Synthesis and transport properties of solid solutions $Sr_{1-x}K_xPbO_{3-y}F_y$ ($0 \leq x, y \leq 0.20$)

V.O. Drozd [a,b,*], A.M. Gabovich [c], S.O. Solopan [d],
S.A. Nedil'ko [b], M. Pękała [e], O.G. Dzyaz'ko [b]

[a] *Center for the Study Matter at Extreme Conditions, Florida International University, University Park, FL 33199, USA*
[b] *Department of Chemistry, Kiev State University, 64 Volodymyrs'ka st, 01033, Kiev, Ukraine*
[c] *Crystal Physics Department, Institute of Physics, Nauka Avenue 46, 03028, Kiev 28, Ukraine*
[d] *Institute of General and Inorganic Chemistry, Kiev, Ukraine*
[e] *Department of Chemistry, University of Warsaw, Al. Żwirki i Wigury 101, PL-02-089 Warsaw, Poland*

Abstract

Polycrystalline solid solutions $Sr_{1-x}K_xPbO_{3-y}F_y$ ($0 \leq x, y \leq 0.20$) were synthesized. Their structural properties were determined by X-ray diffraction. Resistivity and thermoelectric power were measured as functions of temperature. It was shown that a simultaneous substitution of potassium for strontium and fluorine for oxygen results in a transformation of conductivity from semiconductive to metallic at low temperatures. The results are discussed in connection with possible superconductivity in new families of doped perovskites.
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1. Introduction

Strontium plumbate, $SrPbO_3$, is a structural analog of another compound $BaPbO_3$, the latter being a basis of superconducting systems $BaPb_{1-x}Bi_xO_3$ (the maximal critical temperature $T_{c\,max} \approx 13\,K$) [1] and $BaPb_{1-x}Sb_xO_3$ ($T_{c\,max} \approx 3.5\,K$) [2]. Since the ionic radius of Sr^{2+} is smaller in comparison to that of Ba^{2+}, the perovskite-type crystal lattice of $SrPbO_3$ suffers an extremely strong orthorhombic distortion [3]. The ceramics $BaPbO_3$ demonstrates metallic conductivity due to the overlap of O (2p) nonbonding and Pb–O (spσ) antibonding bands at the Fermi level [4]. In the case of $SrPbO_3$, the distances between Pb and O ions increase and the neighboring O–Pb–O angles deviate from the value of $90°$ appropriate to the undistorted perovskites. These structural changes reduce the overlap between 6s and 2p orbitals [3]. Hence, electrical resistivity ρ of $SrPbO_3$ is expected to be larger than that for $BaPbO_3$ and this trend has been actually observed, the former material no longer being a semimetal as $BaPbO_3$ but revealing a semiconductor-like conductivity [5].

As is well known, the search for new oxide superconductors is mostly carried out on the basis of empirical recipes [6,7] (nevertheless, *intuitively* justified by an existing deep theoretical background [8,9]!) rather than first-principles approaches. These prescriptions are similar to Matthias rules for superconducting intermetallides and alloys [10] and more recent superconductivity criteria [11–19].

If one considers phonons as the main bosons, intermediating in Cooper pairing, then there should be certain restrictions on the strength of the required electron–phonon interaction in actual materials. We note, that for oxides the exact nature of the interelectron attraction is not known with confidence [8,9,20,21]. Nevertheless, there is much evidence that the electron–phonon interaction is very important in high-T_c cuprates [22–25] despite the observed predominantly *d*-wave pairing [26,27], more appropriate to the interaction repulsive at short distances [28]. For oxides with lower T_c, there are sound reasons to consider the Cooper pairing isotropic and boson mediators as ubiquitous phonons [29,30].

