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# Non-Arrhenius behavior of the temperature dependence of superionic conductivity of a $La_{0.95}Sr_{0.05}F_{2.95}$ single crystal

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The temperature dependence of the ionic electrical conductivity for a single crystal of the superionic conductor $La_{0.95}Sr_{0.05}F_{2.95}$ with the tysonite-type structure (sp. gr. $P\bar{3}c1$) was studied at 210−1073 K. It was found that, in the temperature range studied, ionic conductivity exhibits non-Arrhenius behavior and satisfies the Vogel-Tammann-Fulcher equation: $\sigma_{dc}T^{1/2} = \sigma_0 \exp\left(-\Delta H_{\mathrm{VTF}}/(T - T_0)\right)$ with parameters $\sigma_0 = 1.2 \cdot 10^2$ SK$^{1/2}$/cm, $\Delta H_{\mathrm{VTF}} = 0.18$ eV, and $T_0 = 85$ K. This behavior of the $\sigma_{dc}(T)$ dependence is apparently due to the energy distribution of ion carrier (fluorine vacancies) hops due to the structural microheterogeneity of the solid solution. The application of the Vogel-Tammann-Fulcher mathematical formalism is of undoubted interest for the study of superionic fluorine-conducting solid solutions.

**Keywords:** ionic conductivity, superionic conductors, strontium and lanthanum fluorides, single crystals, tysonite structure, solid electrolytes.

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## 1. Introduction

Nonstoichiometric fluoride phases (heterovalent solid solutions) $R_{1-y}M_yF_{3-y}$ with tysonite structure in condensed systems $MF_2{-}RF_3$ ($M$ — alkaline earth elements Ca, Sr, Ba and Cd, Pb; $R$ — rare earth elements La−Lu, Y) are advanced superionic conductors with unipolar fluorine-ion conductivity [1–5]. Ion conductivity therein arises as a result of heterovalent substitutions of cations $R^{3+}$ on $M^{2+}$, causing formation of mobile „crystallochemical“ vacancies of fluorine. In Shubnikov Institute of Crystallography (Moscow) a program has been implemented for a long time to consistently study the ion transfer in multi-component fluoride materials [6–10].

Tysonite solid solution $La_{1-y}Sr_yF_{3-y}$ has high fluorine-ion conductivity and is one of most actively studied fluoride conductors. Studies of the ion transport in nonstoichiometric phase $La_{1-y}Sr_yF_{3-y}$ were conducted using single crystals [11–20], polycrystals [21–23], composites [24,25], thin films [26,27] and nanoscale ceramics [28–30]. Structural studies of $La_{1-y}Sr_yF_{3-y}$ crystals were conducted in [31–34].

However, note that fundamental microscopic characteristics of superionic transport in fluoride materials may only be obtained in single-crystal samples. Since the electric conductivity of polycrystals, ceramics and composites is mainly defined by the boundaries of crystalline grains, and electric conductivity of thin films is related to the state of their surface.

To describe the temperature dependence of ion electric conductivity in crystals and glasses, traditionally the mathematical formalism of Arrhenius-Frenkel is used [35]. Use of this formalism makes it possible to visually identify and analyze the electrophysical processes occurring in crystalline and amorphous media, provides important information on the translational (hopping) motion of mobile charge carriers in the structures of superionic conductors. This approach was also used previously to describe the ionic transport in single crystals of solid solution $La_{1-y}Sr_yF_{3-y}$ in [9–12,20,21,28–30].

In [18] it is noted that at high temperatures the ion conductivity of solid solution $La_{1-y}Sr_yF_{3-y}$ deviates from Arrhenius behavior, therefore the Arrhenius-Frenkel equation is applied separately for low- and high-temperature areas of temperature dependence of electric conductivity.

The aim of the paper is to study the non-Arrhenius behavior of temperature dependence of ion conductivity for a single crystal of superionic conductor $La_{0.95}Sr_{0.05}F_{2.95}$ in the wide temperature range 210−1073 K.

## 2. Experiment

Single crystals of tysonite solid solution with composition of $La_{0.95}Sr_{0.05}F_{2.95}$ are grown from the melt by the technique of Bridgman's vertical crystallization in the fluorinating atmosphere and are certified structurally (diffractometer HZG-4, radiation Cu$K_\alpha$, internal standard Si) in the Crystallography Institute [36–38]. The fluorinating atmosphere developed by the tetrafluoroethylene pyrolysis products is necessary for the suppression of the pyrohydrolysis reaction (interaction of fluorides with water vapors), being a specific reaction for inorganic fluorides [39]. The chemical composition of the grown crystals of the solid solution $La_{1-y}Sr_yF_{3-y}$ complied with the composition of the source charge ($y = 0.05$) with relative error of 5 %. The oxygen impurity content in crystals was below 0.01−0.02 mass.%.

