



## Plasma Nitriding of Bored Barrels

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The manuscript was received on 5 November 2010 and was accepted after revision for publication on 15 February 2011.

### **Abstract:**

*The nitriding behaviour of heat-treated alloyed steel was carried out on two samples with various diameters. The influence of nitriding conditions on the thickness and hardness of nitrided layer were investigated. Bored cylindrical barrels were quenched and subsequently tempered. Plasma nitriding was performed at 500°C for 6 hours with mixture of H<sub>2</sub> and N<sub>2</sub> in plasma nitriding appliance. The pressure was changed to 4 and 6 mbar. Microstructure and mechanical properties of nitrided layers were characterized using optical spectrometry, laser microscopy, hardness testing. The nitrided depth was also estimated using cross-sectional microhardness profiles. The results of experiments showed that plasma nitriding process is applicable to cavities. Surface hardness of material was significantly increased. The measurements have shown that the pressure of plasma nitriding process has a remarkable influence on the depth of nitriding in cavities, flat surfaces and on the length of nitriding inside cavities, i.e. the distance from muzzle.*

### **Keywords:**

*Plasma nitriding, nitrided layer, universal hardness, length of nitriding*

### **1. Introduction**

The alloy steels have been generally used in weapon industry for their high hardness and strength. Low corrosion resistance sometimes limits their industrial application. Plasma nitriding process is suitable for surface treatment of barrels of small-bored weapons due to two advantages: improvement of mechanical properties and corrosion resistance. Properties of plasma nitriding layers are not dependent only on parameters of nitriding, such as duration, temperature, pressure, voltage and nitrogen potential, but they are also dependent on nitride-formed elements [1-4]. The aim of the present investigation was to study the creation of nitrided layers inside the small-bored barrels

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with the diameter of 8 mm. This study deals with mechanical properties of nitriding layers on the surface of flat samples and inside the cavities [4, 5]. The other task was to study the length of nitriding in cavity, i.e. the distance from the muzzle of barrel as indicated in Fig. 1.

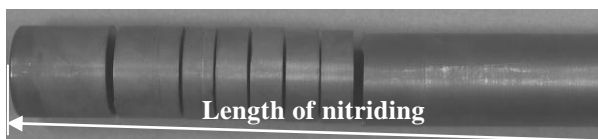


Fig. 1 Image of cut off sample

Chemical composition of steel was verified for selected chemical elements by GDOES/Bulk and QDP methods on LECO SA 2000 spectrometer [6]. Microstructure was evaluated by laser confocal microscope

Olympus OLS 3000. Thickness and microhardness of plasma nitrided layers were measured by microhardness testing method in accordance with standard DIN 50190 [7] on automatic microhardness tester LECO LM 247 AT. Universal hardness was measured at 100 N on Zwick universal hardness tester Zwick ZHU 2.5 [8]. Finally, layers created in two different nitriding environments were compared.

## 2. Experimental

Bars of 32CrMoV12-10 steel in untreated state were bored with diameters of 8 mm. Samples of the length of 500 mm were heat treated in accordance with Tab. 1.

The microhardness of treated material samples was 600 HV 0.05. Plasma nitriding was applied in PN 60/60 RÜBIG furnace according to Tab. 2. The charge consisted of 2 cylindrical small-bored barrels which were plasma nitrided at the pressure of 4 and 6 mbar for 6 hours.

Tab. 1 Temperatures of heat-treated steels

Procedure	Temperature [°C]
Oil quenching	940
Salt tempering	650

Tab. 2 Parameters of plasma nitriding process

Temperature [°C]	500
Duration [h]	6
Gas flow H <sub>2</sub> /N <sub>2</sub> [l.min <sup>-1</sup> ]	24/8
Bias [V]	530
Pressure [mbar]	4, 6
Pulse length [μs]	100

Tab. 3 Chemical composition of 32rMoV12-10 steel

C	Mn	Si	Cr	Mo	V	P	S
GDOES/Bulk							
0.30	0,47	0.25	2.95	0.89	0.28	0.002	0.001
DIN standard							
0.30	<	<	2.80	0.80	0.25	<	<
0.35	0.60	0.35	3.20	1.20	0.35	0.025	0.010

After plasma nitriding process, the barrels with the diameters of 8 mm were cut into small samples. The length of the first sample was 30 mm, each other had 12 mm. The lengths of the samples were following: 30, 42, 54, 66, 78, 90, 102, 114, 126, 138, 150, 162, 174, 186, 198, 210, 222, 234, 246, and 258 mm. All samples were wet ground using silicon carbide paper with grit from 80 down to 2000 and subsequently polished.

Confocal laser microscope LEXT OLS 3000 with outstanding resolution of 0.12  $\mu\text{m}$  and magnification range from 120 $\times$  to 12400 $\times$  was used for observation and cross-structure documentation and compound layer evaluation (Fig. 2). 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 [7]. The major Vickers microhardness numbers were derived from five measurements as an average value according to Fig. 3.

