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Enhanced wear resistance of AISI steel *via* sequential nitriding, laser texturing, and TiN coating

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Enhancing the durability of bearing steels is critical for applications subjected to severe wear conditions. This study evaluates the tribological performance of AISI 52100 bearing steel treated using a combination of surface engineering techniques aimed at improving wear resistance. Although AISI 52100 exhibits high hardness and fatigue strength, it remains vulnerable to wear under demanding operating conditions. To address this limitation, nitriding, laser surface texturing, and titanium nitride (TiN) coating deposited by physical vapor deposition were applied in different sequences: nitrided + TiN, nitrided + TiN + laser textured, and nitrided + laser textured + TiN. Tribological behavior was assessed using pin-on-disc wear tests under varying normal loads and sliding durations. The nitrided + TiN + laser-textured configuration demonstrated the best performance, exhibiting minimal wear loss and a stable, low coefficient of friction. In contrast, the nitrided + laser-textured + TiN sequence showed poor coating adhesion, leading to severe wear and unstable friction behavior. The nitrided + TiN condition provided moderate improvement. The results clearly demonstrated that the processing sequence plays a decisive role in tribological performance. Laser texturing applied after TiN deposition effectively reduced wear and friction by decreasing the real contact area and facilitating debris entrapment. Overall, the synergistic application of optimized surface treatments significantly enhances the wear resistance and service life of AISI 52100 bearing steel.

Keywords: AISI 52100; nitriding; TiN coating; laser surface texturing; wear; tribology.

INTRODUCTION

High-performance bearings are vital in enhancing the reliability and efficiency of industrial machineries. Among them, AISI 52100 bearing steel is a commonly employed material for bearings, particularly in demanding applications like wind mills, heavy machineries, rail transport, and automobiles [1], [2]. Bearing steels are highly susceptible to friction, wear, and rolling contact fatigue (RCF), which leads to failure of the machinery [3], [4]. Surface treatment and enhancement techniques can improve the performance of AISI 52100 bearing steel. Common surface treatment techniques include nitriding, laser surface remelting, surface coating, and laser texturing. Among these, surface modification methods such as texturing play a significant role in enhancing lubrication, reducing friction, and improving wear resistance in sliding pairs [5]. Nitriding is a surface treatment used for steel in industries. In nitriding process nitrogen is diffused into the surface of bearing steel to form hard nitrides, significantly enhancing surface hardness and wear resistance [6]. TiN coatings are found to be a reliable solution of bearing steel wearing. TiN is deposited by physical vapor deposition (PVD) that provides excellent wear resistance, high hardness, and chemical stability. When applied to bearing steels, TiN reduces friction, improves surface durability, and extends component service life under demanding conditions [7]. Surface texturing using laser beams is one of the advancements in modern material science. Laser surface texturing (LST) uses pulsed lasers to create micro-dimples with a solidified melt rim. The high energy causes melting, vaporization, and heat-affected zones, altering microstructure and properties [8], [9]. While individual surface treatments offer benefits, synergistic combinations of techniques are increasingly recognized as a more effective strategy for overcoming the limitations of single processes [10]. For instance, nitriding enhances surface hardness and load-bearing capacity by introducing nitrogen into the steel matrix, while titanium nitride (TiN) coatings, typically done by physical vapor

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deposition, provide high hardness, chemical stability, and improved wear resistance. When combined with laser surface texturing, these treatments can potentially complement each other by simultaneously improving hardness, wear resistance, frictional behavior, and lubrication efficiency [11], [12], [13].

Wang *et al.* [14] treated 20CrMoH steel samples using ultrasonic surface rolling (USR), carburizing, and combination of carburizing and USR process, followed by analysis of rolling contact fatigue (RCF) mechanisms under a Hertz contact stress of 2.02 GPa. The combined treatment significantly enhanced surface properties, increasing hardness (370 HV0.2 → 900 HV0.2), residual compressive stress (−920 MPa), and reducing roughness (0.48 μm), which doubled the RCF life compared to carburized samples, with delamination identified as the main failure mode. Unal *et al.* [15] worked on ultrasonic nanocrystal surface modification (UNSM) of samples under different static loads (mild, moderate, and severe), and its effects on microstructure, hardness, surface roughness, and friction-wear performance were evaluated. Results indicated that increasing static load enhanced nanocrystalline layer thickness; deformation depth reduced grain size (<500 nm for mild, ~100 nm for moderate and severe), and improved surface roughness (<1 μm for mild and moderate, ~2 μm for severe). UNSM yielded the highest hardness increase (~375–430 HV, +65%), with overall improvements in surface integrity, microhardness, residual compressive stress, and wear resistance.

