Turkish Journal of Engineering
https://dergipark.org.tr/en/pub/tuje
e-ISSN 2587-1366

Investigation of the Tribological and the Corrosion Behavior of Liquid Nitrided EN31 Steel

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Cite this study: Alphonse, M., Krishna, P.Mi., Nachiappan, M.N., Lazar, P.J.L., (2025). Investigation of the Tribological and the Corrosion Behavior of Liquid Nitrided EN31 Steel. Turkish Journal of Engineering, 9(3), 591-599.

https://doi.org/10.31127/tuje.1674812

Keywords
EN31
Liquid Nitriding
Pin-on-disk test
Salt spray test
Corrosion resistance

Research Article

Received:12.04.2025
Revised:22.08.2025
Accepted:23.08.2025
Published:30.10.2025

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Abstract

EN31 steel is a high-carbon alloy steel used for several industrial applications like drilling & power tools. The novelty of this study lies in the optimization of liquid nitriding duration for the EN31 steel, which enhances the surface properties and a prolonged service life in tribological applications. In order to overcome the limitations, this study has aimed to investigate the impact of liquid nitriding at 570°C for 30, 60, and 90 minutes to study the hardness, corrosion and wear properties of EN31 steel. As a thermochemical surface treatment for EN31 steel, the study explores the application of liquid nitriding. Compared to traditional methods like gas nitriding, carburizing, hard chrome plating and hardening, the liquid nitriding provides uniform compound layer formation, enhanced surface hardness, and faster processing with less distortion. Performance of the modified EN31 steel post nitriding process was evaluated through various tests, like the pin-on-disk method to assess wear resistance and neutral salt spray testing as per ASTM B117 to measure corrosion resistance. Noticeable improvements in the steels tribological and anti-corrosive properties after nitriding were demonstrated through the test results. Upon analysing the microstructure, a uniform, defect-free nitride layer was revealed. The study evaluated that the 90-minute nitriding cycle resulted in the best performance related to corrosion resistance, wear resistance and surface hardness. A maximum hardness of 31.7 HRC would be achieved during the 90 minute cycle. Interestingly, this cycle also produced the deepest effective case depth thereby enhancing durability of steel under intense load bearing conditions. In addition, the outstanding corrosion resistance of steel was confirmed during 24-hour salt spray testing as no red rust was detected on the nitride samples. The test findings are validated the liquid nitriding as an economical surface treatment process for improving performance as well as durability and also reducing maintenance needs of EN31 steel for applications like drilling tools and for enhancing its reliability in abrasive and corrosive environments.

1. Introduction

The materials science engineering field has played a key role in advancing the high-performance alloys along with improved corrosion protection and wear resistance. Among these materials, EN31 steel is a high-carbon alloy steel and it is mostly utilized in industrial applications due to its excellent mechanical properties [1,2]. It is also exhibiting superior hardness and durability when it is exposed to extreme conditions like high friction and corrosive environments [3,4] and often leads to deterioration on surface over time. To address above said challenges, surface modification techniques like nitriding have been explored to improve the performance of AISI 5140 steel in critical applications, including drilling tools and machine components [5]. Tribology is particularly focus on friction, wear, and lubrication and it is essential for understanding material behaviour under operating loads. Wear and corrosion are two primary factors that influence the service life of components in industrial applications. Wear happened due to mechanical interactions between contacting surfaces and leading to material loss whereas corrosion arises from chemical reactions with environmental elements and it is causing the structural damage. By selecting the effective surface treatments can significantly improve the performance and durability of engineering components by limiting these degradation mechanisms. Liquid nitriding is a process of thermochemical surface treatment that diffuses nitrogen into the surface of ferrous materials and forming a hardened nitride layer which enhances the wear resistance fatigue strength and corrosion resistance [6-8]. By comparing with traditional heat treatment methods [9,10], liquid nitriding is performed at relatively lower temperatures which minimizing distortion while ensuring uniform nitrogen diffusion. This makes it particularly beneficial for materials like EN31 steel, where getting an optimal balance between surface hardness and core toughness is important for excellent performance [11]. There is limited research on the wear and corrosion behaviour of liquid nitride EN31 steel and with most studies focusing on other steel grades [12,13]. Existing research mainly examines wear or corrosion separately and lacking a combined analysis of both properties in EN31 steel. The nitriding parameters influence on its performance is not well explored and comparative study is not there between liquid nitriding and other nitriding techniques. Additionally, the relationship between microstructural changes and performance remains unclear and practical applications are underexplored in industrial environments [14,15]. This study main aim is to bridge these gaps by providing a comprehensive evaluation of liquid nitride EN31 steel.

