



## INVESTIGATION OF THE STRUCTURE AND WEAR RESISTANCE OF NITROOXIDIZED LAYERS

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**Abstract:** This study investigates the influence of plasma nitrocarburizing temperature on the microstructure, phase composition, microhardness distribution, and corrosion resistance of treated steel. Thermochemical treatment was carried out at temperatures ranging from 520 °C to 620 °C in order to analyze the formation mechanisms of the compound layer and diffusion zone. The results demonstrated that increasing the processing temperature significantly affected the development of  $\gamma'$  ( $\text{Fe}_4\text{N}$ ) and  $\varepsilon$  ( $\text{Fe}_{2-3}\text{N}$ ) phases, as well as the thickness and morphology of the compound layer. At 620 °C, an austenitic region was observed beneath the compound layer, indicating intensified diffusion processes and structural transformations. The maximum  $\varepsilon$  phase fraction was detected in samples treated at intermediate temperature, while the highest microhardness values were recorded within the central region of the compound layer formed at elevated temperature. A gradual decrease in hardness toward the substrate confirmed the presence of a structural gradient across the diffusion zone. Corrosion testing revealed that corrosion resistance is predominantly governed by the type, distribution, and morphology of carbonitride phases rather than by the total thickness of the compound layer. The findings emphasize that optimization of phase composition and surface characteristics plays a crucial role in enhancing functional performance.



**Keywords:** Plasma nitrocarburizing; Compound layer;  $\gamma'$  phase;  $\varepsilon$  phase; Microhardness; Corrosion resistance; Carbonitride phases.

### Introduction

Surface engineering methods play a crucial role in improving the operational performance of machine components operating under friction and cyclic loading conditions. Among thermochemical treatments, nitrooxidizing has attracted considerable attention due to its ability to enhance surface hardness, wear resistance, and corrosion resistance without significantly affecting the core properties of the material. This process involves the diffusion of nitrogen into the surface layer followed by controlled oxidation, resulting in the formation of a composite surface structure.

A typical nitrooxidized layer consists of a compound zone formed at the outer surface and a diffusion zone located beneath it. The compound layer generally contains iron nitrides and oxynitrides, which contribute to high surface hardness and improved resistance to adhesive and abrasive wear. The diffusion layer provides gradual hardness transition toward the substrate, reducing internal stresses and improving load-bearing capacity. The thickness and phase composition of these layers depend strongly on treatment temperature, duration, and processing atmosphere.

Compared with conventional nitriding, nitrooxidizing offers additional advantages due to the formation of a thin oxide film on the surface. This oxide layer acts as a protective barrier, reducing friction coefficient and enhancing corrosion resistance. As a result, components treated by nitrooxidizing demonstrate improved tribological performance in aggressive and high-load environments.

Although numerous studies have investigated nitrided and nitrocarburized steels, the relationship between the structural characteristics of nitrooxidized layers and their wear behavior requires further clarification. In particular, the influence of



layer thickness, phase composition, and hardness distribution on wear intensity remains an important research issue.

The objective of this study is to investigate the microstructure, hardness profile, and wear resistance of nitrooxidized surface layers. Special attention is given to the correlation between structural parameters of the formed layers and their tribological performance. The obtained results provide a scientific basis for optimizing nitrooxidizing parameters to improve the durability of treated components.

### **Materials and Methods**

Disk-shaped specimens manufactured from AISI 4140 low-alloy steel were used in this study. The chemical composition of the material (wt.%) was approximately 0.39 C, 0.93 Cr, 0.67 Mn, 0.20 Si, 0.15 Mo, with the balance being Fe. The samples were prepared with a diameter of 20 mm and a thickness of 8 mm. Prior to thermochemical treatment, the surfaces were mechanically ground using successive grades of SiC abrasive papers and subsequently polished with alumina suspension to obtain a mirror-like finish.

Nitrooxidizing treatment was carried out in a pulsed plasma processing unit with a nominal power of 5 kW. The process atmosphere consisted primarily of nitrogen-based gases with controlled additions of hydrogen and carbon-containing components to promote the formation of a compound layer. The treatments were conducted at selected temperatures in the range of 520–570 °C under reduced pressure conditions. The discharge voltage and current were maintained within controlled limits to ensure stable plasma formation. After completion of the thermochemical treatment, the specimens were cooled inside the chamber under controlled conditions to minimize thermal stresses.

For microstructural analysis, the treated samples were sectioned perpendicular to the surface. The cross-sections were mounted and prepared following standard metallographic procedures. To preserve the integrity of the compound layer during preparation, edge protection techniques were applied. The microstructure was



examined using optical microscopy and scanning electron microscopy (SEM). Phase identification in the surface layers was performed by X-ray diffraction (XRD) analysis using  $\text{CuK}\alpha$  radiation. The evolution of phase composition across the modified layer was evaluated by gradual material removal from the surface followed by repeated XRD measurements.

