وزارة التعليم العالي والبحث العلمي
جامعة ميسان
كلية التربية الاساسية

Misan jas
Misan Journal of Academic Studies

مجلة ميسان للدراسات الاكاديمية

العلوم الانسانية والاجتماعية والتطبيقية

ISSN (Paper)- 1994-697X
(Online)- 2706-722X

المجلد 22 العدد 48 السنة 2023

مجلة ميسان للدراسات الاكاديمية

العلوم الانسانية والاجتماعية والتطبيقية

كلية التربية الاساسية - جامعة ميسان - العراق

ISSN (Paper)- 1994-697X
(Online)- 2706-722X

مجلد (22) العدد (48) كانون الاول (2023)

INTERNATIONAL STANDARD SERIAL NUMBER INTERNATIONAL CENTRE

OJS / PKP
www.misan-jas.com

IRAQI Academic Scientific Journals

http://www.misan-jas.com/index.php/ojs

journal.m.academy@uomisan.edu.iq

رقم الايداع في دار الكتب والوثائق بغداد 1326 في 2009

ISSN (Paper) 1994-697X

Online 2706 -722X

https://doi.org/10.54633/2333-022-048-020

Analysis of the surface hardness of niobium carbide coatings deposited on commercially pure titanium and Ti-6Al-7Nb alloy implant materials using the glow discharge plasma technique

Haitham T. Al Qaysi [1], Thekra I. Hamad [2], Thair L. Al Zubaidy [3]

[1] Department of Prosthodontics (Ph.D student), University of Baghdad / College of dentistry, Baghdad 1417, IRAQ.
[2] Department of Prosthodontics, University of Baghdad / College of dentistry, Baghdad 1417, IRAQ.
[3] Department of Prosthetic Dental Techniques, AlEsraa University/ College of Health and Medical Techniques, Baghdad 4001, IRAQ.
Corresponding author: Htadental1979@gmail.com

ORCID ID: Haitham T. Al Qaysi (0000-0002-8908-9851) (orcid.org)

Abstract:

To enhance the properties of titanium and its alloys for implant applications, NbC thin films were deposited on commercially pure titanium and Ti-6Al-7Nb specimens using a low-temperature plasma glow discharge technique with argon gas and C_2H_2 as a carbon source. Vicker's hardness tests were conducted, revealing significantly increased hardness in the coated samples, a finding supported by statistical analysis. This increased surface hardness will potentially improve the wear resistance and load-bearing capabilities, which are essential factors for dental and orthopedic implants. The study aligns with previous research on NbC's properties, positioning it as a promising material for dental implants. Importantly, the study underscores the reliability of the coating technique for implant surface modification.

Keywords: Niobium carbide; Thin films; Plasma glow discharge; Titanium; Surface hardness.

1. Introduction:

Titanium and its alloys are commonly employed in clinical applications as implant materials due to their ability to offer a favorable mechanical characteristic, resistance to corrosion, and biocompatibility (Prasad *et al.*, 2015). Dental or orthopedic implants are designed for long-term placement within the body, protecting such implants against deterioration and damage within the harsh conditions of the bodily fluids is a challenging clinical concern (Freeman and Brook, 2006). One potential solution to address these challenges is the application of a thin, durable coating onto the titanium surface. This coating serves to safeguard titanium from oxidation and enhance its surface hardness, potentially address these issues (Ferro *et al.*, 2004).

The technique of low-temperature plasma glow discharge provides a method for modifying the surfaces of metallic biomaterials with minimal impact on the underlying substrate's microstructure (Sobiecki and Wierzchoń, 2005; Jabur, Hammed and Khalaf, 2021). This approach offers precise control over the development of thin coatings, including the ability to tailor their structure and chemical composition, thereby influencing their properties. Furthermore, it facilitates the treatment of surfaces of complex-shaped components (Czyrska-Filemonowicz *et al.*, 2005).

Niobium carbide (NbC) has gained attention in the field of dental implant materials due to its outstanding combination of properties. This includes impressive chemical stability (Amriou *et al.*, 2003), high hardness (Nedfors *et al.*, 2011), resistance to wear and corrosion, and satisfactory biocompatibility. Recent research has focused on depositing NbC films onto 316L stainless steel using a non-reactive setup, revealing promising osteogenic potential and protective qualities. These findings make NbC an appealing choice for dental implant applications (Xu *et al.*, 2019). Moreover, studies involving the application of NbC coatings onto Ti-6Al-4V through DC magnetron sputtering have demonstrated excellent corrosion resistance and biocompatibility, further emphasizing its potential in the field. Therefore, NbC stands out as a promising material for dental implants due to its remarkable properties and promising research outcomes (Braic *et al.*, 2011).

