Study on factors affecting the nickel plating process on titanium alloy substrates

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Received 10 Jan. 2026; Revised 5 Mar. 2026; Accepted 10 Apr. 2026; Published 25 Apr. 2026.

DOI: https://doi.org/10.54939/1859-1043.j.mst.110.2026.136-142

ABSTRACT

This paper presents the results of research on the factors influencing the electroless nickel plating process on BT6 titanium alloy. Prior to plating, the titanium alloy surface is activated, etched, and zincated using a solution consisting of ZnF_2 *(85 g/L), HF (40%, (60 g/L), and Ethylene glycol (500 mL/L) at a temperature of 25 °C for 1 minute. The electroless nickel plating is performed using a solution of* $NiCl_2$ *(10 ÷ 50 g/L), KF (10 g/L), and Glycine (30 g/L) with a pH of 2.8 ÷ 3.2. Research results indicate that at a* $NiCl_2$ *salt content of 20 ÷ 30 g/L and a plating time of 10 ÷ 15 minutes, the coating surface is quite smooth and uniform, with nickel crystals tightly adhered to the surface. Post-plating heat treatment conducted between 400 ÷ 415 °C promotes the formation of intermetallic phases and transition zones between the layers, enhancing the durability of the Ni coating on the titanium alloy substrate.*

Keywords: BT6 titanium alloy; Nickel plating; Heat treatment of titanium alloy; Titanium alloy plating.

1. INTRODUCTION

Titanium and its alloys possess numerous superior properties, including high strength, low density, and good ductility. Due to its relatively high melting point, titanium serves as a heat-resistant metal; it maintains its durability at low temperatures without becoming brittle or cracking. Titanium is significantly lighter than steel, with a density of approximately 4.5 g/cm^3 compared to steel's 7.8 g/cm^3 [1]. Despite its low weight, titanium is the strongest material, surpassing the hardness of aluminum and even some steels. [1, 2]. However, titanium still has drawbacks such as a high friction coefficient, low electrical and thermal conductivity, and poor weldability. Consequently, it is necessary to plate other metals such as Cr, Ni, Cu, Au, or Ag onto the titanium surface to suit specific functions, such as increasing hardness, enhancing erosion and corrosion resistance, and improving electrical or thermal conductivity [3, 4].

Electroplating on titanium alloys faces significant challenges because the material surface is covered by a stable oxide film that is difficult to remove and extremely quick to reform in air, water, and various solutions. Because titanium is chemically inert and stable, achieving good adhesion between the coating and the substrate is normally very difficult [4, 5]. Solutions for electroless nickel plating on titanium alloy substrates have made significant progress in improving the adhesion strength between the coating and the base metal [3]. The electroless nickel layer is effectively formed following activation and etching processes to remove the native oxide layers on the titanium surface. This paper presents research results on the electroless nickel plating process on BT6 titanium alloy. Once the electroless nickel underlayer is established, further functional coatings such as gold or silver for enhanced electrical conductivity, or chromium for increased hardness and wear and corrosion resistance can be applied to the titanium alloy.

2. EXPERIMENTAL

2.1. Chemicals and equipment

Chemicals used include: NaOH, Na_3PO_4, Na_2CO_3, Na_2SiO_3, H_2SO_4, HCl, HNO_3, HF (40%),

ZnF_2, Ethylene glycol, Glycine, $NiCl_2$, KF, titanium alloy BT6 (primarily composed of titanium, with 6% Al and 4% V).

Equipment includes a heated magnetic stirrer, a vacuum drying oven, and glass beakers.

2.2. Technological process for creating nickel coating on titanium alloy

BT6 titanium alloy samples were mechanically projcessed to dimensions of 30 × 15 × 3 mm, with the surface polished using 2000-grit sandpaper and degreased in a solution of 25 g/L NaOH, 30 g/L Na_2CO_3, 50 g/L $Na_3PO_4.12H_2O$, and 5 g/L Na_2SiO_3 for 5 minutes at a temperature of 60 °C. The process of creating the nickel coating on the BT6 titanium alloy substrate was carried out through the following technological steps:

