



# Anode plasma electrolytic nitrohardening of medium carbon steel



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## ABSTRACT

In this work, we investigate the structure of the medium carbon steel which is formed as a result of plasma electrolytic nitriding with following hardening in the same electrolyte. The cross-sectional microstructure, composition and phase constituents of modified layer under different processing conditions were characterized. It is shown that the electrolyte that contained ammonia chloride and ammonia nitrate provides the saturation of steel with nitrogen up to 5.6 wt.% and formation  $\text{Fe}_4\text{N}$  и  $\text{Fe}_{2-3}\text{N}$  nitrides. The nitrogen diffusion decreases the austenitization temperature and results in formation of martensite after the sample cooling in the electrolyte. The aqueous solution that contained 15 wt.%  $\text{NH}_4\text{Cl}$  allows one to obtain the layer microhardness up to 1060 HV during 5 min at 750 °C. Modified layer thickness that contained iron nitrides, martensite and retained austenite is determined by nitrogen diffusion which depends on the thickness of the surface oxide layer. In its turn, the oxide layer thickness is affected by anode dissolution and oxidation of the steel substrate. It is found that ammonia chloride promotes the decrease in the oxide layer thickness owing to the anode dissolution that results in the growth of the modified layer. The increase in the concentration of ammonia nitrate as a donor of nitrogen leads to the oxidation and the decrease in the modified layer thickness. Surface roughness of samples treated under different processing modes and concentration of electrolyte components is increased due to the insufficient rate of anode dissolution. The minimal roughness is observed after nitrohardening at 700 °C in the electrolyte with 10 wt.%  $\text{NH}_4\text{NO}_3$ .

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## 1. Introduction

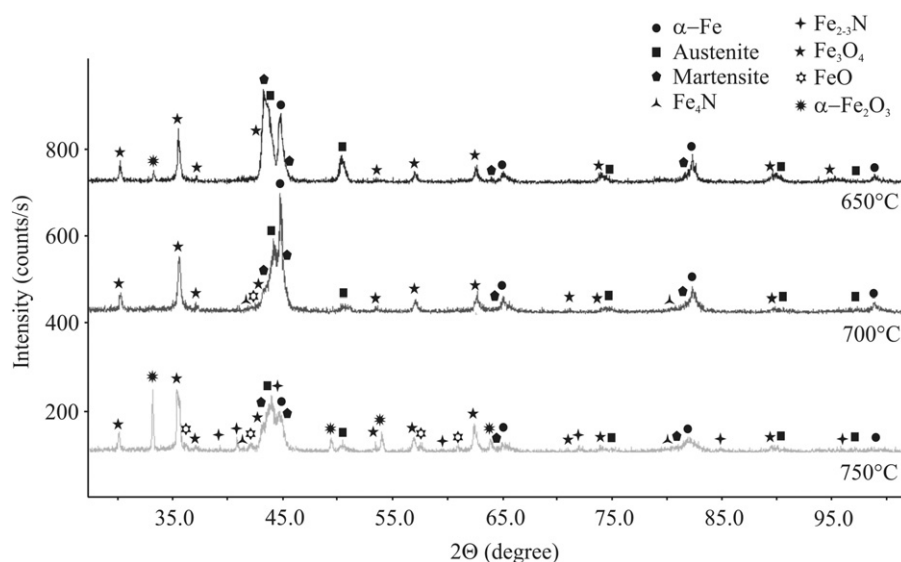
Diffusion saturation of steel with nitrogen increases sufficiently its hardness, endurance limit, wear and corrosion resistance in the atmosphere, water vapor, etc. Nitriding is used initially on medium carbon steels with Cr, Mo, V, and Al to create a case hardened surface and wear resistance owing to formation of  $(\text{Fe}, \text{M})_{2-3}\text{N}$  and  $(\text{Fe}, \text{M})_4\text{N}$  nitrides. Today the nitriding is used on low-carbon and low-alloy steels. This process is carried out mainly in gases that contained ammonia, in salt bath and plasma of glow discharge [1]. Plasma electrolytic nitriding (PEN) of cast steel S0050A in carbamide solution at 280 V (~600 °C) during 8 min results in the formation of nitrated and diffusion layers with thicknesses of 45 μm and 100 μm, respectively [2]. PEN of cast iron shows the similar results. Wear rate of treated samples is decreased in spite of the increase in roughness and friction coefficient. The possibility of cathode nitriding of austenite stainless steel in an aqueous solution of ammonium carbonate [3], high-speed steel R6M5 in electrolyte that contained carbamide and sodium carbonate [4], and medium carbon steel JIS SCM420 using electrolyte with carbamide and potassium hydrate [5] are shown. Anode PEN of austenitic stainless steel 304 and

martensitic stainless steel 403 using the aqueous solution of ammonium chloride and ammonia results in the corrosion rate decrease in the solution that contained boric acid/borax [6].

Saturation of the steel surface with nitrogen results in both the formation of nitrides or solid solution of nitrogen in iron and the decrease in the austenitization temperature. According to Fe–N phase diagram the eutectoidal transformation temperature is decreased to 700 °C in the presence of 0.07 wt.% N. This temperature is 590 °C when steel contains 0.1 wt.% N or more [1]. Therefore, the saturation with nitrogen at 650–750 °C during several minutes results in the austenitization of the surface layer and the formation of the nitrogen-formed martensite in the case of rapid cooling. PEN combines easy with quenching in the same electrolyte; therefore, this process can be named nitrohardness.

