

## Original Research Article

### IMPURITY BANDS AND DENSITY OF STATE IN DOPED SILICATE GLASSES WITH METAL OXIDES (RuO<sub>2</sub>, CuO, MnO<sub>2</sub>)

**Abstract.** In this article, as a result of double alloying with ruthenium, copper and manganese oxides, the formation of an impurity zone in silicate glass and how the density of electron states changes in it was studied by hypothetical and tunneling microprobe spectroscopy. The results of the experiment were processed using Wolfram Mathematica 11 software. It was found that around room temperature, the impurity zone touches or merges with the valence zone of the glass. At a high temperature (around 1000 K), the impurity zone separates from the valence band of the glass, and a pseudo-gap appears between them. As a result, doped silicate glass exhibits metallic conductivity at room temperature, but at high temperatures it has semiconductor (activation) conductivity.

**Key words:** pseudo-gap, thick film resistors, ligature atoms, impurity zone, electrical conductivity, tunneling spectroscopy, thermoEMF coefficient, insulator-metal and metal-insulator transitions

#### INTRODUCTION

Ruthenium oxide-doped silicate glass (DSG) is widely used in electronics and instrumentation as a "thick film resistor" [1], and is expected to be a new thermoelectric material [2]. In this material, the change in electrical conductivity over temperature is strongly different from that of metals and semiconductors (Fig. 1). But in almost 50 years of research, no information was found about the density of states  $g(E)$  in DSG excluding [3], while its numerical value and energy distribution along  $E$  are used in all physical processes involving electrons (holes) (electricity and photoconductivity, formation of thermal and photoelectric power, metal-insulator transitions) play a crucial role [4]. Jacoboni and Rizzi [3] used  $g(E)$  in the form

$$g(E) = \frac{1}{2\pi^2} \left( \frac{2m^*}{\hbar^2} \right)^{3/2} \sqrt{E} \quad (1)$$

only for theoretical calculation of conductivity.  $\hbar$  is Planck's constant,  $m^*$  is the effective mass of electrons. In this article, some properties of the impurity zone formed due to diffusion of metal oxides ( $\text{RuO}_2$ ,  $\text{CuO}$ ,  $\text{MnO}_2$ ) into lead silicate glass as well as the density of states in it are analyzed using tunneling spectroscopy.

### Theoretical analysis

The change in the resistivity of DSG over temperature can be symbolically divided into three regions (Fig. 1): at low temperatures (region I)  $\rho(T) \sim \exp(T_0/T)^\gamma$ ,  $\gamma = 0.4-0.8$  (mainly  $\gamma = 0.5$ ); a minimum appears around room temperature (part region II), then "metallic" ( $\rho \sim T^\zeta$ ,  $\zeta = 1-2$ ) conductivity is observed. At very high temperatures (part region III,  $T > 700$  K),  $\rho$  increases sharply (up to 5-6 times) and reaches a maximum, then decreases according to the activation law, as in semiconductors (activation energy  $E_a = 0.5-1.5$  eV, depending on the composition of the glass). The value of thermoEMF coefficient  $S$  in regions I and II ( $T < 700$  K) is close to metals (15-20  $\mu\text{V}/\text{K}$ ) and changes slowly, but at  $T > 700$  K (region III) it increases almost 100 times (until 1700  $\mu\text{V}/\text{K}$ ) [3].

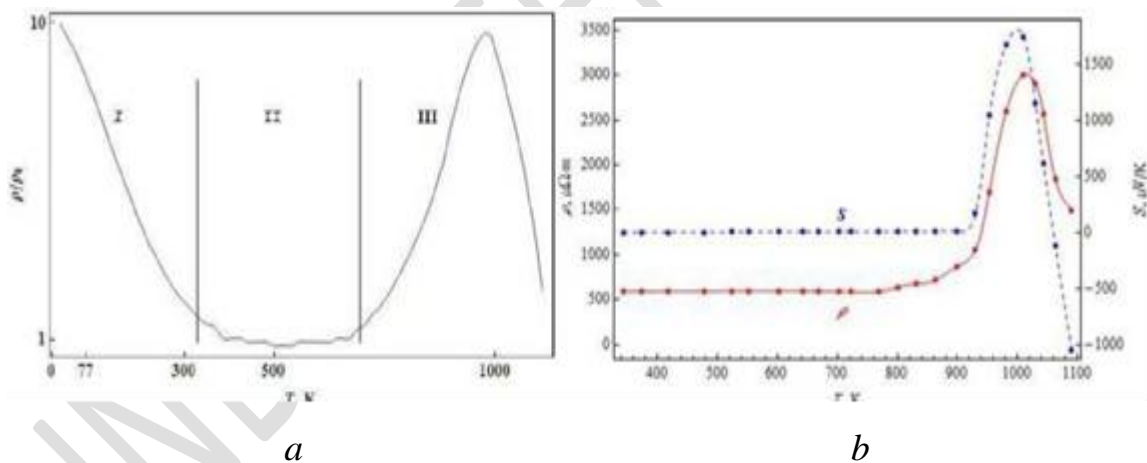


Figure 1. Temperature dependence of resistivity of DSG: schematic (a) and resistivity and thermopower experimental (b)

