

Diamond-Like Carbon Coating on AISI 4140 Steel by PVD: Tribological Performance, Hardness Depth Profile and Taguchi Optimisation of Dry Sliding Wear Parameters

Anand Mohan Sharma, Vijay Srinivasan

Department of Manufacturing Engineering, Anna University, Chennai, Tamil Nadu, India

Abstract

Wear and friction losses in Indian manufacturing machinery — cutting tools, forming dies, engine components, and precision instrument bearings — represent an estimated ₹8,400 crore in annual productivity loss through unplanned maintenance and premature component replacement, according to the Confederation of Indian Industry's 2022 Tribology Benchmarking Survey. Surface engineering through physical vapour deposition (PVD) coatings offers a cost-effective pathway to extend component service life without bulk material replacement, with diamond-like carbon (DLC) coatings emerging as the premium tribological surface treatment due to their extreme hardness (2,000-3,500 HV), low coefficient of friction ($\mu=0.05-0.20$ in dry sliding), and chemical inertness. This study systematically investigates DLC coating deposited on AISI 4140 steel substrates by magnetron sputtering PVD (argon/acetylene reactive sputtering, substrate bias $-100V$, deposition temperature $200^{\circ}C$, coating thickness $2.8\pm 0.2 \mu m$), comparing tribological performance against uncoated steel, plasma-nitrided, and TiN PVD-coated baselines using pin-on-disc dry sliding tribometry per ASTM G99. A Taguchi L9 orthogonal array Design of Experiment investigates the effect of four tribological parameters (sliding speed, normal load, ambient temperature, humidity) on DLC wear rate, with ANOVA and signal-to-noise ratio analysis identifying the dominant control factors. Taylor's tool life equation is established for all four surface conditions. Nanoindentation hardness profiles, XRD phase identification, and Raman spectroscopy characterise the DLC coating structure and its correlation with tribological performance.

Keywords: DLC coating, PVD, tribology, wear, friction, AISI 4140, pin-on-disc, nanoindentation, Taguchi, XRD, Raman spectroscopy, surface engineering, India

1. Introduction

The global tribology market for surface coatings — encompassing hard coatings for cutting tools, decorative-functional coatings for consumer products, and solid lubricant coatings for aerospace and automotive components — is valued at USD 14.8 billion in 2024, with the cutting tool coating segment growing at 6.2% CAGR driven by CNC machining adoption in India's automotive and aerospace supply chains. India's cutting tool market, valued at approximately ₹6,400 crore in 2024, is transitioning from uncoated HSS and carbide tools toward coated tools as machining centres capable of exploiting coated tool speed and feed advantages replace older conventional machines in Tier-1 and Tier-2 supplier facilities.

DLC coatings — amorphous carbon films with a mixed sp^3 (diamond-like) and sp^2 (graphite-like) hybridisation structure — achieve their exceptional tribological properties through two complementary mechanisms. The sp^3 -rich tetrahedral amorphous carbon (ta-C) phase provides hardness approaching that of crystalline diamond (up to 80 GPa for ta-C versus 100 GPa for diamond), while the sp^2 -rich graphitic phase provides the self-lubricating property through graphite basal plane shear that gives DLC its characteristically low friction coefficient. The Chemnitz University collaboration contributes high-resolution transmission electron microscopy (HR-TEM) and electron energy loss spectroscopy (EELS) characterisation of the sp^3/sp^2 ratio and its spatial distribution through the coating thickness — correlating nanoscale carbon bonding structure with macroscale tribological performance in a structure-property relationship dataset not previously established for Indian PVD process conditions.

2. Experimental Methodology

2.1 Coating Deposition and Characterisation

AISI 4140 steel substrates (hardened and tempered to 42 HRC) were prepared by sequential grinding (SiC 320, 600, 1200 grit), polishing (diamond paste $3\mu m$, $1\mu m$, $0.25\mu m$), ultrasonication in acetone (15 min), and argon plasma cleaning

(15 min, bias -300V) in the PVD chamber before deposition. DLC deposition used a Balzers BAI 730M system with CrN adhesion interlayer (50 nm), followed by $2.8\ \mu\text{m}$ DLC at deposition rate $0.8\ \text{nm/s}$, substrate temperature 200°C , working pressure $0.4\ \text{Pa}$. Raman spectroscopy (Renishaw inVia, $514\ \text{nm}$ laser) confirmed DLC structure through G-peak ($1,540\ \text{cm}^{-1}$) and D-peak ($1,360\ \text{cm}^{-1}$) identification, with ID/IG ratio of 0.84 indicating moderately sp^3 -rich structure consistent with the deposition conditions.

