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# MICROSTRUCTURAL AND MECHANICAL RESPONSE OF LOW-CARBON ST 37 STEEL TO A 1 HOUR NITRIDING TREATMENT AT 500 °C

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**Abstract.** Low carbon steel is widely used in engineering applications because of its favorable formability, machinability, and fabrication characteristics; however, its surface properties may limit performance under friction and wear conditions. This study investigated the microstructural and mechanical response of low carbon ST 37 steel subjected to a nitriding treatment at 500 °C for 1 h. Untreated material was used as the reference condition. The chemical composition was characterized using a spectrometer, while microstructural observation, Micro Vickers hardness testing, and Ogoshi wear testing were performed to evaluate changes after treatment. The material contained 0.2080% C and 98.81% Fe, confirming its classification as low carbon steel. The untreated microstructure was dominated by ferrite and pearlite. After the 1 hour treatment, ferrite and pearlite remained observable, accompanied by morphological changes at the specimen edge and fine features described as Widmanstätten like structures. The observed surface layer thickness was approximately 0.200 µm. The average hardness increased from 359.03 kg/mm² for the raw material to 414.50 kg/mm² after treatment. The average specific wear value increased from $2{,}9\times10^{-7}$ mm²/kg to $4{,}0\times10^{-7}$ mm²/kg. Therefore, under the experimental conditions employed, the treatment at 500 °C for 1 hour produced observable microstructural changes, improve the measured average hardness or wear resistance. These results indicate that the selected treatment condition requires further optimization to obtain a more favourable surface response in low-carbon ST 37 steel.

Keywords: low-carbon ST 37 steel; nitriding; mechanical properties

## 1 Introduction

Low carbon steel has a combination of formability, machinability, and good fabrication ability, which makes it one of the widely used materials in engineering.[1], [2]. This material is widely used in components that receive mechanical contact, friction, and repetitive loads. Therefore, the surface properties of components are essential to determine the life of the components and their performance [3], [4]. Under certain operating conditions, the steel surface can undergo plastic deformation [4], abrasion [5], and wear so that a surface modification method is required to obtain mechanical properties and surface resistance that is more in line with the needs of the application [3], [6].

One of the widely used surface modification methods is nitriding [7], [8]. Nitriding is a thermochemical treatment that aims to introduce nitrogen into the surface of the material so that surface areas with different mechanical properties are formed from the base material [9] [10]. The process generally takes place at temperatures below the austenitic transformation temperature of the steel so that nitrogen can diffuse into the ferritic matrix and further form nitride compounds [11], [12]. In carbon steel, the formation of compound layers and diffusion zones can result in increased surface hardness and changes in tribological characteristics [13], [14]. [15] explained that in the nitriding process, compound layers and diffusion zones can be formed, while the increase in hardness is influenced by temperature, holding time, and the number of nitrogen sources used.

Previous research has shown that steel's response to nitriding is strongly influenced by the type of material and process parameters. [16] For example, performing salt bath nitriding on low-carbon steel with a carbon content of about 0.2% and obtaining the formation of a white layer and a nitrogen diffusion zone. The study reported an increase in hardness from 198 HV on the

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substrate to 702 HV on the surface layer after nitriding. The results suggest that nitrogen diffusion and nitride layer formation can provide significant changes to the surface properties of low-carbon steel.

Similar results were also reported by [17] in low carbon steels that are treated with salt bath nitriding. Testing was performed using micro-Vickers hardness, pin-on-disc, and structural characterization to compare conditions before and after nitriding. The hardness of the material increased from 231 HV to 371 HV after nitriding treatment, accompanied by the formation of the FeN nitride phase. The findings show that nitriding can provide changes to the hardness and wear characteristics of the material, although the magnitude of the change is highly dependent on the material and the process conditions used.

