



Characterization of Nb₂O₅-Ni Coating Prepared by DC Sputtering

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ABSTRACT

The Nb₂O₅-Ni coating was processed using DC sputtering on structural steel and study characterization of composite coating SEM/EDS inspection indicated clearly perfect incorporation of Nb₂O₅ within the nickel rich. Increasing in surface roughness and decreasing in average diameters of particles were obtained for coated surface compared with uncoated surface from AFM analysis, in addition, microhardness test and thickness test showed that increasing of the hardness value to 163 HV for Nb₂O₅ - Ni composite coating compared to 132 HV for uncoated samples, the hardness for Ni coating also increasing to 155 HV and the thickness for Nb₂O₅-Ni composite coatings increased significantly compared to uncoated samples.

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1. INTRODUCTION

Coating processes give protection to structure surfaces that exposed to aggressive and corrosion environments in various fields, so considered necessary for some applications [1]. Thin-film or coating is called a new field of chemistry/materials. The structure of thin-films, like all material, is divided into amorphous and polycrystalline structures, depending on the condition of preparation and the material composition. Thin-film consists of two parts: the substrate and the substratum on which the film is mounted [2]. Glow discharges and associated plasma are used in a large number of application fields. When a sample is exposed to the plasma, ionized atoms and molecules may react with the surface exposed. Various effects are commonly observed, including etching, sputtering and material surface alteration [3, 4].

The plasma is generated in a plasma reactor DC glow discharge by supplying a potential DC between two electrodes. The electrodes are in a jar that was evacuated. The vacuum is applied in a plasma forming medium. Natural processes initially ionized a small group of atoms containing the cell. The interaction of a cosmic ray or photon with a molecule or plasma gas atom releases an electron. This will a plasma reactor for ion DC glow discharge; the plasma is created by supplying the potential DC from two electrodes.

Sputtering deposition theory predicts that sputtering caused by the formation of very high local temperatures by bombarding ions, which contributes to the evaporation of target material from those local areas. [5] Sputtering also is a method of ejecting atoms from the solid surface through the action of energetic particles [5]. To protect steel surface there are many methods used of metallic coatings such as Ni, Zn, Cu and alloy coating such as Zn-Ni, Ni-Co, etc. The inclusion of nano or micro-sized particles in the coating has been found to enhance steel corrosion resistance. Such coating was known as composite coating and view higher resistance of corrosion to increase hardness [6].

Niobium oxide has been suggested as a suitable replacement for Ta₂O₅ in solid electrolyte (Ta₂O₅) condensers, where the dielectric oxide layer is produced from porous metal compressed from anodic oxidation. Due to the larger permittivity of Nb₂O₅ compared to Ta₂O₅, it is a suitable replacement due to its similar anodization nature and provides the benefit of greater natural abundance. With a wide variety of technical applications such as sensing materials to assist catalytic processes, niobium oxide films were proposed. The coatings are biocompatible due to the good chemical stability and resistance to corrosion for both acid and base media. [7]. Nb₂O₅ films were prepared using various methods, including magnetron sputtering, electrodeposition, anodization, thermal oxidation, sol-gel processes, chemical vapor deposition (CVD) and epitaxy of the atomic layer [8]. Nb₂O₅ is present in different polymorphic shapes; TT-Nb₂O₅ (pseudo-hexagonal), T-Nb₂O₅ (orthorhombic) and H-Nb₂O₅ (monoclinic) are the most common phases. The most thermodynamically stable form is the H-phase, while the least stable is the TT-phase. It is reasonably easy to transform the TT-phase to the H-phase of sufficient heat treatment. Similarly, at a lower temperature, the amorphous niobium acid can be converted into TT- Nb₂O₅. At 600-800°C, by heating, TT-Nb₂O₅ converts almost continuously into T-Nb₂O₅. H-Nb₂O₅ originates from all other air heating forms at roughly 1100 °C [9]. To achieve good corrosion resistance, the steel chemistry and processing technique need to carefully construct. Nb₂O₅ is believed to show capable of increasing steel strength with no great loss in ductility and durability and improving specific corrosion resistance [10]. In this work, Nb₂O₅- Ni coating was deposited by DC sputtering on carbon steel to investigate the corrosion resistance in seawater at 303 and 333 K and analysis the characterization of the composite coating.

