








IMPACT OF RUTHENIUM DIFFUSION ON THE ELECTRICAL PROPERTIES OF THICK FILM RESISTORS

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The diffusion profile of the RuO₂ into silicate glass and the electrical resistance distribution across diffusion layer have been studied by beveled sample method and energy dispersion spectroscopy. The distribution of content of Ru atoms in the diffusion layer is described by the $\text{erfc}(x)$ what means that the diffusion coefficient is independent of the content of Ru atoms. The correlation of the distribution of Ru atom content and the resistance distribution in the diffusion layer showed that it is the diffusion doping of glass that is responsible for the conductivity of thick-film resistors. Thickness of the diffusion layer is more than 100 μm while average distance between RuO₂ particles is about 0.5-2 μm . It means that all volume of the thick-film resistor comes conductive in firing process at 850°C in 10 minutes.

Keywords: Beveled sample; Diffusion layer; Diffusion coefficient; Resistance distribution; Diffusion profile

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INTRODUCTION

The conduction mechanism of thick film resistors has been the subject of research for more than 50 years [1-15]. The main efforts of researchers were aimed at explaining the mysterious minimum in the temperature dependence of resistance. Variable range hopping (Mott mechanism), percolation, tunneling through a thin layer of glass between crystalline dopant particles (conducting phase, mainly RuO₂ or ruthenates) and combinations of these mechanisms have been proposed. All these proposals are based on the structure of thick-film resistors observed in an electron microscope and X-ray diffraction patterns, in which crystalline particles of the conducting phase are distributed almost uniformly in a glass matrix. Unfortunately, all these proposals could not explain experimentally observed temperature dependence of resistance, including “metallic” conductivity [13-15].

The maximums of resistance and Seebeck coefficient of thick-film resistors at a temperature of about 1000 K [16] have not attracted researchers.

In [17, 18], based on a set of experimental data, another conductivity mechanism for thick-film resistors was proposed, suggesting 1) the existence of nanocrystals in the glass itself; 2) structural transitions in nanocrystals at high temperatures; 3) diffusion of atoms of the conducting phase into the glass during sintering and the formation of an impurity zone near the top of the valence band of the glass; 4) the conductivity of a thick-film resistor is the sum of activation conductivity along the impurity band and hopping conductivity across nanocrystals. This mechanism made it possible to qualitatively explain the temperature dependence of the conductivity of thick-film resistors in the temperature range from liquid helium to 1100 K.

However, the assumption about the correlation between the diffusion of atoms of the conducting phase into glass and the conductivity of the glass itself has not been confirmed experimentally.

The aim of this article is to experimentally confirm the correlation of the diffusion of atoms into glass and the electrical conductivity of the glass itself.

EXPERIMENTAL METHOD

Sample of glass for experiments has a composition (mass %) SiO₂ – 33; PbO – 67 and was boiled at a temperature 1773 K for 1 hour and cast into a steel mold with dimensions 20×4×4 mm. Cooled glass sample was annealed at 723 K for 3 hours with cooling in a switched off furnace. The opposite wide faces of the sample were grinded parallel to each other and polished to mirror finish. A layer of RuO₂ from an aqueous suspension (1 mass %) was deposited on one of the wide faces of the glass prism for 1 min and dried at 423 K for 1 hour. RuO₂ diffusion into glass was carried out at 923 K for 5

hours (Fig. 1a). This low diffusion temperature was chosen to avoid softening the glass and changing the shape of the sample. After diffusion, the sample was polished at an angle of 0.5 degrees to the glass surface (beveled sample, Fig. 1b).

At small polishing angles about 0.5° of a beveled sample, the distance x deep into the diffusion layer is related to the distance l along the surface of the sample as $x = l \tan \varphi \approx l \cdot \varphi = l \cdot \varphi \cdot \pi / 180^\circ \approx l \cdot 0.5^\circ / 57.3^\circ \approx l / 115$, i.e. the diffusion length appears to be increased by approximately 115 times, and a displacement of 0.5 mm along the l axis (Fig. 1b) corresponds to a displacement along the x axis of $4.4 \mu\text{m}$. This makes it possible to correlate the measured resistance distribution $R(l)$ along the glass surface to the resistance distribution $R(x)$ deep into the diffusion layer and compare the profiles of the distribution of atoms in the diffusion layer with the resistance distribution there.

