ISSN 1062-8738, Bulletin of the Russian Academy of Sciences. Physics, 2012, Vol. 76, No. 6, pp. 669–673. © Allerton Press, Inc., 2012.
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# Ion-Induced Modification of Contact Surfaces

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**Abstract**—Iron–nickel contact surfaces subjected to ion-induced modification are studied by AES, XPS and AFM. The corrosion and erosion resistance of the modified contacts is associated with features of the surface topography, and with the formation of iron and nickel nitride layers. The fine energy structure of the atomic state is revealed. Ion irradiation of the contact surfaces yields the formation of pores and cones.

**DOI:** 10.3103/S1062873812060081

## INTRODUCTION

Along with the element- and structure-sensitive techniques based on X-ray, electron, and ion microbeams that became traditional in the 20th century, the scanning probe methods developed over the last few decades (e.g., STM and AFM) that differ in their physical nature and yield data on the atomic profile of the surface layers of solids offer new possibilities for studying complex heterogeneous surfaces. The problem of variations in the atomic states of a solid surface during ion bombardment is well known and remains the focus of attention for many researchers, due to its scientific and practical significance [1–3]. In particular, it is important when investigating surfaces of hermetically sealed contacts after the action of a gas discharge plasma in nitrogen [4]. This work analyzes contact surfaces made of iron and nickel and the results of their ion–plasma modification. To accomplish this, we used a set of complementary and often backup (to increase the reliability of the data on such complex heterogeneous specimens) techniques: Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XDS), and atomic-force and optical microscopy (AFM and OM).

## EXPERIMENTAL

Contact surfaces of hermetically sealed contacts [5] prepared as in [6] served as test specimens. They were pressed from Dilaton permalloy wire (52% Ni, 48% Fe). The surfaces were degreased, annealed in a hydrogen atmosphere, and put into a hermetically sealed cylinder filled with spectroscopically pure (99.999%) nitrogen.

The ion–plasma treatment (IPT) of the contact surfaces was performed using high-voltage pulsed discharges igniting between open contacts (gap $d$ = 27–30 μm) on a special facility [7]. A single-stage IPT lasted 30 s and yielded ion nitriding [8]. Saturation of the near-surface layers with nitrogen was done at relatively high pressures ($P = 33 \times 10^3$–$40 \times 10^3$ Pa). The contact surfaces alternately served as anodes and cathodes.

The state of the modified structure was evaluated based on the results of investigations performed by means of AES, XPS, AFM, and OM. The obtained data were compared with the results from measuring certain characteristics, particularly the intermediate contact resistance. A detailed description of the parameter measurements and tests was given in [5].

The elemental and chemical composition of the contact surface (before and after treatment) was analyzed with an Auger electron spectrometer [9] and a PHI Quantera SMX scanning X-ray photoelectron microprobe. The contact surfaces were also analyzed with the help of an MMP-4 optical microscope. The pressure and composition of the gas filling the cylinder equipped with contact surfaces was controlled with a MI-1201 magnetic mass spectrometer.

The contact surface topography was studied in air using an atomic force microscope (the NTEGRA probe nanolaboratory (PNL) manufactured by NT-MDT) using silicon cantilevers of NSG10/$W_2C$ grade (with a 30 nm-thick hard $W_2C$ current-conducting coating).

