

Ion-Plasma Treatment of Reed Switch Contacts: A Study by Time-of-Flight Secondary Ion Mass Spectrometry

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Received June 11, 2013; in final form, July 22, 2013

Abstract—A TOF-SIMS-5 time-of-flight secondary ion mass spectrometer operating with pulsed 25-keV Bi⁺ ions for analysis and 2-keV Cs⁺ ions for sputter ion-beam etching was employed for studying the near-surface composition of iron–nickel (permalloy) contacts (blades) after the treatment in pulsed nitrogen plasma directly in hermetically sealed reed switches. The formation of 20- to 25-nm thick oxynitride coatings in the contacting region of the blades was observed. It was found that this coating was of the diffusive nature and produced via the plasma nitriding of the contacts.

Keywords: time-of-flight secondary ion mass spectrometry, reed switch, ion-plasma treatment, ion nitriding, cathode sputtering, sputter depth profiling

DOI: 10.1134/S1061934814130115

INTRODUCTION

The reed switch is a miniature electromechanical device composed of two ferromagnetic electrodes (contacts, blades or springs) in a hermetically sealed glass housing [1]. Under the action of an external magnetic field, the contacts touch each other and the reed switch is closed. Reed switches were developed in Bell Telephone Laboratories in 1936, and they are widely used until now in different switchgear and radioelectronic equipment.

The application of protective coatings on the contact surface is a basic technological process in the manufacture of reed switches. As a rule, the electroplating of precious and noble metals such as gold, palladium, rhodium, and ruthenium is used for this purpose [2, 3]. Among the disadvantages of this technology are the high cost of the utilized materials, the electrical energy and material consumption, and expensive equipment. On the electroplating, foreign particles can appear on the contact surface to increase the electrical resistance of the reed switch and, finally, to cause its malfunctioning. Furthermore, plating baths pollute the environment in the course of operation and electrolyte evaporation products are potentially dangerous to the health of the service personnel. Finally, the deposition of thin nonporous coatings or thick coatings with low internal stresses and high adhesion to the contact material is very difficult to perform in this case.

Ion-plasma treatment is widely used for improving the corrosion and erosion resistance of the surfaces of metals and alloys [4, 5]. As an alternative to the electroplating of noble metals, we proposed to create protective nitride coatings on the contact surface of an iron–nickel alloy (permalloy) directly in pressure-sealed devices by ion-plasma treatment. It was found [6–9] that the electrical resistance of reed switches treated by this method remained constant and did not exceed 0.1 Ω after switchgear reliability tests.

The topography and local distribution of electrostatic potential on the contact surface before and after ion-plasma treatment were studied by atomic force and metallographic microscopy [8, 9]. The development of surface pores, cones, and other heterogeneities with specific conducting properties was observed; the formation of different phases of nickel and iron nitrides on the surface and in the near-surface region was confirmed by Auger electron spectroscopy and X-ray photoelectron spectroscopy [7–9].

Previously [10], we studied the contact surfaces of reed switches by time-of-flight secondary ion mass spectrometry (TOF-SIMS). With the aid of this technique, we measured the in-depth profiles of different secondary ions in the working (where the contacts overlap on the closure of reed switches) and nonworking regions of the contacts before and after ion-plasma treatment. The formation of an oxynitride coating with the thickness of $\lambda \sim 350$ nm was detected in the working region. It was established that the formation

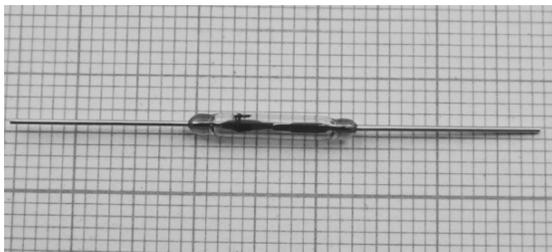


Fig. 1. External view of an MKA-14108 reed switch.

of this coating is related to not only the diffusion of nitrogen and oxygen inside the contacts but also, primarily, to cathode sputtering processes accompanied by the development of a topographic relief and the fusion of some regions on the contact surface.