Therefore, in all cases important in the context of this study the corresponding considerations are actually, although sometimes implicitly, based on a fundamental simple idea that the electron–phonon pairing interaction determines superconduc-

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* Corresponding author. Tel.: +1 305 348 3445; fax: +1 305 348 3070.
E-mail address: drozdv@fiu.edu (V.O. Drozd).

tivity and is strong enough to ensure high T_c's but weak enough to avoid any reconstruction of the parent crystal lattice followed by a concomitant shrinkage of a Fermi surface (FS) and an inevitable drop of T_c [31,32]. Since a current-carrier screening reduces the matrix elements V_{el-ph} of the electron–phonon interaction (see, e.g., [33]), criteria often reduce to a peculiar balance between them and the electronic densities of states (electronic DOSes). Magnetic components should be included into estimates, when necessary, so that the interplay becomes more involved and interesting [16]. On the other hand, the complexity of the problem leads to the inability of existing microscopic theories either to predict new superconducting compounds or to calculate corresponding T_c's with a sufficient accuracy [34]. It is clear that this reasoning is valid for oxide and related materials as well.

Thus, according to general considerations based on the crucial role of the strong electron–phonon interaction, one might expect an appearance of superconductivity in those multi-component oxide systems with variable compositions (bertollides [35]) that occupy phase diagram areas near semiconducting phases. It means that the Cooper pairing should develop in the vicinity of the boundary beyond which a parent electron spectrum is rebuilt and the crystal lattice distorted [36,37]. In particular, it is reasonable to seek for superconducting instability in chemically modified and electrically doped SrPbO$_3$. Interest in studying of SrPbO$_3$ is also stimulated by resistive and magnetic peculiarities, suspiciously similar to manifestations of superconductivity and observed in complex lead oxides. In particular, we mean some evidence in favor of room-temperature superconductivity in Cu$_{24}$Pb$_2$Sr$_2$Ag$_2$O$_x$ [38] and Ag$_x$Pb$_6$CO$_{9+\beta}$ [39], as well as resistive anomalies in the temperature range $190\,K < T < 270\,K$ for a composition Cu$_{24}$Pb$_2$Sr$_2$Ag$_2$O$_x$ [40]. Recently, our group has demonstrated the existence of a diluted superconducting phase in solid solutions Sr$_{1-x}$La$_x$PbO$_{3-\delta}$ hidden for bulk measurements but uncovered by tunnel studies [41].

Besides the strontium plumbate as a basic material, a number of its derivatives with various kinds of substitutions were studied. Thus, a replacement of lead by bismuth, contrary to what happens in the BaPbO$_3$ case [1], results in a current-carrier localization for pristine [42] and doped SrPbO$_3$ [43]. At the same time, doping by antimony almost does not influence electrical conductivity for low concentrations of substituting ions [44]. Heterovalent substitution of La for Sr transfers the system into the metallic state but does not lead to the appearance of the superconducting phase [43,45,46]. Finally, insertion of K$^+$ instead of Sr^{2+} results in an extremely narrow homogeneity region and an electron spectrum transformation, which may reflect either dielectrization or localization effects [47].

All presented doping cases for strontium plumbate are connected to the cation sub-lattice modification. On the contrary, substitution of the oxygen anions by halogens, especially by fluorine, has not been adequately studied, although such substitutions turned out to be quite effective for superconducting cuprates. Thus, upon inserting of fluorine into a semiconducting La$_2$CuO$_4$ halogen ions intercalate into interstitial sites and CuO$_2$

layers oxidize. As a result a superconducting phase emerges with $T_c \approx 40\,K$ [48]. Fluorination of reduced HgBa$_2$Ca$_2$Cu$_3$O$_{8+\delta}$ leads to an enhancement of T_c up to 138 K [49], which is conspicuously higher than the maximal $T_c \approx 134\,K$ achieved in the oxygen flow. When Nd$_2$CuO$_4$ is doped, fluorine substitutes oxygen ions in the crystal lattice sites that leads to the reduction of CuO$_2$ layers and superconducting pairing of negatively charged current carriers [50]. It is worthwhile to indicate the existence of other F-containing superconducting phases Sr$_2$CuO$_2$F$_{2+\delta}$ ($T_c \approx 46\,K$) [51] and YBa$_2$Cu$_3$O$_6$F$_2$ ($T_c \approx 94\,K$) [52].

