

The measurement of the diffusion coefficient of oxygen ions in the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ layer of the electro-resistance memory

Abstract. The paper attempts to determine the chemical diffusion coefficient of oxygen ions in the thin $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ layer of the electro-resistance memory at room temperature. The measurement method was based on the assumption that in the process of switching the sample resistance by means of current pulses there is a stage of diffusion of oxygen ions in the layer of finite thickness from the source located at its surface. This layer is the oxygen depleted layer at the electrode. Its thickness was determined empirically from the amplitude characteristics of the resistive states, while the diffusion time constant was obtained from measurements of the resistance relaxation curves. The value of $5.5 \cdot 10^{-18} \text{ m}^2/\text{s}$ of the oxygen diffusion coefficient in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ obtained in this paper is in the order of magnitude consistent with the literature data obtained under similar conditions.

Streszczenie. W pracy podjęto próbę wyznaczenia współczynnika dyfuzji chemicznej jonów tlenu w cienkiej warstwie $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ pamięci elektrozystancyjnej w temperaturze pokojowej. Metodę pomiaru oparto na założeniu, że w procesie przełączania rezystancji próbki za pomocą impulsów prądowych występuje etap dyfuzji jonów tlenu w warstwie o skończonej grubości ze źródła zlokalizowanego przy jej powierzchni. Warstwą tą jest przyelektrodowa warstwa zubożona w tlen. Jej grubość wyznaczono doświadczalnie z charakterystyk amplitudowych stanów rezystancyjnych, natomiast stałą czasową dyfuzji otrzymano z pomiarów przebiegów relaksacji rezystancji. Otrzymana w pracy wartość $5,5 \cdot 10^{-18} \text{ m}^2/\text{s}$ współczynnika dyfuzji jonów tlenu w $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ jest co do rzędu wielkości zgodna z danymi literaturowymi uzyskanymi w podobnych warunkach. (**Pomiar współczynnika dyfuzji jonów tlenu w warstwie $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ pamięci elektrozystancyjnej**)

Keywords: high-temperature superconductors, electro-resistance memory effect, depleted layer, oxygen-ion electrodiffusion, diffusion coefficient.

Słowa kluczowe: nadprzewodniki wysokotemperaturowe, zjawisko pamięci elektrozystancyjnej, warstwa zubożona, elektrodyfuzja jonów tlenu, współczynnik dyfuzji.

Introduction

The development of computer technology requires better and better memories, both operational and mass. Existing memories have already reached practically their physical limits. Therefore, in recent years, we can observe increased research activity in the search for new materials and phenomena that could be used to build memory elements. In the field of mass storage, one of the most promising directions [1,2] seems to be the use of the phenomenon of electro-resistance memory. This phenomenon occurs in many materials and at different temperatures (from 4 to 400K). Examples are oxide materials with a regular crystallographic structure, such as: TiO_2 [3], NiO , Al_2O_3 [4,5], or having a more complex perovskite structure: $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ [6-9], $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ [10], $\text{Pr}_{1-x}\text{Ca}_x\text{MnO}_3$ [11], $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ [12], SrTiO_3 [13]. The latter also exhibit the properties of high-temperature superconductors (HTS). Superconducting materials have long tempt with low energy losses and high reaction rates.

The phenomenon of electro-resistance memory in oxide materials with a perovskite crystallographic structure occurs as a result of electrodiffusion of O^{2-} oxygen ions through oxygen vacancies [7, 9, 10, 14-16]. Oxygen diffusion inside and outside the perovskite layer changes the oxygen deficit value. The oxygen atoms in some of the nodes of the elementary cell act as acceptor dopants. The increase in oxygen deficit leads to a decrease in the concentration of charge carriers and, as a consequence, to an increase in resistivity. In the structure of the memory element, in the perovskite areas adjacent to the electrodes, there are layers depleted in oxygen, i.e. high resistivity. If we apply a positive potential to the electrode, high enough to provide O^{2-} ions with energy exceeding the diffusion activation energy, they will move through oxygen vacancies to the depleted layer, as a result of which the element resistance will drop. At the negative electrode polarization, the oxygen ions are pushed out of the near-electrode layer into the electro-resistive material. This leads to the recovery of the depleted layer and increase in resistance to a high level.