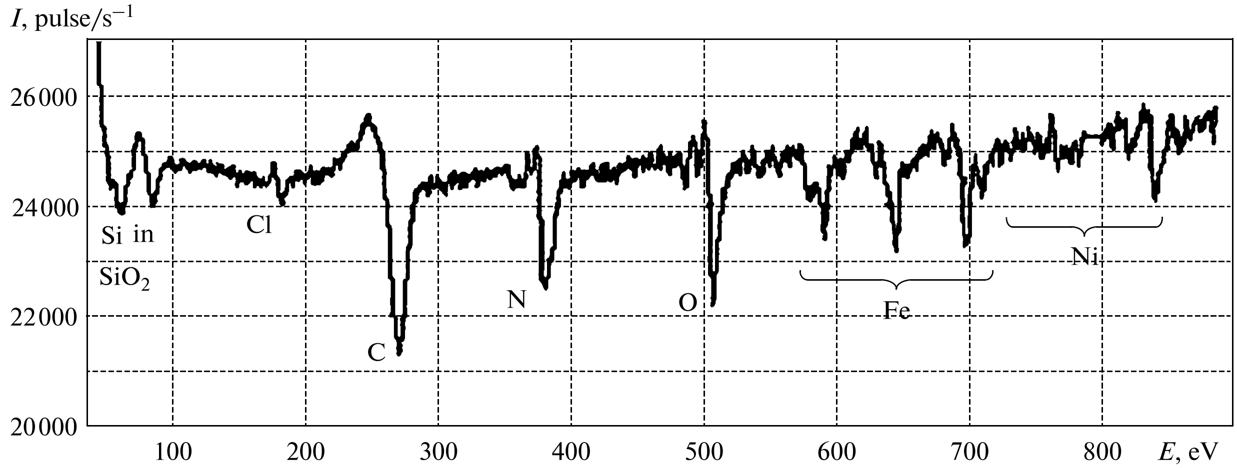


**Fig. 1.** Auger spectrum of a contact surface after hermetic sealing (before IPT). $I$ is the counting rate and $E$ is the kinetic energy of electrons.

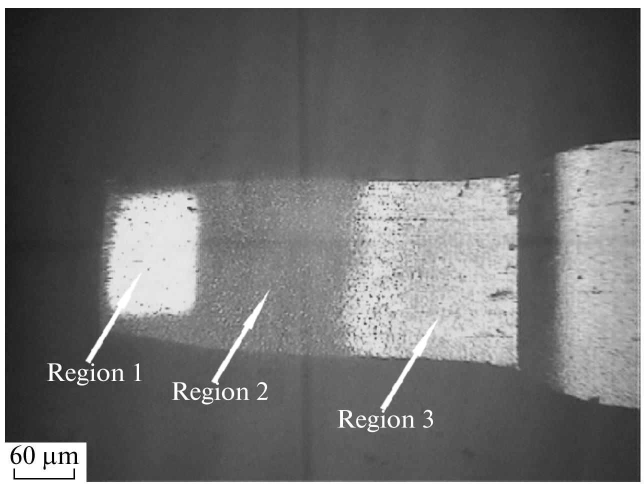


**Fig. 2.** Contact surface after 100 rounds of IPT.

## RESULTS AND DISCUSSION

A typical Auger spectrum of contact surfaces after hermetic sealing is shown in Fig. 1. Apart from the iron and nickel lines, the spectrum shows the lines of oxygen, carbon, silicon oxide, sulfur, chlorine, and nitrogen. We may assume that the up to 0.2–0.4 Ω increase in contact resistances after sealing is related to oxidation of the contact surfaces by glass nanoparticles impacting it, adsorption of oil vapors from the air, and the subsequent formation of polymer films. The results of XPS analysis also point to the formation of iron and nickel oxides, silicon nitride $Si_3N_4$, and different carbon-containing groups.

After IPT, a contact surface can be conditionally divided into three typical (from the standpoint of surface topography) regions, detectable by optical (Fig. 2) and atomic force (Fig. 3) microscopes. Region 1 (Figs. 2 and 3a) is the contact overlapping zone where a gas discharge is ignited when voltage is applied. According to AES and XPS, nitrides (FeN and NiN) are formed in the gas phase of the overlapping zones as a result of reactive cathode sputtering. Some of these nitrides precipitate on the contact surface in region 1 where the surface is nitrated (according to the Kelbel mechanism [8]) under the effect of ion bombardment. Other sputtered nitrides are predominantly precipitated on the surface of the contacts in neighboring region 2, due to diffusion (Figs. 2 and 3b). Judging from the optical image (Fig. 2), region 3 (Fig. 2) either contains no nitrides or their concentration is negligible.

