Creation of Principally New Generation of Switching Technique Elements (Reed Switches) with Nanostructured Contact Surfaces

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Abstract. The cycle of activities of the creation of principally new generation of reed switches with nanostructured contact surfaces was implemented. Experimental justification of the opportunity of reed switches creation with modified contact surface was given (instead of precious metals-based galvanic coating). Principally new technological process of modification of magnetically operated contacts contacting surfaces was developed, based on the usage of the ion-plasma methods of nanolayers and nanostructures forming having specified contact features.

1. Introduction

One of the basic manufacturing operations of the modern reed switches production is operation of the special (corrosion- and erosion-resistant) coatings deposition on working surfaces of permalloy contact springs of reed switches. Meanwhile, a galvanic technology is typically used for coating deposition, and precious and noble metals (gold, palladium, rhodium, ruthenium and so on) are used as materials. [1, 2].

The problems of existing electrodeposition methods of reed switch contact plating are a high precious metal consumption and stock loss thereof, long production time, difficulty and high cost of equipment, large expenditure of energy, complication in alloy deposition of the specified chemical and phase composition and given structure, complication in production of thin nonporous or thick films with low internal voltages and high adhesion to contact blade material [1, 2].

Non-usage of noble metals and methods of deposition thereof will allow producing goods with a competitive price-quality ratio.

The cycle of works [1 - 10] which common purpose is to create a principally new generation of switching technique elements (reed switches) with nanostructured contact surfaces, based on the usage of ion-plasma methods of nitriding and forming of nanolayers and nanostructures with the given contact features, was made.
Ion-plasma nitriding application for modification of surface properties and near-surface layers is known \[11, 12\]. Typically, such properties are hardness, durability, and corrosion stability. Different variations of the method of nitrided layers creation are known as well \[11, 12\]. However, in this case, nitriding was used for creation of the special-purpose surface structure having new special properties.

This paper is a logical follow-up of the cycle of works \[1 - 10\]. The purpose thereof is an experimental investigation of an opportunity of reed switches production with a modified permalloy contact surface (instead of galvanic plating made of precious metals).

The raised task solution is directly connected with study of the special new properties of surface structures being formed as the result of ion-plasma modification of reed switch contact surfaces.

2. Samples and Technique of Experiment

The samples made in a constructive way on the basis of MKA-14103 off-the-shelf devices \[13\] were MKA-14108 reed switches (Figure 1) \[9\]. The major distinctive feature of such reed switch design compared to off-the-shelf devices consisted in the absence of any special coatings on permalloy contacts. Contact springs were shaped from permalloy wire, degreased and fired in the hydrogen atmosphere. The wire was pulled out from permalloy (52% Ni, 48% Fe) of vacuum melting. During vacuum sealing a spectroscopically pure (99.999 %) nitrogen was used as a gas filling. Upon the vacuum sealing a gas pressure in reed switches was 250 – 290 Torr.

![Reed switch design](image)

**Figure 1.** Reed switch design.
Ion-Plasma Treatment (IPT) of contact surfaces was conducted by high-voltage pulse discharges pursuant to the conditions specified in [10]. Discharges were initiated on reed switch open contacts (with a gap $d = 20 – 30 \mu\text{m}$) with the help of ion-plasma treatment machine [4]. Under such conditions [10] the surface modification is implemented as a result of ion nitriding [1, 2, 6, 11, 12].

It is known [1, 2] that the major effect of corrosion and pollutions on electrical contacts consists in conductivity irregularity, so contact resistance to such processes can be characterized by a number of closings, whereby a conductivity changes tragically. Contact erosion resistance can be characterized by a number of closings, whereby conductivity irregularity or non-opening of contacts occurs.

Reed switch experimental samples after treatments by high-voltage discharges, and without such, were subject to the comparative switching tests.

It was tests for active load and in idle mode (under no-load).

The state of contact surface was judged by from diagnostics results by methods of AFM and optical microscopy, Auger-electron spectroscopy as well as integral measurement of electrical resistance $R$ value of reed switch.