Table 1. *Technological steps and solution compositions used.*

Steps	Solution composition and implementation conditions
Activation	15 mL/L HF (40%), 60 mL/L HNO_3 ($d = 1.35\ g/cm^3$), and 925 mL/L H_2O at a temperature of 25 °C for 1 to 1.5 minutes.
Etching	250 mL/L H_2SO_4 ($d = 1.84\ g/cm^3$) and 25 mL/L HCl ($d = 1.19\ g/cm^3$) at a temperature of 65 °C for 2 minutes.
Zincating	85 g/L ZnF_2, 60 g/L HF (40%), 500 mL/L Ethylene glycol at a solution temperature of 25 °C for a treatment time of 1 minute.
Nickel plating	10 to 50 g/L $NiCl_2$, 10 g/L KF, 30 g/L Glycine with a solution pH of 2.8 to 3.2, for a reaction time of 5 to 20 minutes at room temperature.

2.3. Research methods

The elemental composition and structural morphology of the coating were determined using EDX and SEM on a JEOL JSM-6490 device. The thickness of the coating was determined according to the ASTM B487-20 standard using an Axiovert 40MAT device.

3. RESULTS AND DISCUSSION

3.1. Research on nickel plating technology

3.1.1. Results of surface treatment and chemical zincating on titanium substrate

The initial BT6 titanium alloy samples were degreased, followed by pretreatment steps such as activation and etching to remove the highly stable oxide components on the material surface. Subsequently, a chemical zincating process was performed on the surface using a solution of 85 g/L ZnF_2, 60 g/L HF (40%), and 500 mL/L ethylene glycol, at a solution temperature of 25 °C with a treatment time of 1 minute. After the activation, etching, and zincating processes, the surface of the BT6 titanium alloy sample was covered with a layer of Zn crystals (Figure 1).

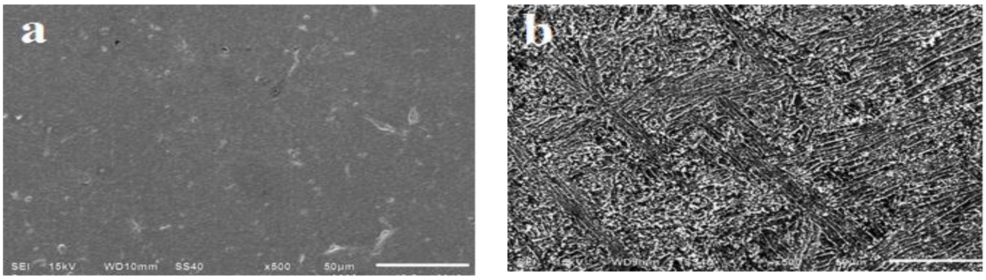


Figure 1. *SEM images of the titanium alloy surface before and after the zincating process: a. Initial surface of the BT6 titanium alloy; b. Zincated surface of the BT6 titanium alloy.*

Observation of the SEM image (Figure 1b) reveals that the zinc crystal grains are uniformly distributed across the etched grooves formed after the activation and etching processes. The zinc crystal layer completely covers and adheres to the surface of the titanium alloy substrate, creating favorable conditions for the growth of the subsequent electroless nickel plating layer.

3.1.2. Influence of solution composition on the properties of the nickel coating

After forming the zinc coating from the zincate solution, the BT6 titanium alloy is plated with nickel using a solution containing, KF, and glycine. Among these, the concentration of salt has a significant impact on the quality of the resulting coating. KF acts as a conductivity enhancer for the electrolyte, the fluoride ions assist in the surface activation of the metallic substrate, whereas glycine acts as a ligand that mediates the reduction of Ni^{2+} ions into elemental nickel.

Figure 2 shows SEM images of the nickel coating surface on the titanium alloy substrate from an electroless plating system with content varying from 10 to 50 g/L $NiCl_2$, 10 g/L KF, and 30 g/L glycine, conducted at room temperature for a reaction time of 15 minutes and a pH range of 2.8 to 3.2.