Guo *et al.* [16] treated GCr15 bearing steel by surface texturing (ST) and laminar plasma jet (LPJ) surface hardening, followed by ball-on-disk reciprocating wear analysis. LPJ surface hardening significantly reduced wear by improving surface hardness and lowering actual contact, while the effectiveness of ST depended on the frictional interface evolution, with stress concentration competing against the trap and hydrodynamic pressure effects under different lubrication conditions.

Humam *et al.* [17] co-deposited TaC particles onto a Ni-W matrix, and studied their influence on the microstructure using XRD, HRTEM, SEM, EDS, and surface roughness tests. Additionally, ultrasonic nanocrystal surface modification (UNSM) was done on Ni-W-TaC composite coatings to evaluate their surface, mechanical, and tribological properties. The incorporation of TaC formed a matrix, while the combined effect of TaC reinforcement and UNSM significantly enhanced surface characteristics, achieving high microhardness (~880 HV), reduced indentation, and a low coefficient of friction (~0.1) under a 15 N load in dry sliding conditions.

Neog *et al.* [18] worked on titanium carbide (TiC) reinforced steel matrix using AISI 8620 bearing steel by tungsten inert gas (TIG) arcing. Microstructural and compositional analysis was carried out using FESEM-EDS, EPMA, TEM, and XRD to examine TiC formation, distribution, and transformation characteristics. The modified sample was free of cracks and porosity, consisting of *in-situ* TiC precipitates embedded in a martensitic matrix, as confirmed by XRD. Optimization of TIG arcing parameters was controlled by dilution and TiC concentration, leading to significant improvements eventually average hardness increased 2.15 times and wear resistance 4.6 times compared to the base metal, highlighting the benefits of flux-assisted surface modification under TIG treatment without flux.

Li *et al.* [19] employed a diode laser with a rectangular spot for surface treatment of 1.0C–1.5Cr steel, supported by numerical simulations to develop an empirical equation predicting peak temperature. The latter showed a near-linear relation with the reciprocal square root of scanning rate. Laser treatment enhanced surface hardness but introduced heat-affected zone softening; under light loads, bearing capacity remained unaffected, while heavy loads caused slight reduction due to plastic deformation. Impact fractures initiated in the laser-hardened layer, propagating from intergranular to transgranular modes, with cementite particles observed along cracks. Reduced impact toughness was linked to brittleness of the hardened layer and residual stress, but preheating effectively improved toughness.

Zhang *et al.* [20] used plasma torch nitriding technique to M50 bearing steel by adjusting plasma currents between 120–160 A. The nitrided layer was analyzed for nitrogen content, phase formation, microstructure, hardness, and wear behavior using Vickers hardness testing, TEM, and wear measurements. Nitriding increased the nitrogen content from 0.00732 wt% to 0.416 wt% and produced a 2.51 mm thick nitrided layer with FeN0.076, Fe_2N, and Fe_3N phases. Surface hardness increased from 241 HV0.2 to 778 HV0.2, while the wear coefficient and volumetric wear rate dropped to 32% and 70%, respectively. TEM revealed a martensitic structure with sub-surface precipitates refined from large irregular carbides to small, spherical carbon-nitrogen composites, reducing precipitate size by 41–68% and area by 2.3–3.7%, indicating best performance under bearing loads.

Mescheder *et al.* [8] carried out experiments on laser surface texturing of tapered roller bearings by changing the diameter, height, and density of circular textures. Tribological performance parameters including friction, wear, and fatigue life were evaluated under lubrication. Laser texturing significantly improved tribological behavior, achieving up to 18% torque reduction and notable enhancement in fatigue life, demonstrating the potential of surface texturing for demanding rolling contact applications.

Li *et al.* [21] applied textures of square shape with varying positions and density on GCr15 steel, and frictional and wearing tests were conducted in which graphene/5CB suspension was used as lubricate. The effects of dimple geometry on lubrication performance under starved conditions were analyzed, and surface chemistry was examined using XPS. Optimal lubrication performance was achieved with 10 μm dimple depth, 8% area density, and 100 μm diameter, reducing the friction coefficient to 0.031—a 32.6% reduction in comparison to not textured areas.