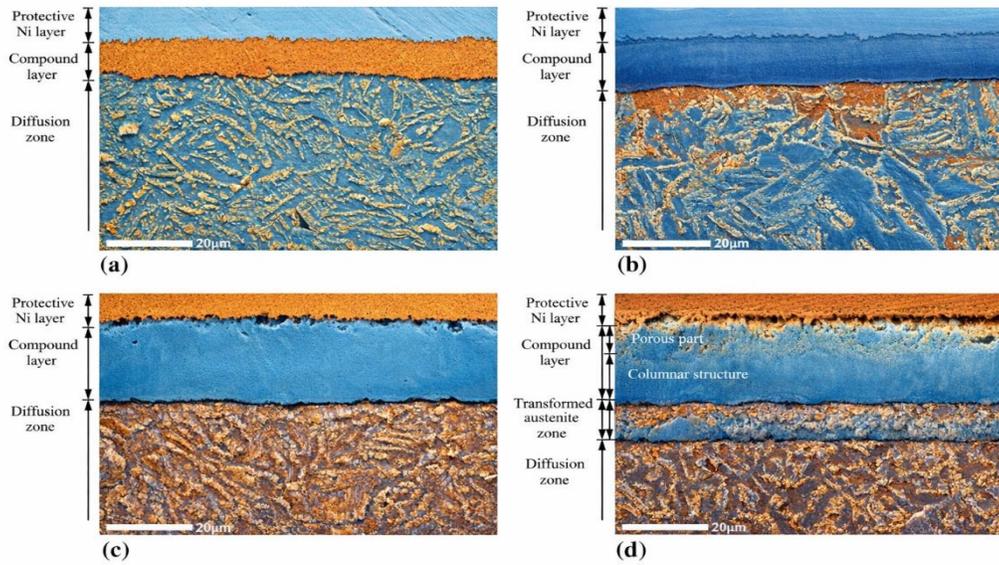
Surface hardness and hardness profiles were determined using a Vickers microhardness tester under different applied loads. Multiple measurements were taken at various depths from the surface to obtain statistically reliable hardness distributions.

Wear resistance was evaluated using a controlled tribological testing setup under dry sliding conditions. The wear rate was calculated based on mass loss measurements and sliding distance. Additionally, the friction coefficient was continuously recorded during testing to assess tribological behavior.

Electrochemical properties of the nitrooxidized layers were investigated using potentiodynamic polarization measurements in a 3.5 wt.% NaCl solution. A conventional three-electrode electrochemical cell was employed, with the treated specimen serving as the working electrode, a saturated reference electrode, and a platinum counter electrode. The polarization scans were conducted at a constant sweep rate to determine corrosion potential and corrosion current density.

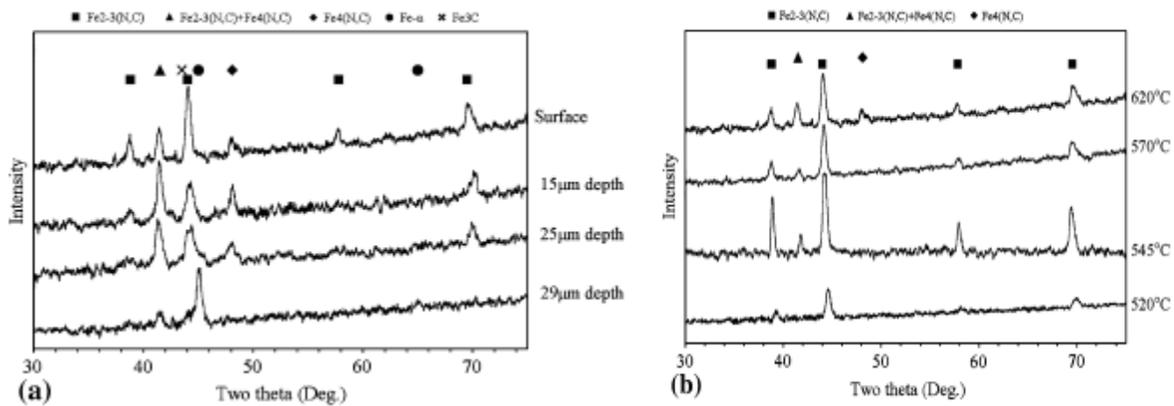
## **Results and Discussion**

Cross-sectional SEM observations revealed that the morphology and thickness of the nitrooxidized layer strongly depend on the treatment temperature. As the processing temperature increased within the selected range, a noticeable growth of the compound layer was observed. This behavior can be explained by the enhanced diffusion kinetics of nitrogen and carbon atoms at elevated temperatures, leading to more intensive interaction between the plasma species and the steel surface. Consequently, higher temperatures promoted the formation of a thicker and more developed surface layer.



**Fig. 1.** Cross-sectional SEM micrographs of AISI 4140 steel plasma nitrooxidized at (a) 520 °C, (b) 545 °C, (c) 570 °C, and (d) 620 °C.

