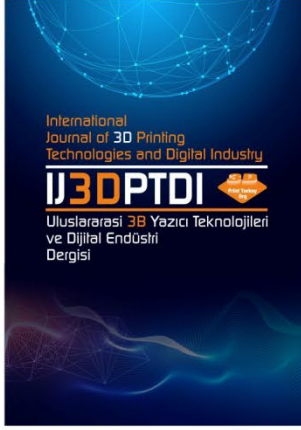




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Yazarlar (Authors): Hatice VAROL ÖZKAVAK , Hatice ASIL UĞURLU 

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CORROSION BEHAVIOR OF Ti-6Al-4V PRODUCED BY ADDITIVE MANUFACTURING METHOD

Hatice VAROL ÖZKAVAK^a , Hatice ASIL UĞURLU^b 

^aIsparta University of Applied Sciences, Technical Sciences Vocational School, Department of Mechanical and Metal Technologies, TÜRKİYE

^bIsparta University of Applied Sciences, Isparta OSB Vocational School, Department of Mechanical and Metal Technologies, TÜRKİYE

* Corresponding Author: haticevarol@isparta.edu.tr

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ABSTRACT

This study investigates the corrosion behavior of Ti-6Al-4V alloys manufactured by the DMLS method and subjected to shot peening using different coverage ratio. The aim of this study was to determine the effect of saturation ratio, a parameter of the shot peening process, on corrosion behavior. After producing Ti alloy by the Direct Metal Laser Sintering (DMLS) method, its microstructure was examined using SEM. Tafel and electrochemical impedance spectroscopy (EIS) tests were performed to analyze the corrosion resistance of the shot peening samples. Corrosion experiments were carried out in a 3.5% by weight NaCl solution. Corrosion potential (E_{corr}), corrosion current density (i_{corr}), and corrosion rate values were determined from Tafel polarization graphs. The sample with a 100% saturation ratio at a shot peening intensity of 12-14A (T100) showed the highest E_{corr} (-1.876 V) value and the lowest i_{corr} (4192 μA) value and corrosion rate (6.193mm/year). The corrosion resistance and capacitive properties of the samples were examined using Nyquist, Bode phase angle, and Bode impedance graphs obtained from the EIS analysis. EIS analyses showed that the best values were obtained from the sample with T100.

Keywords: Ti-6Al-4V, Corrosion, Shot peening, AM

1. INTRODUCTION

Due to their high specific strength, excellent biocompatibility, and corrosion resistance, titanium and its alloys are widely used in many fields, including the aerospace, medical, chemical, and marine industries [1-9]. Widespread use of Ti and its alloys is limited by high manufacturing costs [10]. The use of additive manufacturing (AM), a new production method, helps reduce the manufacturing costs of Ti and its alloys. In addition, it makes it possible to produce parts with complex geometries that are difficult or impossible to produce with traditional manufacturing methods [11-12]. Besides these advantages, material consumption and waste are reduced in the AM method because the material is produced by shaping the part layer by layer [10]. Ti-6Al-4V, a type of titanium alloy, possesses many superior properties such as a high strength-to-weight ratio, excellent

mechanical properties, and corrosion resistance [13]. In addition, due to its two-phase ($\alpha+\beta$) structure, Ti-6Al-4V alloys are a preferred material group for long-term use under extreme conditions [1-14]. Due to these properties, Ti-6Al-4V titanium alloy, preferred in many sectors such as aviation, marine, and biomedical, and the improvement of its properties, is an important research topic. However, it has been observed that in Ti-6Al-4V alloys where the AM manufacturing method is preferred for complex part manufacturing, significant differences are seen in material properties compared to those produced with conventional methods [15]. This situation is explained in the literature as being due to the conversion of β to γ in the structure during manufacturing in AM manufacturing methods such as Selective Laser Melting (SLM) and Direct Metal Laser Sintering (DMLS) [16-17]. Materials produced using the AM method

