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Characteristics of plasma nitrided layers in deep holes

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Abstract

The article deals with mechanical properties of nitrided layers which were created by plasma nitriding technology. Technology of plasma nitriding is generally used to increase the surface hardness, fatigue strength, wear and corrosion resistance of steels. Experiments were focused on using plasma nitriding process for surface treatment of cavities with diameter of 6 and 8 mm. Nitrided layers were applied to steel 42CrMo4 which were subsequently evaluated by metallography, GDOES, microanalysis and microhardness method. The results of experiments have shown that plasma nitriding process is applicable to mentioned cavities. The surface hardness of material was significantly increased. The measurements have shown that the surface hardness and microhardness are dependent on the content of nitrogen in material.

Key words: nitriding, microhardness, nitrided layer, Nht thickness

1. Introduction

The plasma nitriding is a method of surface hardening using d.c. glow discharge to improve elemental nitrogen to the surface of steel with subsequent diffusion into the bulk of material [1, 2]. Generally, two layers are created during plasma nitriding process. The compound layer consists of ε-$Fe_{2-3}N$ and γ-Fe_4N phase [3]. This type of layer is very hard, but unfortunately brittle, with good friction and anticorrosion properties [4]. The thickness and hardness of γ'-Fe_4N depend on quantity and quality of nitride-formed elements [5]. The composition of nitrided layers can be effectively influenced by chemical composition of nitriding atmosphere [6, 7]. This article describes the properties of nitrided layers which were created inside the cavities. The other part of this study is devoted to the creation of nitrided layers inside cavity with the diameter smaller than 6 mm. Nht thicknesses of mentioned samples were subsequently compared with content of nitrogen. This study deals with mechanical properties of nitriding layers which were created by the pressure of 500 Pa. Chemical composition of steel was verified for selected chemical elements by GDOES/Bulk method on LECO SA 2000 spectrometer and local measurement of composition was carried out on SEM microscope with micro analyser Philips Edax 9900. Microstructure was evaluated by laser confocal microscopy Olympus OLS 3000. Thickness and microhardness of plasma nitrided layers were measured by microhardness method in accordance with DIN 50190 standard on automatic microhardness tester LECO LM 247 AT.

Table 1. Temperatures of heat-treated steels

Procedure	Temperature (℃)
Oil quenching	850
Salt tempering	300

2. Experimental work

Bars of 42CrMo4 steel in an untreated state were cylinder bored with diameter of 6 and 8 mm. Samples of length 500 mm were heat treated in accordance with Table 1.

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Table 2. Parameters of plasma nitriding process

Temperature (℃)	500
Duration (h)	6
Gas flow H_2/N_2 ($l\,min^{-1}$)	24/8
Bias (V)	530
Pressure (Pa)	500
Pulse length (μs)	100

Table 3. Chemical composition (at.%) of 42CrMo4 steel

C	Mn	Si	Cr	Mo	Ni	P	S
GDOES/Bulk							
0.40	0.64	0.28	1.14	0.16	0.32	0.012	0.012
DIN Standard							
0.38 0.40	0.50 0.80	< 0.37	0.90 1.20	0.15 1.30	< 0.50	< 0.030	< 0.030

Microhardness of an untreated material of samples was 250 $HV_{0.05}$. Plasma nitriding was carried out in PN 60/60 RÜBIG furnace according to Table 2. The charge consisted of 2 cylindrical samples (cavities) which were plasma nitrided at the pressure of 500 Pa for 6 h.

After plasma nitriding process, the samples with the diameters of 6 and 8 mm were cut off. The length of the first sample was 30 mm, all others were 12 mm longer. The lengths of next samples were as follows: 30, 42, 54, 66, 78, 90, 102, 114, 126, 138, 150, 162, 174, 186, 198, 210, 222, 234, 246, 258 mm.

All samples were wet grounded using silicon carbide paper with grit from 80 down to 2000 and subsequently polished. Confocal laser microscope LEXT OLS 3000 with an outstanding resolution of 0.12 μm and magnification range from 120× to 12400× was used for observation, cross-structure documentation and compound layer evaluation (Fig. 1).

Chemical composition of material was measured by GDOES/Bulk method (Table 3).

The depth profiles were evaluated by GDOES/QDP method and are shown in Fig. 2.

Glow discharge optical emission spectroscopy (GDOES) measurements were performed in LECO SA-2000, with argon glow discharge plasma excitation source, calibration of nitrogen: JK41-1N and NSC4A standards.