To determine the density of states  $g(E)$  according to formula (1), first of all, the effective mass of charge carriers  $m^*$  should be determined. According to experiments [3], the main charge carriers in DSG are holes, and the value of the Hall voltage is below the measurement limit. For this reason, only the upper limit of

the mobility of charge carriers ( $\mu$ ) in DSG was estimated by the noise level for samples of differ resistivity  $\rho$  (table 1)( $t$  is the thickness of the sample):

Table 1. The mobility of charge carriers ( $\mu$ ) in DSG estimation by the noise level for samples of differ resistivity  $\rho$

Resistivity of the sample $\rho$ , Ohm·m	Sheet resistivity $R_s = \rho/t$ , Ohm/□	Mobility $\mu$ , $\text{cm}^2/(\text{V s})$	Holes concentration $p$ , $\text{m}^{-3}$
2.5	$10^5$	$\leq 5 \cdot 10^{-3}$	$\geq 5 \cdot 10^{24}$
$2.5 \cdot 10^{-3}$	$10^2$	$\leq 5 \cdot 10^{-2}$	$\geq 5 \cdot 10^{26}$

One can see that the concentration of holes  $p = 1/(e\rho\mu)$  in DSG is high and the scattering is very strong, and the external magnetic field does not significantly affect their trajectory in the distance of free movement, as a result, the Hall voltage is very small. Here,  $e$  is the elementary charge, and  $t$  is the thickness of the DSG layer. Now we determine the lower limit of the concentration of holes -  $p$  in these samples:  $p = (\rho e \mu)^{-1} \geq 5 \cdot 10^{24} \text{ m}^{-3}$  and  $p \geq 5 \cdot 10^{26} \text{ m}^{-3}$ , respectively. So, in DSG, the gas of the pits is nauseating. According to the information given in [3] about the kinetic properties of DSG, the mobility of charge carriers is  $\mu = \varepsilon/m^*$ ,  $t = l/v$  is the time of free movement of charge carriers,  $\lambda$  is their average free movement distance, and  $v$  is heat average speed corresponding to energy (usually around 105 m/s). We can take  $\lambda \sim 10\text{-}20 \text{ \AA}$  (average distance between ligature atoms in glass). Then  $m^* = (320\text{-}330)m_0$ , that is, DSG is a system with heavy fermions.  $m_0$  is the mass of a free electron. If we put these numbers in the formula (1) (assuming that  $E \sim kT$ ),

$$g(E) = \frac{1}{2\pi^2} \left( \frac{2m^*}{\hbar^2} \right)^{3/2} \sqrt{E} = 1.1 \cdot 10^{26} \text{ eV}^{-1} \text{ sm}^{-3}. \quad (2)$$

It seen that the density of states of holes in the impurity band of DSG is very high, and this material can be a good thermoelectric material [2]. To prove this idea, we calculate the width of the impurity band  $J$  [4]:

$$J = \frac{\hbar^2}{ma_0^2} \quad (3)$$

Here,  $a_0$  is the lattice constant (in LSSh, it can be taken as 18-20 Å - the distance between ligature atoms). As a result,  $J = 23$  meV. Under the influence of heat, the impurity band moves towards the valence band of the glass with a speed of about  $10^{-4}$  eV/K, and around 300 K they combine to form "metallic" conductivity [1,6]. Accordingly, all impurity atoms are ionized at room temperature. The thermoEMF coefficient  $S$  of three-dimensional disordered materials is related to the density of states  $g(E)$  as follows [5]:

$$S(T) \approx \frac{\pi^2 k^2 T}{3e} \left( \frac{d \ln g(E)}{dE} \right)_{E=E_F} = -\frac{\pi^2 k^2 T}{3e} \left( \frac{d \ln g(E)}{dE} \right)_{E=E_F}. \quad (4)$$

It can be seen from this formula that for  $S$  to be large, the numerical value of  $g(E)$  should be small around the Fermi energy, but change sharply. The density of states in valence and conduction bands is small (0.1-10 μV/K) in metals due to formula (1), because  $E_F \gg kT$ .

$$\left( \frac{d \ln N(E)}{dE} \right)_{E=E_F} = \frac{1}{2E_F} \text{ and } S(T) \approx \frac{\pi^2 k^2 T}{6eE_F} \quad (5)$$

The appearance of the minimum of  $\rho(T)$  in region II of Fig. 1 was based on the fact that the impurity band merges with the valence band of the glass under the influence of electron-phonon coupling [3]. The impurity band moves along the temperature with a speed of (2-5)· $10^{-4}$  eV/K [6]. Accordingly, at  $T = 0$  K, the impurity band is located at a distance  $E_g \approx 300 \cdot (2-5) \cdot 10^{-4} = 0.06-0.15$  eV from the ceiling of the valence band of the glass. After the maximum in region III of Fig. 1,  $\rho(T)$  changes according to the activation law as in semiconductors, and depending on the composition of the glass and alloying conditions, the activation energy ranges from 0.1 eV to 1.77 eV [7]. Taking into account that the width of the band gap in the  $2\text{SiO}_2 \cdot \text{PbO}$  glass is 3.3 eV, and that there are energy states (distribution "tail") that can be occupied by electrons in this gap between the impurity band and the valence band, even if it is small, the width at  $T = 0$  K is  $E_g$  it turns out that the slit is actually not a real gap, but a pseudogap [7].