2. Materials and methods:

2.1. Substrate preparation:

For this study, titanium grade II (ASTM f67) and Ti-6Al-7Nb (ASTM F1295) were utilized as substrate materials (Hamad *et al.*, 2018). Commercially pure Titanium (CpTi) rods were cut into disks (Ø 13mm, 5mm thick); while, Ti-6Al-7Nb block was cut into rectangular shapes measuring 12mm by 10mm and with a thickness of 5mm using wire cut machine (Knuth SmartDEM-Germany) (Al-Khafaji and Hamad, 2021). The preparation of the specimens achieved using a sequence of abrasives with grit sizes of 320, 400, 600, 800, and 1000 (SiC paper) in a polishing machine (Wierzchoń *et al.*, 2015). Subsequently, the samples underwent two rounds of sonication, each lasting 10 minutes, successively in acetone, ethanol, and distilled water. After this cleaning process, the smoothed discs were left to air dry for 15 minutes at room temperature (Boyd *et al.*, 2015; Al-Khafaji and Hamad, 2021).

2.2. NbC coating conditions:

The NbC coatings were intended to be produced in three groups: group-1 with (2hrs) deposition time, group-2 with (4hrs) deposition time, group-3 with (6hrs) deposition time.

The NbC coatings were applied onto specimens utilizing a pure Nb target (99.95%) by a plasma glow discharge sputtering process. This process involved the use of Argon gas as an inert gas (99.999%) and C_2H_2 (99.999%), as reactive gas. The distance between the target and the substrate holder was 50 mm. Both a rotary pump and a turbo-molecular pump were employed to evacuate the chamber to a pressure of $1*10^{-4}$ prior to sputtering. The substrate was sputter-cleaned before deposition (Xu *et al.*, 2019) by Ar gas at (25 W) and $5*10^{-2}$ Pa for 1 min to remove the surface oxide layer and any contaminants and prevent any further deposition of Nb; the Ar gas was then stopped. The pressure in the chamber was restored to $1*10^{-4}$ Pa. Acetylene gas C_2H_2 was introduced into the chamber until pressure was maintained at $1*10^{-2}$ Pa. Then, Ar gas was introduced until pressure was maintained at $6*10^{-2}$ Pa, the DC voltage was set at 3.7 Kv and the current was set at 7 mA to achieve a sputtering energy of (25 Watt) which is the material-dependent ion energy (Liu, Chu and Ding, 2004).

www.misan-jas.com ISSN (Paper)-1994-697X (Online)-2706-722X

2.3. Vicker`s hardness measurements:

We used a Vickers micro-hardness tester (Laryee Technology Co. Ltd./China), as illustrated in (Figure 1A). This instrument was employed to measure the surface hardness of specimens with NbC coating (the experimental group) and the uncoated substrates, which served as the control group (Figure 1B). To apply a consistent force, we used a 100-gram weight, resulting in a force of 0.98 N, and maintained this load for a duration of 15 seconds (referred to as "DWELL"). For each sample, we performed three hardness measurements, and the average of these measurements was recorded as the final hardness reading for that specific sample. In total, we tested ten samples for both the coated and uncoated variables for both CpTi and Ti-6Al-7Nb, making a total of 40 samples in the study.