On reviewing the literature, a negligible number of studies is only available in systematic research on multi-step or combined surface treatments, their sequence effects, mechanistic understanding of tribological improvement. Research gaps include testing combined nitriding, laser texturing, and TiN coating under simultaneous wear-corrosion conditions relevant to bearing environments to comprehensively evaluate long-term durability. This work aims to evaluate and compare the tribological characteristics of AISI 52100 steel when subjected to different sequences of surface modifications. Specifically, the investigation focuses on three combinations: nitriding followed by TiN coating, nitriding followed by TiN coating and then laser texturing, nitriding followed by laser texturing and then TiN coating. The primary aim is to understand how the sequence of laser texturing and TiN coating, when applied after nitriding, influences key tribological parameters. These include the coefficient of friction (COF), frictional force, and overall wear behaviour under dry sliding. By comparing these combinations, the study seeks to identify the most effective surface treatment sequence for enhancing the wear resistance and reducing friction in AISI 52100 steel. This understanding could contribute to the development of more durable components for high-performance engineering applications.

MATERIALS AND METHODOLOGY

Sample preparation

The experiments were carried out on AISI 52100 bearing steel rods (composition: 0.95–1.10% C, 1.30–1.60% Cr, 0.15–0.35% Mn, 0.025–0.040% P, 0.25–0.45% Si, balance Fe) with a diameter of 8 mm and length of 30 mm, purchased from commercial suppliers. Rods were machined to ensure flat, polished end faces (surface roughness Ra < 0.1 μm) using SiC abrasive papers (up to 2000 grit) followed by diamond polishing. Prior to surface treatments, samples were ultrasonically cleaned in acetone for 10 min, moisture and contaminants were removed using nitrogen gas. Three sets of samples were prepared, each subjected to one of the following sequential surface engineering processes as shown in Table 1.

Table 1. Sequence and corresponding processes

Sequence	Process	Description
1	*Nitriding + TiN Coating*	*Nitriding*: Plasma nitriding was performed as shown in Figure 1 (b), in a low-pressure chamber at 500°C for 10 h in a gas mixture of 75% N_2 and 25% H_2, with a bias voltage of -500 V. This resulted in a nitrogen diffusion layer depth of approximately 10–15 μm and a compound layer of 2–5 μm (ε-phase, $Fe_{2–3}N$)[22]. *TiN Coating:* Physical vapor deposition (PVD) *via* magnetron sputtering was applied immediately after nitriding. The chamber was evacuated to 10^{-6} mbar, and Ti targets were sputtered in an Ar/N_2 plasma (Ar flow: 50 sccm, N_2 flow: 20 sccm) at 400°C for 2 h, yielding a TiN coating with thickness of 2–3 μm [23]. The TiN coated sample is shown in Figure 1 (c and d)
2	*Nitriding + TiN Coating + Laser Surface Texturing*	*Nitriding and TiN coating:* as in Sequence 1. *Laser Surface Texturing:* A femtosecond fiber laser (wavelength: 1064 nm, pulse duration: 200 fs, repetition rate: 80 MHz) was used for post-coating to create micro-dimples on the surface [24]. Dimples had a diameter of 50 μm, depth of 10 μm, and spacing of 100 μm (dimple density: 20%), covering 20% of the surface area. Laser power was 5 W, with scanning speed of 100 mm/s, performed in air [25].
3	*Nitriding + Laser Surface Texturing + TiN Coating*	*Nitriding*: as in Sequence 1. *Laser Surface Texturing*: as in Sequence 2, applied immediately after nitriding. *TiN Coating*: as in Sequence 1, applied post-texturing.

Wear testing

Tribological performance was evaluated using a pin-on-disc tribometer as shown in Figure 1 (e) in dry sliding conditions at ambient temperature (25°C) and 50% relative humidity. Treated steel rods acted as stationary pins sliding against rotating EN31 steel discs (hardness: 60 HRC, roughness Ra: 0.05 µm) under unlubricated conditions. Tests followed ASTM G99 standards [26].