In addition to temperature parameters [18], holding time [19] is one of the important factors in the nitriding process. Time change [20] treatment may affect the amount of nitrogen that diffuses [21], the formation of surface layers, and the distribution of mechanical properties from the surface to the core part of the material [22]. Research [23] ST 40 steel uses urea-based nitrogen sources with temperature variations of 450, 500, and 550 °C and a holding time of 1, 3, and 5 hours. The study showed that changes in temperature and resistance time provide different responses to the mechanical properties of materials. Research on other low-carbon steels has also shown that different nitriding conditions can result in significant changes in hardness and surface coating [15], [16].

Based on these previous studies, most nitriding studies on carbon steel report a tendency to increase surface hardness due to the formation of nitride layers. However, the response of the material is highly dependent on the chemical composition, initial condition of the material, the method of nitrogen intake, temperature, holding time, and cooling conditions. This is important in the ST 37 low-carbon steel used in this study. Based on the results of chemical composition analysis in previous research, the material used has a carbon content of 0.2080% and an iron content of 98.81%, so it is categorized as low carbon steel.

The purpose of this study was to analyze the microstructure response and mechanical properties of ST 37 low carbon steel after 1 hour of nitriding treatment at a temperature of 500 °C [24], in particular by evaluating changes in microstructure, hardness, and resistance to wear compared to the initial material. The results of the study are expected to provide experimental information on the response of low-carbon steel to surface treatment under the nitriding condition and become a comparative material for the results of the previously reported nitriding research on low-carbon steel.

# 2 Methods

## 2.1 Research Design

This study is an experimental study that aims to evaluate the microstructure response and mechanical properties of ST 37 low carbon steel after nitriding treatment. The main parameter studied in this article is treatment at a temperature of 500 °C with a holding time of 1 hour. As a comparison, raw materials are used that do not receive nitriding treatment.

Overall, the research stages include material preparation, chemical composition characterization, nitriding treatment, microstructure observation, Micro Vickers hardness testing, wear testing using the Ogozi method, and comparative analysis of test results.

## 2.2 Materials and Preparation

The material used is low carbon steel which in the study is referred to as ST 37 steel. The specimen has a thickness of 10 mm and a width of 35 mm. Before nitriding treatment, the surface of the specimen is prepared by cleaning and smoothing using sandpaper so that the surface is relatively flat and smooth. The preparations were carried out to simplify the treatment and subsequent testing process.

Characterization of the chemical composition is carried out before the nitriding treatment to ascertain the carbon content of the material and to know its constituent elements. The test results showed that the material had a carbon content of 0.2080%, so the material was categorized as low carbon steel based on the classification used in the study.



**Fig. 1.** ST 37 Low Carbon Steel



**Fig. 2.** Ammonia

### 2.3 Chemical Composition Testing

The chemical composition of the material is analyzed using a spectrometer. Testing is carried out before the nitriding treatment to obtain the carbon content and alloy elements present in the material. The results of the chemical composition analysis are used as a basis to ascertain the character of the material used in the study as well as as supporting information in interpreting changes in microstructure and mechanical properties after nitriding treatment.

### 2.4 Nitriding Treatment

According to this study, ST 37 low carbon steel was surface treated using ammonia as a nitrogen-containing medium. In accordance with the experimental protocol, the specimens were heated to a nominal temperature of 500 °C before being directly immersed in the ammonia medium. Further verification is necessary because the ammonia content, volume, immersion duration, delivery rate, furnace environment, and cooling conditions were not well reported. The initial experimental explanation suggested that nitrogen from the ammonia medium would interact with the heated steel surface and help with surface change. Nitrogen is introduced to the steel surface in conventional thermochemical nitriding, diffuses into the material, and either occupies interstitial sites or combines with the proper alloying components to produce phases containing nitrogen. These processes may change the microstructure and mechanical properties of the treated surface. The occurrence and extent of nitrogen diffusion are influenced by the nitrogen supply, treatment conditions, temperature, length of exposure, and chemical composition of steel.



