, one can argue that MRA is not able, on a satisfactory level, to accurately model the underlying relationships between passivation parameters and pitting potential. For this reason, a mathematical model of the passivation process was developed by using ANN in combination with a 3^3 full factorial design with three replications. Finally, the ANN model was compared with the MRA model to assess the adequate methodology for further modeling of the similar processes.

EXPERIMENTAL

Biomedical SS

For this research, 81 test samples (3 per each of 27 experimental trials) and 3 control samples were machined. The samples were cylindrical with the diameter of 6 mm and height of 20 mm made of AISI 316LVM SS, containing Cr, Ni, Mo and Mn as main alloy elements. Chemical composition of tested AISI 316LVM SS is in accordance to ISO 5832-1 [21].

Passivation process

Three input variables (X_1: HNO_3 concentration, X_2: temperature of passivation solution, and X_3: passivation time) were selected as passivation parameters. The 3^3 full factorial design with three replications was used. Real and coded values of the parameters and their levels used in the experimentation are given in previous published paper [13].

Prior to each test, the exposed surface of the samples was wet ground with silicon carbide paper up to 1200 grit and polished by using diamond paste with grain size of up to 0.25 μm. Then, the samples were rinsed with distilled water and washed with ethanol in an ultrasonic cleaner. The passivation treatment was performed by immersing samples in nitric acid solutions. Lastly, the samples were rinsed in double distilled water and alcohol, respectively.

Electrochemical measurements

Electrochemical tests for each sample were performed using a three-compartment cylindrical glass cell equipped with a saturated calomel electrode (SCE) as the reference electrode and a platinum foil as the counter electrode. The average of pitting potentials for three samples with the same treatment was chosen as a measure of corrosion resistance. The specimens were immersed 15 s before the start of the potential rise and this time was set by the program. The starting potential was −400 mV with a scan rate of 0.25 mV/s to anodic potential direction. The tests were finished when the current density reached about 0.2 mA/cm^2. The pitting potential (E_p) was chosen as a measure of corrosion resistance and represented a level of potential when the passive film broke down [22].

The electrochemical tests were conducted in Hank's solution, which is a simulated body fluid and most frequently used for *in vitro* tests. During the experiments, the temperature was maintained at 37±1 °C (typical body temperature). The composition and instruction for preparation of the Hank's solution are described elsewhere [23].

Mathematical models

MRA model

In this study, a second order polynomial was selected for mathematical modeling of pitting potential depending on the passivation parameters forms, as follows:

$$E_p = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + \\ + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \quad (1)$$

where E_p is the pitting potential (output), X_j are coded values of the parameters (input), b_0 is the model constant, b_j is the first degree coefficient, b_{jk} are the cross-products coefficients and b_{jj} are the quadratic coefficients.

The regression coefficients, b_0, b_j, b_{jk} and b_{jj}, were estimated by the least squares method. Values of regression coefficients and their statistical significance were determined by using Minitab 15 statistical software package.

ANN model

Three neurons in the input layer (for each of the passivation parameters), one neuron at the output layer for pitting potential, and only one hidden layer were used to define the ANN architecture [18,24]. The number of hidden neurons was selected by considering the following: *i*) too few neurons in the hidden layer can lead to under-fitting, *i.e.*, inability to perform appropriate function approximation, whereas too many neurons can contribute to over-fitting [25], which results in a lack of generalization capability of the developed model; *ii*) the more hidden neurons, the more expressive power of the ANN - however, with the increase of the number of hidden neurons, the number of unknown parameters (weights and biases) to be estimated also increases; (*ii*) the upper limit of the number of hidden neurons can be determined considering that the total number of unknown parameters does not exceed the number of available data for training process. As noted by Sha and Edwards [26], although in the case where the number of the connections to be fitted is larger than the number of available data for training, ANN can still be trained, the case is mathematically undetermined. Therefore, relatively small ANN architecture 3-5-1 was selected to model this passivation process.