exhibit problems such as microstructural defects, porosity, residual stress, and decreased corrosion resistance [18]. This is supported by the literature, and in their study, Dai et al. examined the corrosion resistance of materials manufactured using conventional and SLM methods and determined that the conventional method had higher corrosion resistance [19]. Ti-6Al-4V titanium alloys are exposed to chloride ion-rich environments in their areas of use. Exposure to chloride ions causes surface degradation in these alloys. This degradation reduces their corrosion resistance, shortening their service life. Therefore, improving the corrosion resistance of both conventionally and artificially produced Ti-6Al-4V alloys is crucial. The corrosion resistance of Ti-6Al-4V materials is improved through surface treatments. For this purpose, techniques such as shot peening (SP) [20-21]; mechanical surface etching [22]; ultrasonic shot peening [23]; polishing [24-25]; and surface coating (spraying) [26-27]; are used. Problems arise in surface coating methods due to insufficient bonding between the coating and the substrate. Especially for improving the surface layer properties of Ti-6Al-4V materials produced by the AM method, peening methods such as shot peening (SP), impulse shot peening (ISP), laser shot peening (LSP), ultrasonic shot peening (USP), and cavitation shot peening (CP) are used [28-30]. When these peening methods are applied to Ti-6Al-4V alloys produced by the AM method, improvements in tribological wear resistance, fatigue resistance, and corrosion resistance occur. Avcu et al., who investigated the effect of shot peening on the corrosion resistance of materials produced by the AM method, reported an increase in corrosion resistance [31]. In another study examining the effect of spray pressure on corrosion resistance, Vella et al. concluded that increasing the spray pressure did not improve corrosion resistance [32]. Benedetti and colleagues, in their study, determined that the compressive stress generated by the forging process reduces surface porosity, thus increasing fatigue strength [33]. Ahmed and colleagues, in their study, stated that the deformation occurring on the surface after shot peening can alter corrosion resistance [34].

A review of the literature reveals that problems occur during the production of Ti-6Al-4V alloys manufactured using the AM method, and

that improving surface properties is crucial to eliminate these problems. Improving surface properties can only be achieved by obtaining a smoothly processed surface layer. This clearly demonstrates the importance of correctly selected processing parameters. This study investigates the corrosion resistance of Ti-6Al-4V material manufactured by the DMLS method and shot peened with different coverage ratio. Thus, by determining the effect of coverage ratio on corrosion behavior, as opposed to shot peening intensity, this study allows for the examination of the effect of a different process parameter (coverage ratio) on corrosion behavior. Thus, this study has shown that the corrosion properties can be improved, especially in areas where Ti-6Al-4V material, used as a biomaterial, is employed.