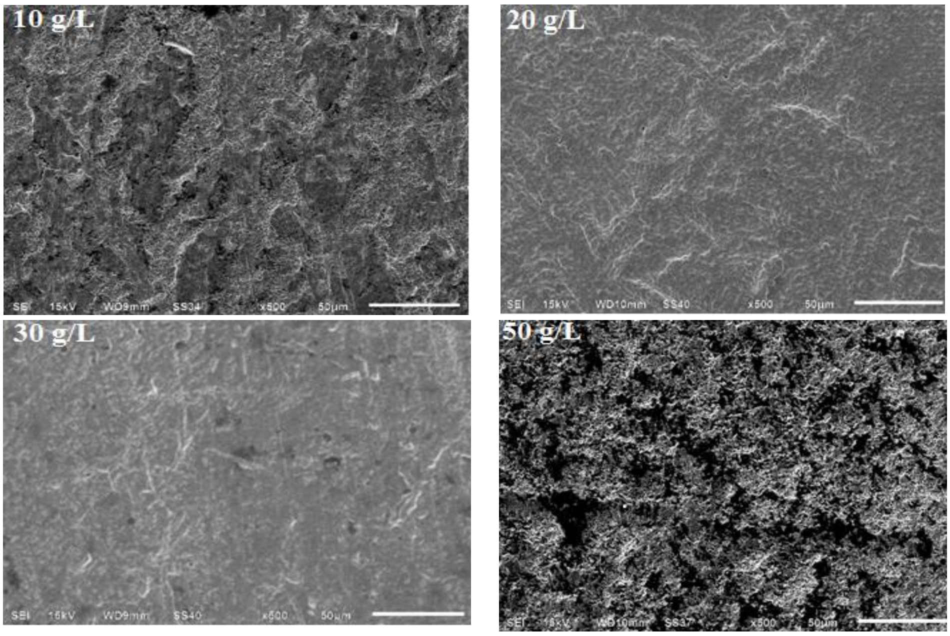


Figure 2. *SEM images of the BT6 titanium alloy surface electrolessly plated with nickel in solutions with different* $NiCl_2$ *contents.*

From the SEM images (Figure 2), it can be observed that at a salt concentration of 10 g/L, the surface of the plated object has not yet been smoothed. This may be due to the slow precipitation rate of nickel at this concentration, which fails to completely cover the sample surface. In the concentration range of 20 ÷ 30 g/L, the coating surface is relatively smooth, flat, and uniform, with nickel crystals tightly and densely adhered to the surface. When the concentration is increased to 50 g/L, the surface exhibits a porous structure, and the nickel crystal grains become larger in size. Thus, to obtain a nickel coating with a smooth, uniform, and dense surface with good adhesion, a content in the range of 20 ÷ 30 g/L should be selected.

3.1.3. Influence of plating time on the properties of the nickel coating

For the chemical plating process, varying the plating time leads to changes in both the thickness and the properties of the coating; therefore, it is necessary to select an appropriate duration to achieve the best quality. Figure 3 presents SEM images of the plated samples at different time intervals from 5 to 20 minutes. It is observed that at a plating time of 5 minutes, the coating surface remains rough and not yet uniform. Increasing the plating time to 10 ÷ 15 minutes results in a smoother, flatter, and more uniform surface quality. When the plating time is further increased to 20 minutes, a phenomenon of nickel crystal agglomeration occurs, making the surface rougher again. Thus, the optimal time to create a chemical nickel coating with good surface quality is within the range of 10 to 15 minutes.