To fluorinate oxide compounds, various agents are used: gaseous F$_2$, XeF$_2$, NH$_4$F and others, which make it possible to carry out a process at the room temperature or by heating up to relatively low temperatures 100–200 °C. Fluorination using transition-metal difluorides MF$_2$ (M = Cu, Ni, Zn, Ag) requires higher T and results in a contamination of the final product by oxides of these metals. High-T synthesis under large pressure is also an effective method of oxide fluorination. For example, a superconducting phase Sr$_2$CuO$_2$F$_{2+\delta}$ has been observed using such a technique [53].

Taking all the aforesaid into account, we formulated our goal as a synthesis of the system Sr$_{1-x}$K$_x$PbO$_{3-y}$F$_y$ with a subsequent investigation of its structural and transport properties. One might expect that a fluorination would reduce the orthorhombic distortion of the crystal lattice for SrPbO$_3$. While a tolerance factor for SrPbO$_3$ calculated using a Sr ionic radius and a coordination number 12 [54] comprises 0.92, its value becomes 0.94 for the composition Sr$_{0.8}$K$_{0.2}$PbO$_{2.8}$F$_{0.2}$. Therefore, one may expect an increase of the metallic character of resistivity and a possible development of a superconducting state for low enough T.

2. Experimental

Polycrystalline samples Sr$_{1-x}$K$_x$PbO$_{3-y}$F$_y$ ($x = 0.00, 0.05, \ldots, 0.30$) were prepared using a solid state synthesis with Sr(NO$_3$)$_2$, KF and PbO as starting reagents. Salts and lead oxide (II) weighted in the needed proportion were ground in an agate mortar under an ethanol layer and were thereafter heated in an aluminum crucible while steadily increasing T up to 700 °C. Then the reacting mixture was sustained several hours at the indicated temperature, once more grounded and heated in air at 900 °C for 48 h. At a final stage of synthesis the samples were again grounded in the agate mortar, pressed into discs 10 mm in diameter and 1.5–2 mm thick and annealed in air at 800 °C.

The X-ray diffraction investigations were carried out to determine a phase composition and lattice parameters of the obtained compounds. A DRON-3 diffractometer (Cu Kα-radiation, Ni filter) was used. α-Quartz served as an internal standard.

Electro-transport properties were measured in the range $16\,K < T < 300\,K$ using a temperature stabilization with an accuracy of 0.02 K. A four-probe technique was exploited for electrical resistivity $\rho(T)$ measurements. For differential thermoelectric power $S(T)$ studies, a small temperature gradient of about 1 K along a sample was created by a milliwatt heater. An HP 3457A voltmeter was used for precise voltage measurements.

3. Results and discussion

It is known that in fluorinated oxide materials, superconducting cuprates including, one or several coexisting mechanisms of the oxygen for fluorine substitution may be realized [53].

Specifically, (i) one O atom may be substituted by one F atom; (ii) one O atom may be substituted by two F atoms; (iii) F atoms may be located in interstitial sites. All three mechanisms lead to substantial structural {(ii) and (iii)} and electronic-configuration {(i) and (iii)} changes for a fluorinated compound.

To define chemical formulae of the obtained compounds one should bear in mind a possibility of fluorine losses during synthesis due to the substitution of F atoms by atmospheric O atoms in the reacting mixture as well as the indicated above substitution of one O atom by two F atoms. Therefore, stoichiometric indices x and y, attributed to K and F, respectively, are equal only at the beginning, whereas for final products $x \geq y$. Stoichiometry of the synthesized samples was checked by energy-dispersive X-ray spectroscopy (EDS) using Oxford Link ISIS energy-dispersive spectrometer fixed on a Hitachi S-4800 scanning electron microscope. The initial cation stoichiometry of the samples was found to be preserved after high-temperature synthesis. For example, sample with the starting composition Sr:K:Pb:O:F = 0.9:0.1:2.9:0.1 according to EDS analysis has Sr:K:Pb:O:F atomic ratio 0.94:0.07:1:3.2:0.05 which is close to the initial one.