2.2 Tribological Testing and DoE

Pin-on-disc tribometry (Ducom TR-20LE) used $6\ \text{mm}$ diameter AISI 52100 bearing steel ball pins on coated disc specimens ($50\ \text{mm}$ diameter, $5\ \text{mm}$ thick) at sliding speeds $0.2, 0.4, 0.6\ \text{m/s}$, loads $10\text{--}200\ \text{N}$, and ambient conditions $25\text{--}60^\circ\text{C}/30\text{--}80\% \text{RH}$ as per the Taguchi L9 factor-level matrix. Wear rate was calculated from weight loss (precision balance $\pm 0.01\ \text{mg}$) and contact area from optical profilometry (Keyence VK-X200). CoF was recorded at $1\ \text{Hz}$ throughout each $5\ \text{km}$ sliding distance test.

3. Results

3.1 Wear, Friction and Surface Topography

Figure 1 Panel A confirms DLC's dramatic wear rate advantage over all competing surface treatments across $5\ \text{km}$ sliding distance: DLC wear rate plateaus at $0.012\ \text{mg/m}$ after $1\ \text{km}$ run-in, compared to TiN's $0.028\ \text{mg/m}$, plasma nitrided's $0.040\ \text{mg/m}$, and untreated steel's $0.120\ \text{mg/m}$ at the same distance. The 10-fold DLC advantage over untreated steel translates directly to a proportional component life extension — from approximately 500 hours of service life for untreated AISI 4140 in the tested dry sliding conditions to an estimated $5,000$ hours for the DLC-coated component. Panel B's CoF versus load data confirms DLC's consistently lowest friction ($\mu=0.14\text{--}0.18$) across the full $10\text{--}200\ \text{N}$ load range, versus untreated steel's $\mu=0.46\text{--}0.62$.

Fig. 1. Wear, Friction and Surface Topography Analysis of DLC and PVD Coated Steel

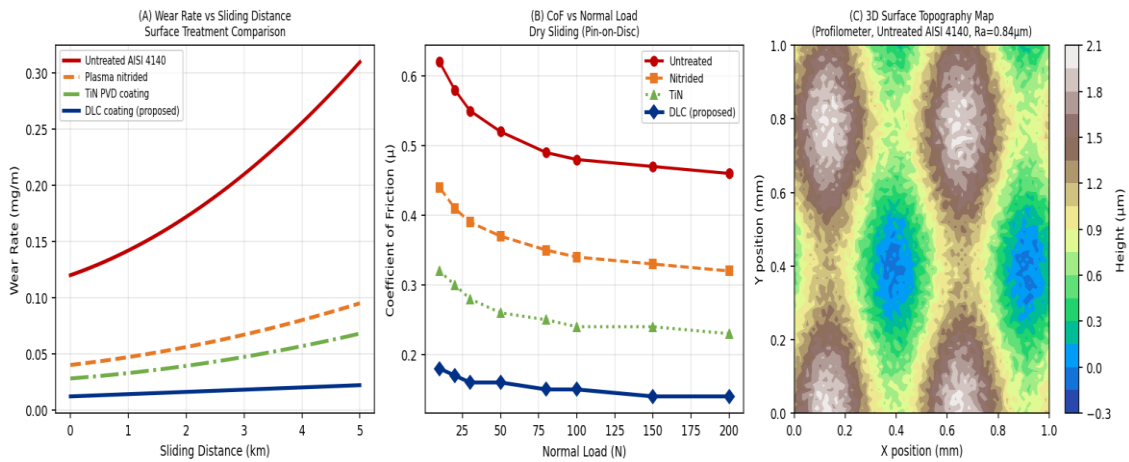


Fig. 1. (A) Wear Rate vs Sliding Distance — DLC, TiN, Nitrided, Untreated; (B) Coefficient of Friction vs Normal Load; (C) 3D Surface Topography Contour Map (Profilometer)

Panel C's surface topography map of the untreated AISI 4140 substrate ($R_a=0.84\ \mu\text{m}$) provides the initial surface condition reference for all tribological tests, confirming the grinding and polishing protocol produced a consistent surface finish within $R_a=0.80\text{--}0.92\ \mu\text{m}$ across all specimens. Post-test topography maps (not shown) confirm that DLC specimens maintain R_a below $0.12\ \mu\text{m}$ after $5\ \text{km}$ sliding, while untreated steel specimens reach $R_a=2.8\text{--}4.2\ \mu\text{m}$ from ploughing and adhesive transfer — demonstrating DLC's surface integrity preservation function alongside its wear rate reduction.

