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PLASMA NITRIDED AND PVD TIN COATED X12CRNI 18 8 STEEL SURFACE

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ABSTRACT
The aim of the article is to describe duplex treatment of austenitic stainless steel X12CrNi 18 8 surface. Combination of both plasma nitriding and PVD coating by TiN as a surface treatment has been used to improve material hardness and wear resistance without decreasing corrosion resistance. GDOES, SEM, microhardness, indentation adhesion test were applied to characterize the chemical composition, surface morphology, adhesion and hardness of duplex treated specimens.
Keywords: stainless steel, duplex surface, adhesion, chemical composition

1. EXPERIMENTAL
Austenitic stainless steel X12CrNi 18 8, has excellent corrosion resistance, superior cryogenic properties, good high-temperature strength and adequate biocompatibility, for this properties is used in the food and chemical industry and in medicine for surgical implants. But low wear resistance and poor hardness limit their applications. Duplex surface system was applied to improve surface and subsurface properties. We used combination of both plasma nitriding and post deposited thin film TiN.
Chemical composition was measured by GDOES method, result is shown in Table.1. Plasma nitriding process was proceed on the PN 60/60 equipment according to parameters shown in Table.2 Subsequent TiN coating was deposited by PVD process main parameters are summarized in Table 3. For experiment were used 4 samples, parameters of their treatment are shown in Tab. 4.
Chemical composition of substrate material was measured by GDOES/Bulk method (SA 2000 spectrometer) depth profiles was evaluated by GDOES/QDP. 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), surface microhardness was measured by indentation method (M400 microhardness tester). Other properties like adhesion were evaluated, too. Relations among chemical composition, structure and diffusion layer properties were briefly discussed.

Table 1. Chemical composition of stainless steel

Chemical composition (%)							
	C	Mn	Cr	Ni	Si	P	S
DIN standard	≤ 0,12	≤ 2,00	17 - 19	8 – 10	≤ 1,00	≤0,045	≤0,03
GDOES/Bulk	0,045	1,78	18,6	8,60	0,45	0,027	0,002
Parameters of GDOES/Bulk analysis: U =800 V, I = 30m A, p_{Ar} = 314 Pa							

2. Results and Discussion
Depth profiles of plasma nitrided layer for carbon and nitrogen conform to the proposed plasma treatment regime. They decrease along the depth layer. There is for carbon concentration maximum 1300 s.

Table 2. Parameters of subsequent coating process

PVD TiN coating		
	TiN 1.5 µm	TiN 5 µm
Bias Voltage (V)	100	100
Substrate temperature (^{0}C)	450	450
Cathode-substrate distance (mm)	150	150
Deposition time (min, h)	80 min	4 h
Nitrogen pressure (Pa)	0,45	0,45

Table. 4 Summary of samples treatment

Sample	Plasma Nitriding	TiN Coating
1.1	XX	5 µm
1.2	XX	1,5 µm
1.7		1,5 µm
1.8		5 µm

Table 3. Parameter of plasma nitriding process

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

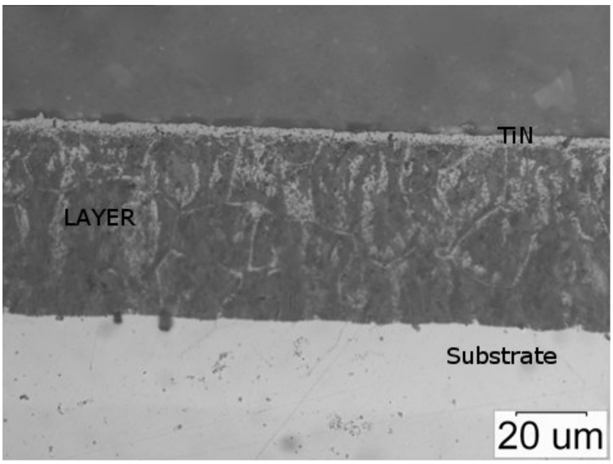

Figure 1. Microstructure of sample 1.1

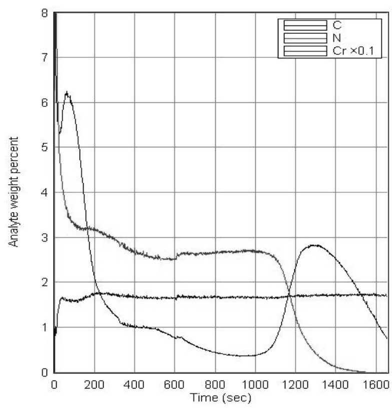

Figure 2. Chemical composition of nitrided layer

Depth profiles of TiN coatings are shown that content of nitrogen on plasma nitrided surface is higher than on the untreated surfaces. Nitrogen layer is decreased and chromium layer is increased more slowly. Adhesion was measured by Rockwell C indentation test, load was 1470 N, and matches to (HF1 – HF3) Figs. 8, 9, 10. Surface microhardness was measured in range of load 0,1N to 9,81N in Table 7. are shown results for all types of treatment. The highest values have samples with duplex surface treatment.

