THE SURFACE MORFOLOGY INSIDE CAVITIES AFTER PLASMA NITRIDING PROCESS

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Abstract

The article deals with chemical composition and mechanical properties of plasma nitrided layers after plasma nitriding process. Experiments are focused on using of plasma nitriding process for surface treatment of cavities with diameter of 8 mm. Nitrided layers were applied to steel 32CrMoV12-10 which were subsequently evaluated by metallographic, GDOES, XRD microanalysis and microhardness methods. The results of measurement showed trends of chemical composition of alloying elements after chemical-heat treated process through the length of cavity. Plasma nitriding process is applied for increasing of surface hardness of material in deep cavities. Mechanical properties of tested material were significantly increased. Experimental work showed characteristics connection among alloying elements. Properties and thickness of nitrided layer are dependent on nitride formed elements contained in core of material. The diffusion coefficient should be addict on nitride formed elements as well.

Keywords: nitriding; chemical composition, alloying elements; nitride layer

1. INTRODUCTION

The plasma nitriding is usually applied to already heat-treated material, i.e. after heat-treatment process [1]. The aim is to achieve an enhanced surface hardness, better wear resistance, reduced friction coefficient, increase fatigue limit or corrosion resistance. The nitriding process developed nitrides of iron in the diffusion layer which caused low increase of microhardness. The main elements that caused increasing of properties are alloying elements as molybdenum, vanadium, aluminum or chrome. During plasma nitriding process, two layers can be created. On the surface of material established the compound layer consisted of ε-Fe2-3N and γ-Fe4N phase [2]. The proportion of individual phases is dependent on carbon concentration in steel [1]. This type of layer has been very hard and brittle with good friction and anticorrosion properties [2]. This layer can be very good evaluated by metallographic methods. Many experiments showed that GDOES evaluation of thickness of compound zone is not expedient. The thickness and hardness of γ'-Fe_4N (diffusion layer) depends on quantity and quality of alloying elements [3]. The composition of diffusion layers can be effectively influenced by chemical composition of nitriding atmosphere [3]. This article describes the chemical and mechanical properties of nitrided layers which were created inside the cavities. Nht thickness of mentioned sample is subsequently compared with content of alloying elements and nitrogen. This study deals with chemical and mechanical properties of nitriding layers which were created by pressure of 400 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 analyzer 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. The thickness of compound layer was measured by optical microscope OLYMPUS GX 51 equipped software program ANALYSIS.

2. MATERIAL AND METHOD

Samples of 32CrMoV12-10 steel in untreated state were cylinder bored with diameter of 8 mm. Samples of length 500 mm were heat treated in accordance with Tab. 1. The initial steel structure was evaluated by confocal laser microscope Olympus OLS 3000. The results showed that the initial structure feature of steel was assessed as homogeneous which corresponded to a low-carbon tempered martensite (Fig. 1).

Table 1 Temperatures of heat-treated steels

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

A microhardness of heat-treated steel of samples was 550 HV0.05. Plasma nitriding was carried out in PN 60/60 RÜBIG furnace according to Tab. 2. The charge was consisted of 3 cylindrical samples (cavities with diameter 8 mm) which were plasma nitrided at the pressure of 400 Pa for 6 hours.

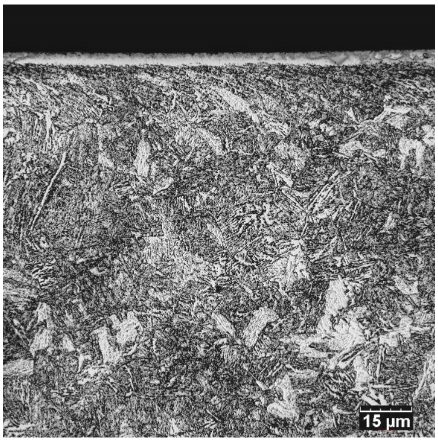


Fig. 1 The chemically etched optical cross-sectional structure of heat-treated 32CrMoV12-10 steel;

Plasma nitriding process was provided by automatized system RUBIG which allows one or two-stage process. The selection of suitable process depends on required layer properties (mechanical and corrosion). After plasma nitriding process, the samples with the diameters of 8 mm were cut. The length of the first sample was chosen greater of the safe nitriding of cavity surface over a length of 30 mm, the annular samples had a length of 12 mm. The lengths of next samples were following: 30, 42, 54, 66, 78, 90, 102, 114, 126, 138, 150, 162, 174, 186, 198, 210, 222, 234, 246, 258 mm. All samples were labelled and again axially slit into halves so that the cutting plane passed though the axis of the cavity as accurately as possible (Fig. 2).