The sample was a disc with polished bases, the thickness and the diameter of the disc are equal to 1.95 and 11.2 mm accordingly. The experiments were made on a non-oriented single crystal in the assumption of the quasi-isotropic behavior of electric conductivity, since anisotropy of ionic conductivity in tysonite crystals $R_{1-y}M_yF_{3-y}$ is minor [18,40].

Electrophysical measurements were performed by impedance spectroscopy in the frequency ranges of $10^{-1}-10^7$ Hz and resistance ranges of $1-10^{10}\,\Omega$ using Solartron 1260 instrument at voltage of 30 mV. A description of the experimental setup is given in [17,41]. Electrodes are made in the form of Ag-contacts. Impedance measurements were carried out in the cooling mode in temperature interval of 210−1073 K in the protective gas atmosphere $N_2$ (high temperatures) and in vacuum $\sim 10^{-3}$ Pa (low temperatures). The error of impedance measurements did not exceed 2 %. The bulk resistance $R_b$ of the crystal was found by the intersection of the impedance hodographs with the axis of the active resistances. The charts were built by non-linear least-square method, using FIRDAC software [42].

## 3. Mathematical formalism of Arrhenius-Frenkel

Figure 1 shows the temperature dependence of ion conductivity in coordinates $\lg(\sigma_{dc}T)$, $10^3/T$ for the studied single crystal $La_{0.95}Sr_{0.05}F_{2.95}$. In the interval of 210−1073 K the ion conductivity increases from $8.4 \cdot 10^{-7}$ to $3.8 \cdot 10^{-1}$ S/cm ($4.5 \cdot 10^5$ times). You can see that the dependence $\sigma_{dc}(T)$ meets the equation of Frenkel-Arrhenius only at low- and high-temperature areas separately:

$$\sigma_{dc} = (\sigma_0/T)\exp[-\Delta H_\sigma/kT], \quad (1)$$

where $\sigma_0$ is the pre-exponential conductivity factor, and $\Delta H_\sigma$ is the ion conductivity activation enthalpy. Values of $\sigma_0$ and $\Delta H_\sigma$ parameters are equal to $5.5 \cdot 10^4$ SK/cm, $0.33 \pm 0.01$ eV and $5.1 \cdot 10^3$ SK/cm, $0.23 \pm 0.01$ eV at $T < T_0$ and $T > T_0$ accordingly. The bend point in temperature dependence $\sigma_{dc}(T)$ is $T_0 = 480$ K.

As noted in [17], in superionics of $La_{0.95}Sr_{0.05}F_{2.95}$ there is a hopping mechanism of ion conductivity related to charge carriers — „impurity" vacancies of fluorine $V_F^\bullet$, formed as a result of heterovalent substitutions of cations $La^{3+}$ at $Sr^{2+}$ in the crystalline lattice of tysonite:

$$La_{La}^{\times} \rightarrow Sr_{La}^{/} + V_F^{\bullet}. \quad (2)$$

Here, the designations of defects are given in the Kroeger-Wink system [43].

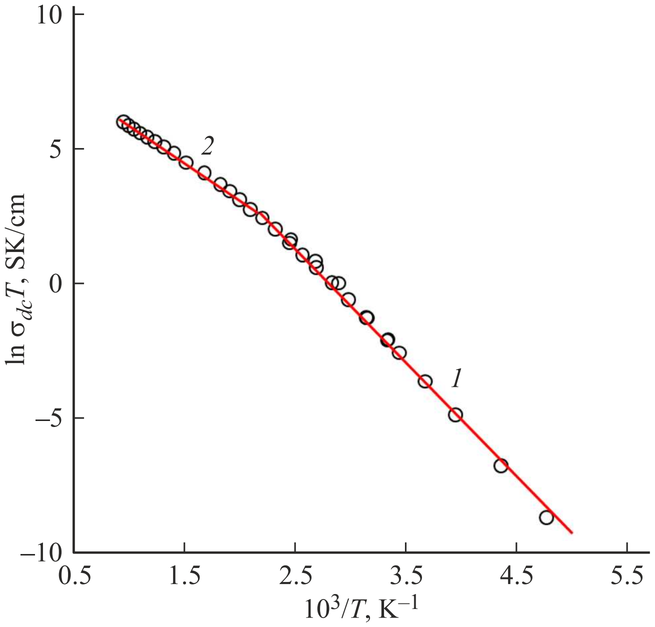

**Figure 1.** Temperature dependence of ion conductivity in coordinates $\lg \sigma_{dc}T$, $10^3/T$ for a superionic conductor $La_{0.95}Sr_{0.05}F_{2.95}$ in the temperature range of 210−1073 K: dots — experiment, straight lines *1* and *2* — calculation using Arrhenius-Frenkel equation at low- and high-temperature areas accordingly.