Since it was assumed that a nonlinear relationship exists between the passivation parameters and pitting potential, the hyperbolic tangent sigmoid transfer (activation) function was used in the hidden layer, and linear transfer function was used in the output layer. According to the selected transfer functions in the input and output layer, all experimental data were normalized in the [−1,1] range. The goal of the ANN training process is to determine (near) optimal values of weights and biases in the hidden and output layer, previously initialized by the Nguyen-Widrow algorithm, in order to minimize the mean squared error between ANN predictions and experimental data. The ANN was trained with gradient descent with momentum by using 23 out of 27 sets of input/output experimental data and the rest was used for testing the ANN's generalization performance capability. Learning rate (η) and momentum (μ) were kept at 0.1 and 0.9, respectively. The training process was finished after 7800 epochs, with the minimal achieved mean squared error of 0.00548.

Statistical evaluation of developed models

Coefficient of determination R^2 was used to evaluate the performance of the developed models, and indicate how well mathematical models fitted experimental data [24]. In addition, for the estimation of the prediction performance of the developed mathematical models, relative error, as one of the most stringent criteria, was calculated by using the following equation:

$$\text{Relative error} = \\ = 100\frac{\left|\text{Experimental value} - \text{Predicted value}\right|}{\text{Experimental value}} \quad (2)$$

The mean relative error (*MRE*) was also calculated.

RESULTS AND DISCUSSION

The results of modeling the passivation process by using *MRA* and ANN are displayed and compared in this section. In addition, the results are discussed and analyzed.

The second order MRA model (full quadratic regression model with interactions), relating passivation parameters and the pitting potential, was obtained as:

$$E_\mathrm{p} = 1.50 + 0.086X_1 + 0.0014X_2 + 0.0334X_3 + \\ +0.077X_1X_2 - 0.056X_2X_3 - 0.227X_1^2 - 0.130X_2^2 \quad (3)$$

Based on the Table 1, it should be noted that insignificant model terms X_1X_3 and X_3^2 were eliminated since they were highly correlated with other variables.

The R^2 value indicates that the passivation parameters explain 53.7 % of variance in pitting corrosion potential. Apart from that, adjusted coefficient of determination and predicted coefficient of determination (given in Table 1) are considerably smaller indicating that the model is inadequate and over-fitted. Consequently, the MRA model is not reliable enough to describe the investigated relationship. Moreover, analysis of variance (ANOVA), shown in Table 2, reveals that F-ratio of 2.19 corresponds to confidence level of 92.2%, which is lower than standard (95 or 99%).

Based on the conducted statistical analysis, one can argue that MRA model is not able, on a satisfactory level, to accurately model the underlying relationships between passivation parameters and pitting potential. For these reasons, modeling of the passivation process was attempted by means of ANN.

By considering the data normalization, transfer functions used in the hidden and output layer, and by using the weights and biases from Table 3, one can obtain a mathematical equation for pitting potential calculation. After denormalization, the mathematical model for pitting potential in terms of the passivation parameters can be expressed by the following equation:

$$E_\mathrm{p} = 0.36\left(\left(\left[\frac{2}{1+e^{-2(X\cdot W_1 + B_1)}} - 1\right]W_2 + B_2\right) + 1\right) + 0.77 \quad (4)$$

Table 1. Regression coefficients of the MRA model; S = 0.174801; R^2 = 53.7%; R^2(adj.) = 29.2%; R^2(pred.) = 0.0%

Coefficient	Calculated coefficient value	*SE* Coefficient	*T*	Probability density *P*
b_0	1.5003	0.9300	16.13	0.000
b_1	0.0856	0.0412	2.08	0.053
b_2	0.0014	0.0414	0.03	0.973
b_3	0.0334	0.0414	0.81	0.431
b_{12}	0.0769	0.0498	1.54	0.141
b_{13}	-0.0297	0.0498	-0.60	0.559
b_{23}	-0.0558	0.0505	-1.11	0.284
b_{11}	-0.2269	0.0781	-2.91	0.010
b_{22}	-0.1300	0.0714	-1.82	0.086
b_{33}	-0.0517	0.0714	-0.72	0.479

