Investigation of the zero resistance and temperature-dependent superconductivity phase transition in Pb-Cu-P-S-O compound

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Abstract – In our previous study, we suggested a synthetic method for the replication of PCPOSOS ($Pb_{10-x}Cu_x[P(O_{1-y}S_y)_4]_6O_{1-z}S_z$) and showed precisely measured zero resistance. Through the synthesis method we named Daecheol-Mingi (DM) method, we measured the phenomenon of superconductivity phase transition depending on temperature. Also, we repeated validation of zero resistance of the samples. This paper presents a specific critical temperature for PCPOSOS, demonstrating consistency with the original authors' data.

Keywords – *superconductor, room temperature, ambient pressure, PCPOSOS.*

I. Introduction

Recently, PCPOSOS [1]($Pb_{10-x}Cu_x[P(O_{1-y}S_y)_4]_6O_{1-z}S_z$) (x= 3 ~6, y+z=0.3 ~ 0.4), which has garnered international attention, has been recognized as a potential room-temperature superconductor (RTSC). Although many research teams have attempted to replicate the it, they replicated PCPOO ($Pb_{10-x}Cu_x(PO_4)O$) (x= 0.9 ~1.1) which is the previously known chemical formula. Research teams concluded that PCPOO is an insulator. However, the possibility of superconductivity emerging from the lanakite structure has been suggested through DFT simulation[2]. Also, Kim [3] suggested that the superconductivity of PCPOSOS can be interpreted by BR-BCS theory. To date, unfortunately, only a few studies have seriously attempted to replicate PCPOSOS. However, the potential Meissner effect has been discovered by some research groups[4]. Furthermore, in our previous study, we repeatedly confirmed the zero resistance of PCPOSOS [5]. In this study, we focused on the phase transition depending on temperature, which is crucial evidence of superconductivity. We have repeatedly validated the phase transition and present the critical temperature. The estimated critical temperature aligns with the claims of the original authors [6]. Moreover, for those who may not understand despite the zero resistance data in our previous paper, we kindly provide additional explanations.

II. Details of the synthesized samples

All samples were synthesized using the DM method, same to the approach suggested in the previous study[5]. The samples used in this study are samples #5 and 6, which already showed zero resistance [5], along with the newly synthesized sample #8. As shown in Fig. 1, the synthesized samples uniformly showed a coppery or dark-gray surface color. We already explained that the cross-section of the samples consistently appears with a silver-gray or dark-gray color. To measure the electrical characteristics of the samples, it is recommended to apply probes to the dark-gray or silver-gray areas. Table 1 shows the size of each sample.

(a) sample #5 (b) sample #6 (b) sample #8

Fig. 1. The shape and appearance of the synthesized samples.

Table 1. Size of the samples and copper plate.

	Length (mm)	Width (mm)	Thickness (mm)
Sample #5	17.0	13.0	4.0
Sample #6	17.0	14.0	3.0
Sample #8	16.1	14.2	4.4

III. Current (I) -Voltage (V) measurements

In the measurements, the used current source and voltage measurement equipment were the Keithley models 6221 and 2182A, respectively. The utilized 4-point probe station is M.S.Tech M4P302 model which uses gold-coated probes. We used the well-known four-probe method setting a distance of 1 mm. Fig. 2 shows the measured voltages of the sample #5 for applied current in the range of -100 to 100 mA. Fig. 3 shows estimated resistances of sample #5. The calculated resistances are already lower than copper. when the current approaches zero, the calculated resistance increases rapidly. This behavior is not due to an actual increase in resistance, but rather a limitation of the measuring equipment's minimum voltage. The fact that the measured voltages are already assumed to be zero, similar to the noise level of the equipment, and the slope becomes zero indicates that the resistance is already zero in that applied current range. Pure metals and conductors do not exhibit these behaviors. These behaviors were also exhibited for sample#6 and 8 as well (Figs. 4 and 5) Also, we calculated the average voltage eliminating contact resistance and wire resistances as follows: $V \equiv \Delta V = \frac{V_+ - V_-}{2}$, where V_+ is a voltage in a positive current I_+ and V_- is a voltage in a negative current I_- (see Fig. 6).