The microhardness was measured by Vickers microhardness method on the automatic microhardness tester LM 247 AT LECO at 50 g load and 10 s dwell time. The major Vickers microhardness numbers were

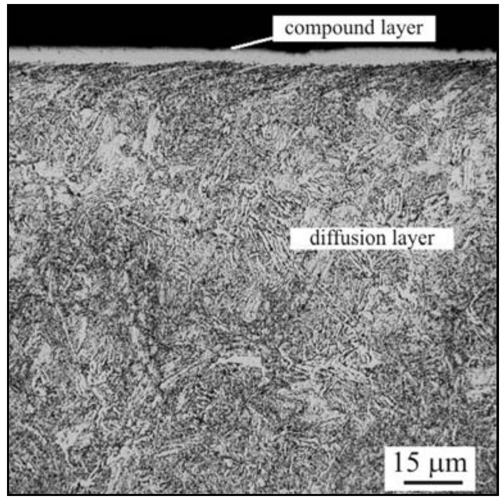

Fig. 1. Chemically etched confocal cross-sectional structure of tempered steel, compound layer (top of surface) and diffusion layer below.

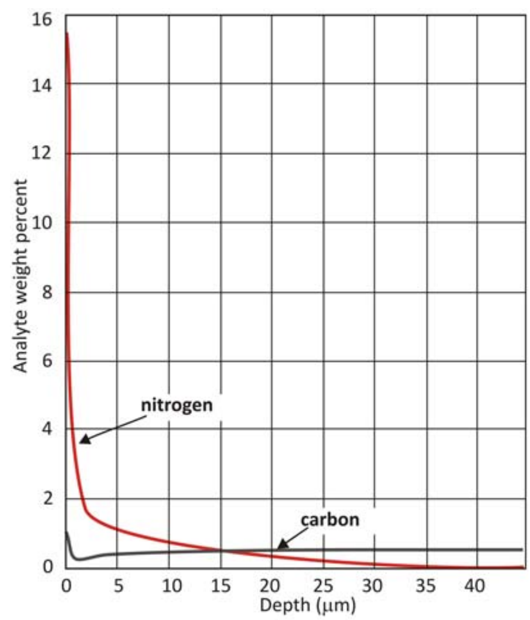

Fig. 2. GDOES depth profile, measured on reference sample; plasma nitriding 500 ℃/6 h/500 Pa.

derived from five measurements as an average value according to Fig. 3 and the results are displayed in Fig. 4.

Following equation was used for calculation of Nht thickness X (1) in accordance with DIN 50190 standard:

$$X = [[Y * 0.1] * 10] + 50, \tag{1}$$

where X is Nht thickness in mm, Y is the average mi-

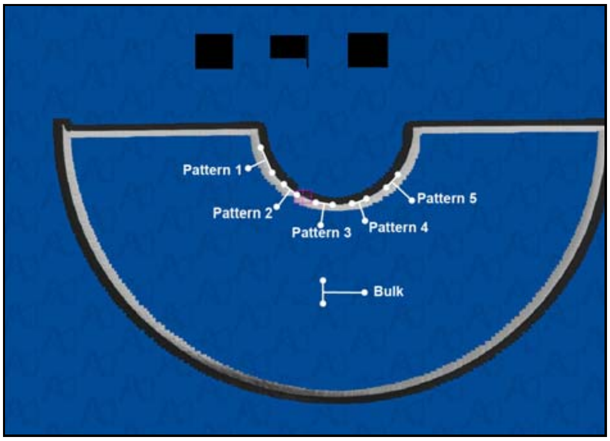

Fig. 3. Real image of measured sample from LM 247.

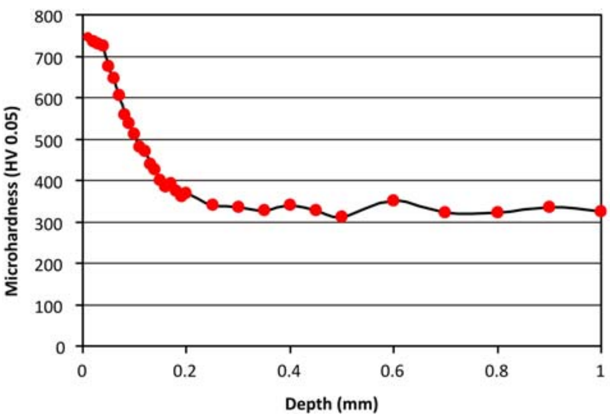

Fig. 4. Microhardness depth profile measured in length 30 mm from forepart; plasma nitriding process 500 °C/ 6 h/500 Pa.

crohardness number from five indentation's patterns in $HV_{0.05}$ [kg].

The local chemical compositions of plasma nitrided layers in length of cavity were observed by SEM method in combination with energy dispersive micro analyser PHILIPS EDAX 9900. Measurement of nitrogen content was performed from two local spaces by 25× magnification. The results of analysis are shown in Table 4 and Fig. 5.

3. Results

Heat-treated samples with diameters of 6 and 8 mm which were plasma nitrided at pressure of 500 Pa were investigated and subsequently compared. Microhardness of depth profiles of plasma nitrided layers confirmed enhancement of microhardness about 400 $HV_{0.05}$ which is shown in Fig. 4. Nht thickness of plasma nitrided layer was measured in accordance with DIN 50190 standard [1].