For a deeper understanding of the mechanism of operation of such memories and conducting quantitative

analyses, there is a need for mathematical modeling of oxygen diffusion processes in the structures of the electro-resistive memory. One of the most important parameters of such a model is the O^{2-} ions diffusion coefficient determined in the conditions of future memory work. However, there is not enough reliable data on the subject in the scientific literature. The diffusion coefficient is usually determined at high temperatures ($300\div 850^\circ\text{C}$), adequate to the processes of manufacturing memory structures, and not to the conditions of their operation. In addition, the tracer diffusion coefficient D^* is usually determined, while the chemical diffusion coefficient \check{D} is rather needed to model the work of the elements.

The aim of this work is to determine the chemical diffusion coefficient of O^{2-} ions in the layer of polycrystalline $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ high-temperature superconductor at room temperature.

Measurement method

The chemical diffusion coefficient \check{D} of oxygen ions can be determined using the following relationships:

$$(1) \quad \check{D} = \frac{d^2}{4\tau},$$

resulting from the diffusion model in the layer of finite thickness d from the source located at the surface [8,11, 17]. The parameter τ is the time constant of this process. In the case of structures with electro-resistive properties, the constant τ can be determined on the basis of the resistance relaxation curves after switching off the current (or voltage) [11, 16]. The thickness d of the diffusion layer can be obtained (using the method proposed in [18]) from the amplitude characteristics of the resistive states of the memory element after switching off the current.

Experimental research was carried out on a memory element in the form of a superconducting structure (HTS) consisting of a thin film of the superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ applied on a sapphire substrate (Al_2O_3) with dimensions of $10 \times 6 \text{ mm}$ [19]. The superconductor layer with a thickness of $h \approx 0.5 \mu\text{m}$ was obtained by magnetron sputtering. The crystallographic studies showed a polycrystalline structure

with the predominant orientation of the crystallite axis c perpendicular to the plane of the layer. Next, the HTS film was formed with the photolithography method in the shape of a microbridge of the width of $w=200\mu\text{m}$ and a length of $l=2\text{mm}$, and then a layer of silver contacts was sputtered and formed (the $\text{Ag}/\text{YBa}_2\text{Cu}_3\text{O}_{7-x}/\text{Ag}$ structure). Parameters of the HTS microbridge measured immediately after it was formed amounted to: critical temperature $T_c=87.5\text{ K}$, superconducting transition temperature width $\Delta T_c=1.4\text{ K}$ and critical current $I_c\approx 550\text{ mA}$ at the temperature of 78 K [19]. The test sample was placed in a nitrogen flow-through cryostat designed for measurements in the temperature range from 77 to 300 K , fastening it to the "cold finger" with silver paste. During the measurements, the sample was in a vacuum of 10^{-2} hPa .

The measurements consisted in determining the resistance characteristics of the HTS microbridge at different temperatures and with different history of the preceding interactions. Resistance was determined using a technical method in a 4-wire system, supplying microbridge from a current source. Such a supplying mode is more beneficial when testing the phenomenon of electro-resistance in comparison to the voltage source [9]. With current stabilization, changes in the resistivity of one area do not cause changes in the electric field in neighboring areas, which allows better identification of physical phenomena depending on the intensity of this field. The measuring system uses the Keithley 2000 multimeter and the Keithley 6221 current source (which, in addition to direct current, can also generate periodic waveforms of the current). The measuring instruments were controlled by a PC via the GPIB interface. During the measurements, the voltage-current, amplitude and relaxation characteristics of the HTS microbridge resistance at room temperature T_a and the temperature of liquid nitrogen T_{LN} were determined. Between the measurements of the characteristics, the sample was subjected to several minutes of DC current with different directions and values from 20 to 50 mA . Some of these results are presented in paper [18]. The present work uses the results of measurements of relaxation and amplitude characteristics obtained at room temperature ($T_a\approx 294\text{K}$).

