The potential zero-resistance phenomenon of the Pb-Cu-P-S-O compound and its synthesis method

Huk Geol Kim[1] and Dae Cheol Jeong[2]

[1]*Independent Researcher, Seoul 06132, South Korea*

[2]*SuperConductor Technology LAB Corp. Ltd., Seoul 06132, South Korea*

Abstract – We have developed a new synthesis method for producing the Pb-Cu-P-S-O compound samples in order to replicate zero resistance of the PCPOSOS ($Pb_{1-x}Cu_x[P(O_{1-y}S_y)_4]_6O_{1-z}S_z$), which we refer to as the Daecheol-Mingi (DM) synthesis method. The potential zero resistances of the samples were observed through I-V measurements.

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

I. Introduction

Recently, the potential room-temperature (RT) superconductor at atmospheric pressure has garnered global attention [1,2]. However, over the past 6 months, despite numerous attempts by researchers worldwide to replicate it, the zero-resistance phenomenon has not been observed. Many researchers have produced samples exhibiting semiconductor characteristics [3] contrary to assertions made by the original research team. This has sparked significant doubt. The primary cause of this problem is considered to be the mis-specification of $Pb_{1-x}Cu_x(PO_4)_6O$ (PCPOO) (LK 99) (0.9 <x<1.1) in the papers that were published [1,2]. Subsequently, the research team announced a new chemical formula ($Pb_{1-x}Cu_x[P(O_{1-y}S_y)_4]_6O_{1-z}S_x$) (PCPOSOS x= 0.3 ~0.6, y+z=0.3 ~ 0.4) including a superconducting component, CuS, interpreted by BR-BCS theory [4,5]. A second reason is the considerable difficulty in synthesizing an RT superconducting sample, despite its apparent simplicity. Addressing these two problems is crucial to usher in the era of room-temperature superconductors. Furthermore, the M-H curve was measured in PCPOSOS [6]. The flat bands were theoretically found [7].

In this letter, we present a novel and straightforward synthesis method for producing the Pb-Cu-P-S-O compound samples akin to single crystals and for measuring potential zero resistance phenomenon in the synthesized samples.

II. Synthesis of the Pb-Cu-P-S-O compound

The synthesis process is as follows: powders of Pb, Cu, P, and S are placed into a ceramic crucible. Oxygen can be absorbed in the air atmosphere. An oxygen cutter is used for this synthesis method, with a mixture of LPG gas and oxygen set at a 1:1 ratio. Due to the influence of surrounding temperature and pressure conditions during synthesis, significant variations in the synthesized product exist depending on the heating time. However, generally, the heating process can be done within the range of 2000 to 3000°C for approximately 0 to 10 minutes. The resultant material is quenched in water, aiming for a metallic appearance with a copper-colored surface and a silver-colored interior.

In general, the resulting products are either a ceramic or a combination of ceramic and metal phases, or a single crystal with a CuS peak in XRD data as a characteristic of PCPOSOS. When breaking the ceramic, only the metal part contained inside should be collected. Magnetic properties can be investigated for those metal parts. If carbon is introduced during the synthesis process, black-colored ceramic occurs. It is important to minimize carbon involvement during the process.

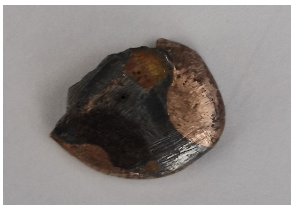
If the electrical resistance of the metallic resultant is higher or its diamagnetic characteristics are weaker than that of the same-sized copper, the heat treatment process can be repeated. Repeating the synthesis process may enhance the diamagnetic characteristics of the samples. This repetition of heat treatment can involve reheating a single sample or reheating multiple samples for synthesis. Due to the intellectual propriety issue, the detailed mass ratio of each component One important thing is, S and P must be added in greater mass ratio than Cu because of evaporation. We release a video of the synthesis process online. powder information will be addressed in our future work. Fig. 1 shows the appearance of the heat treatment in the synthesis process.