This allowed optimizing modes of contact spring surface modification of reed switch experimental samples, and studying physical processes occurring with all treatment stages and with reed switch operation.
3. Experimental Results and Discussion

3.1. Switching Tests

Comparative switching tests of pilot MKA-14108 reed switches and MKA 14103 off-the-shelf devices showed that pilot MKA-14108 reed switch running time, pretreated by high-voltage pulse discharges, generally corresponds to requirements raised to durability of MKA 14103 off-the-shelf devices (Table 1).

| Mode of switching tests | Reed switch type | Without failing, % | Rmax before tests, Ohm | Rmax after tests, Ohm |
|-------------------------|------------------|--------------------|------------------------|-----------------------|
| 50 mV - 5 µA, 100 Hz, 10^6 operations | MKA-14103 | 100 | 0.1 | 0.1 |
|                        | MKA-14108, without treatment | 21 | 0.25 | 100 |
|                        | MKA14108, with treatment | 100 | 0.07 | 0.08 |
| 5 V - 10 mA, 100 Hz, 10^6 operations | MKA-14103 | 100 | 0.17 | 0.11 |
|                        | MKA14108, without treatment | 27 | 0.35 | 22.7 |
|                        | MKA14108, with treatment | 100 | 0.08 | 0.08 |
| 20 V - 0.5 A, 50 Hz, 5·10^6 operations | MKA-14103 | 38 | 0.16 | 40 |
|                        | MKA14108, without treatment | 47 | 0.31 | 0.11 |
|                        | MKA14108, with treatment | 100 | 0.07 | 0.10 |
| 24 V - 400 mA, 50 Hz, 5·10^5 operations | MKA-14103 | 100 | 0.13 | 0.5 |
|                        | MKA14108, without treatment | 100 | 0.29 | 0.11 |
|                        | MKA14108, with treatment | 100 | 0.07 | 0.01 |
| 36 V - 15 mA, 50 Hz, 5·10^6 operations | MKA-14103 | 25 | 0.2 | 8.7 |
|                        | MKA14108, without treatment | 53 | 0.49 | 0.34 |
|                        | MKA14108, with treatment | 100 | 0.08 | 0.07 |
| 50 V - 50 mA, 50 Hz, 5·10^5 operations | MKA-14103 | 100 | 0.14 | 0.22 |
|                        | MKA14108, without treatment | 100 | 0.38 | 0.25 |
|                        | MKA14108, with treatment | 100 | 0.07 | 0.08 |
| 100 V -100 mA, 50 Hz, 5·10^5 operations | MKA-14103 | 2 | 0.17 | 0.19 |
|                        | MKA14108, without treatment | 100 | 0.27 | 0.23 |
|                        | MKA14108, with treatment | 100 | 0.07 | 0.07 |

For treatment mode optimization and identification MKA-14108 reed switch pilot samples tests before and after treatment under no-load (idle) were implemented. Switching quantity changed step-by-step from 0 to 10^7 collisions. At any stage of reed switch testing R value was measured (Figure 2).
Figure 2. Relation between MKA14108 reed switch resistance (R) across median and operations number N in «dry» circuit (1 – untreated contact blades, 2 - contact blades after IPT). Reed switch quantity within a batch is 100 pcs.

Contact surface morphology changes in terms of switching number were studied under metallurgical and atomic-force microscopes (Figures 3 - 6).

Figure 3. MKA-14108 reed switch contact surfaces after N operations under no-load, where N: a - $10^4$; b - $10^5$; c - $10^6$; d - $10^7$. Magnification is 300X.

With operation number increasing a reed switch resistance pretreated by high-voltage pulse discharges remains stable and does not exceed 0.1 Ohm (Figure 2, Curve 2). On the contrary, with operation number increase an untreated reed switch resistance raises about 100 times, and with $10^7$ operations it reaches 10 Ohm (Figure 2, Curve 1).

With an operation number of $10^4$, independently in contacting area, some black-brown spots emerge on reed switch contact surface having no special coatings (Figure 3). It is thermal decomposition products of polymer films formed from adsorbed carbon, oxygen and hydrogen on a contact surface during switchings. With a switching number increase up to $10^7$, size and degree of such spots blackening increase, and in step with this process R-value increases. Within range of $10^5$ – $10^6$ polymer films damage and R-value decrease are observed (Figures 2, 3). With a further increase of switching number (up to $10^7$)
instead of films broken by collisions new polymer coatings start developing that is accompanied by R increase [1, 2, 6] (Figures 2, 3). Reasons for such island coatings origin were discussed in [1, 2, 6]. Here we will discuss an issue of contacts erosion.