This study aims to examine the effects of liquid nitriding process in EN31 steel by observing the wear and corrosion resistance. The novelty of this study lies in the optimization of liquid nitriding duration for the EN31 steel, which enhances the surface properties and a prolonged service life in tribological applications. The investigation involves subjecting EN31 steel samples to nitride at a controlled temperature of 570°C for different durations (30, 60, and 90 minutes) to analyse the impact of process parameters on material characteristics [16]. Wear behaviour is studied by using a pin-on-disk tribometer to simulate high-friction conditions typical in drilling operations. Corrosion resistance is studied through neutral salt spray testing and replicating exposed to aggressive environments [17-19]. Additionally, surface hardness, case depth, and microstructural features are analysed to establish a relationship between the improvements in material properties and the nitriding process.

This research mainly focuses to optimize the liquid nitriding process for EN31 steel and highlighting its potential to enhance the service life and reliability of components used in demanding industrial applications. By systematically investigating the influence of nitriding cycle duration, the study provides valuable insights into selecting optimal process parameters for achieving desired performance improvements. The findings contribute to the field of materials engineering by presenting an economical and effective solution for enhancing the tribological and corrosion resistance properties of high-performance steels.

2. Method

2.1. Surface Preparation

The preparations of EN31 steel specimens were essential to ensure consistent and accurate results. The process mainly consists of cleaning, machining and polishing to remove contaminants and to achieve the uniform dimensions. It is also useful to create a smooth surface suitable for nitriding. The first step was cleaning and in this process the steel bars were thoroughly washed using chemical agents and degreasers to eliminate dirt, oil, grease and other impurities. This step was very important as a clean surface improves nitrogen diffusion and making the nitriding process very effective. After cleaning activity, the specimens were machined into cylindrical shapes with a diameter of 10 mm and a length of 30 mm using precision cutting machines as shown in Figure 1. This ensured uniformity among all specimens to reducing variability in testing. The final step was polishing and its involved grinding with different abrasives to achieve a smooth and reflective surface. A well-polished surface increases wear testing accuracy by ensuring consistent frictional contact. Following this systematic preparation process helped to remove contaminants, maintain uniformity and optimizes the specimens for reliable testing. EN31 steel specimen was tested in lab for its chemical composition and results shown in Table 1. Figure 2 shows the EN36 material prepared for disc.

Figure 1. EN31 Steel Material prepared for Testing

Figure 2. Disk specimen

Table 1. EN31 Steel compositions

Elements	% weight
C	0.963
Si	0.246
Mn	0.351
P	0.013
Cr	1.44
S	0.006

2.2. Liquid Nitriding Process

Liquid nitriding is a widely used surface treatment method and it is significantly increasing the mechanical properties of steel by improving surface hardness, corrosion and wear resistance. This study mainly focus on the liquid nitriding of EN31 steel to find out the improvements in surface characteristics and involving the immersion of steel samples in a molten salt bath containing nitrogen-releasing compounds and in this process it's allowing nitrogen atoms to diffuse into the surface and form a hardened nitride layer. The main key factors included in this process are salt bath composition, nitriding duration, treatment temperature, cooling and post-treatment procedures and these were carefully controlled to get the optimal results. The composition of the molten salt bath played an important role in nitrogen diffusion with Sodium Cyanide (NaCN) acting as the primary nitrogen donor, Sodium Carbonate (Na2CO3) maintaining bath stability and Potassium Carbonate (K2CO3) improving nitrogen solubility to ensure a uniform and hardened surface. Treatment temperature and duration were also crucial with steel specimens treated at 570°C to facilitate nitrogen absorption while maintaining toughness [20].