Table 2. ANOVA results for the MRA model;DF - degree of freedom; SS - sum of squares; MS - mean square; F - value of Fisher's distribution; P - probability density

Source	*DF*	*SS*	*MS*	*F*	*P*
Regression	9	0.60322	0.06702	2.19	0.078
Residual Error	17	0.51944	0.03056		
Total	26	1.12267			

where X is a column vector that contains the normalized values of HNO_3 concentration, temperature of passivation solution and passivation time. Coefficient of determination R^2 for this model reveals that the passivation parameters explain 81% of variance in pitting corrosion potential, suggesting that the model has a good fit. Therefore, the obtained ANN model is better and more reliable than the MRA model. Details related to the ANN model are given in Table 3.

Comparison of the models

In order to compare the models as well as effectiveness of the passivation process, the measured and predicted values of the pitting potential for all experimental trials are listed in Table 4. Apart from the results, relative errors of the models were calculated as well as standard deviation for measured data. Moreover, the measured pitting potentials for the control sample are shown in Table 4. Maximal effect of passivation was measured for passivation condition in 12th experimental trial.

At first, positive effect of the passivation on the corrosion resistance is obvious according to measured pitting potential values. Standard deviation of measured pitting potentials is about of 5% indicating high reliability of the measured values. Then, taking into consideration the coefficient of determination for both models one can notice significantly higher value for the ANN model. Additionally, MRE shows better prediction performance of the ANN model since it produces two times less MRE than the MRA model. In other words, ANN model is more suitable for the analysis process with a large non-linearity such as SS passivation. Therefore, the influence of the passivation parameters on the corrosion resistance of the SS is considered by using the ANN model only.

Effects of passivation parameters on pitting potential

The first part of the analysis is concerned with the analysis of main effects of passivation parameters on pitting potential. To this aim, Eq. (4) was plotted by changing one passivation parameter at a time, while keeping the other two constant at the center level (Figure 1).

It is evident that the mathematical relationships, presented graphically in Figure 1, are highly non-linear. Quantitatively, based on the analysis of the main effects, concentration is the most influential parameter, followed by temperature and passivation time as less influential, respectively. While passivation temperature and time are on central level, the highest corrosion resistance is achieved when the concentration is about 20%. For central levels of concentration and passivation time the highest corrosion resistance is achieved when the temperature is slightly lower than 30 °C. Finally, when the concentration and temperature are set on the central level, the highest corrosion resistance is achieved when the duration of the process is about 25 min.

In order to determine the interaction effects of the passivation parameters on the pitting potential, 3-D surface plots were generated considering two parameters at a time, while the third one was kept constant at the center level (Figure 2).

From Figure 2 it can be observed that the pitting potential is highly sensitive to the selected passivation parameters. It is also obvious that the effects of the parameter are variable depending on their own level, since there are significant interaction effects of passivation parameters on the pitting potential. The functional dependence between the pitting potential and the passivation parameters is strongly nonlinear, therefore the effect of a given parameter on the pitting potential must be considered through the interaction with the other parameters.

For instance, if the passivation time is set on the central level (40 min), Figure 2a, and temperature on the low level, an increase in concentration of the solution leads firstly to the pitting potential increase up to some extreme value, which corresponds with the middle level of the concentration. Further increase in concentration leads to the reduction of the pitting potential. In this case, the pitting potential is very low when the concentration is on the high level, while the middle one provides a fairly high pitting potential. When the temperature is set on the high level, with an increase in concentration from the low level the pitting potential firstly decreases, then increases, and the nearby high level starts to impair.

