UDC 539. 4

Duplex Surface Treatment of Stainless Steel X12CrNi 18 8

J. Kadlec[1,a] and M. Dvorak[2,b]

[1] Department of Mechanical Engineering, University of Defence in Brno, Brno, Czech Republic

[2] Department of Mechanical Technology, Faculty of Mechanical Engineering, Brno University of Technology, Brno, Czech Republic

[a] jaromir. kadlec@unob.cz, [b] dvorak.m@fme.vutbr.cz

Described technology of stainless steel duplex surface treatment is based on the plasma nitriding of the component in micropulse plasma and subsequent coating by Ni and composite Ni/diamond film. The formed duplex coating is characterized by very good mechanical properties, e.g., an excellent abrasion resistance, a low friction coefficient and a high hardness.

Keywords: plasma nitriding of stainless steel, chemical composition, structure, properties.

Experimental. Duplex coating technology is based on combination of both plasma surface treatment (ion nitriding) and subsequent deposition of thin film [1, 2]. In this case, the plasma nitrided layer was either electroplated by nickel film or electroplated by composite Ni/diamond film (Watts bath) containing codeposited diamond particles with average particle size of 0.5 μm in diameter. Thickness of electroplated films reached units of micrometers. Steel X12CrNi 18 8 (1.4300) widely used in food-processing industry and in medicine for surgical instruments was applied for experiment. Chemical composition according to DIN standard, measured by GDOES method and verified for selected chemical elements by EDS method is presented in Table 1. Plasma nitriding process was carried out on the PN 60/60 equipment according to parameters given in Table 2. For experiment two samples were used. Parameters of subsequent coating process are listed in Table 3.

T a b l e 1

Chemical Composition of Stainless Steel X12CrNi 18 8

Method	Chemical Composition (wt.%)							
	C	Mn	Si	Cr	Ni	P	S	Al
DIN standard	≤ 0.12	≤ 2.00	≤ 1.00	17–19	8–10	≤ 0.045	≤ 0.030	–
GDOES/Bulk	0.045	1.78	0.45	18.6	8.60	0.027	0.002	–
EDS	–	–	0.58	18.9	8.45	–	–	0.39

Notes: Parameters of GDOES/Bulk analysis: $U = 1002$ V, $I = 34.9$ mA, $p_{Ar} = 450$ Pa. Parameters of EDS analysis: $U = 30$ kV, $M = \times 250$, $I = 134$ pA, $WD = 21.20$ mm.

Chemical composition of substrate alloy was measured by GDOES/Bulk method (SA 2000 spectrometer) and by EDS method (Noran system Six), depth profiles was evaluated by GDOES/QDP and EDS methods. Calibration of nitrogen: JK41-1N and NSC4A standards. Microstructure and surface morphology was evaluated by electron and light microscopy (Vega TS 5135 electron microscope and Neophot 32 light microscope), respectively. Surface structure was tested by the 3D topography method (TALYSURF CLI 1000) with confocal gauge CLA before and after treatment. Layer thickness and microhardness were measured by indentation method (M400 microhardness tester). From microhardness behavior the layer depth in accordance with DIN 50 190 standard as Nht

T a b l e 2

Parameters of Plasma Nitriding Process

Parameter	Plasma cleaning	Plasma nitriding (sample 3.4)	Plasma nitriding (sample 3.2)
Temperature (℃)	520	450	550
Time/Duration (min, h)	30 min	8 h	8 h
Flow H_2 (l/min)	20	8	8
Flow N_2 (l/min)	2	32	32
Flow CH_4 (l/h)	0	1.5	1.5
Voltage (V)	800	530	530
Pulse length (μs)	100	100	100
Pressure (Pa)	80	280	280

T a b l e 3

Parameters of Subsequent Coating Process

Coating	Temperature (℃)/ Duration (min)	Current density (mA/cm^2)	Diamond particles (wt.%)
Nickel	60/5	10–15	–
Nickel/diamond	60/5	10–15	6–10

parameter was determined. Other properties (adhesion, corrosion resistance) were evaluated, too. Relations among chemical composition, structure and diffusion layer properties were briefly discussed.

Results and Discussion. Depth profiles of plasma nitrided layers (Figs. 1 and 2) for both carbon and nitrogen are in good agreement with the proposed plasma treatment regimes. Carbon and nitrogen contents decrease along the layer depth (from surface to substrate). As for carbon concentration there is local maximum ten micrometers from the surface. Existence of this maximum was verified by microstructure evaluation, too.