This work is the continuation of our previous studies [10]. For the optimization of coating application conditions from the standpoint of energy saving and reliability, we considerably decreased the duration of voltage pulses supplied to the contacts of reed switches in the process of ion-plasma treatment. In this case, the total time of contact treatment remained the same. Anticipating things, note that a change in the pulse duration led to a change in the type of the discharge excited in a gap between the contacts, and, as a consequence, to the formation of coatings with different thicknesses and structures in the working region. The aim of this work was to study this coating by TOF-SIMS and to compare the experimental results with previously published data [10].

EXPERIMENTAL

We studied the prototype units of MKA-14108 reed switches (Fig. 1) manufactured by OAO Ryazanskii Zavod Metallokeramicheskikh Priborov (Ryazan Metal Ceramics Instrumentation Plant Joint Stock Company) on the basis of commercially produced MKA-14103 devices; the production cycle of these latter was described in detail in [6–9]. The contacts were punched from a Dilaton™ wire, which was stretched from a vacuum-melt permalloy workpiece (52% Ni and 48% Fe). Then, the contacts were degreased and annealed in an atmosphere of hydrogen. On sealing, the glass housings of the reed switches were filled with pure nitrogen (99.99%) to a pressure of $(33\text{--}40) \times 10^3$ Pa.

As compared with a standard technology, the operation of electroplating in the production of the prototypes was replaced by ion-plasma treatment performed by pulsed discharges, which were initiated in a gap between the contacts with the aid of specially developed pulse generators. In the test reed switches, this gap was $d = 27\text{--}30$ μm in the working region of the contacts, which was about 400 μm in length and 680 μm in width. Actually, the reed switch is the simplest diode sputtering system: the contacts are its elec-

trodes, alternately, a cathode and an anode, a target and a backing, and the glass bulb serves as a working chamber. The discharge in the gap between the contacts can be categorized as symmetrical because it is allowed between parallel electrodes of the same surface area, which are sputtered at the same intensity.

The voltage pulses with an amplitude of about 2 kV, a duration of 2 μs , and a frequency of 1 kHz were nearly rectangular in shape, and they followed in groups (packets). Each particular packet consisted of five pulses, whose polarity changed at a frequency of 50 Hz. The duration of single treatment was 30 s, the duration of a pause between the single treatments was 30 s, and the number of treatments was as high as 30. Note that 20- μs pulses were used previously [10], and all of the other parameters of ion-plasma treatment were the same as those used in this work. The distinctive features of discharges initiated by pulses of different durations will be considered in the Discussion section.

The mass spectra and in-depth profiles of secondary ions were measured at the Institute for Physics of Microstructures, Russian Academy of Sciences (Nizhni Novgorod) using a TOF.SIMS-5 instrument from ION-TOF (Muenster, Germany) with a reflectron time-of-flight mass analyzer. The spectrometer operated with two ion beams: a pulse ion beam of 25-keV Bi⁺ was used for analysis, and the sputter depth profiling was performed with 2-keV Cs⁺ ions. The cesium ion current was about 120 nA, and the beam was scanned over an area of 200 \times 200 μm . The zone of analysis was selected at the center of an etching crater, and it was about 4% of the crater area. The intensity of different atomic and cluster secondary ions of negative polarity was measured. Preliminary experiments showed that the negative secondary ions formed upon the bombardment of nitrogen-containing coatings by bismuth ions with sputtering by cesium ions were more intense than positive ones. The depth of the etching crater after performing the analysis was determined with the aid of a Talysurf CCI-2000 optical profilometer from AMETEK Taylor Hobson (Leicester, the United Kingdom). The experimental conditions of the TOF-SIMS measurements were described in detail elsewhere [11].

Directly before the measurements, the glass housing of a reed switch was mechanically destroyed, and the contacts were extracted outside. The contacts were not specially treated before the measurements except for the removal of glass remainders using a jet of pure nitrogen.