In the tysonite structure, fluorine ions are distributed in three crystallographic positions of the spatial group $P\bar{3}c1$ at the ratio of $F_1 : F_2 : F_3 = 12 : 4 : 2$ [44–46]. The coordination number of fluorine anions by cations is 4 for $F_1$ and 3 for $F_1, F_2$. The crystallochemical difference between positions $F_2$ and $F_3$ is small, therefore, when interpreting physical properties, they are often grouped into the general group $F_{2,3}$. It is generally assumed [47–50] that the ion transfer below 480 K is mainly associated with the migration of fluorine vacancies $V_F^\bullet$ along structural positions $F_1$.

## 4. Mathematical formalism of Vogel-Tamman-Fulcher

Previously to describe the non-Arrhenius behavior of ion conductivity in cation conductors $KTiOPO_4$ [51] and $Li_{2/3-x}Li_{3x}TiO_3$ [52,53] the Vogel-Tamman-Fulcher equation was used [54–56]:

$$\sigma_{dc}T^{1/2} = \sigma_0 \exp\big(-\Delta H_{VTF}/(T - T_0)\big), \quad (3)$$

where $\sigma_0$ — pre-exponential factor of electric conductivity, $\Delta H_{VTF}$ — enthalpy of electrotransport activation and $T_0$ — characteristic temperature that meets the start of structural disordering of conductivity ions ($Li^+, K^+$). Figure 2 shows the temperature dependence of ion conductivity in Vogel-Tamman-Fulcher $\ln(\sigma_{dc}T^{1/2})$, $10^3/(T - T_0)$ for a single crystal $La_{0.95}Sr_{0.05}F_{2.95}$. You can see that $\sigma_{dc}(T)$

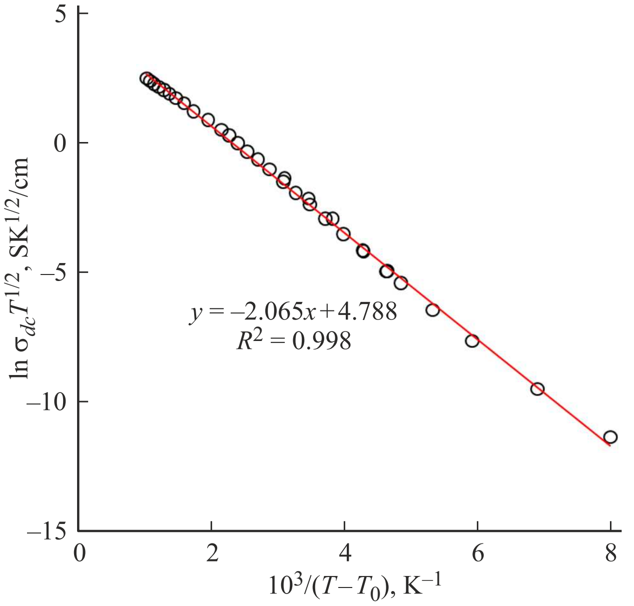

**Figure 2.** Temperature dependence of ion conductivity in coordinates $\ln \sigma_{dc} T^{-1/2}$, $10^3/(T - T_0)$ for a superionic conductor $\mathrm{La_{0.95}Sr_{0.05}F_{2.95}}$ in the temperature range of 210−1073 K: dots — experiment, straight line — estimate using Vogel-Tamman-Fulcher equation.

dependence meets this equation in the entire studied interval of temperatures (correlation coefficient $R^2 = 0.998$). The values of parameters $\sigma_0$, $\Delta H_{\mathrm{VTF}}$ and $T_0$ are equal to $1.2 \cdot 10^2$ SK$^{1/2}$/cm, 0.18 eV and 85 K accordingly.

The cause for non-Arrhenius behavior of dependence $\sigma_{dc}(T)$ seems to be the presence of energy distribution of ion carriers in the studied single crystal as a result of structural microheterogeneity of the solid solution. The process of ion transport in the crystalline structure of solid solution $\mathrm{La_{1-y}Sr_yF_{3-y}}$ is related to fluorine ions overcoming the energy (potential) barriers. In [48] it was found that the height of potential barriers for motion of fluorine ions in tysonite crystal $\mathrm{LaF_3}$ has probabilistic logarithmic Gaussian distribution.

The study of the effect of superionic conductivity at microscopic level and determination of concentration and mobility of charge carriers are relevant objectives for ionics of fluoride materials. In $\mathrm{La_{1-y}Sr_yF_{3-y}}$ superionic conductor with a tysonite structure, hopping conductivity mechanism is implemented and anion carriers — fluorine vacancies $V_{\mathrm{F}}^{\bullet}$ participate in charge transport in thermal activation conditions.