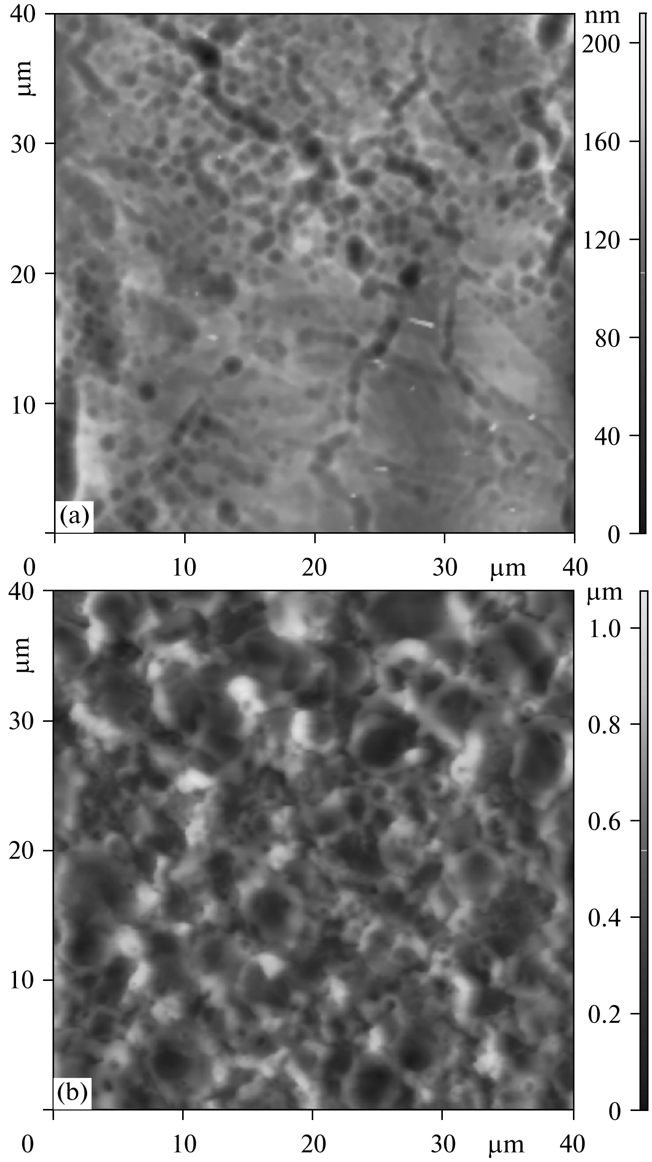


**Fig. 3.** 2D AFM: image of a contact surface after 100 rounds of IPT; (a) region 1; (b) region 2.

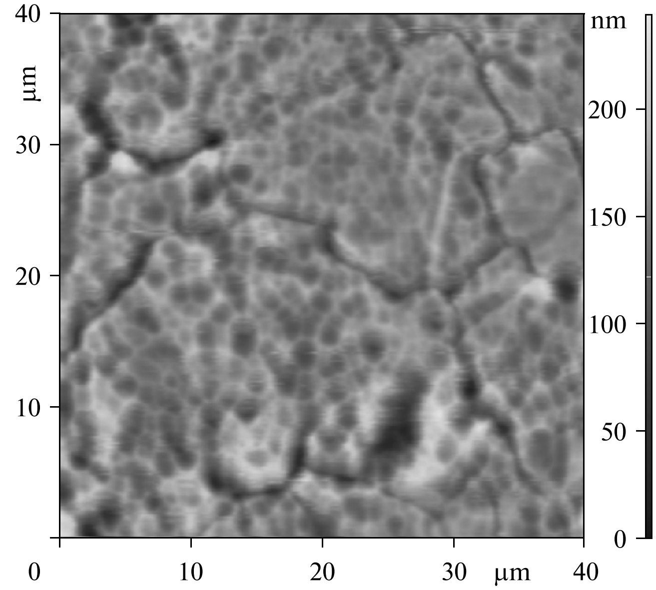


**Fig. 4.** 2D AFM: image of region 1 of a contact surface after 200 rounds of IPT.

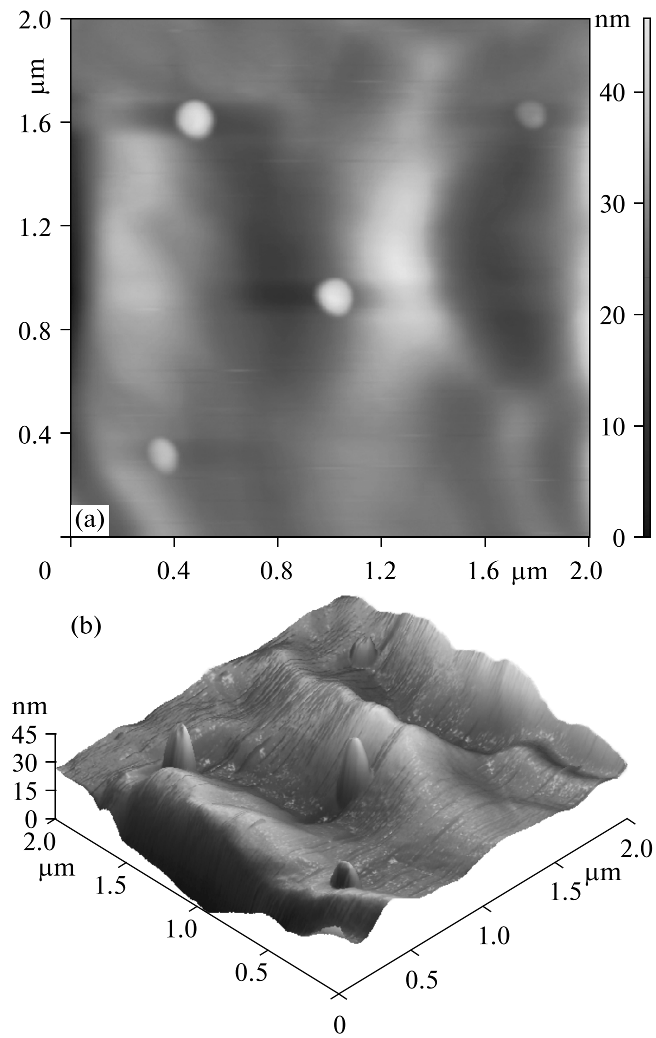


**Fig. 5.** AFM image of region 1 of a contact surface after 200 rounds of IPT: (a) 2D, (b) 3D.

IPT yields the formation of surface pores on the contact surfaces in region 1 (Figs. 3a, 4, 5), followed by the growth of conical prominences on the pore bottoms (Fig. 5). It follows from the figures that their concentration increases with the length of the discharge's duration. The conditions for the formation of such surface pores and prominences, and their development on the surface of a solid irradiated by ions, were considered in [1]. The author believed that their formation was mainly related to processes caused by ion-induced stresses, and to the displacement of atoms in the surface layer. This would be due to ion-accelerated diffusion, the displacement of dislocations, and recrystallization.

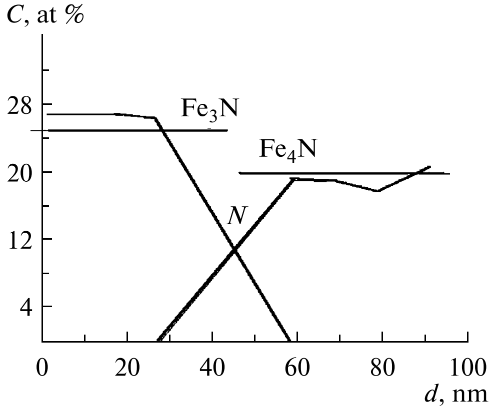