From images of contact surfaces shown in Figures 4-6 it is evident that resistance of reed switches with untreated contact blades increases also due to surface erosion as a result of actual contact area decreasing. In Figure 4, one can clearly see areas of surface erosion of untreated contact blade. 2D, 3D- image of one of these areas (it is indicated by an arrow in Figures 4-6) is presented in Figures 5, 6, respectively.

Contact surface of nitrided contact blades, on the contrary, was found to be more resistant to erosion process due to higher hardness of nitrided layers, therefore resistance of reed switches while switching remains stably low (Figure 2, Curve 2). A similar stability is demonstrated by MKA-14103 reed switches having a gold-ruthenium coating while switching dry circuits [6].

![Image of untreated contact blade surface of MKA-14108 reed switch after 3·10⁷ operations in a dry circuit. Magnification is 300X.](image-url)
**Figure 5.** 2-D-image of untreated contact blade surface of MKA-14108 reed switch after $3 \cdot 10^7$ operations in a dry circuit.

**Figure 6.** 3-D-image of untreated contact blade surface of MKA-14108 reed switch after $3 \cdot 10^7$ operations in a dry circuit.
Let's examine possible origins of erosion being observed.

Processes which take place in a gas filling, on surface and in volume of reed switch contacts, determine an evolution of contact blades surface of reed switches while switching, and in most cases, have a negative influence on functionality of devices.

A geometrical surface of contacts is such that an actual metal contact is realized only in separate points [13-15]. Even the smoothest metal surfaces have microasperities of 0.01-0.1 µm in height [16]. These asperities are located usually on some wavy surface, period of which in different conditions is $10^3 - 10^4$ µm, and height of 1-10 µm [17]. Just because waviness and asperities are present, it results in that two surfaces come in contact only in separate points [17].

During switching, under the influence of pressure forces of contact surfaces, in places of asperities, metal will be deformed and they will be transformed into conductivity centres. The more pressure forces of contact surfaces occur the more asperities may be deformed: partly plastically and (or) partly elastically [14, 15].

If the contacts of reed switch are closed, which are being under some voltage, so with a critical approaching thereof, a vacuum breakdown occurs [17] which is similar to that discussed in works [18, 19]. After closing contacts, a force arises which may lead to the bounce thereof. This force is conditioned by resistance of contact springs [20] and by high pressure of vapour and metal plasma as a result of micro-explosions in places of contact areas [17].

While switching dry circuits (without load), contacts of reed switch are also under voltage of 10 - 30 mV. It has been found that the origins of this voltage (dynamic noise) are electro-induction processes in a ferromagnetic material during reconstruction of its domain structure, under the influence of mechanical stresses and magnetic field [21].

While current breaking, due to the surface irregularity, contacts diverge non-simultaneously. The whole circuital current will flow through separate spots. Therefore, the current constriction area will be melted, and a molten metal bridge is formed [17] which, being exploded, creates a specific surface relief of reed switch contacts, that has a negative impact on operability of device [21].

Thus, if the contact material is relatively soft and fusible (permalloy is exactly such a material compared to Ruthenium), as well as there are no corrosion- and erosion-resistant coatings, then while switching dry circuits (without load), it has to be expected that a bridge erosion will appear. This is observed in case of untreated contacts (Figures 5,6).

3.2. Auger Spectrum Investigations

Elemental composition of the contact surface (before and after treatment) was analyzed using an Auger electron spectrometer [22]. The device is equipped with a cylindrical mirror electron energy analyzer (resolution 0.25%) with an integrated electron gun having a beam current up to 1 µA at a beam diameter of 100 µm. To clean the surface of contact blades of reed switches and for layer-
by-layer Auger analysis thereof, an ion gun, with a differential pumping of working gas (Ar) and ion current density up to 3.5 mA/cm$^2$, was used. All measurements were performed in vacuum of $2 \times 10^{-7}$ Pa to eliminate effects of electron-stimulated adsorption of residual gas molecules on surfaces being researched.

Results of layer-by-layer Auger analysis of contact near-surface layers of reed switches after ion-plasma surface treatment thereof are shown in Figure 7. In the course of investigations, samples were subjected to sputtering by an ion probe with a simultaneous Auger analysis of primary element lines.

Etching was carried out using Ar$^+$ ions and 2.5 keV energy, in-beam current of 200 µA/cm$^2$. All that provides an ion-beam sputtering rate of 64.2 nm/min across permalloy.