As shown in Fig. 2, the synthesized samples uniformly exhibit a coppery surface color. However, upon inspecting the cross-section, the interior of the samples consistently appears with a silver-gray color. We would recommend measuring the dark-gray or silver-gray region rather than the surface when conducting I-V measurements. Table 1 shows the size of each sample and the copper plate.

Fig. 1. Heat treatment process of the Pb-Cu-P-S-O compound using oxygen-cutter.

(a) sample #3

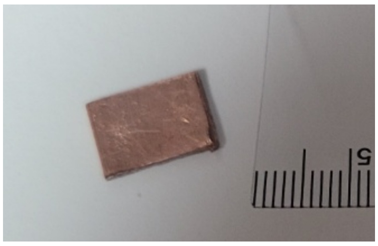
(b) sample #5



(c) sample #6

(d) sample #7

(e) cross-sectional view of sample #3



(f) copper plate

Fig. 2. The shape and appearance of the synthesized samples and copper plate.

Table 1. Size of the samples and copper plate.

	Length (mm)	Width (mm)	Thickness (mm)
Copper plate	14.0	10.0	2.0
Sample #3	21.0	5.0	2.0
Sample #5	17.0	13.0	4.0
Sample #6	17.0	14.0	3.0
Sample #7	12.0	8.0	2.0

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. We used the well-known four-probe method with a V prob electrode interval of the distance of 2 mm. We have measured the I-V behaviors of the samples and a copper plate. Fig. 3 shows the measured voltages of the copper plate for applied current in the range of -100 to 100 mA. The measured source data show some differences in the range of -50 to 50 mA, but overall, it displays a reasonably typical linear I-V behavior for copper (Fig. 3). We calculated the average voltage of all data through $V \equiv \Delta V = \frac{V_{+}-V_{-}}{2}$ removing contact resistance and wire resistances, where V_{+} is a voltage in a positive current I_{+} and V_{-} is a voltage in a negative current I_{-}. The resistance of the copper is determined to be approximately 2.8749 ($\pm$1) $\times$ $10^{-6}\Omega$ from slope through the fitting of the averaged I-V curve, as shown in Fig. 4. Calculating the resistivity of 1.875 ~3.875 $\times$ $10^{-8}\Omega$m confirms its alignment within the typical range of the known resistivity for copper.

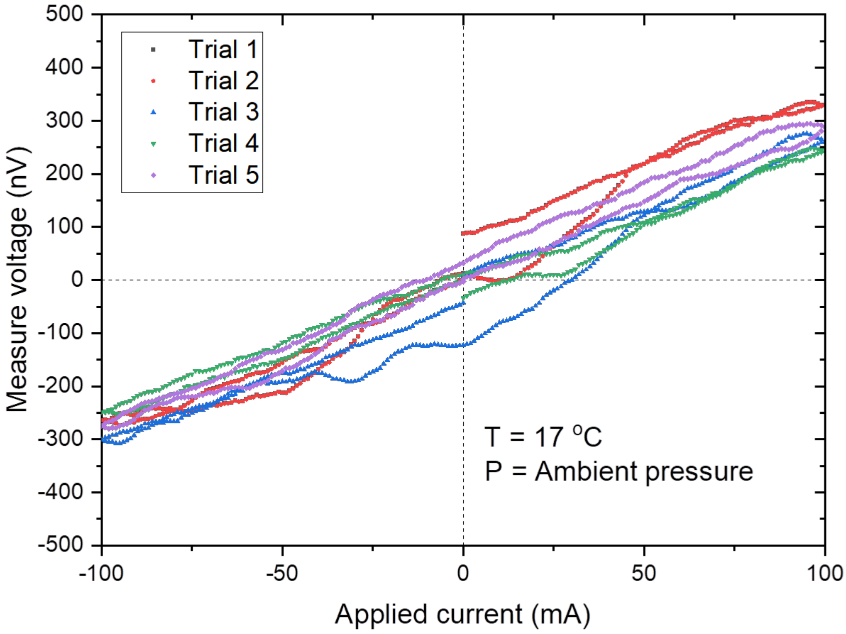


Fig. 3. Measured voltages for applied current ranging from -100 mA to 100 mA (copper plate).