As shown in Figs 2, 4, and 5, all samples showed zero resistance. The voltages are not proportional to the applied current (Ohmic behavior) in the range of -100 mA to 100 mA. This aligns precisely with the zero resistance observed in previous research[5]. Additionally, we repeatedly confirmed that this zero resistance phenomenon did not occur when measuring copper plate and insulator for verification and validation of the measurement process and method. Furthermore, Fig. 6 clearly showes that the sampled are in condensed state for the entire range of applied current. Although the sample is amorphous material, the calculated average voltage being 0 indicates zero resistance, providing decisive evidence of the presence of a superconducting component or phase in the current channel.

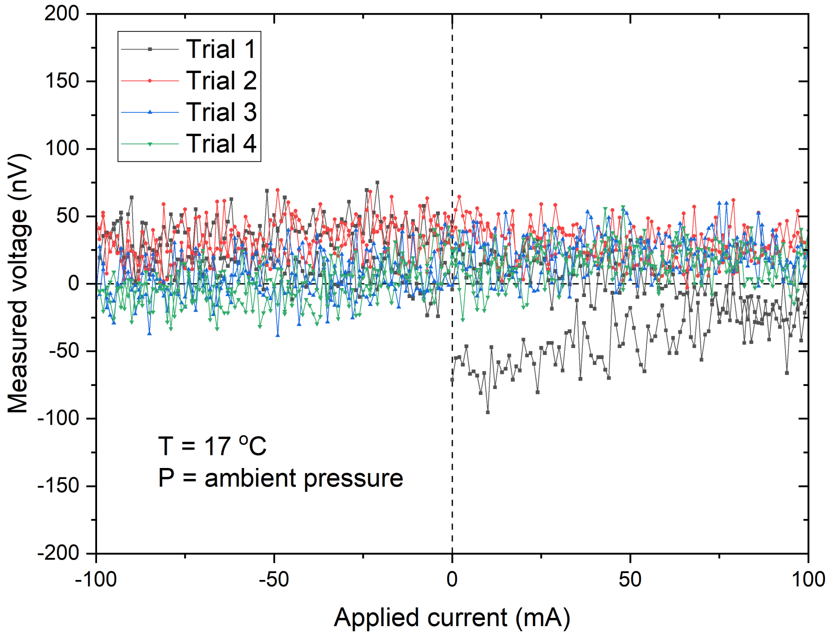

Fig. 2. Measured voltages for applied current ranging from -100 mA to 100 mA (sample #5), (newly measured data).

