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From high-temperature superconductivity to room-temperature superconductivity: From ambient to high pressure; from very high pressure to ambient again!?

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Abstract. This article will first briefly review the impressive advancements made in high-temperature superconductivity (HTS) before the arrival of room-temperature superconductivity (RTS). Accompanying the advancements made in superconductivity science and technology over the last century, a solid experimental framework concerning the search, development, and even authentication of new discoveries has been established. All these can serve as valuable references in the infancy of RTS research. In this spirit, we will comment on the current status of rare-earth hydride RTS and present our preliminary negative results on Lu-N-H and LK-99, the two most studied materials in the search for RTS in the last few months, although several more reports of negation than affirmation have appeared.

1. Introduction

Ever since the discovery of superconductivity in Hg with a transition temperature T_c of 4.2 K by Kamerlingh Onnes in 1911, its scientific excitement and technological potential has lured innumerable great minds to expend tremendous efforts in physics and material science. As a result, great progress has been made in all areas of superconductivity research and development. Raising the superconducting temperature T_c has become the major driving force for the sustained effort and has well served the advancement of superconductivity science and technology. As the search for superconductors with higher T_cs has proceeded, the targeted T_c has risen to match operational temperatures of devices that have become realistic, as shown in Fig. 1. Such practical operating temperatures have included 20.3 K, the boiling point of liquid hydrogen, after stoichiometric Nb_3Ge film with a T_c of 23.2 K was obtained in 1974 [1]; 77 K, the boiling point of liquid nitrogen, when the $ReBa_2Cu_3O_{7-\delta}$ series with a T_c above 93 K was discovered in 1987 [2,3]; 100 K, the cargo bay temperature on the Space Shuttle in orbit opposite the sun, when $Bi_2Sr_2Ca_2Cu_3O_{10-\delta}$ with a T_c of 110 K was discovered in 1988 [4]; 120 K, the boiling point of natural gas, when $Tl_2Ba_2Ca_2Cu_3O_{10-\delta}$ with a T_c of 120 K was discovered in 1988 [5]; and 145 K, the boiling point of Freon, when the T_c of $HgBa_2Ca_2Cu_3O_{8+\delta}$ was pushed up to 164 K under 30 GPa in 1994 [6]. The ultimate goal is to sustain superconductivity at or above 300 K, so-called "room-temperature superconductivity" (RTS), to exploit this exceptional property for its many applications in science and technology so that we may create an environmentally sustainable world for us all. Following the recent intense activities of scientists and so-called "citizen scientists" on "room-temperature superconductors," such a goal appears to be within reach. In this article, we shall present our preliminary results on two systems, Lu-N-H and Pd-Cu-S-P-O (LK-99), that have been covered extensively by the news media and discussed by the scientific communities, following a brief recount of the long and tedious, yet exciting road to high-temperature superconductivity (HTS), which may provide lessons for moving the science forward more efficiently and reducing the confusion RTS currently faces.

