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Effect of Surface Hardening by Gas Nitriding on the Fatigue Resistance of Low Alloy Steel EN 1.8509 (41CrAlMo7-10)

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Abstract: In this research, the fatigue resistance of low alloy steel was improved by using surface hardening by gas nitridation at different temperatures, 475 to 610°C, and in a medium containing 70% nitrogen and 30% hydrogen. The results obtained showed a significant improvement in the fatigue resistance of the samples used after the nitridation process, where the highest fatigue resistance was obtained (580 MPa) at the temperature range 475 to 510°C. At the same time, there was a decrease in the fatigue resistance when using high temperatures in the gas nitridation process, which decreased to 400 MPa at 610 °C. In comparison, the value of the fatigue resistance before nitridation was 325 MPa. Through the results of the micro hardness and tensile test, it was found that there was an increase in the hardness tensile strength values on the surface and in general for all groups of samples treated by nitriding, It was also observed that the hardness gradually decreased smoothly as we moved away from the surface towards the depth, as the metal retains good hardness up to a depth of 300 microns. It helps to avoid the sudden and non-gradual transition from a rigid surface to a soft center. Thus, a structure of sufficient thickness is formed from the nitriding layer, coherent with the core. The results of the X-ray diffraction examination also showed the formation of nitrides of various alloying elements, Fe_3N, Fe_4N, Cr_2N, Fe_2Al_5, Al_2N, which have a significant role in increasing the hardness and improving the fatigue resistance.

Keywords: Fatigue Resistance, Gas Nitriding, Hardness, Low Alloy Steel, Surface Hardening, Tensile Strength.

1 INTRODUCTION

Low-alloy steel is characterized by important physical, electrical, thermal, and mechanical properties. EN 1.8509 low alloy steel is extensively used for many industrial and automotive applications, such as structural parts with high surface hardness — piston rods, extruders, cylinders, shafts, straightening tools, measuring tools, etc. This type of steel is very suitable for surface heat treatment by nitriding, as this process leads to improvement in the mechanical properties and resistance to fatigue and wear of steel [1]. Previous studies have shown that gas nitriding significantly increases the fatigue strength of 18Ni-300 maraging steel by creating compressive residual stresses and shifting crack initiation deeper into the material, ultimately improving fatigue strength by 30% to 100% compared to heat-treated samples [2]. AISI 4340 steel suffers from low fatigue strength due to post-weld brittleness. Plasma nitriding has been proposed as an alternative post-weld treatment, which improves surface hardness and fatigue performance by forming double nitride layers, making it superior to conventional nitriding [3].

According to [4], gas nitriding significantly increases the fatigue resistance of low alloy steel, resulting in a 16% to 32% improvement in fatigue life, depending on the carbon potential and treatment time, as demonstrated by metallurgical evaluations and tests. However, the effect of gas nitriding surface hardening on the fatigue strength of EN low alloy steel is not explicitly discussed in [5], which instead focuses on the impact of plasma nitriding on the hardness and toughness of 25Cr2Ni4W low alloy steel. Other studies have shown that plasma nitriding significantly improves the fatigue resistance of 50CrV4 low alloy steel, increasing the fatigue limit from 450 MPa to 705 MPa due to surface hardening effects [6]. Lakshmi D. Tadepalli et al. [7] studied the effects of nitriding on the mechanical properties of AISI 409 ferritic stainless steel. Their work demonstrated that surface treatment using nitriding improves these mechanical properties and broadens the areas of application of such steels.

F. Borgioli, A. Fossati, E. Galvanetto, and T. Bacci [8] investigated glow-discharge nitriding of AISI 316L austenitic stainless steel and found that the nitridation process can be carried out at low temperatures to avoid a decrease in corrosion resistance due to chromium nitride formation. They reported high hardness values (from ~1450 to ~1550 HK0.01) in the modified layer, with a steep decrease to matrix values. In another study, low-temperature nitriding of nickel-free, high-manganese austenitic stainless steel was investigated [9]. Nitriding at 360–380°C for up to 5 hours improved surface hardness and corrosion resistance through the formation of expanded austenite (γN), while preventing harmful nitride precipitation.

2 EXPERIMENTAL PROCEDURE

2.1. Metal Used

In this research, EN 1.8509 low-alloy steel was used. This type of alloy steel is characterized by its good ability to undergo surface heat treatment with nitriding. Table 1 shows the chemical composition of the metal used in this research, as a spectrometer device was used to examine it in the laboratories of North Gas Company.