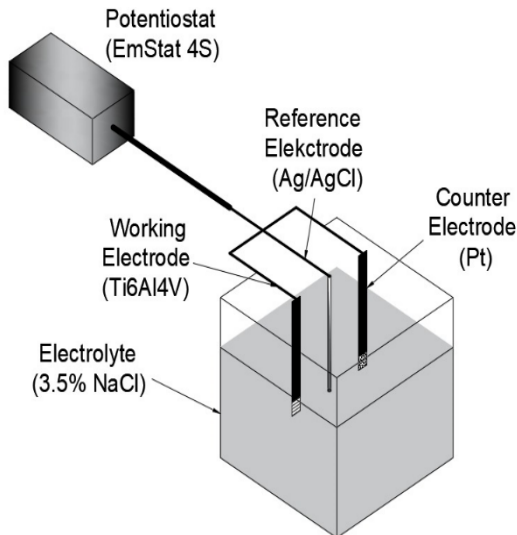
2. MATERIALS AND METHODS

In this study, Ti-6Al-4V samples were produced using the DMLS method, which is one of the AM methods. Ti-6Al-4V powder with a size of 25-60 μm and a density of 2.32 g/cm³ was used in sample production. Samples were produced using an EOSINT M280 (Germany) instrument. Sample production consists of the following steps: creating a CAD model; converting the created model to STL format; selecting process parameters; and producing the sample. Sample dimensions were selected as 10x10x4mm. For Ti-6Al-4V samples, the following processing parameters were used, as recommended by EOS GmbH, Germany Company: 0.03mm layer thickness; 1250mm/s laser speed; 170W laser power; and 0.1mm scanning distance. In samples produced using the DMLS method, residual stresses occur due to the high energy density of the laser and rapid cooling. To eliminate these stresses, the samples were subjected to stress-relieving annealing heat treatment in a controlled atmosphere furnace. The produced samples were then subjected to a shot peening process. For the shot peening process, S110 balls with a diameter of 0.22-0.28 mm and a hardness of 62 HRC were used. In this study, the shot peening intensity applied to the samples was selected to be constant at 12-14 Almen intensity. The only treatment parameter modified in the study was the coverage ratio, which was set at 100% and 200%. The abbreviations of the samples used in the study are given in Table 1.

Table 1. Abbreviations of the samples used in the study

Processed Sample	Sample abbreviation
No shot peening process applied (Untreated)	UT
Shot peening process applied at 12-14 A intensity with 100% saturation rate	T100
Shot peening process applied at 12-14 A intensity with 200% saturation rate	T200

An Olympus BX51TRF-6 optical microscope was used for microstructure analysis before the shot peening process, and a FEI QUANTA FEG 250 SEM instrument was used for surface morphology analysis after the shot peening process. Corrosion experiments were conducted to determine the corrosion behavior at different coverage ratio levels. An EmStat 4S Potentiostat/Galvonostat/Impedance Analyzer device was used for corrosion experiments. The system used in the corrosion experiments is shown in Figure 1. In the experiments, a Ti-6Al-4V sample was used as the working electrode; Ag/AgCl was used as the reference electrode; and a 3-electrode system consisting of a counter electrode and a counter Pt plate was used.

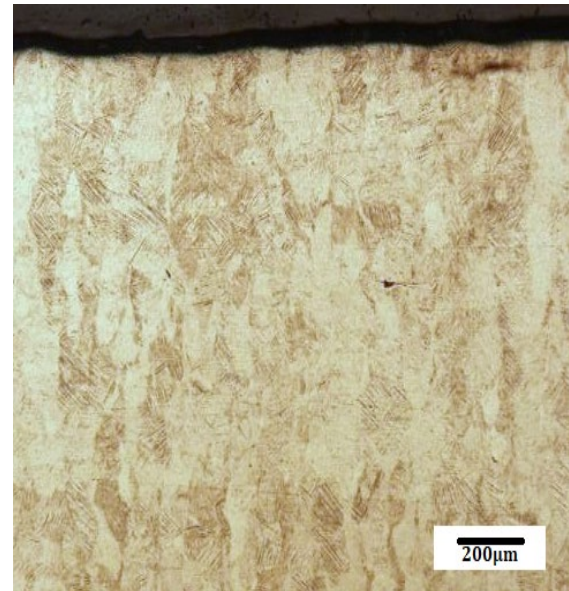
**Figure 1.** Corrosion test setup.

The samples were immersed in a 3.5% NaCl solution for 30 minutes prior to the experiment to allow equilibrium, and Open Circuit Potential (OCP) values were measured. The working surface area for corrosion experiments was calculated as 0.5 cm². A signal amplitude of 5 mV and a frequency range of 0.01 Hz-100 kHz were selected for the study. Corrosion experiments were performed using these

parameters. After the experiment, corrosion current density (i_{corr}) and corrosion potential (E_{corr}) values were determined using Tafel curves. Experiments were performed using fresh electrolyte each time, and the samples were washed with deionized water after the corrosion experiments.

3. EXPERIMENTAL FINDINGS

In this study, the microstructure of Ti-6Al-4V alloy produced by additive manufacturing method was examined using an optical microscope and is given in Figure 2. From Figure 2, it was determined that the structure is $\alpha+\beta$. This structure was formed by the decomposition of the martensitic (α') structure (β) during the stress relief process applied to the 3D-printed material [35].

