

## Determination of the effect of NaH<sub>2</sub>PO<sub>4</sub> film obtained on Ti45Nb by sol-gel method on corrosion resistance and surface wettability

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**Abstract** – In this study, NaH<sub>2</sub>PO<sub>4</sub> film was obtained by sol-gel method on Ti45Nb material, which is frequently used in the biomedical field. The aim of this study is to compare the surface wettability and corrosion resistance of the samples untreated Ti45Nb and coated with NaH<sub>2</sub>PO<sub>4</sub>. XRD and SEM were used for the structural characterization of the samples. Surface wettability was determined using a surface tension meter. Electrochemical measurements in simulated body fluid (SBF) solution were performed. Corrosion tests were carried out with the GAMRY Series G750TM Potentiostat/Galvanostat/ZRA device using Potentiodynamic Polarization and the OCP (Open Circuit Potential) Technique (Gamry Instruments, Warminster, USA). According to the results obtained, higher corrosion resistance and better surface wettability were obtained in the NaH<sub>2</sub>PO<sub>4</sub> coated sample.

*Keywords – Hydrophilicity, Sol-Gel, Ti45Nb, Corrosion, Nah<sub>2</sub>po<sub>4</sub>*

### I. INTRODUCTION

The number of biomedical applications is growing as a result of scientific research and technological advancements [1]. Biomedical applications need continuous renewal according to the emerging need [2]. This renewal generally includes studies that aim to eliminate or minimize the disadvantages in biomedical applications. It has been observed that in most studies where the disadvantages of biomedical applications are tried to be eliminated, the mechanical, chemical, electrochemical, or tribological properties of the biocompatible materials used are tried to be improved [3–6].

Titanium and its alloys, which are preferred for use in most biomedical applications, are among the most studied materials [7]. Because titanium and its alloys are preferred due to their low specific gravity, high strength, and self-healing biocompatible oxide layer [8]. Among them, Ti45Nb is often preferred for use in biomedical applications.

However, in some cases, the surface wettability and corrosion resistance of the Ti45Nb alloy may be insufficient [9]. It is desired that the Ti45Nb alloy,

which is preferred to be used especially in biomedical applications integrated into the body, has a long service life, and it is also important to complete the adaptation period more quickly after the integration process with the body [10]. Therefore, it will be of great advantage that the studies to be carried out on Ti45Nb alloys will provide both increasing corrosion resistance and improving surface wettability.

This study, it was aimed to improve both corrosion and surface wettability properties by coating NaH<sub>2</sub>PO<sub>4</sub> on Ti45Nb alloy with the sol-gel method. XRD and SEM were used for the structural characterization of the samples. Surface wettability was determined using a surface tension meter. In addition, Corrosion tests were carried out with the GAMRY Series G750TM Potentiostat/Galvanostat/ZRA device using Potentiodynamic Polarization and the OCP (Open Circuit Potential) Technique (Gamry Instruments, Warminster, USA).

## II. MATERIALS AND METHOD

Ti45Nb samples were polished with 80-1200 mesh SIC abrasives and then all samples were dried after cleaning with alcohol. Prior to the sol-gel process, the following procedures were carried out in the following order: acetone degreasing; alkaline etching in a solution containing 5 g/L Na<sub>2</sub>S and 60 g/L NaOH at 60°C for 2- 3 minutes; dipping in 30%-50% (volume fraction) HNO<sub>3</sub> at room temperature for 1 minute; and finally thorough rinse in deionized water. NaH<sub>2</sub>PO<sub>4</sub> was added to pure water at a concentration of 5 mM, and the peptide amphiphile was placed in each solvent to create a solution with an 80 M PA concentration at 25 °C. Without stirring, the peptide amphiphile was dissolved instantly in the prepared solvent. As a result, the solution needed for the NaH<sub>2</sub>PO<sub>4</sub> thin film coating obtained from the dip-coating is prepared [11].