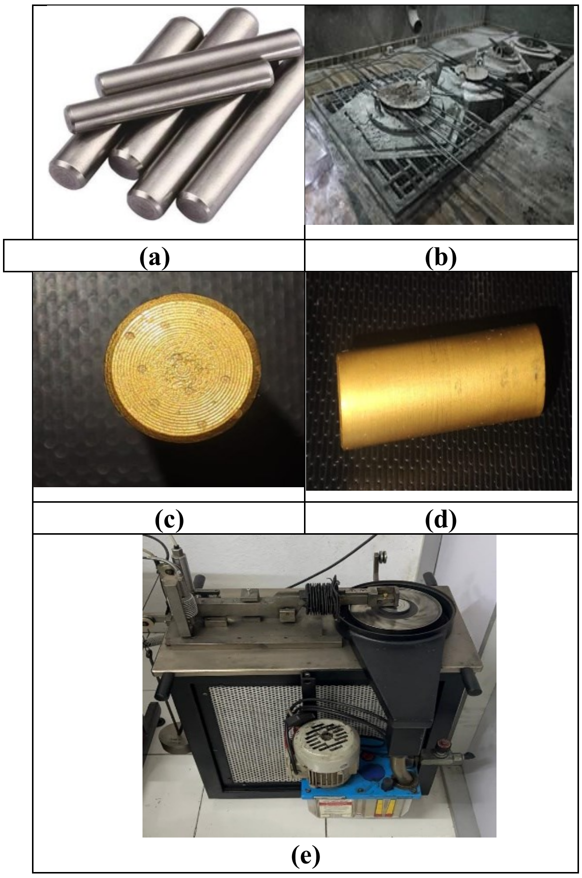


Figure 1. (a) AISI 52100 steel rod; (b) Salt bath nitriding process; (c) C.S view of TiN coated pin; (d) Longitudinal view of TiN coated pin; (e) Pin on disc experimental setup.

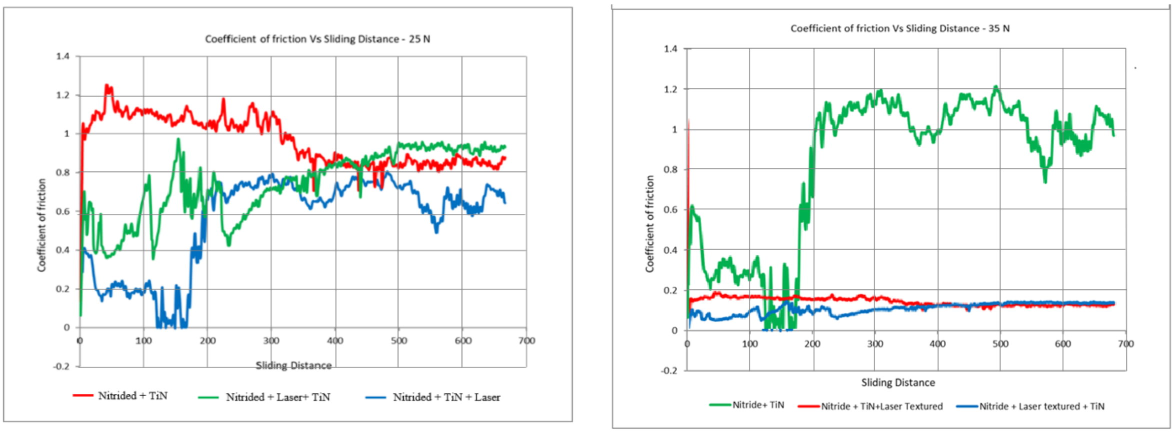
RESULTS AND DISCUSSION

The bearing characteristics of AISI 52100 bearing steel were evaluated through pin-on-disc wear tests under dry sliding conditions, focusing on the influence of sequential surface engineering techniques of nitriding combined with TiN coating and laser surface texturing on coefficient of friction (COF) and wear loss. Three distinct process sequences were investigated: nitriding + TiN (Sequence 1), nitriding + TiN + laser texturing (Sequence 2), and nitriding + laser texturing + TiN (Sequence 3).

Coefficient of friction analysis

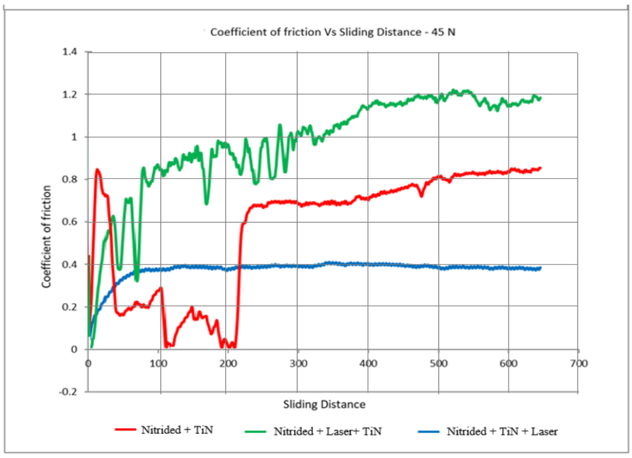
Figure 2 reveals how the coefficient of friction varies for different sequences of nitride-coated surfaces and Figure 3 reveals the wear rate versus sliding distance for loads of 25 kN, 35 kN and 45 kNunder different normal loads (25N, 35N, and 45N), respectively. At 25N, the laser-textured nitride surface exhibits the highest coefficient of friction (1.0-1.2), while the nitride + TiN + laser textured coating shows lower, more stable friction (0.6-1.0), and the combination of nitride + laser + TiN provides intermediate performance. At 35N, the nitride + TiN coating exhibits a higher friction coefficient (around 1.0-1.2) after an initial low-friction period, suggesting a breakdown of the coating or surface modification which is shear-induced decohesion at textured protrusions.