**Figure 2.** Microstructures of Ti-6Al-4V samples produced by DMLS method.

SEM analyses of un shotpeened and shot peened samples with 100% and 200% coverage ratio levels and 12-14A intensity are given in Figure 3.

In shot peening, increasing Almen intensity leads to an increase in surface roughness. Samples produced using additive manufacturing methods exhibit significantly higher surface roughness as a result of this manufacturing process. In the shot peening process, these roughnesses are reduced at the peak points of the roughness due to the impact of the balls on the surface, resulting in a decrease in surface roughness. In ball striking, as the coverage ratio increases, a greater

reduction in surface roughness will occur due to the increased number of balls impacting the surface. As the coverage ratio increases, the surface roughness will first decrease, then increase, and tend to stabilize after a certain period, a finding consistent with the literature [36].

Corrosion tests were performed on both shot-peening and unshot-peened samples in a 3.5% NaCl solution, and the resulting Tafel polarization curve is given in Figure 4.

Examination of Figure 4 reveals that a passivation zone formed during corrosion progression in both shot-peening and unshot-peened samples. For all samples, the passive region is approximately in the potential range of -1.5 V to -0.5 V, and it is observed that the anodic current slows down in this potential range. This indicates the formation of a passivation film in the 3.5% NaCl solution, which prevents corrosion of the material.

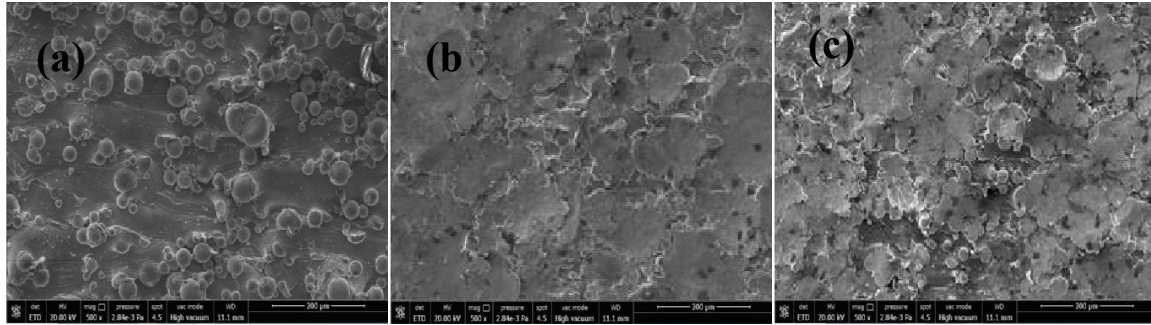


Figure 3. SEM images of samples before and aftershot peening (a) unshotpeened (b) shot peened samples at 100% coverage ratio (c) shot peened samples at 200% coverage ratio.

The corrosion potential (E_{corr}), corrosion current density (i_{corr}), and corrosion rate values obtained from the polarization curves are given in Table 2. Table 2 shows that the highest current density value was obtained in the sample unshot-peening process, at $1.084 \times 10^4 \mu A$. High current density causes instability in the formation of the passivation film. This instability results in low corrosion resistance [37]. In the study, the highest corrosion rate was obtained in the unpeened sample. In addition, a positive shift in corrosion potential occurred with the shot peening process. This positive shift reduced the tendency of the materials to corrode, and the results obtained are consistent with the findings reported in the literature [38, 39]. Therefore, a more positive E_{corr} value indicates that the sample is more stable against corrosion, and when Table 2 is examined, the E_{corr} value of the sample named T100 was calculated as -1.876 V. Compared to other samples, the T100 sample has a lower tendency to corrode and is thermodynamically more stable. The surface roughness values of Ti-6Al-4V samples produced by additive manufacturing are reduced by the shot peening process. This leads to an improvement in corrosion resistance. It has been reported in the literature that reducing surface roughness by shot peening has a positive effect on inhibiting

pitting corrosion [18, 40]. However, an increase in surface roughness is inevitable with increasing coverage ratio. This situation has caused a decrease in corrosion potential (E_{corr}) and consequently in corrosion resistance.