## Material and methods

The shift of the impurity band causes this band to merge with the valence band around room temperature (Fig. 2b), and as a result, "metallic" conductivity appears in DSG, because the concentration of holes does not change anymore, and the electrical conductivity decreases with temperature the change along is only due to the scattering of the cavity. If the temperature rises further, structural transitions characteristic for silicates begin to occur around 700 K, the distance between atoms increases, the overlap of the wave functions of the cavities decreases, and the impurity band shifts away from the valence band of the glass and moves to the middle of the forbidden band. Around 1000 K, the structural transitions in silicates are completed, the impurity band no longer shifts (Fig. 2c), resulting in the activation conductivity characteristic of semiconductors. In this case, the value of  $E_g$  goes up to 1.5 eV depending on the composition of DSG, and the density of states decreases to an insignificant level, that is, the pseudogap turns into a real gap.

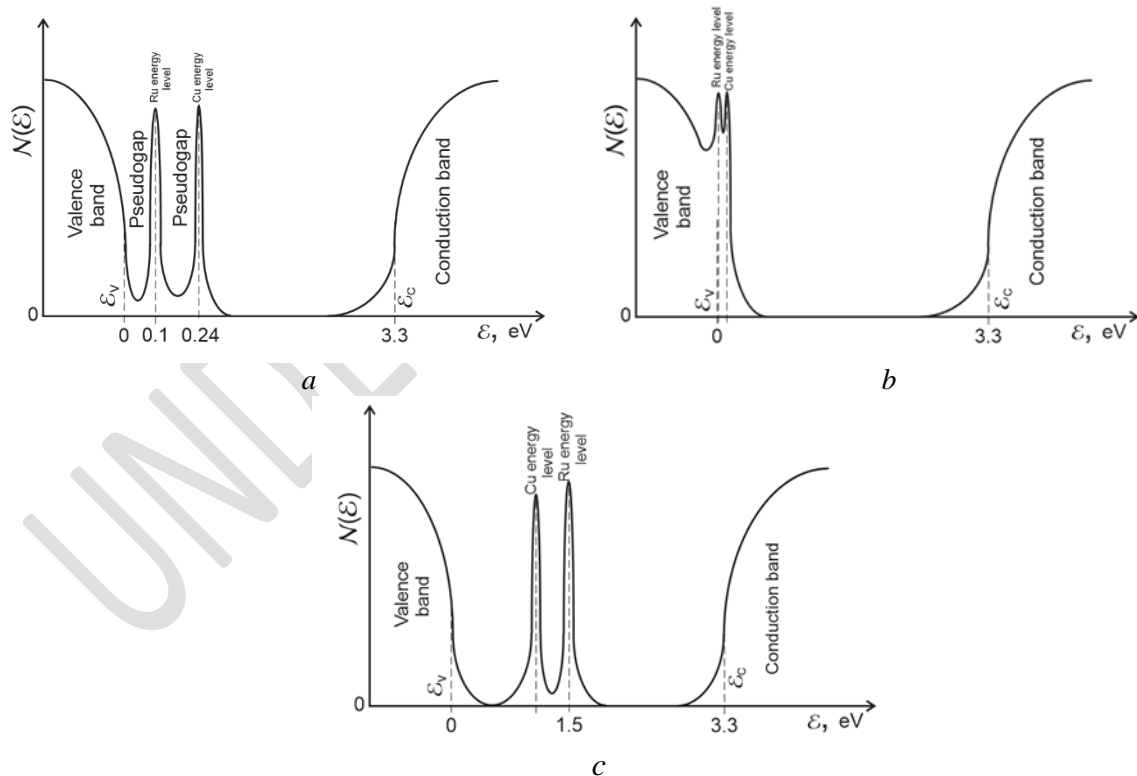


Figure 2. Distribution of the density of states in silicate glass doped with ruthenium and copper at different temperatures  $T$  (K): (a) 0; (b) 300; (c) 1000

In DSG, the location of the impurity band and the density of states in it undergo serious changes under the influence of temperature, as a result of which two

consecutive insulator-metal and metal-insulator transitions occur. The density of electron states  $g(E)$  is one of the factors determining all the electrical properties of a solid (electrical conductivity, photoconductivity, thermoEMF coefficient, etc.). For example, in semiconductors, the concentration of electrons (holes)  $n$  is directly related to  $n(E)$  [8]:

$$n(E) = N_0(E) \exp[-E_a/kT] \quad (6)$$

According to Drude's theory, the energy of free charge carriers inside the substance is expressed as  $E(p) = p^2/2m^*$ , where  $p$  is their momentum. Accordingly, the density of states (for example, the number of states that can be occupied by electrons corresponding to the energy range of 1 eV in the volume of 1 cm<sup>3</sup> of the sample

$$g(E) = 2\pi \left(\frac{2m^*}{\hbar^2}\right)^{\frac{3}{2}} E^{\frac{1}{2}}. \quad (7)$$

In many semiconductors  $(E)$  has been studied well [9], but in doped silicate glass information on the density of states has not been found. According to the formula for the first energy level of ruthenium, the hydrogen-like model of energy levels [10] gave a value of 0.124 eV, where  $m^* = 330m_0$  is the effective mass of charge carriers in DSG,  $m_0$  is the mass of a free electron,  $\epsilon_{st} = 190$  is the measured static dielectric constant of DSG,  $R_{\square} = 13.605$  eV - Rydberg constant.