The GNR-Explorer model X-ray diffractometer was used for the XRD analyses (XRD). XRD characterization was performed using a Cu-K cathode with a wavelength of = 1.54059 Å, and the chemical compositions of the phases formed were determined by comparing the results to the International Centre for Diffraction Data (ICDD) standard cards. The FEI QUANTA 250 scanning electron microscope was used to create surface images of the base material and surface treatment samples. All specimens' contact angles were measured using (Attension Theta Lite C204A).

Electrochemical measurements in simulated body fluid (SBF) solution and SBF content were performed. Corrosion tests were carried out with the GAMRY Series G750TM Potentiostat/Galvanostat/ZRA device using Potentiodynamic Polarization and the OCP (Open Circuit Potential) Technique (Gamry Instruments, Warminster, USA). Polarization readings The Ag/AgCl reference electrode (RE) was created in a corrosion cell using three-electrode techniques, with the graphite rod serving as the counter electrode (CE).

## III. RESULTS

Fig. 1 shows the XRD graphs of untreated and NaH<sub>2</sub>PO<sub>4</sub> coated samples. When the XRD graphs are examined, it is seen that the Ti45Nb sample consists of a β-ti structure. When the XRD graph of the film obtained after the sol-gel surface treatment is examined, it is seen that the NaH<sub>2</sub>PO<sub>4</sub> structure is formed on the surface of Ti45Nb [12].

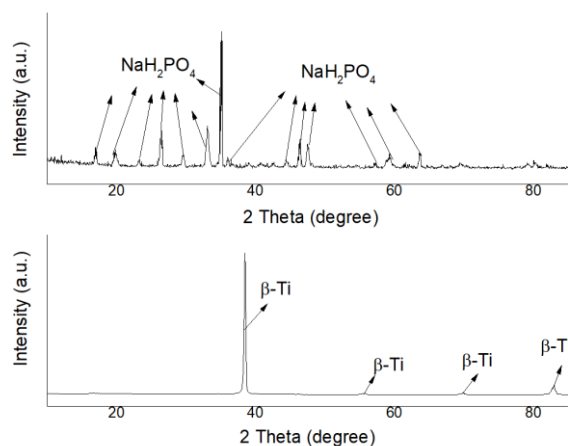


Fig. 1 XRD graphs untreated Ti45Nb and NaH<sub>2</sub>PO<sub>4</sub> coated sample.

Top view SEM images of untreated Ti45Nb and NaH<sub>2</sub>PO<sub>4</sub> coated samples and cross-section SEM image of NaH<sub>2</sub>PO<sub>4</sub> coated sample are given in Fig. 2. In the untreated sample, only the traces left after the partitioning process are observed. In the NaH<sub>2</sub>PO<sub>4</sub> coated sample, it is observed that the morphological structure of the surface changes after the sol-gel process. When the cross-sectional image of the NaH<sub>2</sub>PO<sub>4</sub> film is examined, it can be said that the film has a homogeneous structure.

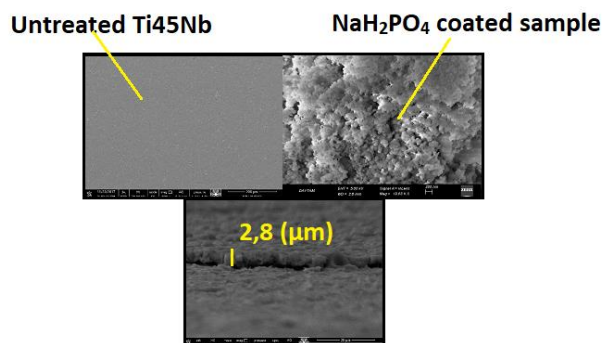


Fig. 2 SEM images of untreated Ti45Nb and NaH<sub>2</sub>PO<sub>4</sub> coated sample.

The contact angle images of untreated Ti45Nb and coated samples are given in Fig. 3. As shown in Fig. 3, Ti45Nb surface wettability increased after the sol-gel process. The contact angle of the untreated sample was approximately 53°, while the contact angle of the NaH<sub>2</sub>PO<sub>4</sub> coated sample was measured to be approximately 20°. According to the results obtained, it can be said that the surface wettability improves after the applied surface treatment [13].