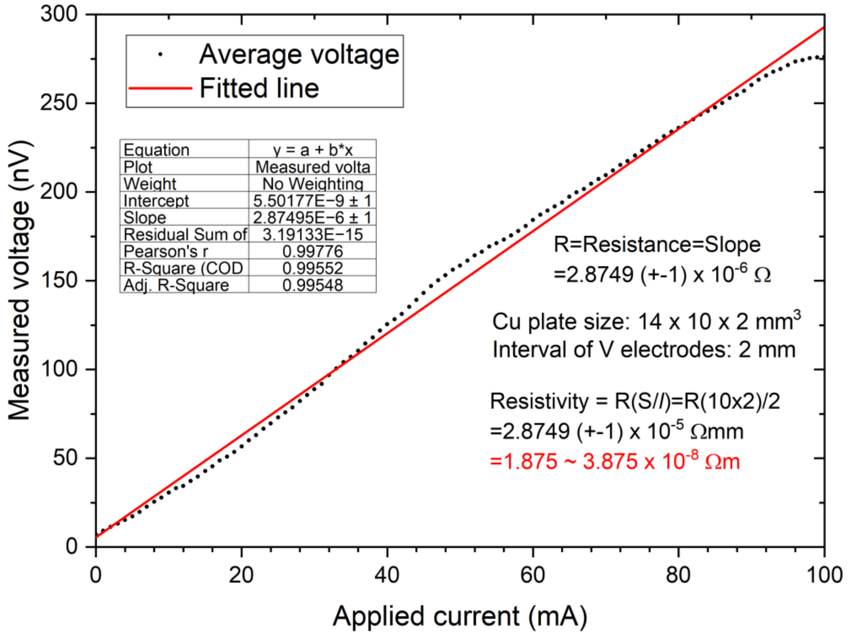


Fig. 4. Averaged voltage (black dots) for applied current ranging from 0 mA to 100 mA (copper plate) and its fitting (red line).

The measured voltage versus applied current for sample #5 is shown in Fig. 5. Interestingly, the measured voltage for sample #5 showed a very low voltage for the applied current, even when compared to the copper plate. Furthermore, the voltage did not appear to be proportional to the current (Ohmic behavior) in the range of -50 mA to 50 mA. We calculated the upper, lower, and total average values of the measured voltage for the range of 0 to 100 mA (Fig. 6), upper (lower) average means measuring with increasing (decreasing) current to 100 mA, respectively. The average voltage of all data is extracted through$V \equiv \Delta V = \frac{V_+ - V_-}{2}$, removing contact resistance of electrodes and wire resistances, where V_+ is a voltage in a positive current I_+ and V_- is a voltage in a negative current I. This is a technique to measure zero resistance. The measured voltage remains noise within the range of 50 mA, which is regarded as zero resistance of the superconducting condensed state. Upon calculating the resistance at A point of a pair breaking or forming (Fig. 6), it is found to be approximately $1.5 \times 10^{-7}\Omega$, indicating one order lower than resistance compared to copper (Fig. 4). Resistivity is evaluated as approximately $3.9 \times 10^{-10}\Omega$m, when assumed that sample is 100% single phase (a cross-section of sample is equal to a cross-section of the current channel), although homogeneity of real sample is much smaller than the single phase. The obtained resistivity of sample #5 becomes the maximum value, but the true value is much smaller due to the mall cross-section of current channel. Thus, the presence of the condensed state of R=0 below 50mA is decisive evidence that sample #5 has a superconducting component or phase.

Such low resistance was consistently observed in samples #6 and 7 as well. Fig. 7 shows the I-V curve measured in sample #6. Comparing it with Fig. 5, it can be seen that the voltage approaches zero within almost the same range. It should be noted that this flat slope was confirmed in the dark-gray part of sample #6. The surface of the sample was relatively smooth and was confirmed to conduct electricity well. Moreover, Fig. 8 shows zero resistances with the superconducting condensed state extracted by the zero resistance measurement method for sample #6 using another data set.

