I. INTRODUCTION

The fascinating properties of strontium ruthenates \( \text{Sr}_{n+1}\text{Ru}_n\text{O}_{3n+1} \) (\( n = 1, 2, ..., 8 \)) have garnered enormous attention \([1]\) since the discovery of high-temperature superconductivity in cuprates. Superconductivity in ruthenates was found only for \( n = 1, 2 \) case \([2]\) with \( T_C \) as high as 1.5K \([3]\). Other representatives of this Ruddlesden-Popper family possess peculiar magnetic properties. The most pronounced magnetic order takes place at \( n = \infty \) \( \text{SrRuO}_3 \) is a ferromagnet with Curie temperature \( T_C \sim 165 \text{ K} \); at \( n = 2 \), \( \text{Sr}_3\text{Ru}_2\text{O}_7 \) is an anomalous paramagnet; at \( n = 3 \), \( \text{Sr}_3\text{Ru}_3\text{O}_{10} \) is a metamagnet \([3]\). The Cooper pairing in \( \text{Sr}_2\text{Ru}_4\text{O}_7 \) was well known as a textbook example of the spin-triplet state (odd parity \( S = 1 \), see reviews \([3–6]\) and references therein). Recently, NMR spectroscopy reinvestigation \([7]\) has given compelling evidence that the superconductivity in \( \text{Sr}_3\text{Ru}_4\text{O}_7 \) is likely to be even parity, which unequivocally demonstrates that research on the physical properties of ruthenates is far from being complete.

Another confirmation of this statement comes from the recent fascinating discovery of high-temperature superconductivity in calcium ruthenate, \( \text{Ca}_2\text{Ru}_4\text{O}_7 \) \([8]\). The stoichiometric composition of this material in a single-crystalline form is a Mott insulator, while single crystals with excess oxygen are metallic above 160 K \([9]\). The excess amount of oxygen results in important crystallographic change: the lattice symmetry changes from \( \text{Pbca} \) to \( \text{P}2_1/c \) with \( c-\text{axis} \) extension from 11.9613 Å to 12.3719 Å. Interestingly, electrically induced insulator-metal transition have been detected via infrared nano-imaging and optical-microscopy measurements on bulk single crystal \( \text{Ca}_2\text{Ru}_4\text{O}_7 \) \([10]\). Much more drastic changes occur when the thickness of \( \text{Ca}_2\text{Ru}_4\text{O}_7 \) crystal is reduced to the nanometer range: novel quantum states including high-temperature superconductivity via resistive and magnetic measurements at 64 K have been observed \([8]\). This remarkable finding in micronanocrystals demonstrated how rich the superconducting phenomena in ruthenates can be. It also invigorates the value of polycrystalline materials (ceramics), in which the samples in \([8]\) were originally prepared before subsequent sonification to obtain nanomicrocrystals.

In this rapid communication, we present spectroscopic data in the THz – FIR range obtained for polycrystalline samples of initial stoichiometric composition of \( \text{Sr}_3\text{Ru}_4\text{O}_{4-x}\text{S}(\text{Se})_x \). The choice of this composition was made in result of series of experiments in which the oxygen was partially replaced by S or Se in presence of Cl as a flux at the synthesis of \( \text{Sr}_3\text{Ru}_4\text{O}_{4-x}\text{S}(\text{Se})_x \) and subsequent cationic substitutions for Ru (Fig. 1).

As follows from Fig. 1 typical \( \rho(T) \) dependence of \( \text{Sr}_3\text{Ru}_4\text{O}_7 \) drastically changes into typical strange metal behavior \([11]\) at application of Cl–flux and vacuum during synthesis (details can be found in \([12]\)). Moreover, inclination towards zero resistivity at \( T \rightarrow 0 \text{ K} \) appears at further substitution of chalcogens for oxygen. Since this inclination might have been related with a superconducting phase, we focused our efforts on deepening it using, in particular, cationic substitutions for Ru. The best results were observed with Re–ions \([12]\). At the time of publication \([12]\), the critical temperatures in the range of 20 – 30 K appeared very unusual for ruthenates; however they were later supported by the findings of Ref. \([8]\). Our current spectroscopic data is in support of supercon-
heat treatment of the pellet was performed in high vac-
peratures, powder thermogravimetry was used. Next,
∼a weight loss
increase and decrease at a similar rate (4 hours each, with

