Chapter 18

Surface Engineering of 316L Stainless Steel Using Duplex CrN/TiN Coatings via DC Magnetron Sputtering: Microstructural, Tribological, and Hardness Evaluation

Karunakaran K [a*] Ramasubramanian S [a], Sivabalan S [a], Ruban M [a], Naresh D [b], Saranya Kumaresan [c]

[a] *Department of Mechanical Engineering, Vels Institute of Science, Technology & Advanced Studies, Chennai, India.*

[b] *Student, Department of Mechanical Engineering, Vels Institute of Science, Technology & Advanced Studies, Chennai, India.*

[c] *Centre for Sustainable Materials Research, Department of Physics, Academy of Maritime Education and Training (AMET) Deemed to be University, Kanathur, Chennai, India. saranyakumaresh28@gmail.com*

** Corresponding Author: kkaran.se@velsuniv.ac.in*

Abstract

Surface engineering plays a critical role in improving wear resistance, corrosion resistance, and overall service life of industrial components. This study presents the deposition and characterization of duplex chromium nitride/titanium nitride (CrN/TiN) coatings on AISI 316L stainless steel substrates using physical vapor deposition (PVD) via DC magnetron sputtering. Substrate temperature, working pressure, and nitrogen gas flow rate were selected as primary control variables, identified from literature review and response surface methodology (RSM) considerations. Coatings of approximately 4 µm thickness were deposited and examined using X-ray diffraction (XRD), scanning electron microscopy (SEM), and tribological testing according to ASTM G99 standards. XRD analysis confirmed distinct CrN and TiN phases,

ISBN 978-819871346-9

while SEM micrographs revealed uniform coating coverage with good adhesion and minimal porosity. Wear testing demonstrated that duplex CrN/TiN coatings significantly outperformed both uncoated and single-layer TiN-coated samples, showing reduced wear rates and lower coefficients of friction even under increased normal loads. Hardness measurements indicated that duplex coatings possessed hardness values nearly five times greater than the base 316L substrate. These improvements are attributed to the synergistic combination of TiN's lubricity and CrN's high hardness and corrosion resistance. The findings suggest that duplex CrN/TiN coatings are highly promising for applications requiring enhanced durability, such as plastic molding tools, die-casting dies, and mechanical components subject to severe wear.

Keywords: CrN/TiN duplex coating; DC magnetron sputtering; PVD; wear resistance; hardness; surface engineering.

1. Introduction

The continuous demand for high-performance engineering components has driven advances in surface engineering, particularly in thin film deposition technologies. In many industrial applications — such as plastic injection molding, die-casting, and cutting tools — surface degradation due to wear, corrosion, and oxidation is the primary factor limiting component life. Surface engineering methods, particularly those involving hard coatings, are essential in combating these effects without compromising the bulk properties of the base material.

Among various surface modification techniques, Physical Vapor Deposition (PVD) has emerged as a leading method due to its ability to produce high-purity, well-adhered, and uniform coatings at relatively low process temperatures. Chromium nitride (CrN) and titanium nitride (TiN) are two widely used hard coatings with complementary properties: TiN offers excellent lubricity and aesthetic appeal, while CrN exhibits superior hardness, oxidation resistance, and corrosion protection. Combining them in a duplex coating architecture can leverage the strengths of both materials, resulting in improved mechanical and tribological performance.

This study focuses on the deposition of CrN/TiN duplex coatings on AISI 316L stainless steel substrates using DC magnetron sputtering — a PVD technique known for precise control over coating thickness and composition. The process parameters were chosen based on an extensive literature review, with emphasis on substrate temperature, working pressure, and nitrogen gas flow rate as key variables influencing coating quality. The coatings were evaluated for their microstructure, wear performance, and hardness, with results compared to uncoated and single-layer TiN-coated specimens.

2. Literature Review

Several researchers have investigated the deposition of CrN and TiN coatings using PVD, highlighting the sensitivity of coating properties to process parameters. Hetal N. Shah et al. reported that low working pressures can lead to residual stress build-up, while higher pressures decrease microhardness due to increased crystal imperfections. B. Subramanian et al. demonstrated that increasing nitrogen flow rate increases grain size and changes electrical resistivity from metallic-like to semiconducting behavior.

Yucong Wang et al. observed that CrN, TiN, and TiAlN coatings provide significant resistance to molten aluminum corrosion, with CrN maintaining superior performance at higher oxidation

ISBN 978-819871346-9

temperatures. S. Ortmann et al. found that bias voltage has a significant influence on surface morphology, with coarse structures offering improved lubricant retention. J.-D. Kamminga et al. showed that substrate hardness directly affects scratch resistance, while Jianliang Lin et al. studied CrN/AlN superlattice coatings, revealing that reduced layer thickness increases hardness due to higher interface density.

These studies establish that coating quality is strongly dependent on deposition parameters, justifying the need for controlled experimentation in optimizing CrN/TiN duplex coatings for stainless steel substrates.