Table 1. The chemical composition of alloy steel EN 1.8509 used in this work

Element	C	Si	Mn	Cr	Mo	V	Al	Fe
Wt%	0.41	0.40	0.65	1.65	0.33	0.50	1.00	Balance

2.2. Heat Treatment before Nitriding Process

The samples used in this research, and to be nitrided (before nitridation), were treated with some heat treatments to prepare them for use. These treatments are in the following sequence:

1. Hardening: Heating to a temperature of 870-930°C and then quenching in oil after a thorough heating hold for 15 to 30 minutes.
2. Slow heating to tempering temperature of 450-600°C immediately after hardening and hold at this temperature at least 2 hr according to the thickness of the specimens.

The gas nitriding process must be preceded by the necessary heat treatments, such as hardening and tempering, and its purpose is to obtain a solid core. These heat treatments of the steel samples used in this research lead to a homogeneous structure of bainite, and the presence of chromium in a high percentage obstructs the process of carbon diffusion when using a rapid cooling medium and the formation of carbides, which results in an increase in the hardness of the samples used.

2.3. Gas Nitriding Heat Treatment

The nitriding process is widely used to improve the properties of machine and tool elements. The heat treatment of the steel, which involves hardening, quenching, and tempering, is performed to achieve high durability, ensuring no separation between the nitride layer and the heart of the specimens. The gas nitriding process was carried out in an insulated nitriding furnace, thermal insulated with a ventilation fan and a sampling chamber tightly closed connected to the gas inlet tube connected nitrogen and hydrogen gas cylinders and valves gas flow control, on the other side of the output gas tube, where the nitridation process was carried out in a medium consisting a mixture of nitrogen with a ratio of 70% and hydrogen 30%, at temperatures between 475-610°C Temperature was controlled using K-type thermocouple. The specimens were held at these temperatures for 10 hours, after cleaning them of oils, grease, and other impurities with ethyl alcohol.

The sample groups were placed suspended inside a temperature-resistant crucible. The process takes place in a medium of ammonia gas. The furnace is turned on to the temperatures shown in Table 2 and settled at this temperature for 10 hours. Atomic nitrogen is produced that penetrates the metal surface from the decomposition of ammonia gas, as in the following chemical equation:

$$2NH_3 \rightarrow N_{atomic} + 3H_2 \quad (1)$$

where the atomic hydrogen gas is expelled, along with part of the ammonia gas, through the intake tube in the gate because its continuous presence with the temperature causes the explosion [10].

Table 2. The sample groups and Nitriding temperature

S. No.	Specimens Group	Nitriding Temperature (°C)
1.	A	Without Nitriding
2.	B	475
3.	C	510
4.	D	540
5.	E	580
6.	F	610

2.4. Tensile Test

A tensile test was conducted for the steel samples used in this research, as one sample was manufactured for each heat treatment according to the standard specifications of the tensile test sample. The tensile test was conducted on a tensile test device type (Universal Testing Instron) in the laboratories of the North Gas Company [11].

2.5. Fatigue Test

The metal was received in the form of bars and cut into final models with diameters of 10 mm and lengths of 80 mm. These models were manufactured on a lathe machine according to the standard specifications of the fatigue test for rotary bending, and after obtaining samples with high accuracy in dimensions, especially in the curvature area. Before the fatigue test, a smoothing and polishing process was carried out for the sample surface in two stages. The first stage included the use of silicon carbide grinding paper with degrees of fineness 300, 500, 800, 1000, and 1200. In the second stage, the polishing process of the samples was carried out using diamond paste with its cloth, Fatigue tests were carried out at constant amplitude loads and stress ratios (R=-1), where the test piece in this device is subjected to a load that is shed from the correct side holding of the sample perpendicular to the sample axis. Thus, the surface of the piece is subjected to successive tensile and compression stress when rotating, and the device is equipped with a counter to record the number of stress cycles and a rotational speed of 600 rpm.

The process of shedding stress is carried out using an iron block that slides over a stepped iron beam of length 30 cm. By calculating the bending moment, the stress value is represented by the movement of the iron mass on the sample. The maximum load of the device with the use of additional blocks is 900 MPa. This test was carried out on different sample groups to obtain the S-N curve for each group, and the relationships between stress and number of cycles were drawn [12].

2.6. Fatigue Test

The examination was carried out by X-ray diffraction of the samples that were heat-treated by nitriding using an X-ray diffractometer device of the type located in the General Company for Geological Survey and Mining. The results showed the presence of high iron nitrogen phases on the surface, with a percentage of hard chromium and aluminium nitrides.