calced at 695°C for 10 hours which incurred 6% of
weight loss. The calcined powder was again powder-
tized and heat treated in air, linearly increasing tempera-
ture up to 1350°C and down during 8 hours with 25% of
weight loss. This powder was pelletized and heat treated
again at 1350°C in air for 5 hours with linear temperature
increase and decrease at a similar rate (4 hours each, with
a weight loss ~5%). For optimizing heat treatment tem-
peratures, powder thermogravimetry was used. Next,
heat treatment of the pellet was performed in high vac-
uum (~10^-6 mbar, 650°C, 500 min). The weight of the
pellet did not change noticeably, but the resistivity be-
came smaller. No changes in the sample’s characteristics
were obtained at further vacuum heat treatments.

The crystalline structure of the sample is shown in
Fig. 2. Using EDX well-matching data taken by mul-
tiple analysis of various crystalline areas of this sam-
ple, the composition of the sample was determined as
Sr2.61(Ru1.20,Re0.13)O4. In view of > 25% of

II. EXPERIMENTAL DETAILS

Details on the preparation of Sr2Ru1−xRe2O4−ySey
samples can be found in [12]. Here, we will briefly sum-
marize them. The precursors, RuO2, SrSe, ReO3, SrCO3,
and SrCl2·6H2O were powdered and mixed in stoichio-
metric proportions. A combination of hand and mechan-
ical grinding and mixing was applied. The powder was

weight loss at the thermal treatment quoted above, the
change of the initial stoichiometry is not surprising
(for the convenience, we will keep calling our sample
Sr2Ru1−xRe2O4−ySey). For Se content, EDX microanalysis
is not sufficiently sensitive. WDX analysis revealed
one of the regions from which the compositional data were
taken.

For XRD structural studies (Rigaku Miniflex 600; mea-
surement using Cu-Kα line in the angle interval 2θ =
3–140° with the step 0.01° and scanning rate 0.1–0.5°/s;
phase content and refinement of atomic structural mod-
els by the Panalytical HighScore Plus 3.0e software),
0.5 mm3 of sample was ground in agath mortar. Analy-
sis revealed the presence of three phases: Sr3Ru2O7
(50–60%), Sr2RuO4 (20–30%), and SrRuO3 (10–20%).
The lattice parameters for the phase Sr2(Ru, Re)(O, Se)4
are: a = b = 3.8745(5) Å, c = 12.648(2) Å, space group
I4/mmm. For comparison, the pure n = 1 phase has pa-
rameters a = b = 3.8724 Å, c = 12.7423 Å with the same
space group [13]. This means that the lattice parameters
of this cell are squeezed by 0.74% along the c–axis, and
extended by 0.054% within the ab–plane. This can be as-
sociated with an influence of the uniaxial pressure which
significantly affects the Tc of Sr2RuO4 [1]. The lattice
parameters for the phase Sr3(Ru, Re)2(O, Se)7 are: a =
b = 3.8804(2) Å, c = 20.664(1) Å, space group I4/mmm.
Reference data for n = 2 [15] are: a = b = 3.8872(4) Å,
c = 20.732(3) Å. In this case, all the lattice parameters
are squeezed (0.17% for a, 0.33% for c).

Physical characterization of properties of this sample’s
magnetoresistance, heat capacity, DC and AC magnetic
susceptibility were reported in [12] (sample #643), and
we will use them later when discussing the major topic
of this communication.

For far-infrared (FIR) measurements, a thin (about
50 μm) disk-type slice was dry cut from cylindrical sam-

FIG. 1. Drastic change in resistivity of Sr2RuO4 ce-
ramic samples. While pure samples typically demonstrate
semiconductor-type temperature dependence, the chlorine
flux and vacuum treated samples demonstrate the so-called
strange metal behavior, and chalcogen addition introduces a
downturn. Both features are intriguing (more details can be
found in [11]). The curve corresponding to Re, as well as
the modification of its resistivity in the magnetic field, are
discussed in the text.