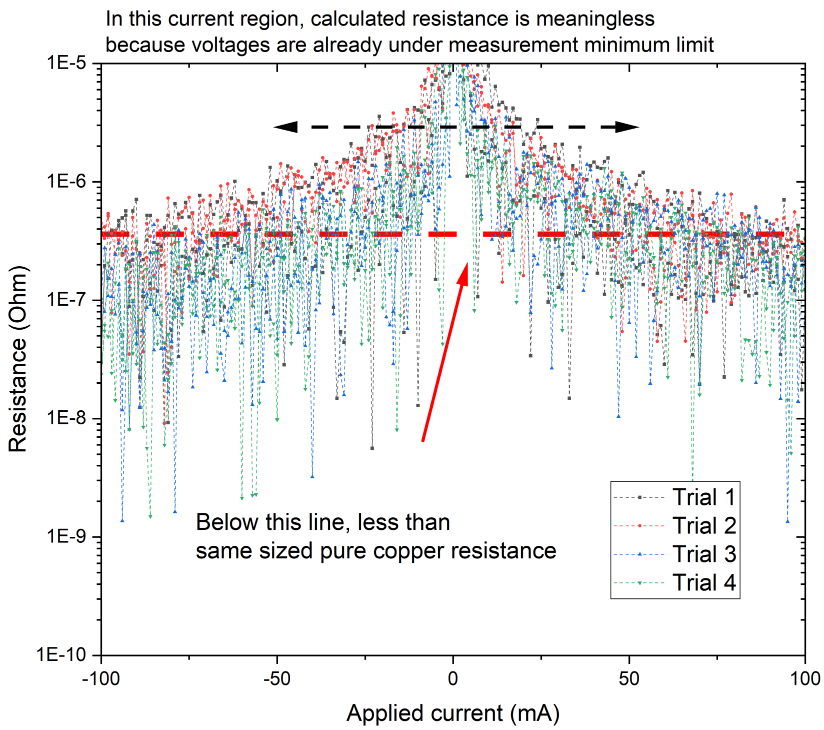

Fig. 3. Resistances of sample #5 for applied current ranging from -100 mA to 100 mA (data from Fig. 2).

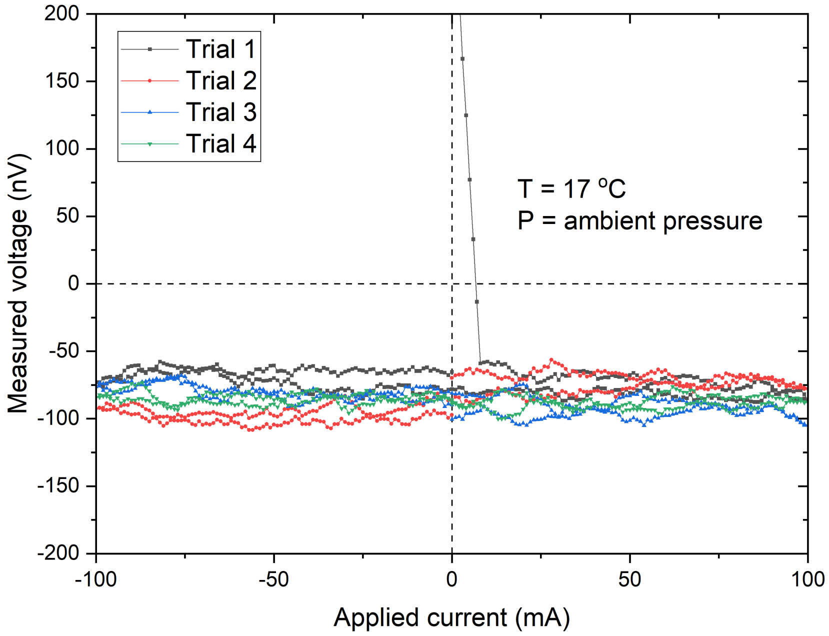

Fig. 4. Measured voltages for applied current ranging from -100 mA to 100 mA (sample #6).

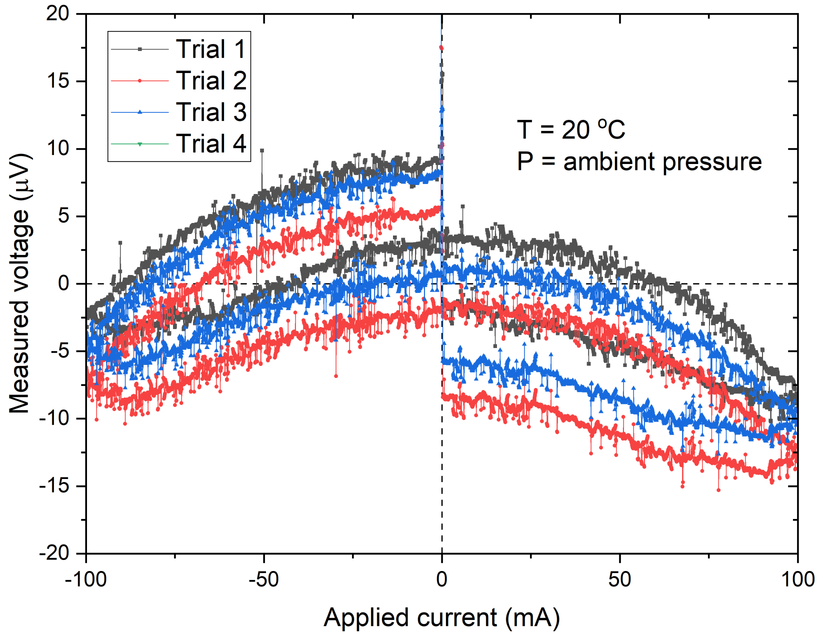

Fig. 5. Measured voltages for applied current ranging from -100 mA to 100 mA (sample #8).

