PLASMA NITRIDING OF DIN 16MnCr5 STEEL

PILCH Ondrej, KUSMIC David, DOAN Thanh Van, LUKAC Tomas

University of Defence, Department of Mechanical Engineering, Brno, Czech Republic, EU
ondrej.pilch@unob.cz

Abstract

This paper deals with explanation of plasma nitriding fundamentals of and specific technical features of this technology. Preparation of samples and performing of plasma nitriding process is described in the experimental part. Plasma nitriding process was carried out on the DIN 16MnCr5 steel at 500 °C under pressure of 280 Pa in the nitriding atmosphere mixture of H_2 and N_2 (l / h) for 10, 20 and 30 h. The nitrided steel samples were studied using the GDOES spectrometry, optical microscopy, and hardness and microhardness testing. Measurements of nitride layers properties of selected steel were evaluated using the experimental methods in accordance with standards. The influence of diffusivity and of plasma nitriding conditions on the nitride layers depth was comprehensively assessed. Relation between plasma nitriding duration and depth of nitride layer was found and explained. The measurements have shown that the plasma nitriding duration has a significant influence on the nitride layer depth.

Keywords: Plasma nitriding, microhardness, nitrided layer, nitriding duration

1. INTRODUCTION

The surfaces of steel components are subjected to a wide variety of stresses, such as wear, rolling and bending stresses. In such cases, it suitable to increase the resistance to above mentioned stresses acting on the surface of the component by the way of appropriate hardening. Nitriding is hardening process with particularly low distortion. In contrast to flame, induction and case hardening, where the hardness increase is achieved by microstructural transformation of the steel, low treatment temperatures are sufficient for nitriding, mostly in the range of 490 to 530 °C in technical praxis [1].

The aim of nitriding is to form a diffusion layer (precipitation layer) with a material-dependent nitriding depth of up to 0.9 mm. The precondition for successful nitriding process is using of suitable alloyed steel containing nitride-forming elements (Al, Cr, V, Mo, Mn). Creation of nitrides in the nitrided steels compressive stresses are generated in the formed nitride layer. The increased surface hardness is dependent not only on the nitriding temperature and the amount of nitrogen available, but particularly also on the quantity of nitride-forming elements and the strength of the components in and tempered state [2].

The increased content of these elements, the higher surface hardness after nitriding is generated. Aluminium is a particularly effective element when it comes to increasing surface hardness by nitriding. Surface hardness of approx. 1000 HV can be achieved in a steel containing of 1 % Al. Steel containing roughly of 1% Cr attains surface hardness about 600 HV. In order to obtain a surface hardness of approx. 1000 HV in Cr-alloyed steels, the chromium content has to be increased up to approx. 5 %. The recommended carbon content of nitrided steels should not exceed 0.5 %C, for carbides forming suppression and supporting nitrides forming of alloying elements and iron with nitrogen [3].

2. EXPERIMENTAL PART

DIN 16MnCr5 steel was selected for this experiment. DIN 16MnCr5 steel is a kind of alloyed structural steel and gear steel, under DIN and EN 10084-1998 standard. This steel has a good hardenability and machinability.

DIN 16MnCr5 steel was widely used for higher stressed components production, such as gears, shafts, crankshafts, connecting rods, cam shafts, etc. [4].

For the experimental purpose was round steel bar used with diameter of 40 mm and 200 mm length. The 10 disc samples were prepared with thickness of 8mm and subsequently heat treated. The parameters of heat treatment are summarized in **Table 1.**

Table 1 Heat treatment parameters of DIN 16MnCr5 steel

Steel	Water quenching	Water tempering
	Temperature (°C)	Temperature (°C)
DIN 16MnCr5	860	570

Following the heat treatment the microhardness measuring using the Vickers method was performed on the automatic microhardness tester LM 247 AT LECO [5, 6]. The Vickers microhardness method consists of 5 measurements, which were used to calculate the average value [7]. The chemical composition of steel was verified by GDOES method on GDOES LECO analyzer SA 2000. The results of the BULK mode measurements, which is designed for the volumetric chemical composition analysis of materials, are displayed in **Table 2** and **3** [8].