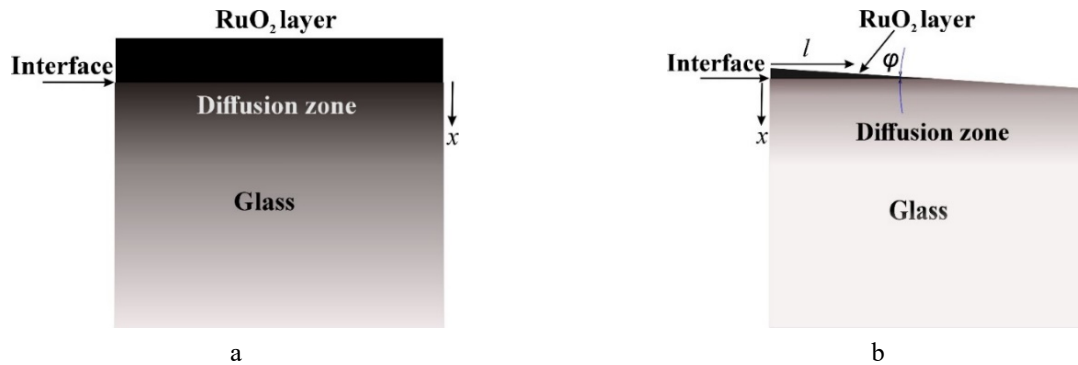


Figure 1. Sample of the glass after diffusion (a) and the beveled sample (b)

The distribution of Ru atoms along and across the glass surface was measured on a JEOL JSM-IT200 scanning electron microscope with energy dispersive spectrometer (Uzbek-Japanese Innovation Center of Youth, TSTU, Tashkent, Uzbekistan). The resistance R_s distribution along the sample surface (Fig. 2) was measured using a simple-probe (spreading resistance probe – SRP) method [19, 20] with a digital multimeter Rigol DM3058E. The radius a of the tip of the tungsten carbide probe was about $50 \mu\text{m}$. The resistivity ρ of the diffusion layer was calculated from the measured resistance R_s as $\rho = 4aR_s$ [19, 20].

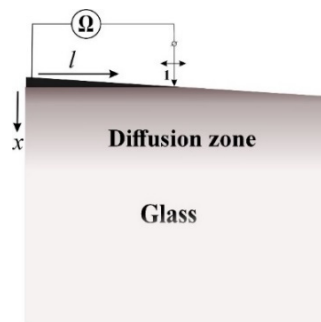


Figure 2. Simple-probe method.

1 – mobile probe, Ω - ohmmeter (multimeter). Noise of the energy dispersive spectra was filtered by Fourier and inverse Fourier transform in Wolfram Mathematica 13 program, which also used to design all graphs

RESULTS AND DISCUSSION

Two types of experiments were carried out to determine the diffusion profile of Ru atoms into glass. In the first experiment, the diffusion profile was measured across the interface between the RuO_2 layer and the glass surface (Fig. 3).

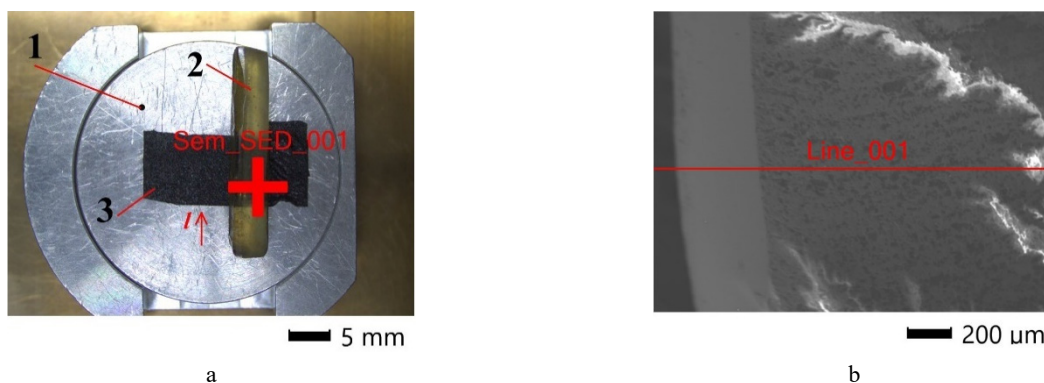


Figure 3. The sample of the glass in the SEM chamber (a) and the scanning line (b): 1 – sample holder; 2 – glass sample; 3 – two-sided conductive scotch tape. The scanning line in the Fig. 3b is perpendicular to the plane of Fig. 3a (along the x -axes in Fig. 2)

The sample of the glass after diffusion is shown in Fig. 3a in the SEM chamber and the scanning line for EDS analysis is in Fig. 3b. This experiment is basic for comparison with the profile, obtained in the second experiment – along the glass surface in beveled sample. Such a comparison is necessary due to the fact that it is impossible to measure the resistance distribution in a diffusion layer with a thickness of units and tens of micrometers, and to obtain this distribution we are forced to use a beveled sample.