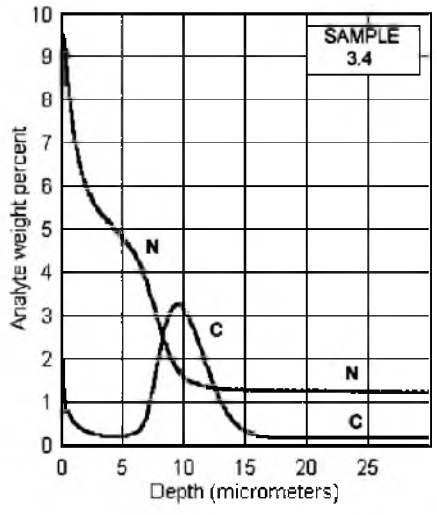

Fig. 1. Chemical composition (sample 3.4).

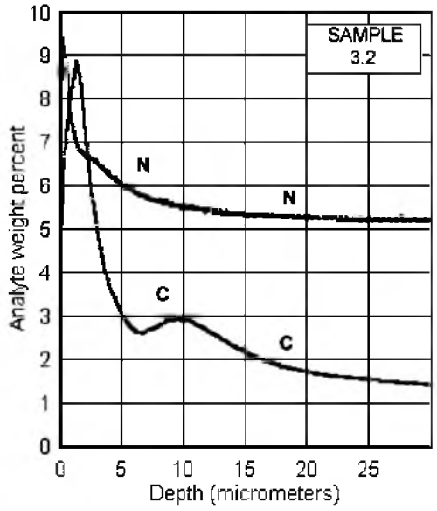

Fig. 2. Chemical composition (sample 3.2).

A very sharp interface between substrate material and plasma nitrided layer was found out (Figs. 3 and 4). Thickness/depth of plasma nitrided layer was evaluated by way of microhardness behavior measurement in compliance with DIN 50 190 standard. Attained Nht value is Nht 270 HV 0.05 = 0.08 mm (Fig. 4). This result is in conformity with spectrometric and metalographic measuring and evaluation. The highest value of measured microhardness was observed for plasma nitrided layer of sample 3.2 and reached 1578 HV 0.05.

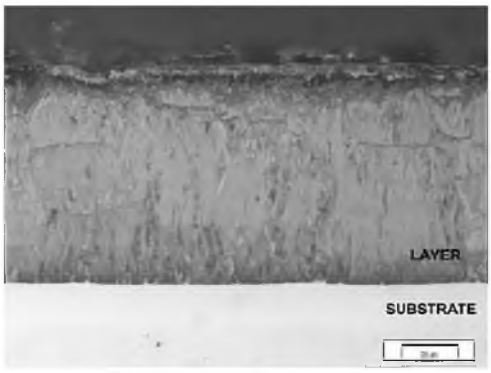

Fig. 3

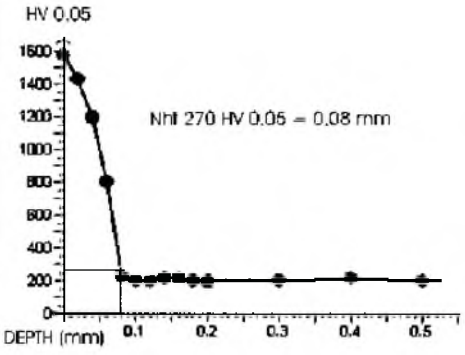

Fig. 4

Fig. 3. Microstructure of sample 3.2 (Vilella Bain).
Fig. 4. Microhardness behavior of sample 3.2 (determination of layer depth).

Results of surface morphology of Ni/diamond coating evaluation, indentation adhesion test of plasma nitrided layer (is equal to HF1–HF2) and Calotest of Ni coating, respectively, are in Figs. 5–7.

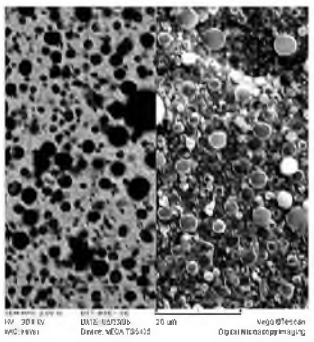

Fig. 5

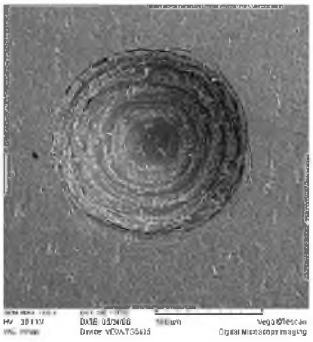

Fig. 6

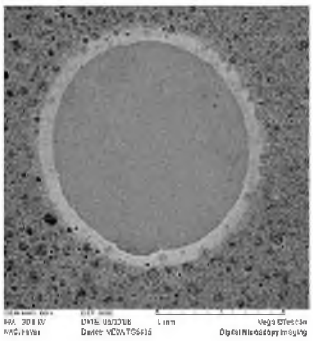

Fig. 7

Fig. 5. Surface morphology of Ni/diamond composite.
Fig. 6. Indentation adhesion test (plasma nitrided layer).
Fig. 7. Result of thickness measurement – Calotest.