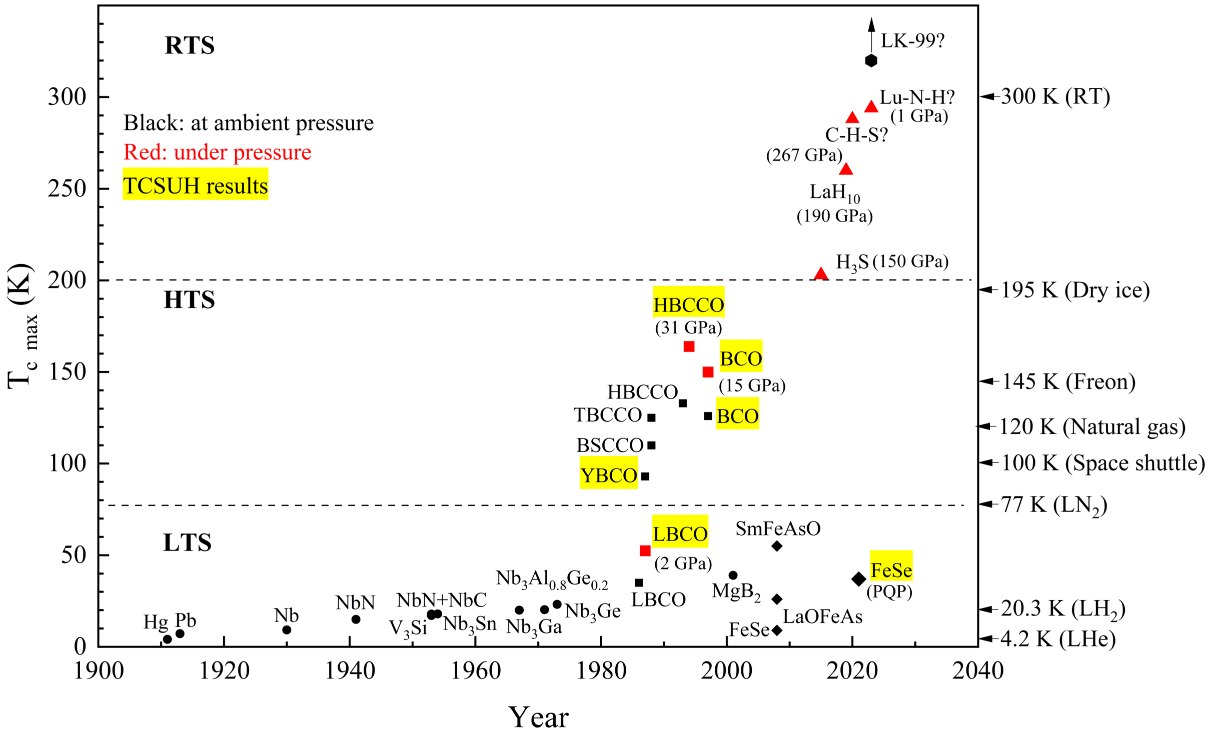

Fig. 1. Evolution of T_c with time, *i.e.* low-temperature superconductivity (LTS), high-temperature superconductivity (HTS), and room-temperature superconductivity (RTS).

2. Low-temperature superconductivity (LTS), high-temperature superconductivity (HTS), and room-temperature superconductivity (RTS)

Superconductivity is the manifestation of the macroscopic quantum state of a superconductor. Its occurrence requires electron-pairing, which in turn requires an additional attractive force to overcome the Coulomb repulsion. The higher the T_c, the greater the attractive interaction required. In the Bardeen-Cooper-Schrieffer (BCS) theory, the electron-phonon interaction provides the attraction. As T_c has increased, different types of interactions of increasing strength have been proposed, although they remain within the BCS framework. However, the ever-increasing strength of interaction between electrons will invariably trigger different kinds of electron and/or lattice instabilities in the materials that forbid the realization of higher T_c. This is the main challenge for scientists in the field.

For the simplicity of discussion, we have roughly grouped the long history of superconductivity research into three periods according to the superconducting transition temperature T_c achieved, namely, the low-temperature superconductivity (LTS) period, the high-temperature superconductivity (HTS) period, and the room-temperature superconductivity (RTS) period, as shown in Fig. 1, where the record high T_c and compound are shown as a function of discovery time, and the targeted operational temperatures are also shown on the right vertical axis. We take the liberty of defining low-temperature superconductors as materials with a T_c up to ~ 21 K, the boiling point of liquid hydrogen (mostly intermetallics); high-temperature superconductors as those with a T_c above 77 K, the boiling point of liquid nitrogen (mostly cuprates); and room-temperature superconductors as those with a T_c above 200 K (mostly hydrides), which Eremets first claimed in H-S. Such a grouping is by no means definitive nor

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rigorous, especially for RTS. For instance, in the 1970s, when asked where to find room-temperature superconductors, Bernd Matthias, the most successful discoverer of new superconductors at the time, simply said “go to the edge of the universe”. He was correct because the residual temperature of the universe after the Big Bang is 3 K, and so is the temperature at the edge of the universe. Superconductors with a T_c above 3 K could thus be considered “room-temperature superconductors” since they can be operated at the ambient condition of the edge of the universe. However, it would be impossible for a mortal to reach this distance since it would take tens of billion years, even traveling at the speed of light. If recent reports of superconductivity at ~ 300 K are proven, there will be no need for such impossible travel to achieve the goal of RTS.

As mentioned earlier, we have plotted the evolution of record high T_cs of major superconductors as a function of the time of their discoveries in Fig. 1. The discovery of superconductivity at 4.2 K in Hg started the epoch of superconductivity. Ensuing studies showed that superconductivity is a thermodynamic state [7], independent of the history or the thermodynamic path taken to reach it. A genuine quantum superconducting state must therefore possess two characteristics: zero resistivity, an intrinsic property of the sample that is independent of its dimension, and expulsion of the magnetic field from within, different from an ordinary good or even perfect conductor that can repulse external magnetic fields. For a superconductor to function, it must be operated in a three-dimensional space (Fig. 2) at a temperature below its transition temperature T_c, in a magnetic field below its critical field H_c, and when the electric current density through it does not exceed the critical value J_c. Raising these three critical values, especially T_c, has become the major research focus of scientists and engineers in the field.