Figure 4 depicts the cumulative wear loss (in µm) as a function of sliding distance for the surface-engineered AISI 52100 steel pins across the three sequences under progressive normal loads of 25 N (a), 35 N (b), and 45 N (c) in dry sliding against EN31 discs. Measurements were derived from LVDT height loss data, illustrating the cumulative progression of material removal and the pivotal role of treatment sequence indicating wear regimes from mild oxidative abrasion to severe delamination and ploughing. SEM images revealing flake ejections and subsurface cracks, while the laser-textured surfaces maintain relatively low and stable friction as shown in Figure 5.



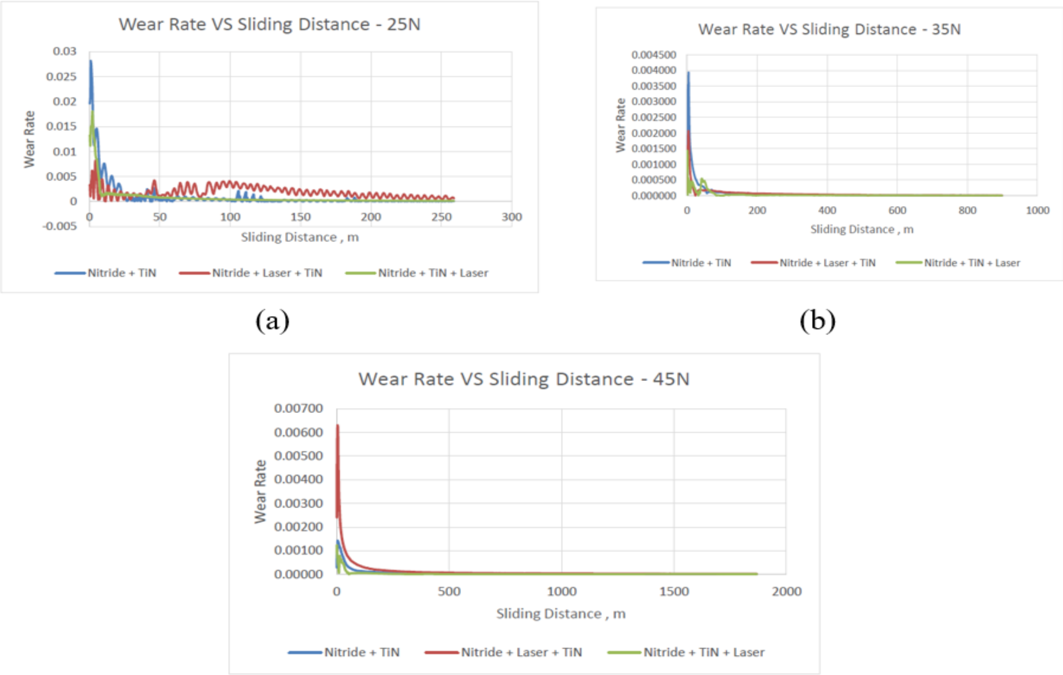
(a)

(b)



(c)

Figure 2 (a-c). Coefficient of friction *vs.* sliding distance for loads of 25 kN, 35 kN and 45 kN



(a) (b)