The most prevalent donor of nitrogen is ammonia which is added directly in solution for PEN or liberated in the saturating media due to some reactions. In particular, the electrolyte contained 10%  $\text{NH}_4\text{Cl}$  and 5%  $\text{NH}_4\text{OH}$  permits one to obtain the nitride layer with thickness 40 μm on the pure iron during 10 min at 750 °C [7]. Plasma electrolytic nitrohardening (PENH) of medium carbon steels results in a significant improvement of their mechanical properties. Hardened layer on the steel with 0.45% C has a thickness of 0.3 mm, surface microhardness of 950 HV after 5 min treatment at 750 °C. It will be noted that the martensitic transformation occurs in the layer where nitrogen content is sufficient for the steel austenitization. For this reason the martensite layer thickness is determined by nitrogen diffusion which depends on

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**Fig. 1.** X-ray diffraction patterns of surface layers after anode PENH in a solution of ammonium nitrate (10%) and ammonium chloride (10%) during 5 min for different treatment temperatures.

the processing time. Anode PENH of steels results in the sufficient increase in their endurance limit by a factor of 2 or more [8] because of increase in steel strength and creation of residual compressive

stresses [7]. Steel PENH is found to increase in the wear rate at dry condition against sintered TiC [9,10].

A disadvantage of electrolyte with ammonia is low resource due to the rapid evaporation of ammonia. In this article, we study PENH of medium carbon steel (0.45% C) in aqueous solution that contained ammonia chloride and ammonia nitrate that include some electrochemical aspects. Carbon diffusion is known to be prevented by a surface oxide layer [11]; the thickness of this layer is determined by the competition of anode dissolution of steel and its oxidation [12]. Also this competition affects surface roughness of low-carbon steel carburized in electrolyte with glycerol [13]. Nitrogen diffusion is suggested to be prevented also by oxide layer, and its thickness depends on the processing mode and electrolyte component concentrations. This hypothesis is verified by studying the dependence of the thickness of the structural components of the modified layer on the concentration of ammonium chloride, ammonium nitrate, temperature of samples and treatment time.

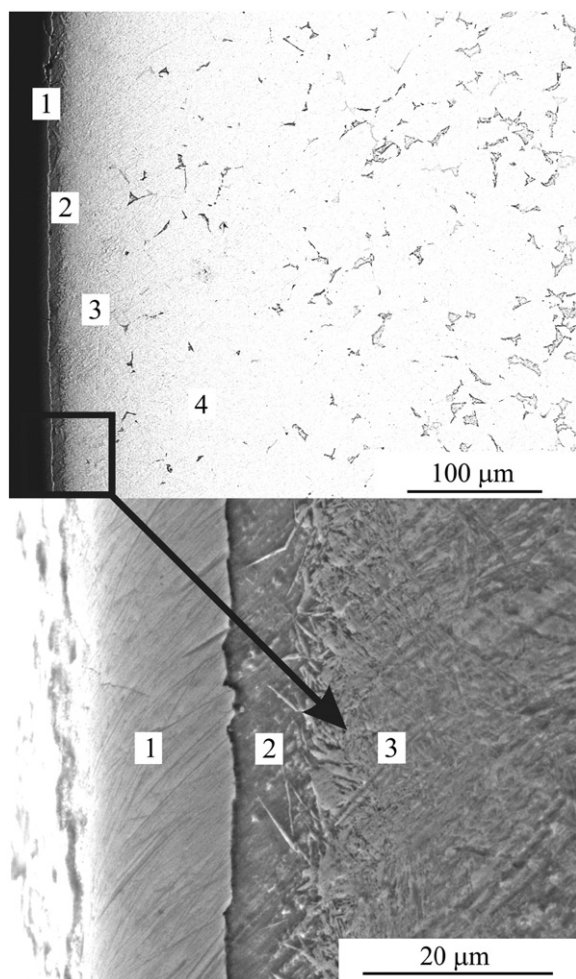
## 2. Experimental procedure

### 2.1. PENH treatment

Cylindrical samples ( $\varnothing 12 \times 15$  mm) of medium carbon steel (0.45% C) were anode nitrohardened in a cylindrical working chamber with an axially symmetric electrolyte flow supplied through a nozzle located at the bottom of the chamber [14]. In the upper part of the chamber, the electrolyte overflows into the sump and is further pumped through a heat exchanger at a rate of 3 l/min. The volume flow rate was measured with the use of a RMF-0.16 GUZ flowmeter (possessing an accuracy of  $\pm 2.5\%$ ). The electrolyte temperature was measured with the use of an MS-6501 digital thermometer and rapid response thermocouples (possessing an accuracy of  $\pm 3\%$ ) and was maintained at  $20 \pm 1$  °C. The samples were connected as the positive output and the chamber was connected as the negative output of the power supply.

After switching the voltage, the samples were immersed in the electrolyte to a depth equal to their height. The voltage was measured with the use of an LM-1 voltmeter (possessing an accuracy of  $\pm 0.5\%$ ). The current was probed with an MS8221 multimeter. The sample temperature is measured with the use of a second MS8221 multimeter and M89-K1 thermocouple was inserted into the sample approximately 1 mm to the low surface by drilling a hole (accuracy to within  $\pm 2.0\%$ ).

An aqueous solution of ammonium chloride and of ammonium nitrate was used as the working electrolyte. The concentration of the



**Fig. 2.** SEM image of cross-section of the steel surface after anode PENH in a solution of ammonium nitrate (10%) and ammonium chloride (10%) at 750 °C for 5 min. 1—surface oxide layer; 2—nitride-martensite layer; 3—martensite-ferrite layer; and 4—initial pearlite-ferrite structure.

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