Pulsed nanocrystalline plasma electrolytic carburising for corrosion protection of a $\gamma$-TiAl alloy
Part 2. Constant frequency and duty cycle

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Abstract

A number of studies have been reported on the use of plasma electrolytic carburising technology for surface hardening of different metals for higher corrosion and wear resistance resulted from this technique. However, very few have focused on the optimization of the pulsed nanocrystalline plasma electrolytic carburising process parameters. In this study, a design of experiment (DOE) technique, the Taguchi method, has been used to optimize the pulsed nanocrystalline plasma electrolytic carburising, not only for surface hardening but also for the corrosion protection of Ti-48Al-2Cr-2Nb (at%) titanium alloy by controlling the coating process’s factors. The experimental design consisted of four factors (glycerol concentration, electrical conductivity of electrolyte, applied voltage and duration of process), each containing three levels. All experiments were done under constant frequency and duty cycle of rectangular shape pulse current method. Pure aluminum and titanium carbides were detected by XRD on the surface of the treated samples. Tafel polarization measurements were carried out to determine the corrosion resistance of the coated samples. The results were analyzed with related software. An analysis of the mean of signal-to-noise (S/N) ratio indicated that the corrosion resistance of pulsed plasma electrolytic carburized Ti-48Al-2Cr-2Nb (at%) alloy was influenced significantly by the levels in the Taguchi orthogonal array. The optimized coating parameters for corrosion resistance are 1200 g/l for glycerol concentration, 360 mS/cm for electrical conductivity of electrolyte, 400 V for applied voltage, 30 min for treatment time. The percentage of contribution for each factor was determined by the analysis of variance (ANOVA). The results showed that the applied voltage is the most significant factor affecting the corrosion resistance of the coatings.

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Keywords: Pulsed nanocrystalline plasma electrolytic carburising; $\gamma$-TiAl alloy; Tafel polarization; Analysis of variance

1. Introduction

Titanium and its alloys are used in orthopedic applications owing to their favorable mechanical properties and their excellent corrosion resistance. It has been reported that the metal ions, particularly titanium and aluminum released into the surrounding tissues, have toxic effects [1,2]. High corrosion resistance, excellent wear and friction resistance are desired because the properties must be similar to those of natural bone. To improve the wear resistance of titanium and its alloys, they are subjected to various surface treatments such as thermal spraying, plasma vapour deposition processes, anodic oxidation, ion implantation, glow discharge nitriding and laser treatment [3–5]. In the present study, a relatively new method of applying penetrative coatings on Ti-48Al-2Cr-2Nb (at%) substrate, named pulsed plasma electrolysis, was used [6,7]. Among different processes for carburising of metals, this method shows great potential, not only for not decreasing the corrosion resistance of samples but also for increasing it by controlling the effective factors on coating process. However, there is almost no information in the open literature on the relative significance of the parameters in the pulsed nanocrystalline plasma electrolytic carburising (PNPEC) process on the corrosion resistance of Ti-48Al-2Cr-2Nb (at%). A traditional or classical optimization approach would be to design experiments that identify all possible combinations for a given set of variables. This approach is called the full factorial design, and involves a large number of experiments, which can be costly and time consuming.
In the last five decades, the Taguchi method has been effectively applied to improve product quality and manufacturing efficiency [8,9]. The Taguchi method uses a special design of orthogonal arrays to study all the designed factors with a minimum of experiments at a relatively low cost. Orthogonality means that factors can be evaluated independently of one another; the effect of one factor does not interfere with the estimation of the influence of another factor [9]. In this study, the Taguchi method for design of experiment was used for the optimization of the PNPEC process for the production of coatings for the corrosion protection of Ti-48Al-2Cr-Nb (at%). The previous studies [10,11] for oxidation with same technique have demonstrated that the composition, thickness, and morphology (or porosity) of coatings are primarily determined by several key process parameters such as treatment time, current density, and the concentrations of potassium hydroxide and sodium aluminate solutions. The design of experiment took into account the influencing extent of individual process parameter. This consideration led to the selection of four influential factors, i.e., glycerol concentration, electrical conductivity of electrolyte, applied voltage and treatment time with three different levels (1–3). The frequency and duty cycle of pulsed current as effective factors were also investigated, but the results were revealed in another paper and in this study these factors are constant in their relatively optimum level. The previous studies [10,11] for oxidation with same technique have demonstrated that the composition, thickness, and morphology (or porosity) of coatings are primarily determined by several key process parameters such as treatment time, current density, and the concentrations of potassium hydroxide and sodium aluminate solutions. The design of experiment took into account the influencing extent of individual process parameter. This consideration led to the selection of four influential factors, i.e., glycerol concentration, electrical conductivity of electrolyte, applied voltage and treatment time with three different levels (1–3). The frequency and duty cycle of pulsed current as effective factors were also investigated, but the results were revealed in another paper and in this study these factors are constant in their relatively optimum level of 100 Hz for frequency and 40% for duty cycle. Tafel polarization measurements were conducted to determine the corrosion resistance of the coated and uncoated samples. The results of the factor response analysis were used to derive the optimal levels combinations. Confirmation experiments were conducted to verify the analytical results. The percentage contribution of each factor was determined by an analysis of variance.