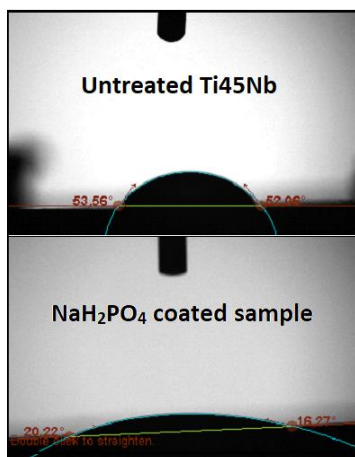


Fig. 3 The contact angle of untreated Ti45Nb and NaH<sub>2</sub>PO<sub>4</sub> coated sample.

Open circuit potentials and potentiodynamic polarization curves graphs of untreated Ti45Nb and coated samples are given in Fig.4 and Fig.5 respectively. When the graphs are examined, it is seen that the corrosion resistance increases thanks to the film obtained on Ti45Nb. The resulting film prevented the SBF liquid from contacting Ti45Nb, which led to an increase in corrosion resistance. When the studies conducted on titanium and its alloys in the literature were examined [14], it was emphasized that the films obtained showed a barrier effect and thus provided an increase in corrosion resistance.

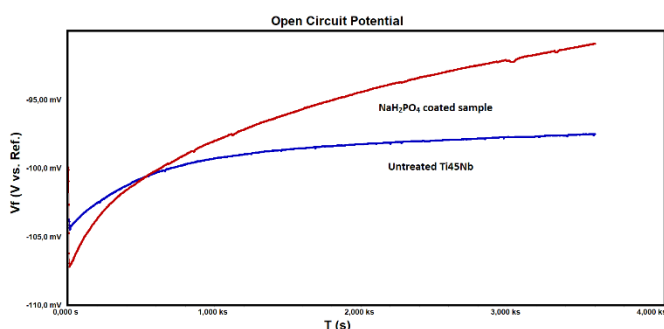


Fig. 4 Open circuit potentials of untreated Ti45Nb and NaH<sub>2</sub>PO<sub>4</sub> coated sample.

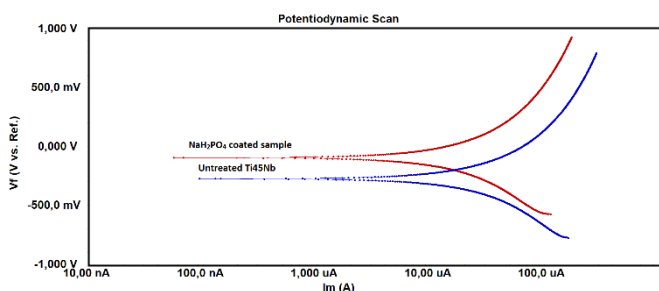


Fig. 5 Potentiodynamic polarization curves of untreated Ti45Nb and NaH<sub>2</sub>PO<sub>4</sub> coated sample.

#### IV. DISCUSSION

In this study, NaH<sub>2</sub>PO<sub>4</sub> was coated on Ti45Nb alloy by the sol-gel method and the surface wettability and corrosion resistance were examined after examining the structural analysis. According to the results obtained, when the XRD graphics were examined, NaH<sub>2</sub>PO<sub>4</sub> coating was successfully obtained on the Ti45Nb surface after the sol-gel procedure. When the SEM images are examined, it is seen that the surface morphology changes after the applied surface treatment, and the structural formations of NaH<sub>2</sub>PO<sub>4</sub> can be seen. It has been seen that these structures increase surface wettability in contact angle analyses. Electrochemical experiments showed that the resulting film reduces the contact between SBF liquid and Ti45Nb surface, resulting in an improvement in corrosion resistance.

#### V. CONCLUSION

As a result, the film obtained by the sol-gel method on the Ti45Nb surface changed the surface morphology. Changing surface morphology both increased surface wettability and improved corrosion resistance.