3. Materials and Methods

3.1 Substrate Material

AISI 316L stainless steel was selected as the substrate due to its excellent corrosion resistance, moderate hardness (312.76 Hv), and high coefficient of thermal expansion. Specimens were prepared with dimensions of 50 × 40 × 3 mm, and cylindrical pins (Ø10 × 50 mm) were machined for tribological testing.

3.2 Coating Deposition Process

The coatings were deposited using DC magnetron sputtering in a high-vacuum chamber. The deposition system was equipped with pure chromium (99.99%) and titanium targets. The chamber was evacuated to a base pressure below 5×10^{-6} Torr, and argon was introduced for plasma generation. Nitrogen gas was used as a reactive species to form CrN and TiN compounds.

3.2 Control variables

- **Working pressure:** 10–20 mTorr

- **Substrate temperature:** 300–500 °C
- **Nitrogen gas flow rate:** 5–15 sccm

The coatings were deposited in a duplex configuration: TiN as the first layer for improved adhesion and lubricity, followed by CrN for hardness and corrosion resistance. Coating thickness was approximately 4 µm.

3.3 Surface Preparation

Prior to coating, specimens were cleaned ultrasonically to remove oils and contaminants, followed by liquid honing with fine abrasive powder to achieve a surface roughness of ~0.108 µm.

3.4 Characterization Techniques

- **X-Ray Diffraction (XRD):** Cu-Kα radiation was used to identify coating phases.
- **Scanning Electron Microscopy (SEM):** Morphological analysis and cross-sectional imaging.
- **Hardness Testing:** Vickers microhardness tester with 0.1 kg load.
- **Wear Testing:** Pin-on-disc tribometer (Ducom TR-20 LT) under dry sliding conditions, track diameter 100 mm, sliding speeds 98 and 110 rpm, loads 12 and 15 N, as per ASTM G99.

4. Results and Discussion

4.1 Phase Identification (XRD)

XRD patterns confirmed the presence of CrN and TiN phases, with characteristic peaks at 37.5° (CrN) and 36.6° (TiN). The sharpness of peaks indicated good crystallinity, while the absence of unwanted phases suggested high purity.

ISBN 978-819871346-9
9 788198 713469

4.2 Surface Morphology (SEM)

SEM images showed uniform coating coverage without visible delamination or cracks. Cross-sectional imaging confirmed layer thickness and strong adhesion. The duplex structure exhibited a compact, dense microstructure with minimal porosity — essential for wear resistance and corrosion protection.

4.3 Wear Behavior

The wear rate of uncoated 316L increased sharply after ~250 s due to poor lubricating properties and adhesive wear. TiN-coated samples showed reduced wear, with maximum wear depth of ~9 µm at 230 s. In contrast, CrN/TiN duplex coatings exhibited significantly lower wear rates and maintained performance even under higher loads (15 N) and speeds (110 rpm).

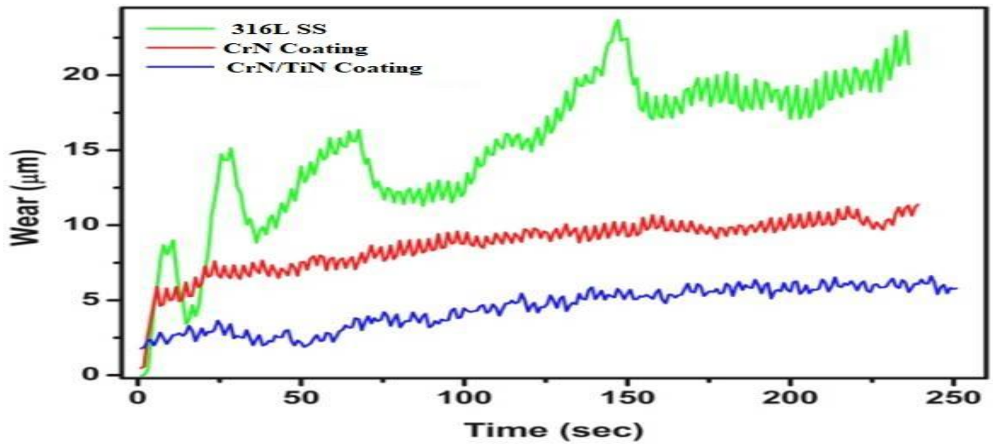

Figure. 1: Wear vs. Time

The superior wear performance of duplex coatings can be attributed to:

Synergistic effect: TiN's lubricity reduces initial friction, while CrN resists abrasion and deformation.

Improved load distribution: Duplex layers spread contact stresses more effectively.

Strong adhesion: Enhanced resistance to delamination during sliding.

4.4 Friction Coefficient

Friction factor measurements showed that duplex coatings maintained lower and more stable coefficients of friction compared to both uncoated and TiN-coated specimens, particularly under high-load conditions.

