

Evaluation of gas nitrocarburizing effects on impact-sliding wear behaviour of AISI H13 die steel

Gaz nitrokarbürizasyonunun AISI H13 kalıp çeliğinin darbeli-kayma aşınma davranışı üzerindeki etkilerinin değerlendirilmesi

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Abstract

To simulate the combined impact and sliding forces encountered during the extrusion process, a novel inclined ball-on-plate impact-sliding test was developed to evaluate the wear behavior of the nitrocarburized surface layer on hardened H13 (QT) steel. The treated steels were structurally characterized using a light optical microscope (LOM), accompanied by hardness evaluations. Gas nitrocarburizing notably improved the surface properties of QT steel, increasing surface hardness from 406 HV_{0.05} to 881 HV_{0.05}. The treated layer consisted of an ~8.9 µm white layer and a 110 µm diffusion zone. Wear tracks formed after impact-sliding wear tests were analyzed under the LOM and SEM to identify the underlying wear mechanisms. For the QT steel, severe plastic deformation and material transfer from the counter-body (AISI 52100 ball) contributed to the development of three-body abrasive wear. As a result, the wear rates of the nitrocarburized H13 steel (QT+NC) were reduced by 89% in the impact region, 20% in the sliding region, and 35% overall compared to the QT steel, a performance attributed to the presence of a protective oxide layer on the continuous white layer.

Keywords: AISI H13 die steel, Impact-sliding wear, Nitrocarburising, Steel surface modification

Öz

Ekstrüzyon süreci sırasında karşılaşılan birleşik darbe ve kayma kuvvetlerini simüle etmek amacıyla, sertleştirilmiş H13 (QT) çeliği üzerindeki nitrokarbürleşmiş yüzey tabakasının aşınma davranışını değerlendirmek için yeni bir eğimli bilye-plaka darbeli-kayma testi geliştirilmiştir. İşlem görmüş çelikler, optik ışık mikroskobu (LOM) kullanılarak yapısal olarak karakterize edildi ve sertlik ölçümleriyle desteklendi. Gaz nitrokarbürizasyon, QT çeliğinin yüzey özelliklerini önemli ölçüde iyileştirerek yüzey sertliğini 406 HV_{0.05}'ten 881 HV_{0.05}'e artırdı. İşlem görmüş katman, ~8,9 µm kalınlığında beyaz bir tabaka ve 110 µm derinliğinde difüzyon bölgesinden oluşmaktadır. Aşınma mekanizmalarını belirlemek amacıyla darbeli-kayma aşınma testlerinden sonra aşınma izleri, LOM ve SEM ile incelendi. Su verme ve temperleme işlemine tabi tutulmuş H13 çeliğinde, karşı malzeme AISI 52100 bilyeden malzeme transferi ve şiddetli plastik deformasyon, üç elemanlı abrasif aşınmanın gelişimine katkıda bulundu. Sonuç olarak, nitrokarbürleme işlemine tabi tutulan H13 çeliğinin (QT+NC) aşınma hızları, QT çeliğine kıyasla darbe bölgesinde %89, kayma bölgesinde %20 ve genel olarak %35 oranında azaldı; bu performans, sürekli beyaz tabaka üzerinde oluşan koruyucu oksit tabakasının varlığından dolayıdır.

Anahtar kelimeler: AISI H13 kalıp çeliği, Darbeli-kayma aşınma, Nitrokarbürleme, Çelik yüzey modifikasyonu

1. Introduction

In many forming applications, dies are preheated, and during operation, the surface temperature can exceed 550 °C. As a result of combined mechanical and thermal loading, the die surfaces undergo high-temperature tempering. After several forming cycles, significant wear occurs, leading to dimensional degradation. Additionally, the relative sliding between the workpiece and the die materials contributes to a reduction in die hardness, further compromising die performance (Kumar et al., 2023). These thermal and mechanical challenges are particularly evident in aluminum extrusion processes, where dies are exposed to elevated

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temperatures due to both the preheated billet and the frictional heat generated during deformation. In such environments, not only does die surface tempering and wear occur, but the dimensional accuracy and surface quality of the extruded profiles are also adversely affected (Akhtar et al., 2010; Gasem, 2013). Moreover, high contact pressures and the rapid formation of a hard oxide layer on the extruded aluminum further accelerate wear on the die bearing surfaces. To withstand these demanding conditions, AISI H13 is widely used for the production of extrusion dies and mandrels due to its favorable combination of toughness, hardenability, elevated temperature strength, machinability, and cost-effectiveness (Berrais et al., 2023; Luo et al., 2023).

Among various surface engineering techniques, gas nitrocarburizing stands out as one of the most effective methods for enhancing the surface properties of extrusion dies. This process involves the simultaneous diffusion of carbon and nitrogen at relatively low temperatures, leading to the formation of a hard compound layer and an underlying diffusion zone. However, since it typically affects only the top 0.2-0.5 mm of the surface, the treatment may need to be reapplied after each die regrinding cycle (Bhavar et al., 2016). In addition to its practicality in industrial applications, nitrocarburizing significantly improves surface hardness, wear resistance, fatigue strength, and corrosion resistance without compromising the core mechanical properties of the steel (Boztepe et al., 2018; Dalla Nora et al., 2025). Compared to conventional nitriding, it produces a denser and more uniform compound layer, making it especially suitable for components like H13 extrusion dies that operate under severe mechanical and thermal loads (King et al., 2005).