FIG. 2. Polycrystalline surface morphology of the measured
sample (JEOL JCM 6000Plus SEM). The rectangle indicates
one of the regions from which the compositional data were
taken.
ple #643 (diameter of 4 mm) using Princeton Scientific WS25 High Precision diamond-impregnated wire saw. One face of this polycrystalline slice was carefully polished with the Precision polishing system Allied Multi-Prep 8” using diamond disc with 1 μm grade to obtain a shiny, highly planar (within 1 – 2° accuracy) surface. The infrared reflectivity spectra were measured near normal incidence (≈ 11°) in the spectral range of 40 – 670 cm⁻¹ (5 – 83 meV) at various temperatures between 5 K and 300 K using a conventional Fourier transform IR spectrometer (IFS 125HR, Bruker) equipped with a liquid He–cooled Si–bolometer and a multi-layer mylar beam splitter. For IR measurements, the polished slice was mounted with the STYCAST 2850ft epoxy glue to the tip of the cone to avoid parasitic back-reflection. A similar cone supports the gold reference mirror. Both cones were attached to the two-position sample holder on the cold finger of the vertical Konti Spectro A continuous-flow cryostat with TPX windows. The design of the cryostat provides a sliding heat exchanger with the precision positioning system controlled by stepping motors. The possible uncertainties related to misalignments during the taking of reference measurements, especially at low frequencies, can be greatly reduced in relative measurements by cycling the temperature without moving the sample [16]. The advantage of this technique is that all temperature-driven distortions of the optical set-up are already frozen around 20 K, thus making it unnecessary to take a reference measurement at every temperature in the range 5-45 K. At energies below 5 meV, the small size of the sample, combined with strong oscillations that stem from standing waves between the optical elements of the spectrometer and cryostat windows, prevent accurate measurements and set a lower limit in our experiment.

III. RESULTS

Final outcome of our FTIR spectroscopic study is shown in Fig. 3 (top panel). The spectral curves for Sr₂Ru₁₋ₓReₓO₄₋ySeᵧ were taken at 5, 10, 15, 20, 25, 35 and 45 K temperatures, then normalized by the 45 K curve. For wavenumbers above 220 cm⁻¹ the set of curves for different temperatures becomes horizontal up to small vertical translations due to noise. At the lower range of wave numbers (40 – 225 cm⁻¹), one can observe a more complex structure. The most noticeable is the first dip which occurs within the range 75 – 175 cm⁻¹ and after the peak at 50 – 60 cm⁻¹. The curve corresponding to T = 35 K has the lowest deviation (i.e., it does not have as deep of a dip compared to other curves). The other curves are more packed together and reach roughly the same peak elevation for the range 50 – 60 cm⁻¹. Moreover, one can notice that the lower the temperature, the higher the peak. This behavior closely resembles that of a typical superconductor (e.g. boron-doped diamond [17, 18]) as shown in the bottom panel of Fig 3. It is important to note that for the graph in the bottom panel the curves correspond to temperatures both above and below the critical temperature (T_c = 6 K). In our case, T_c was not known; however, one can suggest, based on the relative flatness of the T = 35 K curve, that T_c should be slightly below 45 K.

A second dip, though less strong in amplitude, occurs within the range 200 – 225 cm⁻¹. This dip is not accompanied by as high of a peak as the one that was discussed above. One can theorize that this is due to another, larger gap that occurred within the range 75 – 175 cm⁻¹. Based on the fact that the opening of a superconducting gap below T_c results in the behavior shown in the bottom panel, one can suggest that in the top panel at T_c ~ 35 K, two gaps of different magnitude opened up.

The dip in the case of the boron-doped diamond is
located at $\sim 18$ cm$^{-1}$. The major dip in the case of Sr$_2$Ru$_{1-x}$Re$_x$O$_{4-y}$Se$_y$ corresponds to $\sim 125$ cm$^{-1}$, i.e., the gap is by a factor of seven larger than that of the doped diamond. This means that $T_c$ should be about by a factor of seven higher as well: $T_c \sim 42$ K. Physically, the dip in reflectance corresponds to the maximum of absorption, which takes place at the photon energy $\omega = 2\Delta$ in the “dirty” limit. Let us estimate the gap value from our data. The wave number $k \sim 125$ cm$^{-1}$ corresponds to photon energies $\sim 15.5$ meV. If $T_c \sim 42$ K, i.e., about 3.6 meV, then $2\Delta/T_c \sim 4.3$, which is not far from the BCS value 3.53; typically, higher $T_c$ materials have $2\Delta/T_c$ ratio higher than 3.53.

The second gap mentioned above may correspond to another phase or even have a non-superconducting origin. Because of fluctuations, its functional form is not defined as well as that of the larger dip. Thus, it is hard to definitively deduce the critical temperature, as well as other relevant parameters, corresponding to this gap. We will postpone quantitative analysis of this gap until further exploration.