Measurement results

The relaxation characteristics were determined by passing through HTS microbridge rectangular current pulses with amplitude $I_m = 20\text{ mA}$ and the course shown in Fig. 1 in the form of curve $I(t)$. The duration of the pulse was 10 s , followed by switching off the current for 20 s , during which the relaxation of the resistance was observed.

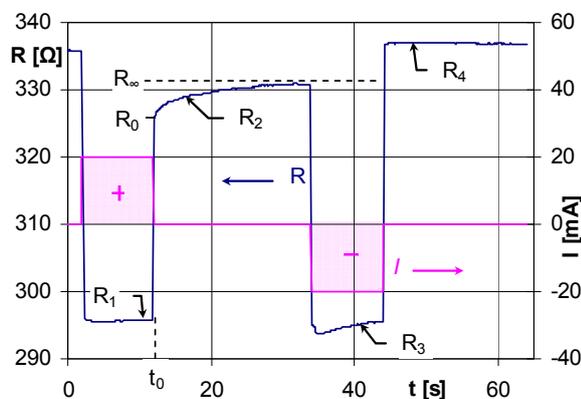


Fig.1. Runs of resistance response $R(t)$ of the HTS microbridge to the action of current pulses $I(t)$ in measurement no.10. R_1 , R_2 , R_3 , R_4 - symbols of the resistance response stages.

In practice, "switching off" the current meant switching it to the level of $I_0=100\text{ }\mu\text{A}$ (called the "zero" current), so that the resistance of the element could be measured. Then an analogous pulse with opposite polarization was given. The positive direction of the current was assumed conventionally due to the symmetry of the examined microbridge, and it was used consistently in all measurements. Before each pulse, the microbridge conductivity was stabilized for 2 s using I_0 current with the polarity of the following pulse. Simultaneously with the current run, voltage measurements were taken with a step of 100 ms , which allowed to calculate the resistive response $R(t)$ of the microbridge.

A typical course $R(t)$ is shown in Fig.1. Qualitatively similar resistive responses were observed in [6,16,20] for $\text{Au}/\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ structures. In the experiment from Fig. 1, with the impact of $+20\text{ mA}$ current (stage R_1), the resistance drops from the initial state 336Ω to the value 296Ω (by approx. 40Ω). Such non-linearity of resistance for structures based on $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ was presented both in our papers [9, 18] and in the work of other authors [21]. The reason for this lies in the mechanism of electric conductivity of carriers induced from trap levels, just O^{2-} ions (Poole-Frenkel mechanism) [22, 23]. After switching off the current at the moment t_0 (Fig.1), the resistance increases, but only up to the level $R_0=326\Omega$, which is lower than the initial one. This effect can be explained by the previously discussed oxygen ion electrodiffusion. The diffusion of these ions under the influence of the electric field towards the positive electrode reduces the oxygen deficit x in the depleted layer. After switching off the field, they remain in the new nodes of the crystal lattice, causing an increase in the concentration of traps, and thus a decrease in resistance (relative to the initial one). The distribution of O^{2-} ions concentration in the depleted layer is, however, very uneven, namely they are concentrated just at the electrode. Thus, a concentration gradient appears that causes the oxygen ions to retract within the depleted layer by thermodiffusion. The result is a relaxation increase of resistance in the section R_2 (Fig.1) from the value R_0 to R_∞ . Turning on the current in the reverse direction (-20 mA) causes, as previously, a step drop in resistance. At this stage (R_3) the O^{2-} ions are pushed out by the electric field from the nearelectrode layer into the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ material, which leads to the recovery of the depleted layer. As a result, after switching off the current, the resistance increases to the initial level of 336Ω . In the further course (R_4), however, we do not observe the relaxation of the resistance by thermodiffusion. The reason for this may be too small a gradient of concentration of oxygen ions inside the superconductor due to its much greater thickness compared to the depleted layer.