Numerous widely recognized testing techniques, including the ball-on-disk test (Das et al., 2016; Wang et al., 2018), pin-on-disk test (Pérez et al., 2016; Berrais et al., 2023), scratch test (Vega-Moron et al., 2018), impact test (Gasem, 2013; Peng et al., 2020), and impact-sliding test (Akyıldız et al. 2019), have been employed to assess the performance of H13 steel subjected to various nitriding treatments (salt, gas, and plasma). However, there is a lack of comprehensive studies examining the wear behavior of nitrocarburized H13 extrusion die steel under combined impact and sliding conditions at extremely high contact stress levels. A testing method is still required to evaluate the wear produced by successive impact and sliding movements of nitrocarburized H13 steel under conditions that simulate the extrusion process. To bridge this gap in the literature, particularly for nitrocarburized H13 steel under extrusion-like conditions, it is important to adopt test methods that simulate combined impact and sliding actions. In this context, Chen and Nie (2011) developed the ball-on-plate inclined impact-sliding test, a method specifically designed to replicate the wear mechanisms experienced by materials subjected to repetitive impact-sliding loads in practical applications.

In the present study, the impact-sliding wear behaviour of the as-quench & tempered and as-quench & tempered and gas-nitrocarburized AISI H13 die steels used by a local aluminum extrusion plant was investigated to determine their performance during the extrusion process by a homemade impact-sliding wear tester. This study also clarified how gas nitrocarburizing affects the wear performance of quench & tempered H13 die steel.

2. Material and method

The die steel used in this study matches the chemical composition of AISI H13, as detailed in Table 1. AISI H13 is a standard hot - work die steel, commonly applied in tools like hot forging and extrusion dies due to its excellent toughness and thermal stability (Berrais et al., 2023; Kohli et al., 2023). Circular discs, 30 mm in diameter and 4 mm thick, were sectioned from a rod supplied in the annealed state. An industrial quenching and tempering process was applied to these steels, resulting in a rod hardness of $406 \pm 16 \text{ HV}_{0.05}$. The heat treatment process, used for H13 steels in this work, is depicted schematically in Figure 1. The resulting steels are referred to as quench & tempered (QT) steels.

Prior to gas nitrocarburizing, the QT steels were mechanically finished using 2000-grit emery paper and then mirror polished. They were subsequently cleaned ultrasonically in acetone for 15 minutes before being placed in a gas nitrocarburizing furnace. The industrial gas nitrocarburizing process employed, shown in Figure 2, was performed in a closed NITREX furnace. The volume percentages of gases used during nitrocarburizing were given in Table 2. A modern nitrocarburizing system equipped with advanced hardware and intuitive software was employed in collaboration with a local industrial facility. The system enabled full automation of the process, including all operational steps, safety protocols, and closed-loop control of processing parameters. Steels treated through this method are designated as quench & tempered and gas nitrocarburized (QT+NC) steels.

Table 1. Alloy composition of H13 die steel (annealed condition).

Elements	C	Si	Mn	Cr	V	Mo	Fe
Weight %	0.38	1.00	0.37	5.15	1.00	1.35	Remaining

Table 2. Volume percentages of gases used during nitrocarburizing.

N ₂	60.72±4.1 % (9.9±1.09 m ³ /hrs)
NH ₃	37.95±4.35 % (6.16±0.58 m ³ /hrs)
CO ₂	1.33±0.26% (0.21±0.05 m ³ /hrs)

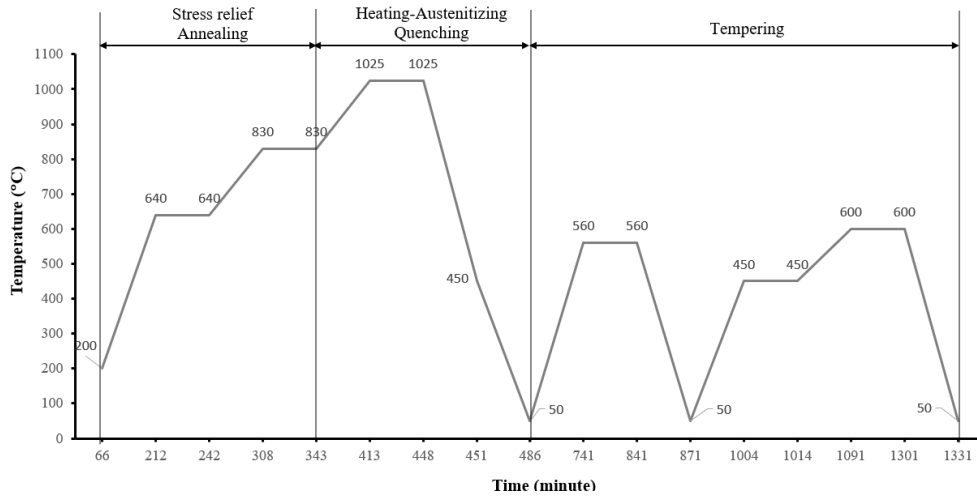


Figure 1. Schematic diagram of quench-tempering heat treatment process.