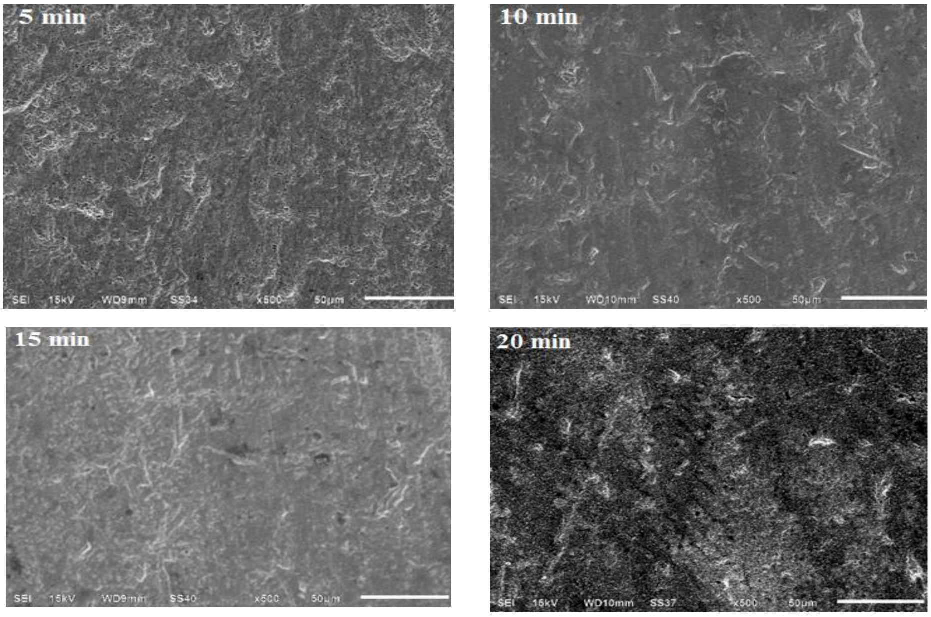
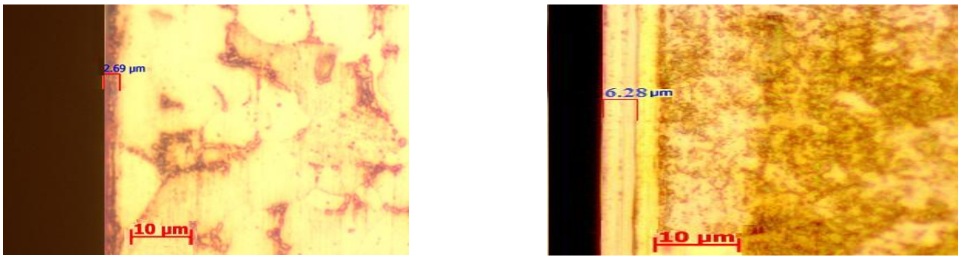


Figure 3. *SEM images of the titanium alloy plated with nickel at different concentrations.*

The results of measuring the thickness of the electroless nickel coating from the solution system containing 30 g/L $NiCl_2$, 10 g/L KF, and 30 g/L glycine, with reaction times of 5 minutes and 15 minutes conducted at room temperature, reached values in the range of 2.69 to 6.28 µm (Figure 4).



Plating time 5 minutes (thickness 2.69 µm). *Plating time 15 minutes (thickness 6.28 µm).*

Figure 4. *Thickness of electroless Ni coating at plating times of 5 and 15 minutes.*

In order to further increase the thickness of the nickel coating, a subsequent electroplating process can be performed using the Watts solution system [2].

3.2. Study on the effects of heat treatment

When the annealing process is performed, the intermediate Zn coating diffuses into the titanium alloy substrate and into the outer Ni plating layer. This diffusion process creates intermetallic phases, resulting in better bonding of the Ni coating. Phase diagrams for Ni-Zn, Ti-Zn, Al-Zn, and Al-Ni-Zn have proven that these interactions are entirely possible during heat treatment [6-11]. Thus, Zn serves not only as an intermediate layer to facilitate better plating; after annealing, it also acts as a bonding agent, creating intermetallic phases and transition zones between layers, which significantly improves the durability of the Ni coating.

This study selected an annealing temperature range of 400 °C to 415 °C, which is less than the melting point of zinc. Some studies on the diffusion reaction between Ni and Zn have reported that at 200 ÷ 350 °C, bonding phases such as $NiZn_8$ (δ), and Ni_2Zn_{11} (γ) are formed; at temperatures of 350 °C and 400 °C, bonding phases such as Ni_3Zn_{22} (δ) and Ni_3Zn (γ) can form. Studies on the Ti-Zn system show a complex phase equilibrium with several intermetallic compounds such as $TiZn_3$, $TiZn_5$, Ti_3Zn_{22}, $TiZn_{16}$ and Ti_2Zn_3, and formed during the heat treatment process. For Al and Zn during heat treatment, phases with formulas and Al_2Zn_3, Al_3Zn_2, and other phases γ, ε with compositions varying by temperature, will be formed. [10,11]

Figure 5 presents an SEM cross-sectional image of the electroless nickel coating on the BT6 titanium alloy substrate before heat treatment.