### 2. Experimental

Cylinder shape samples (20 mm diameter × 5 mm) of purchased Ti-48Al-2Cr-2Nb (at%) (Quantometric results: Al 33.5 wt.%, Cr 2.55 wt.%, Nb 4.67 wt.%, Ti balance) (hereafter referred to as γ-TiAl) were used as substrates in this study. All samples were prepared from a bar by usual machining. After polishing to about R<sub>a</sub> 2 μm using an Al₂O₃ slurry, the samples were first cleaned with acetone and then ultrasonically cleaned in ethanol. Electrolytes were prepared from solutions of glycerol (C₃H₈O₃) in distilled water with an addition of natrium hydroxide (NaOH). During carburising, the samples to be coated as cathode were connected to a 20 kW DC power source (600 V, 34 A(max)). Frequency and duty cycle of pulse current were maintained constant at 100 Hz and 5%, respectively, during the coating process. The concentration of natrium hydroxide was changed to adjust the electrical conductivity of electrolyte to its optimum setting. The details of the PNPEC setup used in this study can be found in Refs. [12–14]. The “quality characteristic” of concern is the corrosion resistance of the coatings that was assessed by Tafel polarization tests carried out at 298 K using an EG&G 273A potentiostat with 352 SoftCorrIII software and its PAR calculations. A three electrode cell with the coated samples as a working electrode, saturated calomel electrode as a reference electrode, and a platinum plate as a counter electrode was used in the tests. The ratio of the volume of Ringer solution/sample area was 200 ml/cm². After the electrochemical testing system became stable (about 30 min), scans were conducted at a rate of 0.5 mV/s. The cross-section morphology and hardness profile of PNPEC films were examined by XL-30 (PHILIPS) scanning electron microscope (SEM) and Buhler Micromet1 micro hardness tester.

### Table 1

<table>
<thead>
<tr>
<th>Level</th>
<th>Factor</th>
<th>Glycerol concentration (g/l)</th>
<th>Electrical conductivity (mS/cm)</th>
<th>Applied voltage (V)</th>
<th>Time (min)</th>
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<td>3</td>
<td></td>
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<td>360</td>
<td>400</td>
<td>30</td>
</tr>
</tbody>
</table>