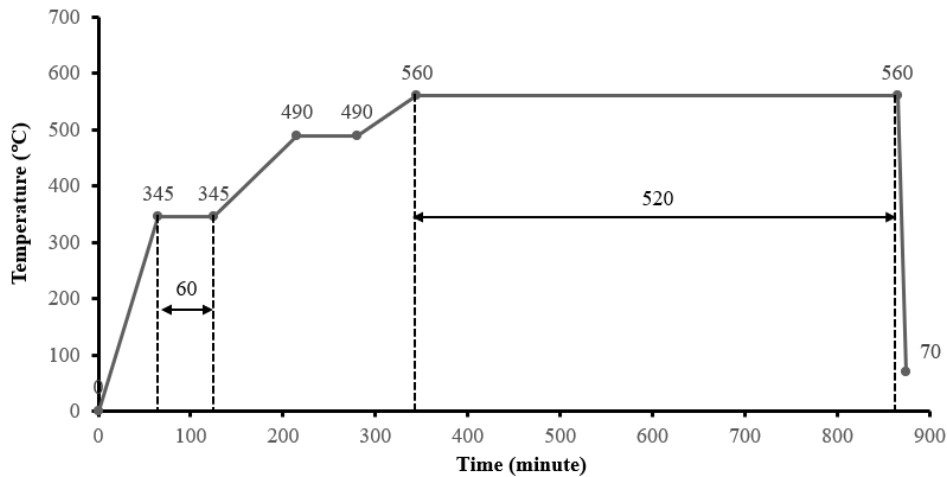


Figure 2. Schematic diagram of industrial gas nitrocarburizing process used in this study.

Cross-sectional analysis of the treated steels was conducted using a Nikon Eclipse LV150 light optical microscope (LOM) following proper polishing and etching with a 2% nital solution. The variation in hardness with depth was assessed using a Shimadzu HVM microhardness tester, applying a 50 g load for 15 seconds.

Figure 3 presents the schematic of the inclined impact-sliding tester developed in our laboratory to replicate the impact-sliding forces generated when billets contact extrusion dies. Figure 3 also includes a typical representation of an impact-sliding wear scar. For each test, the flat treated steels are fixed on a sloped support at a 30° angle. A spring and pneumatic cylinder controlled the applied loading during testing, operating at a frequency of 2.5 Hz. The sample holder, acting as a rocker, rotated around a fixed roller bearing in response to the indenter ball's vertical motion caused by the piston's movement. The spring and pneumatic cylinder were

calibrated using a load cell before testing, ensuring maximum loads of 40 N and 240 N, respectively. Each treated steel was subjected to 4297 impact cycles, reflecting the maximum service life of extrusion dies used in the production of aluminum profiles at a local extrusion facility. The tests were conducted under dry conditions using a commercially available 10 mm-diameter 100Cr6 steel ball counter-body with a hardness of approximately 830 ± 20 HV to simulate steel-steel contact, at a temperature of 20 ± 5 °C and relative humidity of $30 \pm 5\%$. After the impact-sliding wear test, two separate areas of the wear track (impact and sliding regions) having total length of ~ 7 mm were analyzed using a 2D surface profilometer to measure the amount of wear loss. To determine the wear rates of the steels in mm^3/cycle , profilometric measurements were taken at the central points of the impact and sliding regions. The average wear volumes in these regions, derived from the corresponding 2D profiles, were divided by the total number of loading cycles, following the method outlined by Ates et al. (2024). To ensure data consistency, each impact-sliding wear test was repeated at least three times on the treated steels. Finally, the wear tracks were examined in more detail using both the LOM and field-emission scanning electron microscopy (FE-SEM) equipped with an energy dispersive spectrometer (EDS).

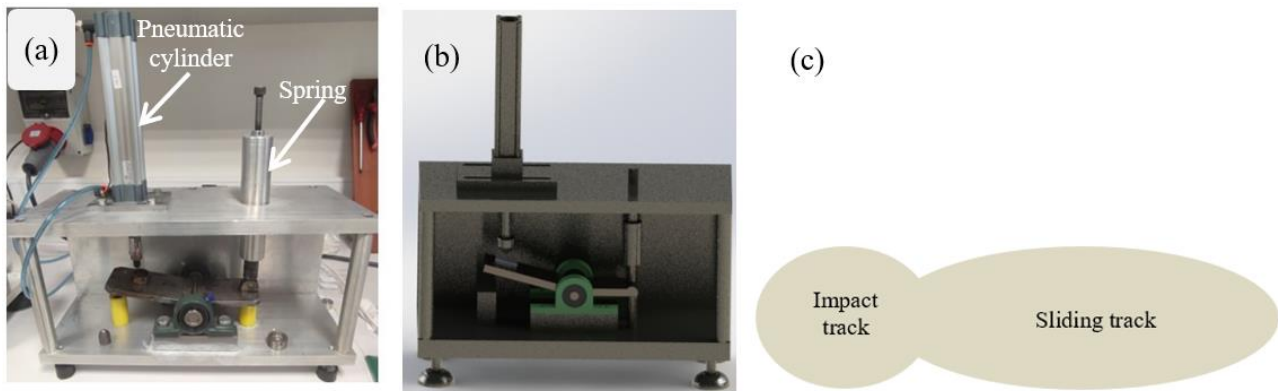


Figure 3. (a) Experimental configuration, (b) 2 D view of inclined impact-sliding wear tester, and (c) schematic view of the wear track after wear tests.