Table 2. Results of polarization curve performed on the samples before and after SP

Samples	E_{corr} (V)	i_{corr} (μA)	Corrosion rate (CR) (mm/year)
UT (Not shot peened)	-1.987	1.084×10^4	16.02
T100 (12-14A %100 coverage ratio)	-1.876	4192	6.193
T200 (12-14A %200 coverage ratio)	-1.911	7677	13.34

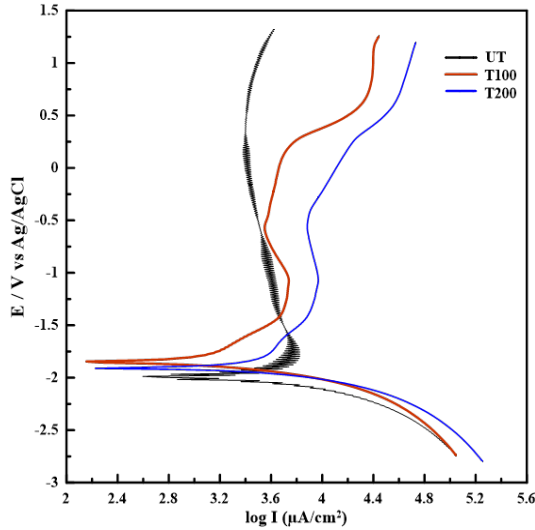


Figure 4. Tafel polarization curves for Ti-6Al-4V samples.

In Nyquist plots, the diameter of each semicircle represents the polarization resistance of the material. A larger diameter of the semicircle indicates higher corrosion resistance [41]. Figure 5 shows the electrochemical impedance spectroscopy (EIS) results for Ti-6Al-4V samples in a 3.5% NaCl solution. Examining the Nyquist plots obtained after EIS analysis (Figure 5a), it is observed that the capacitive loop radii of the samples subjected to 100% coverage ratio and shot peened are larger than those not subjected to shot peened and shot peened with a 200% saturation. This indicates that the impedance values of the Ti-6Al-4V alloy increase with ball beating [42]. Examining the Bode plot given in Figure 5b, it was determined that all samples exhibited a peak in the phase angle that first increased and then decreased as the frequency increased. The phase angle represents the capacitive character of the material and, consequently, its ability to prevent the electrolyte from penetrating into the peened surface. An increase in the phase angle is an indicator of increased corrosion resistance of the peened surface. An increased phase angle can be expressed as an increase in corrosion resistance. The higher phase angle peak ($\sim 61^\circ$) exhibited by the T100 sample over a wide frequency range in the Bode Phase Angle graph shows that the passive oxide layer formed on its surface is more stable, homogeneous, and has superior capacitive properties compared to the UT and T200 samples.

