

POROSITY IN SURFACE LAYERS AFTER CHEMICAL-HEAT TREATMENT

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Abstract

The article deals with porosity of compound layers after gas and plasma nitriding. Experiments are focused on using of gas and plasma nitriding processes for surface treatment. Nitriding technologies were applied to steels C 35 (sample A1) and steel 34CrNiMo6 (sample A6), which were subsequently evaluated by electron microscopy, GDOES, XRD and microhardness methods. The results of measurement showed characteristics of chemical composition of alloying elements in core of material and in created nitrided layer after chemical-heat treatment process. Main task was to compare the porosity after plasma and gas nitriding and finally prepare the documentation of pores from surface and from cross sectional structure. Plasma and gas nitriding process were applied for increasing of surface hardness of material in depth and improve mechanical and tribological properties. Mechanical and tribological properties of tested material were significantly increased.

Keywords: Gas nitriding; plasma nitriding; alloying elements; nitrided layer; porosity

1. INTRODUCTION

The chemical-heat treatment is in many cases applied to already heat-treated steels, i.e. after heat-treatment process [1]. Non-equilibrium structure is due to redistribution of alloying elements in crystal lattice more suitable for diffusion process. The aim of this paper is to achieve an enhanced surface hardness, better wear resistance, reduced friction coefficient, increase fatigue limit or corrosion resistance with low occurrence of porosity. During nitriding process the nitrides of iron are primarily created. These type of nitrides caused low increasing of microhardness. Main elements that caused increasing of mechanical properties are alloying elements as molybdenum, vanadium, aluminium or chrome. During nitriding process, two layers are mostly created. The compound layer created on the surface of steel is consisted of ϵ -Fe₂-3N and γ -Fe₄N phase [2]. The proportion of individual phases is dependent on carbon concentration in steel [1]. The compound layer has been very hard and brittle with good friction and anticorrosion properties [2, 3]. Unfortunately, the porosity is very often presented in these surface compound layers. The thickness and hardness of γ '-Fe₄N (diffusion layer) depends on quantity and quality of alloying elements [3, 4, 5, 6]. This article describes the influence of type of nitriding technology to formation of areas of pores in surface compound layers. The porosity is presented in surface layer and be able to have bad influence on mechanical properties, esp. the wear resistance [7, 8, 9]. Chemical composition of steel was verified by GDOES/Bulk method on LECO SA 2000 spectrometer and local measurement of composition was carried out on SEM microscope Hitachi Tabletop 3000 equipped by XRD Oxford Instruments Stream-2. Microstructure was evaluated by electron microscopy method on Hitachi Tabletop 3000. Thickness and microhardness of nitrided layers were measured by microhardness method in accordance with DIN 50190 standard on automatic microhardness tester LECO LM 247 AT [10].

2. MATERIAL AND METHOD

Steel C35 (sample A1) and 34CrNiMo6 (sample A6) in untreated ferrite-pearlite state were heat-treated. Tempering and quenching was performed due to reach different microstructure with different parameters of microhardness and different redistribution of atom in crystal lattice.

Table 1 Chemical composition of selected steel

Sample	Chemical composition of steel [w%(X)]						
	C	Mn	Si	Cr	Ni	Mo	Al
A1 (C35)	0.36	0.68	0.33	0.08	0.04	-----	0.003
A6 (34CrNiMo6)	0.35	0.76	0.23	1.64	1.62	0.19	0.024

The chemical composition of material was measured by GDOES/Bulk method on reference samples (**Table 1**). Glow discharge optical spectroscopy (GDOES) measurements were performed in LECO SA-2000, with argon glow discharge plasma excitation source, calibration of nitrogen: JK41-1N and NSC4A standards.

Table 2 Conditions of heat-treatment

Steel	Sample	Process	
		Quenching	Tempering
C35	A1	860 °C, water	600 °C, air
34CrNiMo6	A6	850 °C, oil	600 °C, water

The steel structure was evaluated by electron microscopy on SEM Hitachi Tabletop 3000 before chemical heat-treatment. The structure after quenching is displayed in **Figure 1**. The conditions of heat-treatment process are given in **Table 2**.