The original distribution of the EDS spectra of components in the diffusion layer (across the interface between the RuO_2 layer and the glass surface, line 001 in the Fig. 3b) are shown in the Fig. 4. Note that the number of counts (intensity) in Fig. 4 and below is proportional to the concentration of the corresponding atoms (oxygen, silicon, lead and ruthenium).

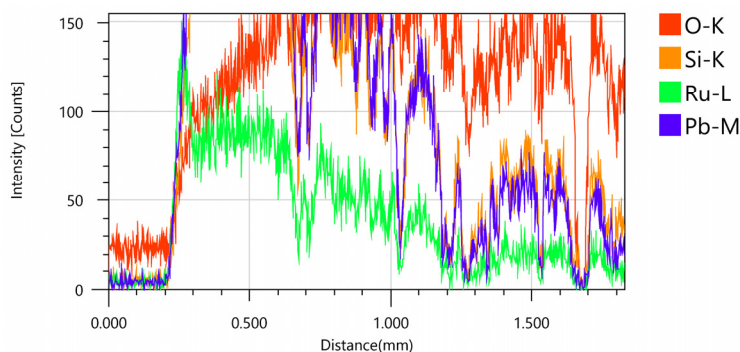


Figure 4. The original EDS spectra of the sample components along the line 001 in the Fig. 3b

The total content of components in the diffusion layer is given in the Table. The content of SiO_2 and PbO oxides, recalculated from these data, corresponds to the initial composition of the glass, while the RuO_2 content is about 4 wt. %. Last fact contradicts to conclusion [21, 22], that solvability of RuO_2 in a silicate glass is less 10^{-4} atomic %, and conforms results of Abdurakhmanov [23], Flachbart et al. [24].

Table. The total content of the sample components from EDS analyses

Element	Line	Mass%	Atom%
O	K	44.38±0.15	82.47±0.28
Si	K	10.05±0.06	10.64±0.06
Ru	L	2.36±0.09	0.69±0.03
Pb	M	43.20±0.21	6.20±0.03
Total		100	100
Line 001 wholespectrum		Fitting ratio 0.2875	

Apparently, the conclusion about the weak solubility of ruthenium dioxide in silicate glass arose from studies of the solubility of metallic ruthenium in various silicate melts, including those used for nuclear waste disposal [25-27]. Indeed, pure metals are poorly soluble in silicate melts, but the solubility of oxides of the same metals can be tens of wt. %. For example, the solubility of metallic lead in a silicate melt is hundredths of a percent, while PbO with SiO_2 forms homogeneous glass with a PbO content of up to 90 wt. %. The glass we study in this article contains 67 wt. % PbO .

EDS spectrum of Ru with very intensive noise (Fig. 4) after filtration is shown in Fig. 5. The wide double maximum at $0.25 \text{ mm} < x < 0.55 \text{ mm}$ corresponds to the RuO_2 layer (see Fig. 3b). Distribution of the Ru atoms in diffusion layer, described as $I(x) = 10 + 320 \operatorname{erfc}(1.8 x)$, indicates that the diffusion coefficient is constant (does not depend on the concentration of ruthenium atoms at the point in question).

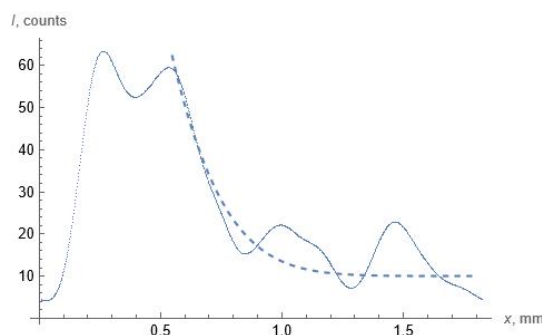


Figure 5. Filtered EDS spectrum of Ru across the diffusion layer (see Fig. 4).
The dotted line is the approximation by the function $10 + 320 \operatorname{erfc}(1.8 x)$

It is also seen that the diffusion length in the glass (about 0.335 mm) under diffusion conditions (873 K, 5 h) significantly exceeds the thickness of the glass layer (about 25 μm) and the diameter of the glass powder particles

(0.1-0.2 μm) in thick-film resistors. Standard firing duration of the thick-film resistors at peak temperature is $\tau_s = 10$ min, so diffusion length l_d will be shorter: $l_d = 0.335\sqrt{\tau_s/\tau} = 0.06$ mm. This once again confirms the conclusion of our previous studies [23, 30] that during sintering the entire volume of glass is doped almost uniformly and becomes conductive.