Reviews of literature provide some researches, which is done in this field, it's:

Yinsong et al. In (2003), prepared Nb₂O₅ films by DC reactive sputtering process. These structural features include solid electrochemical stability, high reversibility of Li⁺ injection/extraction and better Electrochromic property. The colorless blank and brownish-gray color of DC sputtered Nb₂O₅ films may be a promising candidate for electro chrome applications [11].

Nilgiin et al. In (1996), prepared Electrochromic niobium coatings by the sol-gel spin-coating and DC magnetron sputtering techniques. XRD analysis shows the sol-gel process and magnetron sputtering films for temperatures below 450 °C. was amorphous. However, the structure was crystalline at a temperature higher than 450 °C. X-ray photoelectron spectroscopy (XPS) Studies indicated that Nb₂O₅ stoichiometry of films. The electrochemical activity or structural changes in 1 M LiClO₄/propylene carbonate solution was observed [12].

H. Chen et al. (2015), applied electroless plating to deposit ternary Ni-P-W coatings forming dispersed Nb₂O₅ particles mostly on sintered NdFeB substrates. During electroless plating, the influence of experimental process conditions was investigated on the deposition rate. Experimental research tested the performance of coatings, like corrosion resistance, interlayer adhesion strength and micro-hardness [13].

M.F. Pillis et al. in (2016), applied niobium oxide thin-films to stainless steel substrates AISI 316 by sputtering the reactive DC magnetron. Niobium oxide films have been studied in their structure,

composition, and corrosion resistance. Electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization evaluated the corrosion behavior of the specimens. X-ray diffractometry analyzed the structure of the film. The Nb_2O_5 layers increased the corrosion resistance of the substratum. The best performance in protection was achieved at 15 min deposition time [14].

R. M. Reddy et al. in (2017) prepared the Ni– Nb_2O_5 composites by the pulse electrodeposition method. Optimization study was achieved to obtain a uniform deposition layer. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were used to study both coating's structure and surface morphology. The coefficient of texture and hardness of the deposits was determined. Traditional weight loss and electrochemical approaches were used to analyze the coating's corrosion behavior. Comparisons of the corrosion behavior of straight current (DC) and pulse current (PC) coatings have been investigated [15].

2. EXPERIMENTAL

I. Sample Preparations

Steel (AISI 1015) specimens prepared to study the electrochemical test. Firstly, cutting the specimen is considered an important parameter in corrosion resistance and a uniform surface has to be prepared. Square specimens cut of the electric saw (2 cm \times 2 cm \times 1 cm) and then collected as a final. Specimen shape. Secondly, grinding and polishing the specimen's accomplished, though, series of 400, 600, 800 and 1000 silicon carbide papers to achieve smooth, scratch-free surface and show the chemical composition test for carbon steel samples in TABLE 1.

TABLE I: Chemical composition of carbon steel.

| Element | C | Si | Mn | P | S |
|----------|-----------|-------|-----------|--------|--------|
| Wt. % | 0.161 | 0.271 | 0.425 | 0.0073 | 0.0049 |
| Standard | C | Si | Mn | P | S |
| Wt. % | 0.13-0.18 | 0.29 | 0.30-0.60 | 0.040 | 0.050 |

II. Preparation of Target coating and technique

Powders of nickel and niobium pentoxide have been used during this work. Powders were used in the coating were both Ni and Niobium pentoxide. X-ray diffraction (XRD) test performed to verify the powders showed in Figure 1. Also, the preparation of the powders achieved by mixing and pressed by a pneumatic specimen punch device. The load was 8 bars for 1 min. which are used during press with the steps, as shown in Figure 2.