According to the X-ray analysis, the synthesized compounds were single-phase ones with an exception of the composition $x = 0.20$, diffractograms of which revealed PbO reflections (see Fig. 1). It is remarkable that a homogeneity region in the system $Sr_{1-x}K_xPbO_{3-y}F_y$ is much wider than its counterpart in the case of $Sr_{1-x}K_xPbO_3$. In the latter substance, the strontium substitution by potassium was observed in the narrow range of K concentrations $x \leq 0.10$ [47]. On the other hand, the simultaneous replacement of Sr by K and O by F ensures the charge balance in the final compounds. A wider homogeneity interval in $Sr_{1-x}K_xPbO_{3-y}F_y$ as compared to that in $Sr_{1-x}K_xPbO_3$ may be considered as one of the proofs of the fluorine inclusion into the crystal lattice of $SrPbO_3$. In this connection one should take into consideration that to guarantee the electrical balance in the case of the heterovalent substitution of K^+ for Sr^{2+} vacancies must be created in the oxygen sub-lattice. Low oxygen vacancies concentration range in the structure $SrPbO_3$ causes the low solubility of potassium in it.
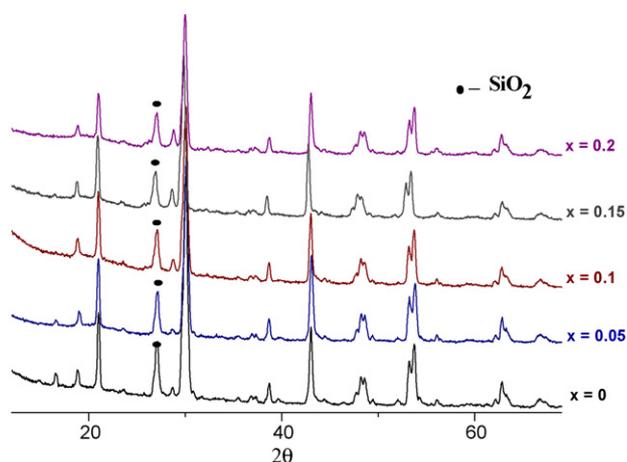

Fig. 1. X-ray diffractograms for samples $Sr_{1-x}K_xPbO_{3-y}F_y$.

Table 1
Crystal lattice parameters for the system $Sr_{1-x}K_xPbO_{3-y}F_y$

x	a (nm)	b (nm)	c (nm)	V (nm^3)
0	0.5885(6)	0.5978(5)	0.8371(9)	0.2945(9)
0.05	0.5879(4)	0.5978(4)	0.8367(9)	0.2941(7)
0.10	0.5900(6)	0.5977(4)	0.8366(8)	0.2950(8)
0.15	0.587(1)	0.599(1)	0.846(1)	0.297(1)
0.20	0.5874(7)	0.5989(7)	0.837(1)	0.2944(9)

All X-ray diffraction patterns of the $Sr_{1-x}K_xPbO_{3-y}F_y$ samples were indexed in the orthorhombic syngony. Calculated crystal lattice parameters are presented in Table 1.

T-dependences of the resistivity ρ for investigated samples are demonstrated in Fig. 2a. It is easy to observe that with increasing x the minima of the curves $\rho(T)$, which correspond to the sign change of the derivatives $d\rho/dT$, i.e. to the formal transition from the metallic to semiconducting conductivity type, shift to lower T. The minima positions fall within the limits 126 K for $x = 0$ and 40 K for $x = 0.20$. For any of the compositions with $x > 0.10$ resistivity is almost T-independent for low enough T.

In [43], the quantum interference phenomena were invoked to explain the shallow minima of $\rho(T)$ and the upturn at low T for the undoped $SrPbO_3$. Such effects are inherent to the disordered systems and involve a weak charge-carrier localization and an inter-electron Coulomb interaction, the strength and character of which depends on the impurity (defect) concentration [55,56]. The indicated quantum mechanical behavior is conspicuous mostly when the effective dimensionality of the electron (hole) sub-system is less than three. The reduced dimensionality may arise not only in layered compounds or wires but also in the case when conductivity is determined by a percolative network [55,57].