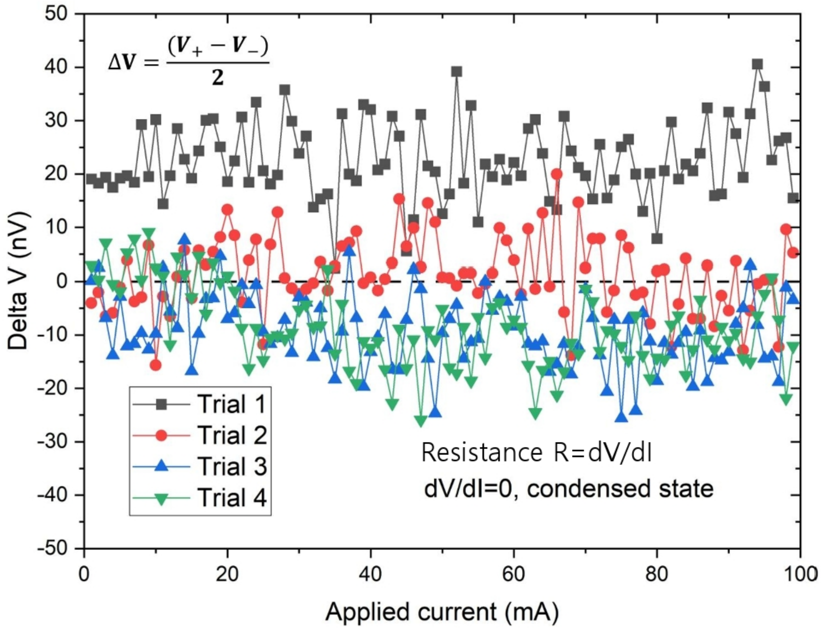

Fig. 6. Calculated averaged voltage for sample#5.

IV. Measurement of the temperature dependence of resistance and phase transition

We measured the temperature dependence of samples #5 and 7. For the measurements, we utilized a ceramic-coated hot plate which uses Mica plate heater (HK Science, Model: GLHP-D). Figs. 7 and 8 show the measured voltages depending on the temperature for samples #5 and 8 when applying a current of 1 μA, respectively. The samples exhibited phase transitions at specific temperatures, clearly indicating the presence of the superconducting phase in PCPOSOS. These transitions were consistently observed. We repeatedly performed the measurement of the voltage versus temperature for sample #8 under applied current conditions of 10 and 100 mA (Fig. 9). Considering heat conduction and temperature gradient, the estimated critical temperatures are around the range of $373 \sim 400\,\mathrm{K}$. Interestingly, the temperature range of the phase transition aligns with the values provided by the original authors [7].