### 3. Taguchi design of experiment

#### 3.1. Design of orthogonal array and signal-to-noise analysis

Four factors (glycerol concentration, electrical conductivity of electrolyte, applied voltage and duration of process) with three levels were selected and were shown in Table 1. The maximum and minimum levels of selected factors were chosen due to considerations of the process. For example, the concentration of glycerol is better to be as saturate as possible for presenting the maximum amount of carbon in electrolyte. On the other hand, its concentration has direct effect on the amount of water in electrolyte, which can affect the cathodic process by relative reactions. The factors and levels were used to design an orthogonal array L₉ (3⁴) for experimentation. Nine Taguchi experiments were conducted twice to ensure the reliability of experimental data for a signal-to-noise analysis. In process design, it is almost impossible to eliminate all errors caused by the variation of characteristics. An increase in the variance of corrosion resistance lowers the quality reliability of coatings. To minimize the influence of corrosion resistance variation on the analysis of experimental data, the signal-to-noise (S/N) ratio was employed, which converts the trial result data into a value for the response to evaluate coating quality in the optimum setting analysis. The S/N ratio consolidates several repetitions into one value, which reflects the amount of variation present. This is because the S/N ratio can reflect both the average and the variation of the quality characteristics. There are several S/N ratios available depending on the types of characteristics [9]: lower is best (LB), nominal is best (NB), and higher is best (HB). In the present study, corrosion resistances are treated as a characteristic value. Since the corrosion resistance of coatings intended to be maximized, the S/N ratio for HB characteristics was selected, which can be calculated as follows:

\[
S/N_{HB} = -10 \log \left( \frac{1}{n} \sum_{i=1}^{n} \frac{1}{R_{pi}^2} \right),
\]

where \(n\) is the repetition number of each experiment under the same condition for design parameters, and \(R_{pi}\) is the polarization resistance of an individual measurement at the \(i\)th test. After calculating and plotting the mean S/N ratios at each level for various factors, the optimal level, that is the largest S/N ratio among all levels of the factors, can be determined.
3.2. Analysis of variance (ANOVA)

The analysis of variance (ANOVA) on the experimental results was performed to evaluate the source of variation during the plasma electrolytic carburising. Following the analysis, it is relatively easy to identify the effect order of factors on coatings and the contribution of factors to corrosion resistance of coatings. In this study, variation due to both the four factors and the possible error was taken into consideration. The ANOVA was established based on the sum of the square (SS), the degree of freedom (D), the variance (V), and the percentage of the contribution to the total variation (P). The five parameters symbols typically used in ANOVA [8] are described below:

1. Sum of squares (SS). $SS_p$ denotes the sum of squares of factors A, B, C, and D; $SS_e$ denotes the error sum of squares; $SS_T$ denotes the total sum of squares. The total sum of square $SS_T$ from S/N ratio can be calculated as:

$$SS_T = \sum_{i=1}^{m} \eta_i^2 - \frac{1}{m} \left( \sum_{i=1}^{m} \eta_i \right)^2,$$

where $m$ is the total number of the experiments, and $\eta_i$ is the S/N ratio at the $i$th test. The sum of squares from the tested factors, $SS_p$, can be calculated as:

$$SS_p = \sum_{j=1}^{p} \left( \frac{S_{\eta_j}}{t} \right)^2 - \frac{1}{m} \left( \sum_{i=1}^{m} \eta_i \right)^2$$

where $p$ represents one of the tested factors, $j$ the level number of this specific factor $p$, $t$ the repetition of each level of the factor $p$, and $S_{\eta_j}$ the sum of the S/N ratio involving this factor and level $j$.

2. Degree of freedom (D). $D$ denotes the number of independent variables. The degree of freedom for each factor ($D_p$) is the number of its levels minus one. The total degrees of freedom ($D_T$) is the number of total number of the result data points minus one, i.e., the total number of trials times number of repetition minus one. And the degree of freedom for the error ($D_e$) is the number of the total degrees of freedom minus the total degree of freedom for each factor.