For an EN31 steel, extending the nitriding duration beyond 90 minutes may often leads to excessive compound layer thickness, which could result in cracking, brittleness on the surface, and also reduces the fatigue strength. Based on the hardness-depth and microstructural stability, 90 minutes is the optimal maximum, and 30 minutes is the minimum for the effective surface hardening. The higher durations of nitriding may risk the tribological properties. In this process, three nitriding durations were studied: first one 30 minutes and it is provided a moderate increase in hardness but limited nitrogen penetration, second one 60 minutes and it is improved nitrogen diffusion hence leading to better hardness and wear resistance and the third one 90 minutes which achieved the most extensive nitrogen diffusion and it is enhancing surface properties as shown in Figure 3. After nitriding, the specimens were naturally cooled in air to prevent thermal shock and avoid cracks or defects and finally cleaning process to be provided to remove any remaining salt deposits, ensuring a contaminant-free and durable surface.

Figure 3. Liquid Nitride EN31 steel samples

2.3. Wear Testing

In this testing Pin-on-Disk Tribometer was used for testing the nitride EN31 steel samples to know the wear resistance under controlled conditions [21,22] as shown in Figure 4. This method preciously measured material loss and frictional behaviour during wear. Before testing, the tribometer was calibrated for accuracy [23].

The setup included a rotating hardened steel disc EN26 and a nitride EN31 steel pin pressed against the disk under a constant load. A 3 kg load was applied to the pin, with a sliding velocity of 2 m/s and a total sliding distance of 200 meters. Three different sample (30-minute, 60-minute and 90-minute) were tested for 12 minutes to evaluate wear characteristics. During the test, frictional force and wear depth were recorded in real-time and finally the wear track on the disk was analysed to measure material removal. The wear rate is calculated after all the experiments were conducted by using the equation 1 & 2.

Wear Volume, V= $w_1 - w_2/\rho$ ——— 1

Where w1-w2 is the mass loss and ρ is the density of the pin material

Wear Rate = V/Fd ——— 2

Where F is the load in N, d is the sliding distance

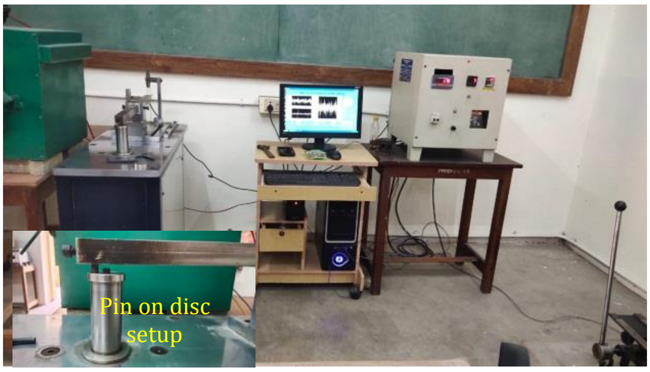

Figure 4. Pin-on-Disk Test setup

2.4. Corrosion Testing

The liquid nitride EN31 steel samples were further tested using a Salt Spray Test following the ASTM standards [24] to know the corrosion resistance. Harsh condition was simulated in this test to know the samples' ability to resist corrosion. In this test, the samples were placed in a controlled salt mist chamber at a temperature between 34.5°C and 35.5°C and positioned at an angle of 15-30 degrees for uniform exposure. A salt solution containing 4.8-5.3% sodium chloride (NaCl) was used with a pH level maintained between 6.65 and 6.85. The samples will be kept in the chamber for 24 hours to observe corrosion effects. Periodic inspections were carried out to detect discoloration or surface damage and rust. This result helped to know how different nitriding durations affected corrosion resistance and confirming the effectiveness of liquid nitriding in protecting EN31 steel in harsh environments.

2.5. Case Depth and Hardness Measurement

The Liquid nitriding on EN31 steel creates a hard surface layer called as surface hardening, with a specific penetration called as case depth. This supports the hardened surface under various load, better wear resistance and fatigue life. The Optimal balance ensures the performance and durability, which is crucial in demanding applications. Also, by studying the case depth and surface hardening provides an optimal solution for the factors maximizing the service life and reliability [25].