It follows from the above considerations that the relaxation of the resistance on the section R_2 (Fig. 1) takes place due to the thermodiffusion of O^{2-} ions inside the depleted layer. Therefore, dependence (1) is true for this process. The time constant τ can be determined from the course $R(t)$ on the R_2 section, approximating it with the exponential function of the type: $\exp(-x^\tau)$ [16]. For this purpose, the course $R(t)$ obtained from the experiment will be presented in the coordinate system $(t', \Delta R')$. Coordinate $t'=t-t_0$ is the time counted from the moment of switching off the current pulse (Fig.1), while $\Delta R'$ - is the relative difference of the resistance of the microbridge at the moment t' , defined as:

$$(2) \quad \Delta R' = \frac{R_\infty - R}{R_\infty - R_0}$$

where: R_0 - the resistance at the moment t_0^+ ; R_∞ - asymptotic value of the resistance at $t' \rightarrow \infty$ (Fig.1).

The approximating function is then:

$$(3) \quad \Delta R' = e^{-\left(\frac{t'}{\tau}\right)^n},$$

where: τ - time constant of the course; n - exponent power. Function (3) takes the linear form in the coordinate system $(\ln t', Y)$ if

$$(4) \quad Y = \ln(-\ln \Delta R')$$

The equation of this line can then be written as follows:

$$(5) \quad Y = n \cdot \ln t' - n \cdot \ln \tau$$

After the linear regression procedure of experimental runs

$Y = f(\ln t')$, we obtain the value n from the slope of the approximating line and the time constant from the dependence:

$$(6) \quad \tau = e^{-\frac{Y(t'=1s)}{n}}$$

using the value of free term of function (5).

In Fig. 2, runs $R_2(t)$ obtained in six experiments carried out at room temperature are shown in the system $(\ln t', Y)$. Dashed lines have functions that approximate the each course.

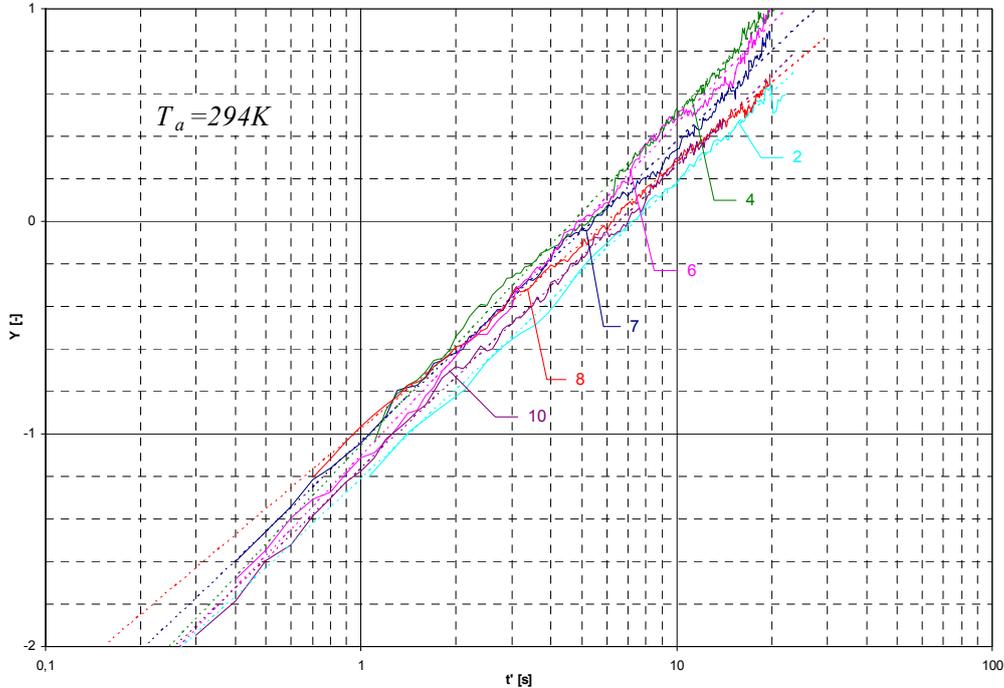


Fig.2. Runs of resistance relaxation of HTS-bridge after a +20mA current pulse, in coordinates $(\ln t', Y)$ (solid lines), and linear regression lines of these runs (dashed lines). The numbering of the curves corresponds to the order in which the measurements are taken.