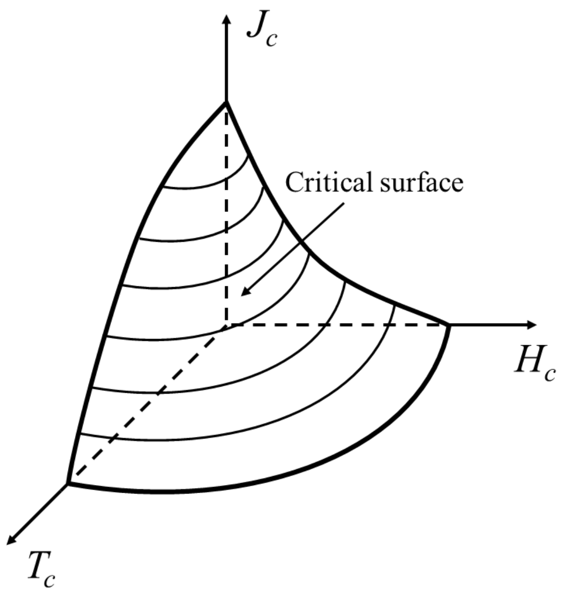

Fig. 2. Critical surface of a superconductor. T_c, H_c, and J_c represent critical transition temperature, critical magnetic field, and critical current density, respectively. Values of the applied field, transport current, and temperature corresponding to points below the critical surface are in the superconducting region, and points above this surface are in the normal region.

3. LTS

The LTS era started with the discovery of superconductivity in Hg with a T_c of 4.2 K in 1911 (Fig. 3) [8] and ended roughly with the entrance of liquid-nitrogen superconductivity. The highest T_c was 23.2 K achieved in the Nb_3Ge thin film in 1974 [1]. The T_c of a compound was found to vary qualitatively with its valence electrons per atom (Fig. 4a), known as the Matthias rule [9], which was later shown to be related to the variation of the electron density of states, in general accord with the BCS theory. Those compounds with the highest T_c, such as V_3Si, Nb_3Sn, Nb_3Ge, *etc*., display the A15 structure with three orthogonal linear chains of transition-metal elements (Fig. 4b). Later, extensive studies showed the importance of structural integrity and the significant role of transition-metal elements to high T_c, as well as raising concerns about structural and electronic instabilities that might limit the achievement of higher T_c. However, further investigation showed that such concerns might have been overstated [10].

The highest T_c achieved during this period was lower than the theoretical limit of ~ 30 K set by BCS. Although there was a great effort to search for new materials and novel mechanisms for higher T_c [11], the field indeed faced a serious crisis in confidence.

Almost all superconducting materials found during the LTS era were intermetallic. Due to the heroic efforts of Larbalestier and colleagues, practical and long superconducting wires have been fabricated from such intermetallics, such as NbTi, to form the backbone of today's superconducting technology [12].

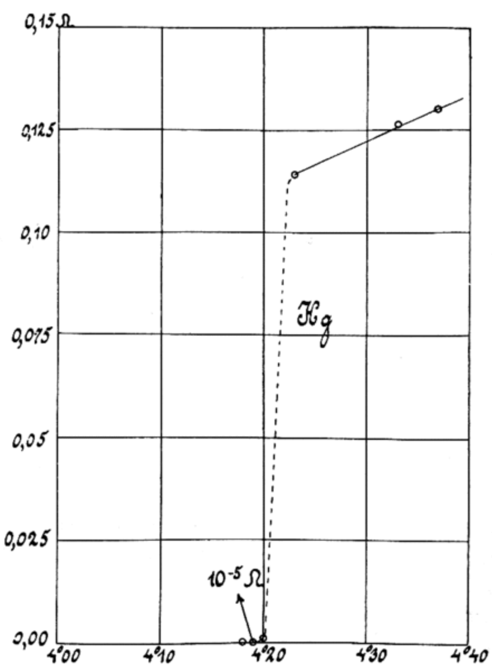

Fig. 3. First measurements on superconductivity: resistivity of a capillary of mercury as a function of temperature. [Onnes H K 1911 *Leiden comm.* 120b]