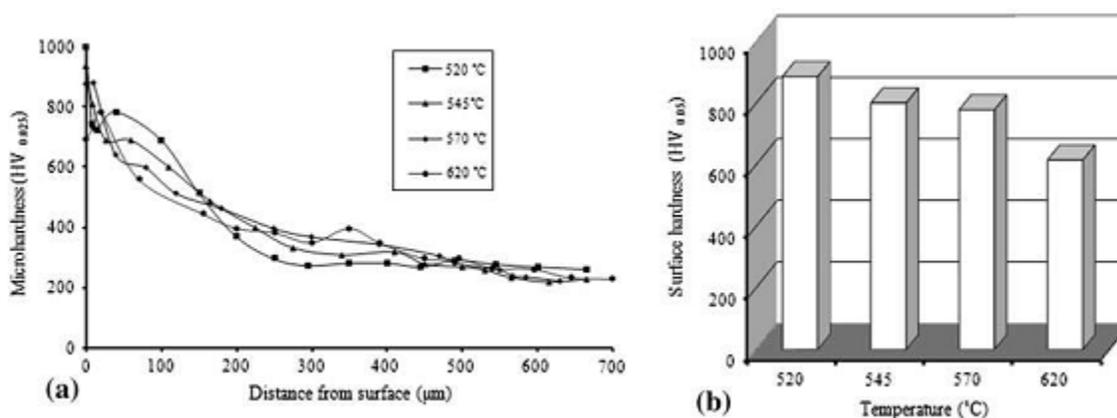
At moderate treatment temperatures, the modified surface exhibited a typical two-zone structure consisting of an outer compound layer and an underlying diffusion zone. However, when the treatment temperature approached the upper limit of the applied range, the microstructure became more complex and an intermediate transformed zone appeared between the compound layer and the diffusion region. This zone is attributed to phase transformations occurring during cooling, particularly in areas enriched with nitrogen and carbon.



**Fig. 2.** XRD results of plasma microcarburized AISI 4140 steel at different depths of the compound layer formed at 620 °C and at different treatment temperatures.



Detailed SEM analysis showed that the compound layer formed at higher temperatures consisted of two distinct sub-layers. The outermost region exhibited a relatively porous morphology, whereas the inner region was characterized by a more compact columnar structure. The porosity of the outer surface is associated with the accumulation and recombination of nitrogen at grain boundaries during plasma treatment. Beneath the compound layer, a darker transition zone was detected, corresponding to a transformed structure formed during post-treatment cooling. Slow cooling conditions favor the transformation of nitrogen-enriched austenitic regions into a mixture of ferrite and nitride phases, resulting in a refined microstructure.



**Fig. 3.** (a) Microhardness profiles and (b) surface hardness of AISI 4140 steel plasma microcarburized at different temperatures.

X-ray diffraction (XRD) analysis confirmed the presence of iron nitride and carbonitride phases within the compound layer. The phase composition varied with depth from the surface. The outer porous sub-layer was predominantly composed of  $\epsilon$ -type carbonitride phase, while the inner region showed a mixed structure containing both  $\epsilon$  and  $\gamma'$  phases. A gradual decrease in the  $\epsilon$ -phase fraction and a relative increase in  $\gamma'$  phase content were observed toward the bottom of the compound layer. This distribution suggests that nitrogen concentration decreases with depth, influencing the stability of the formed phases.



At greater depths, near the interface between the compound layer and diffusion zone, minor amounts of ferrite and carbide phases were detected. The presence of these phases is related to carbon redistribution during the thermochemical treatment. In low-alloy steels, carbon enrichment near the lower boundary of the compound layer can promote the formation of carbides during cooling.

With increasing treatment temperature, the intensity of diffraction peaks corresponding to carbonitride phases initially increased, indicating enhanced phase formation. At even higher temperatures, changes in peak intensity suggested partial structural modification associated with surface porosity development and phase transformation during cooling. Due to the limited penetration depth of Cu K $\alpha$  radiation in steel, the XRD results primarily represent the outer portion of the compound layer rather than its full thickness.

### **Conclusions**

The conducted research confirmed that the formation of the  $\gamma'$  phase and the development of compound layer thickness in nitrided steel are strongly dependent on treatment temperature. Increasing the nitriding temperature from 520 °C to 620 °C significantly intensified phase transformations within the surface layer. In the sample treated by plasma nitrocarburizing at 620 °C, an austenitic zone was formed beneath the compound layer, while the type and distribution of carbonitride phases varied along the cross-section from the surface toward the substrate.

The compound layer obtained at 545 °C exhibited the highest volume fraction of the  $\epsilon$  phase, whereas the maximum microhardness in the 620 °C treated sample was recorded in the middle region of the compound layer. A noticeable decrease in hardness was observed toward the substrate, which is associated with structural gradient changes across the diffusion zone.

The results demonstrate that nitrocarburizing treatment improves corrosion resistance primarily due to the formation characteristics of carbonitride phases and the morphological features of the compound layer surface. It was determined that



corrosion resistance is influenced more significantly by the type and distribution of carbonitride phases and surface porosity than by the total thickness of the compound layer. Therefore, optimizing phase composition and surface structure is more critical for enhancing corrosion performance than merely increasing compound layer thickness.

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