$$E_1 = \frac{m^*}{m_0} \frac{1}{\epsilon_{st}^2} R_T \quad (8)$$

Now we will try to estimate the density of states in DSG for copper and manganese oxides in a different way. Let's assume that in a number of semiconductor (insulator) materials there is a correlation between the shallow energy levels characteristic of a chemical element. Based on this assumption, before estimating the energy levels of the input in DSG, we should consider how valence and conduction zones appear in lead-silicate glass. In general, electrons in a crystalline solid behave complexly in a periodic field. As a result, the density of states in it (2) is characterized by a relatively complex function and is divided into several parts [11]. The bottom of the conduction band of lead-silicate glass is formed by the

crossing of the 3s orbitals of oxygen and the 6d orbitals of lead (Fig. 1), and the top of the valence band is formed the 2p orbitals of oxygen and the 6p orbitals of lead. As a result, a bandgap with a width of about 3.3 eV is formed in a glass with 2SiO<sub>2</sub>·PbO composition [16].

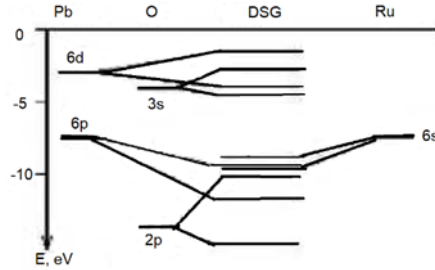


Figure 3. Emergence of energy bands in lead silicate glass

When alloyed with RuO<sub>2</sub>, an impurity band is formed as a result of the splitting of the 6s orbitals of the Ru atom near the ceiling of the valence band of lead-silicate glass, that is, the holes are the main charge carriers in such DSG. The density of states in this zone is  $N_d = 4.5 \cdot 10^{22} \text{ eV}^{-1} \text{ sm}^{-3}$  [3]. The impurity band moves with temperature at a rate of  $(2-5) \cdot 10^{-4} \text{ eV/K}$  [6], near room temperature the gap between the impurity band and the top of the valence band disappears and a "metallic" conductivity originated in DSG. From the results of measuring  $\rho(T)$  in DSG in the range from helium temperature to 1100 K one can say that the gap width between the impurity band and the top of the valence band is 60-150 meV at  $T = 0 \text{ K}$ . In the impurity band, the energy states are generally distributed according to the Gaussian formula, and the edge of this distribution reaches the valence band. A similar distribution applies to the valence band. According to this, the gap between the impurity band and the valence band is actually not a real gap, but a pseudo-gap [7] (Fig. 2a), the concentration of the cavity gas is high and it is variable: the specific resistance is  $\rho \sim 2.5 \text{ Ohm}\cdot\text{m}$  the mobility of holes in the sample is  $\mu < 5 \cdot 10^{-3} \text{ sm}^2 \text{ V}^{-1} \text{ s}^{-1}$  and  $p = (\rho e \mu)^{-1} > 5 \cdot 10^{24} \text{ m}^{-3}$ . It should be noted that the width of the forbidden zone,  $E_g$ , is usually much smaller than that of the conduction and valence zones. For example, in silicon,  $E_g = 1.1 \text{ eV}$ , and valence and conduction bands are 12 eV and  $> 7 \text{ eV}$ , respectively. To determine the location of impurity bands in lead-silicate glass doped with Cu and Mn oxides, we

use the first ionization potential of elements in it (Table 2) and the energy of shallow entry levels in Si and Ge (Table 3) (deep levels are not taken into account).

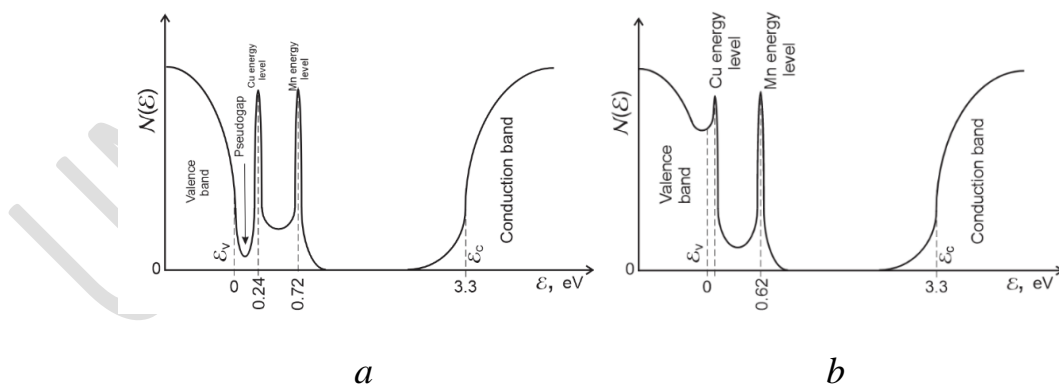
Table 2: First ionization potentials of Si, Ge, Cu, Mn, Fe, Pb, Ru and O, V [9]

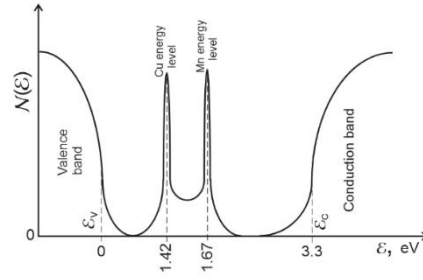
Si	Ge	Cu	Mn	Fe	Ru	Pb	O
8.140	7.88	7.724	7.432	7.896	7.36	7.415	13.614

Table 2 shows that the first ionization potential of copper, manganese and ruthenium is close to that of lead in glass. In Table 3, the energy levels of these elements in silicon are also close to each other. Based on this, the location of the impurity energy levels formed in lead-silicate glass when alloyed with copper and manganese oxides at different temperatures is shown in Fig. 2.