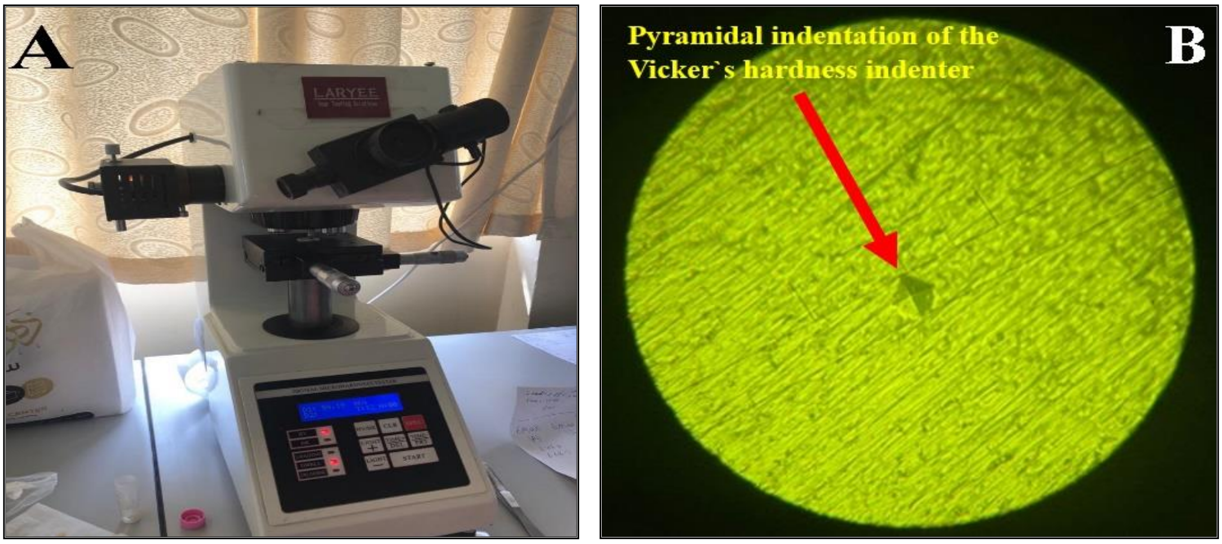

Figure 1: A. The Vicker`s hardness measurement device. B. The pyramidal indentation after loading the surface.

3. Results:

The results show a significant increase in hardness for the coated samples in comparison to the uncoated ones, and this difference has been statistically confirmed using a t-test. More detailed information and the statistical analysis in (Table 1). Additionally, (Figure 2) provides a bar chart illustrating the descriptive statistics and the results of the t-test analysis for the hardness measurements under different experimental conditions.

Table 1: The hardness measurements and the results of the t-test for both CpTi and Ti-6Al-7Nb.

Hardness	Group	N	Mean	SD	t	df	Sig. (2-tailed)
CpTi	Uncoated	10	205.27	14.11	-30.136	18	0.000*
	Coated	10	436.29	19.71			
Ti-6Al-7Nb	Uncoated	10	292.57	7.22	-43.098	18	0.000*
	Coated	10	483.60	12.01			

www.misan-jas.com
ISSN (Paper)-1994-697X (Online)-2706-722X

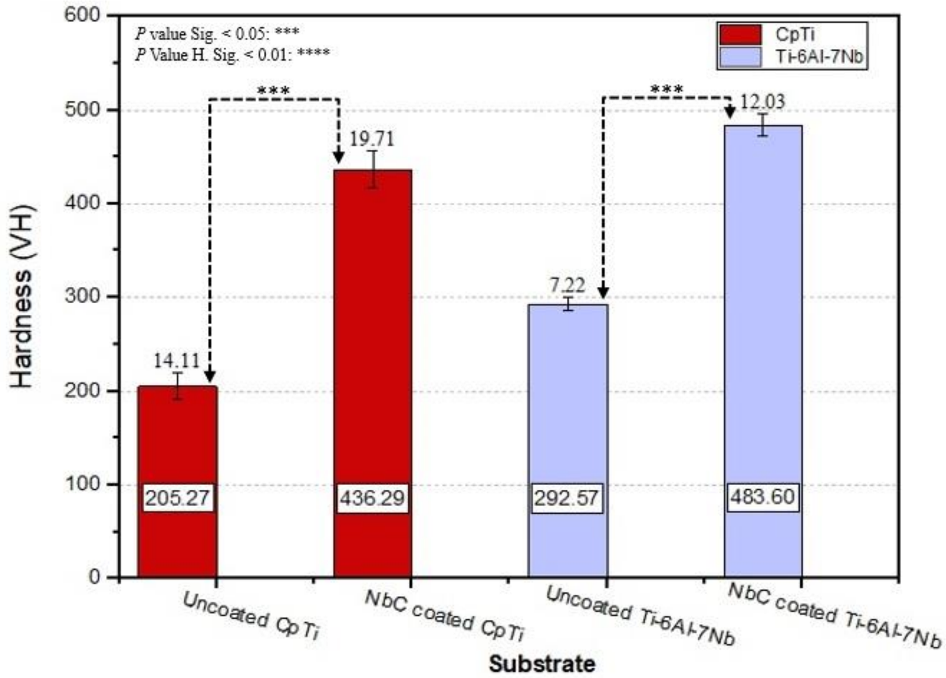

Figure 2: Bar chart demonstrates the descriptive statistics and t-test analysis of hardness measurements for different experimental conditions.

4. Discussion:

The increase in surface hardness observed in the coated specimens has several important implications for practical applications. Firstly, improved hardness is often associated with enhanced wear resistance (Sen, 2004, 2005). In conditions where materials are subject to friction and abrasion, such as dental or orthopedic implants, a harder surface is less prone to wear and, consequently, offers increased durability and reliability in performance (Sen, 2005; Cai and Xu, 2017). This attribute is particularly crucial in situations where materials frequently experience mechanical stresses and come into contact with opposing surfaces (Mohammed and Hamad, 2021).