The very good approximation quality is indicated by the fact that the square of the linear correlation coefficient in all cases was greater than 0.994. Parameters n and τ were obtained from linear regression equations and summarized in Table 1. The power n of the exponent of the function (3) assumes values in the interval $(0.54 \div 0.68)$. The relatively small spread of n confirms a very similar slope of the waveforms, shown in Fig. 2. Time constants τ are from 4.7 to 7.4s, so they are also similar to each other.

Table 1. The values of the exponent n and the time constant τ obtained from the approximation of relaxation curves from Fig.2 by means of the function (3) and the resistance switching voltage U_s^- and the thickness of the depleted layer d^- determined from the amplitude characteristics (Fig.3). Chemical diffusion coefficient \tilde{D} calculated from dependence (1).

Test no.	n [-]	τ [s]	U_s^- [V]	d^- [nm]	\tilde{D} [$10^{-18} \text{m}^2/\text{s}$]
2	0,604	7,42	4,2	10,7	3,9
4	0,678	4,73	4,4	11,2	6,6
6	0,678	5,10	4,6	11,7	6,7
7	0,612	5,43	4,6	11,7	6,3
8	0,543	6,06	4,6	11,7	5,7
10	0,617	6,59	4,0	10,1	3,9

Thickness d of the depleted layer, which is necessary to determine the diffusion coefficient from dependence (1), can be obtained from the amplitude characteristics $R_o(U_m)$ of resistive states of the memory element, as proposed in [18]. For this purpose, the examined microbridge were subjected

to quite long (about 4s), rectangular current pulses with an amplitude I_m changing in the range of ± 20 mA. As the amplitude U_m , the voltage was assumed on the sample at this current. The resistance R_o was determined after switching off the current, i.e. at the "zero" current $I_o = 10 \mu\text{A}$. Selected amplitude characteristics $R_o(U_m)$ are shown in Fig.3. Their course is similar to that obtained in the work of other authors, e.g. [6, 12]. These characteristics show that in the HTS microbridge structure there is a switching of resistance states under the influence of current interactions. This phenomenon is clearly threshold. Resistance switching takes place above a certain voltage value, called the switching voltage U_s . Fig. 3 presents the method of determining this voltage at positive currents: U_s^+ and negative: U_s^- , on the example of the curve from measurement 3. This voltage is related to the thickness of the depleted layer. This relationship was derived by Tomasek et al. [7] using the proposed model of switching the resistance of the structure $\text{Ag}/\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$. This model is based on the mathematical description of O^{2-} ions diffusion processes in the field of oxygen vacancy potential.

To determine the diffusion coefficient, the thickness d of the depleted layer is needed in this state of the HTS microbridge, in which the time constant τ was determined (Tab.1). Therefore, the amplitude characteristics were determined just before the measurements of relaxation characteristics. In addition, the constant τ of oxygen ions diffusion was obtained for the stage R_2 (Fig. 1) of the course $R(t)$, i.e. following the positive pulse.