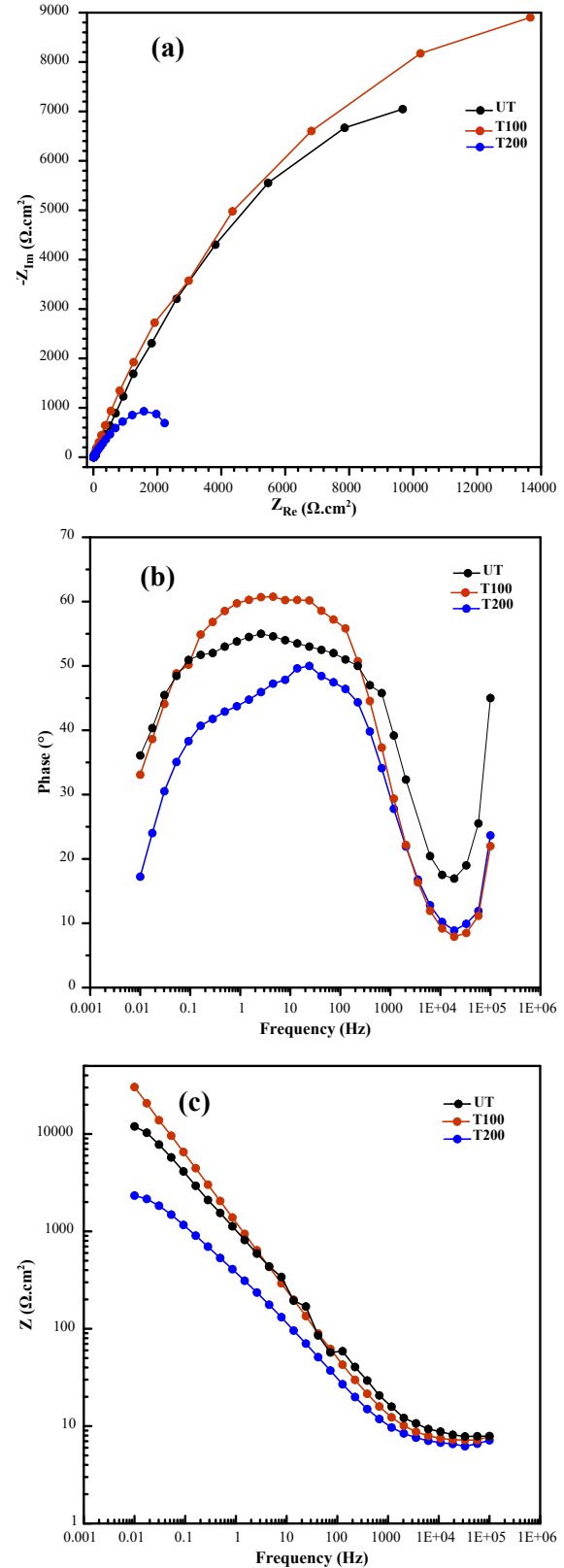


Figure 5. Effect of shot peening on the EIS of Ti-6Al-4V titanium alloy in 3.5% NaCl solution (a) Nyquist diagram; (b) Bode diagram, phase angle (c) Bode diagram, impedance.

The Bode impedance graphs are consistent with the capacitive semicircle observed in the Nyquist diagram. Furthermore, the high impedance values obtained in the low-frequency region are an indicator of high corrosion resistance. As the Z value increases, corrosion resistance also increases (Figure 5c). A decrease in corrosion resistance was observed in samples with a coverage ratio of 200%. This is because, in samples with a 200% coverage ratio, the plastic deformation limit is exceeded, leading to flaking or microcracks on the surface, allowing the corrosive solution to penetrate the material and paving the way for pitting corrosion. Additionally, increasing the coverage ratio increases the surface area by creating craters. This increased surface area leads to increased contact with the corrosive environment, resulting in an increased corrosion rate.

4. RESULTS

The results obtained from the analysis of Tafel and EIS (Nyquist and Bode phase and Bode impedance) graphs are consistent with each other for the T100 sample. The consistent and significant superiority exhibited by the T100 sample in both methods proves that the T100 sample forms a more effective corrosion protection under steady-state conditions. However, the Tafel and EIS results of the unpeened sample and the sample with a 200% coverage ratio are inconsistent. This inconsistency stems from the passivation behavior of the material and the nature of the analytical methods. Tafel analysis scans the material potentially, and this process can disrupt, dissolve, or cause the accumulation of corrosion products in the passive layer formed. EIS analysis, on the other hand, measures the material in steady-state conditions by applying a very small signal.

In conclusion, it was found that the T100 sample has better corrosion resistance than the unpeened and T200 samples. Corrosion experiments conducted in 3.5% NaCl medium increase the usability of the T100 sample as a biomaterial. Considering these study results and the mechanisms in the living organism, it would be beneficial to investigate the tribocorrosion properties in future studies.

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