Table 3: Energy levels of Cu, Ge, Fe, Pb, and Ru in Si, Ge, and GaAs, eV [9, 13]

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	Cu	Mn	Fe	Pb	Ru
Si	0.24	0.33	D:0,4	0.37	A: $E_c-0.45$
Ge	0.04	0.16	0.31		
GaAs	0.023	0.095	0.37	0.12	





c

Figure 4. Distribution of the density of states in silicate glass doped with copper and manganese at different temperatures  $T$  (K): (a) 0; (b) 300; (c) 1000

The effective mass of charge carriers in DSG is  $m^* = 330 m_0$ , where  $m_0 = 9.1 \cdot 10^{-31}$  kg is the mass of a free electron. Therefore, when  $E \sim kT$  (weak external electric field)

$$N(E) = 2\pi(2m^*/\hbar^2)^{3/2}E^{1/2} = 10^{24} \text{ eV}^{-1}\text{cm}^{-3} \quad (9)$$

23 meV was obtained by calculating the width  $J$  of the entrance zone using the formula  $m^* = \hbar^2/(Ja_0^2)$  [14]. Here  $\hbar$  is Planck's constant,  $a_0$  is lattice constant (in glasses, it can be taken as the average distance between cations - copper or manganese atoms). When the proportion of alloying oxides is 10 mass %. In the impurity band, the density of states  $N_A$  is also related to the effective mass of charge carriers:

$$N_A = 2J^{-1} [2\pi m^* kT / \hbar^2] \approx 1.2 \cdot 10^{22} \text{ cm}^{-3} \text{ eV}^{-1} \quad (10)$$

These figures show that DSG can be an effective thermoelectric material.

The production of electricity from waste heat has a special place in ensuring energy efficiency, because almost 70% of all energy produced in the world is eventually converted into heat and dissipated into the environment. Thermoelectric generators (TEG) are the most promising in solving this task [3]. TEGs work like heat engines, but have no moving parts. Therefore, one of the challenges facing modern science and technology is to increase the efficiency and reduce the cost of TEG. This task, in turn, leads to the creation of a new thermoelectric material (TEM). One of the promising TEMs is DSG with various metal (mainly ruthenium, copper, manganese and tin) oxides, which is currently used in electronics as thick film resistors, in environmental and labor protection, robotics, human prostheses, mechanical and are widely used as sensors (sensing elements) of chemical effects [3]. However, the mechanism of electrical conductivity and thermal conductivity of this material has not been well studied. In particular, it is not clear how the impurity bands are formed during the alloying process and how they are affected by the composition and amount of ligature, how these levels are connected with the

thermoEMF coefficient (Zeebeck coefficient).

Efficacy of TEG

$$\eta_{el} = \frac{T_i - T_s}{T_i} \frac{\sqrt{ZT+1} - 1}{\sqrt{ZT+1} + T_s/T_i} \quad (11)$$

The unitless quality of TEM depends on  $ZT = S^2 \sigma T / k$ . Here  $S$  is the thermoEMF (Zeebeck) coefficient,  $\sigma$  is electrical conductivity,  $k$  is thermal conductivity,  $T = (T_1 + T_s)/2$  is the average temperature. The possibilities of increasing  $ZT$  by electrical conductivity  $\sigma$  are limited by the Wiedemann-Franz law:  $k/\sigma = LT$ ;  $L$  is Lawrence's number. Accordingly, the main way to increase  $ZT$  is to increase  $S$ , and according to Mott's formula [8], the density of electron states  $N(E)$  and its rate of change over energy are the main factors:

$$S(T) \approx \frac{\pi^2 k^2 T}{3e} \left( \frac{d \ln \sigma(E)}{dE} \right)_{E=E_F} = \frac{\pi^2 k^2 T}{3e} \left( \frac{d \ln N(E)}{dE} \right)_{E=E_F} \quad (12)$$

As in alloyed semiconductors, the location of the impurity band in the forbidden zone and the density of states in it play a key role in DSG. But there was no information about this in DSG. Therefore, it is necessary to estimate the location of the impurity band and the density of states in the lead-silicate glass doped with copper and ruthenium oxides with the help of the measuring method used in [10] and the data in it.