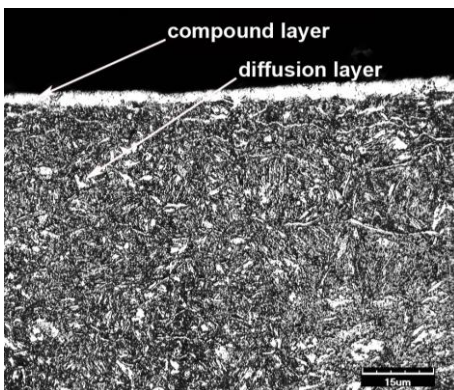


Fig. 2 The chemically etched confocal cross-sectional structure of tempered steel

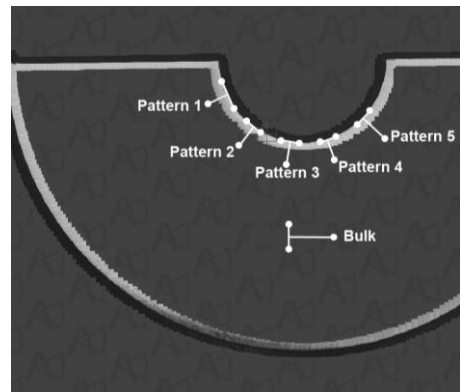


Fig. 3 Real image of measured sample from LM 247

On the base of microhardness measurements in the defined distances from muzzle the limited thicknesses (Nht thickness) were determined (Tab. 4). These Nht thicknesses show the thicknesses of created nitrided layers in the defined distances from muzzle (length of nitriding) and the results are displayed in Figs 4a, b.

The following equation was used to calculate of Nht thickness  $X(1)$  in accordance with DIN 50190 standard:

$$X = [(Y \cdot 0.1) \cdot 10] + 50 \quad (1)$$

where  $X$  is Nht thickness in mm,  $Y$  is the average microhardness number from five indentation's patterns in HV 0.05.

Instrumental hardness measurements were carried out on a Zwick universal testing machine with a hardness measurement head (Zwick ZHU 2.5) [8]. The initial head speed approaching the sample was 300 mm/min. After the head touched the sample, the approach speed of the diamond indenter until initial contact with the sample was 50 mm/min. Indentations were made on the surface of sample. The tests were carried out at 100 N indenter loads. Working test force has been maintained for 12 s. The result values of universal hardness were calculated as an average value of 5 measurements.

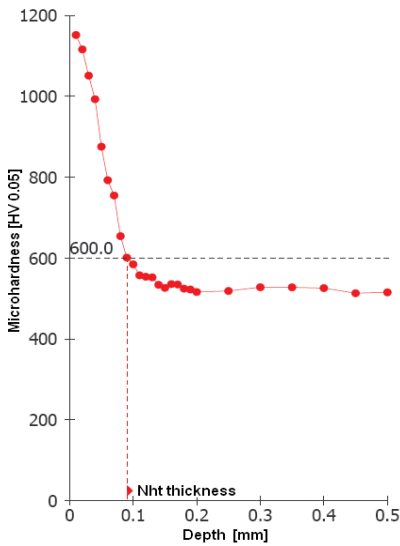


Fig. 4a Microhardness depth profile; measured in distance 66 mm from muzzle; plasma nitriding process 500 °C/6h/4mbar

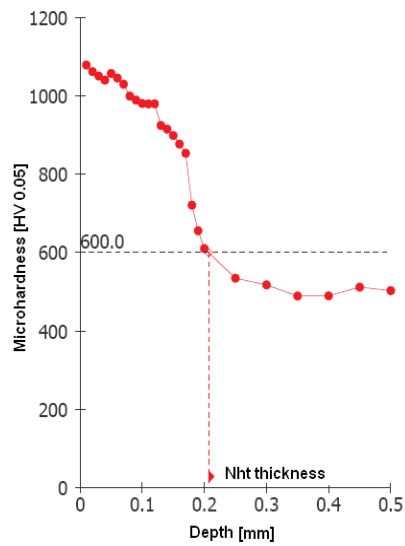


Fig. 4b Microhardness depth profile; measured in distance 66 mm from muzzle; plasma nitriding process 500 °C/6h/6mbar