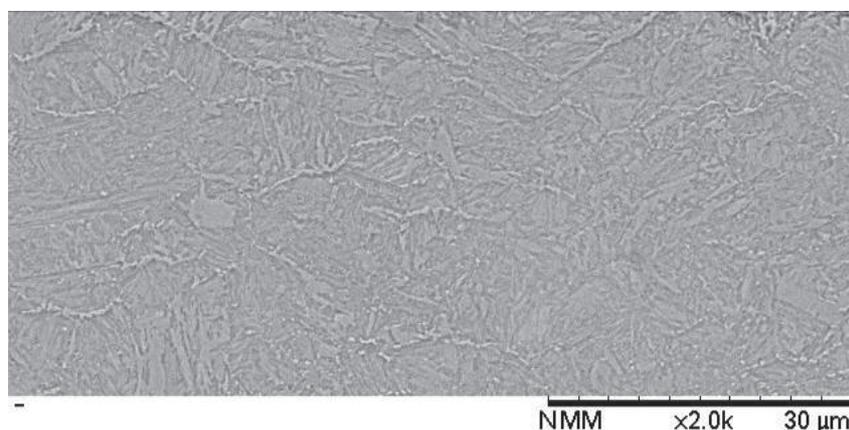

Figure 1 The chemically etched optical cross-sectional structure of sample A1 after heat-treatment

Table 3 Depth of nitrated layers after gas and plasma nitriding

Technology	Depth (μm)			
	A1		A6	
	Compound layer	Diffusion layer	Compound layer	Diffusion layer
Plasma nitriding 30 h	5.36	0.26	4.24	0.29
Gas nitriding 6 h	9.72	0.22	11.45	0.20

The microhardness of steel was evaluated by Vickers microhardness method on the automatic microhardness tester LM 247 AT LECO. Load set at 50 g and 10 s dwell time. This method was used for microhardness measurement of surface and the measurement of created diffusion layer in depth after gas and plasma nitriding

process (Table 3, Figure 2). A microhardness of heat-treated sample A1 was analysed to value 499 ± 31 HV 0.05 after quenching and 266 ± 6 HV 0.05 after tempering. In case of sample A6 it was reached the value of microhardness 658 ± 44 HV 0.05 after quenching and 310 ± 20 HV 0.05 after tempering.

The initial microhardness after quenching is usually during process of chemical-heat treatment decreased due to high temperature close to A_1 curve Fe-Fe₃C (Figure 2).

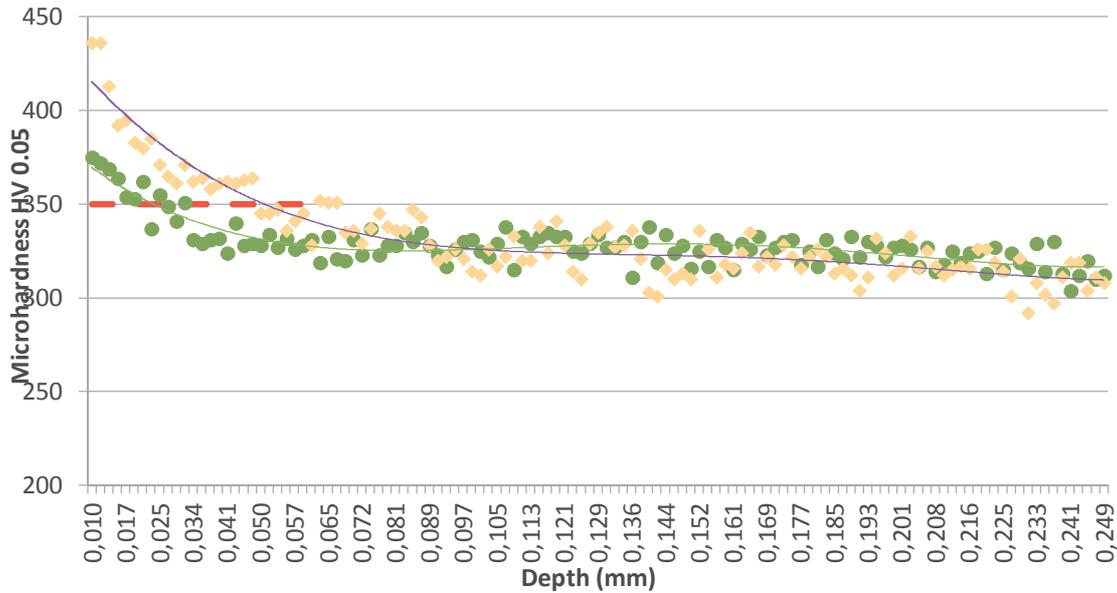


Figure 2 The increasing of surface microhardness caused by different microstructure of sample A1 after gas nitriding process (green curve tempered, yellow curve quenched)

After preparation and nital etching, the electron microscope Hitachi Tabletop 3000 was used for observation of surface morphology and cross-sectional structure documentation (Figure 1).