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#### REFERENCES

- [1] M. Niinomi, Recent research and development in titanium alloys for biomedical applications and healthcare goods, *Sci. Technol. Adv. Mater.* 4 (2003) 445.
- [2] C.N. Elias, J.H.C. Lima, R. Valiev, M.A. Meyers, Biomedical applications of titanium and its alloys, *Jom.* 60 (2008) 46–49.
- [3] O. Çomaklı, M. Yazıcı, M. Demir, A.F. Yetim, A. Çelik, Effect of bilayer numbers on structural, mechanical, tribological and corrosion properties of TiO<sub>2</sub>-SiO<sub>2</sub> multilayer film-coated  $\beta$ -type Ti45Nb alloys, *Ceram. Int.* 49 (2023) 3007–3015.
- [4] V. Sawyer, X. Tao, H. Dong, B. Dashtbozorg, X. Li, R. Sammons, H.-S. Dong, Improving the tribological properties and biocompatibility of Zr-based bulk metallic glass for potential biomedical applications, *Materials.* 13 (2020) 1960.
- [5] S. Prakash, A. Favas, I. Ameeth Basha, R. Venkatesh, M. Prabhakar, V.P. Durairaj, K. Gomathi, H. Lenin, Investigation of Mechanical and Tribological Characteristics of Medical Grade Ti6Al4V Titanium Alloy in Addition with

Corrosion Study for Wire EDM Process., *Adv. Mater. Sci. Eng.* (2022).

[6] W. Yang, D. Xu, Y. Gao, L. Hu, P. Ke, J. Chen, Microstructure, adhesion, in vitro corrosion resistance and tribological behavior of (Si: N)-DLC coated pure Ti, *Diam. Relat. Mater.* 92 (2019) 109–116.

[7] X. Liu, P.K. Chu, C. Ding, Surface modification of titanium, titanium alloys, and related materials for biomedical applications, *Mater. Sci. Eng. R Rep.* 47 (2004) 49–121.

[8] M. Geetha, A.K. Singh, R. Asokamani, A.K. Gogia, Ti based biomaterials, the ultimate choice for orthopaedic implants—a review, *Prog. Mater. Sci.* 54 (2009) 397–425.

[9] S. Laketić, M. Rakin, M. Momčilović, J. Ciganović, \DJor'dje Veljović, I. Cvijović-Alagić, Influence of laser irradiation parameters on the ultrafine-grained Ti45Nb alloy surface characteristics, *Surf. Coat. Technol.* 418 (2021) 127255.

[10] W.K. Czaja, D.J. Young, M. Kawecki, R.M. Brown, The future prospects of microbial cellulose in biomedical applications, *Biomacromolecules.* 8 (2007) 1–12.

[11] T. Otsuka, T. Maeda, A. Hotta, Effects of Salt Concentrations of the Aqueous Peptide-Amphiphile Solutions on the Sol–Gel Transitions, the Gelation Speed, and the Gel Characteristics, *J. Phys. Chem. B.* 118 (2014) 11537–11545. <https://doi.org/10.1021/jp5031569>.

[12] M. Banach, Z. Kowalski, Z. Wzorek, K. Gorazda, A chemical method of the production of “heavy” sodium tripolyphosphate with the high content of Form I or Form II, *PJCT.* 11 (2009) 13–20. <https://doi.org/10.2478/v10026-009-0018-x>.

[13] A. Çelik, M.T. Acar, T. Yetim, H. Kovacı, A.F. Yetim, Improving structural, tribological and electrochemical properties of Ti6Al4V alloy with B-doped TiO<sub>2</sub> thin films, *Tribol. Int.* 146 (2020) 106210.

[14] O. Çomaklı, Improved structural, mechanical, corrosion and tribocorrosion properties of Ti45Nb alloys by TiN, TiAlN monolayers, and TiAlN/TiN multilayer ceramic films, *Ceram. Int.* 47 (2021) 4149–4156.