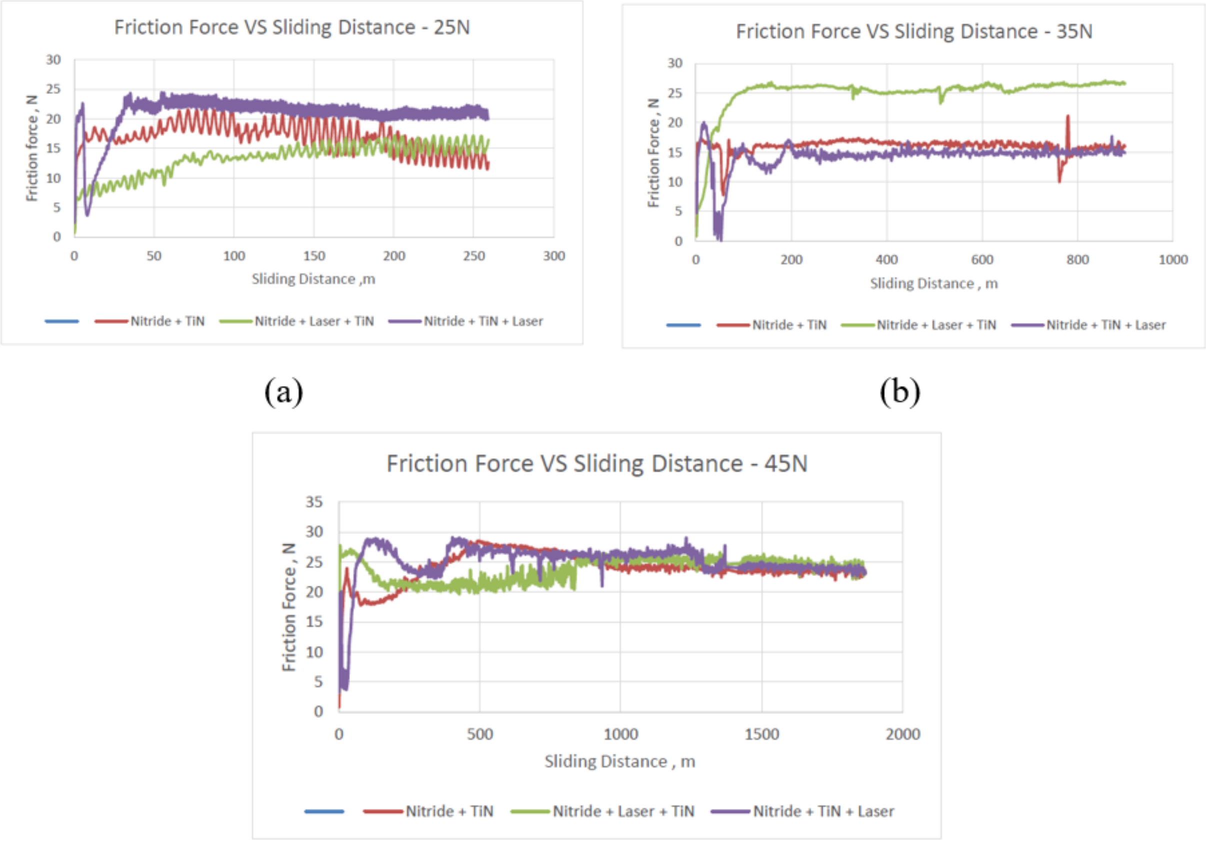
(c)

Figure 3 (a-c). Wear rate *vs.* sliding distance for loads of 25 kN, 35 kN and 45 kN

The wear rate *versus* sliding distance plots at 25 N, 35 N, and 45 N loads show that all treated samples exhibit an initial running-in stage with a relatively high wear rate, which rapidly decreases and stabilizes over longer sliding distances. At 25 N, nitriding + TiN + laser treatments provide lower and more stable wear rates compared to nitriding + laser + TiN, indicating that the sequence of TiN deposition plays a crucial role in reducing wear. At 35 N, all samples demonstrate minimal wear rates after the initial stage, with negligible differences among treatments, reflecting the effectiveness of surface hardening and coating in withstanding moderate loads. The SEM examination. Figure 5 (a and b) reveals micro-fractures at dimple edges post-run-in, with EDX indicating transient Fe-Ti mixing from adhesive pull-out in nitriding + laser + TiN sequence (Figure 6 (a and b). At 45 N, the wear rates are very low and nearly identical across all treatments after an early transient stage, suggesting that the combined impact of nitriding and TiN coating provide resistance to wear, regardless of laser processing order. Overall, nitriding combined with TiN coating ensures superior wear resistance, and while laser processing contributes to surface modification, its effect is less significant at higher loads where the coating's hardness and load-bearing capacity impact the performance. The dimple-induced contact fragmentation is visible in SEM cross-sections confirming intact overlayers and

EDX homogeneity in Ti/N (Figure 5 (b). Sequence 1 (Figure 5 (a)) shows initial peaks (~0.004 µm/m) decaying to baseline, indicative of controlled oxidative wear on the smooth TiN, though EDX detects Ti oxidation. Sequence 3 initiates with the highest rate (~0.008 µm/m), plummeting after delamination exposes the nitride, there is also residual instability. EDX image shows fragmented TiN ejecta and EDS spectra. Figure 6(b) shows elevated subsurface O/Fe ratios.