RESULTS

Figure 2 shows the working region of the contact of an MKA-14108 reed switch with a sputtering crater after TOF-SIMS analysis. Figures 3 and 4 show typical in-depth profiles of negative secondary ions measured in the MKA-14108 reed switch after 30-fold

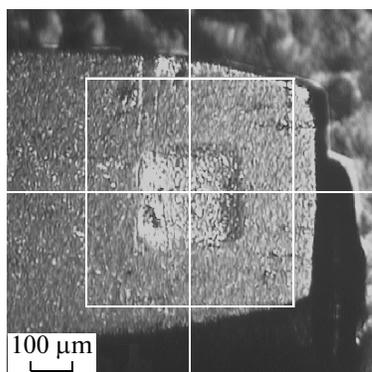


Fig. 2. Working region of the contact of an MKA-14108 reed switch with a sputtering crater after TOF-SIMS analysis.

ion-plasma treatment for working and nonworking contact regions, respectively. All of the profiles are represented on a semilogarithmic scale, and the sample sputtering time was converted into the depth of layer-by-layer analysis at a constant sputtering rate of ~ 50 nm/min, which was calculated from data on the depth of a crater after the end of sputtering. Earlier, Baldwin et al. [12] found that, for iron alloys treated in a nitrogen plasma, the rate of sputtering remained constant both inside the modified layer and at the interface between this layer and the base material. Therefore, the measurement of the final depth of the sputtering crater is sufficient for calibrating the depth scale of the layer-by-layer analysis.

We selected the cluster ions $[\text{FeN}]^-$, $[\text{NiN}]^-$, $[\text{CN}]^-$, and $[\text{NO}]^-$ as the characteristic secondary ions for nitride coatings. The signal intensities of $[\text{FeN}]^-$ and $[\text{FeO}]^-$ were normalized to a signal from Fe_2^- cluster ions; other signals were normalized to the intensity of Ni_2^- . The ratio of normalized signals $(\text{Fe}_2/\text{Ni}_2)^-$ was constant throughout the entire depth of the sputtering crater in all of the samples tested in this work. The intensity of the signals of the secondary atomic and molecular ions of nitrogen was very low; therefore, the distribution profiles of these ions are not shown in Figs. 3 and 4.

Now, we consider the nature of secondary ions whose profiles are given in these figures. It is most likely that the ions $[\text{FeN}]^-$ and $[\text{NiN}]^-$ are the dissociation products of Fe_nN and Ni_nN molecules (where $n = 2-4$) under the action of local surface heating and cathode sputtering processes on ion-plasma treatment and also due to the ion bombardment of the contacts in the course of TOF-SIMS analysis. The ions of iron and nickel oxides, as well as the ions of atomic oxygen and nitrogen oxide, can originate from natural oxide layers, which always occur on contact surfaces. The ions of carbon are mainly residual pollution products from lubricants used in permalloy wire drawing. The