3. Results and discussion

3.1. Microstructure characterization

The quench & tempering heat treatment process in Figure 1 shows that the H13 steel is first hardened and then tempered, which creates a tempered martensitic microstructure (shown in Figure 4). This structure is designed to achieve maximum hardness, thereby improving wear resistance (Berrais et al., 2023). As shown in Figure 4, LOM cannot accurately identify the presence of carbides in the tempered martensite matrix because it has low resolution.

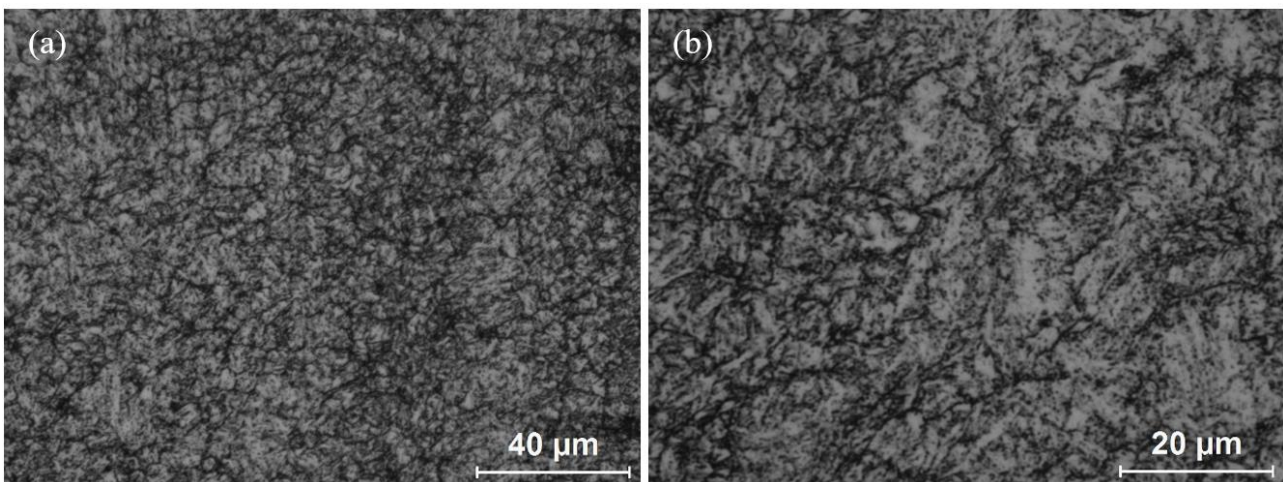


Figure 4. (a) Low and (b) high magnification LOM micrographs of the QT steel.

It is suggested that the nitrocarburized layer consists of two distinct regions: a white (bright) compound layer at the surface, followed by a gray diffusion zone enriched with carbon (C) and nitrogen (N), as shown in Figure 5. The LOM observation indicated that the thickness of the diffusion zone was approximately 110 μm . It is believed that the white layer, approximately 8.9 μm thick, is dense, well-adhered, and mainly consists of $\epsilon\text{-Fe}_{2-3}(\text{C}, \text{N})$ (Psyllaki et al., 2006; Taczala et al., 2023). It is hypothesized that the diffusion zone mainly contains $\gamma\text{-Fe}_4\text{N}$ and $\alpha\text{-Fe}(\text{C}, \text{N})$ nitrides or carbonitrides. A simplified schematic illustration of the typical white layer components formed after gas nitrocarburizing is provided as Figure 6 (Taczala et al., 2023). Furthermore, the possible formation of molybdenum and vanadium-based alloy nitrides or carbonitrides cannot be excluded.