The thickness of created compound layer and the surface morphology after nitriding process were evaluated by electron microscope Hitachi Tabletop 3000 (Table 3, Figures 3, 4) equipped by XRD Oxford Instruments Stream-2.

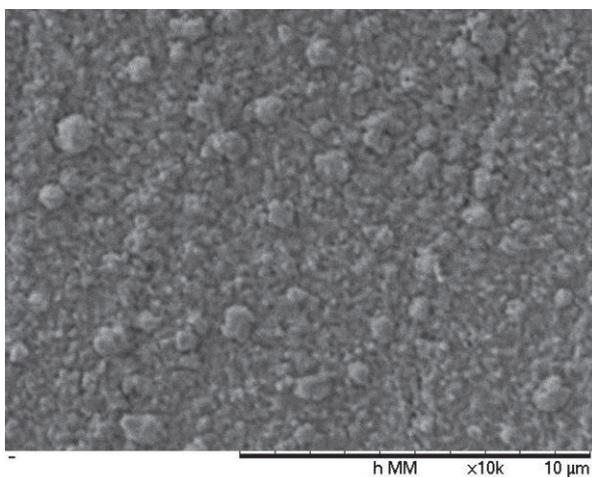


Figure 3a The surface morphology of A1 after plasma nitriding process

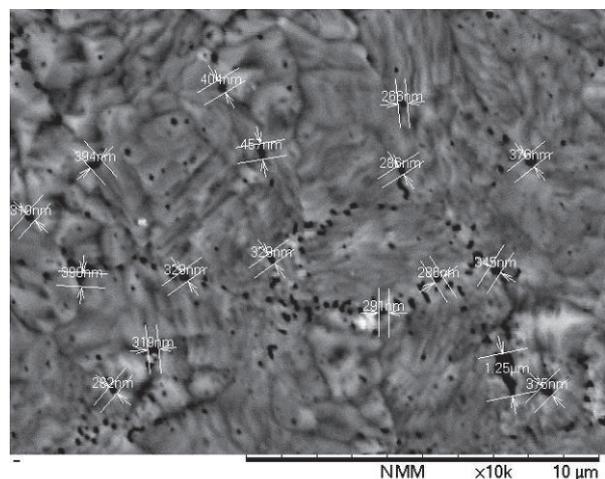


Figure 3b The surface morphology of A1 after gas nitriding process

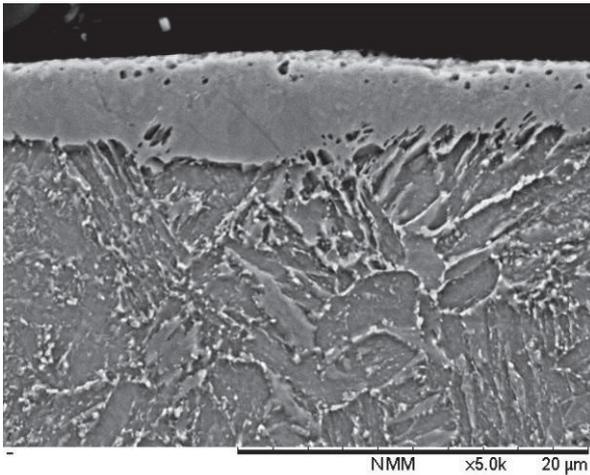


Figure 4a Cross-sectional structure of A1 after plasma nitriding process

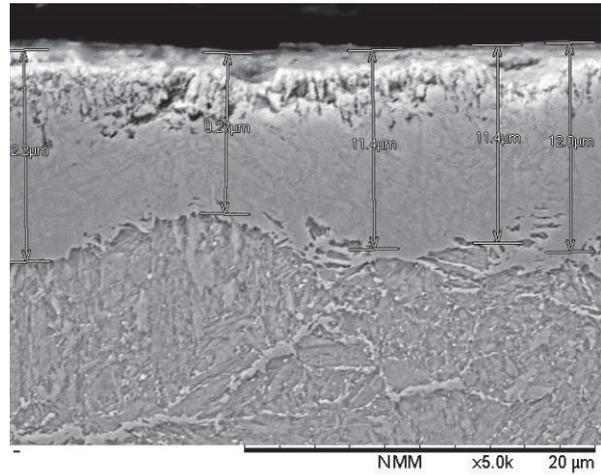


Figure 4b Cross-sectional structure of A1 after gas nitriding process; overview of porosity

The results of measurements of nitrogen (N K), oxygen (O K), molybdenum (Mo L), chrome (Cr K), manganese (Mn K) and iron (Fe K) were performed from two local spaces by magnification 2000x by method of surface analysing (mapping). Results are displayed in **Table 4**. The changes of the chemical composition were measured at an accelerating voltage of 20kV and exposure time 50 sec.

