

Enhanced Tribocorrosion Resistance of 3D Printed Ti- Implants: PVD multilayer coating on PEO-Treated Surface

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INTRODUCTION: Recently, significant progress has been made in using 3D printed Porous Ti-6Al-4V implants in orthopedics. Despite many advantages offered by the 3D printed implants, such as anatomical design and lower mismatch in the elastic modulus with bone, they face some challenges in tribocorrosion resistance under micro-movements and corrosive physiological environments¹. This undesirable tribocorrosion performance leads to the release of metal particles and ions, causing local and systemic toxicity, inflammation, adverse tissue reaction, and finally implant failure². To address this issue, surface treatment through the deposition of coatings by various technologies has been proposed. Recently, the strategy of deposition of hybrid coatings to fabricate multifunctional implants has been of interest³. For example, plasma electrolyte oxidation (PEO) techniques to develop porous TiO₂ on the surface of Ti-based alloy implants for improving the biological properties, increasing the adhesion of the top layer, and enhancing the load-bearing, followed by deposition of hard nitride layer by physical vapor deposition (PVD) methods to enhance the tribocorrosion of samples through covering the micropores induced by the PEO method, have been used⁴. Therefore, in this study, based on the results of our previous study⁵, where the PEO-treated samples with two layers of Niobium/Niobium nitride (Nb/NbN) Nb/NbN coating could exhibit superior wear behavior and decreased wear rate by 90% compared to untreated samples, we aimed to investigate the effect of TiO₂/2-layer Nb/NbN coatings on the tribocorrosion properties of samples. We hypothesized that applying TiO₂ and Nb/NbN multilayer coating by hybrid methods of coating, including PEO followed by PVD, will increase the tribocorrosion performance of Ti-6Al-4V implants.

METHODS: Ti-6Al-4V porous implants with gyroid structure were designed and fabricated via laser powder bed fusion (LPBF). Based on our previous work⁵, the implants were treated with PEO and then coated with a multilayer Nb/NbN film using PVD (**Figure 1a**). The tribocorrosion test was conducted using a hip simulator system⁶ (**Figure 1b**). Samples were loaded against the alumina (Al₂O₃) ball, rotated ±30° in an oscillatory pattern in the bovine-calf serum (BCS, 30 g/L of protein concentration) under the normal load of 16±1 N (to achieve the contact pressure of 50 MPa). The test was performed with a frequency of 1 Hz for 3600 s. Tribocorrosion tests were performed under open circuit potential (OCP) and potentiostatic (PS) conditions. For the PS tests, the potential value of -0.1 V was applied. The wear-corrosion synergistic analysis was done, as per Stack's approach; $K_{wc} = K_w + K_c$, where K_{wc} , K_w , and K_c represent the total weight loss of material due to tribocorrosion wear, and corrosion, respectively⁷. The worn surfaces were examined by scanning electron microscopy (SEM, JOEL, MA, USA).

RESULTS: The PEO/PVD-treated samples demonstrated increased tribocorrosion performance under OCP conditions, evidenced by a lower potential drop and coefficient of friction (COF) value (**Figure 1c**). Moreover, the PEO/PVD treatment reduced corrosion current and COF of samples during the tribocorrosion test under PS conditions, which indicates higher tribocorrosion performance and the anti-friction performance of the coatings (**Figure 1d**). The total weight loss of material due to tribocorrosion (K_{wc}), wear (K_w), and corrosion (K_c) for PEO/PVD-treated was lower than that of others. Lower K_w value obtained for PEO/PVD-treated was related to their higher wear resistance. The K_c values of all samples were significantly lower than the K_w values, indicating minimal effect of corrosion in the synergistic degradation process. All samples exhibited abrasive and adhesive wear mechanisms based on the SEM images of the worn surface after tribocorrosion. The grooves were shallower and narrower for PEO/PVD-treated samples, indicating their higher wear resistance than others. However, both PVD-treated and PEO/PVD-treated samples exhibited the coating delamination mechanism (**Figure 1e**).

DISCUSSION: This study aimed to improve the tribocorrosion performance of porous Ti-6Al-4V implants by applying PEO-TiO₂ and PVD-2 layers of Nb/NbN coatings. The results confirmed this hypothesis and showed that deposition of PEO/PVD multilayer coatings on Ti-6Al-4V implants could successfully enhance tribocorrosion performance. A porous TiO₂ layer developed by PEO, as a load-bearing interlayer, and the PVD-Nb/NbN layers, as a hard and wear-resistant coating, improved the tribocorrosion performance of samples. Moreover, as shown in our previous study, deposition of two layers of Nb/NbN on PEO-treated samples by filling PEO-induced micropores and decreasing surface roughness decreased the wear rate of samples, which positively affected the enhancement of tribocorrosion properties. Based on the results, the PEO/PVD-treated samples showed the lowest potential drop under OCP conditions and the lowest current under PS conditions, and reduced the COF in both conditions. Hence, this approach may be a promising idea for future implant designs to enhance the lifetime of titanium-based implants under tribocorrosion conditions. The study has many limitations, as it is considered only a short-duration test; further long-term tests will be conducted together with biological evaluation of the metal products.

SIGNIFICANCE: This study addresses the challenges related to the tribocorrosion performance of Ti-6Al-4V porous implants, proposing a solution by

depositing PEO/PVD coatings. However, further research is needed to optimize these coatings and evaluate their long-term application under tribocorrosion conditions. Moreover, antibacterial agents can be added to the coating to minimize inflammation from the degradation products.

ACKNOWLEDGEMENTS:

Blazer Foundation and UIC International Office.

REFERENCES: [1] Cheng *et al.*,2019, [2] Costa *et al.*,2023, [3] Kumar *et al.*,2024, [4] Zhang *et al.*,2022, [5] Ebrahimi *et al.*,2024, [6] Mathew *et al.*,2011, [7] Stack *et al.*,2005.

Figure: 1a) The LPBF Ti-6Al-4V porous implants with different surface modifications. **b)** Hip simulator schematic, **c)** OCP and COF curves of samples during sliding in BCS at 37°C, **d)** Current and COF curves of samples during sliding in BCS at 37°C, **e (e1-e8))** SEM images of worn area after tribocorrosion test.