Table 2 DIN 16MnCr5 steel chemical composition [in wt. %]

C	Mn	Si	Cr	Ni	Al	P	S
GDOES/BULK							
0.20	1.29	0.34	0.89	0.02	0.04	0.021	0.021
Standard DIN							
0.14 - 0.19	1.10 - 1.30	max 0.40	0.80 - 1.10	-	-	max 0.035	max 0.035

Method: GDOES / Bulk; Device: SA 2000 Leco; Calibration Standards CKD 180A to 189A;
Reported are the average of 10 measurements.

For creation of nitride layers on heat treated steel samples was the plasma nitriding technology [4]. The plasma nitriding process was carried out on the DIN 16MnCr5 steel at 500 °C under pressure of 280 Pa in the nitriding atmosphere of H_2 and N_2 (l / h) for 10, 20 and 30 h. The basic plasma nitriding parameters are displayed in **Table 3**. Plasma nitriding treatment was performed by RÜBIG PN 70/120 device. For plasma nitrided sample see **Figure 1**.

Figure 1 Plasma nitrided DIN 16MnCr5 steel sample

Table 3 Parameters of plasma nitriding process

Temperature (°C)	Nitriding duration (h)	Gas flow H_2/N_2 ($l \cdot min^{-1}$)	Voltage (V)	Pressure (Pa)	Pulse length (μs)
500	10/ 20/ 30	24/8	520	280	100

The nitride layer concentration profile was evaluated using GDOES Leco SA 2000 device in the mode of QDP (Quantitative Depth Profiling) [10]. The depth of one measurement was set to 20 µm. The results of the measurement are shown as the concentration dependence of C and N (in wt. %) on depth (see **Figure 2**). Calibration nitrogen standards used for measurement: JK41-1N and NSC4A [7, 8].

The microstructure formed after heat treatment and subsequent performed plasma nitriding process was analysed and documented using the optical microscope OLYMPUS GX51. The microstructure of steel was reviewed using the magnification of 200x and 500x (see **Figure 3**).

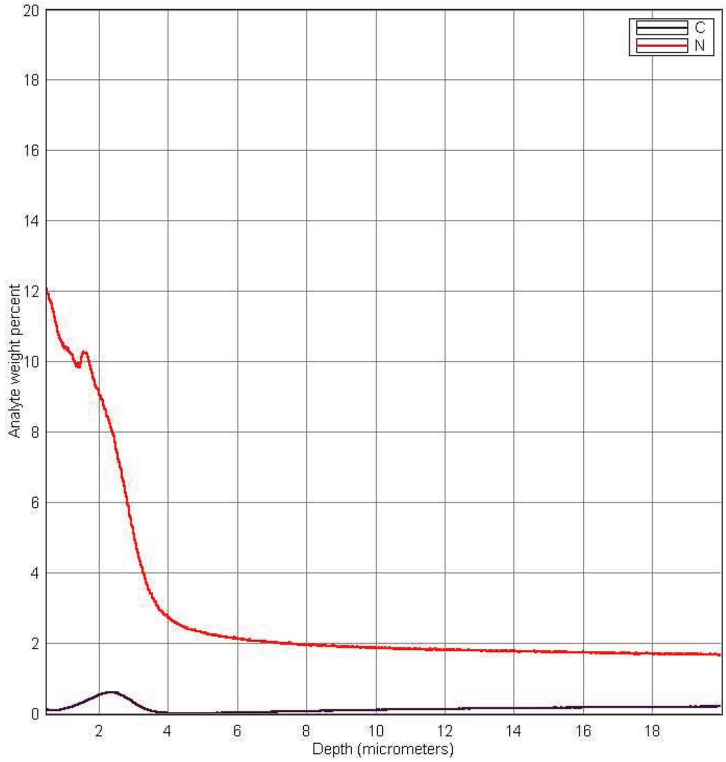

Figure 2 The concentration profile plasma nitride DIN 16MnCr5 Steel (PN 5 h)