**Fig. 3.** Furnace



**Fig. 4.** Cooling process

### 2.5 Microstructure Observation

Microstructure observation was carried out to compare the microstructure condition of the initial material with the material after nitriding treatment. The specimens to be observed are first prepared by grinding and grinding processes to level the surface. Furthermore, the surface is sanded until smooth and polished using autosole to produce a shinier surface so that the reflection of light during microscope observation is better.

In the research procedure, microstructure observation was carried out with a magnification of 100×. Microstructure documentation is carried out after hardness testing so that Micro Vickers penetration marks can be seen on micro photographs.

Microstructure analysis was performed comparatively between raw material and specimen after 1 hour nitriding treatment. The observed parameters included the morphology and distribution of the perlite ferrite structure as well as structural changes in the periphery of the specimen.

### 2.6 Hardness Test

The hardness of the ST 37 steel specimens was assessed both before and after treatment using a Vickers hardness tester.

The Vickers hardness test is based on the indentation of a diamond indenter with a square-based pyramidal

geometry and an included angle of 136°. During testing, the indenter is pressed against the specimen surface under a predetermined load for a specified dwell time. Once the load is released and the indenter is removed, the lengths of the two diagonals of the resulting square indentation are measured using an optical measuring system. The mean diagonal length is subsequently used to determine the Vickers Hardness Number (VHN).

In the present study, a test load of 30 kgf was applied with a dwell time of 10 s. Three measurement locations, labeled P1, P2, and P3, were selected on each specimen to evaluate variations in surface hardness. The hardness values obtained at each measurement location were recorded, and the arithmetic mean of the three measurements was calculated to determine the representative hardness value for each specimen.

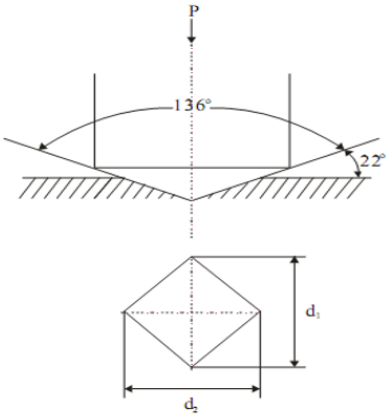



**Fig. 5.** Hardness Vickers Indentor

$$Hv = \frac{1.854P}{d^2} \quad \frac{kg}{mm^2}$$

Where:
P = pressing load N (kg)
d = diagonal penetrator (mm)
α = 136o angle for diamond pyramid (*Vickers*)

The average hardness is calculated using:

$$\overline{HV} = \frac{HV_1 + HV_2 + HV_3}{3}$$



**Fig. 6.** Vickers Hardness Tester

### 2.7 Wear Test

Wear testing of a wear testing machine (Ogoshi High Speed Universal Wear Testing Machine type OAT-U) to measure the wear rate of a material through the shear method. The basic principle of the Ogoshi test is the frictional contact between a hard revolving disc and a specimen with a flat surface. The rotor disc is stressed on the test piece with a specific load. The disc is then rotated at a certain linear speed within a predetermined mileage. As a result of this friction, a depression or wear mark will form on the surface of the test specimen that is shaped like a circular arc. The calculation of wear is carried out using the following formula:

$$Ws = \frac{B \times B_o^3}{8 \times r \times P_o \times L_o} \quad \frac{mm^2}{kg}$$

Where:
Ws = Specific wear ($mm^2/kg$)
B = Width of the disk (mm)
$B_o$ = Width of wear on specimens (mm)
r = Radius of the disk (mm)
$L_o$ = mileage in the wear process (m)
$P_o$ = The compressive force on the wear process takes place (kg)

The principal specifications of the Ogoshi High-Speed Universal Wear Testing Machine (Type OAT-U) are presented in table 2.