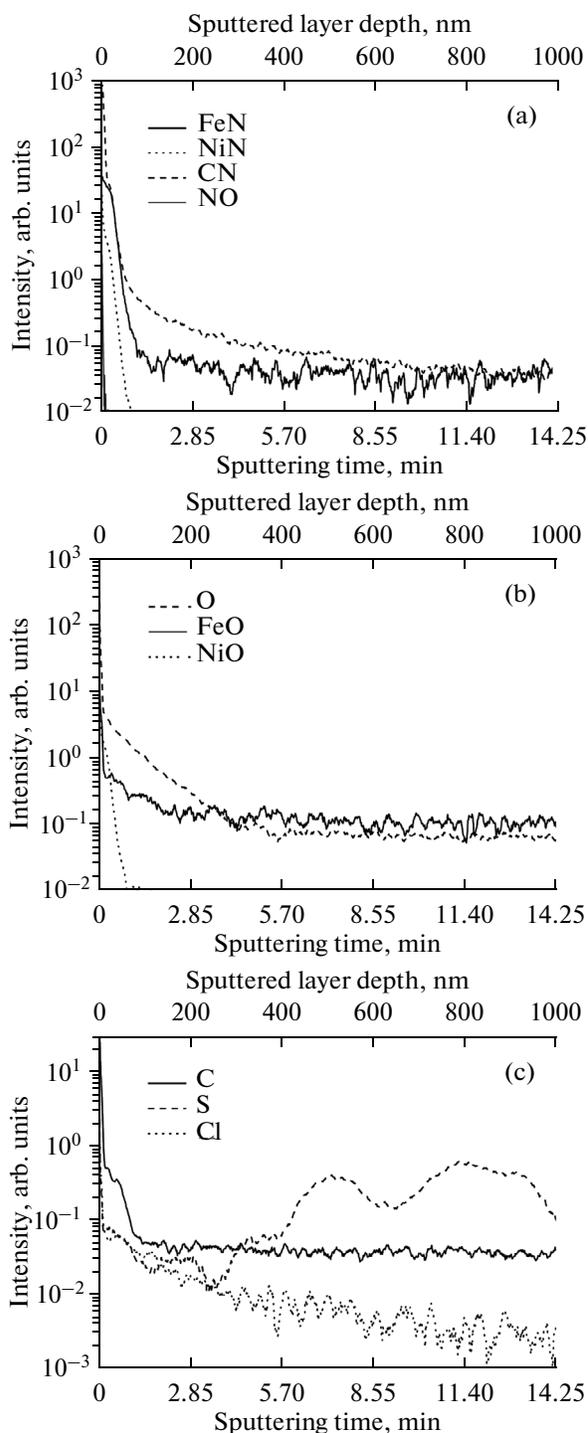


Fig. 3. In-depth profiles of the relative intensities of negative secondary ions measured in the working region of a reed switch contact after ion-plasma treatment: (a) nitrogen-containing ions, (b) oxygen-containing ions, and (c) impurity ions.

ions of nitrogen carbide can be formed as a result of the interaction of carbon and nitrogen both on the contact surfaces and in the plasma at the surface; these chemical elements are prone to the formation of