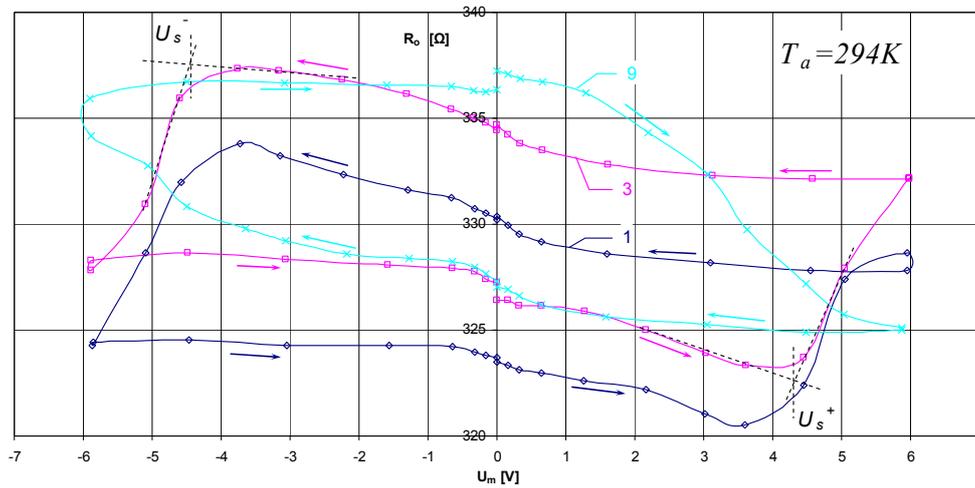


Fig. 3. Amplitude characteristics of resistance states R_o of HTS microbridge at room temperature T_a . The numbering of the curves and the direction of the arrows are in accordance with the order in which the measurements are taken.

Therefore, the thickness of the depleted layer should be determined for the analogous resistive state, i.e. based on the voltage U_s^- (Fig. 3). The values of voltage U_s^- and thickness d^- obtained in this way are summarized in table 1. The switching of the resistive state of the microbridge took place at close to each other voltages: from 4.0 to 4.6 V, and the thickness of the depleted layer ranged from 10.1 to 11.7 nm. Then, using the relation (1), the values of the chemical diffusion coefficient \tilde{D} were calculated for the corresponding values of τ and d^- , and they were summarized in table 1. The average value of the diffusion coefficient is $5.5 \cdot 10^{-18} \text{ m}^2/\text{s}$ with standard uncertainty (type A) equal to $0.5 \cdot 10^{-18} \text{ m}^2/\text{s}$. The uncertainty at the level of 9% of the measurement result is quite satisfactory, taking into account the estimated nature of the measurement.

Analysis of results

Materials with a perovskite structure are characterized by a very high anisotropy of kinetic properties. This also refers to the oxygen ions diffusion coefficient. In the case of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$, the anisotropy measured by the ratio of the diffusion coefficient D_{ab} in the plane ab to the coefficient D_c in the direction of c -axis can be 10^3 [24-26] and even 10^6 [27]. Therefore, a question arises, with which values obtained by other authors can be compared results obtained by us? As mentioned earlier, the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ film present in our structures was polycrystalline, with the predominant orientation of crystallites along the c -axis. Thus, we can expect an isotropic diffusion with a D_{ab} coefficient [28]. The validity of this hypothesis is confirmed by the n values obtained by us (Tab.1) of the power of the exponent of the function approximating the resistance relaxation characteristics. According to the theory of diffusion of atoms in a crystal by trap states [16, 29], power n assumes the value of $1/3$ in a one-dimensional diffusion, $1/2$ in two-dimensional and 0.6 in three-dimensional. The great majority of n values obtained in our measurements are in the range (0.60 \div 0.68), which proves the three-dimensional (isotropic) diffusion of O^{2-} ions in our sample and confirms earlier assumptions.