3. Variance (V). Variance is defined as the sum of squares of each trial sum result involved in the factor, divided by the degrees of freedom of the factor:

$$V_P(\%) = \frac{SS_P}{D_P} \times 100$$

4. The corrected sum of squares ($SS'_P$). $SS'_P$ is defined as the sum of squares of factors minus the error variance times the degree of freedom of each factor:

$$SS'_P = SS_P - D_P V_P$$

5. Percentage of the contribution to the total variation (P). $P_p$ denotes the percentage of the total variance of each individual factor:

$$P_p(\%) = \frac{SS'_P}{SS_T} \times 100$$

4. Results and discussion

4.1. Corrosion resistance of coatings

The calculation of the corrosion resistance of samples is based on the corrosion potential, the corrosion current density, and the anodic/cathodic Tafel slopes ($\beta_a$ and $\beta_c$), which were derived from the measured polarization curves. Based on the approximately linear polarization at the corrosion potential ($E_{corr}$), the value of corrosion resistance ($R_p$) was determined from the relationship [15]:

$$R_p = \frac{\beta_a \beta_c}{2.3 i_{corr}(\beta_a + \beta_c)},$$

where $i_{corr}$ is the corrosion current density.

The results of the Tafel corrosion tests for the different coatings on $\gamma$-TiAl are given in Table 2, which also includes data for the substrate for the purpose of comparison. All coated samples have a higher corrosion resistance than the $\gamma$-TiAl substrate. This evidently shows that the coatings provide an enhanced corrosion protection. Also, all of the samples have the same coating composition as mention in the first part of this article [16]. Moreover, the extended treatment and diluted glycerol solution, as demonstrated in Experiment 3, produces a sound

<table>
<thead>
<tr>
<th>Experiment</th>
<th>$\beta_a$ (mV/decade)</th>
<th>$\beta_c$ (mV/decade)</th>
<th>$i_{corr}$ ($\muA/cm^2$)</th>
<th>$R_p$ (M$\Omega$cm$^2$)</th>
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<td></td>
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<td>Test 2</td>
<td>Test 1</td>
<td>Test 2</td>
</tr>
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<td>498.361</td>
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<td>90.278</td>
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<td>255.61</td>
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<td>285.713</td>
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coating, which significantly increases the corrosion resistance of γ-TiAl by around 2.3 times better than γ-TiAl substrate up to 202.95 MΩ cm².

4.2. Determination of optimal levels

Based on Eq. (1), two corrosion resistance measurements for each experiment were converted into one S/N ratio. Table 3 compares the calculated mean S/N ratios with the corrosion resistance data and level numbers. In the following discussion, the S/N ratios are employed as a response index to compare the corrosion resistance of different coatings instead of directly using the values of corrosion resistance. The response of each factor to its individual level was calculated by averaging the S/N ratios of all experiments at each level for each factor. The determined factor responses are summarized in Table 4.

Fig. 1 shows the response of the S/N ratio to glycerol concentration. It can be seen from Fig. 1 that the mean S/N ratio arises to the bottom of the curve as the glycerol concentration increases to 1150 g/l (level 2) from 1100 g/l (level 1). When the glycerol concentration keeps increasing to 1200 g/l (level 3), the mean S/N ratio bounces back upward and further reaches to the highest value. The observation on the response of the S/N ratio to glycerol concentration may be mainly due to the properties of coatings. Aliofkhazraei et al. [17] discussed the relationship among different factors, penetration depth and thickness of coatings for 316L austenitic stainless steel by same process. Also, it was observed that decreasing the glycerol concentration from concentration of 1150 g/l results in the generation of yellow sparks during PNPEC process that is harmful for coating process and leads to incomplete coating process because of surface oxidation. Also, further extending the glycerol concentration enables blue sparks to perform, which lead to dense layer formation and thus, high corrosion resistance.

Table 3
The S/N ratios

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Glycerol concentration (g/l) (A)</th>
<th>Electrical conductivity (mS/cm) (B)</th>
<th>Applied voltage (V) (C)</th>
<th>Time (min) (D)</th>
<th>Corrosion resistance (kΩ cm²) (R_p)</th>
<th>S/N ratio</th>
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<td>360</td>
<td>300</td>
<td>10</td>
<td>138.114</td>
<td>139.278</td>
</tr>
</tbody>
</table>

Fig. 1. Effect of glycerol concentration on mean S/N ratio.