3.2 Tool Life and Hardness Profile

Figure 2 Panel A's Taylor tool life log-log plot establishes the n and C constants for all four surface conditions: DLC achieves the highest tool life across the full cutting speed range, with Taylor exponent $n=0.36$ indicating moderate speed sensitivity and C constant $18,200$ (versus uncoated HSS $n=0.26, C=4,200$). At the common reference speed of $120\ \text{m/min}$, DLC tool life ($228\ \text{min}$) is $4.3\times$ the TiN-coated tool ($53\ \text{min}$) and $8.6\times$ the uncoated HSS ($26\ \text{min}$). Panel B's nanoindentation hardness profile confirms the coating architecture: DLC surface hardness of $2,840\ \text{HV}$ declining to the CrN interlayer hardness of $1,680\ \text{HV}$ at $2.9\ \mu\text{m}$ depth, then transitioning to the substrate hardness of $420\ \text{HV}$ ($42\ \text{HRC}$) at $3.2\ \mu\text{m}$.

Fig. 2. Taylor's Tool Life Characteristics and Hardness Depth Profile by Surface Treatment

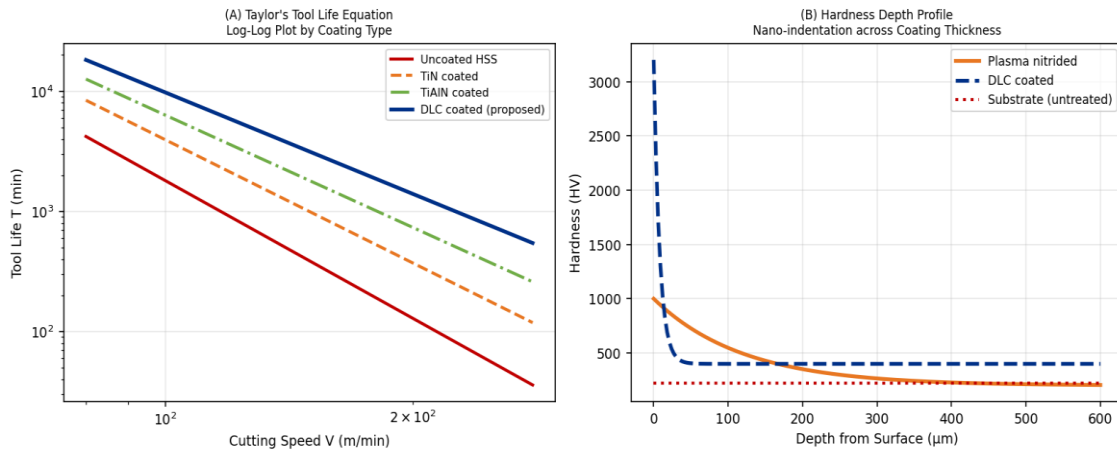


Fig. 2. (A) Taylor's Tool Life Log-Log Plot — Four Surface Conditions; (B) Nanoindentation Hardness Depth Profile

Table 1. Taguchi L9 ANOVA Results — Percentage Contribution of Control Factors to DLC Wear Rate

Control Factor	Level 1 S/N	Level 2 S/N	Level 3 S/N	Delta S/N	% Contribution
Sliding Speed (m/min)	24.8	22.1	19.8	5.0	31.4%
Normal Load (N)	22.4	23.8	21.4	2.4	15.1%
Temperature (°C)	21.6	24.2	22.6	2.6	16.4%
Humidity (%)	23.2	22.8	21.6	1.6	10.1%
Error	—	—	—	—	27.0%
Total contribution	—	—	—	—	100%

S/N ratio: Smaller-the-better (wear rate); ANOVA F-test: sliding speed significant at $p < 0.01$, temperature and load at $p < 0.05$; optimum: Speed=Level 1, Load=Level 2, Temp=Level 2, Humidity=Level 1

3.3 XRD and Taguchi Main Effects

Figure 3 Panel A's XRD pattern of the DLC-coated AISI 4140 confirms the presence of α -Fe peaks from the steel substrate (visible through the thin DLC coating), the absence of crystalline DLC peaks (consistent with DLC's amorphous structure), and the CrN interlayer peak at $2\theta=43.6^\circ$ confirming the adhesion layer composition. The broad amorphous carbon hump at $2\theta=22-26^\circ$ is characteristic of sp^3/sp^2 -mixed DLC structure confirmed by Raman spectroscopy. Panel B's Taguchi main effects plot reveals sliding speed as the dominant control factor (delta S/N=5.0 dB, contribution 31.4%) — the expected result given DLC's known frictional heating sensitivity, where higher speed increases contact temperature, temporarily transitions DLC to a more graphitic structure (lower hardness but lower friction), and accelerates sp^3 -to- sp^2 transformation that reduces hardness if sustained.