In the second experiment, the distributions of the concentration of ruthenium atoms $I(l)$ and the spreading resistance $R(l)$ along the beveled sample were measured (Fig. 1b and Fig. 6). Since the length of the beveled sample is more than 15 mm, and the maximal electronic scanning zone in the SEM is 3 mm, the total EMF was compiled by stitching together 5 separate spectra (Fig. 6). This spectrum has been filtered by Fourier transform as well (Fig. 7).

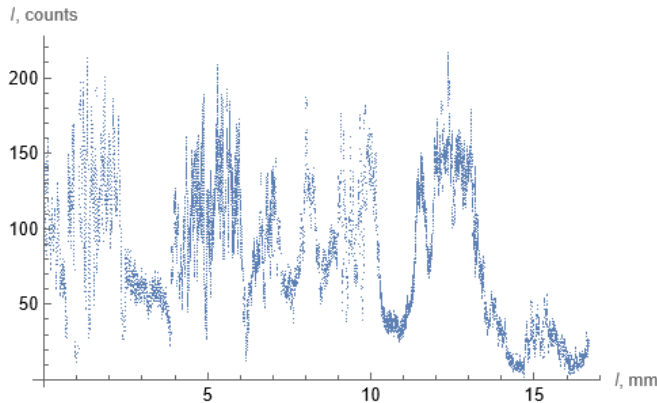


Figure 6. The original EDS spectrum of the Ru along the diffusion layer, stitched from 5 separate scan sections

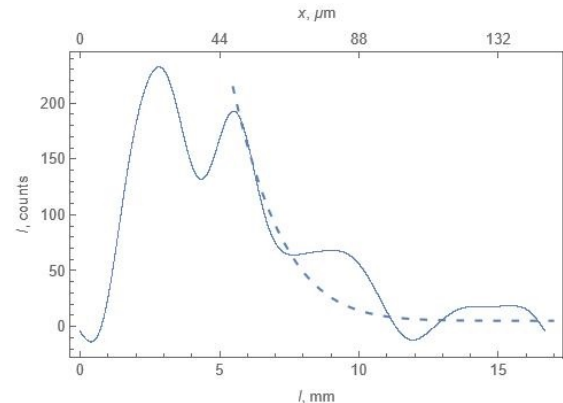


Figure 7. The filtered EDS spectrum of the Ru along the diffusion layer. The dotted line –function $5 + 1600 \operatorname{erfc}(0.195 l)$. The meaning of the lower and upper scales, see Fig. 1b

One can estimate the diffusion coefficient $D(873 \text{ K})$ from the data in Fig. 7, keeping in the mind that $\operatorname{erfc}(0) = 1$ and $\operatorname{erfc}(1) = 0.1559$. The initial intensity of the EDS spectra $I_0 = I(x = 0.53) \approx 55$ counts, therefore the intensity $I_0 \cdot \operatorname{erfc}(1) = 55 \cdot 0.1559 = 8.57$ is achieved at the point $x = 1.2 - 0.53 = 0.67$ mm. Taking into account the fact that the argument of the function $\operatorname{erfc}(z)$ is equal to $z = x/2l_d$, we have $l_d = x/2 \approx 0.335$ mm. Then $D(873 \text{ K}) = l_d^2/\tau \approx (3.35 \cdot 10^{-4})^2/18000 \approx 6.2 \cdot 10^{-12} \text{ m}^2/\text{s}$, which is in good agreement with data on diffusion in oxide glasses [28, 29].

The rescaled distribution of the Ru atoms in Fig. 7, described as $5 + 1600 \operatorname{erfc}(0.195 l)$, agrees well with the similar distribution in Fig. 5. The double maximum at $0.25 \text{ mm} < x < 0.55 \text{ mm}$ corresponds to unpolished layer of RuO_2 (see above on Fig. 5). This confirms the possibility of using a beveled sample to establish a correlation between the concentration of ruthenium atoms and the resistivity distribution in the diffusion layer.