Secondly, the increased hardness can also result in improved load-bearing capabilities, as pointed out by (Ramezani and Ripin, 2023). In applications where materials must bear heavy loads, such as implants or prosthetics, surface hardness plays a significant role in the material's ability to withstand external forces without deforming or failing prematurely. A harder surface can effectively distribute applied loads, reducing the risk of bulk material breakdown before its expected lifespan (Ibrahim *et al.*, 2017; Moghadasi *et al.*, 2022).

The observed increase in hardness in the NbC-coated specimens aligns with findings in relevant references. For instance, Zhang et al. in 2014 explored the tribological and mechanical properties of sputtered niobium carbide coatings, highlighting niobium carbide's inherent hardness due to strong atomic bonding and crystalline structure (Zhang *et al.*, 2014). Additionally, a study by Xu et al. in 2019 investigated the biocompatibility and mechanical properties of NbC/a-C:H coatings on 316L stainless steel, demonstrating the correlation between hardness and wear resistance (Xu *et al.*, 2019), a concept consistent with the idea that a harder surface is less susceptible to wear (Cai and Xu, 2017).

However, it's important to acknowledge certain complexities that may deviate from these discussed implications. Cai et al. in 2016 highlighted in their study that while hardness and wear resistance can improve with coatings, the presence of a coating can introduce new considerations, such as coating adhesion and the potential for coating delamination (Cai *et al.*, 2016). This suggests

www.misan-jas.com ISSN (Paper)-1994-697X (Online)-2706-722X

that while enhanced hardness offers advantages, careful attention must be paid to the coating-substrate interface to ensure optimal performance and durability in practical applications.

5. Conclusion:

In conclusion, this research highlights the significant potential of niobium carbide (NbC) coatings to enhance the properties of titanium and its alloys for clinical implant applications. The process of applying NbC coatings using the reactive glow discharge plasma indicates the efficacy of the deposition technique as a viable method for surface modification of implant materials. The study underscores the implications of increased surface hardness of the deposited NbC thin films with the potential enhancement of wear resistance and load-bearing capabilities, positioning it as a highly attractive material for dental implant applications and other clinical uses. Overall, this research contributes valuable insights into the potential benefits of NbC coatings, offering improved performance and durability for titanium-based implants in various clinical applications.

References:

Al-Khafaji, A.M. and Hamad, T.I. (2021) ‘Surface Analysis of the PEKK Coating on the CP Ti Implant Using Laser Technique’, *International Medical Journal*, 28(1), pp. 29–32. https://www.researchgate.net/profile/Aseel-Al-Khafaji/publication/352002071

Amriou, T. *et al.* (2003) ‘FP-LAPW investigations of electronic structure and bonding mechanism of NbC and NbN compounds’, *Physica B: Condensed Matter*, 325, pp. 46–56. https://doi.org/10.1016/S0921-4526(02)01429-1

Boyd, A.R. *et al.* (2015) ‘Strontium-substituted hydroxyapatite coatings deposited via a co-deposition sputter technique’, *Materials Science and Engineering: C*, 46, pp. 290–300. https://doi.org/10.1016/j.msec.2014.10.046

Braic, M. *et al.* (2011) ‘Preparation and characterization of biocompatible Nb–C coatings’, *Thin Solid Films*, 519(12), pp. 4064–4068. https://doi.org/10.1016/j.tsf.2011.01.193

Cai, X. *et al.* (2016) ‘Investigation of the adhesion strength and deformation behaviour of in situ fabricated NbC coatings by scratch testing’, *Surface and Coatings Technology*, 299, pp. 135–142. https://doi.org/10.1016/j.surfcoat.2016.05.004

Cai, X. and Xu, Y. (2017) ‘Microstructure, friction and wear of NbC coatings on a Fe substrate fabricated via an in situ reaction’, *Surface and Coatings Technology*, 322, pp. 202–210. https://doi.org/10.1016/j.surfcoat.2017.05.046

Czyrska-Filemonowicz, A. *et al.* (2005) ‘Transmission electron microscopy and atomic force microscopy characterisation of titanium-base alloys nitrided under glow discharge’, *Acta Materialia*, 53(16), pp. 4367–4377. https://doi.org/10.1016/j.actamat.2005.05.035