The friction force *versus* sliding distance plots under different loads (25 N, 35 N, and 45 N) is shown in Figure 4. The graphs reveal that the sequence of surface treatments plays a decisive role in tribological performance. At 25 N, the nitrided + TiN + laser combination shows a more stable and relatively lower friction force compared to the other sequences although treatments involving laser modification exhibit higher initial fluctuations. Nitride + TiN sequence has high frictional force that leads to abrasive wear in exposed nitride surface irregularities, as SEM (Figure 5 (a)) pronounces ploughing tracks and EDX (Figure 6 (a)) reveals Fe-O enrichment from oxidative debris. At 35 N, nitriding + TiN and nitriding + TiN + laser maintain steady friction behavior in the range of 15–20 N, whereas nitriding + TiN + laser results in lower and less stable friction, indicating effective lubrication retention. Under the highest load of 45 N, all samples exhibit a running-in stage followed by stabilization, but nitriding + TiN + laser provides consistently lower friction compared to other sequences. Overall, the results indicate that nitriding enhances hardness and load support, while TiN coating significantly reduces adhesive wear and stabilizes friction [27].

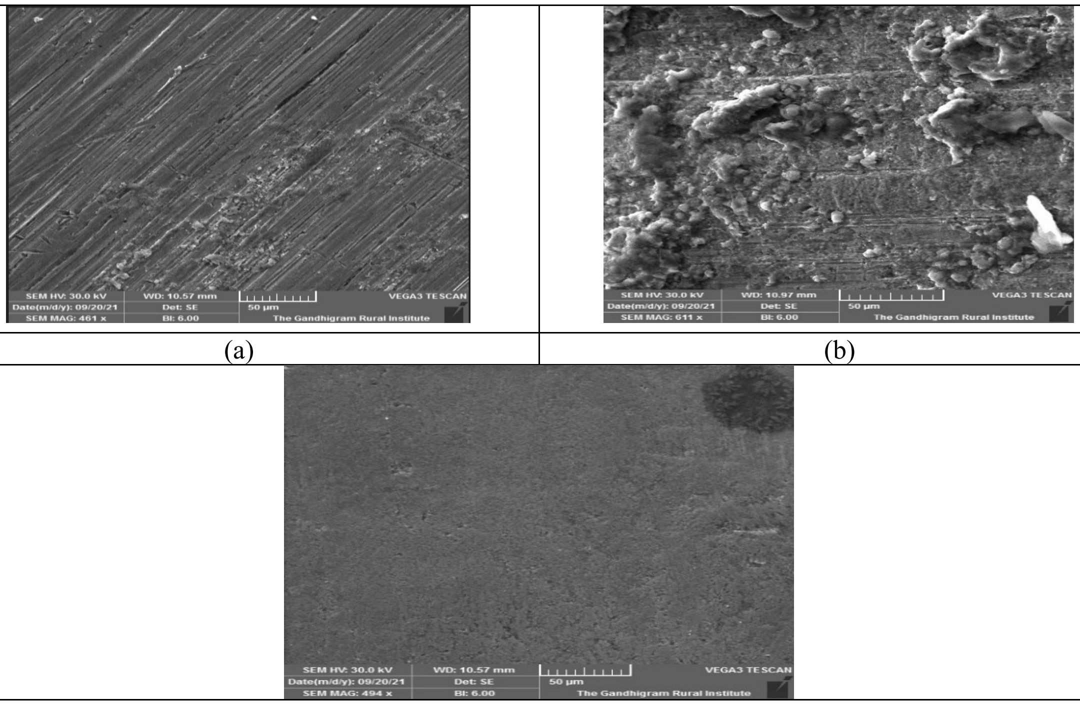


(a)

(b)