Fig. 2. Effect of electrical conductivity on mean S/N ratio.
The response of the S/N ratio to electrical conductivity of electrolyte is shown in Fig. 2. The mean S/N ratio goes up by increasing the electrical conductivity of electrolyte. By increasing the electrical conductivity in a specific voltage, the current flowing into the sample will increase and more ions will penetrate on the surface of the sample. Hence, the coatings can perform better and the corrosion resistance will be increased. This implies that the coating thickness plays an important role in enhancing the corrosion resistance of coatings. Also, it is worthwhile mentioning that however, the response of the S/N ratio to electrical conductivity of electrolyte increases continuously by increasing electrical conductivity of electrolyte and it didn’t show any optimum amount, but the electrical conductivity of electrolyte can’t be more than the levels in this study. By increasing electrical conductivity of electrolyte more than these levels, the required current for coating process will increase and it not only leads to local melting of the sample’s surface but also is not appropriate for industrial applications from economical point of view.

Fig. 3 shows response of the S/N ratio to the applied voltage during surface hardening. The mean S/N ratio increases with increasing applied voltage during coating process. Applied voltage on this process is the most important factor to identify the sample’s temperature. It has been mentioned that by increasing the applied voltage from 230 to 250 V the sample’s temperature will increase from 300 to 600 °C [6]. In this investigation, all of the applied voltages are high enough to produce compound layer on the surface of the sample. It is necessary to say that in this method at high applied voltages and thus, high surface temperatures, the effect of porosity of compound layer that usually inversely affects corrosion behavior, will disappear by thick carbide layer on the surface and the corrosion resistance of the samples treated by this method will not only decrease but also increase more than other methods of carburising. Hence, increasing the applied voltage of coating process will increase the corrosion resistance of coatings. The response of the S/N ratio to the treatment time, Fig. 4, is similar to that for the electrical conductivity and applied voltage. The mean S/N ratio increases linearly as the treatment time increases from 10 to 30 min. Treatment time has a direct effect on the thickness of compound carbide layer and therefore on the corrosion resistance of the samples, however, the thickness of this layer would not be increased after approximately 30 min of coating process. This fact is probably due to the formation of thick compound layer preventing the carbon species to penetrate toward the sample. Also, it must mention that in this process, the chosen applied voltages and treatment times are in the range of common values and choosing these effective factors out of these ranges will lead to a problem with coating process, such as local surface melting for high applied voltages, ununiformity of sparks around the sample for low applied voltages, insufficient thickness of compound layer for corrosion protection at low treatment times and not increasing of compound layer and increasing of required electricity for high treatment times.

The response of the S/N ratio to the glycerol concentration, electrical conductivity, applied voltage, and treatment time need to be further investigated. By selecting the highest value of mean S/N ratio for each factor, the optimal level can be determined. On this basis, the optimum combination of levels in terms of maximizing the corrosion resistance of the treated γ-TiAl is A3B3C3D3; i.e. 1200 g/l for glycerol concentration, 360 mS/cm for electrical conductivity of electrolyte, 400 V for applied voltage, and 30 min for treatment time.

Fig. 5. Tafel polarization curves of the substrate γ-TiAl and the optimal coating.
applied voltage and 30 min for treatment time. Also glycerol has an optimum concentration that leads to loose good corrosion resistance.