3 Results and Discussion

3.1. Ultimate Tensile Strength and Hardness

The results of the tensile test showed an increase in the ultimate tensile strength (UTS) of the steel after nitriding at 475°C, from 1130 MPa (as received) to 1635 MPa—the highest UTS value after nitriding was achieved at 510°C. However, a noticeable decrease in UTS was observed at higher nitriding temperatures, with the UTS dropping to 1175 MPa at 610°C, as shown in Table 3. During the nitridation process, iron-nitrogen phases form. The solubility of nitrogen in α-Fe (ferrite) at 590°C is approximately 0.42% N, while in γ-Fe (austenite) at the same temperature, the solubility increases to 2.35%. The γ-phase composition is primarily Fe_4N. As the temperature decreases, the nitrogen concentration in ferrite decreases, eventually reaching 0.01–0.15%, and forms a surface layer rich in nitrogen phases, primarily the ε-phase (Fe_2N).

In the inner layers, nitrogen-diffused ferrite is present, where the nitrogen concentration gradually decreases with depth from the surface. The nitridation layer typically extends to a depth slightly less than 1 mm. Although the layer depth increases at temperatures above 600°C, the formation of the brittle ε-phase also intensifies, which may lead to cracking. As a result, both tensile strength and yield strength tend to decrease at elevated nitriding temperatures. Therefore, it is preferable to increase the nitridation depth by extending the treatment time rather than raising the temperature, to maintain good mechanical properties.

Additionally, at high temperatures, there is a reduction in the precipitation of carbide phases that contribute to strength, along with poor cohesion between the carbides and grain coarsening. Prolonged thermal exposure during the nitriding process without nitrogen gas feeding (i.e., only tempering) can lead to over-hardening and reduced ductility. Hence, the mechanical properties of nitrided samples are influenced not only by the hardness of the nitrided layer but also by improvements in the mechanical properties of the core [13].

Table 3. Mechanical properties of alloy steels used before and after the nitridation process

S. No.	Specimens Group	Tensile Strength		Ductility Ɛ (%)
		бu (MPa)	бy (MPa)	
1.	A	1130	940	8
2.	B	1680	1450	3
3.	C	1635	1345	4
4.	D	1550	1280	5
5.	E	1300	1115	6
6.	F	1175	1055	7

In this paper, the effect of nitriding process temperature on the microhardness of the nitride layer was also studied. The results showed a significant and remarkable increase in microhardness within the temperature range of 475–510°C.

The surface layer exhibited high hardness values of approximately HV 1310 at 510°C and HV 1120 at 475°C, as shown in Fig. 1. It was also observed that when the temperature exceeded 510°C, the microhardness of the nitrided layer began to decrease gradually, with a slow decline of approximately 3 MPa/°C. However, at nitriding temperatures above 540°C, a sharp decrease in microhardness was noted, reaching up to 50 MPa/°C per degree rise in temperature. These results indicate that high microhardness values were consistently found on the surface and to a depth of more than 100 microns at all nitriding temperatures used. This improvement is attributed to the presence of alloying elements such as aluminium, chromium, molybdenum, and vanadium in the steel, which play a key role in the formation of complex and stable nitrides within the hardened surface layer. The aluminium content enhances nitrogen diffusion and contributes to the stability of the nitrides formed.

Additionally, the results demonstrate a gradual and smooth decrease in microhardness from the surface toward the core of the samples. This trend corresponds to a uniform reduction in nitrogen content with increasing depth, observed consistently across all treatment temperatures. The gradual decline in microhardness is associated with a reduced degree of nitride phase precipitation and a lower degree of cohesion among the formed nitrides. These observations are consistent with findings reported in previous studies.

