

XXII Международная конференция  
Взаимодействие ионов с поверхностью «ВИП-2015»

XXII International Conference  
Ion-Surface Interactions «ISI-2015»

РОССИЙСКАЯ АКАДЕМИЯ НАУК  
НАЦИОНАЛЬНЫЙ ИССЛЕДОВАТЕЛЬСКИЙ ЯДЕРНЫЙ УНИВЕРСИТЕТ «МИФИ»  
МОСКОВСКИЙ ГОСУДАРСТВЕННЫЙ УНИВЕРСИТЕТ  
САНКТ-ПЕТЕРБУРГСКИЙ ГОСУДАРСТВЕННЫЙ ПОЛИТЕХНИЧЕСКИЙ УНИВЕРСИТЕТ  
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ЯРОСЛАВСКИЙ ФИЛИАЛ ФИЗИКО-ТЕХНОЛОГИЧЕСКОГО ИНСТИТУТА РАН  
ИНСТИТУТ ПРОБЛЕМ ТЕХНОЛОГИИ МИКРОЭЛЕКТРОНИКИ И ОСОБОЧИСТЫХ МАТЕРИАЛОВ

# Взаимодействие ионов с поверхностью

## ВИП-2015

### Труды XXII Международной конференции

20 - 24 августа 2015 г.  
Москва, Россия

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# Ion-Surface Interactions

## ISI–2015

Proceedings  
of the XXII International Conference

August 20–24, 2015  
Moscow, Russia

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Volume 3

## ALLOYING AND NITRIDING STEELS BY COMPRESSION PLASMA FLOWS TREATMENT

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Mixing of a “coating/substrate” system by ion, electron, plasma and laser beams allows alloying the substrate material with the coating elements. This process is of special interest in investigating nonequilibrium phase diagrams or in producing nonequilibrium, immiscible and metastable compounds the existence of which in equilibrium phase diagrams is forbidden. In contrast to well-known ion-beam mixing based on energy transfer in atomic elastic collisions or on diffusion in thermal spikes, mixing with high energy fluxes can provide formation of deeper alloyed layers with the thickness of 0.1 - 100  $\mu\text{m}$ . This effect is explained by the action of an other mechanism including melting of the coating and substrate surface layers, convection mixing in the melt and subsequent crystallization under high cooling speed. The use of such a technique for steel treatment leads to formation of surface layers with improved properties. Treatment by plasma flows generated by a gas discharge allows incorporating additional atoms of a plasma-forming gas, thus expanding the number of compounds to be synthesized in the surface layer.

Earlier conducted investigations showed that compression plasma flows (CPF) generated in a gas-discharge magneto-plasma compressor could be effectively used for alloying the steel surface layer [1] as well as for metals nitriding [2]. Element and phase composition can be controlled by plasma treatment parameters of (e.g. energy absorbed by the surface layer). Investigation of the pulses number influence on the element and phase composition of the surface steel layer and its mechanical properties after CPF treatment was the main aim of this work. Ti and Nb atoms were chosen for plain carbon steel alloying in the experiments. These elements are already used for steel alloying [3]. At the same time the melting temperature of these elements ( $T_m^{\text{Ti}}=1671\text{ }^\circ\text{C}$ ,  $T_m^{\text{Nb}}=2477\text{ }^\circ\text{C}$ ;) is substantially different which can be crucial for uniform distribution of the alloying element in the melt existing for a short time ( $\sim 100\text{ }\mu\text{s}$ ) for this type of alloying technique.

The samples used were made of carbon steel 3 (0.2% C, 0.2% Si, 0.5% Mn, in wt.%, Fe - balance) and instrumental steel U9 (0.9% C, 0.25% Cu, 0.25% Ni, 0.2% Si, 0.2% Mn, 0.2%

Cr in wt.%, Fe - balance). The titanium coating on steel 3 samples and niobium coating on steel U9 samples were formed using cathodic arc vapour deposition with the following process parameters: the arc current was 100 A for titanium, 190 A for niobium, the bias voltage was -120 V, the deposition time was 10 min (the corresponding coating thickness  $\sim$  2.5  $\mu\text{m}$ ).