There is a lot of data in the world literature about the oxygen diffusion coefficient in the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ polycrystalline films or in the ab -plane of the single crystals that were obtained (or extrapolated) at room temperature ($T_a=300\text{K}$). The most representative values can be considered: $10^{-18} \text{ m}^2/\text{s}$ [8] measured in the epitaxial layer on the LaAlO_3 substrate and $2.7 \cdot 10^{-22} \text{ m}^2/\text{s}$ [30] and $10^{-24} \text{ m}^2/\text{s}$ [24] obtained for polycrystalline samples. However, the

discrepancy between these results is huge, since they differ by 10^6 times. However, the orders of magnitude are similar to the values obtained in room temperature for other materials with a perovskite structure. For example, the oxygen diffusion coefficient in $\text{GdBaCo}_2\text{O}_{5+x}$ was $2.6 \cdot 10^{-17} \text{ m}^2/\text{s}$ [31], in $\text{Pr}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ ($5 \cdot 10^{-20} \div 5 \cdot 10^{-18}$) m^2/s [11], and in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ was received $1.6 \cdot 10^{-21} \text{ m}^2/\text{s}$ [32].

Such a large spread of literature data with oxygen diffusion coefficient in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ may result from differences in the properties of samples used, measurement methods or measurement conditions. Depending on the measurement method, different diffusion coefficients can be determined: tracer D^* or chemical \tilde{D} . Chemical diffusion is caused by a chemical potential gradient. In contrast, the tracer diffusion occurs under the influence of the oxygen concentration gradient and does not take into account the gradient of the chemical potential. These coefficients are coupled by the thermodynamic factor θ , namely $\tilde{D} = \theta \cdot D^*$. In turn, the parameter θ depends on various factors: stoichiometric composition, purity and porosity of the material, content and type of defects of the crystalline structure, temperature and others. For example, the decrease in oxygen deficit x in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ from 0.7 to 0 is accompanied by an increase in θ from 1 to 400 [33]. The porosity of the material has an even greater impact. Taking into account the contribution of various factors, the thermodynamic factor can reach values $10^4 \div 10^6$ [28], i.e. the number of times the literature data differs.

Another reason for differences in D values obtained by different authors may be extrapolation of results obtained at one temperature to another, e.g. room temperature. Measurements of the diffusion coefficient are usually carried out in the temperature range: 300 \div 850°C, which is much higher than room temperature. Then the results are extrapolated using the Arrhenius law, that is, the relation $D(T) = D_o \exp(-E_a/kT)$, where: E_a – activation energy of oxygen diffusion; D_o - pre-exponential factor; k - Boltzmann's constant. The assumption is that the D_o and E_a parameters are constant. Meanwhile, this is not true, because they can change significantly depending on various factors, including temperature. For example, the activation energy values E_a for $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ found in the literature range from 0.4 eV [34] to 1.3 eV [25]. Meanwhile, the change of E_a by 0.3 eV results in a 10^3 -fold change in the diffusion coefficient according to Arrhenius law, while a change of 0.6 eV changes D 10^6 times. Thus, the indeterminacy of only

activation energy can explain such a large scatter of literature data.

The type of sample, methodology and conditions for determining the oxygen diffusion coefficient closest to ours were at work [8]. The sample in the form of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ epitaxial layer deposited on the LaAlO_3 substrate with the orientation along the c -axis was investigated there. The electrodes were also applied from silver. Similarly, a time constant τ was determined based on the resistance relaxation curves. The measurements were carried out at room temperature. All these similarities, to a large extent, explain similar D values obtained in our work and work [8] ($5.5 \cdot 10^{-18}$ and 10^{-18} m^2/s respectively). Taking into account the above considerations, it should be recognized that the values of oxygen diffusion coefficient in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ obtained by us are sufficiently compatible with the literature.

Summary

Conducted experimental investigations of the phenomenon of electroresistance memory in thin-film $\text{Ag}/\text{YBa}_2\text{Cu}_3\text{O}_{7-x}/\text{Ag}$ structures allowed to determine the chemical diffusion coefficient of oxygen ions in the high-temperature superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ at room temperature. The time constant of the oxygen diffusion process in the depleted layer was determined from the resistance relaxation curves after the current pulse. The thickness of this layer was determined from experimentally obtained amplitude characteristics of the resistive states of the test sample. The value of $5.5 \cdot 10^{-18}$ m^2/s of oxygen diffusion coefficient in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ determined on their basis is on the order of magnitude consistent with the literature data obtained under similar conditions.

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