Distribution of resistivity in the diffusion layer

Distribution of spread resistance $R_s(l)$ and resistivity $\rho(l)$ along beveled sample is shown in Fig. 8. This distribution consists of two parts: 1) a linear part at $x < 5$ mm, corresponding to the unpolished RuO_2 layer on the glass; 2) nonlinear part at $x > 4$ mm, described by the function $R_s(l) = 0.9 + 0.185 l / ((1 - 0.065 l) \operatorname{erfc}(0.023 l))$.

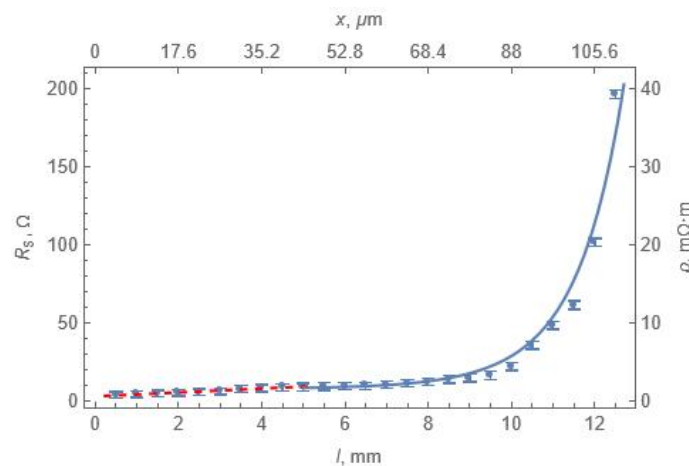


Figure 8. The spread resistance R_s and resistivity ρ distribution along (lower scale) and across (upper scale) the diffusion layer (Fig. 2). The dotted line is the function $R_s(l) = 0.0011 + 0.551 l$, the solid line is the function $R_s(l) = 0.9 + 0.185 l / ((1 - 0.065 l) \operatorname{erfc}(0.023 l))$. See text for explanations of these functions

We are not interested in the linear part of $R(l)$ in the context of this article, and let us consider its second part in more detail. The nonlinear function $R(l)$ above may be expressed as

$$R_s(l) = 0.9 + 0.185l(1 - 0.065l)^{-1}/\operatorname{erfc}(l/\sqrt{D\tau}), \quad (1)$$

with $1/\sqrt{D\tau} = 0.023 \text{ mm}^{-1}$. Here coefficient 0.185 includes all the constant parameters of the experiment, i. e. radius a of the movable probe for the spread resistance $R_s = \rho/4a$, geometric factors (namely, the constant width of the beveled sample), elementary charge e and the mobility of charge carriers (holes) μ_h , etc. The material resistivity ρ is considered to be inversely proportional to the charge carrier concentration p , which in turn is proportional to the concentration of ruthenium atoms or intensity of EDS spectrum for Ru (Fig. 6). This consideration is expressed by the multiplier $1/\operatorname{erfc}(l/\sqrt{D\tau})$.

The factor $l/(1 - 0.065l)$ reflects a change in the geometry of the beveled sample – an increase in the distance between the fixed contact and moving probe (l in the numerator) and a decrease in the cross-section of the sample with distance ($1 - 0.065l$ in the denominator) in accordance with the definition of the conductor resistance $R = \rho \cdot l/s$ (s is the cross-sectional area of the conductor).

The distribution of ruthenium atoms in the diffusion layer (Fig. 5 and 8) and the distribution of resistivity in the same layer (Fig. 9) are in good agreement with each other. This means that the entire volume of the thick-film resistor is involved in electrical conductivity, but not only conducting chains (endless clusters) formed from dopant (conducting phase) particles.

The fact that the entire volume of a thick-film resistor is electrically conductive is also evidenced by an experiment with laser scribing along current lines [30] and a study of the distribution of piezosensitivity on the nanoscale across the interface glass-RuO₂ layer [31-33].

Totokawa et al. [31-33] have showed that thin film of bismuth-borosilicate glass, doped with ruthenium, 1) contains trivalent as well as tetravalent states of ruthenium; 2) exhibits electrical conductivity, that can be described as variable range hopping; 3) has high strain sensitivity due to the spatial expansion of wave functions of charge carriers (holes) in localized states; 4) ruthenium atoms diffuse into the glass during firing; 5) the diffusion coefficient of Ru into bismuth-borosilicate glass is about $1.4 \cdot 10^{-13} \text{ m}^2/\text{s}$ and the diffusion length is about 100 nm; 6) piezoresistive properties depend on the distance from the interface of layers of glass and RuO₂.

