Study on Microstructure and Mechanical Properties of Nitrided and Titanized Carbon Steel

Jiawei Xu*, Lingbo Tang*, Long Chen
Hubei Key Laboratory of Digital Textile Equipment, Wuhan Textile University, Wuhan, China

*Corresponding author e-mail: 864669630@qq.com, *cy11211@163.com, b231499196@qq.com

Abstract. In this paper, the structure of nitrided titanium on carbon steel is studied, the interaction mechanism of nitrided titanium is analyzed, and the orthogonal process design of nitrided titanium is given. The mechanical properties of nitrided layer in different depth were studied, and the microstructure and mechanical properties of nitrided layer under different temperature and nitrogen-hydrogen ratio were analyzed. The microstructure, surface morphology, wear marks and element composition of the nitrided layer were observed and analyzed by means of Om, XRD, SEM and EDS, and the gradient hardness distribution, friction and wear properties of nitrided layer were tested and analyzed.

1. Introduction
A plasma generator is installed in the vacuum chamber. Through the discharge of the plasma generator, the nitrogen-containing gas in the vacuum chamber is ionized the sample is ionized and nitrided by nitrogen-containing plasma produced after ionization. Because the plasma production is no longer dependent on the sample itself, the defect edge effect and arcing phenomenon of plasma nitriding are eliminated, the increase of the Surface roughness of the sample after nitriding is small, and the sample is surrounded by plasma during nitriding Nitriding speed, suitable for all kinds of materials with complex shape, reduce energy consumption. Although Plasma nitriding technology has been developed for many years, and has been widely used, but a new type of plasma generator makes plasma nitriding technology constantly vibrant [1].

Plasma nitriding is to place the workpiece in the vacuum furnace which can produce glow discharge equipment, use the workpiece as cathode, use the vacuum furnace wall or other metal screen as anode, apply high negative bias voltage to the workpiece, under the effect of High Voltage Electric Field, under the attraction of the negative voltage of the work piece, the gas is bombarded to the cathode sample, and finally the energy is lost and absorbed by the work piece, thus providing a difference of nitrogen concentration on the surface of the sample The nitriding layer is formed by diffusion to the inside of the workpiece [2, 3].

PLASMA SOURCE PLASMA NITRIDING MAKES THE PLASMA PRODUCED IN PLASMA nitriding process independent of each other. A plasma generator is installed in the vacuum chamber [4, 5]. Through the discharge of the plasma generator, the nitrogen-containing gas in the vacuum chamber is ionized the sample is ionized and nitrided by nitrogen-containing plasma produced after ionization.
[6]. Because the plasma production is no longer dependent on the sample itself, the defect edge effect and arcing phenomenon of plasma nitriding are eliminated, the increase of the Surface roughness of the sample after nitriding is small, and the sample is surrounded by plasma during nitriding Nitriding speed, suitable for all kinds of materials with complex shape, reduce energy consumption.

2. Experimental materials and equipment

2.1. Experimental materials
The test material is M50NiL MARTENSITIC steel. Before the test, the steel needs to be quenched to make the alloy elements evenly distributed and fully arranged in the lattice to obtain the high quality penetration layer. The tempering and plasma diffusion processes are carried out simultaneously to stabilize the quenched martensite structure. The hardened steel is cut by uniform wire into sizes of 12 ~ 12 ~ 4 mm. The surfaces of the samples were smoothed with 240-800 # water sandpaper before plasma penetration.

2.2. Nitriding equipment
The nitriding test was carried out in the TGP-650 MULTI-ARC ION plating machine produced by Beijing Tecno Technology Co, Ltd. Mainly by the system control cabinet, Vacuum Chamber and Vacuum System, bias power supply, water system, air system, motor Bogie and so on. The Vacuum System Adopts Mechanical Pump and FF-200 / 1200 molecular pump double-stage pump, and the limit vacuum can reach 810-5 PA. The chamber heating system adopts several groups of stainless steel armored heaters, and then uses iodine tungsten lamp to heat the sample separately the surface temperature of workpiece can be directly measured by armored thermocouple, and the nitriding temperature can be accurately controlled.