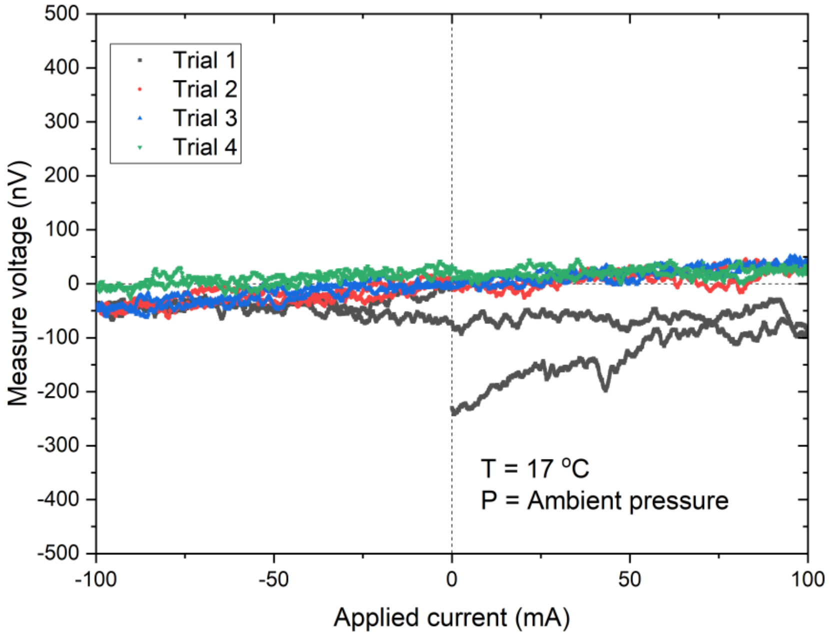


Fig. 5. Measured voltages for applied current ranging from -100 mA to 100 mA (sample #5).

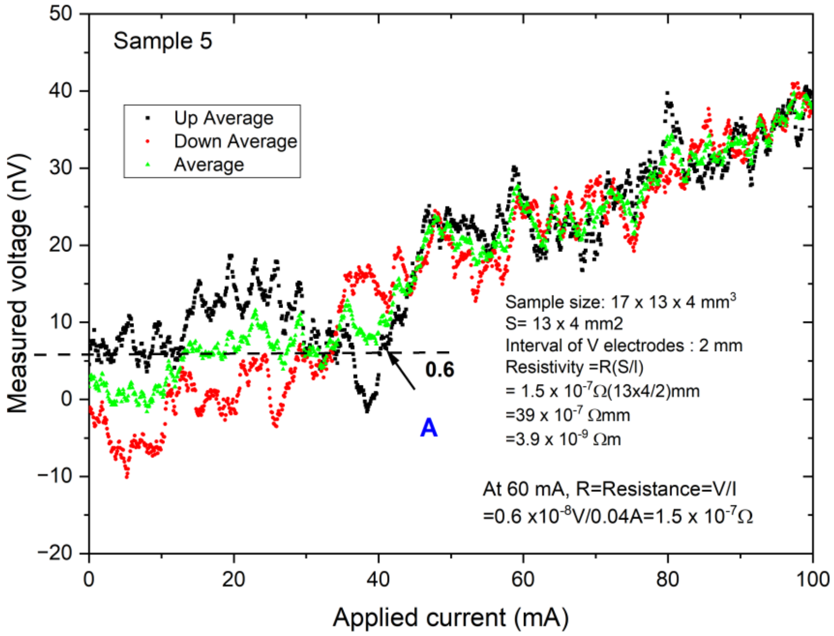


Fig. 6. Averaged voltage for applied current ranging from 0 mA to 100 mA (sample #5).