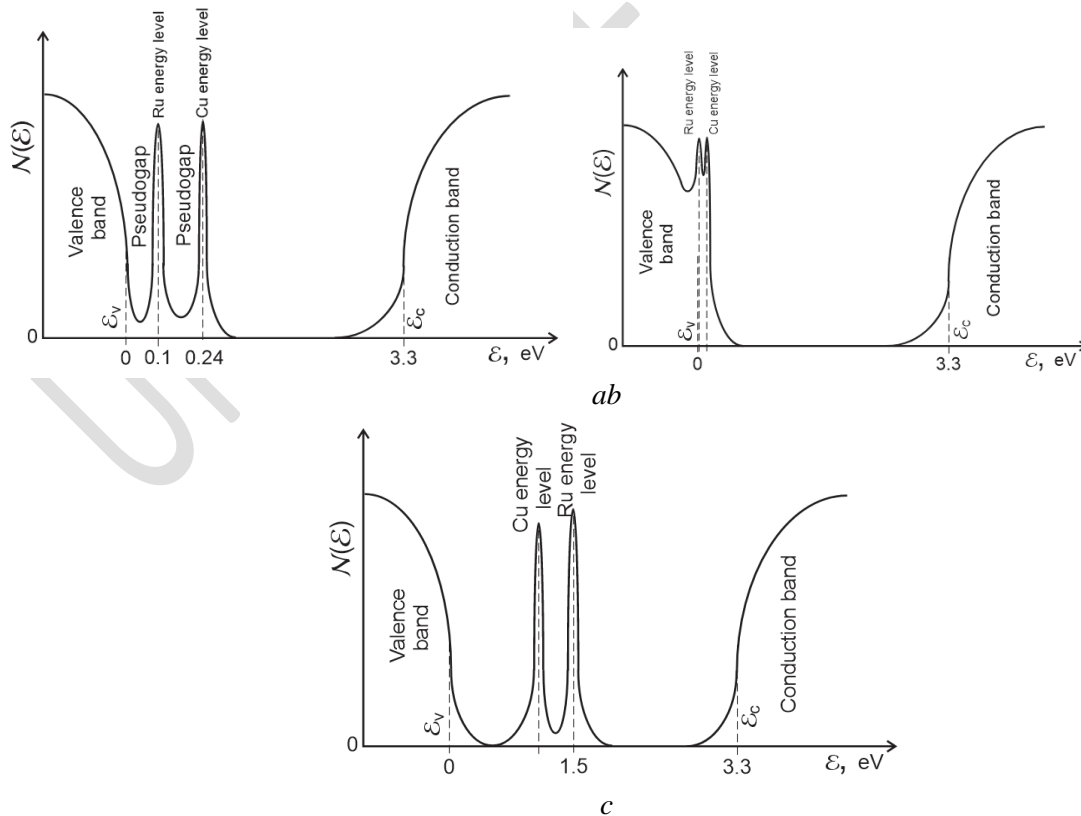


Figure 5. Location of entrance zones in silicate glass doped with ruthenium and copper oxides at different temperatures: (a) – 0 K; (b) — 300 K; (c) = 1000 K

From these estimates, it became clear that the energy levels of ruthenium and copper are located very close to each other (Fig.5 a), as the temperature increases, they move towards the valence band of glass under the influence of electron-phonon coupling, and close to room temperature joins the valence band (Fig.5 b). In addition, the distance between the ruthenium and copper zones also decreases. When the temperature exceeds 700 K, due to the structural transitions that occur in the silicate nanoparticles in the glass, the impurity band move towards the middle of the forbidden band of the glass (Fig. 5c). It is worth noting that when the temperature reaches 1000 K and structural transitions are completed, the band of copper is lower than the band of ruthenium. As can be seen from the pictures, even in the glass doped with copper and ruthenium oxides between helium and room temperatures, the energy gap between the impurity band and the valence band is not a real gap, but a pseudo-gap [24].

Considering that  $S \approx 1700 \mu\text{V/K}$ ,  $\rho \approx 2.5\text{-}25 \text{ m}\Omega\text{cm}$  DSG around the temperature at which structural transitions are completed, we are sure that DSG can be an effective TEM, because its thermal conductivity  $k = 0.5\text{-}1 \text{ W}/(\mu\text{K})$  [18].

### Analysis of results & discussion

As the temperature increases, the band gap of glass narrows due to electron-phonon coupling, but this narrowing (0.06-0.15 eV) is not significant compared to the width of the band gap (3.3 eV). In DSG doped with RuO<sub>2</sub> and MnO<sub>2</sub>,  $g(E)$  theoretical considerations about how it can be distributed [23-25].

The study of  $g(E) \sim d^2I/dV^2$  in LSSH doped with RuO<sub>2</sub> and MnO<sub>2</sub> by the method of microcontact spectroscopy [21] showed the general similarity of the experimental results and theoretical considerations, while at the same time they differ significantly (6-7-8 picture). Here,  $I$  is the constant current passing through the microcontact, and  $V$  is the voltage between the microcontact and the sample. In particular, theoretically, the energy levels formed by RuO<sub>2</sub> should be almost connected to the valence band of the glass, and the MnO<sub>2</sub> band should be separated from it by a much larger energy gap. In the experiment, the location of these levels corresponds to the model. But the MnO<sub>2</sub> band is located closer to the conduction zone of the glass in the model, while in the experiment this zone is much closer to the RuO<sub>2</sub> zone (Fig. 4c). It was found in the experiment that the input levels are spread quite widely across the energy range. It is because of this wide distribution that the



Figure 6. Mutual arrangement of sample and microcontact in microcontact spectroscopy

continuous energy distribution mentioned above may have occurred. Another noteworthy aspect of Fig. 4c is that the density of states is much higher in the interval from the valence band of the glass to the  $\text{MnO}_2$  band, which means that this energy gap is actually a pseudo-gap.