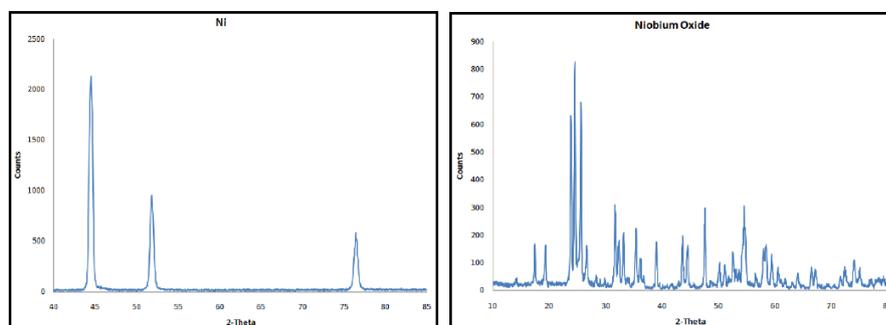


Figure 1: XRD of a pattern of Ni powder and Nb_2O_5 powder.

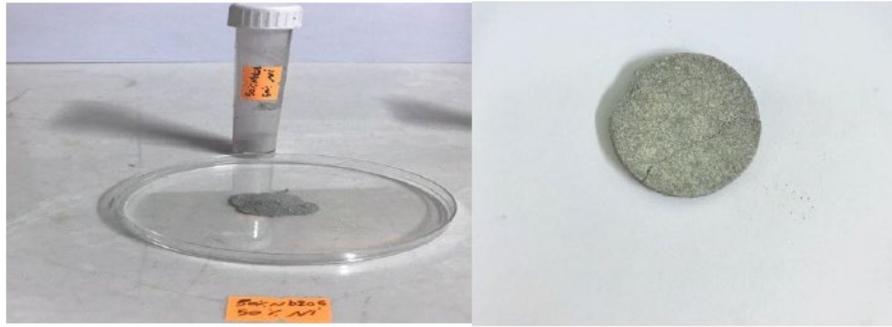


Figure 2: Preparation of Target coating.

DC sputtering technique was consisted of the target (cathode) and anode disk of stainless steel. The cathode opposite the anode and provides the gas discharge with the electrical field. The plasma chamber consisted of electrical electrodes and a 4KV DC power supply connected with them. The cathode electrode's bottom change is protected by an insulator disk (ceramic) and quartz tube with a diameter of 14.5 cm while the target electrode with an area of 7.5 cm and the distance between them is 3.5 cm. The gas source-flow controller system is responsible for supplying the feedstock at the desired flow rate and gas pressure to the plasma chamber. It is consisting of dual-stage regulators, tubing and fittings for the gas storage cylinders (Argon). The dual-stage needle valve regulated the flow pressure to the plasma chamber. This system consists of a turbopump (Variant.V-1000HT) assisted by a hydraulic rotary pump (60 m³/h. Balzer). The installation of penning (Edward CP25-K with controller 1102) and Perani gauges (LH-Thermovac with Combitron CM350) in the plasma chamber was required to monitor the actual pressure and the partial pressure of the discharge gases.

3. CHARACTERIZATION METHODS

Using the scanning electron microscope (SEM) and its calibrated energy dispersive spectrometer (EDS), the morphologies and chemical compositions of the obtained coating were studied and this test was carried out at the University of Sanjay Sheriff Tehran-Iran. The coating roughness was assessed at the College of Science at the University of Baghdad using an AFM (Model AA3000 Scanning Probe Microscope) image analysis.