The case depth and surface hardness of the nitride EN31 steel samples were measured to know the effectiveness of the nitriding treatment [26]. Case depth measurement involved to know the hardness profile at various depths from the surface ranging from 0.1 mm to 1.0 mm. A Vickers hardness tester was used for this purpose to know the precise hardness readings at different depths. This analysis provided very crucial data on the extent of nitrogen diffusion into the steel and the depth at which the nitride layer retained its enhanced hardness. Along with case depth measurement, surface hardness measurements were also conducted using the Rockwell hardness test to determine the overall hardness improvement achieved through liquid nitriding [27,28]. Hardness values were recorded for samples treated for different durations like 30 minutes, 60 minutes, and 90 minutes to know the impact of varying nitriding time. The results were compared to analyse how nitriding duration influenced not only surface hardness but also wear resistance, corrosion resistance and overall mechanical performance of EN31 steel. These findings played an important role in determining the effectiveness of liquid nitriding as a surface treatment method for enhancing the durability and reliability of steel in demanding industrial applications.

3. Results and discussions

3.1. Wear Test Results

A pin-on-disk test was conducted to evaluate the effect of liquid nitriding on the wear resistance of EN31 steel by replicating real-world friction conditions found in industrial machinery. Figure 5 shows the pin and disc before and after the tribological investigation. Three steel samples were nitride for 30, 60, and 90 minutes before testing to measure material loss and analyse the impact of nitriding duration on wear resistance [29-31]. The results showed that longer nitriding durations leads to lower wear rates, with the 90-minute nitride sample exhibiting the least material loss due to the formation of a deeper and more uniform nitride layer by reducing friction and minimizing material degradation. In contrast, the 30-minute nitride sample had the highest wear rate due to an underdeveloped nitride layer by making it more prone to wear.

The By comparison of the three samples and found that the 30-minute sample had the highest wear rate of 3.12 mm^3/Nm, followed by the 60-minute sample with 2.45 mm^3/Nm and while the 90-minute sample showed the lowest wear rate of 1.97 mm^3/Nm. These findings showed that increasing nitriding duration enhances wear resistance and making EN31 steel more durable under frictional conditions. The key findings from this investigation are a material loss for 30 minute is 0.011 grams, 60 minute is 0.0093 grams and 90 minute is 0.0061 grams as shown in Figure 6. A prolonged nitriding process leads to thicker and uniform nitriding layer resulted in less material loss. The tribological performance was enhanced because of larger diffusion of nitrogen in the surface of EN31 material. Based on the depth of diffusion depth, the 90 minute nitriding process enhanced the load bearing capacity.

Figure 5: Pin & Disk a) Before test b) After test

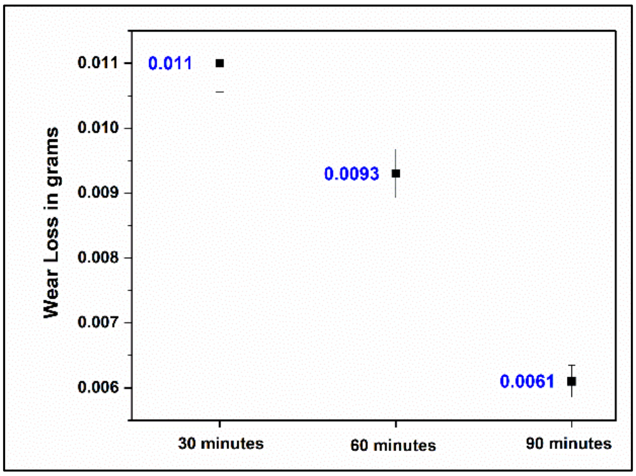

Figure 6. Wear loss in the Liquid Nitride Sample

Figure 7 illustrates the Coefficient of Friction observed during the nitriding process. The experimental results demonstrate a direct correlation between the friction reduction and duration of nitriding. As the duration of nitriding increases, the CoF decreases, indicating improved tribological performance. Specifically, CoF values diminish from 0.01024 to 0.0038, and further to 0.0026 at 30, 60, and 90 minutes, respectively. This reduction helps in enhancing the tribological properties logically to the formation of iron nitrides on the surface of samples. Results revealed a more uniform and controlled diffusion of nitrogen layers has formed into the steel surface, which has promoted the formation of a consistent and high-quality iron nitride layer. Iron nitrides is well known for wear resistance and high hardness, form a robust surface layer. The longer nitriding durations helps in promoting the formation of a thicker and more robust iron nitride layer. This thicker layer minimizes the direct contact between the sliding surfaces, which logically reduces the friction and wear by providing a more effective and durable interface.