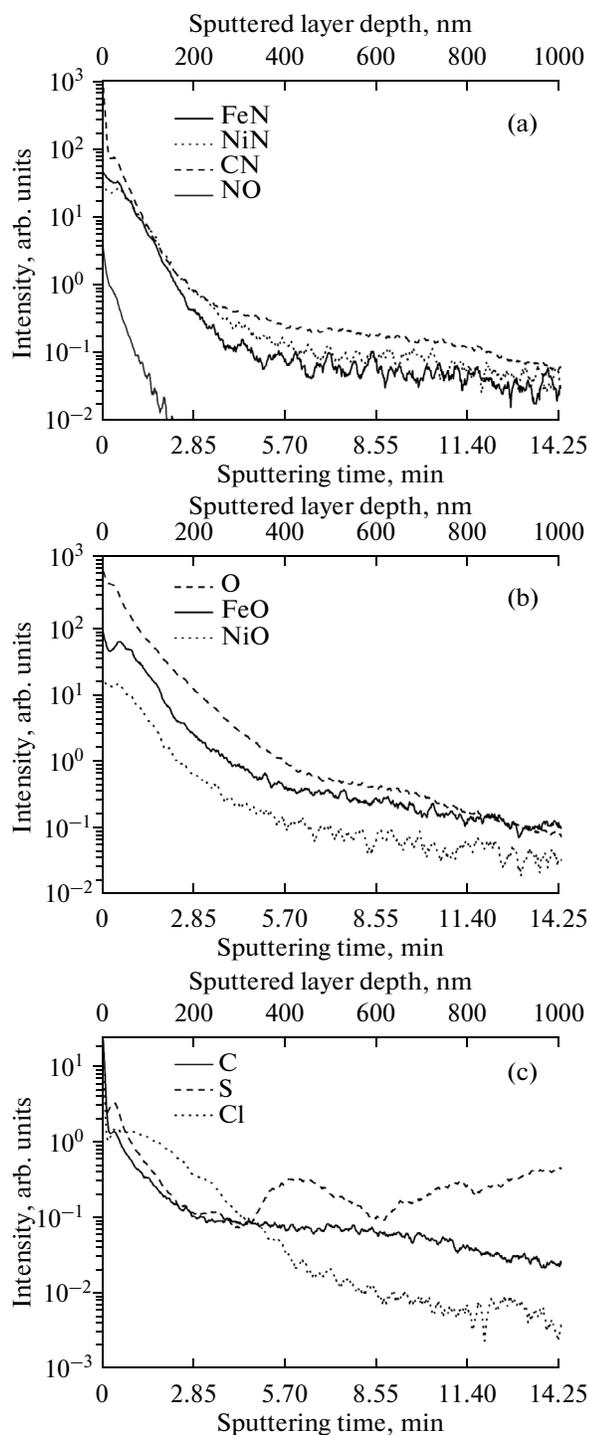


Fig. 4. In-depth profiles of the relative intensities of negative secondary ions measured in the nonworking region of a reed switch contact after ion-plasma treatment: (a) nitrogen-containing ions, (b) oxygen-containing ions, and (c) impurity ions.

strong chemical bonds, including univalent ones [13]. Chlorine is a widespread surface pollutant, which is characterized by a very high yield of negative secondary ions; in our case, chlorine-containing solvents

used for the cleaning and degreasing of permalloy wire can be the source of chlorine. Finally, the ions of sulfur, which also belongs to the elements with high electron affinity, are a bulk pollutant of permalloy.

At present, we cannot perform quantitative TOF-SIMS analysis because we do not have available information on the efficiency of formation of negative secondary atomic and cluster ions under the Bi^+ ion bombardment of nitride compounds on the surfaces of reed switch contacts. Nevertheless, using the normalized signal intensities of secondary ions, we can compare the results represented in Figs. 3 and 4, estimate the thickness of coatings in the working and nonworking regions of contacts, and correlate these data with the results published previously [10].

DISCUSSION

Earlier, we found [6–9] that, depending on the duration of exciting pulses, the ion-plasma treatment of reed switch contact surfaces occurs in either glow discharge ($\tau_1 = 2 \mu\text{s}$) or combined discharge ($\tau_2 = 20 \mu\text{s}$) plasma. We measured the in-depth profiles of different secondary ions in the case of a combined (anomalous glow and arc) discharge [10]; reactive ion-plasma sputtering [14, 15] in combination with ion nitriding [4, 5] was the predominant mechanism of coatings in the working region.

In the case of a glow discharge, the diffusion saturation of a surface with nitrogen atoms with the formation of nitride compounds predominantly due to ion nitriding will be observed in the working region of contacts. Although this method is widely used in industrial production, up to now, there has been no universal theory developed to explain the entire set of physicochemical processes that form its basis. According to simplified models [4, 5, 16–18], in the process of ion nitriding, nitrogen-containing particles (atoms, excited molecules, and radicals formed in the plasma) are chemisorbed on the surface of treated materials (in our case, iron–nickel contacts), and they diffuse into their bulk. In this case, the ions of nitrogen and fast neutral molecules bombard the contact surface, and a substantial part of their energy (to 90%) is consumed for the heating of contacts. The erosion of contacts accompanied by the cathode sputtering of surface atoms and molecules (Fe, Ni, their nitrides and oxides, C, O, S, Cl, and other impurities) occurs due to this bombardment. Other processes such as the scattering of ions and neutral particles, electron and ion emission, and ion implantation also occur. The sputtered and scattered particles in a volume at the contact surface can collide and chemically interact with gas molecules to form different nitrogen compounds in this case; these compounds are deposited on the treated contact surfaces and on the walls of the glass bulb of a reed switch. At a high surface temperature (higher than 400–500°C), nitrides are partially decomposed, and the formed nitrogen diffuses into

the bulk of the sample and evaporates into the plasma. In the region of relatively low temperatures (lower than 300°C), the surface implantation of nitrogen ions can influence the density, phase composition, and microstructure of the nitrided layers to accelerate their formation [19].