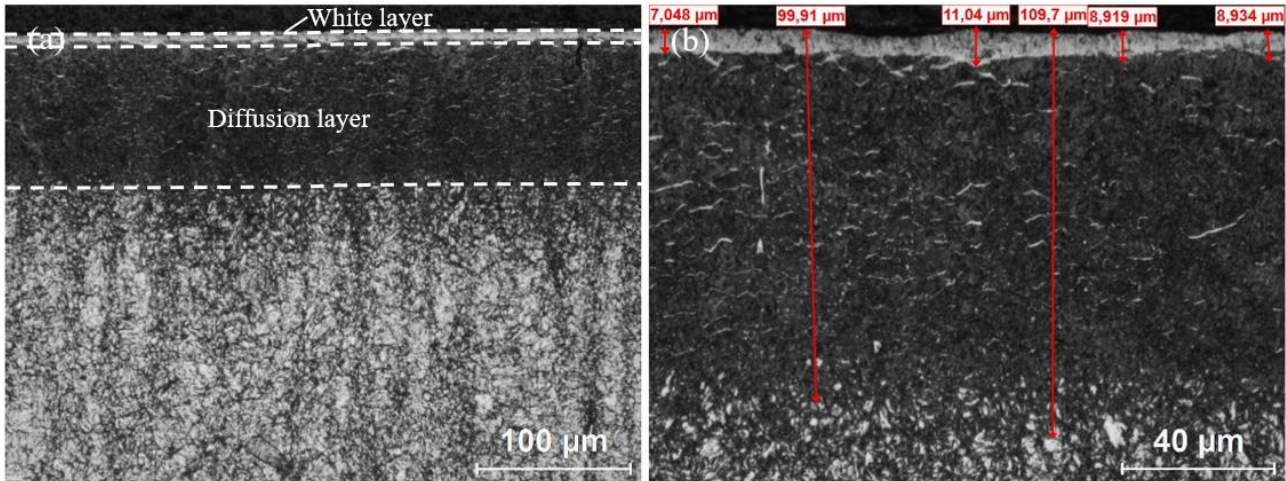


Figure 5. (a) Low and (b) high magnification cross-section LOM micrographs of the nitrocarburized layer.

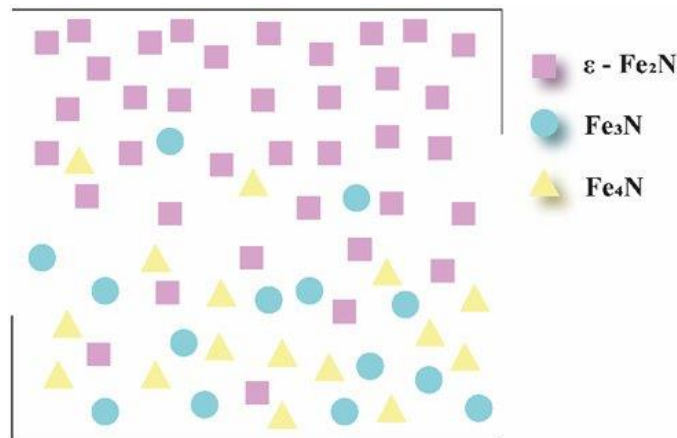


Figure 6. Schematic representation of the phases formed in the layers after gas nitrocarburizing (Taczala et al., 2023).

3.2. Hardness measurements

Figure 7 presents the hardness-depth profile of the treated steels. The surface hardness values for the QT and QT+NC steels are approximately $406 \pm 16 \text{ HV}_{0.05}$ and $881 \pm 18 \text{ HV}_{0.05}$, respectively. According to established criteria, the effective case depth of a nitrocarburized layer is defined as the depth at which the hardness drops to the substrate hardness plus 50 HV (Wang et al., 2018). Using this criterion, the effective case depth of the nitrocarburized AISI H13 steel was determined to be around 110 μm , consistent with the microstructural features observed in Figure 5. The nitrocarburizing treatment notably increased the surface hardness of the QT steel. This hardened region gradually merges with the core material, with the observed decline in hardness resulting from the diminishing C and N content. Akhtar et al. (2010) obtained a shallow effective case depth of approximately 90 μm by adjusting the nitriding potential. This resulted in a thin compound layer, 4-10 μm thick, predominantly composed of monophase $\epsilon\text{-Fe}_3\text{N}$, which is less brittle, more ductile, and better able to withstand cracking under compressive stresses typical in extrusion die applications.

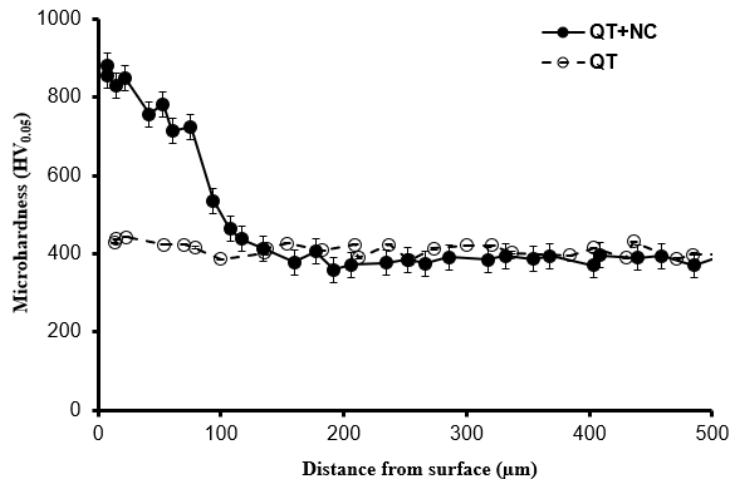


Figure 7. Hardness profiles of both QT and QT+NC steels.