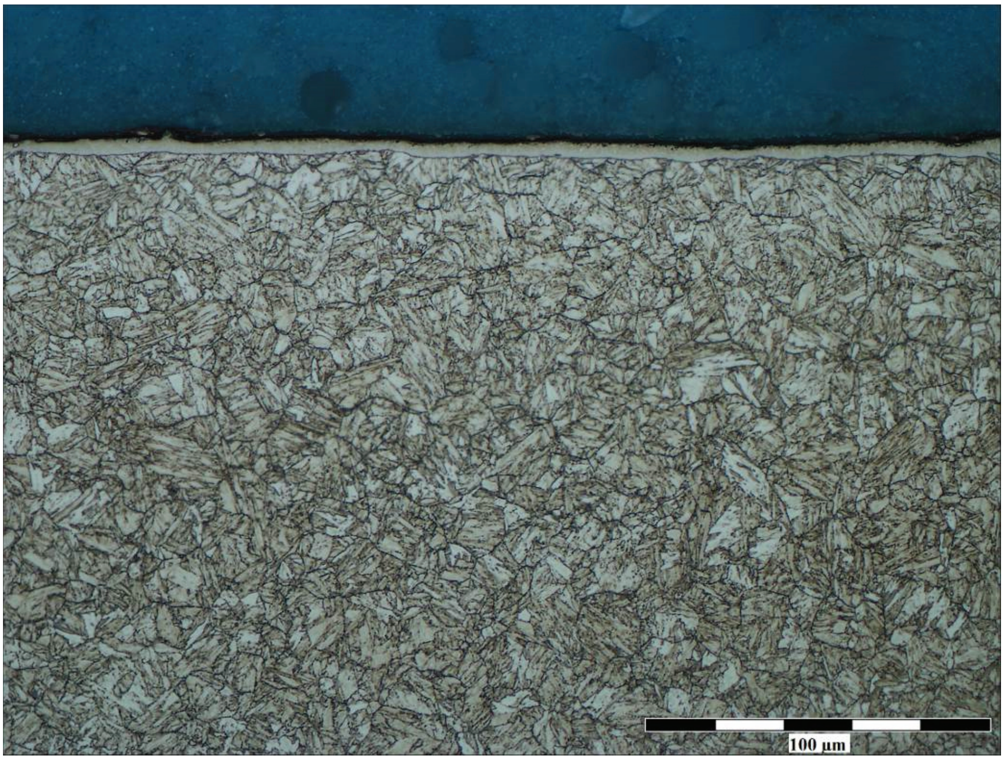

Figure 3 Microstructure of plasma nitrided sample; PN 500 °C/30 h/280 Pa

The diffusion layer microhardness and nitride layer thickness was evaluated by means of the automatic microhardness tester LM 247 AT LECO equipped by AMH43 software. The load for the test was set at 50 g and 10 s dwell time [9]. The calculation of Nht thickness was determined in accordance with DIN 50190 standard. Individual microhardness depth profiles are presented in **Figure 4**. The microhardness profiles are plotted in **Figure 4** represents microhardness of diffusion layers. All measurements were performed under the same conditions. Microhardness measuring was performed on each of samples. The results of all microhardness measurements are summarized in **Table 4** and **Table 5** [5, 6].