**Fig. 7.** Universal Wear Testing Machine

**Table 1.** Ogoshi High-Speed Universal Wear Testing Machine (Type OAT-U) specifications

| **Parameter** | **Specification** |
|---|---|
| Contact presure | 30 - 400 kg/cm2 (5 steps) |
| Abrasion speed | 0,05 – 4, 0 m/sec (22 steps ) |
| Abrasion distance | 60 – 600 m (5 steps) |
| Maximum load | 180.19 kg |
| Gross weight | 200 kg |
| Power required | 1 HP |
| Dimensions | 840 mm (width)×1090 mm (height)×580 mm (depth) |

# 3 Results and Discussion

## 3.1 Optical Emission Spectroscopy

Chemical composition testing is performed using a spectrometer, which automatically displays the results of chemical composition measurements from samples for each 1 hour hold time test.

**Table 2.** Raw Material Chemical Composition Test Results

| **Substance** | **Percent (%)** |
|---|---|
| Carbon (C) | 0.2080 |
| Silicon (Si) | 0.0103 |
| Sulfur (S) | 0.0386 |
| Phosphorus (P) | 0.0615 |
| Manganese (Mn) | 0.4728 |
| Nickel (Ni) | 0.0563 |
| Chrome (Kr) | 0.0529 |
| Molybden (Mo) | 0.0038 |
| copper (Cu) | 0.2196 |
| Tungsten (W) | 0.0054 |
| Titanium (Ti) | 0.0019 |
| Tin (Sn) | 0.0552 |
| Aluminum (Al) | 0.0032 |
| Lead (Pb) | 0.0000 |
| Calcium (Ca) | 0.0000 |
| Zinc (Zn) | 0.0001 |
| Ferro (Fe) | 98.81 |

The results of the composition show that the carbon content of the material is relatively low, which is 0.2080%. This composition became an early characteristic used to compare changes in microstructure and mechanical properties after nitriding treatment.

## 3.2 Microstructure Test

Microstructure observations were carried out on raw materials and specimens after nitriding treatment. The results of observations in the study were documented with a magnification of 200×, while the microstructure analysis section also included images with a magnification of 100× and a scale of 10 μm.

In raw material conditions, the microstructure observed consists of ferrite and perlite with relatively large granular morphology. The structure is the initial condition of the material before getting the nitriding treatment.

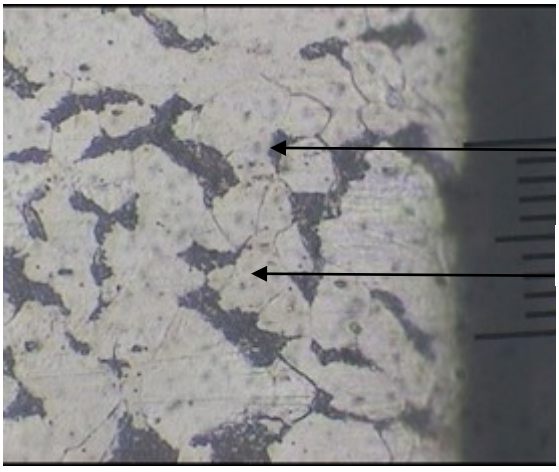



**Fig. 8.** *Raw material*

After 1 hour of nitriding treatment at 500 °C, observation of the microstructure showed changes in the specimen's edge area. The observed structure still shows the presence of ferrite and perlite, but the grain morphology of the affected part appears to be smaller than in the initial condition.

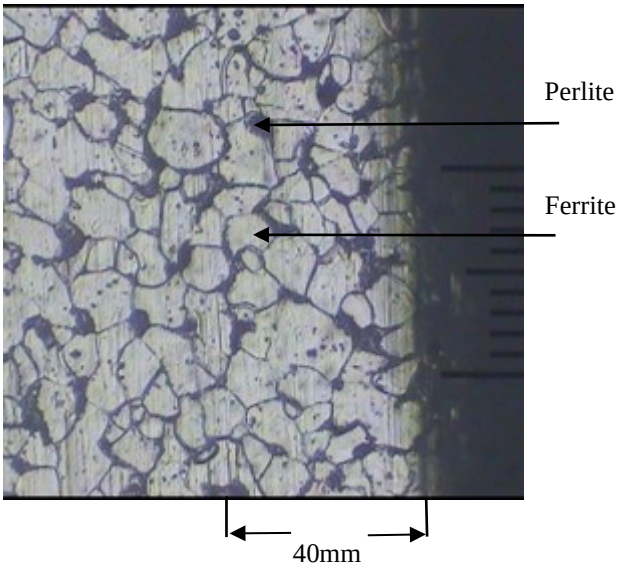



**Fig. 9.** Holding time 1 hour

The change in the microstructure shows a morphological difference between the initial area of the material and the area that underwent nitriding treatment.