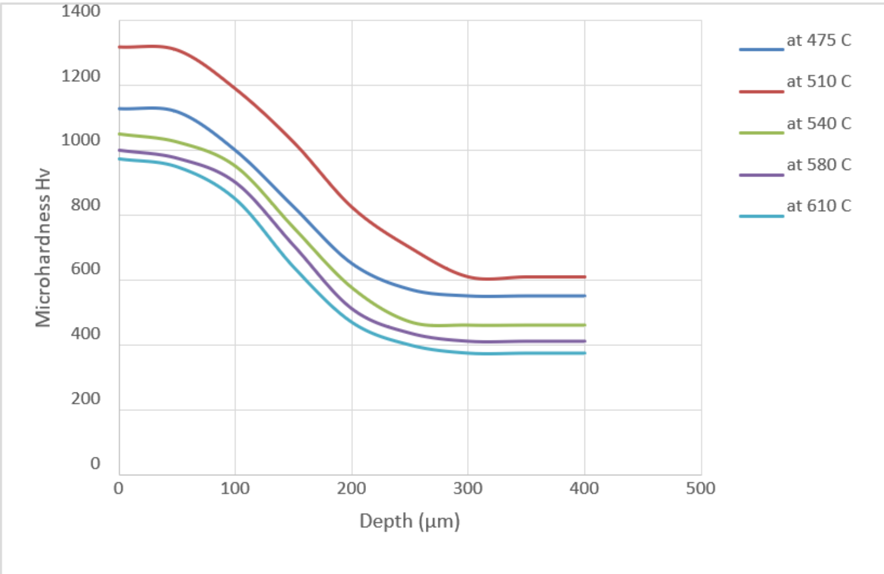

Fig. 1. Relation between microhardness and depth of the hardened Layer

3.2. Fatigue Test Results

The results obtained from the fatigue test, conducted under fixed-amplitude loads, indicate an increase in the fatigue limit for all sample groups after undergoing the nitridation process at various temperatures. The most notable improvement was observed in the samples nitrided at 510°C, where the fatigue limit reached 580 MPa. Conversely, a decline in fatigue limit was observed with increasing nitridation temperatures, with the lowest value recorded at 400 MPa for samples nitrided at 610°C. These findings confirm a significant enhancement in fatigue resistance and an increase in the number of cycles required to cause failure, compared to the untreated samples.

This improvement is attributed to the presence of a hardened surface layer rich in stable and hard nitrides, which resists the initiation and propagation of fatigue cracks due to its high hardness and adequate depth exceeding 130 µm. During the nitridation process, nitrogen diffuses into the iron lattice in atomic form at the treatment temperature, forming atomic bonds with iron and the various alloying elements in the steel to produce iron nitrides.

However, at higher nitriding temperatures, atomic nitrogen tends to recombine into molecular nitrogen, which does not dissolve or diffuse effectively into the steel. These molecular nitrogen species cannot dissociate at elevated temperatures, limiting their contribution to nitride formation. In contrast, at lower temperatures, the presence of alloying elements facilitates the formation of a solid solution of stable nitrides on the surface.

As illustrated in Fig. 2 (a–f), these effects support the observed trends in fatigue behavior [14]. Additionally, previous research [15] on gaseous nitriding of low-carbon bainitic steel demonstrated a 13% increase in fatigue strength compared to untreated samples. However, it also reported that brittleness and reduced ductility can limit the overall improvement in fatigue resistance.

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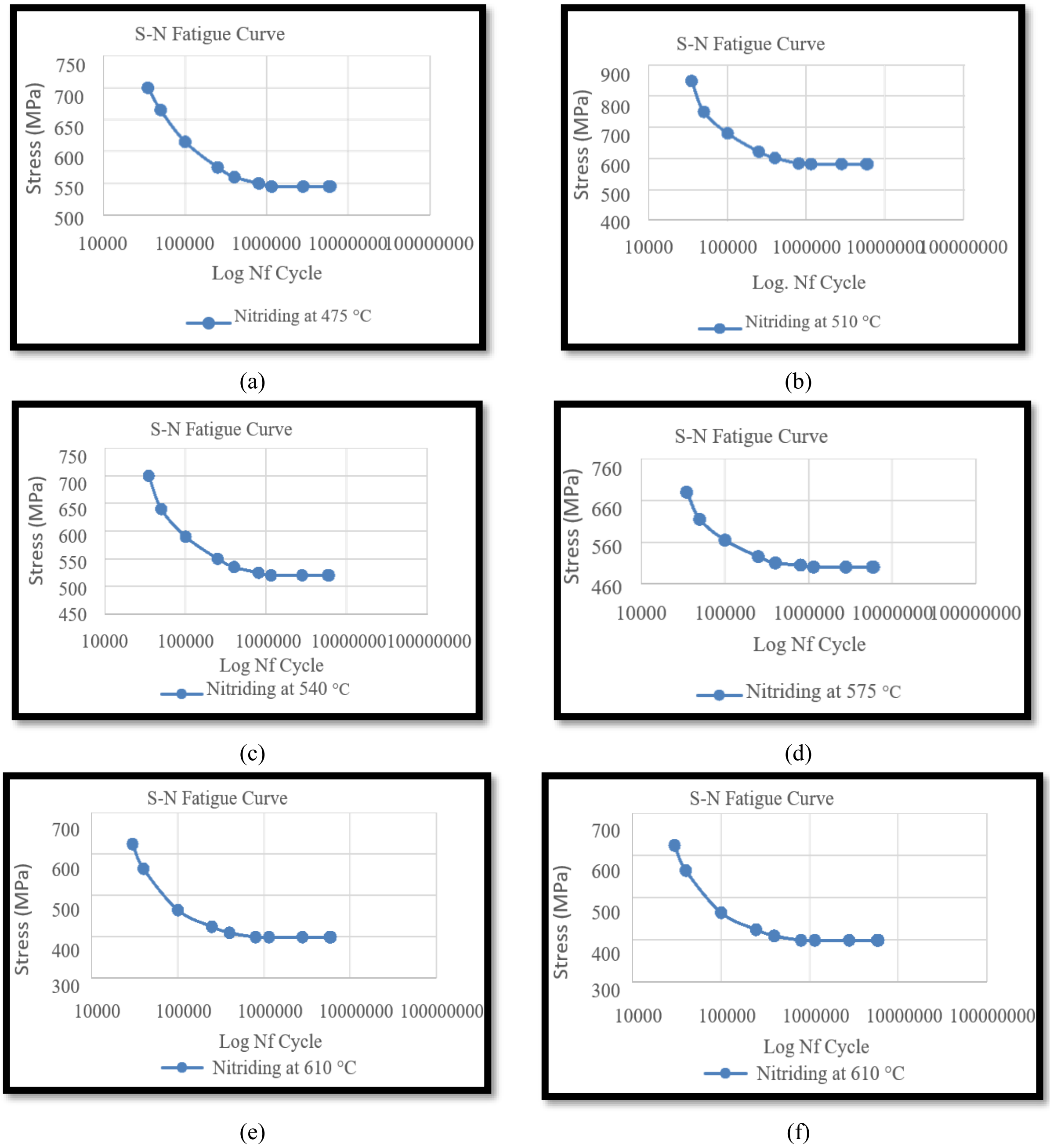