When the temperature is set on the middle level (Figure 2b) and concentration is on the low level, an

Table 3. The weights and biases of the developed ANN mode; W_1: weights between input and hidden layer; W_2: weights between hidden and output layer; B_1: biases of the hidden neurons; B_2: bias of the output neuron

W_1			W_2	B_1	B_2
-0.26021	-2.5744	-0.78867	0.36462	2.1845	-1.2035
-1.2355	-2.2487	-1.317	-1.1196	1.3122	
1.4764	1.5512	0.95885	-1.1753	-0.27368	
-1.9939	0.97443	0.30408	-1.0401	-2.539	
-1	-1.6398	-1.4029	-1.0435	-2.4896	

Table 4. Comparative review of the measured pitting potential and predicted values for the pitting potential by means of MRA and ANN models; important remark: shaded rows - testing data for ANN model performance

Exp. trial	Passivation parameters			Experimental		MRA Model		ANN Model	
	HNO_3 concentration, %	Temperature, °C	Passivation time, min	E_p, V	Standard deviation, %	E_p, V	Relative error, %	E_p, V	Relative error, %
Control	-	-	-	0.68	4.04	-	-	-	-
1	10	17	20	0.85	4.51	1.08	26.89	0.86	1.15
2	10	17	40	1.16	5.13	1.13	2.21	1.13	2.49
3	10	17	60	1.19	6.11	1.19	0.01	1.47	23.85
4	10	40	20	1.12	4.16	1.19	6.03	1.14	1.37
5	10	40	40	1.44	5.51	1.19	17.53	1.35	6.14
6	10	40	60	1.18	4.58	1.19	0.64	1.22	3.38
7	10	60	20	1.08	3.06	1.04	4.03	1.05	2.49
8	10	60	40	0.77	2.52	0.98	27.35	0.87	12.79
9	10	60	60	0.81	5.20	0.92	14.17	0.78	3.48
10	30	17	20	1.0	4.93	1.29	29.49	1.03	3.00
11	30	17	40	1.39	4.04	1.35	2.83	1.46	5.08
12	30	17	60	1.49	5.03	1.41	5.60	1.47	1.32
13	30	40	20	1.41	3.21	1.46	3.53	1.46	3.27
14	30	40	40	1.47	5.14	1.46	0.70	1.33	9.56
15	30	40	60	1.39	3.04	1.46	5.02	1.30	6.16
16	30	60	20	1.16	5.77	1.36	17.63	1.20	3.37
17	30	60	40	1.41	2.52	1.31	7.18	1.30	7.79
18	30	60	60	1.33	4.62	1.25	5.79	1.26	5.02
19	65	17	20	1.28	4.20	1.10	14.37	1.32	3.35
20	65	17	40	1.29	5.00	1.15	10.71	1.31	1.26
21	65	17	60	0.94	3.55	1.21	28.47	1.04	10.44
22	65	40	20	1.13	2.89	1.36	20.24	1.11	1.94
23	65	40	40	1.14	5.77	1.36	19.18	1.19	4.20
24	65	40	60	1.43	4.93	1.36	4.99	1.48	3.64
25	65	60	20	1.42	3.79	1.36	4.13	1.34	5.40
26	65	60	40	1.17	3.21	1.31	11.59	1.34	14.89
27	65	60	60	1.34	4.15	1.25	6.73	1.31	2.38
Mean relative error (*MRE*)							11.00		5.53

increase in passivation time leads firstly to the pitting potential increase up to some extreme value and then decrease. In this case, the pitting potential is very low when the passivation time is on the low and high level, while the middle one provides pretty high pitting potential. When the concentration is set on the high level, with increasing passivation time from the low level the pitting potential firstly increases rashly, then slightly decreases up to about 30 min, and then starts to grow again up to the high level.