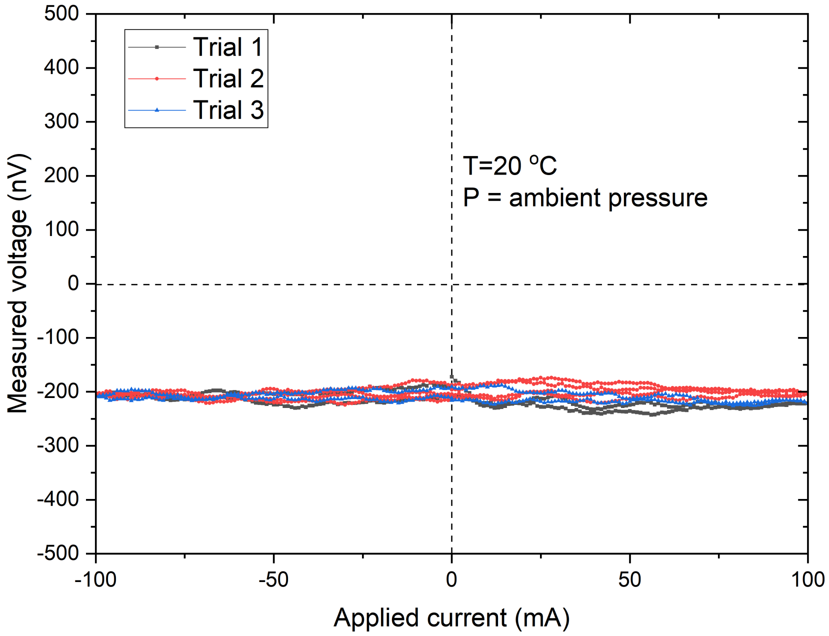


Fig. 7. Measured voltages for applied current ranging from -100 mA to 100 mA (sample #6).

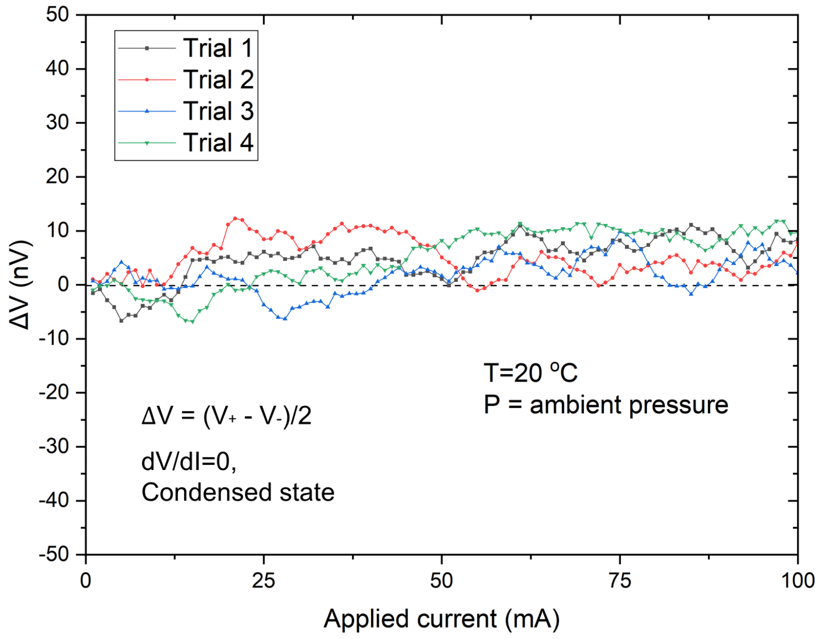


Fig. 8. Zero resistances measured for sample #6.

Furthermore, we conducted measurements on sample #5 and the copper plate using the 4-point probe station with gold-coated probes for cross-validation. The utilized 4-point probe station is M.S.Tech M4P302 model. Figs. 9 and 10 represent the measured voltages of the copper plate for applied current ranging from -100 to 100 mA for each copper plate and sample #5 , respectively.

As shown in Fig. 8, the copper plate exhibits typical linear I-V behavior of a conductor, indicating successful cross-validation when compared to Fig. 3. However, as it can be seen in Fig. 10, for sample #5, the voltage remained remarkably consistent across the range of -100 mA to 100 mA, exhibiting distinctly different I-V behavior from the copper plate. The voltage tends clearly toward zero, suggesting a condensed state where zero resistance is measured.