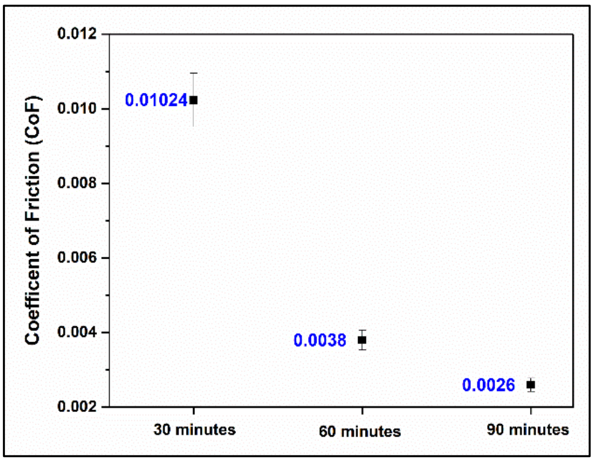

Figure 7. Coefficient of friction (CoF) in the Liquid Nitride Sample

3.2. Salt Spray Test Results

The liquid nitride EN31 steel samples were further tested using a Salt Spray Test following the ASTM standards to know the corrosion resistance. Harsh condition was simulated in this test to know the samples' ability to resist corrosion. Steel samples were placed in a controlled chamber and exposed to a fine mist of saltwater for 24 hours to examine their resistance to rust formation and corrosion [12]. The results evaluated that nitride samples exhibited excellent corrosion resistance along with no red rust observed and indicating that the nitride layer effectively protected the steel by acting as a barrier against moisture and salt, the main causes of rust Longer nitriding durations provided even better corrosion resistance due to the formation of a denser and more uniform protective layer, making nitride EN31 steel suitable for applications exposed to moisture, chemicals or humid conditions. Industries such as automotive, aerospace and marine engineering can benefit from its enhanced durability, confirming that liquid nitriding is an effective treatment for improving both corrosion resistance and overall performance in demanding environments.

3.3. Case Depth Measurement

The effect of liquid nitriding on the hardness of EN31 steel was evaluated by analyzing the case depth of three samples treated for different durations to understand how nitriding time influences the hardened layer. Since nitriding enhances wear resistance by hardening the surface and determining the extent of hardness penetration is essential for optimizing performance [32,33]. The Vickers Hardness (HV) test was conducted at various depths and results converted to Rockwell Hardness (HRC) for better comparison. Three samples like 30 minutes, 60 minutes and 90 minutes samples were tested by showing a clear correlation between nitriding duration, case depth, and hardness retention. All samples had high surface hardness, gradually decreasing with depth due to the diffusion-based nature of nitriding. The results observed has shown in Figure 8. The 30 minutes sample had a surface hardness of 35.9 HRC at 0.10 mm but dropped to 15.2 HRC at 0.70 mm, indicating limited hardness penetration. 60 minutes sample exhibited slightly lower surface hardness at 32.8 HRC at 0.10 mm but maintained 15.2 HRC up to 1.0 mm. 90 minutes sample showed the most significant improvement, with a surface hardness of 38.4 HRC at 0.10 mm and retaining 17.6 HRC at 1.0 mm by indicating the deepest and most effective hardened layer. These findings confirm that longer nitriding durations increase surface hardness and case depth, improving wear resistance and durability. While all samples underwent successful case hardening and 90 minutes sample had the highest surface hardness and deepest case depth, making it the most resistant to wear and mechanical stress.