4. ANALYSES AND DISCUSSION

1. Characterization of Nb₂O₅-Ni coating

SEM/EDS inspection was performed for coated surfaces to confirm the presence of Nb₂O₅-Ni composite as a coating. Figure 3 shows the typical very small nodular structure. The nodules were uniformly and densely distributed, but pores were still present in the coating. This figure exhibited the deposition of Ni film on carbon steel, while the SEM of Nb₂O₅-Ni coating that deposited by DC sputtering on carbon steel at the size of 200 nm. The growth of crystallite nodules is seen as deposits that are dispersed on the surface of the steel. This obviously indicates a great integration of Nb₂O₅ within the rich nickel. Also, it can be notified that there are two characteristic phases showed within the interface, the first is nodular like solid flakes and the second phase is like homogeneous clusters at the background as also observed by Oluyori and co-workers when they deposited Zn-Nb₂O₅ coatings by electrodeposition technique on mild steel [16]. Ni coating showed some of the agglomerations of particles be caused Ni coatings seem to be denser and compact in comparison with Nb₂O₅-Ni coatings due to faster deposition rate of DC sputtering as also observed by Jahanzeb Bhatti and co-workers when they Investigation of the mechanical properties of electrodeposited nickel and magnetron sputtered chromium nitride coatings deposited on mild steel substrate [17].

EDS analysis of Ni coated carbon steel shows 77 wt% Ni and 23 wt% O confirms the deposition of nickel and the oxygen content is due to oxidizing of some nickel. EDS of Nb₂O₅-Ni coating illustrates the presence of Nb content as 38.1 wt%, The presence of both nickel and oxygen in EDS of composite coatings was attributed to both oxidation and niobium oxide.