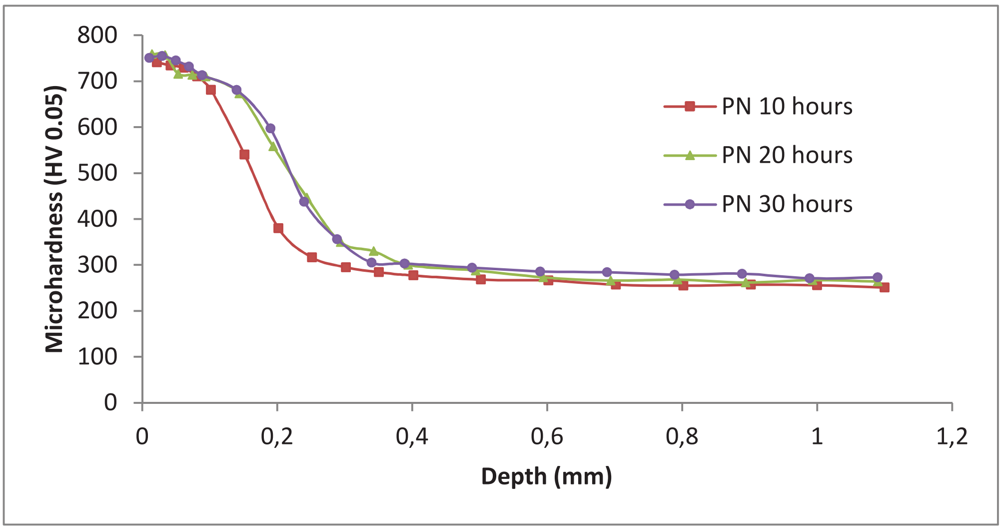

Figure 4 The microhardness profiles of plasma nitrided DIN 16MnCr5 steel

Table 4 Results of microhardness depth profiles

DIN 16MnCr5 steel			
Depth (mm)	Microhardness (HV 0.05)		
	10 h	20 h	30 h
0.01	741	759	751
0.03	734	757	755
0.05	729	716	745
0.07	710	714	732
0.09	681	710	713
0.15	540	673	681
0.20	380	558	597
0.25	316	447	438
0.30	295	350	356
0.35	284	330	305
0.40	277	300	303
0.50	268	288	294
0.60	266	273	286
0.70	257	266	284
0.80	255	268	279
0.90	257	262	281
1.00	256	267	271
1.10	251	264	273

Table 5 Nitrided layer thickness summarization

	DIN 16MnCr5 steel		
Plasma nitriding duration (h)	**10**	**20**	**30**
Nht thickness (mm)	0.265	0.360	0.325
Compound layer thickness (µm)	5.78	4.05	6.94

The surface hardness of plasma nitride layer was evaluated using the automatic microhardness tester LM 247 AT LECO equipped by AMH43 software. The test load was set at 1000 g. The won results are summarized in **Table 6** as average values of 10 measurements.

Table 6 Surface hardness after plasma nitriding

	Surface hardness (HV1)			
	Reference sample	**PN 10 h**	**PN 20 h**	**PN 30 h**
DIN 16MnCr5 steel	247	745	740	755