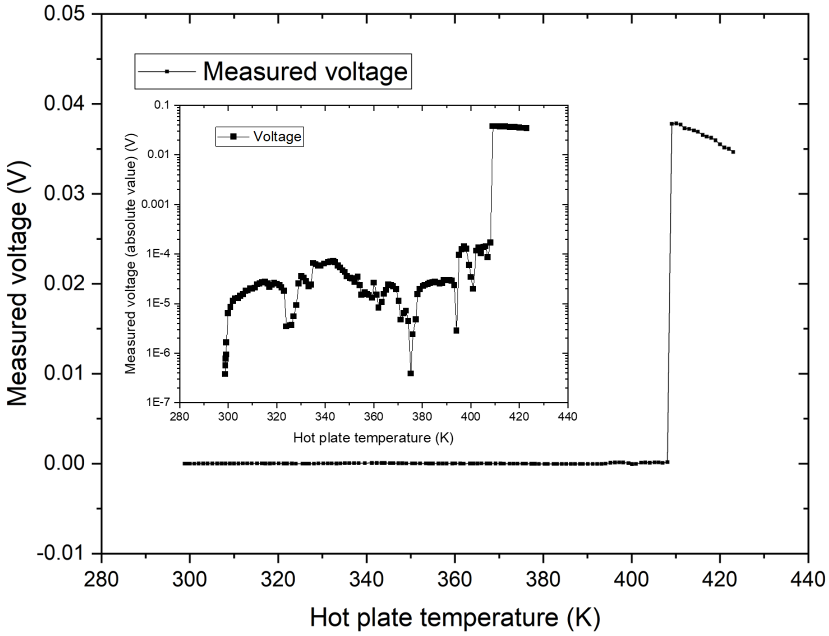

Fig. 7. Temperature dependence and phase transition of sample#5 for applied current of 1 μA.

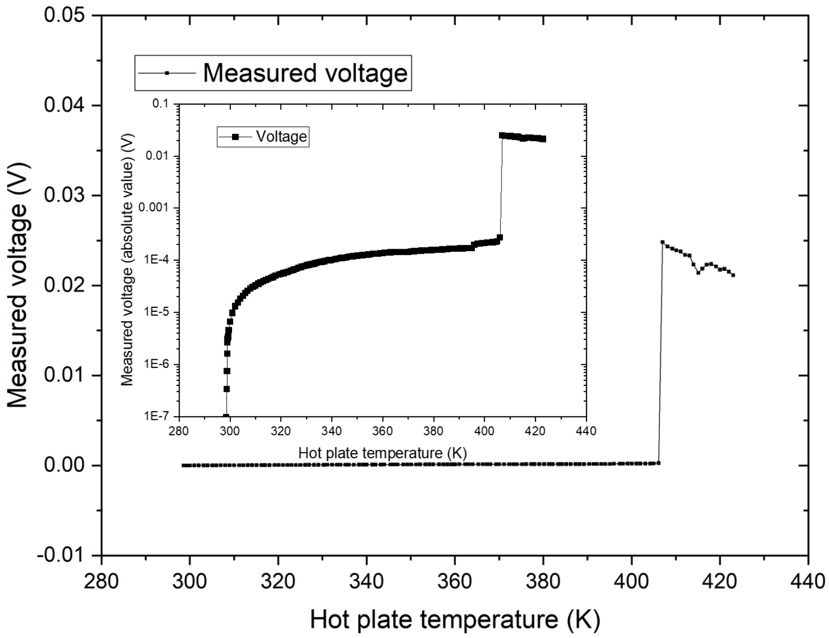

Fig. 8. Temperature dependence and phase transition of sample#8 for applied current of 1 μA.