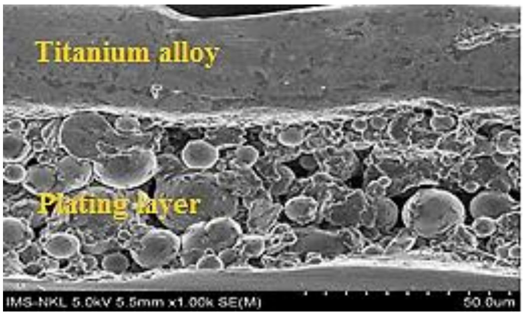


Figure 5. *SEM cross-sectional image of the coating before heat treatment.*

From the SEM image in Figure 5, it can be observed that before the heat treatment process is performed, a clear delamination between the plating layer and the titanium alloy substrate is visible. This separation is the cause of the very poor adhesion between the plating layer and the substrate, which leads to the plating layer easily peeling off from the base.

Figure 6 shows a cross-sectional SEM image of the nickel plating on the titanium alloy substrate after the heat treatment process was conducted at a temperature of 415 °C, under a vacuum of 0.1 Pa, for a duration of 3 hours.

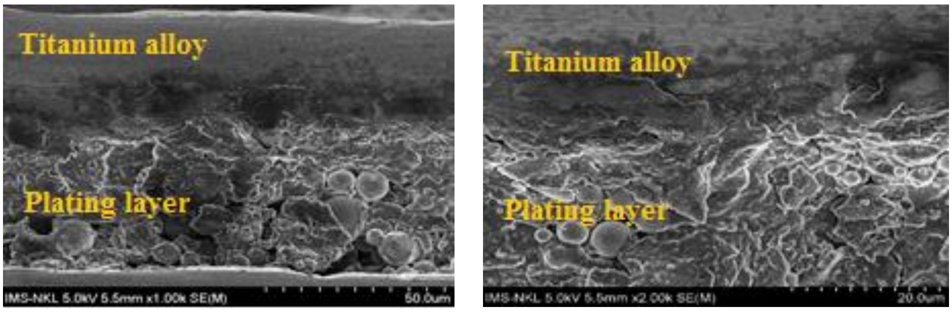
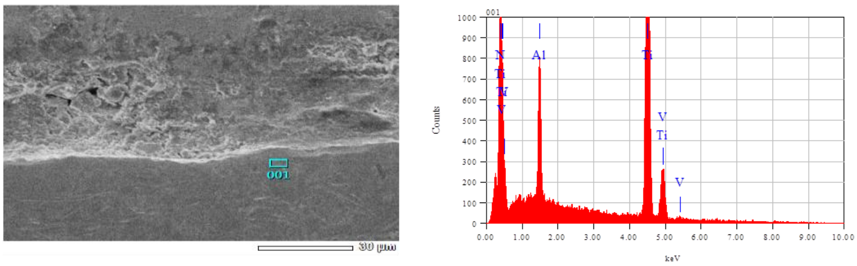


Figure 6. *Cross-sectional image of the Ni coating on the titanium alloy after heat treatment.*

Observation of the SEM cross-sectional image of the coating after heat treatment shows that the delamination between the coating and the substrate is no longer visible. At the boundary between the coating and the titanium alloy substrate, there is a more uniform and homogeneous bond between the layers and the base. This can be explained by the diffusion process, which creates bonding phases between Zn and the Ti and Al in the titanium alloy substrate, as well as the diffusion of Zn with the outer Ni coating. This process helps increase the adhesion of the coating and creates a strong bond with the titanium alloy substrate, and also removes any residual stresses caused after the plating process.

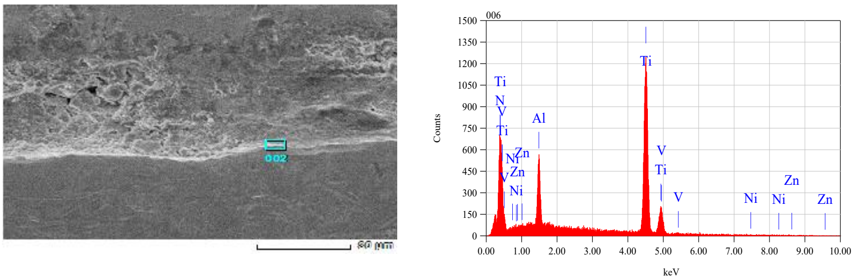
Figure 7 shows the results of the EDX elemental analysis on the cross-sectional surface at the boundary between the coating and the titanium alloy substrate at various positions. The results show that the elemental composition in this region includes elements from the titanium alloy substrate, such as Ti, Al, and V, as well as elements from the coating, namely Zn and Ni.