Note that the nonworking contact surface is mainly heated due to heat exchange with a more heated working region. Because of the low temperature, the dissociation of iron and nickel nitride molecules and, therefore, ion nitriding in the nonworking region are less probable than those in the working region. The products of cathode sputtering will make a more significant contribution to the formation of coatings in the nonworking region and on the internal surface of the glass envelope of a reed switch [20]. Note that this coating is darker in color than that in the working region.

Usually, the process of nitriding in a glow discharge plasma occurs at a nitrogen pressure of 10–10³ Pa and a potential difference between the cathode and the anode of about 0.3–1 kV [10]. In this case, the electric field strength E is 10–50 V/cm. In our case, at both exciting pulse durations, the nitriding of the contact surface was performed at the pressure $p = 3 \times 10^4$ Pa and $E = 0.5 \times 10^6$ V/cm, which are considerably higher than these parameters under ordinary ion nitriding conditions. Consequently, the processes occurring at the contact surface and in the plasma in our case are more complex than those described above.

Let us consider the experimental results shown in Figs. 3 and 4. The in-depth profiles of secondary nitrogen-containing ions measured in the working and nonworking contact regions after ion-plasma treatment (Figs. 3a and 4a) have approximately the same shapes with a decrease in the intensity on penetrating deep into the contact material; this decrease is more rapid in the working region than that in the nonworking one. The character of this decrease is close to a diffusion (exponential) one. We approximated the profiles of [FeN]⁻ and [NiN]⁻ by the relationship

$$I \sim \exp(-Az), \quad (1)$$

where I is the corresponding signal intensity, and z is the depth of a sputtered layer (or the layer-by-layer analysis time). The values of A were found to be 0.15 and 0.17 or 0.030 and 0.026 for [FeN]⁻ and [NiN]⁻ in the working or nonworking region, respectively. The profiles were approximated for $z \geq 25$ nm in the working region and for $z \geq 50$ nm in the nonworking region.

Based on these data, we can state that the rate of formation of a nitrogen-containing coating in the working region of contacts was higher than that in the nonworking region. Note that similar profiles measured previously [10] in the working region at longer exciting pulses were clearly of nondiffusion nature. We obtained the following values of coefficients A for the nonworking region: 0.044 for [FeN]⁻ and 0.048 for [NiN]⁻. They are somewhat greater than analogous

data for the nonworking region of the contacts of the reed switch studied in this work; at the same time, they are much lower than the values obtained for this reed switch in the working region of contacts. We can conclude that the processes responsible for the formation of nitride coatings in the nonworking regions of contacts at different types of discharges in the gap between the contacts are the same, namely, ion nitriding with a contribution from cathode sputtering. They are different in the working region: these are predominantly ion nitriding for short exciting pulses ($\tau_1 = 2 \mu\text{s}$) and cathode sputtering with a contribution from ion nitriding for longer pulses ($\tau_2 = 20 \mu\text{s}$).

We also estimated the diffusion coefficient of nitrogen D (in the case of short pulses) in a near-surface layer of permalloy in the working region according to the formula [6–9]

$$D = z_e^2/T, \quad (2)$$

where z_e is the depth of the sputtered layer at a level of the signal decreased by a factor of e with respect to its maximum value, and T is the contact treatment time, which can be calculated from the formula [6–9]

$$T = \tau N F k \Delta / 2, \quad (3)$$

where τ is the voltage pulse duration, N is the number of single-polarity pulses in a packet, F is the pulse packet repetition frequency, Δ is the duration of single treatment, and k is the number of treatments.

From the experimental profile of the secondary ion [FeN]⁻, which is shown in Fig. 3a, we can estimate the value of z_e at about 20 nm.

At the process parameters of ion-plasma treatment ($\tau = 2 \mu\text{s}$, $N = 5$, $F = 50$ Hz, $\Delta = 30$ s, and $k = 30$), we obtained $D \sim 1.8 \times 10^{-11}$ cm²/s, which is consistent with the results of the calculations of nitrogen diffusion coefficients in the samples of steel treated in a pulsed glow-discharge plasma [21].