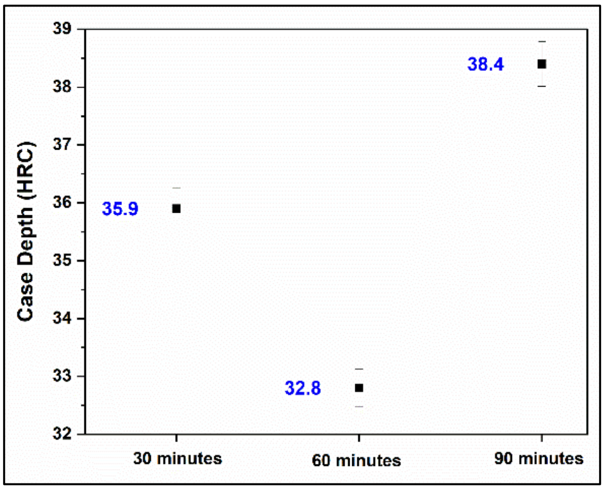

Figure 8. Case Depth of each Samples

3.4. Surface Hardness Analysis

Surface hardness is critical for determining a material's resistance to friction, wear and mechanical stress and it especially in high-friction environments like gears, bearings, and cutting tools [17]. This study examined the effect of liquid nitriding duration on the surface hardness of EN31 steel by analysing three samples as shown Figure 9 and the hardness measured along the surface is shown in Figure 10.The results evaluated a direct correlation between nitriding time and surface hardness with 30 minutes sample exhibiting the lowest hardness 29.3 HRC due to limited nitrogen diffusion, 60 minutes sample showing moderate improvement 29.77 HRC) with increased wear resistance and whereas 90 minutes sample achieving the highest hardness 30.03 HRC due to deeper nitrogen penetration. The progressive increase in hardness from 30 minutes sample to 90 minutes sample confirms that longer nitriding durations increases surface hardening, improving performance and durability. Despite slight variations, all samples displayed uniform hardness, ensuring even wear and minimizing the risk of localized soft spots. Among them, 90 minutes sample was the most suitable for high-wear applications due to its superior hardness, while 60 minutes sample provided a balanced improvement and 30 minutes sample was better suited for moderate wear conditions. This study highlights that prolonged nitriding improves surface hardness and making it a reliable method for enhancing the mechanical properties of EN31 steel.

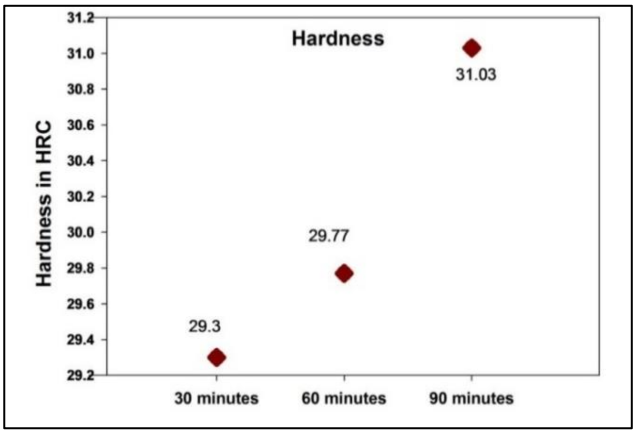

Figure 9. Hardness Measured on the Surfaces

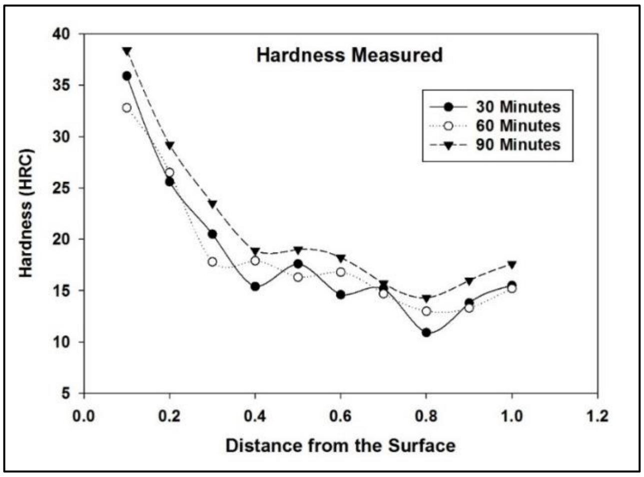

Figure 10. Hardness Measured on the Surfaces

3.5. Microstructural Analysis

To evaluate the effectiveness of the hardened layer, quality and uniformity developed during the liquid nitriding process, the microstructural analysis of nitrided EN31 steel samples was performed [34,35]. It is essential for optimizing the nitriding process for understanding the diffusion of nitrogen into the material and the impact on mechanical properties, such as wear resistance, hardness, and corrosion resistance. To examine the nitride layer in detail, a high-magnification imaging at 20 µm was employed as shown in the Figure 11, which focuses on potential defects, consistency, and the influence of different nitriding durations on the steel's microstructure.