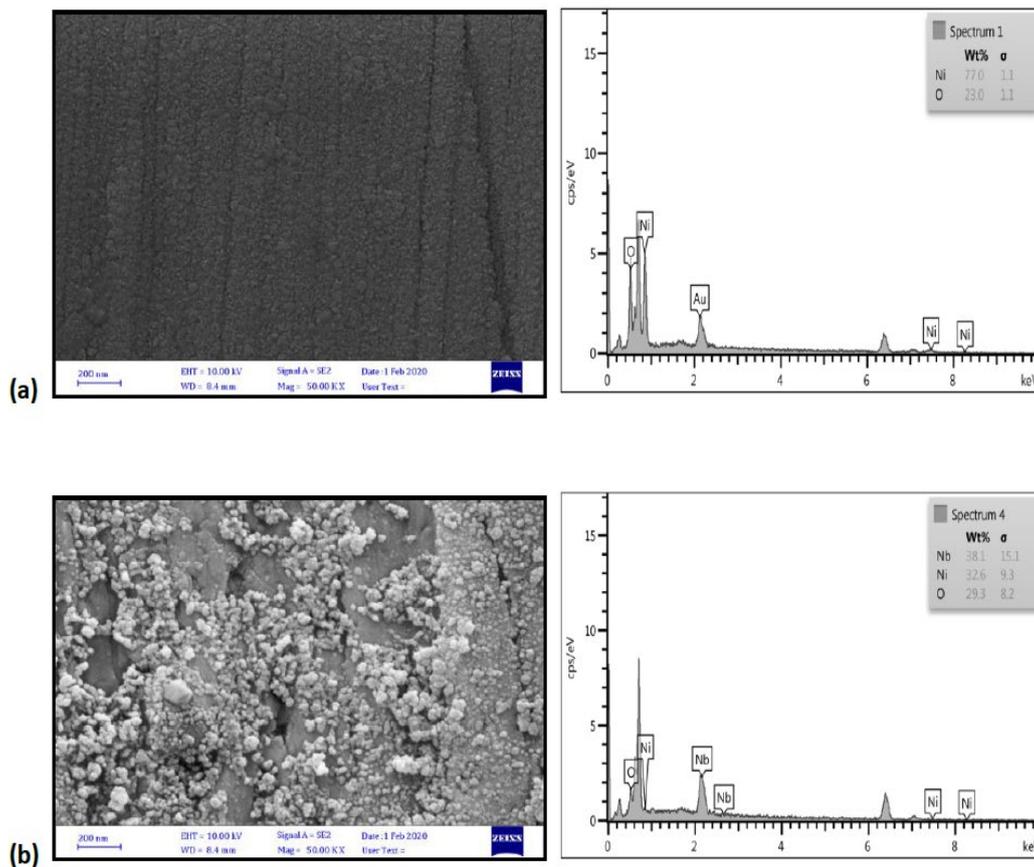


Figure 3: SEM/EDS of coated (a) Ni coated (b) Nb₂O₅-Ni coated.

AFM facilitates surface topography study, including measures of surface roughness. Additionally, because of the rough film shape and size, AFM records topographical images. Therefore, AFM images and force vs. distance graphs are therefore used to provide additional knowledge about surface film properties. Figure 4 illustrated the 2D and 3D images of uncoated and coated steel. Smooth surface can be shown on the uncoated steel surface with very little roughness equal to (3.1 nm). As shown in the TABEL 2. This result could be attributed to atmospheric corrosion and further confirmed by a random distribution of particles as shown in Figure 4 with a very high average diameter of (148.05 nm). For Ni coated surface, this result was a few summits and valleys on the surface that attributed to deposition of Ni particles. In addition to oxidizing some nickel to nickel oxide that gave a roughness average (9.52 nm) with uniform distribution of particle Figure 4. The average diameters equal to (81.13nm). Coating with Nb₂O₅-Ni composites gave a smoother surface compared with Ni coated samples and smoothness increases at increasing weight percent for Nb₂O₅ to the composite as indicated from average roughness was obtained values that listed in TABEL 2. However more average diameter than Ni coating. A decrease in surface roughness is attributed to the Nb₂O₅-incorporated Ni film resulting in the enhancement of grain growth to achieve a large particle size.