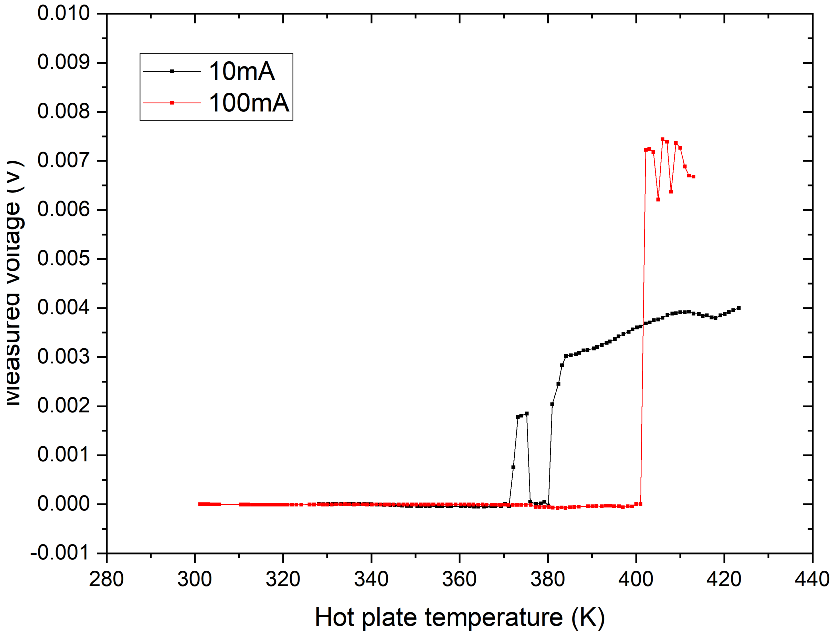

Fig. 9. Temperature dependence and phase transition of sample#8 for applied current of 10 and 100 mA.

We employed delta mode to mitigate the distortion of measured voltage. Delta mode applies a step function shaped current to correct the voltage noise. Fig. 10 shows the temperature-dependent measured voltage of sample #5 in delta mode. When viewed on a logarithmic scale, it is evident when the transition occurs at the same temperature. Despite the use of delta mode in previous transitions, distortion in the measured voltage due to increased resistance of wires and probes with temperature rise is observed. That is, what is significant in Figs. 7 to 10 is not the measured voltage values but the transition phenomenon with respect to temperature. To reiterate, when temperature is applied, the measured voltage is distorted due to the increase in temperature of the measuring device. This implies that before the transition temperature, the calculated resistance is much larger than the actual value. Similarly, when copper is measured using the same method, despite a 10K temperature change, the measured voltage increases tenfold. It is already clear fact that the resistance of copper does not increase so abruptly. This is the reason that we presented zero resistance data for applied current with fixed temperatures in Section III. The most important aspect is the transition phenomenon, which is not observed when measured with a copper plate. To accurately measure voltage, it is necessary to stabilize the measuring device for at least 2 hours at each temperature, which is not an easy task. In this study, we focused on transitions and providing raw data openly.

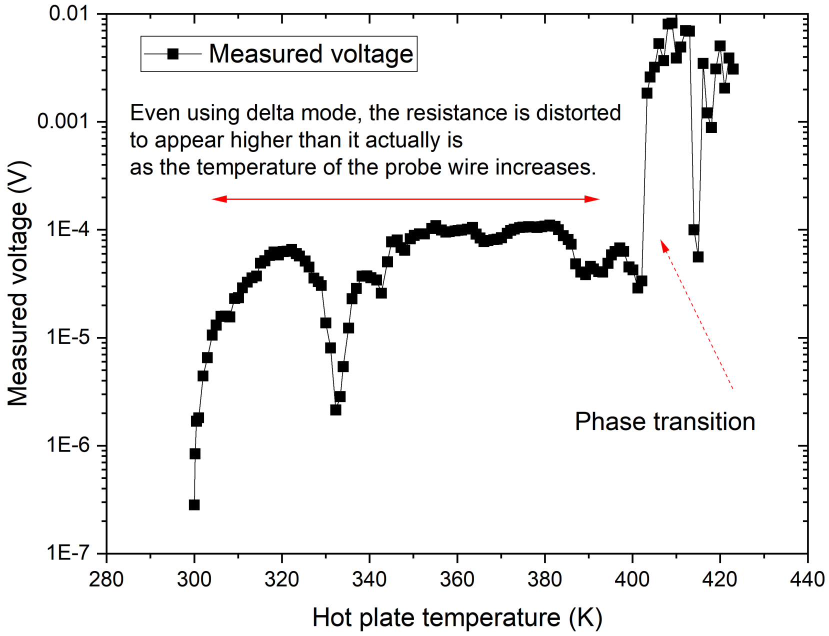

Fig. 10. Temperature dependence and phase transition of sample#5 for applied current of 10 mA, delta mode.