The microstructural characteristics varied depending on the nitriding duration. During 30-minute nitriding duration the analysis revealed the presence of a distinct diffusion layer near the surface. The nitriding surface appeared very smooth, but in the localized areas fine nitrides were observed. During 60-minute nitriding duration a diffusion layer was evident, also accompanied by the formation of a precipitation zone beneath the surface. Fine nitrides were also observed a few places, though their distribution was not uniform. In the 90-minute nitriding duration the microstructure exhibited a more homogenous distribution of nitrides throughout the treated zone and a well-defined diffusion layer were observed. In general, a high-magnification imaging at 20 µm revealed a well-developed and consistent nitride layer in all the samples, which suggests even wear resistance across the surface. This uniformity is very essential for the industrial applications where components experience constant mechanical stress and well as tribological issues. No structural defects, such as porosity, cracks or any other surface irregularities were detected, this confirms the successful execution of the nitriding process without introducing material weaknesses. The absence of defects ensures that the EN31 steel which maintains its strength while benefiting from enhanced surface properties.

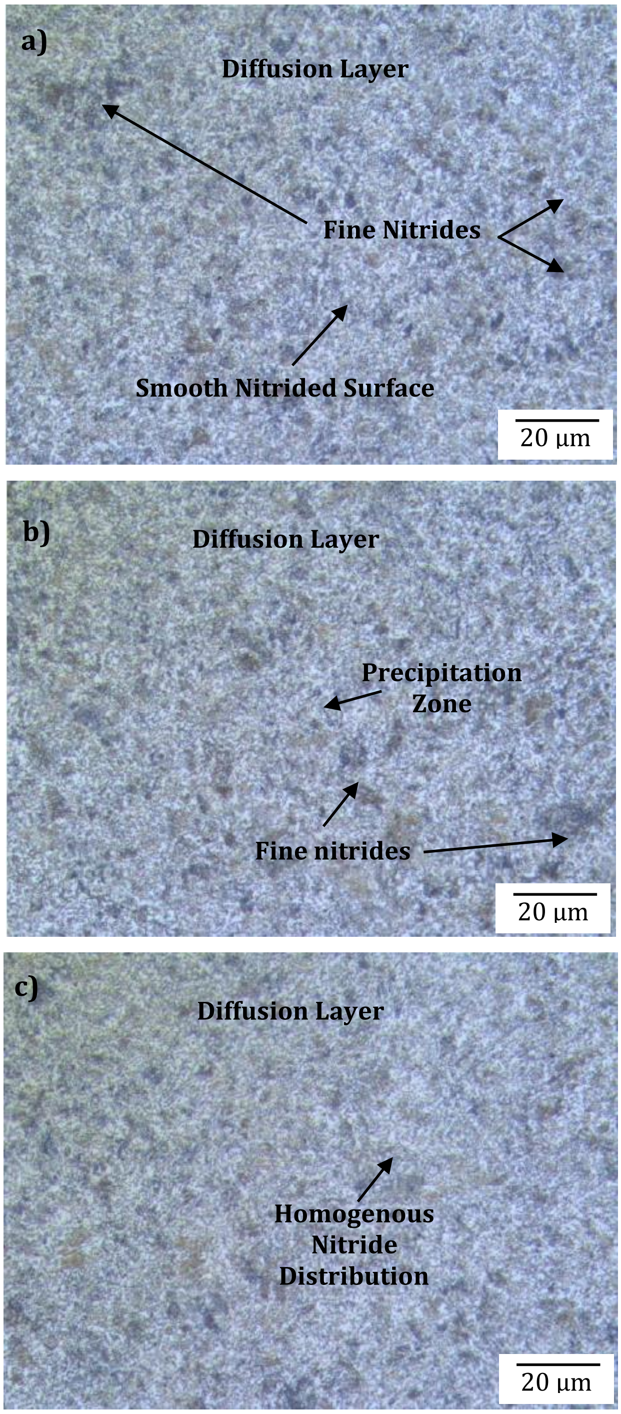

Figure 11. Micro Structure for Samples a) 30 minute, b) 60 minute, c) 90 minute

The figure 11 a) indicates the ferritic matrix with the dispersed dark region, the dark region represents carbide presence and light region represents ferrite. From figure 11 b) revealed a fine dispersion of dark phases with light ferritic matrix, which suggests pearlitic. In figure 11 c) displays a fine ferrite matrix with dark rounded and elongated particles. The pattern revealed an annealed EN31 steel, better hardness.