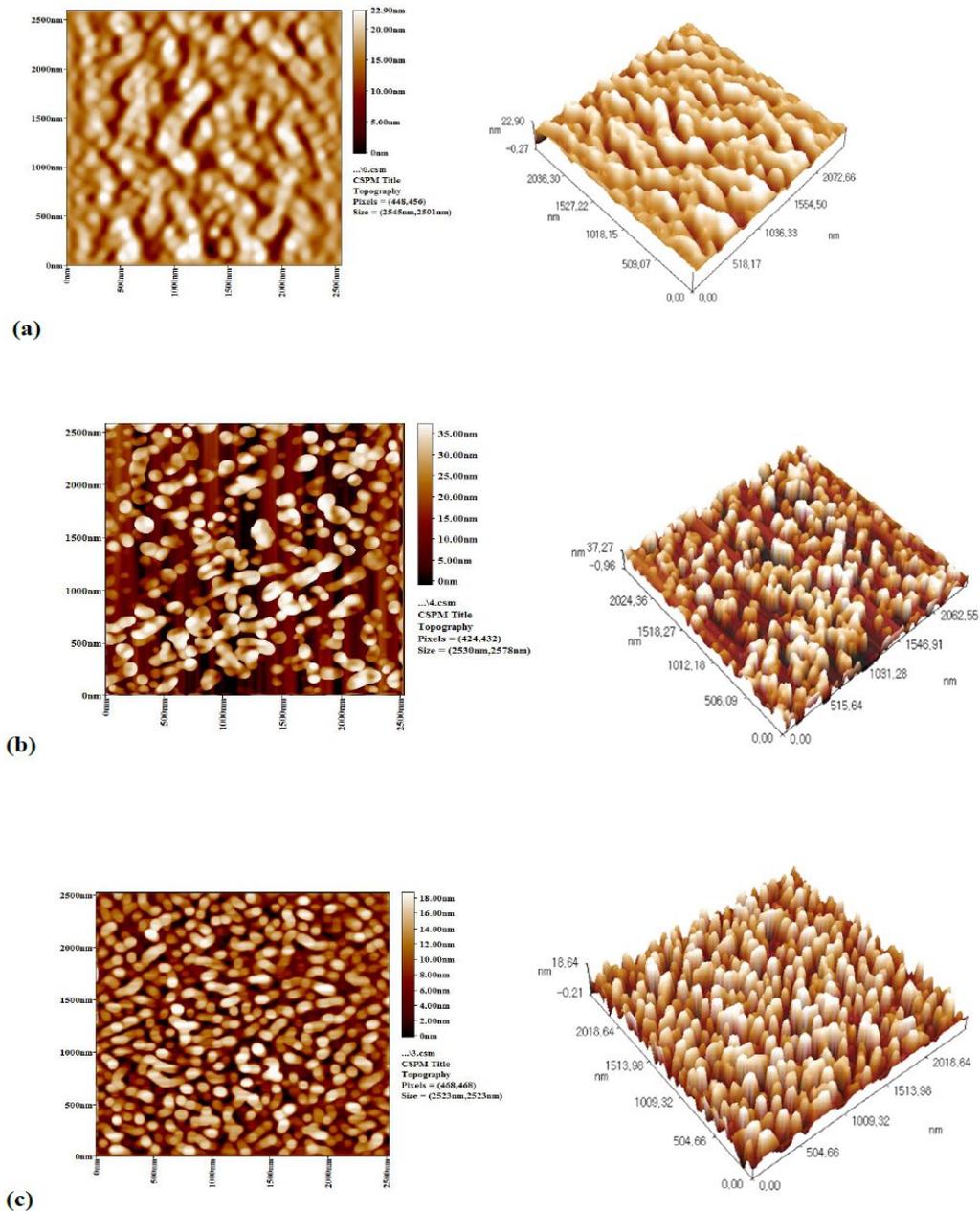


Figure 4: AFM images for (a) uncoated, (b) Ni coated (c) and Nb₂O₅-Ni coated.

TABLE II: AFM data in uncoated and coated samples.

| Material coating | Roughness average (nm) | Average diameter (nm) |
|--|------------------------|-----------------------|
| Uncoated steel sample | 3.01 | 148.05 |
| Ni coating sample | 9.52 | 81.13 |
| Nb ₂ O ₅ -Ni coated sample | 4.53 | 74.44 |

II. Microhardness and Thickness Test

The microhardness test was done on coated specimens and results are listed in TABLE 3. The data shows the higher hardness of the surface Ni coated (155 HV) compared with the uncoated surface (133 HV). While increasing the hardness value of Nb₂O₅-Ni composite coating to 164 HV compared with a hardness value of Ni coating surface. The incorporation of Nb₂O₅ with Ni coating

led to the getting small increase to thickness coating by $\approx 6\mu\text{m}$ as shown in TABLE 3 because of filling any space between Ni particles by Nb_2O_5 particles.

TABLE III: Microhardness and thickness.

| Coating type | Microhardness (HV) | Thickness (μm) |
|---|--------------------|-----------------------------|
| Uncoated steel sample | 133 | --- |
| Ni coating sample | 155 | 5 |
| Nb_2O_5 -Ni coated sample | 164 | 6.4 |

5. CONCLUSIONS

- 1) Ni and Nb_2O_5 -Ni composite coatings were deposited by DC sputtering to get uniformly and densely distributed thin-film with some pores that characterized by SEM/ EDS analysis and AFM examination.
- 2) Microhardness results showed an improvement in hardness value after DC sputtering.
- 3) Deposition other materials with nickel such as TiO_2 -Ni, ZnO-Ni and Al_2O_3 -Ni composite coatings.
- 4) Deposition of Nb_2O_5 -Ni coatings with other techniques such as electrophoretic deposition, thermal spraying and RF sputtering.

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