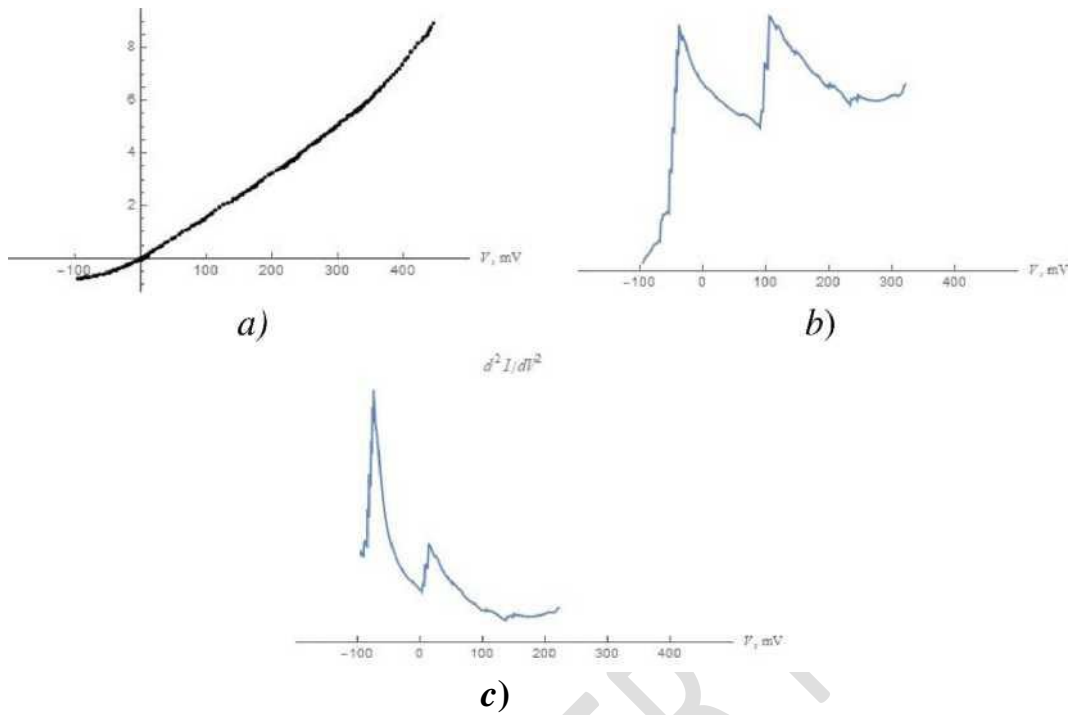


Figure 7. a) low-voltage volt-ampere characteristic of silicate glass doped with  $\text{RuO}_2$  and  $\text{MnO}_2$  (result of microcontact measurement); b) electrical conductivity (derivative of the volt-ampere characteristic); c) density of electron states (the second derivative of the volt-ampere characteristic)

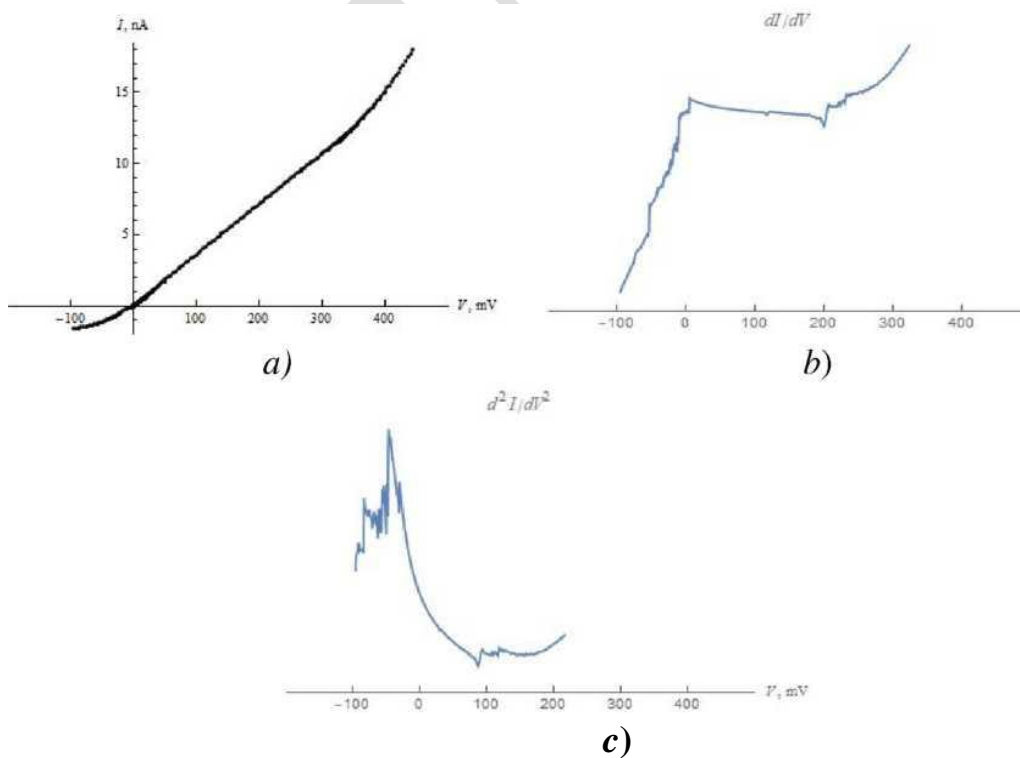


Figure 8. a) low-voltage volt-ampere characteristic of silicate glass doped with RuO<sub>2</sub> and CuO (result of microcontact measurement); b) electrical conductivity (derivative of the volt-ampere characteristic); c) density of electron states (volt-ampere second derivative of the characteristic)

