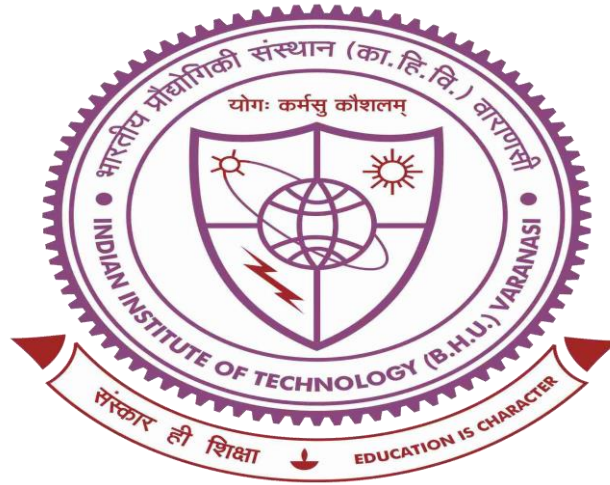


# **Wear, Corrosion, and Biocompatibility of Ta-Coated 316L Stainless Steel for Orthopedic Applications**



*Thesis submitted in partial fulfillment for the  
Award of Degree*

**DOCTOR OF PHILOSOPHY**

By

**DILEEP PATHOTE**

Department of Metallurgical Engineering  
Indian Institute of Technology  
(Banaras Hindu University)  
Varanasi-221005  
INDIA

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## 7.1 Summary

Austenitic stainless steel (grade 316L) was coated with the Tantalum target (99.95%) with varying coating thicknesses by the Direct Current Magnetron Sputtering (DCMS) System. To achieve high-quality coatings, argon (99.99%) gas was used, and proper monitoring of process parameters as per standard procedure to obtain strong adhesion between the tantalum coating and the 316L stainless-steel substrate was also followed.

The **pin-on-disc** wear test was performed on a Ducom (**TR-701**) at 37°C in a Simulated Body Fluid (**SBF**) environment. **Microhardness** and **elastic modulus** of austenitic stainless-steel sample 316L SS and Ta-coated 316L SS were evaluated. The **Electrochemical Corrosion** measurements, using Open Circuit Potential (OCP), electrochemical impedance spectroscopy (EIS), and Potentiodynamic polarization (PDP) tests, were performed in SBF of 7.4 pH at 37°C to obtain corrosion behavior. Bare and Ta-coated 316L stainless steel samples with a dimension of  $(20 \times 20 \times 2)$  mm were used for the cell culture (Cell Adhesion and Cell Proliferation) investigation. Cells of human bone osteosarcoma named **MG-63** were utilized for the **biocompatibility evaluations**. The **wettability** of the films' surface was evaluated using contact angle measuring equipment called Drop Shape Analyzer-DSA 25 (Kruss). The adhesive coating strength was assessed using the Scratch test conducted with the Scratch Tester TR-101 Ducom. After the scratch test, the **2D/3D Profilometer** evaluated the worn surfaces (coated and uncoated) of 316L stainless steel.

The surface of the 316L stainless steel (coated and uncoated) was investigated using various **characterization techniques** [i.e. Optical Microscope (**OM**), Energy Dispersive X-ray Spectrometry (**EDS**), Scanning Electron Microscopy (**SEM**), Scanning Probe Microscope (**SPM**),

Photoelectron Spectroscopy (XPS), Optical Emission Spectroscopy (OES), Inductively Coupled Plasma Mass Spectrometry (ICP-MS), and Fluorescent Microscopy (FM)].

## 7.2 Major Conclusions

The bare 316L stainless steel releases toxic ions into the body due to its poor tribological properties, susceptibility to corrosion, low biocompatibility and adverse impact on the human body, harming long-term implant application. To address this issue, a thin layer of tantalum coating was applied on the 316L SS to mitigate these challenges. The following major conclusions were drawn:

- ❑ The successfully applied a thin layer coating of tantalum over the 316L SS with the help of the DC Magnetron Sputtering system.
- ❑ Materials characterization revealed Ta's strong metallurgical adhesion over the 316L SS and confirmed the significant improvement in wear behavior.
- ❑ After Ta-coating on 316L SS, hardness increased, and the modulus of elasticity decreased to 132.16 GPa against the bare 316L SS. The wear rate of the bare sample is very high ( $62.50 \times 10^{-5} \text{ mm}^3/\text{Nm}$ ) as compared to Ta-coated 316L SS at an applied load of 40N, while Ta-coated 316L SS for 60 minutes found a very low ( $3.75 \times 10^{-5} \text{ mm}^3/\text{Nm}$ ) Ta-coated wear rate at the same load has better wear resistance compared to bare 316L SS.
- ❑ The results also confirmed that the wear mechanism in the SBF environment and developed coating is so effective in preventing the wear on the surface of 316L SS that Ta-coating may be recommended as a preferable coating.

- ❑ The Ta-coated (60 min) demonstrated the highest corrosion resistance, while the bare 316L SS exhibited the lowest. Analysis of the potentiodynamic polarization measurements revealed a consistent decrease in corrosion rate with increasing Ta thickness.
- ❑ The bare 316L SS sample showed the highest corrosion rate of **0.2053 mm/y**, whereas the sample with a 60-min Ta coating displayed the lowest rate of **0.0047 mm/y**.
- ❑ The XPS results reveal a **significant reduction** in the amount of unoxidized stainless steel susceptible to corrosion after the coating.
- ❑ The corrosion performance of the Ta-coated samples in **simulated body fluid** is much better than the 316L SS.
- ❑ Ta coating (1.504 $\mu$ m, 3.809  $\mu$ m, and 6.083  $\mu$ m thickness) was deposited on 316L SS to increase the biocompatibility and hydrophilicity. The application of human osteoblast MG 63 cells in cell culture on specimens for **1, 7, and 14 days** of incubation showed improved biocompatibility. The Ta-coated (60 min) 316L SS demonstrated a significantly greater capacity to promote the formation of an apatite layer that mimicked bone, suggesting that Ta coating can enhance the bioactivity of 316L stainless steel >70 % of alive cells and a few dead cells are obtained for the Ta-coated specimen, which ensures improved cell viability, cell proliferation, and biocompatibility.
- ❑ The Adhesion strength of the Ta-coated (60 min) sample was the highest (23  $\mu$ N  $\pm$ 0.296), whereas for bare 316L stainless steel, it was the lowest (4  $\mu$ N  $\pm$ 0.724).
- ❑ Based on the materials' characterisation results after wettability, adhesion, and in vitro biocompatibility tests, the tantalum coating over 316L stainless steel is a promising candidate for orthopedic applications.