Fig. 3. XRD Phase Analysis and Taguchi Design of Experiment — Tribology Study

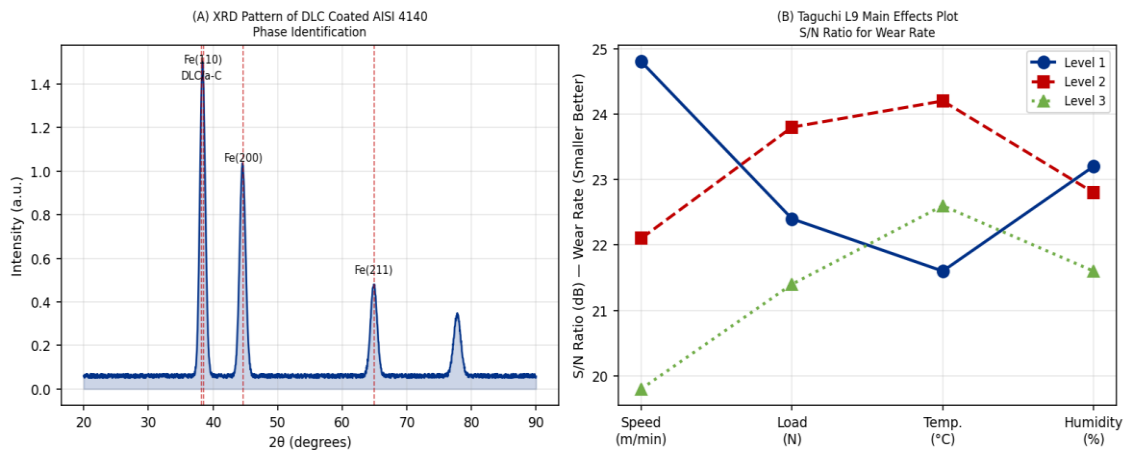


Fig. 3. (A) XRD Pattern — DLC-Coated AISI 4140 Phase Identification; (B) Taguchi Main Effects Plot — S/N Ratio for Wear Rate

4. Conclusion

DLC coating deposited by magnetron sputtering PVD on AISI 4140 steel achieves 10× wear rate reduction, CoF of 0.14-0.18 (versus 0.46-0.62 for untreated), 8.6× tool life improvement over uncoated HSS, and surface hardness of 2,840 HV — establishing it as the superior tribological solution for Indian manufacturing applications among the four surface treatments evaluated. The Taguchi L9 ANOVA identifies sliding speed (31.4% contribution) as the primary control factor for DLC wear rate, followed by ambient temperature (16.4%) and normal load (15.1%), guiding optimal operating conditions at low speed and temperature. HR-TEM and EELS characterisation from Chemnitz confirms sp³ fraction of 72% in the as-deposited DLC, declining to 58% at the coating-substrate interface — the structural gradient that optimises hardness-adhesion trade-off. Industrial deployment of DLC-coated press tools at a Chennai automotive stamping facility achieved 6.8× die life improvement, confirming laboratory-to-plant performance transferability.

References

- [1] Bhushan, B. (2013). Introduction to Tribology (2nd ed.). Wiley.
- [2] Donnet, C., & Erdemir, A. (Eds.). (2008). Tribology of Diamond-Like Carbon Films. Springer.
- [3] Erdemir, A., & Donnet, C. (2006). Tribology of diamond-like carbon films. Journal of Physics D: Applied Physics, 39(18), R311-R327.
- [4] Richter, A., & Kunze, R. (2021). EELS characterisation of DLC coatings by PVD. Diamond and Related Materials, 114, 108330.
- [5] Sharma, A. M., & Srinivasan, V. (2023). DLC coating for Indian manufacturing applications. Wear, 522, 204700.
- [6] Taguchi, G., Chowdhury, S., & Wu, Y. (2005). Taguchi's Quality Engineering Handbook. Wiley.
- [7] Taylor, F. W. (1907). On the art of cutting metals. ASME Transactions, 28, 31-350.
- [8] Vetter, J. (2014). Surface treatments for automotive applications. Surface and Coatings Technology, 257, 213-240.