3.3. Impact-sliding wear

Following impact-sliding wear testing, both QT and QT+NC steels exhibited characteristic two-dimensional wear tracks, as shown in Fig. 8. In QT steel, the impact region was deeper and wider than the sliding region. For QT steel, shear lips formed along the edges of the impact and sliding regions, with more pronounced formation in the impact region. This data indicates that massive plastic deformation played a role in wear progress in QT steel. However, the nitrocarburizing treatment did not significantly influence shear-lip formation. Instead, nitrocarburizing led to reduced wear track depth and width in both regions. In nitrocarburized steel, the depth of wear tracks caused by the steel ball was less than that of the white layer ($\sim 8.9 \mu\text{m}$) in both the impact and sliding regions.

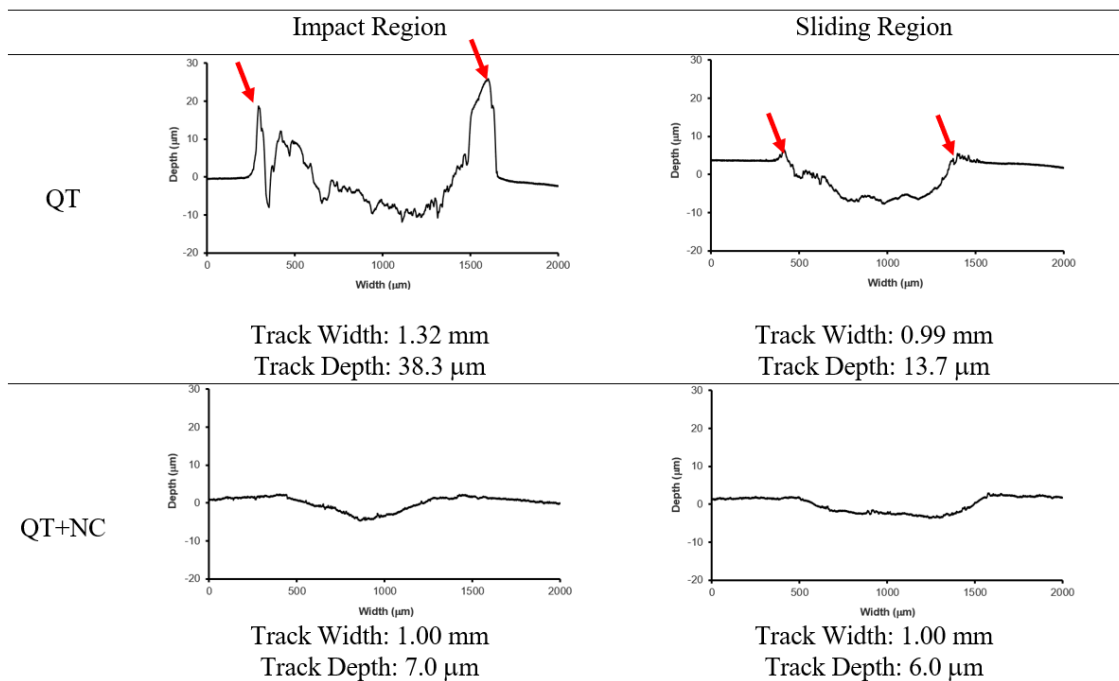


Figure 8. Two-dimensional profiles of the wear tracks for both QT and QT+NC steels, with arrows indicating the shear lips.

Figure 9 shows the average wear rates of the QT and QT+NC steels in the impact and sliding regions. Nitrocarburizing reduced the wear rate in the impact region by approximately 89%, but only by 20% in the sliding region. On the other hand, gas nitrocarburizing resulted in a $\sim 35\%$ decrease in the overall wear rate. Figs. 10 and 11 show the LOM images of the worn surfaces formed on the QT and QT+NC steels. The

evaluation of wear rate data in Figure 9, together with the worn surfaces and counter-body images in Figure 10, indicates that nitrocarburizing had a notable impact on the wear performance of the treated steels. Figure 8 implies that the white layer on the surface of the nitrocarburized steel did not completely peel off in both the impact and sliding regions. Nitrocarburizing made the H13 steel better at resisting wear from both the impact and sliding regions by forming a continuous white layer on its surface (Figure 5). Studies have shown that nitrocarburized H13 steel exhibits a notable enhancement in wear resistance, primarily due to the formation of $\epsilon\text{-Fe}_{2-3}(\text{C}, \text{N})$ and $\gamma'\text{-Fe}_4\text{N}$ phases in the white layer (Basso et al., 2007). As illustrated in Figure 9, the repetitive impact-sliding conditions revealed that the increased surface hardness (Figure 7), primarily attributed to the formation of $\epsilon\text{-Fe}_{2-3}(\text{C}, \text{N})$ and $\gamma'\text{-Fe}_4\text{N}$ phases within the white layer, contributed significantly to the enhanced wear resistance of the QT+NC steel in both the impact and sliding regions. Low-magnification LOM examinations showed that the nitrocarburized steel created diminished wear marks in terms of depth and width across both the impact and sliding regions (Figure 10). Upon impact, the QT steel exhibited a distinct and deep crater on its surface (Figure 10). The sliding region of QT steel (Figure 10) displays numerous grooves and scratches, typical of abrasive wear. The wear marks are noticeably larger and deeper compared to those on QT+NC steel. The formation of these grooves and scratches may be attributed to plastic deformation of the QT steel resulting from contact with the counter-body (Figure 10), or they could be the result of abrasive action by hardened and oxidized wear particles during sliding. Additionally, adhered particles are visible in the sliding region (Figure 11). On the other hand, the wear track in the sliding region for QT+NC steel had oxide patches (Figures 11 and 12). The existence of the oxide layer was confirmed by the EDS result (Figure 12 b). This is a common sign of tribo-oxidation that happens when higher test loads cause a lot of friction heat (Peng et al., 2018).