2.3. Ancillary equipment
The nitriding test is carried out in the ZXT-500 high vacuum coating machine produced by Beijing Tecno Technology Co, Ltd. The nitriding test consists of system control cabinet, Vacuum Chamber, Vacuum System, bias power supply gas system and so on. The Vacuum System Adopts Mechanical Pump and FF-200 / 1200 molecular pump double-stage pump, the limit vacuum can reach 810-5pa. The Chamber Heating System Adopts Iodine Tungsten lamp to heat the sample separately, and uses armored thermocouple to measure the surface temperature of the workpiece directly Nitriding temperature can be controlled accurately.

3. Experimental Results and analysis of the effect of temperature

3.1. On the microstructure of nitried titanium coating
Temperature and time are two important factors in the process of plasma penetration, especially temperature. The diffusion coefficient of N and TI atoms in plasma can be influenced by temperature, which has an important effect on the precipitation and growth of the second phase and the stress between different phases. Mainly in the microstructure, phase structure and relative content, steel surface morphology, wear resistance and corrosion resistance and so on. When the temperature is low, the layer thickness is thin, so the modified steel cannot bear the influence of friction and wear for a long time, and cannot meet the service requirements. When the temperature is too high, the thickness of the layer increases obviously, but the structure coarsens easily, the size of the second phase is too large, the strengthening effect is weakened, and the hardness, wear resistance, corrosion resistance and other properties are greatly reduced. Therefore, it is of great significance to study the effect of temperature on the microstructure and mechanical properties of the infiltrated layer.

Fig. 1 is a metallographic picture of re-nitrided titanium layer in M50NiL steel at different temperatures. Compared with the samples treated at 460 °C, the corrosion morphology of Martensite
lath in 540 °C is clearer, which shows that the increase of temperature reduces the corrosion resistance of the coating.

![Figure 1](image1.png)

**Figure 1.** Microstructure of plasma rare earth nitrided titanium coating on M50NiL steel at different temperatures (a) 460°C, (b) 540°C

On the other hand, the thickness of the corrosion layer is only about 11 m at 460 °C, and about 50 m at 540 °C, which shows that the thickness of the corrosion layer is greatly increased by increasing the treatment temperature. In addition, it should be noted that the thickness of the interface between the diffusion layer and the diffusion layer is uneven. The lower the treatment temperature is, the more uneven the thickness (relative to the thickness of the corrosion layer) is. This is because the diffusion ability of nitrogen and titanium atoms is weak at low temperature, and it is difficult to diffuse inside the grains. With the increase of the treatment temperature, the diffusion ability of nitrogen and titanium atoms is enhanced, and it is easier to pass through the grain, and the difference of the velocity of nitrogen and titanium atoms diffused at the grain boundary is decreased.

3.2. Effect of hydrogen-nitrogen ratio on microstructure of nitrided layer

Nitrogen-hydrogen ratio is directly related to nitrogen concentration in plasma nitridation process. The higher the ratio of nitrogen to hydrogen, the higher the concentration of nitrogen ions in the furnace atmosphere, the higher the probability of nitrogen ions impacting the surface of steel, and the higher the concentration of nitrogen ions on the surface of steel in the same time, which makes the nitrogen concentration on the surface of steel more easily exceed the upper limit of solid solution. The compound phase is easier to precipitate out. The kind and proportion of the compound phase in the co-permeation layer play a vital role in the performance of the co-permeation layer.

Fig. 2 shows the metallographic structure of the re-nitrided titanium layer of M50NiL steel at 500 °C with different N / h ratios. The thickness of the corrosion layer is slightly 40 m when the ratio of nitrogen to hydrogen is 2:2, while the thickness of the corrosion layer is about 37 m when the ratio of nitrogen to hydrogen is 2:4. It should be noted that there are two phases of Fe4N and Fe2-3N in the surface layer after different N / h ratio treatment, but there is no white layer in the metallographic photos. It has been reported in the literature that the formation of bright white layers containing Fe4N and Fe2-3N phases needs to satisfy the condition that the nitrogen potential of the surface layer exceeds a certain critical value. It is shown that the nitrogen potential of the surface layer of the co-infiltrated layer does not reach the critical nitrogen potential of the white-bright layer formed by M50NiL steel at 500 °C, which results in the existence of Fe4N and Fe2-3N phases in the co-infiltrated layer. However, the appearance of the white layer is not found in the metallographic photographs.
Figure 2. Microstructure of plasma nitrided titanium coating with different nitrogen-hydrogen ratio at 500 °C  (a) N: H 2:2, (b) N: H 2:4