The concentration of charge carriers does not depend on the temperature and is defined by the structural mechanism of heterovalent substitutions of $\mathrm{La^{3+}}$ on $\mathrm{Sr^{2+}}$ cations. Heterovalent substitutions of $\mathrm{La^{3+}}$ on $\mathrm{Sr^{2+}}$result in charge heterogeneity of cation sublattice and spatial heterogeneity of anion sublattice (appearance of point defects $V_{\mathrm{F}}^{\bullet}$). The structurally disordered state of the anionic sublattice has a crystallochemical nature and persists at low temperatures.

With account of the scheme (2), the concentration of mobile fluorine vacancies $V_{\mathrm{F}}^{\bullet}$ is equal:

$$n_{mob} = 2Zy/(\sqrt{3}a^2c), \tag{4}$$

where the number of formula units in the tysonite lattice cell $Z = 6$, $y = 0.05$ is the mole fraction of $\mathrm{SrF_2}$ in solid solution, $a$ and $c$ are the lattice cell parameters. The value of $a$ and $c$ lattice parameters are taken from [31]. For $\mathrm{La_{0.95}Sr_{0.05}F_{2.95}}$ crystal, charge carrier concentration calculated by equation (4) is equal to $9.1 \cdot 10^{20}\,\mathrm{cm^{-3}}$ [18] and is 1.7 % of the total number of anions.

Ion conductivity of crystals $\mathrm{La_{1-y}Sr_yF_{3-y}}$ is determined by characteristics of charge carriers:

$$\sigma_{dc} = q n_{mob} \mu_{mob}, \tag{5}$$

where $q, n_{mob}$ and $\mu_{mob}$ — charge, concentration and mobility of vacancies $V_{\mathrm{F}}^{\bullet}$. Having taken the obtained values of $\sigma_{dc}$ and $n_{mob}$, you can assess the average value of charge carrier mobility. $\mu_{mob}$ mobility values at 210, 400 and 1073 K, calculated using equation (6) are equal to $5.8 \cdot 10^{-9}$, $6.9 \cdot 10^{-5}$ and $2.6 \cdot 10^{-3}$ cm$^2$/(sV) accordingly.

The mobility of charge carriers (fluorine vacancies $V_{\mathrm{F}}^{\bullet}$) in the superionic tysonite nonstoichiometric crystal $\mathrm{La_{0.95}Sr_{0.05}F_{2.95}}$ is considerably higher than the mobility of charge carriers (interstitial ions $\mathrm{F}^{/}_{mob}$) in the fluorite nonstoichiometric crystal $\mathrm{Ba_{0.5}La_{0.5}F_{2.5}}$ ($1.1 \cdot 10^{-7}$ cm$^2$/(sV) at 400 K [50]). High conductometric characteristics of a superionic conductor $\mathrm{La_{0.95}Sr_{0.05}F_{2.95}}$ make it possible to consider it as a promising superionic fluoride conductor for practical applications in ionics of fluoride materials.

## 5. Conclusion

Heterovalent $\mathrm{La_{1-y}Sr_yF_{3-y}}$ solid solution with the tysonite structure is a model system for nonstoichiometric fluoride superionic materials. In a superionic conductor $\mathrm{La_{1-y}Sr_yF_{3-y}}$ the charge carriers are fluorine vacancies $V_{\mathrm{F}}^{\bullet}$, which are formed at heterovalent substitutions of matrix cations $\mathrm{La^{3+}}$ on impurity cations $\mathrm{Sr^{2+}}$.

In the interval of 210−1073 K the ionic conductivity of single crystal $\mathrm{La_{0.95}Sr_{0.05}F_{2.95}}$ increases from $8.4 \cdot 10^{-7}$ to $3.8 \cdot 10^{-1}$ S/cm ($4.5 \cdot 10^5$ times) and has non-Arrhenius behavior. The analysis of the temperature dependence of ionic conductivity is carried out using Vogel-Tamman-Fulcher equation. The parameter values in Vogel-Tamman-Fulcher equation are equal to pre-exponential factor $\sigma_0 = 1.2 \cdot 10^2$ SK$^{1/2}$/cm, activation enthalpy $\Delta H_{\mathrm{VTF}} = 0.18$ eV and temperature characterizing the start of the structural disordering of the anionic sublattice $T_0 = 85$ K.

Non-Arrhenius behavior of ion conductivity seems to be caused by energy distribution of charge carrier hops as a result of structural microheterogeneity of the solid solution. The mechanism of ion transport is discussed in the superionic conductor $\mathrm{La_{0.95}Sr_{0.05}F_{2.95}}$. The application of the mathematical formalism of Vogel-Tamman-Fulcher is of certain interest for further studies of fluoride superionics.

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## Conflict of interest

The authors declare that they have no conflict of interest.

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