Table 4. Trends of microhardness thickness and nitrogen concentration

Length (mm)	Nht thickness (mm)	Content of nitrogen (%)
30	0.33	5.69
52	0.30	5.09
64	0.34	6.55
76	0.33	5.71
88	0.32	5.72
100	0.32	5.71
112	0.34	6.26
124	0.31	5.64
136	0.31	6.15
148	0.18	4.26
160	0.10	0.95
172	0.00	1.11

Table 5. Thickness of nitrided layer in cavities with diameter of 6 and 8 mm

<table>
<tr><th rowspan="3">Length (mm)</th><th colspan="2">Cavities with diameter of</th></tr>
<tr><th>8 mm</th><th>6 mm</th></tr>
<tr><th colspan="2">Nht thickness (mm)</th></tr>
<tr><td>30</td><td>0.20</td><td>0.33</td></tr>
<tr><td>150</td><td>0.15</td><td>0.10</td></tr>
<tr><td>162</td><td>0.18</td><td rowspan="8">0.00</td></tr>
<tr><td>174</td><td>0.19</td></tr>
<tr><td>234</td><td>0.04</td></tr>
<tr><td>246</td><td rowspan="5">0.00</td></tr>
<tr><td>258</td></tr>
<tr><td>270</td></tr>
<tr><td>282</td></tr>
<tr><td>294</td></tr>
</table>

The sample with the diameter of 6 mm attained Nht thickness 0.33 mm in length 30 mm. In cavity with diameter of 8 mm the value of Nht thickness 0.20 mm was attained in the same length (Fig. 6, Table 5). In cavity with diameter of 6 mm the nitrided layer was found in length 162 mm while in cavity with diameter of 8 mm the nitrided layer was found in length 246 mm (Fig. 6). The plasma nitriding of cavity with diameter of 8 mm caused the increasing of length of nitriding (Fig. 6).

4. Conclusion

Experiment have shown that plasma nitriding process is applicable not only for flat surfaces but for cav-

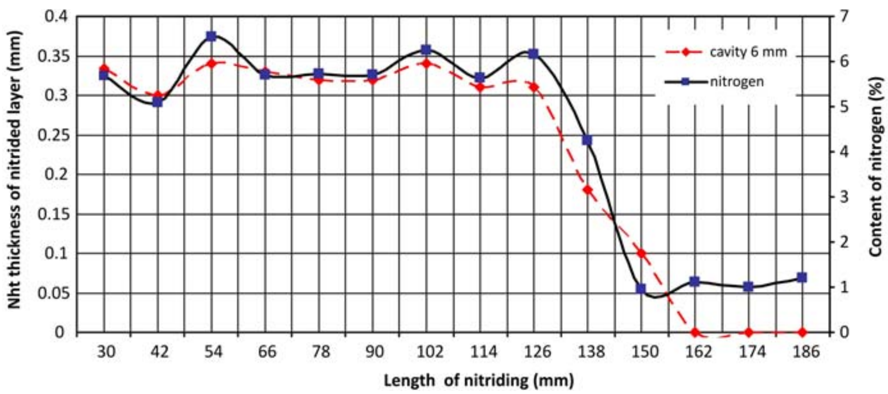

Fig. 5. Thickness of nitrided layer and nitrogen concentration.

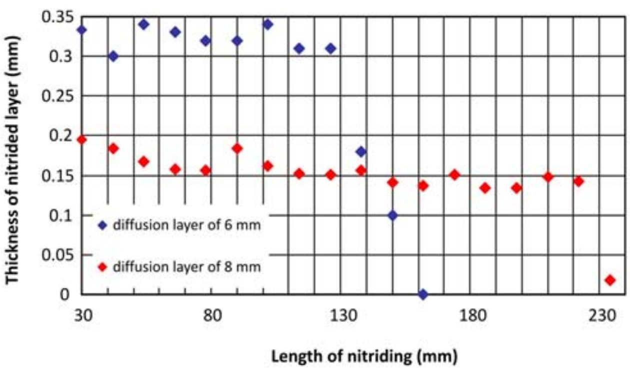

Fig. 6. Comparison of the course of microhardness in cavities with diameter of 6 and 8 mm.

ities, too. These created nitrided layers improve corrosion resistance and mechanical properties of steel. It has been proven that the diameter of cavity has remarkable influence on the plasma nitriding on the length in accordance with Fig. 6. Peaks which were established during plasma nitriding process (Fig. 6) have a connection with the plasma density and concentration of nitrogen. Figure 5 shows that concentration of nitrogen has significant influence to the thickness of nitrided layers. Diffusion layer and nitrogen concentration are interdependent. Created plasma nitrided layer improves mechanical properties.

Acknowledgements

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