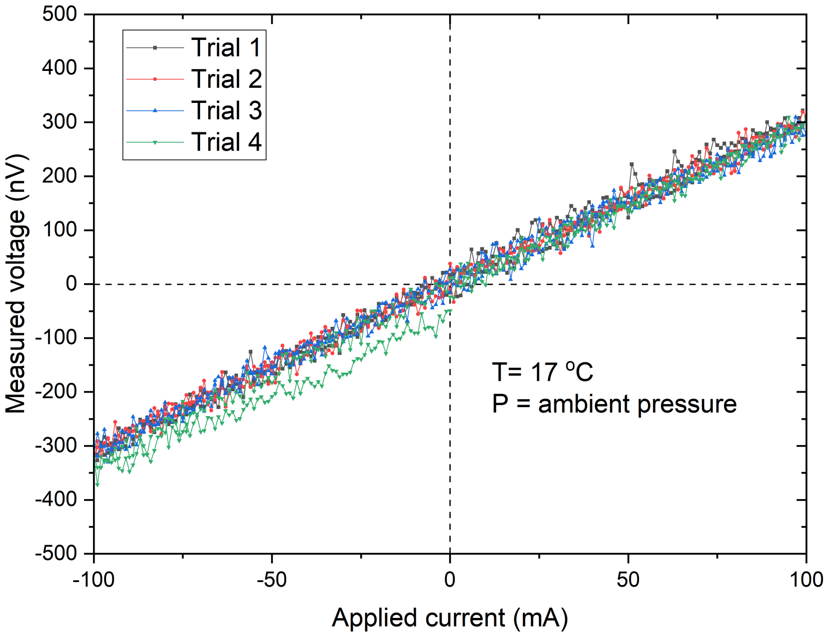


Fig. 9. Measured voltages for applied current ranging from -100 mA to 100 mA using the 4-point probe station (copper plate).

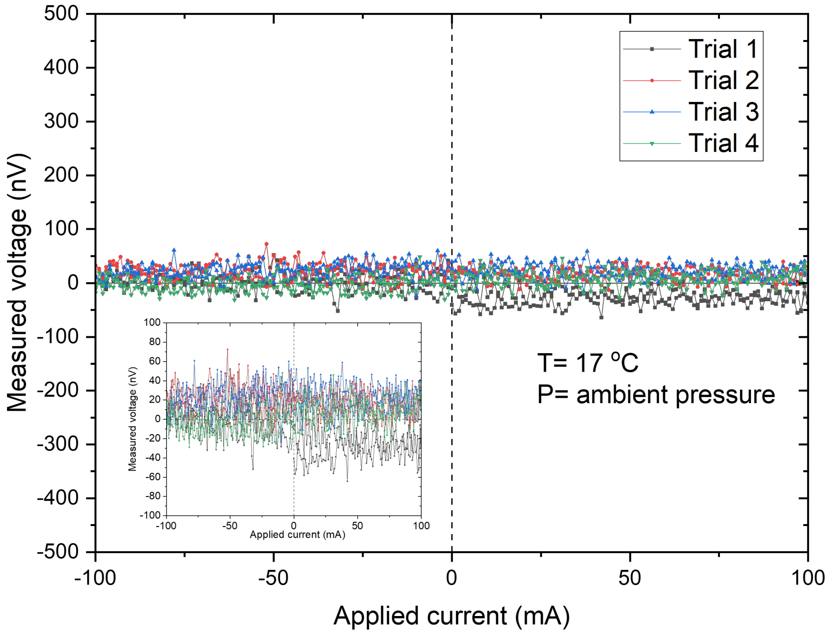


Fig. 10. Measured voltages for applied current ranging from -100 mA to 100 mA using the 4-point probe station (sample #5).

V. Conclusion

In summary, in order to resolve an emerging problem in the field of the RT superconductor, we developed a synthesis method of the Pb-Cu-P-S-O compound similar to possible RT superconductor, PCPOSOS, and measured the potential zero resistances (noises) regarded as the superconducting condensed state. These technologies can be applied to applications of RT superconductors. Despite measuring equipment limitations, our findings suggest promising avenues for further investigation. We will release the raw data included in the paper online. Future research will focus on overcoming these limitations and exploring the compound's behavior under higher currents. We will release our research content to include results such as temperature dependence, ZFC-FC, and M vs H measurements soon.

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Competing interests

Authors declare that they have no competing interests.

*Corresponding author : Hyun-Tak Kim, hkim22@wm.edu

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