V. Discussion on voltage decay phenomenon of samples

We discovered voltage decay phenomenon in the samples in voltage measurements using a power supply for DC currents (Fig. 11). In the case of sample #8, the voltage decayed exponentially at 500 mA, ultimately converging to around 300 nV. The voltage decay phenomenon is known to occur in superconducting wires or coils. We speculate that if the channels due to the one-dimensional superconducting state have a curved shape, such a phenomenon may occur. We observed a similar phenomenon in sample #6. For sample #6, the voltage decreased ultimately converging to 500nV at 6A.

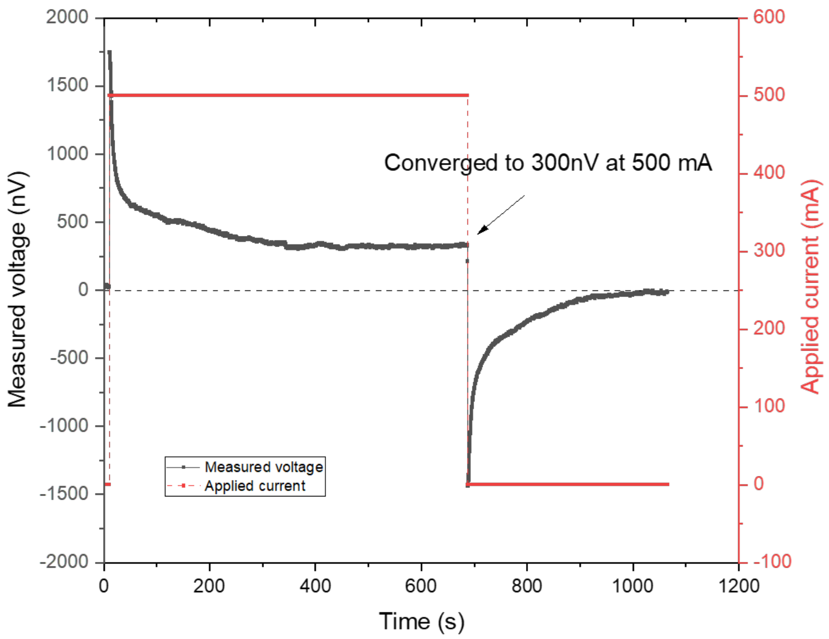

Fig. 11. Voltage decay phenomenon of sample #5 for applied current of 500 mA.

VI. Conclusion

In this study, we have clearly showed the superconductivity of PCPOSOS. Each sample exhibited zero resistance and superconductor-conductor phase transitions, with zero resistance values and critical temperatures clearly matching the data provided by the original authors. We release the raw data and videos of the zero resistance and temperature-dependent measurements publicly available online. Also, we will sequentially disclose XRD analysis, SQUID M vs H Meissner effect, and critical current data in future papers. A scholarly mindset entails a critical attitude. It is important to distinguish between skepticism and denial and maintain an attitude aimed at uncovering the truth. We welcome constructive discussions and questions regarding our samples and data.

Acknowledgment

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Competing interests

Authors declare that they have no competing interests.

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APPENDIX A. RAW DATA AND MEASUREMENT VIDEO LINKS

1.zero resistance of samples, comparison with copper [raw data]:
https://docs.google.com/spreadsheets/d/1JIiuzRpCd_huiwih_y4NqJiq1u4Vb1he/edit?usp=drive_link&ouid=116167257581004355028&rtpof=true&sd=true

2.measurement video of zero resistance for sample#5 [video]:
https://www.youtube.com/watch?v=jKu1qzonsbc

3.raw data of this paper [raw data]:
https://docs.google.com/spreadsheets/d/1tjfgD6fo1bORhAeYMYwIkDZQjOgPSjmJ/edit?usp=sharing&ouid=116167257581004355028&rtpof=true&sd=true

4.measurement video of zero resistance for sample#6 [picture]:
https://drive.google.com/file/d/175kRGcgMc7u6uoAcRK7Y6QERmfsKoCFa/view?usp=sharing

5.measurement video of phase transition for sample#8 [video]:
https://youtu.be/OCLInsjfM9A