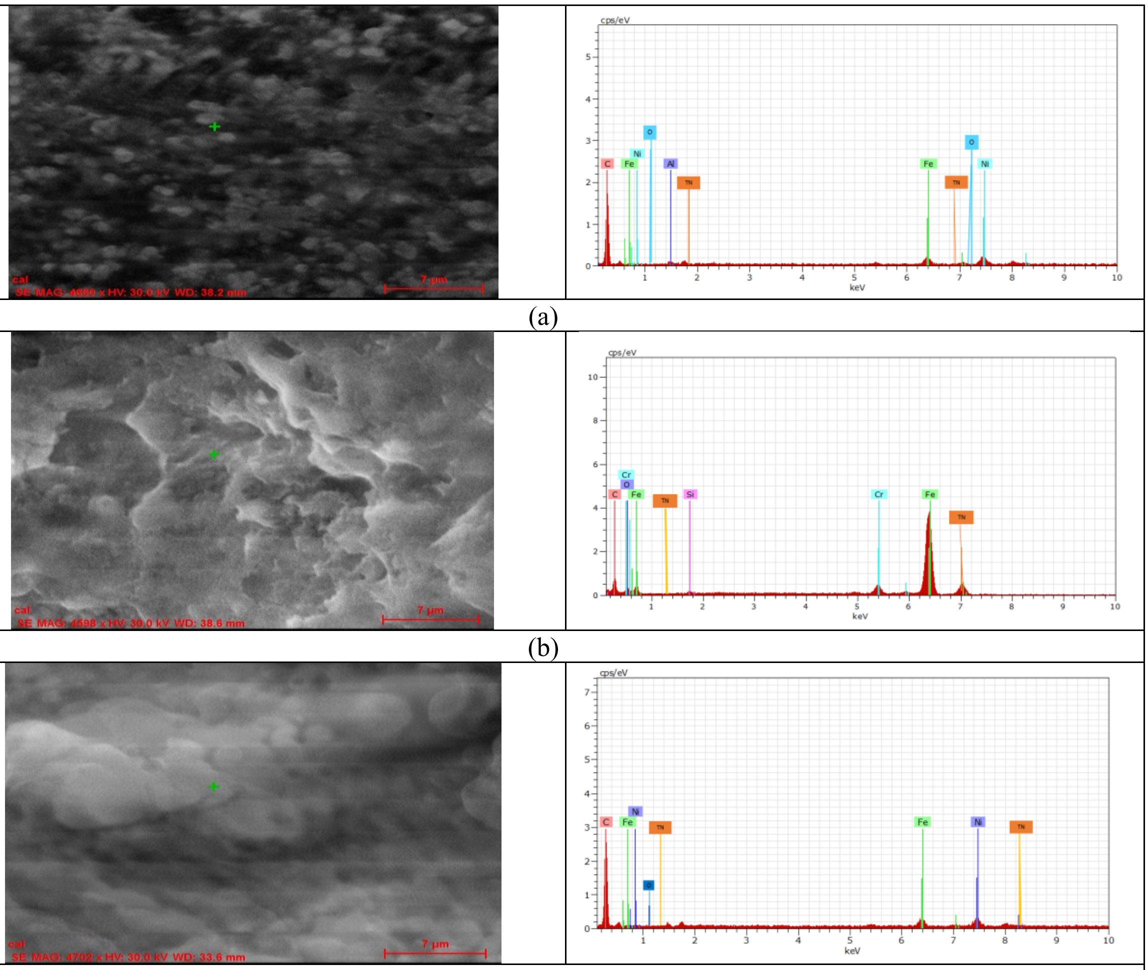
(c)

Figure 4 (a-c). Friction force *vs.* sliding distance for loads of 25 kN, 35 kN and 45 kN



(a) (b) (c)

Figure 5. SEM images of (a) nitride + TiN, (b) nitride + laser + TiN and (c) nitride + TiN + laser



(a) (b) (c)

Figure 6. EDX images and spectra of (a) nitride + TiN, (b) nitride + laser + TiN and (c) nitride + TiN + laser

CONCLUSION

This study demonstrates that the sequence of surface engineering treatments critically governs the tribological performance of AISI 52100 bearing steel under severe dry sliding conditions. Among the investigated configurations, the nitride + TiN + laser sequence exhibited the best performance, achieving wear losses below 5 µm and a stable low coefficient of friction (~0.13) even at 45 N over 2000 m of sliding. SEM/EDX analyses confirmed intact TiN layers, effective debris trapping within laser-textured micro-dimples, and minimal oxidation. In contrast, the nitride + laser + TiN sequence suffered catastrophic wear (>30 µm) and unstable friction due to poor TiN adhesion and coating delamination. The nitride + TiN baseline provided only moderate improvements (15–25 µm wear), highlighting the necessity of textural synergy. Overall, post-coating laser texturing was identified as a key strategy for maximizing wear resistance and frictional stability, with significant potential to extend bearing service life in high-load engineering applications.

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Data availability statement: *The authors confirm that the data supporting the findings of this study are available within the article.*

Research ethics: *Not applicable.*

Use of large language models, AI and machine learning tools: *ChatGPT was used for language improvement.*

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