The thickness of the layer from the 1 hour nitriding process in can be the thickness of the carbon layer as:

$$40 \text{ mm} = x \text{ µm}$$

$$\frac{20}{40} = \frac{100}{x}$$

$$x = \frac{40\ x\ 100}{20}$$

$$x = \frac{400}{20}$$

$$x = 200 \text{ mm}$$

$$= 0.200 \text{ µm}$$

### 3.3 Hardness Test

The results of the hardness test using Micro Vickers showed a difference between the raw material and the specimen after nitriding treatment. The test was carried out at three points, namely P1, P2, and P3. Complete data for the conditions that were the focus of the study are shown in table 2.

**Table 3.** Hardness Test Results

| Variabel | P1 (HV) | P2 (HV) | P3 (HV) | Average (kg/mm²) |
|---|---|---|---|---|
| Raw material | 370.80 | 397.28 | 309 | 359.03 |
| Holding time 1 hour | 429.17 | 385.18 | 429.17 | 414.50 |

For the conditions that were the focus of the article, namely nitriding for 1 hour, the hardness values at the three points were 370.80 HV, 397.28 HV, and 309.00 HV, respectively, with an average value of 359.03 HV. In contrast, have an average value of 414.50 HV.

### 3.4 Wear Test

Wear testing is carried out with the Ogoshi High Speed Wear Testing Machine (Type OA-U) machine. The results of the measurements at three points for each condition are used to obtain the average value of specific wear. Raw material and specimen data with a treatment time of 1 hour are shown in table 3.

**Table 4.** Wear Test Results

| Variabel | K1 (mm²/kg) | K2 (mm²/kg) | K3 (mm²/kg) | Average (mm²/kg) |
|---|---|---|---|---|
| Raw material | $3.3\times10^{-7}$ | $2.0\times10^{-7}$ | $3.3\times10^{-7}$ | $2.9\times10^{-7}$ |
| Holding time 1 hour | $3.5\times10^{-7}$ | $3.7\times10^{-7}$ | $4.7\times10^{-7}$ | $4{,}0\times10^{-7}$ |

The data in table 3 show that the raw material has an average specific wear value of $2.9 \times 10^{-7}$ mm²/kg, while the specimen after nitriding for 1 hour has an average value of $4.0 \times 10^{-7}$ mm²/kg.

## 4 Conclusion

The results of the ST 37 low carbon steel research after treatment at a temperature of 500 °C with a holding time of 1 hour, it was concluded that the research material has a carbon content of 0.2080% and an Fe content of 98.81%, so it is categorized as low carbon steel. Observation of the microstructure of raw materials, dominated by ferrite and perlite with relatively large granular morphology. After the nitriding treatment for 1 hour, observation of the microstructure showed changes in the edge area of the specimen. The observed structure still shows the presence of ferrite and perlite, but the grain morphology of the treated part appears to be smaller than the initial condition, in the image also found by Widmanstätten, which is described as fine lines on the material.

Treatment of nitriding at 500 °C for 1 hour did not increase the measured average hardness. The average hardness value changed from 359.03 HV on raw materials to 414.50 HV after nitriding treatment.

The average specific wear value increased from $2.9 \times 10^{-7}$ mm²/kg on raw materials to $4.0 \times 10^{-7}$ after heat treatment.

Overall, treatment at 500 °C for 1 hour resulted in microstructural changes in ST 37 steel, providing an increase in hardness and wear resistance based on the method and measurement points used. Further

optimization of process parameters is required to obtain better surface response.

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