**Fig. 6.** Reconstructed profile of phase distribution over depth $d$, showing concentrations of nitrogen in stoichiometric compounds of $Fe_3N$ and $Fe_4Ni$ after one round of IPT.

After 30 rounds of IPT, the intermediate resistance dropped to 0.08–0.1 Ω. The observed increase in erosion and corrosion resistance of the contacts is related to the nitriding that occurs during IPT.

Indeed, AES revealed that IPT yields the formation of a nitride phase of the $Fe_3N$ type on the surface of iron–nickel contacts. At depth, it is replaced by a layer consisting of the $Fe_4N$ nitride phase (Fig. 6). Depending on the duration of ion nitriding, the thicknesses of the nitride layers varies from several tens to several thousands of nanometers.

The results from processing the XPS spectra of contact surfaces (Fig. 7) are in agreement with the results of our Auger analysis. The fine energy structure of the XPS spectrum lines also points to the formation of iron and nickel oxides and different carbon-containing groups. In addition, sealing the contact surfaces in a cylinder yields the synthesis of $Si_3N_4$ silicon nitride. This is confirmed by the N 1*s* and Si 2*p* photoelectron spectra (Figs. 8 and 9).

Figure 8 shows the state of nitrogen N present in the specimens. The principal maximum, with an energy of about 397.3 eV, corresponds to the nitride state, while $Si_3N_4$ is present in the source specimen; this also follows from the binding energy of the Si 2*p* maximum (102.5 eV) in Fig. 9.