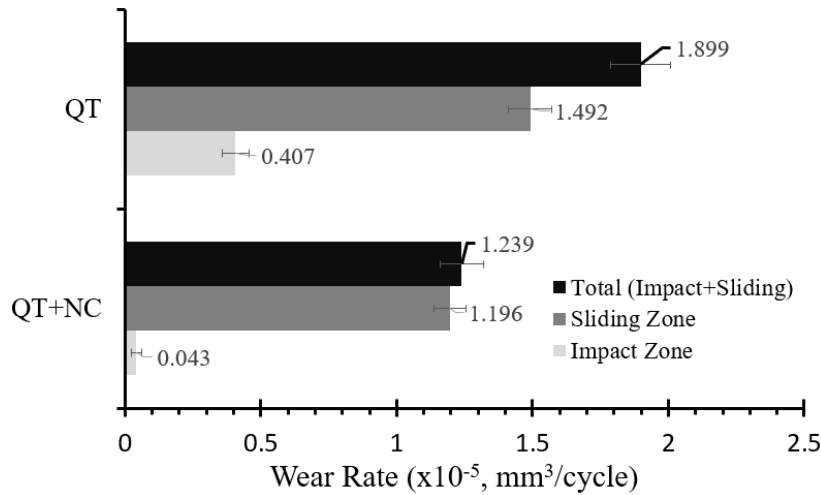


Figure 9. Wear rates of the wear tracks developed on the treated steels' surfaces.

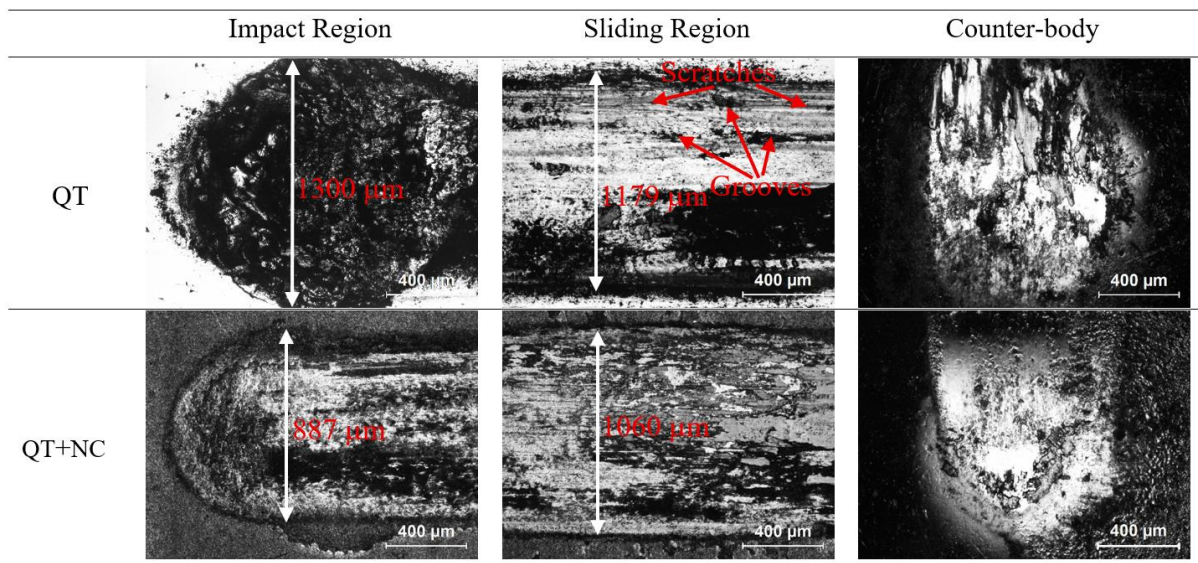


Figure 10. Low-magnification LOM micrographs on impact-sliding tracks of the QT and QT+NC steels.