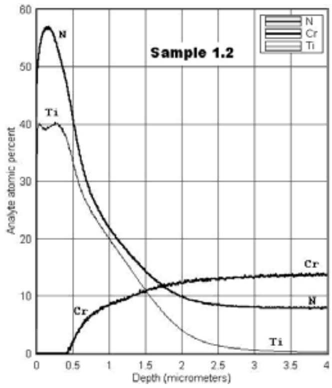

Figure 3.Chemical composition (sample 1.2) TiN 1,5 µm on plasma nitrided surface

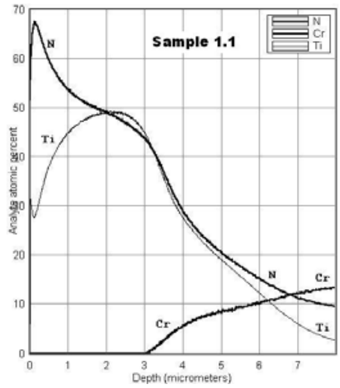

Figure 4. Chemical composition (sample 1.1) TiN 5µm on plasma nitrided surface

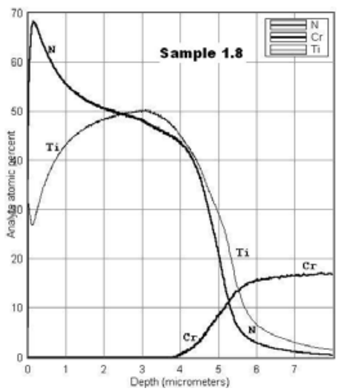

Figure 5.Chemical composition (sample 1.8) TiN 5µm on untreated surface

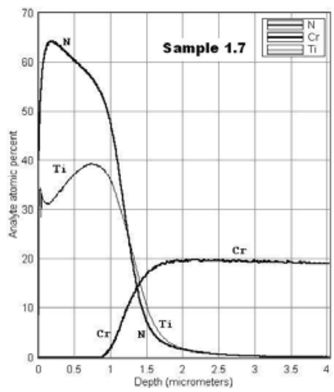

Figure 6. Chemical composition (sample 1.7) TiN 1,5µm on untreated surface

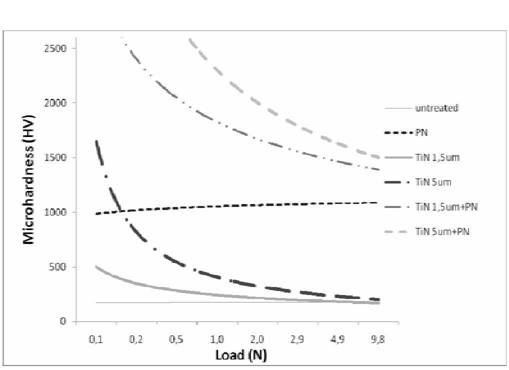

Figure 7. Surface microhardness

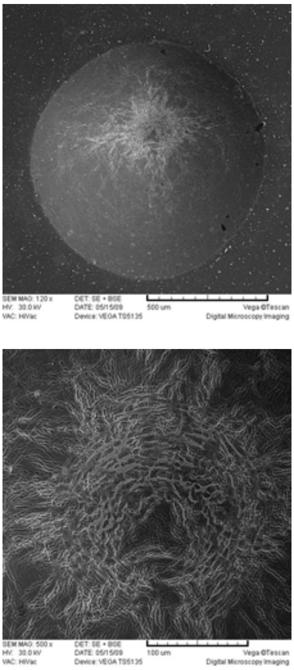

Fig. 8.Indentation adhesion test of sample 1.8 (TiN 5 um)

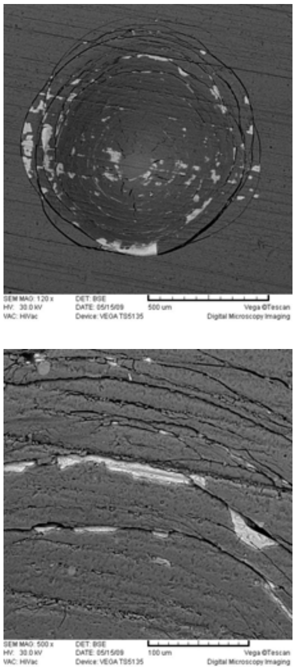

Fig.9.Indentation adhesion test of sample 1.1 (Plasma Nitrided + TiN 5um)

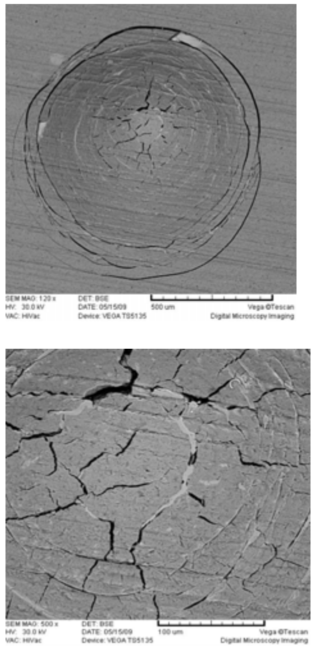

Fig. 10.Indentation adhesion test of sample 1.1 (Plasma Nitrided + TiN 1,5um)

3. CONCLUSION

Duplex surface treatment on the steel X12CrNi 18 8 surface was investigate. The aim of the research was found the relation between chemical composition, structure and properties of duplex system. This system improved the hardness differences between the coating and substrate. It leads to better wear properties of whole system. Results from indentation test shown that duplex system with 1,5µm TiN coating has better adhesion than with 5µm TiN coating. Results from demonstrated that the highest values of microhardness has duplex system of sample 1.1 (Plasma nitrided + TiN 5um).

4. ACKNOWLEDGMENTS

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