It should be noted based on the results of works [31-33] that the dependence of piezoresistive properties on the distance from the interface between glass and RuO₂ layers indicates a correlation of these properties with the concentration of Ru atoms, but the distribution of resistance depending on this distance has not been studied.

Abe et al. [34, 35] studied the diffusion of atoms from a RuO₂ layer into a glass and vice versa, glass atoms into a ruthenium dioxide layer, using energy dispersive spectrometry. It turned out that ruthenium atoms diffuse into glass to a depth of more than 1 μm , while the diameter of glass particles in pastes for thick-film resistors is less than 1-2 μm , i.e. the entire layer of glass between the dopant particles is doped fairly uniformly. However, the correlation between such glass doping and the electrical conductivity of resistors has not been studied by these authors.

The series of temperature characteristics of resistance for thick film resistors and their analyses from the point of view of possible mechanisms responsible for $R(T)$ are discussed in [13, 36]. The consideration is carried out mainly with an emphasis on the metallic conductivity of RuO₂, as in many other publications [1-15], [37-41] and therefore sufficient correlation with experimental data was not achieved.

The authors hope that the results presented here can help in elucidating the role of glass phase and dopant particles in the mechanism of electrical conductivity of thick film resistors.

In the future, it is necessary to study in more detail the diffusion of transition metal atoms from their oxides into glass for a better understanding of the mechanism of electrical conductivity of thick film resistors.

CONCLUSIONS

The experiment showed that in silicate glass the distribution of the concentration of ruthenium atoms $N(x)$ and the electrical conductivity $\sigma(x) = 1/\rho(x)$ along the depth of the diffusion layer obey the same law $\operatorname{erfc}(x)$. This confirms that

1) the diffusion coefficient D of ruthenium atoms in silicate glass is constant and does not depend on their concentration;

2) due to the diffusion of ruthenium atoms, the glass itself becomes electrically conductive;

3) the electrical conductivity of glass is proportional to the concentration of ruthenium atoms. At 873 K over a duration of 5 hours, the diffusion length of Ru atoms in lead-silicate glass was determined to be $l_d = 0.335 \text{ mm}$ based on energy-dispersive spectroscopy data. This value significantly exceeds the diameter of the glass particles typically used in thick-film resistor pastes, indicating that the entire glass volume is uniformly doped. The corresponding diffusion coefficient was estimated to be $D(873 \text{ K}) = 6.2 \times 10^{-12} \text{ m}^2/\text{s}$, in good agreement with known values for diffusion in oxide glasses.

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Conflict of interests

The authors declare that there are no conflicts of interest.

Authors contribution

A. Dekhkonov: First author, Investigation, Methodology, Calculation of results, formal analysis, Visualization, Validation, Writing-draft, editing draft.
 G. Abdurakhmanov: Supervision, Provide Resources, Investigation, Methodology, Calculation of results, formal analysis, Visualization, Validation, editing draft.
 M. Tursunov: Investigation, Methodology, Calculation of results, formal analysis, Visualization, Validation, editing draft.
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 G. Vokhidova: Investigation, Methodology, Calculation of results, formal analysis, Visualization, Validation, editing draft.
 D. P. Rai: Investigation, Methodology, Calculation of results, formal analysis, Visualization, Validation, editing draft, Submission.
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ВПЛИВ ДИФУЗІЇ РУТЕНІЮ НА ЕЛЕКТРИЧНІ ВЛАСТИВОСТІ ТОВСТОПЛІВЧНИХ РЕЗИСТОРІВ

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Профіль дифузії RuO₂ у силікатне скло та розподіл електричного опору по дифузійному шару досліджували методом скошеного зразка та енергетично-дисперсійної спектроскопії. Розподіл вмісту атомів Ru в дифузійному шарі описується $erfc(x)$, що означає, що коефіцієнт дифузії не залежить від вмісту атомів Ru. Кореляція розподілу вмісту атомів Ru та розподілу опору в дифузійному шарі показала, що саме дифузійне легування скла відповідає за провідність товстоплівкових резисторів. Товщина дифузійного шару становить понад 100 мкм, а середня відстань між частинками RuO₂ становить близько 0,5-2 мкм. Це означає, що весь об'єм товстоплівкового резистора стає провідним в процесі випалу при 850°C протягом 10 хвилин.

Ключові слова: скошений зразок; дифузійний шар; коефіцієнт дифузії; розподіл опору; дифузійний профіль