4.5 Hardness Enhancement

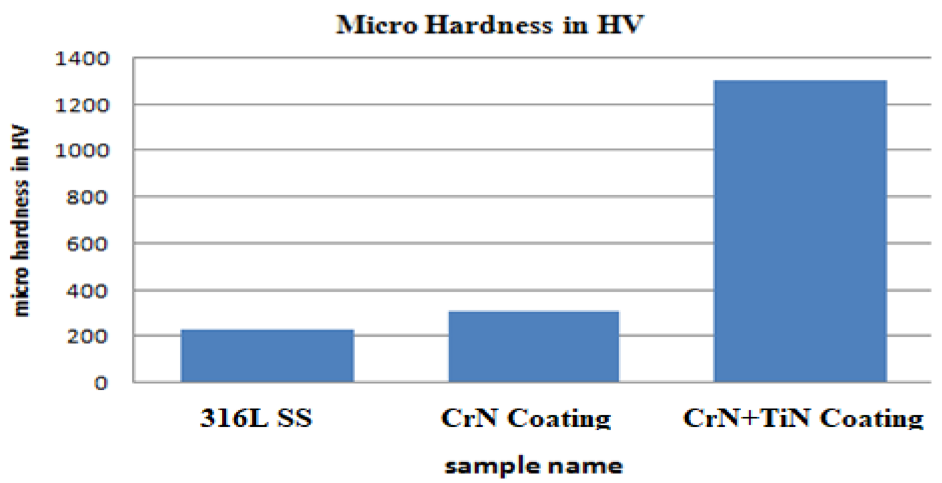

Figure. 2: Micro hardness vs. Samples

Vickers micro hardness testing revealed that duplex coatings achieved hardness nearly five times greater than the base 316L steel. This improvement is linked to grain refinement, solid solution strengthening, and the presence of hard CrN phases.

5. Cost Analysis

The cost estimation for coating and testing included material cost (₹6,250) and labor cost (₹2,900), bringing the total to ₹9,150 for the

ISBN 978-819871346-9

project. This relatively low cost, combined with improved service life, supports the economic viability of CrN/TiN duplex coatings in industrial applications.

Table 1: Cost Analysis

S.No	Description	Quantity	Material cost
1	316 L Stainless Steel	1 kg	850
2	Coating	5 (Sample)	2500
3	Hardness test	2 (Sample)	1200
4	Wear Test	2 (Sample)	1000
5	Density Test	2 (Sample)	700
Total			Rs. 6,250

6. Conclusion

This study demonstrated that CrN/TiN duplex coatings deposited via DC magnetron sputtering significantly enhance the mechanical and tribological performance of 316L stainless steel. The optimized process produced coatings with:

- Superior hardness (~5× improvement over base metal)
- Substantially reduced wear rate under high load
- Lower and more stable friction coefficients
- Uniform, defect-free morphology

Given their combined wear resistance, hardness, and corrosion protection, these coatings are promising for tooling, plastic molding, and other high-demand applications where both performance and cost-effectiveness are critical.

References

[1] H. N. Shah, V. Chawla, R. Jayaganthan, and D. Kaur, "Microstructural characterizations and hardness evaluation of d.c. reactive magnetron sputtered CrN thin films on stainless steel substrate," Bull. Mater. Sci., vol. 33, no. 2, pp. 103–110, Apr. 2010.

[2] B. Subramanian, K. Prabakaran, and M. Jayachandran, "Influence of nitrogen flow rates on materials properties of CrNx films grown by reactive magnetron sputtering," Bull. Mater. Sci., vol. 35, no. 4, pp. 505–511, Aug. 2012.

[3] T. Björk, R. Westergård, and S. Hogmark, "Wear of surface treated dies for aluminium extrusion—a case study," Wear, vol. 249, no. 3–4, pp. 316–323, Aug. 2001.

[4] Y. Wang, "A study of PVD coatings and die materials for extended die-casting die life," Surf. Coat. Technol., vol. 94–95, pp. 60–63, 1997.

[5] J. Lin, J. J. Moore, B. Mishra, M. Pinkas, X. Zhang, and W. D. Sproul, "CrN/AlN superlattice coatings synthesized by pulsed closed field unbalanced magnetron sputtering with different CrN layer thicknesses," Thin Solid Films, vol. 517, no. 19, pp. 5798–5804, Aug. 2009.

[6] J.-D. Kamminga, P. van Essen, R. Hoy, and G. C. A. M. Janssen, "Substrate dependence of the scratch resistance of CrNx coatings on steel," Tribol. Lett., vol. 19, no. 2, pp. 107–112, Jun. 2005.

[7] J. C. Avelar-Batista, L. P. P. Rodrigues, and H. A. Santos, "Wet stripping of Cr–N coatings deposited by different PVD techniques," Surf. Coat. Technol., vol. 142–144, pp. 118–123, Jul. 2001.

[8] S. Ortmann, A. Esser, and C. Leyens, "In-process structuring of CrN coatings for improved tribological performance," Surf. Coat. Technol., vol. 113, no. 3, pp. 114–120, 1999.

[9] K. Holmberg and A. Matthews, Coatings Tribology: Properties, Mechanisms, Techniques and Applications in Surface Engineering, 2nd ed. Amsterdam, Netherlands: Elsevier, 2009.

[10] P. J. Kelly and R. D. Arnell, "Magnetron sputtering: A review of recent developments and applications," Vacuum, vol. 56, no. 3, pp. 159–172, Mar. 2000

ISBN 978-819871346-9