Based on Figure 2a and b, it can be concluded that the highest pitting potentials correspond with a combination of parameters concentration-temperature and concentration-passivation time slightly below the

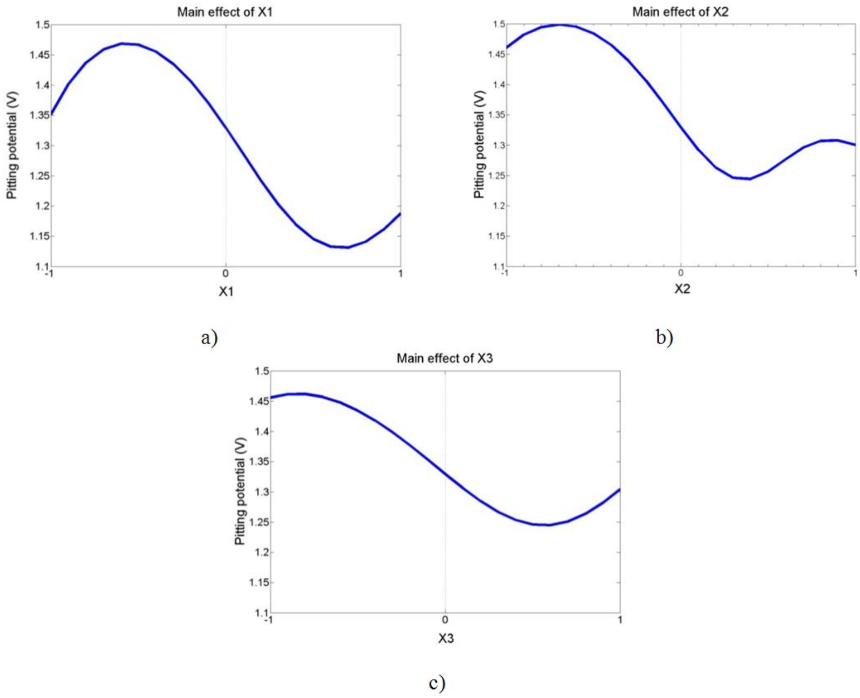

Figure 1. Main effects of passivation parameters on pitting potential.