A key observation during the observation was the refinement of the grain structure during nitriding process. The 90-minute nitriding sample showing the most refined and densely packed nitride layer. This finer microstructure revealed a deeper nitrogen diffusion, which is potentially leading to a superior wear resistance and higher hardness. The progression from localized fine nitrides at 30 minutes to a homogenous nitride distribution at 90 minutes suggests a time-dependent diffusion process. The findings confirm that longer nitriding durations improve the nitrided layer's quality, resulting in a potentially more durable and harder surface.

The study results confirm that liquid nitriding significantly improves the mechanical properties of EN31 steel particularly in wear resistance, corrosion resistance, surface hardness and case depth. In all the samples 90 minutes sample confirmed the lowest wear rate and demonstrating that longer nitriding durations improves wear resistance and also making it ideal for high-friction applications like drilling tools and machine components. Corrosion resistance also enhanced with increased nitriding times due to the formation of a thicker nitride layer that protected the surface from oxidation and degradation. Subsequently increased nitriding time resulted in a deeper hardened layer and higher surface hardness by improving the material's load-bearing capacity and durability under mechanical loads. Microstructural analysis confirmed the uniformity of the nitride layers, with no defects such as cracks or porosity by ensuring reliability and an extended service life. These findings highlight liquid nitriding as an effective surface treatment for improving the performance of EN31 steel in industrial applications.

This study results are highly relevant to industries like manufacturing where drilling tools and wear resistant components require improved mechanical properties. The 90 minute nitriding process provides a cost-effective way to improve performance, durability and extend the service life of crucial components. For applications with cost limitations the 60-minute nitriding process offers a balanced solution by delivering good performance at a lower cost and making it suitable when maximum wear resistance is not essential.

4. Conclusion

For enhancing the tribological properties of EN31 steel, the liquid nitriding was used as a surface treatment for evaluating the effectiveness. The experimental analysis yielded the following key findings:

- The wear resistance of EN31 steel was improved significantly by increasing the nitriding duration. The 90-minute sample attributed to a deeper nitrided layer which exhibited the lowest wear rate of 1.97 mm^3/Nm. But the 30-minute sample showed a higher wear rate of 3.12 mm^3/Nm, which is likely due to the underdeveloped nitrided layer.
- Superior corrosion resistance was observed for the Nitrided EN31 steel, with no red rust observed after 24 hours in a salt spray test. The longer nitriding durations helps in forming a denser protective layer which enhances the corrosion resistance.
- The extended nitriding duration increased both the surface hardness and case depth of the EN31 steel. The 90-minute sample achieved the deepest case depth and the highest hardness of 38.4 HRC. This indicates an improved durability and wear resistance while compared to 30-minute nitriding durations.
- The longer nitriding duration led for a better surface hardness. The 90-minute sample attained the highest hardness of 31.7 HRC, which results from a deeper nitrogen diffusion and confirming improved wear resistance and durability for high-friction applications.
- The most refined and uniform nitrided layer, contributing to better hardness, wear resistance, and a defect-free surface quality, thereby enhancing overall tribological performance.

The future research will focus on optimizing the liquid nitriding process for industrial applications requiring high wear resistance. Further the optimization of nitriding process, includes the adjustments to treatment parameters and bath composition, this could yield even greater improvements in surface properties for specific real-time applications

Author contributions

Mathew Alphonse: Conceptualization, Methodology, Writing-Original draft preparation. **Padala Murali Krishna:** Data curation, Writing-Original draft preparation. **Murugu Nachippan Nachippan:** Validation, Writing-Reviewing and Editing**. Jeya lal Prince**: Investigation. Visualization**.**

Conflicts of interest

The authors declare no conflicts of interest.

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