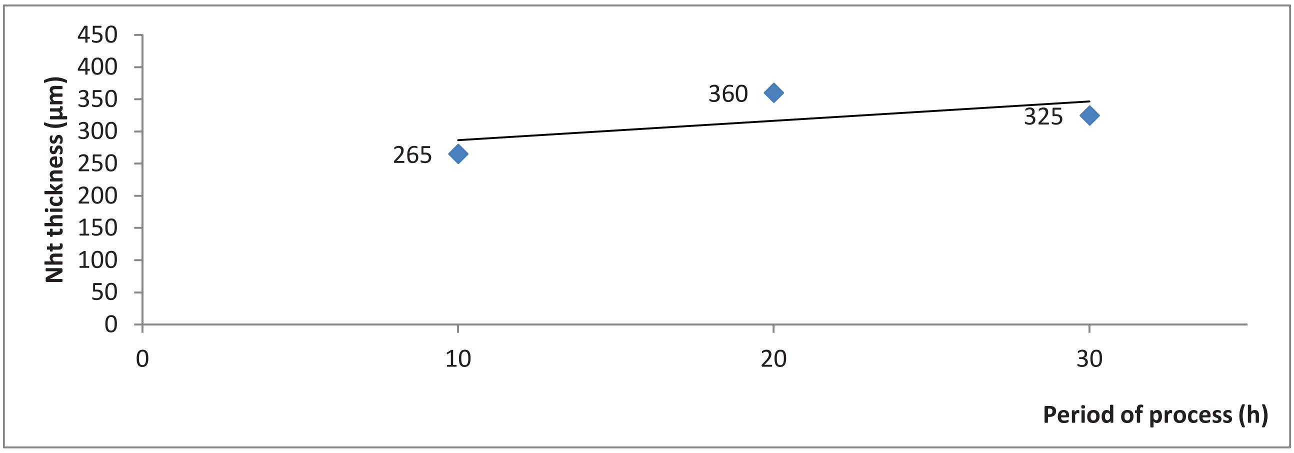

Figure 5 The thickness of nitrided layers

3. RESULTS

Experimentally was confirmed that the influence of nitriding duration increases the thickness of the nitride layer on the DIN 16MnCr5 steel. Course of diffusion layer limit thickness corresponds to the course of the white layer thickness and, at the same time, to the concentration profile of nitrogen. The surface hardness of plasma nitrided DIN 16MnCr5 steel increased from 247 HV1 to 755 HV1, values are summarized in **Table 6**. Measurements of microhardness have shown that the duration of plasma nitriding process had a significant influence on the nitride layer thickness. Analyzing the results was found that Nht thickness reached after 10 h of plasma nitriding depth of 252 µm. The nitride layer thickness was changed by altering duration of plasma nitriding process. The nitride layer thickness of 271 µm was created after 20 h of process. After 30 h of plasma nitriding process the thickness of nitride layer was increased to 324 µm. It is obvious that increasing of plasma nitriding duration increases the thickness of the nitride layer, which is shown in **Figure 5**.

4. CONCLUSION

The experiments showed that during the plasma nitriding process was a nitride layer formed on the DIN 16MnCr5 steel surface, consisted of white and diffusion layer. Three different plasma nitriding durations were applied for plasma nitriding of the DIN 16MnCr5 steel. The parameters of the nitride layers are summarized in **Table 4** and **Table 5**. After plasma nitriding, the mechanical properties of steels were improved. Following the plasma nitriding process, the microhardness increased slightly from 741 HV0.05 to 759 HV0.05 (see **Figure 4**). The experimental results showed that the duration of nitriding process changed the thickness of the nitride layer of DIN 16MnCr5 steel (**Figure 5**).

ACKNOWLEDGEMENTS

The paper has been prepared thanks to the support of the project *"The Development of Technologies, Design of Firearms, Ammunition, Instrumentation, Engineering of Materials and Military Infrastructure VÝZBROJ (DZRO K201)."* and *"Surface technology in applications special techniques SV16-216."*

REFERENCES

[1] KUSMIČ, D., DOBROCKÝ, D. Corrosion Resistance of Plasma Nitrided Structural Steels. *Manufacturing Technology*, 2015, vol. 15, no. 1, p. 64-69. ISSN 1213-2489.

[2] POKORNÝ, Z., STUDENÝ, Z., HRUBÝ, V. Effect of nitrogen on surface morphology of layers. *Metallic materials*, 2016, vol. 54, no. 2, p. 119-124. ISSN 1338-4252.

[3] POKORNÝ, Z., STUDENÝ, Z., POSPÍCHAL, M., HRUBÝ, V., JOSKA, Z. Characteristics of plasma nitrided layers. *Manufacturing Technology*, 2015, vol. 15, no. 3, p. 403-409. ISSN 1213-2489.

[4] HRUBÝ, V.: Povrchové vrstvy ocelí a jejich příprava iontovou nitridací, Brno 1991.

[5] EN ISO 6507-1 Metallic materials - Vickers hardness test - Part 1: Test method. 1998.

[6] STN EN ISO 14577-1: Metallic materials - Instrumented indentation test for hardness and materials parameters - Part 1: Test method.

[7] KADLEC, J.: *Metodika hodnocení chemickeho složení a vlastností povlaků $FeFe_2O_4$*, 2008, 18s. Univerzita obrany, Brno ISBN: 978-7231-474-4.

[8] PAYLING, R.; JONES, D.; BENGTSON, A. *Glow Discharge Optical Emission Spectrometry*. England: John Wiley & Sons Ltd. 1997. ISBN 0-471-96683-5.

[9] DIN 50190-4:1999, Hardness depth of heat-treated parts - Part 4: Determination of the diffusion hardening depth and the diffusion depth.

[10] HRUBÝ, V., POKORNÝ, Z., BARBORÁK, O. Characteristics of plasma nitrided layers in deep holes. *Metallic materials*, 2012, vol. 3, no. 50, p. 209-212. ISSN 0023-432X.