Ferro, D. *et al.* (2004) ‘Hardness of electron beam deposited titanium carbide films on titanium substrate’, *Journal of materials science*, 39(1), pp. 329–330. https://d1wqtxts1xzle7.cloudfront.net/45340311/Hardness_of_electron_beam_deposited_tita20160504-6279-1rwiftq-libre.pdf

Freeman, C.O. and Brook, I.M. (2006) ‘Bone response to a titanium aluminium nitride coating on metallic implants’, *Journal of Materials Science: Materials in Medicine*, 17, pp. 465–470. https://doi.org/10.1007/s10856-006-8474-9

ISSN (Paper)-1994-697X (Online)-2706-722X

Hamad, T.I. *et al.* (2018) 'Biomechanical evaluation of nano-zirconia coatings on Ti-6Al-7Nb implant screws in rabbit tibias', *Current medical science*, 38, pp. 530–537. https://doi.org/10.1007/s11596-018-1911-4

Ibrahim, M.Z. *et al.* (2017) 'Biomedical materials and techniques to improve the tribological, mechanical and biomedical properties of orthopedic implants–A review article', *Journal of Alloys and Compounds*, 714, pp. 636–667. DOI: https://doi.org/10.1016/j.jallcom.2017.04.231

Jabur, Y.K., Hammed, M.G. and Khalaf, M.K. (2021) 'DC glow discharge plasma characteristics in Ar/O2 gas mixture', *Iraqi Journal of Science*, pp. 475–482. https://doi.org/10.24996/ijs.2021.62.2.13

Liu, X., Chu, P.K. and Ding, C. (2004) 'Surface modification of titanium, titanium alloys, and related materials for biomedical applications', *Materials Science and Engineering: R: Reports*, 47(3–4), pp. 49–121. https://doi.org/10.1016/j.mser.2004.11.001

Moghadasi, K. *et al.* (2022) 'A review on biomedical implant materials and the effect of friction stir based techniques on their mechanical and tribological properties', *Journal of Materials Research and Technology*, 17, pp. 1054–1121. https://doi.org/10.1016/j.jmrt.2022.01.050

Mohammed, A.A. and Hamad, T.I. (2021) 'Assessment of Coating Zirconium Implant Material with Nanoparticles of Faujasite', *Journal of Baghdad College of Dentistry*, 33(4), pp. 25–30. https://doi.org/10.26477/jbcd.v33i4.3016

Nedfors, N. *et al.* (2011) 'Structural, mechanical and electrical-contact properties of nanocrystalline-NbC/amorphous-C coatings deposited by magnetron sputtering', *Surface and Coatings Technology*, 206(2–3), pp. 354–359. https://doi.org/10.1016/j.surfcoat.2011.07.021

Prasad, S. *et al.* (2015) 'Biomaterial properties of titanium in dentistry', *Journal of oral biosciences*, 57(4), pp. 192–199. https://doi.org/10.1016/j.job.2015.08.001

Ramezani, M. and Ripin, Z.M. (2023) 'An Overview of Enhancing the Performance of Medical Implants with Nanocomposites', *Journal of Composites Science*, 7(5), p. 199. https://doi.org/10.3390/jcs7050199

Sen, U. (2004) 'Kinetics of niobium carbide coating produced on AISI 1040 steel by thermo-reactive deposition technique', *Materials Chemistry and Physics*, 86(1), pp. 189–194. https://doi.org/10.1016/j.matchemphys.2004.03.002

Sen, U. (2005) 'Wear properties of niobium carbide coatings performed by pack method on AISI 1040 steel', *Thin Solid Films*, 483(1–2), pp. 152–157. https://doi.org/10.1016/j.tsf.2005.01.008

Sobiecki, J.R. and Wierzchoń, T. (2005) 'Glow discharge assisted oxynitriding of the binary Ti6Al2Cr2Mo titanium alloy', *Vacuum*, 79(3–4), pp. 203–208. https://doi.org/10.1016/j.vacuum.2005.03.008

Wierzchoń, T. *et al.* (2015) 'Glow discharge assisted oxynitriding process of titanium for medical application', *Applied Surface Science*, 334, pp. 74–79. https://doi.org/10.1016/j.apsusc.2014.08.071

Xu, Z. *et al.* (2019) 'Potential of niobium-based thin films as a protective and osteogenic coating for dental implants: The role of the nonmetal elements', *Materials Science and Engineering: C*, 96, pp. 166–175. https://doi.org/10.1016/j.msec.2018.10.091

Zhang, K. *et al.* (2014) 'Reactive magnetron sputtering deposition and characterization of niobium carbide films', *Vacuum*, 99, pp. 233–241. https://doi.org/10.1016/j.vacuum.2013.06.012