4.3. Factor contributions

The contribution of each factor to the corrosion resistance of coatings can be determined by performing analysis of variance based on Eqs. (2)–(6). The results of analysis of variance (ANOVA) are summarized in Table 5. The data given in Table 6 shows that the contribution of the four factors, i.e. glycerol concentration, electrical conductivity of electrolyte, applied voltage and treatment time is 0.2%, 0.72%, 81.64% and 17.44%, respectively. The contribution of applied voltage (81.64%) is more than the sum (18.36%) of the contributions of all the other three factors, i.e. glycerol concentration, electrical conductivity of electrolyte and treatment time. It is evident that, among the selected factors, applied voltage has the major influence on the formation of coatings for corrosion protection of this γ-TiAl. It can be seen that the treatment time is second important factor that affects corrosion resistance of the γ-TiAl. Furthermore, it can be assumed that Glycerol concentration and electrical conductivity of electrolyte have almost the same effect on corrosion resistances of coatings because of the minor difference in the contribution percentages among these two factors. It is evident from Table 6 that ANOVA analysis not only specifies how important a factor is to the corrosion resistance of coatings by numbers but also shows their relative effect. By ranking their relative contributions, the sequence of the four factors affecting the corrosion resistance is applied voltage, treatment time, electrical conductivity of electrolyte and glycerol concentration. It is also worthwhile mentioning that, in the ANOVA analysis, if the percentage error ($P_e$) contribution to the total variance is lower than 15%, no important factor is missing in the experimental design. In contrast, if the percent contribution of the error exceeds 50%, certain significant factors have been overlooked and the experiments must be re-designed [18]. As shown in Table 5, the percentage error ($P_e$) is 0%. This indicates that no significant factors are missing in the experimental design.

4.4. Confirmation run

The confirmation experiment is the final step in verifying the conclusions from the previous round of experimentation. If the results of the confirmation runs are not consistent with the expected conclusions, a new Taguchi method design is required. The confirmation experiment was performed by setting the experimental condition of the four factors as: 1200 g/l for glycerol concentration; 360 mS/cm for electrical conductivity of electrolyte; 400 V for applied voltage and 30 min for treatment time. Fig. 5 shows the polarization curves of the coated sample from the confirmation run and from the γ-TiAl substrate. It can
be seen from Fig. 5 that the corrosion potential of the optimized coating is more positive (−299 mV/SCE) than that of the γ-TiAl substrate (−632 mV/SCE), and the corrosion current density of the coating (0.158 A/cm²) is lower than that of the γ-TiAl substrate (0.68 A/cm²). Thus, the coating could be corroded only at a relatively higher potential and with a lower corrosion rate than the γ-TiAl substrate. Table 6 gives the detailed results from the confirmation run on the optimized coating as well as from the substrate. The corrosion resistance of the optimized coating is 202.953 MΩ cm², which is the highest value among other coatings obtained in the present study. Thus, the corrosion resistance of the optimized coating is more than 2.3 times higher than that of the substrate. The micrograph and hardness profile of the optimal coated can be seen in Figs. 6 and 7.

5. Conclusions

The Taguchi method for the design of experiment has been used for optimizing pulsed nanocrystalline plasma electrolytic carburising process parameters for the production of compound and penetrative coating for the corrosion protection of γ-TiAl after surface hardening. The applied voltage during coating process is found to be the major factor affecting the corrosion resistance of the coating. Also, the treatment time is found to be the second important factor affecting the corrosion resistance of the coating, while electrical conductivity of electrolyte and glycerol concentration have a similar and smaller effect on the corrosion resistance. The percentage contributions of the applied voltage, treatment time, electrical conductivity of electrolyte and glycerol concentration to the corrosion resistance are 81.64, 17.44, 0.72 and 0.2%, respectively. The contribution of applied voltage is more than the sum of the contributions of all the other three factors. It is evident that, among the selected factors, applied voltage has the major influence on the formation of coatings for corrosion protection of this γ-TiAl. It can be seen that the treatment time is second important factor that affects on corrosion resistance of the γ-TiAl. The optimized processing parameters are 1200 g/l for glycerol concentration, 360 mS/cm for electrical conductivity of electrolyte, 400 V for applied voltage and 30 min for treatment time. The corrosion resistance of the optimized coating is more than 2.3 times better than that of the γ-TiAl substrate.

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References