In doped and pristine $SrPbO_3$ the mentioned disorder might originate from defects in the oxygen sub-lattice. However, the derivatives $d\rho/dT$ for the investigated solid solutions change their signs at such high T, when inelastic scattering had to completely destroy the phase coherence of the electronic waves. On the other hand, the smeared transition to the semiconductor-like behavior of $\rho(T)$ observed in [43,47] and this work might be induced by the electron spectrum gapping, when definite electron and hole sections of the FS are nested [1,36,37], or by strong on-site Coulomb repulsion (Mott–Hubbard metal–insulator transition) [58]. In the latter case the observed weakening of the tendency towards a transition into the semiconducting state in the doped samples should be considered as a quite natural trend, since extra current-carriers screen the direct Coulomb interaction.

The observed trend towards the semiconducting behavior might also originate from strong localization effects [59]. This point of view is supported by our previous findings of gapped and V-shaped differential tunnel conductivity in various samples of $SrPbO_{3-\delta}$ [42].

Finally, one should make a reservation concerning all possible interpretations of transport phenomena in polycrystalline samples based on "bulk" scenarios. Such speculations do not take into account macro-structural manifestations. At the same time, general runs of curves $\rho(T)$ may be governed by a current-carrier
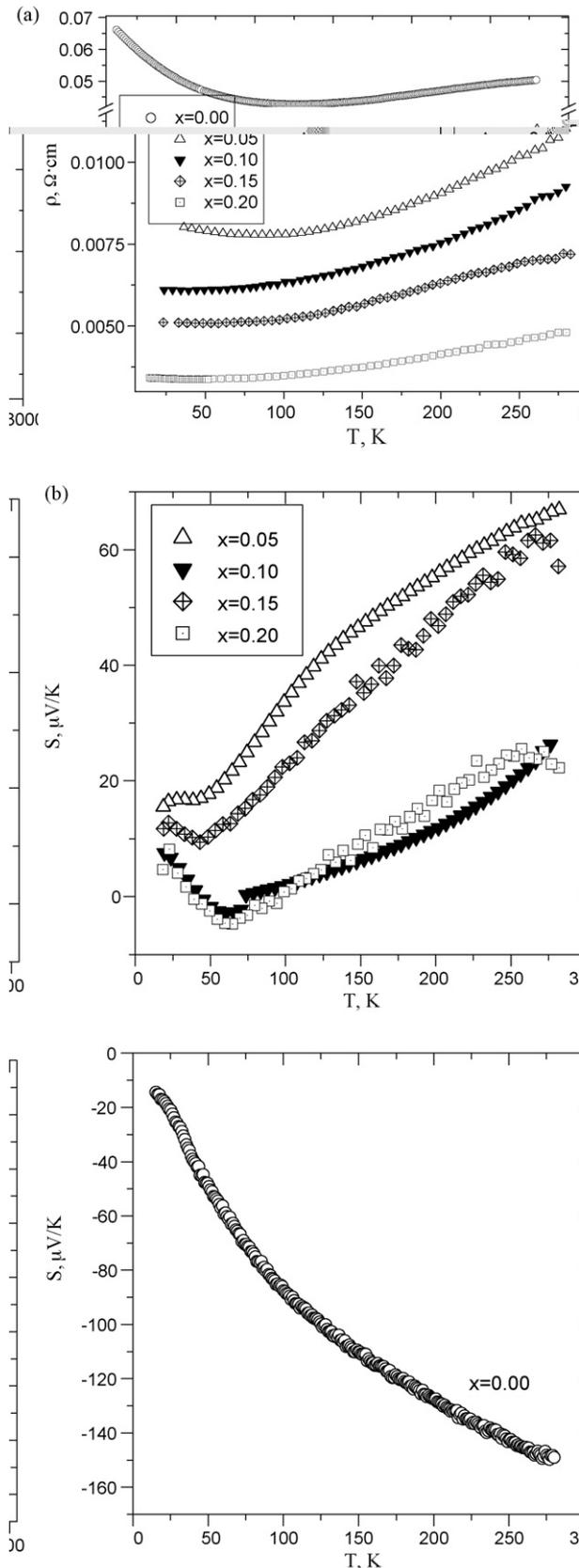
Fig. 2. The temperature, *T*, dependences for the same samples as in Fig. 1 of the resistivity ρ (a) and thermoelectric power (Seebeck coefficient) *S* (b).