CPF were obtained using a gas-discharge magneto-plasma compressor of compact geometry powered with the capacitive storage of 1200 mF and operating at the initial voltage of 4 kV. Nitrogen was used as a plasma forming gas. The pressure of the plasma forming gas was 400 Pa during the discharge. The discharge duration amounted to  $\sim$  100  $\mu\text{s}$ . The treatment of the formed Ti/steel and Nb/steel systems was carried out by one, three and six pulses (n) of compression plasma flows. The time interval between pulses was 5 s. The heat flux absorbed by the surface layer (registered by calorimetric measurements) was  $\sim$ 13 J/cm<sup>2</sup> per pulse.

Structure, element and phase composition of the surface layer were characterized by the X-ray diffraction analysis (XRD), Auger electron spectroscopy (AES), scanning electron microscopy (SEM) and energy-dispersive X-ray microanalysis (EDX). Vickers microhardness and tribological tests were carried out.

The element composition analysis showed that the growth of pulses the number led to the decrease of the Ti concentration in the bulk of the mixed layer (Fig. 1). The corresponding concentration for n=1, n=3, n=6 amounted to 12 at. %, 9 at. % and 7 at. %, respectively. These data are in good agreement with the results of the EDX analysis. Similar regularity was found for Nb/steel samples. The reason for such behaviour can be erosion of the surface during plasma impact. According to the measurements of Ti/steel samples weight the mass loss was  $\sim$  1 mg/cm<sup>2</sup> per pulse. Erosion resulted in the removal of the coating surface layer during CPF impact (n=1) or in the removal of the surface layer containing the alloying element (n>1), thus diminishing the alloying element volume that could be redistributed in the melted layer.

AES spectra also contained carbon, nitrogen and oxygen peaks that appeared due to diffusion of vacuum chamber residual atmosphere elements into samples during treatment. The maximum nitrogen concentration in the surface layer was about 35-37 at. % and it was almost constant for all treated samples. The distribution of titanium was not uniform in the surface layer. The Ti distribution had a surface segregation peak (just in the layer containing light impurities). The underlying layer lacked titanium. This effect was also observed in the previous investigations [1].

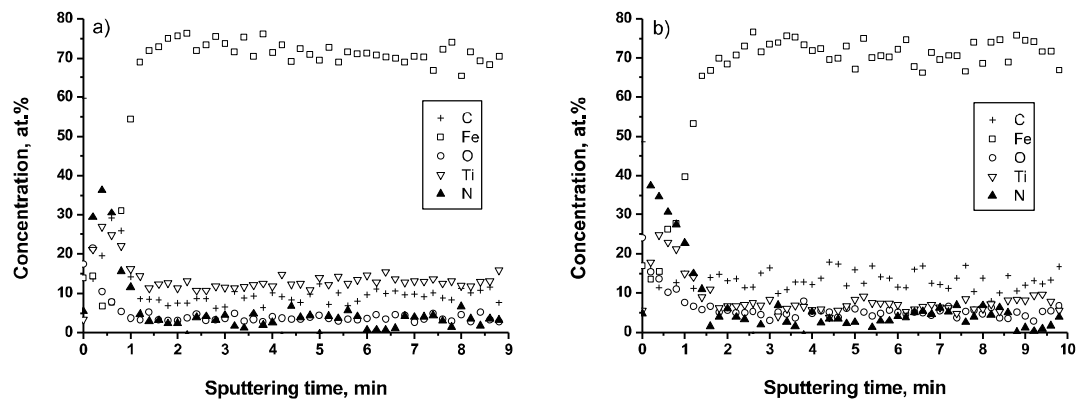


Fig. 1 AES elements depth profiles of the Ti/steel samples treated by CPF with  $n=1$  (a) and  $n=6$  (b).