The comparison of the diffusion coefficient obtained in this work with the mobility of nitrogen atoms in iron upon treatment in a continuous glow discharge [22] showed that the pulse discharge used for the ion-plasma treatment of the reed switch increased the mobility of nitrogen by a factor of about 2.

Figures 3b and 4b show the in-depth profiles of the secondary ions of atomic oxygen and nickel and iron oxides. These distributions are very similar to the distributions of nitrogen-containing secondary ions for the corresponding regions discussed above. We can say that, under the conditions of our experiments, the ion-plasma treatment of the contact surface of reed switches is the process of nitriding combined with oxidation. Natural nickel and iron oxides present on the contact surface and oxygen-containing gases released from the walls of a glass bulb under the action of glow discharge plasma can serve as a source of oxygen.

The in-depth profiles of the secondary ions of impurities (carbon, sulfur, and chlorine) in the working region exhibited an increased intensity near the

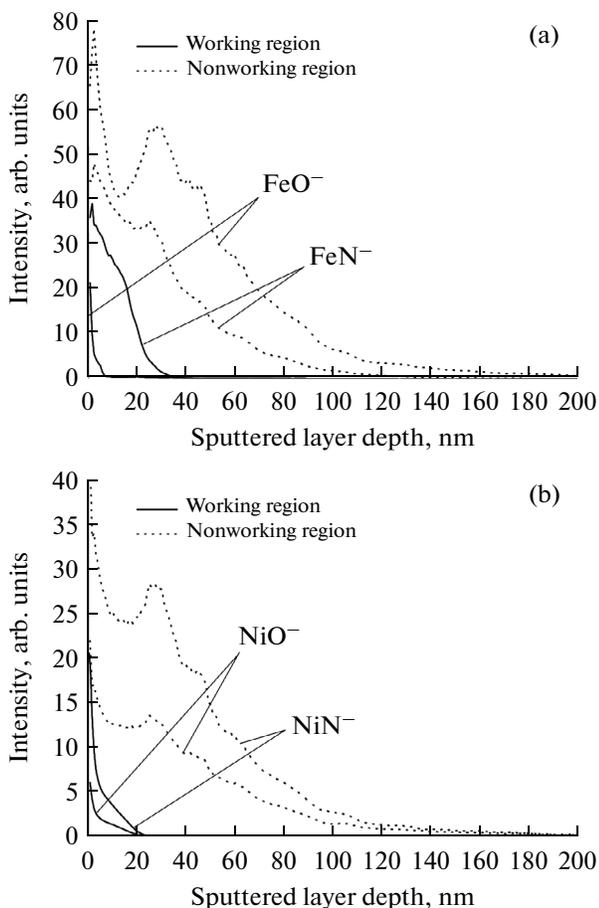


Fig. 5. In-depth profiles of negative secondary ions measured in a reed switch after ion-plasma treatment in the working and nonworking regions of the contact: (a) iron-containing $[\text{FeN}]^-$ and $[\text{FeO}]^-$ and (b) nickel-containing $[\text{NiN}]^-$ and $[\text{NiO}]^-$.

surface (Fig. 3c); this is likely due to an increase in the ion yield, which is typical of the layer-by-layer SIMS analysis of electronegative elements. The intensity of all impurity ions considerably decreased and reached a level of the bulk impurities of permalloy as the material of contacts was sputtered. In the nonworking region, the profiles were more pulled deep into the sample. Let us note humps in the distributions of the secondary ions of sulfur in both working and nonworking regions of the contacts. These irregularities could be caused by the processes of sulfur segregation in the production of permalloy wire, and the detailed consideration of them is beyond the scope of this work. It is necessary to consider that the yields of the negative secondary ions of sulfur and chlorine are very high. Therefore, although the intensity of the mass peaks of the secondary ions of these elements is high, their absolute concentration in the contacts is low; probably, it is lower than 10^{18} atom/cm³. In our opinion, they do not exert a substantial influence on the properties of the nitride coating formed on the surface of contacts.