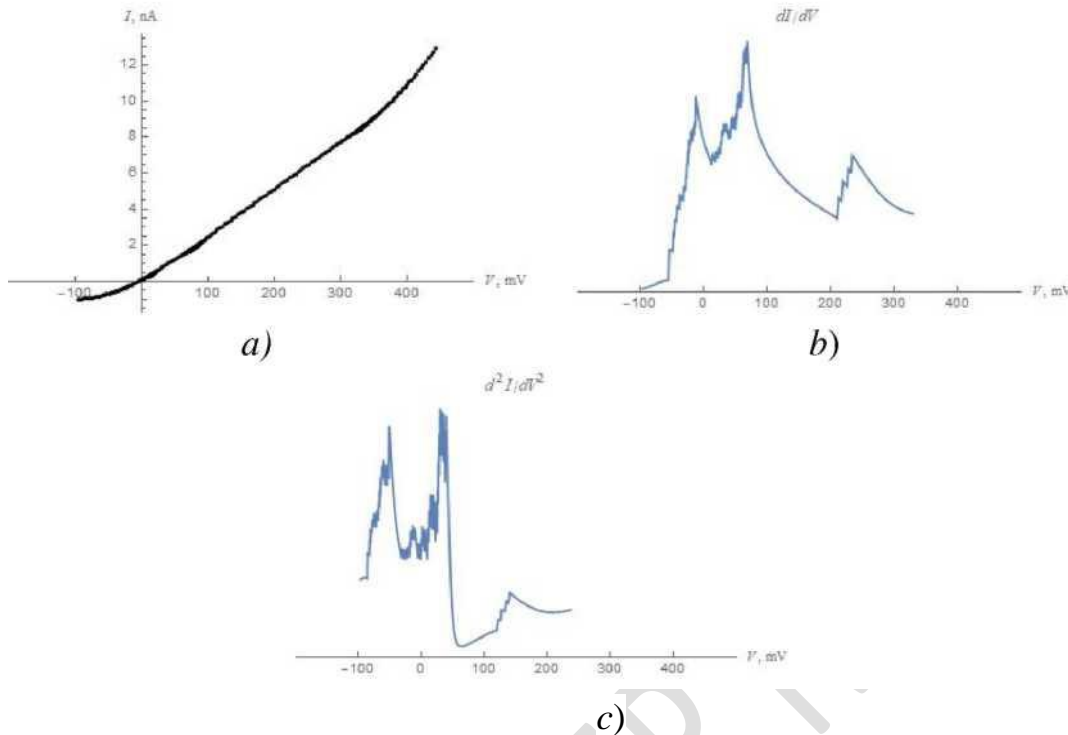


Figure 9. a) low-voltage volt-ampere characteristic of silicate glass doped with CuO and MnO<sub>2</sub> (result of microcontact measurement); b) electrical conductivity (derivative of the volt-ampere characteristic); c) density of electron states (the second derivative of the volt-ampere characteristic)

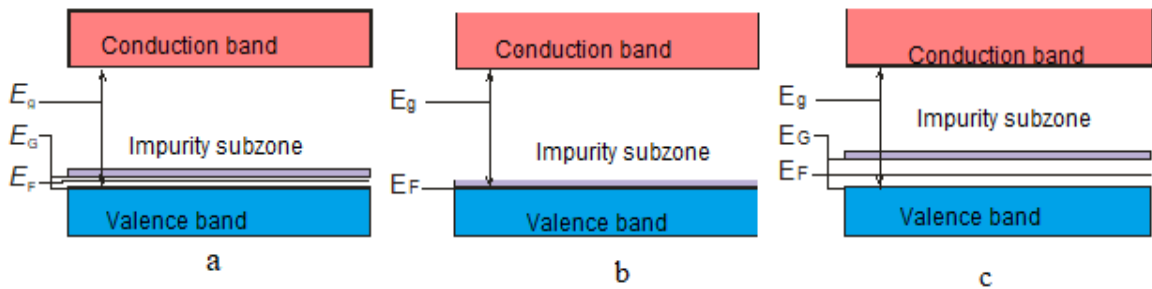


Figure 10. Energy bands in doped glass at T (K):0 (a); 300 (b); > 970 (c)

According to the results of the analysis mentioned above, it is possible to imagine the location of the energy zones in DSG and their changes along the temperature as shown in Fig. 10. The band gap of the 2SiO<sub>2</sub>·PbO glass is 3.3 eV at T = 0 K, and decreases with a rate of 0.1 meV/K as the temperature increases, to 0.1 eV at T = 1000 K, i.e., negligible compared to its initial value. But the impurity band is separated from the valence band by a pseudogap of only 0.05 eV (Fig. 10a), and at T = 300 K, this pseudogap decreases to 0.03 eV, becomes very close to the valence band, and may even merge (Fig. 10b). If the temperature increases further (Fig. 10c), due to the structural transitions in the silicate nanocrystals, the impurity will go to the middle of the forbidden band, and DSG will have activation conductivity.

## Conclusion

1. The location of the impurity band formed in the silicate glass doped with two metal oxides in the forbidden band and the density of states in them change under the influence of temperature, leading to dielectric-metal and metal-dielectric transitions.
2. The impurity band that appear as a result of double doping can be separated by a mutual pseudo-gap (CuO and MnO<sub>2</sub>, RuO<sub>2</sub> and MnO<sub>2</sub>) or can be combined to form a single impurity band with a large width (RuO<sub>2</sub> and CuO).

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