3.3. Effect of voltage on microstructure of nitrided layer
Fig. 3 is a metallographic picture of nitrided layer processed for 4 hours at 510 °C and the applied voltage of Cathode Plate from-0.4 kv to-0.8 Kv. It can be seen that the thickness of nitrided layer increases with the increase of applied voltage of cathode plate. The increase of the voltage applied to the cathode plate results in the increase of the density of the nitrogen plasma reaching the surface of the sample. The plasma density produced by the Cathode Plate with-0.8 kv voltage is three times that of-0.4 KV The thickness of the diffusion layer, as observed in the metallograph, did not increase much, but it was noted that the corrosion of the diffusion layer increased a lot, indicating an increase in the concentration of nitrogen atoms in the diffusion layer. There was no white compound layer on the surface of the nitriding layer, and there was no vein structure in the diffusion layer, and the nitriding layer had good structure. However, compared with ion source assisted nitriding, the distribution of nitrided layer obtained by this nitriding method is not uniform.

With the increase of the cathode plate voltage, the sample will fire because it is too close to the cathode. As can be seen from FIG. D, the distribution of nitrides in the nitriding layer is extremely uneven due to sample firing. The main reason for sample firing is the change of the surface temperature of the sample, thus changing the diffusion rate of nitrogen. The temperature inhomogeneity also seriously affects the nitriding layer's organization, even can produce the vein organization.

Figure 3. Microstructure of nitrided layer of M50 steel under different voltage
a) -0.4kV; b) -0.6kV; c) -0.7kV; d) -0.8kV
3.4. Effect of air pressure on microstructure of nitrided layer

Fig. 4 shows the hardness of nitriding cross-section and the thickness of nitriding layer treated for 4 hours at 510 °C and the voltage applied to the cathode plate at -0.5 kV under different nitrogen pressure. The thickness of nitrided layer and nitrides in nitrided layer increased with the increase of air pressure, and the density of nitrogen plasma increased with the increase of air pressure, and reached the highest at about 30 Pa. And then decrease as the air pressure increases. At nitrogen pressure of 200 Pa, the plasma density is the lowest, but at the order of 10^14, the thickest nitrided layer is obtained.

This shows that in glow discharge assisted nitriding, not only the influence of plasma density on nitriding, but also the influence of cathode glow discharge characteristics on nitriding should be considered. We know that the thickness of the cathode drop region produced by glow discharge is negatively correlated with the gas pressure, that is, the higher the gas pressure is, the thinner the cathode drop region is, and the Langmuir probe is in the cathode drop region. The relative velocity of Nitrogen Ions is very high, and the Langmuir probe itself has a negative voltage and is in the region of saturated ion current, so the measured plasma density is high. However, it can be observed in the experiment that, although the sample is in a suspended state, it occasionally produces an anodic glow which puts the sample in the positive column of the glow discharge. The electrons attracted by the glow from the anode are much hotter than the gas, meaning that the electron energy is enough to ionize the nitrogen-containing gas around the sample again, producing nitrogen ions for plasma nitriding.

![Figure 4. Microstructure of nitrided layer of M50 steel at 510 °C and different atmospheric pressure](image)

4. Conclusion

A 35 m thick nitriding layer can be obtained after 4 hours of auxiliary nitriding in the temperature range of 430-530 °C by glow discharge of Cathode Plate, and no white-light compound can be found on the surface of the nitriding layer. There is no vein-like tissue in the diffusion layer, and the low content of nitriding layer makes the metallographic picture difficult to be recognized. The low content of surface hardness in nitriding layer makes the metallographic picture difficult to be recognized. The hardness gradient decreased slowly; the surface roughness increased very little and increased to the...
highest 0.034 m compared with 0.015 m after polishing; the wear rate decreased by 75-85% compared with that of the non-nitrided sample.

The nitriding layer of more than 25 m can be obtained by raising the nitrogen concentration gradient between the surface and the center of the sample, and the nitriding layer depth increases by 15% with the increase of the nitrogen ion density by 3 times. When a voltage of -0.8 KV is applied to the cathode plate, when the sample meets the cathode and Anode, the sample meets the cathode and anode glow effect, which causes the Surface roughness to increase to 0.050 m and the friction coefficient to increase to more than 1.0.

The 25-30 m thick nitriding layer can be obtained by plasma nitriding in pure nitrogen 15-200 PA for 4 hours. The roughness of M50 sample after nitriding is between 0.03-0.048 MS, which is 100-160% higher than that of the original sample without nitriding.

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Corresponding author: Tang Lingbo.

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