Figure 5 shows the in-depth profiles of the nitride and oxide negative secondary ions of iron and nickel on a linear scale of intensity. From these data, we estimated the thickness of an oxynitride coating (at the level of a 10% decrease in the signal intensity with respect to a maximum value), which varied over a range of 20–25 nm for the working region or 80–90 nm for the nonworking region. For comparison, let us give analogous data obtained previously with long exciting pulses [10]: 320–380 nm in the working region and <75 nm in the nonworking region of contacts. The data for the nonworking regions of contacts approximately coincide because the processes of coating formation in these regions are identical. At the same time, much different values were obtained for the working regions; they confirm that the processes responsible for the formation coatings in these regions are different. Note that the development of a diverse topographic relief (irregularities, cones, cavities, etc. [6–9]) was observed on the surface of contacts in the working region at long exciting pulses, and this relief was more pronounced than that observed at short pulses. Selective cathode sputtering could be a reason [20]. The presence of a surface relief can distort the data of layer-by-layer TOF-SIMS analysis leading to an increase in the thicknesses of sputtered layers calculated from the in-depth profiles. In our opinion, the surface relief induced by the cathode sputtering of contacts is one of the main reasons for different colors of working regions in the optical images of contacts—they appeared lighter colored at short pulses [6–9].

The peaks observed in the in-depth distribution profiles of nitrogen-containing and oxygen-containing ions in the nonworking region of contacts at a depth of 25–30 nm have engaged our attention. In principle, peaks of this kind also occurred in the distribution profiles of other secondary ions in the nonworking region of contacts. Less pronounced peaks at approximately the same depth were also observed in this region at long exciting pulses [10].

So far, we cannot comprehensively explain the nature of these peaks, although we can reliably state that they are not related to the instability of instrumentation. It is believed that the structure of an applied coating changes at this depth; in turn, this also leads to a change in the yield of secondary ions.

CONCLUSIONS

This work characterizes in detail the near-surface layers of reed switch contacts before and after their treatment in the plasma of a glow discharge initiated by short voltage pulses ($\tau_1 = 2 \mu\text{s}$) directly in the pressure-sealed device. The layer-by-layer TOF-SIMS profiles obtained by a sputtering beam of Cs^+ ions demonstrate the presence of a layer of iron and nickel nitrides with the thickness $\lambda \sim 20\text{--}25$ nm in the working region of the contacts. It was shown that this layer has a diffusion character, and its origin is predomi-

nantly related to ion nitriding processes. The in-depth profiles of secondary atomic negative ions of oxygen and oxygen-containing clusters indicate that we have oxynitride coatings. An oxynitride coating was also formed in the nonworking region of contacts, but it was thicker (approximately 80–90 nm) and formed due to the cathode sputtering and ion nitriding.

A comparison with our results published earlier [10] for longer pulses ($\tau_2 = 20 \mu\text{s}$) showed that the coating of contacts tested in this work was thinner in the working region or of approximately the same thickness in the nonworking region. According to the switchgear tests of reed switches, both types of coatings possess sufficient corrosion and erosion resistance and high electrical conductivity; however, the coating technology with the aid of short ($\tau_1 = 2 \mu\text{s}$) pulses is less power-consuming and hence more advantageous to be introduced into industrial production. In general, both of the technologies for the ion-plasma treatment of reed switch contacts were found competitive with traditional electroplating methods based on rare and noble metals.

ACKNOWLEDGMENTS

This work was supported by OAO Ryazanskii Zavod Metallokeramicheskikh Priborov (Ryazan Metal Ceramics Instrumentation Plant Joint Stock Company), the Ministry of Education and Science of the Russian Federation (contract no. 14.V37.21.0895), the Portuguese Foundation for Science and Technology (project PTDC/CTM-ENE/2514/2012), and via the programs of the Presidium of the Russian Academy of Sciences, and the Russian Foundation for Basic Research (project no. 12-02-00548). The equipment of the Institute for Physics of Microstructures, Russian Academy of Sciences (Nizhni Novgorod) was used in this study.

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Translated by V. Makhlyarchuk