All samples were wet grounded using silicon carbide paper with grit from 80 down to 2000 and subsequently polished. After nital etching, the confocal laser microscope LEXT OLS 3000 with outstanding resolution of 0.12 µm and magnification range from 120x to 12400x was used for observation and cross-structure documentation (Fig. 1).

Table 2 Parameters of plasma nitriding process

Temperature [°C]	500
Duration [h]	6
Gas flow H2/N2 [l.min-1]	24/8
Bias [V]	530
Pressure [Pa]	400
Pulse length [μs]	100

The thickness of created compound zone was evaluated by optical microscope Olympus GX 51 equipped software Analysis (Tab. 4). As a part of the experiments the chemical composition of material was measured by GDOES/Bulk method on reference samples (Tab. 3). 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. The microhardness was measured by Vickers microhardness method on the automatic microhardness tester LM 247 AT LECO. Load set at 50 g and 10 s dwell time. The major Vickers microhardness numbers were derived from five measurements as an average value. The overview of results is displayed in Tab. 4.

Table 3 Chemical composition of 32CrMoV12-10 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 0.35	< 0.60	< 0.35	2,80 3.20	0,80 1.20	0,25 0.35	< 0.025	< 0.010

Following equation was used for calculation of Nht thickness X (1) in accordance with DIN 50190 standard [4]:

$$X = [[Y * 0.1] *10] + 50. \qquad (1)$$

Where, X is Nht thickness in mm, Y is the average microhardness 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 analyzer PHILIPS EDAX 9900. 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 25x magnification by method of surface analysis. Results are displayed in Tab. 4, Fig. 2. The measurement of clusters is performed in Fig. 5.

Table 4 Selected results of microhardness measurements

Length [mm]	Nht thickness [mm]	Compound layer thickness [μm]	Cluster size [nm]	Chemical composition [wt.%]					
				N	Mo	V	Cr	Mn	Fe
30	0.10	1.9	118	4.06	1.37	0.49	4.12	0.85	82.52
138	0.06	1.4	97	4.33	1.30	0.24	3.29	0.56	86.78
222	0.04	0.9	90	4.23	1.35	0.32	3.32	0.56	85.84
258	0.05	1.1	103	3.49	1.15	0.36	3.40	0.70	86.66

The changes of the chemical composition of cavity surface were measured at an accelerating voltage of 20kV and exposure time 50 sec.