Conclusions on the concentration and phase states as well as thickness of modified layer were drawn according to the conventional schedule: based on amplitude measurement of differentiated Auger lines of atoms, relation thereof, line profile stability in depth and shape of the lines which specify atomic Auger processes depending on atom coordination environment in various phase states.

Since a concentration stabilization of nitrogen atoms for depths from 14 up to 26 nm and from 58 up to 90 nm (Figure 7) is observed, when it is different in each case, and a shape of Auger line is typical for iron nitrides [21], it was assumed that all nitrogen atoms form a single-phase bonding only with iron atoms, but with various stoichiometry for every stabilization zone.

System solution: $C_{Fe} + C_N = 100$ [atom. %] and $C_{Fe}/C_N = K$ for two values obtained by calculation $K = 2.7$ and 4.07 (two stabilization zones) allows estimating a concentration of nitrogen atoms without regard to other present elements (Ni, C, and O), what is shown in Figure 8 in combination with nitrogen atomic concentration for stoichiometric compounds of Fe$_3$N and Fe$_4$N.

Due to the examination, a zonal, typical for intermetallic phases, distribution of atoms composing the sample, including doping nitrogen in depth, is revealed. This is the first transition-type zone about 14 nm in thickness (see fracture observed on atomic profiles) which is identified by a lateral nano-roughness of relief of surface atomic layers and by a crater effect known in probe ionic diagnostics (nonuniform ionic layer-by-layer etching across surface). All the above considered, the following zone specifies a stable stoichiometric relation of the main elemental components of heterogeneous sample, namely Fe and N, with a typical equidistant scaling. Regular breaks in concentration profile justify this assumption which comes into the open from transformations of shape of Auger lines that are typical for nitride phases, as shown below in Figure 7. In accordance to the data from scientific and technical literature, zone 2 is to be identified with nitride phase of Fe$_3$N type. It will be replaced (on the basis of typical breaks in profile of layer-by-layer ionic etching, line shape, and typical chemical displacements of Auger transitions) by Fe$_4$N phase which is regularly alloyed using interatomic additives of excess oxygen and carbon (Figures 7, 8).

Typical breaks and cophased nature of concentration decline on layer-by-layer atomic profiles (C, O) at a simultaneous relative stability of amplitudes of
lines of Ni atoms allow ascertaining a heterogeneous lamination of phase state of examined samples which can be interpreted as nickel carbides and oxides. The findings conform to the working data [21].

Characteristics of Fe-Ni contact blades examined at atomic and molecular level, subjected to activation doping in a low-temperature plasma of nitrogen, specifically: transient resistance, potential surface relief (provided by attained values and lateral distribution of electron output work in a region of overlapping contact blades, and therefore, by a contact potential difference in conduction bands), effective mechanical and erosion wear-resistance, congruous stability of phase state of reed switch blades in bench and performance tests – all that is intentionally achieved due to reproduction processes of singular phase structure in range of nanoscaling.

Thereby, the results of integrated researches with corpuscular probing methods and received physical-chemical characteristics combined with reached results of comprehensive tests suggest on a purposeful reproduction of a new reed switch class unknown in world practice, commercial development thereof will permit to expand a market segment of the Russian science-intensive industrial products.

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**Figure 7.** Elements concentration profile in surficial region of ion-plasma treated reed switch and shape of nitrogen Auger line, typical for nitride phase (E – kinetic energy of electrons; N – quantity of electrons with energy E).
Figure 8. Regenerated phase distribution profile in depth specifying nitrogen concentration in stoichiometric compounds of Fe$_3$N and Fe$_4$N.

3.3. AFM- Research

In Figure 9 AFM-image of nitrided surface of reed switch contact spring is provided, that has grain boundaries and nanorelief thereon. One can see well interchange of dark and light round-shaped nanosized regions that points to the change of surface relief height. Profile of AFM-image and statistical analysis of height distribution, provided in Figures 10 b and 10 c, respectively, give additional and detailed information on the obtained relief.

Statistical analysis has revealed that an average peak height is 50 nm. Lateral dimensions of peaks at the bottom were 70-100 nm. On the average, width of peak at half height is 70 nm.