The following equation was used to calculate of Nht thickness  $X(1)$  in accordance with DIN 50190 standard:

$$X = [(Y \cdot 0.1) \cdot 10] + 50 \quad (1)$$

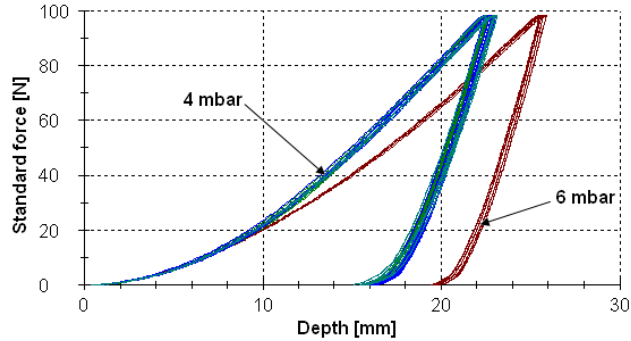
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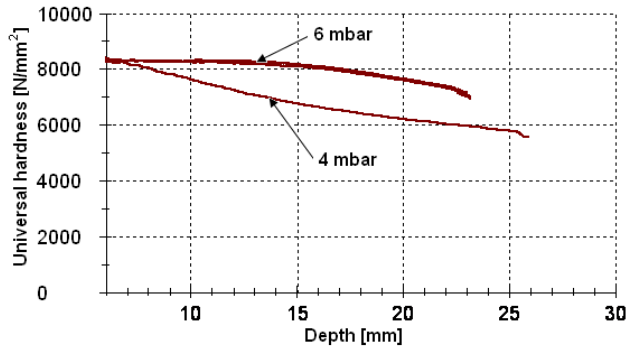
The universal hardness was evaluated automatically by software (TestXpert®) and was expressed as volume hardness. Universal hardness was measured on samples nitrided in pressure 4 mbar and 6 mbar and the curves are displayed in Figs 5a, b. Vickers indenter was used for indentation test. Following equation was used in calculating universal hardness  $HM$  (2)

$$HM = \frac{F_2}{f_{II} h_2} \quad (2)$$

where  $HM$  is universal hardness in  $N/mm^2$ ,  $F_2$  is the force at the point of load application after dwell time in N,  $f_{IT}$  is factor for indenter (26.43 for Vickers),  $h_2$  is indentation depth at the maximum force in mm [7].



*Fig. 5a Standard force on depth of nitrided layer; measured on reference samples; plasma nitriding process 500°C/6h/4mbar and 500°C/6h/4mbar*



*Fig. 5b Universal hardness; dependence of universal hardness (2) on depth of nitrided layer; measured on reference samples; plasma nitriding process 500°C/6h/4mbar and 500°C/6h/4mbar*

Chemical composition of material was measured by GDOES/Bulk method (Tab. 3). The depth profiles were evaluated by GDOES/QDP method and are shown in Fig. 6. 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.

### 3. Results

After plasma nitriding the surface hardness of plasma nitrided layers increased from 500 to 1150 HV 0.05 which is shown in Figs. 4 a, b. Microhardness depth and Nht thickness of nitrided layers were measured in accordance with DIN 50190 standard

(Eq. 1) [7]. Measurements of microhardness have shown that the pressure of plasma nitriding process has remarkable influence on the surface of materials. The pressure of 6 mbar caused an increasing of Nht thickness about 0.1 mm (Figs 4a, b). Also GDOES depth profiles and universal hardness confirmed these results (Figs 5a, b, 6). Especially in Figs 5a, b the change of hardness of nitrided layers is visible which is represented by higher depth of indentation.

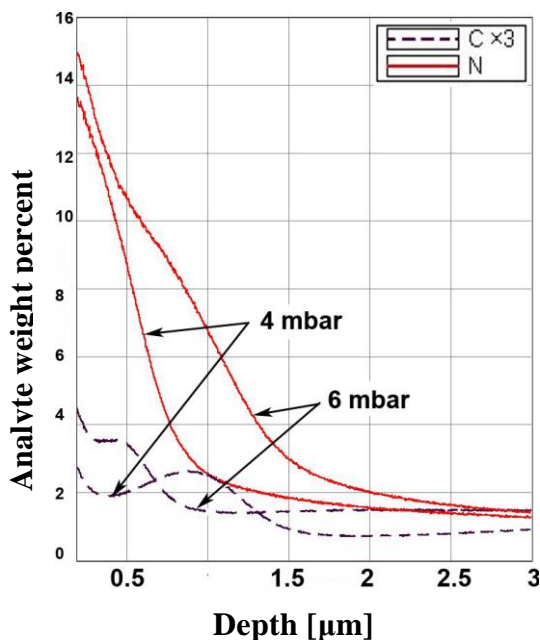


Fig. 6 GDOES depth profile; measured on reference sample; comparing btw plasma nitriding process 500°C/6h/4mbar and 500°C/6h/6mbar

Tab. 4 Results of Nht thicknesses; values are counted from microhardness measurements

Length [mm]	Cavities with diameter of 8 mm	
	pressure 4 mbar	pressure 6 mbar
	Nht thickness [mm]	
30	0,10	0,27
42	0,10	0,25
90	0,08	0,17
102	0,08	0,16
114	0,07	0,16
150	0,06	0,14
198	0,06	0,11
210	0,05	0,10
222	0,04	0,10
234	0,02	0,09
246	0,04	0,08
258	0,05	0,04