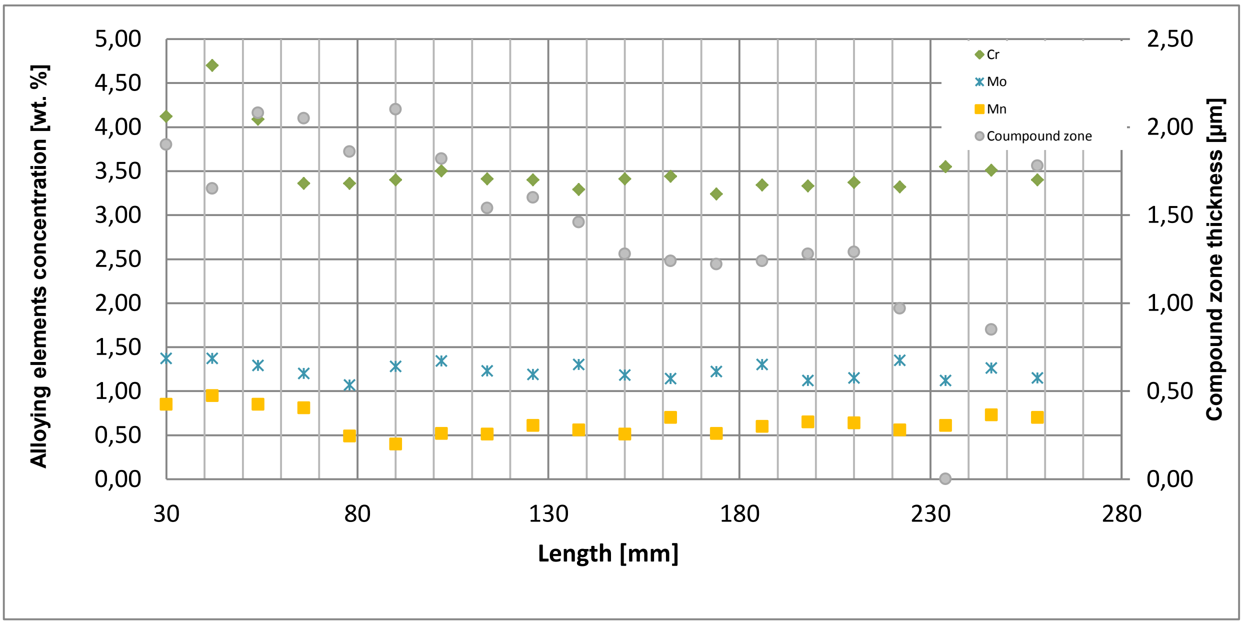


Fig 2 Chemical analysis of alloying elements

3. RESULTS AND DISCUSSION

The main effort of experimental work was aimed at analysis of chemical and mechanical properties along nitride cavities (cavity length). From previous experiments were known that diameter of cavity has remarkable influence on length of nitriding. This part of experiments is devoted to the description of effect of alloying elements to nitrogen concentration in mentioned steel after plasma nitriding. Heat-treated samples with diameter of 8 mm which were plasma nitrided at pressure of 400 Pa were investigated. Nht thickness of plasma nitrided layer was measured in accordance with DIN 50190 standard (1).

The cavity attained Nht thickness 0.10 mm in length 30 mm. The value of microhardness corresponds to the value of surface hardness. By increasing length in cavity the nitriding gradient is going down what is shown in Fig. 3. In the middle of length of cavity Nht thickness was decreased about 0.4 mm to value 0.06 mm. The last value (1) of measured microhardness was found in length 234 mm (Tab. 4, Fig. 3)

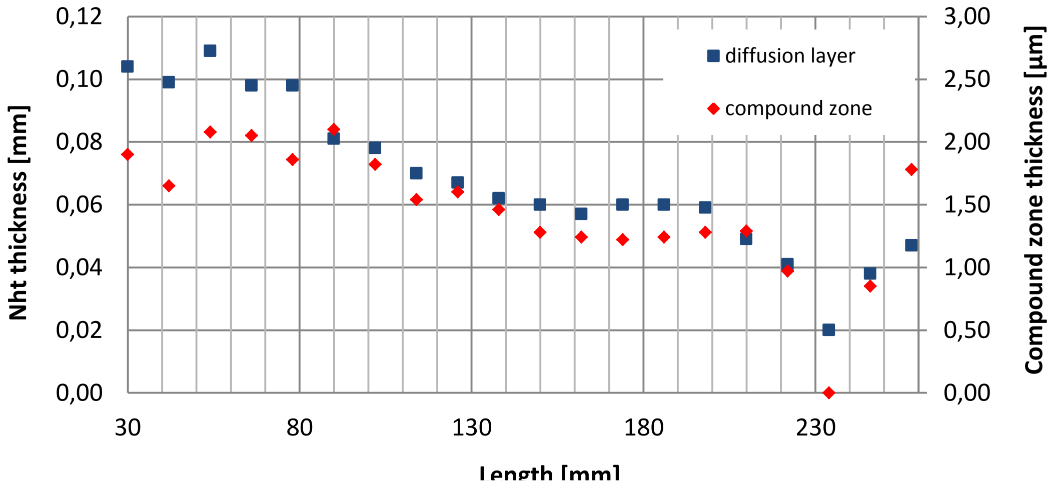
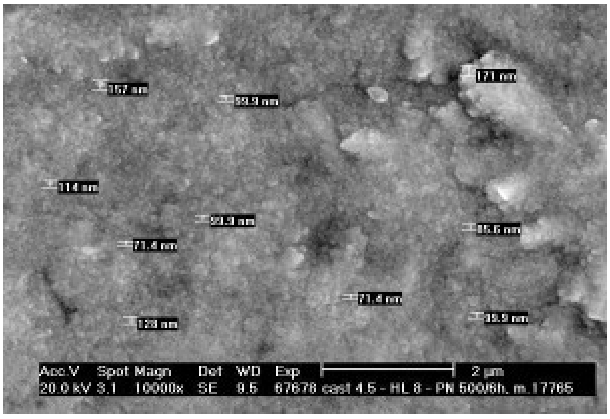