IV. DISCUSSION

Let us consider how this spectroscopic result relates with other facts reported previously on possible superconductivity in this material [12]. We will first compare it with the heat capacity measurement which we will replot in a more elucidating way (Fig. 4). The curve in this figure is compatible with the BCS behavior of the heat capacity of a superconductor with a broad transition, which most likely characterizes superconductivity in the heterophase Sr$_2$Ru$_{1-x}$Re$_x$O$_{4-y}$Se$_y$. To characterize its behavior, as shown in Fig. 4 we applied a 5 T magnetic field to the sample, which reduced the superconducting phase volume mimicking its normal state value for the heat capacity. One can conclude that at about 23 K, the heat capacity has an upturn compared to its normal value, and far below transition, it has values lower than in its normal state (as should be expected from the qualitative BCS pattern of superconductivity). An important question here is why the critical temperature at this measurement is smaller by a factor of two than in the spectroscopic case. A possible answer can be found in the recent results on superconductivity in calcium ruthenates [8]. Unlike 60 K superconductivity in Ca$_2$RuO$_4$ microcrystals, superconductivity in bulk polycrystalline (as well as in macroscopically large crystalline samples) is fully absent [9]. If the mechanism of superconductivity in Sr$_2$Ru$_{1-x}$Re$_x$O$_{4-y}$Se$_y$ is similar to that of Ca$_2$RuO$_4$ (it is hard to expect that the mechanisms are much different!) then $T_c$ in the bulk of Sr$_2$Ru$_{1-x}$Re$_x$O$_{4-y}$Se$_y$ pellet may easily be lower than at the surface layer, even by a factor greater than 2: the heat capacity reflects the bulk property while the IR reflectance is related with the surface layer. The micro-crystallites in the surface layer should be relatively free from the effects of the surrounding material.

The magnetization measurements support this conclusion, Fig. 5. Typically, for AC magnetic susceptibility measurements, the superconducting transition reveals itself as a small jump at $T = T_c$ of the imaginary part of the magnetic susceptibility [19]. Such a jump is indeed observable on the $M''$—curve of our sample at about 40 K, which comes close to the FIR data (Fig. 3). After further cooling, the polycrystalline samples with intergranular connections may have a broad hump [21, 22] similar to the one seen in Fig. 5. Interestingly, the major downturn of the real part of $M'$, as well as the downturn of the magnetic moment measured by the DC magnetometer, starts at about 20 K. It appears that the relative
contribution of the surface effects is small in the case of these quantities, similar to the heat capacity (Fig. 4). These downturns may be indicative of the Meissner effect. These downturns become suppressed at the lower temperatures, as Fig. 1 indicates (see also the enlarged pattern in its inset).

To complete our discussion, we should mention that features similar to the ones which we mentioned here have been reported in the past for the composition Sr$_2$Ru$_{1-x}$Re$_x$O$_{4-y}$Se$_y$ [7]. They were attributed to magnetic fluctuations, similar to other cases [24–28]. Leaving aside the applicability of the magnetic fluctuations to all other facts pointing towards superconductivity in our samples, it is hardly possible that magnetic fluctuations would be able to quantitatively explain the spectroscopic data presented in Section 3 (with the ratio of the gap to the temperature of its opening being close to the BCS-value).

V. SUMMARY

Terahertz spectroscopy, taken together with other observational data on Sr$_2$Ru$_{1-x}$Re$_x$O$_{4-y}$Se$_y$, delivered indications of high temperature superconductivity with $T^\text{onset} \sim 40$ K (Fig. 3). This spectroscopic value for $T_c$ comes close to the estimate of $T_c$ from the measurements of an imaginary part of the AC susceptibility (Fig. 5). Both properties are likely to be determined by the surface layer of our polycrystalline sample. Bulk characteristics, such as the heat capacity (Fig. 4) or the real part of the magnetic susceptibility (Fig. 5) also point towards superconductivity and reveal themselves at lower temperatures. That means that the bulk properties are different from the properties of the surface layer, which sets up a bridge between our findings and the recently discovered superconductivity at 60 K in solitary micronanocrystals of Ca$_2$RuO$_4$ [8]. It is very likely that the mechanism of superconductivity is the same in both cases.

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