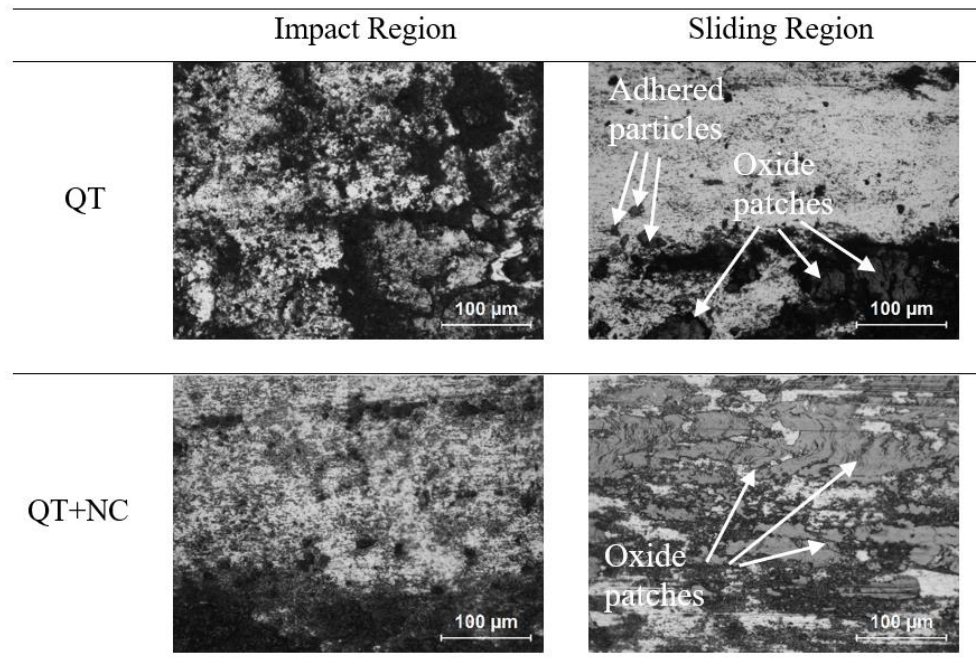


Figure 11. High-magnification LOM micrographs on impact-sliding tracks of the QT and QT+NC steels.

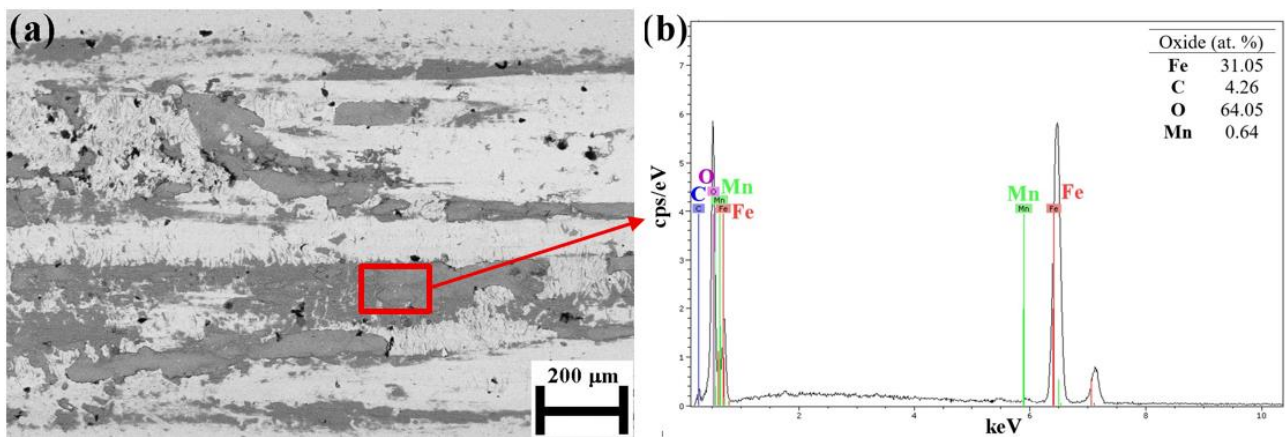


Figure 12. (a) FE-SEM image and (b) EDS result of the wear track formed on the sliding region of the QT+NC steel.

4. Conclusions

The study used a cyclic wear test with impact and sliding under an inclined setup on treated H13 steels (QT and QT+NC). The current research allows us to conclude the following:

- 1- The gas nitrocarburizing process significantly enhanced the surface hardness of QT steel. Measured surface hardness values for QT and QT+NC steels were 406 ± 16 HV_{0.05} and 881 ± 18 HV_{0.05}, respectively.
- 2- The nitrocarburized layer was visibly composed of a continuous white layer approximately 8.9 µm thick, beneath which lay a gray diffusion zone measuring around 110 µm.
- 3- The wear rate of QT+NC steel decreased by 89% in the impact region and by 20% in the sliding region compared to QT steel.
- 4- Nitrocarburizing led to a substantial 35% decrease in the total wear rate.
- 5- Based on the wear rates observed in the study, the QT+NC steel, having higher hardness, demonstrated superior wear resistance compared to QT steel, as evidenced by its lower total volumetric wear loss.

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Author contribution

Tunahan Fatih Sekili: Investigation, Experimental Work, Writing Review & Editing. Elif Ozturk: Investigation, Experimental Work, Writing Review & Editing. Harun Mindivan: Conceptualization, Investigation, Experimental Work, Writing Original Draft, Writing Review & Editing.

Declaration of ethical code

The authors declare that this study does not require ethical committee approval or any legal permission.

Conflicts of interest

The authors declare no competing interests.

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