Table 4 Selected results of chemical composition measured by XRD SEM mapping method

Element	Technology			
	Plasma nitriding		Gas nitriding	
	C 35	34CrNiMo6	C 35	34CrNiMo6
	A1	A6	A1	A6
Fe	88.2 ± 0.5	79.4 ± 0.7	85.6 ± 0.6	76.4 ± 0.1
C	5.1 ± 0.4	11.1 ± 0.6	6.3 ± 0.5	13.7 ± 1.0
N	3.7 ± 0.2	3.3 ± 0.4	4.8 ± 0.3	4.6 ± 0.6
O	1.7 ± 0.2	2.3 ± 0.2	1.6 ± 0.2	2.1 ± 0.3
Ni	-	1.4 ± 0.2	-	0.7 ± 0.3
Cr	-	1.2 ± 0.1	-	0.2 ± 0.2
Mn	0.7 ± 0.1	0.5 ± 0.1	0.9 ± 0.1	1.0 ± 0.2
Si	0.3 ± 0.0	0.5 ± 0.0	-	0.3 ± 0.1
Mo	-	0.2 ± 0.1	-	-

3. RESULTS AND DISCUSSION

The main effort of experimental work was aimed at analysing of chemical and mechanical properties of the surface layers created by gas and plasma nitriding process. Main task was to prove the differences in compound layer which is created after plasma and gas nitriding. In compound layers the porosity was created without difference of using technologies, i. g. present after plasma and gas nitriding. The porosity was significantly increased after gas nitriding (**Figures 4a, 4b**). The porosity was larger after gaseous technologies in all experiments. The occurrence of pores was reduced after plasma technologies (**Figure 4a**). In compound layers created by plasma technologies there were presented only independently pores what is documented in **Figure 4a**. This result was confirmed by surface morphology (**Figure 3a**). In case of gas nitriding the porosity

was visible from the surface of created compound layer (**Figure 3b**). The influence of chemical composition on porosity development was not proved. Part of samples A1 and A6 were hardened and quenched (**Table 2**). The results showed the interesting difference in surface microhardness. In dependent on chemical composition (concentration of alloying elements) the depth of microhardness was changeable. Chemical heat-treatment caused decreasing of initial microhardness of steel due to long time process of nitriding (**Figure 2**).

4. CONCLUSION

Gas nitriding process caused increasing of porosity in compound layer. It was reached significant reductions in case of using plasma nitriding process. It is obvious that compound layer is partially diffusion layer. The top part of compound layer was formed due to condensation of particles in atmosphere (**Figure 5**). We assume that the pores are predominantly formed in non-diffusion part of compound layer. The experiments showed that the chemical composition of surface compound layer after plasma and gas nitriding has different value of substitution elements. The chemical composition has not remarkable influence on porosity. The formation of pores is dependent on selected technology. The diffusion process is more effective in gaseous atmospheres than in plasma. After 6 hours' process of gas nitriding it was reached more suitable parameters of layers than after 30 hours' plasma process (**Table 3**).

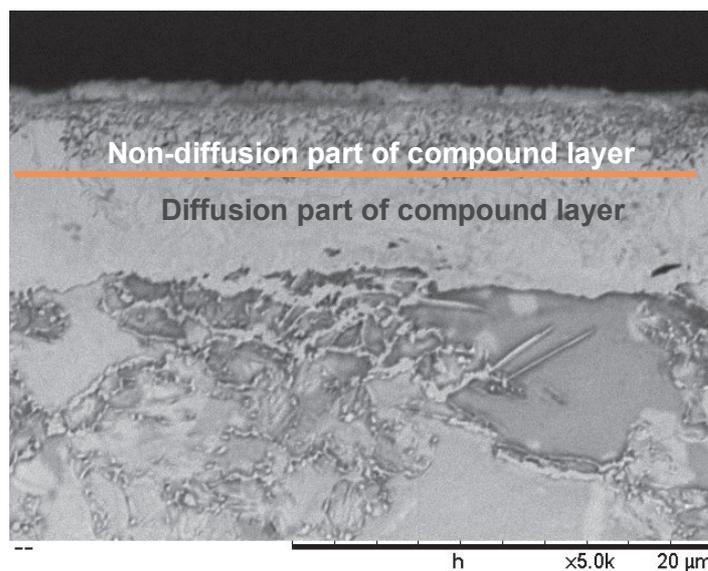


Figure 5 Overview of formation of pores in the compound layer after gas nitriding process

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