Surface segregation of the alloying element took place due to the interaction between the surface atoms and the residual gases of the vacuum chamber. The formation of TiN having high crystallization temperature can occur at the surface of the melt. Further growth of the surface nitride layer during or after crystallization resulted in upward diffusion of the alloying element from the bulk and diffusion of nitrogen from residual atmosphere. The latter effect can explain formation of the titanium depleted subsurface layer.

The change of the alloying element concentration in the surface layer influences its phase composition (Fig. 2). CPF treatment with  $n=1$  resulted in the formation of  $Fe_2Ti$  phase and TiN phase with (200) preferred orientation. The XRD analysis showed a shift of  $\alpha$ -Fe diffraction lines to smaller angles with the growth of the titanium concentration. A possible reason for this effect is the formation of a supersaturated  $\alpha$ -Fe(Ti, C) solid solution where Ti atoms substitute Fe atoms in the bcc lattice. Ti solubility in  $\alpha$ -Fe does not exceed 2.9 at.% even at 773 K in equilibrium conditions. Increase of the pulses number ( $n=3$ ) resulted in the decrease of the Ti concentration thus leading to diminishing  $Fe_2Ti$  diffraction lines intensity. After CPF treatment with  $n=6$ ,  $\alpha$ -Fe(Ti, C) diffraction lines and (200)TiN line of weak intensity are seen only in the diffraction pattern. Similar regularities were found in the Nb/steel system after CPF treatment.

SEM investigations showed more homogeneous distribution of alloying elements (Ti, Nb) when the number of CPF pulses increased. Convective mass transfer time growth was the main reason for this effect. With the growth in the pulses number, the process of convective motion in the liquid is repeated, thus leading to increase in the total duration of convective motion.

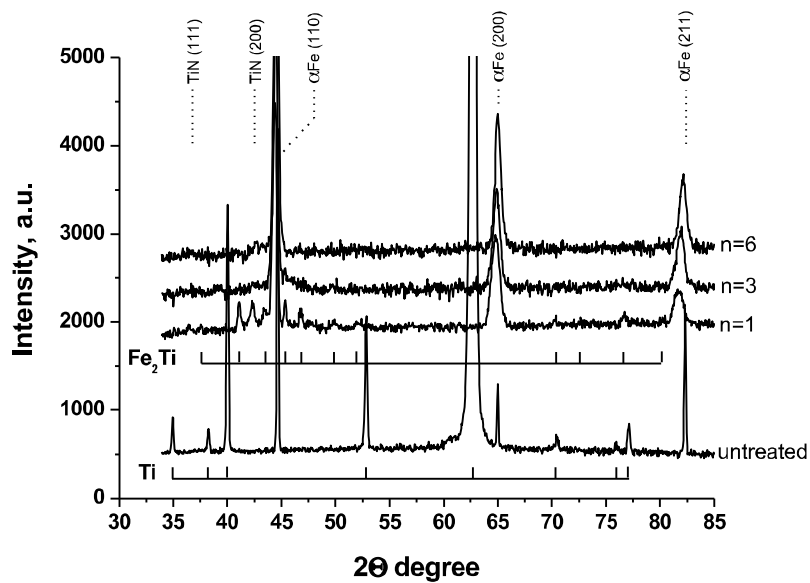


Fig. 2 XRD patterns of untreated Ti/Steel sample and samples treated by CPF with different number of pulses.

The change of phase composition and quenching effects resulted in 2-2,5 times microhardness increase and 2-3 times friction coefficient decrease.

One can see that treatment of a “coating/steel” system by compression plasma flows generated in nitrogen atmosphere allows alloy the steel surface layer with the coating element and nitrogenizing it simultaneously. The variation of the pulses number results in the formation of a range of phases in the steel surface layer, improving its mechanical properties. Such an approach allows modifying mechanical, corrosion, magnetic and other properties of the steel surface layer by an appropriate choice of alloying elements and gas discharge atmosphere.

This work was supported by the grant #3226/GF4 of Ministry of Education and Science of Kazakhstan

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