In the sample with the diameter of 8 mm which was nitrided at the pressure of 6 mbar the Nht thickness attained 0.27 mm in the distance of 30 mm from muzzle, in case of cavity nitrided at 4 mbar the value of Nht thickness was 0.10 mm. The significant part of results is shown in Tab. 4. In case of both cavities with the diameter of 8 mm, the nitrided layer was presented along all the length of cavity. The values of Nht thickness decreased to 0.04 mm in the length of 246 and 258 mm and then increased back to higher values because all samples were nitrided from both sides. It shows that plasma nitriding process is not symmetric from both sides.

#### 4. Conclusion

After plasma nitriding process the nitrided layers were created. These layers consisted of compound and diffusion layers (Fig. 2).

The hardness and microhardness of created nitrided layer were researched and compared (Tab. 4, Fig. 7). Experiments have shown that plasma nitriding process increased the surface hardness about 100%. Results are displayed in Figs 4a, b. The length of nitriding (distance from muzzle) was 234 mm at the pressure of 4 mbar and 258 mm at the pressure of 6 mbar (Fig. 7). Nht thickness of nitrided layer was higher in case of pressure 6 mbar (Fig. 7).

Results of the experiment have shown that the pressure of nitriding process changed the length of nitriding (distance of nitriding layer inside cavity from muzzle). It is clear that pressure has remarkable influence not only on the depth of nitriding layer, but also on the length of nitriding (Fig. 7).

Experiment has shown that plasma nitriding process is applicable not only for flat surfaces but for cavities, too.

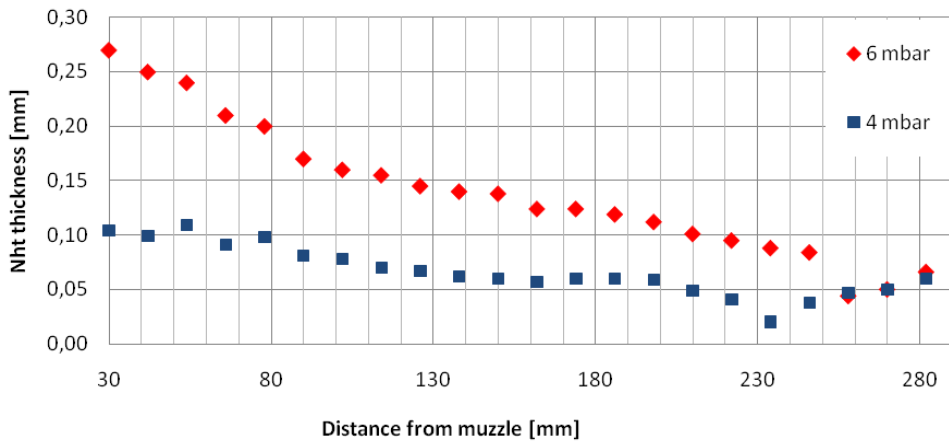


Fig. 7 Course of microhardness in cavities with diameter of 8 mm at various pressure

### Acknowledgements

The work was supported by a research project of the Ministry of Defence of the Czech Republic, project No. MO0FVT 0000404 and by Specific research of K-216.

### References.

- [1] PYE, D. *Practical nitriding and ferritic nitrocarburizing*. Ohio : ASM International Materials Park, 2003. 256 p.
- [2] JOSKA, Z. et al. Characteristics of Duplex Coating on Austenitic Stainless Steel. *Key Engineering Materials*, 2011, vol. 465, no. 1, p. 255-258.
- [3] KADLEC, J. and DVORAK, M. Duplex surface treatment of stainless steel X12CrNi 18 8. *Strength of Materials*, 2008, vol. 40, p. 118-121.
- [4] POKORNÝ, Z., HRUBÝ, V. and KUSMIČ, D. Plasma nitriding of barrel boring of small-bore arms (in Czech). *Hutnické listy*, 2010, vol. LXIII, No. 2, p. 46-50.

- [5] POKORNÝ, Z. and HRUBÝ, V. Plasma nitriding of deep narrow cavities. *Key Engineering of Materials*, 2011, vol. 465, no.1, p. 267-270.
- [6] JELINEK, M. et al. Gradient titanium-carbon layers grown by pulsed laser deposition combined with magnetron sputtering. *Laser Physics*, 2003, vol. 10, p. 1330-1333.
- [7] DIN 50190-4:1999 *Hardness depth of heat-treated parts - Part 4: Determination of the diffusion hardening depth and the diffusion depth.*
- [8] ISO 14577-4:2007 *Metallic materials – Instrumented indentation test for hardness and materials parameters – Part 1: Test method.*