Results of the elemental composition analysis at the cross-section of the plated sample at various positions show that there is an appearance of metallic elements, including Ti, Al, V, Ni, and Zn, in the interface zone. This can explain the formation of bonding types and the creation of intermetallic phases during the heat treatment process.



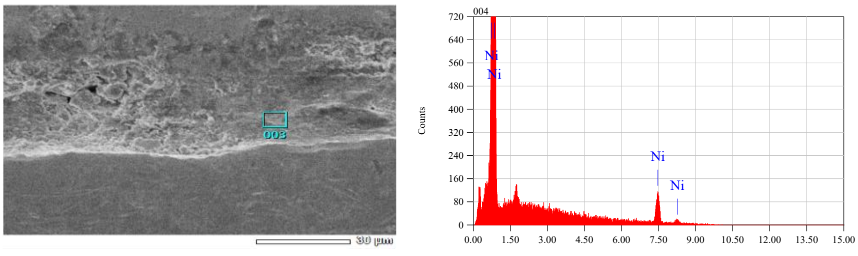
Elemental composition:
Ti: 90.29%
Al: 45.78%
V: 3.93%

Region 1: Titanium alloy substrate.



Elemental composition:
Ti: 89.64%
A:l 4.81%,
Ni: 0.27%,
Zn: 3.83%.

Region 2: Interface zone between the coating and the substrate.



Elemental composition:
Ni 100%.

Region 3: Ni coating layer.

***Figure 7.** EDX spectra of the cross-section of the Ni-plated sample on BT6 titanium alloy.*

4. CONCLUSIONS

To create an electroless nickel coating on a BT6 titanium alloy substrate, after forming a chemical zincate layer, a solution with the following composition is used: $NiCl_2$ 20 to 30 g/L, KF 10 g/L, and Glycine 30 g/L, with a solution pH of 2.8 to 3.2 at room temperature. For a plating time of 10 to 15 minutes, the coating thickness reaches 6.28 µm; the coating surface is smooth, flat, and uniform, with nickel crystals tightly and densely covering the surface. Following nickel plating, a heat treatment process is performed at a temperature range of 400 to 415 °C under a vacuum of 0.1 Pa for 3 hours. This process facilitates the formation of intermetallic phases and transition zones between the layers, thereby improving the durability of the Ni coating on the titanium alloy substrate.

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TÓM TẮT

Nghiên cứu một số yếu tố ảnh hưởng đến quá trình tạo lớp mạ niken trên nền hợp kim titan

Bài báo trình bày kết quả nghiên cứu ảnh hưởng của một số yếu tố đến quá trình tạo lớp mạ niken hóa học trên nền hợp kim titan BT6. Trước khi mạ niken hóa bề mặt hợp kim titan được hoạt hóa, tẩm thực và tạo lớp kẽm hóa học bằng dịch ZnF_2 *85 g/L, HF (40%) 60 g/L, Etylenglicon 500 mL/L, nhiệt độ dung dịch 25 °C, thời gian xử lý 1 phút. Lớp mạ niken hóa được thực hiện bằng dung dịch* $NiCl_2$ *10 ÷ 50 g/L, KF 10 g/L, Glycin 30 g/L, pH của dung dịch 2,8 ÷ 3,2. Các kết quả nghiên cứu chỉ ra ở hàm lượng muối* $NiCl_2$ *từ 20 ÷ 30 g/L, thời gian mạ từ 10 ÷ 15 phút thì bề mặt lớp mạ khả nhẵn phẳng, đồng đều, các tinh thể niken bám kín sít trên bề mặt. Sau khi mạ hóa niken thực hiện quá trình ủ nhiệt trong khoảng 400 ÷ 415 °C, có tác dụng tạo pha liên kim loại, các vùng chuyển tiếp giữa các lớp mạ với nhau giúp cho độ bền lớp mạ Ni trên nền hợp kim titan tốt hơn.*

Từ khóa: BT6 titanium alloy; Nickel plating; Heat treatment of titanium alloy; Titanium alloy plating.