Fig 3 Thickness of nitrided layers in cavities with diameter of 8 mm



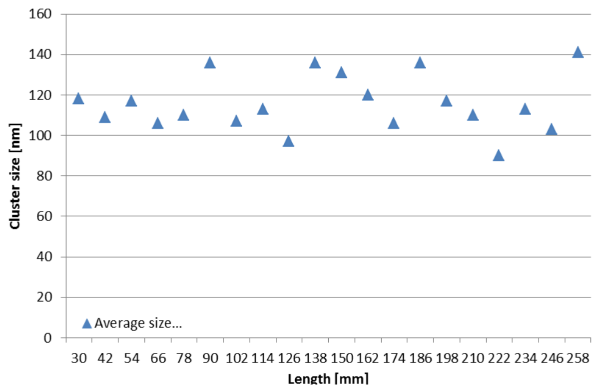


Fig 4 Clusters measurement on SEM, 10000x **Fig 5** The comparison of cluster sizes

The same progress is visible on trend of compound zone thickness. In length 30 mm was measured 1.9 µm but by increasing length in cavity, the thickness of compound zone is going down. The last value of thickness was measured in length 234 mm (Fig. 2, 3).

The results data from measurement of mechanical and chemical analysis were arranged graphically, according to the position of plasma nitrided sample surface from the cavity side. The courses of them provided the basic information about process effectivity. Mentioned effectivity of nitriding process is given on Fig. 3.

The thickness decreasing of nitrided layers in deep cavity is caused by capability of plasma nitrided process. It is technically very difficult to keep process of ionisation in the inner side of deep narrow cavity. The results show very close connection among alloying elements. Results show that many alloying elements have positive influence on plasma nitriding process. Unfortunately, many elements caused decreasing of plasma nitriding effect, what is visible in [3, 4, 5] Experimental results show very close link among concentration of chromium and nitrogen their influence on formation of nitrided layer. The surface morphology is influenced by sizes of clusters created on the surface of plasma nitrided steel (Fig. 4).

4. CONCLUSION

Influence of diameter of cavity to length of nitriding was solved in previous works [3, 5, 6]. The focus of this work was to found connection between all alloying elements, clusters created on the nitrided surface and depth of nitriding. All showed results explain mutual bond among all elements which is shown in Fig. 2, 3. Some elements as chromium, manganese and molybdenum keep hold saturation from surface to core of steel (Fig. 3). Fig. 3 confirmed the hypothesis that saturation and diffusion process is dependent on concentration and type of alloying elements in steel. On the surface of nitride material the clusters about changeable sizes were formed (Fig. 4, 5). Graphical representation was given the connectivity between diffusion of nitrogen and influence of selected alloying elements displayed in Fig. 3, 5. Influence of nitrogen to compound zone thickness is undisputed. In case of cavity with inner diameter of 8 mm close connection is visible between compound zone and diffusion layer, and nitrogen concentration and created clusters. The presented trends are equidistant (Fig. 2). Fig. 2 shows that concentration of nitrogen has significant influence on the thickness of nitrided layers their trends are equidistant as well. The main significant to surface hardness has the content of nitrogen and nitride formed elements.

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