Qualitative and quantitative results of 3D surface topography measurements are shown in Figs. 8 and 9. The most important parameters of surface structure are presented at the same time.

Conclusions. Plasma nitrided layer on the steel X12CrNi 18 8 surface at two different temperatures with subsequently deposited Ni based films was carried out. The focus was on the relations between chemical composition, structure and properties of the formed coatings. It follows from GDOES measurements that a variable composition depth profile can be fabricated. The highest value of microhardness 1578 HV 0.05 was observed for plasma nitrided layer (550°C/8 h). To restore surface corrosion resistance, two types of

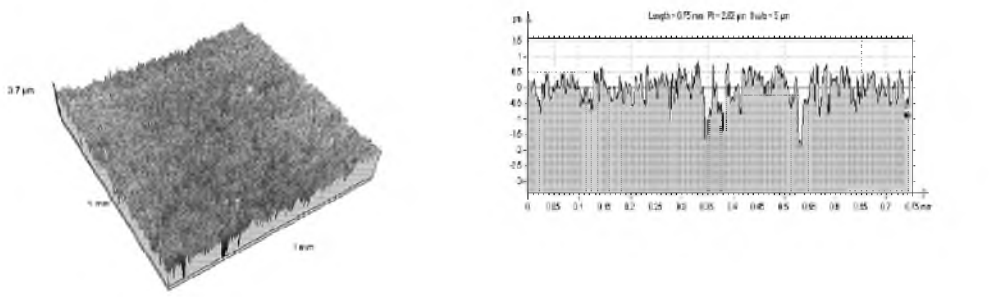

Fig. 8. 3D surface topography (sample 3.2, plasma nitriding 550℃/8 h): $P_a = 0.345$ μm; $P_q = 0.452$ μm; $P_t = 2.83$ μm; $R_a = 0.358$ μm; $R_q = 0.47$ μm; $R_t = 2.73$ μm; $W_a = 0.043$ μm; $W_q = 0.0512$ μm; $W_t = 0.168$ μm.

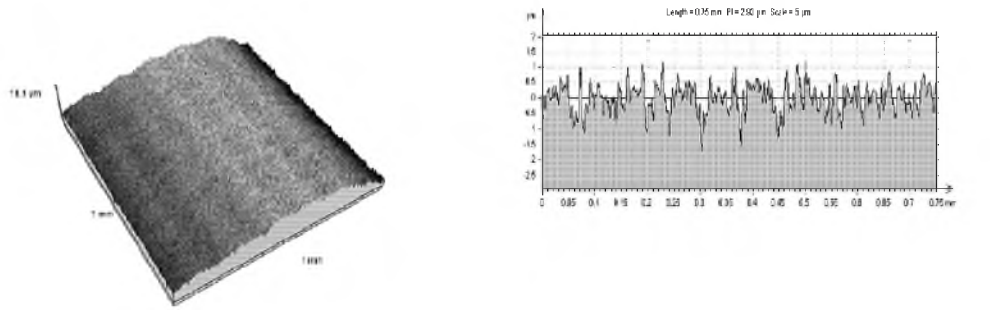

Fig. 9. 3D surface topography (sample 3.4, plasma nitriding 450℃/8 h): $P_a = 0.373$ μm; $P_q = 0.469$ μm; $P_t = 2.93$ μm; $R_a = 0.375$ μm; $R_q = 0.477$ μm; $R_t = 2.75$ μm; $W_a = 0.0265$ μm; $W_q = 0.0298$ μm; $W_t = 0.114$ μm.

highly adhesive electrolytic nickel-based thin films with thickness in units of micrometers were subsequently deposited. These thin films improve not only corrosion resistance, final surface structure and surface mechanical properties, but also perfect the appearance of treated surface.

Acknowledgements. The work was supported by research project BTU Brno BD13713313 and by Ministry of Defence of the Czech Republic, project No. FVT 0000404.

1. J. Kadlec, V. Hruby, and M. Novak, *Vacuum*, **41**, 2226 (1990).
2. W. S. Baek, S. C. Kwon, J. J. Rha, et al., *Thin Solid Films*, **429**, 174 (2003).

Received 28. 06. 2007