## CONCLUSIONS

Iron–nickel contact surfaces after the action of a pulsed gas discharge plasma (ion-induced modification) were investigated by means of AES, XPS, and AFM. It was shown that the corrosion and erosion resistance of the modified contacts is related to features of the surface topography and the formation of nitride layers (iron and nickel) alternating with one another at depth. It was found that the ion irradiation of

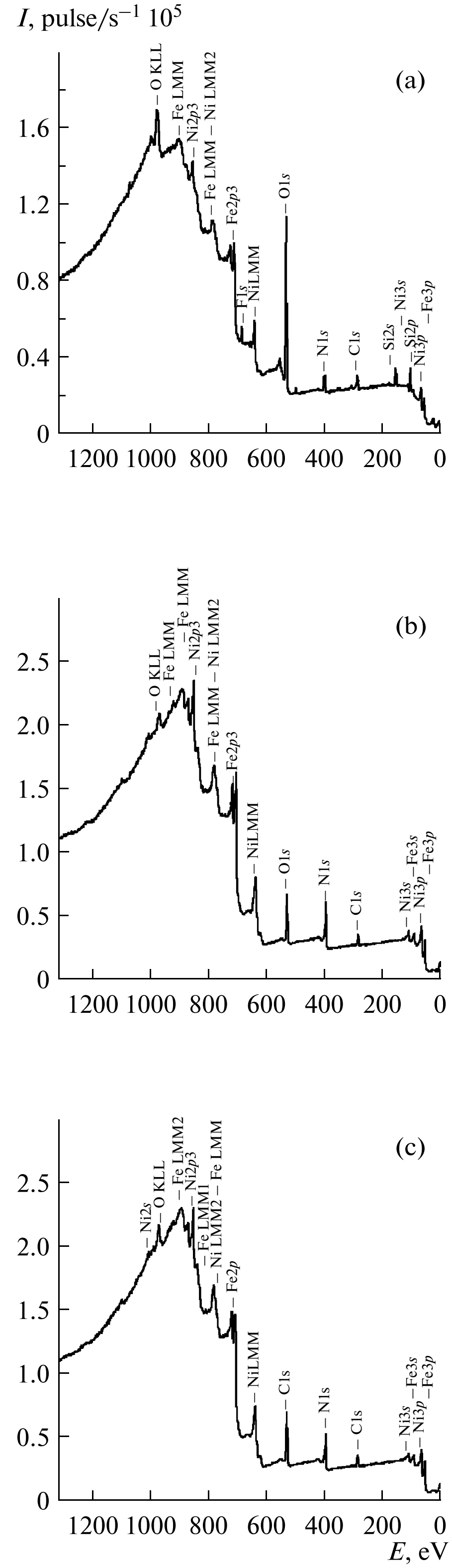


**Fig. 7.** Survey XPS spectrum of a contact surface (a) without IPT; (b) after 30 rounds of IPT; (c) after 100 rounds of IPT ($I$ is the counting rate, $E$ is the binding energy).

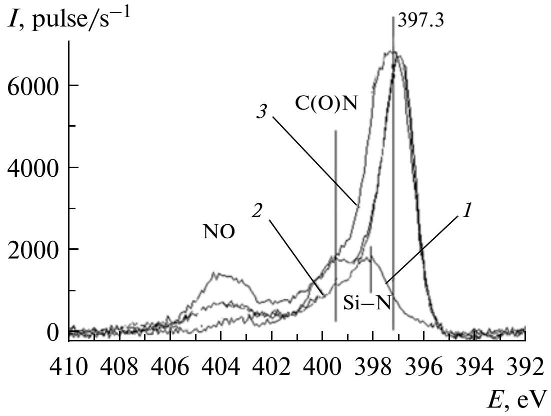


**Fig. 8.** Photoelectron N 1*s* spectra of a surface (*1*) without IPT; (*2*) after 30 rounds of IPT; (*3*) after 100 rounds of IPT (*I* is the counting rate, *E* is the binding energy).

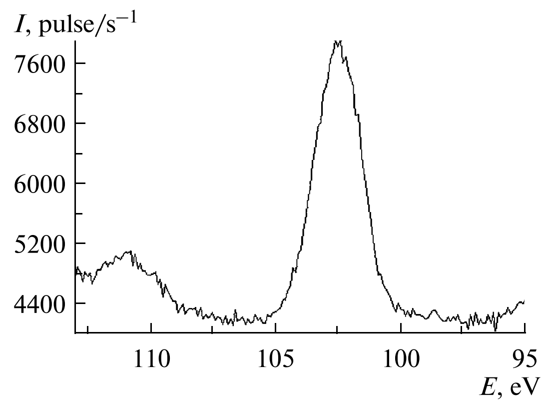


**Fig. 9.** Photoelectron Si 2*p* spectrum (*I* is the counting rate, *E* is the binding energy).

contact surfaces is accompanied by the formation of pores and cones whose density increases with the duration of irradiation. Energy features of the atomic state of the contact surfaces, manifesting themselves in the energy distribution of photoelectrons, were observed.

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