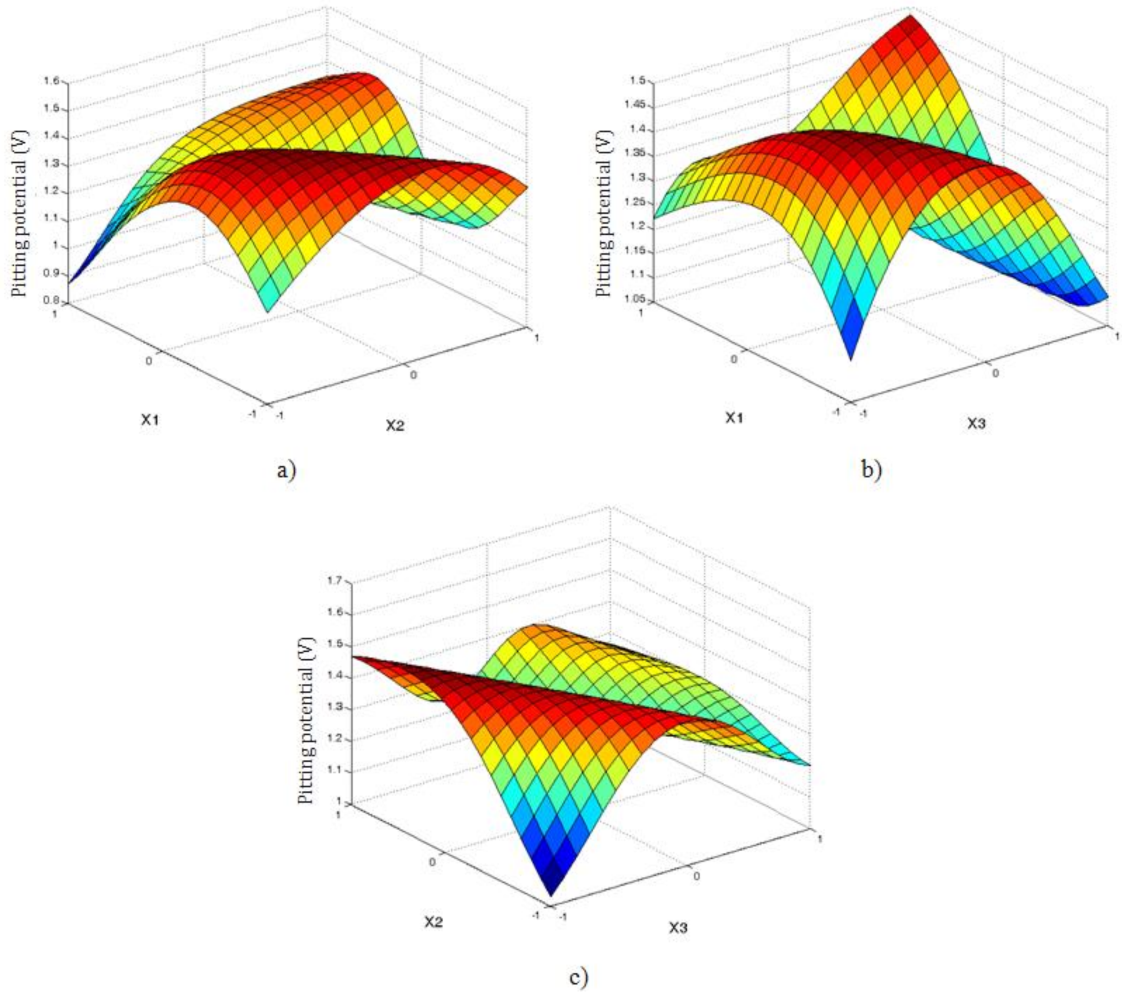

Figure 2. Interaction effects of passivation parameters on pitting potential.

middle levels. As it can be seen in Figure 2c (where concentration is on the middle level), the lowest pitting potential predicted for temperature of 17 °C and passivation time 20 min; the highest pitting potentials is predicted for the temperature and passivation time slightly below the middle level.

CONCLUSION

Passivation by immersing in nitric acid solution is an effective method to improve corrosion resistance of biomedical SS. In biomedical SS passivation process, MRA and ANN were introduced to model functional relationship between pitting potential and passivation parameters such as nitric acid concentration, temperature and passivation time. A non-linear functional dependence between the passivation parameters and the pitting potential was determined. Hence, the ANN model proved to be more suitable for modeling the processes such as chemical passivation of SS. Nitric acid concentration has maximum influence on the pitting potential followed by the temperature and passivation time. The best experimental result was achieved by a combination of parameters: HNO_3 concentration – 30%; temperature – 17 °C; passivation time – 60 min.

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NAUČNI RAD

UTICAJ PARAMETARA PASIVIZACIJE NA PITING POTENCIJAL BIOMEDICINSKOG NERĐAJUĆEG ČELIKA

Pasivizacija predstavlja hemijski process u kome se površina metalnih legura dovodi u stanje elektrohemijske pasivnosti. Biomedicinski nerđajući čelik AISI 316LVM može se pasivizirati potapanjem u azotnu kiselinu, jer se time poboljšava zaštitini oksidni sloj;na taj način se povećava i koroziona postojanost ovog materijala u fiziološkim rastvorima. U ovom istraživanju, za matematičko modeliranje procesa pasivizacije korišćene su višestruka regresiona analiza i veštačke neuronske mreže. Za izlazni (zavisni) parametar modela izabran je piting potencijal, koji predstavlja meru korozione postojanosti. Kao parametri pasivizacije razmatrani su: koncentracija azotne kiseline, temperatura i vreme pasivizacije. Upoređeni su eksperimentalni rezultati i rezultati modela. Pokazalo se, da jedino model dobijen pomoću veštačkih neuronskih mreža ima statistički zadovoljavajuću tačnost predikcije. Na kraju, na osnovu modela dobijenog pomoću veštačkih neuronskih mreža, izvedena je analiza uticaja parametara pasivizacije na piting potencijal biomedicinskog nerđajućeg čelika.

Ključne reči: nerđajući čelik, azotna kiselina, pasivizacija, višestruka regresiona analiza, veštačke neuronske mreže.