tunneling between granules (grains) in disordered ceramics with random variation of the grain capacitances [55,60]. In this case, the optimization of current paths results in the following overall semiconductor-like resistivity of granular composites

$$\rho(T) \propto \exp\left[\left(\frac{T_0}{T}\right)^{1/2}\right], \tag{1}$$

whatever is the type of intrinsic intra-grain conductivity. It is remarkable that the same stretched-exponential dependence is seen in regular periodic systems [61]. Thus, the observation of the dependence (1) may be not the smoking gun of the capacitance-disorder dominance but rather an evidence of elastic or inelastic tunneling through intermediate virtual intra-grain states [61]. Such hopping processes are similar to those in amorphous semiconductors with strong Coulomb effects [59].

As for a decrease of the ρ magnitude while doping, it is most probably induced by a rise of the current-carrier mobility rather than with an increase of their density. Indeed, if one considers a scheme $Sr_{1-x}^{2+}K_x^+Pb^{4+}O_{3-x}^{2-}F_x^-$ when each oxygen anion is replaced by one fluorine ion and simultaneous substitutions of Sr^{2+} by K^+ and O^{2-} by F^-, the current-carrier concentration should not be altered by doping. Nevertheless, one should keep in mind a possibility of at least partial increase of conductivity in samples with substitutions due to a reduction of their oxygen non-stoichiometry. The dual substitution may also cause minor structural distortions with concomitant electron band modifications, which may also provoke concentration changes. All aforementioned effects are interdependent, so that their unambiguous separation is problematic.

To elucidate the nature of current carriers in complex compounds, measurements of the thermoelectric power, *S*, can be very important and useful [62]. Dependences *S*(*T*) obtained for the compositions indicated above are presented in Fig. 2b. As is readily seen, the increase of the substitute concentrations while doping leads to a transition from the electron type of the conductivity (*x* = 0) to the hole one (*x* > 1) for almost all *T*. At the same time, in the case of solid solutions $Sr_{1-x}K_xPbO_3$ the overall thermoelectric power is always negative for all nominal *x* [47].

The attention should be called to the fact that the modifications of the magnitudes and character of the dependences *S*(*T*) for ceramics $Sr_{1-x}K_xPbO_{3-y}F_y$ are non-monotonic. Similar behavior has been also observed for the non-fluorinated samples $Sr_{1-x}K_xPbO_3$ [47]. It is remarkable that for compositions with *x* = 0.1 and *x* = 0.2 the thermoelectric power, being positive at room temperatures, recovers the electron character inherent to undoped ceramics in a rather narrow *T*-range. It argues for the interplay of (at least) two groups of current carriers with opposite charge signs as well as for their distinct behavior while cooling. Hence, the resistivity must be determined by a combined action of several current-carrier groups, so that the observed almost complete absence of the *T*-dependence for the resistivity ρ in the composition $x \geq 0.1$ at low temperatures suggests a compensation of contributions from "metallic" and "semiconducting" components.

4. Conclusions

We carried out studies, which made it possible to find limits for the existence of solid solutions $Sr_{1-x}K_xPbO_{3-y}F_y$ while substituting K for Sr and F for O. These limits constitute up to 20 at%. Substitutions in cationic and anionic sub-lattices transform a semiconductor-like conductance into a metallic one in the low-T region when substitute concentrations increase. The thermoelectric power changes its sign to positive upon doping. The totality of data argues for the existence of several current-carrier groups. The data obtained show that a further investigation of the fluorinated plumbates seems promising to find good conductors and, probably, superconductors with low charge-carrier densities.

Acknowledgements

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