For the first time depictions of reed switch contact surface after ion-plasma treatment within the reflection of diffluence resistance were obtained (Figure 11), that is a positive evidence of the presence of special conductive properties of nanopeaks.

Before our researches there were no data about the formation of such nanostructures on a permalloy surface as a result of nitriding.

Contacting in reed switches always takes place in separate points on some areas because it is impossible to make and form a construction so, that contacting could be realized across the whole contact surface. In fact the number of contacting points is limited and their position changes during reed switch operation. With foreign dielectric particles are within contact region between
two contacting planes, transient resistance increases because a gap is formed between contacting planes at once, and the number of contacting points may considerably decrease.

Our usage of special nitriding technology creates required properties of contacting surfaces of reed switch, improves stability its operation in different modes during long period of time, simplifies technology of contact creation, eliminates influence of foreign particles on contact properties and usage of comparatively “dirty”, labour-intensive and complicated to use galvanic technologies of deposition of precious-metal-based coatings. Galvanic technology is one of the main sources of foreign particles in reed switch that leads to device failures in the process of operation. A new galvanic technology, that eliminates the usage of such main source of foreign particles, significantly improves reliability of reed switch operation. Level of failures (number of faulty reed switches under testing is 1 million pieces), with application of the new technology, decreases from 200 to 50 ppm and less (about 4 times).

Surface nanostructure (nanorelief with peaks of a higher conductivity) provides realization of reliability principle - multilevel echeloned protection of contact surface from corrosion, erosion and mechanical damages, increases amount of contacting points, improves noise immunity from foreign particles, and finally solves a problem of reed switches quality, substantially decreasing amount of service failures.

![AFM image](image.png)

**Figure 9.** AFM – image of reed switch contact spring surface nitrided in discharge, field width 7 x 6 µm.
Figure 10. a) AFM – image of reed switch contact spring surface nitrided in discharge, field width 2x2 µm; b) profile of AFM - image in X-direction along a solid line 175 (see a); c) statistical distribution of relief height on surface area of reed switch contact spring (see a).
Figure 11. AFM-image of nitrided surface area of reed switch contact spring: a, c – method of constant effective force; b, d - method of reflection of diffluence resistance.

Origination of defective-deformative instability is shown in a number of researches devoted to investigation of results of energy deposition on surface of condensed matters, namely, laser irradiation, ion fluxes, and gradient fields [24, 25]. This stipulates realization of critical conditions for display of synergistic effect that leads to the development of surface structures of relief. Particularly, in our case, combination of temperature of permalloy surface, irradiation
intensity and duration by nitrogen ions, forms a surface relief in the form of system of nanostructured peaks, as a result of atomic synergistic self-assembly.

4. Conclusion

Principally new technological process of modification of Reed Switch (RS) contacting surfaces was developed, that is after sealing of contact springs into a glass tube filled with nitrogen, across RS being opened, current impulses are passed which cause formation of nanosized layers with the specified contact features in a near-surfacial region of RS.

As a result of these researches we have managed to create such a discharge electrophysical condition at which corrosion- and erosion-resistant nanosized high-conductivity layers are formed in the near-surfacial region of permalloy contacts of reed switches. In our opinion, it enables not to use the galvanic technology of the special precious-metal based coatings.

Profile of element concentration distribution in near-surfacial region and images of nanostructured contact peaks (50nm) (Figures 9-11) on new heterogeneous Fe$_3$N (30nm) and Fe$_4$N (40nm) base (Figures 7, 8), obtained by atomic-force microscope and electronic Auger spectrometer, indicate the formation of nanostructured surface contact layer. Moreover, cardinal change of contact layer characteristics also indicates the achievement of nanostructured states of near-surfacial atomic layers.

Surface nanostructure (nanorelief with conductive peaks) provides realization of high reliability principle - multilevel echeloned protection of contact surface from corrosion, erosion and mechanical damages, increases amount of contacting points, improves noise immunity from foreign particles, and, finally, solves a problem of reed switches quality, substantially decreasing amount of service failures.

So, one of the main problems of modern technology of reed switch production – relatively high level of service failures – has been overcome, that will not only improve reed switch reliability but also significantly enlarge the range of its application, competitive capacity and enable to expand the segment of world market of Russian science-intensive commercial nanoproducts.

The developed modification process was tested during the commercial manufacture of the principally new reed switch sample with nanostructured contact surfaces (MKA 14108 type) (see Figure 1).
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