Fig. 2. Stress vs Cycle for Nitriding at different Temperatures

3.3. X-Ray Diffraction Results

The results obtained showed that the gas nitriding process led to an improvement in the fatigue resistance of the samples at all the different temperatures used in this work. This improvement is attributed to the increased hardness of the surface layer—up to 130 µm—which consequently enhances the fatigue life of the nitrided samples. The hardened surface layer acts as a barrier that hinders the initiation and propagation of fatigue cracks. The increase in surface hardness is primarily due to the formation of nitrides of various alloying elements. The most critical nitrides identified include Fe_3N, Fe_4N, Cr_2N, Fe_2Al_5, and Al_2N, along with other nitrides in varying proportions. These phases were confirmed by the X-ray diffraction (XRD) results, as shown in Fig. 5.

Aluminium, and to a certain extent chromium, vanadium, and molybdenum, exhibit a strong tendency to bond with nitrogen. Aluminium in particular forms extremely hard nitrides near the surface, which restricts the diffusion of nitrogen to deeper layers. This results in the formation of a tough but relatively thin nitrided layer, with a thickness not exceeding 1.0 mm. Chromium also contributes to the increase in surface hardness through the formation of chromium nitrides.

Since chromium nitrides tend to form at greater depths compared to aluminium nitrides, they help ensure a gradual transition from the hardened surface to the softer core. This gradual transition reduces the risk of surface delamination or peeling. Molybdenum, in addition to enhancing surface hardness, also plays a role in improving the strength of the material's core.

4 CONCLUSIONS

By analyzing the mechanical and tribological properties of the low-alloy steel used in this work after nitriding at different temperatures, a clear improvement in these properties was observed. Among all the tested conditions, nitriding at 510°C produced the most favorable results. This temperature led to the formation of a nitrided layer of sufficient thickness that was well bonded with the core, offering high surface hardness while maintaining the required toughness and good ductility of the core material. High microhardness values were recorded on the surface and to a depth exceeding 100 microns across all nitriding temperatures. The best surface microhardness values were HV 1310 at 510 °C and HV 1130 at 475 °C, confirming the effectiveness of the process in enhancing surface hardness. The nitriding process also improved the fatigue resistance of all sample groups. The highest fatigue resistance value was obtained at 510 °C, while a noticeable decline was observed at higher temperatures, such as 610°C. This behavior underscores the role of the hardened surface layer in preventing crack initiation and propagation, thereby increasing the fatigue life of the steel used in this study. X-ray diffraction analysis confirmed the formation of nitrides of various alloying elements because of the nitriding process. The most significant nitrides identified were Fe_3N, Fe_4N, Cr_2N, Al_2N, and $FeAl_3$, along with other nitrides in varying proportions. These phases are primarily responsible for the improved hardness and fatigue resistance of the nitrided samples.

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ETHICS STATEMENT
This study did not involve human or animal subjects and, therefore, did not require ethical approval.

STATEMENT OF CONFLICT OF INTERESTS
The authors declare no conflicts of interest related to this study.

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