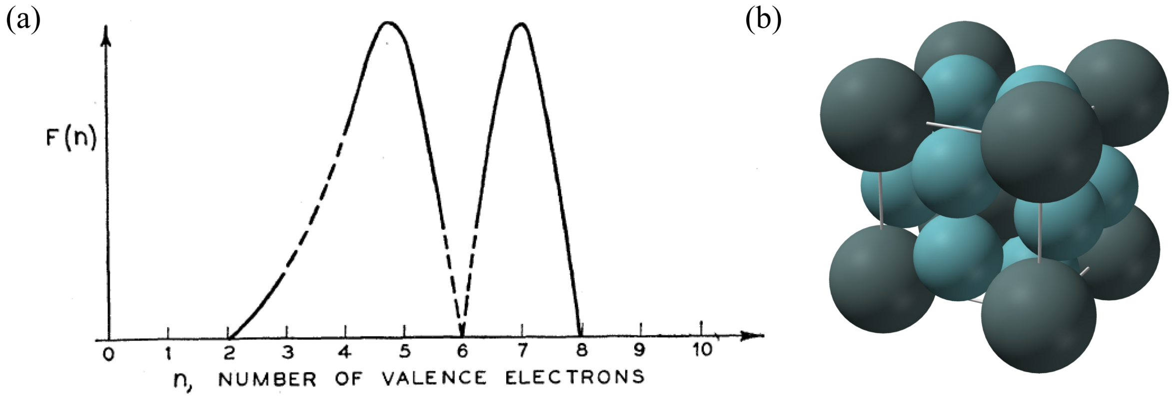

Fig. 4. (a) Matthias' empirical relation between superconductivity and the number of valence electrons per atom. F(*n*): superconductivity transition temperature as a function of the number of valence electrons per atom (*n*). [Matthias B T 1955 *Phys. Rev.* **97** 74] (b) Unit cell of A15 phase of Nb_3Sn. Image source: https://en.wikipedia.org/wiki/A15_phases. Pale blue spheres: niobium (Nb) atoms; grey spheres: tin (Sn) atoms.

4. HTS

The HTS era was inaugurated by the publication of the paper entitled "Possible high T_c superconductivity in the Ba-La-Cu-O system" by Bednorz and Mueller in September 1986 (Fig. 5) [13], in which they announced the achievement of superconductivity in the 30s K range in mixed-phase samples of $Ba_xLa_{5-x}Cu_5O_{5(3-y)}$, as evidenced by their resistance results. The paper was initially met with skepticism and did not receive the attention it deserved, perhaps due, in part, to its modest title and to the time of publication, around which many false alarms were reported. Only the four die-hard groups in Tokyo, Houston, Bell Labs, and Beijing, each with a long history in the search for higher T_c in oxides, paid attention to the report. They reproduced the resistive data and identified $(La,Ba)_2CuO_4$ (214) to be the phase responsible for the superconductivity, announcing their findings on December 4, 1986, at the Fall MRS Meeting. The genie was out of the bottle. At the same time, the Houston group demonstrated the importance of pressure in raising the T_c of LBCO, first to 40.2 K and then to 52.5 K at an unprecedentedly high $\Delta T_c/\Delta P$ rate, breaking the theoretical limit. During this time, we in Houston detected sporadic signs of superconductivity, both resistive and magnetic, up to 90 K in the mixed-phase LBCO samples (Fig. 6) but not in the pure 214 samples. The observations suggested to us that superconductivity must exist; the challenge at the time was how to identify and stabilize the new superconducting phase. The unusually large pressure effect on T_c suggested that reducing the interatomic distance might help overcome the challenge. We therefore decided to replace the La with a smaller Y based on the concept of chemical pressure. The superconducting phase with a T_c of 93 K was finally stabilized and reported on March 2, 1987, in the paper entitled "Superconductivity at 93 K in a new mixed-phase Y-Ba-Cu-O compound system at ambient pressure" by Wu *et al.*/Chu *et al.* (Fig. 7) [2]. March 2, 1987, was called by many "a Super Day of Physics" not just for the discovery of the 90 K superconductor, but also the announcement of the detection of a supernova, not to mention the now-defunct Superconducting Super Collider that was about to be built in Texas. The excitement of the discovery and the burden of certainty were both extremely high. One of us (Chu) still vividly remembers asking his students whenever he passed their offices, "think hard to see whether you can come up with any reason, other than superconductivity, to account for our observations", right up to the appearance of our *PRL* paper. He realized the possible dire consequence—being banned from superconductivity research for life—that he might have to endure if it were not actually superconductivity.

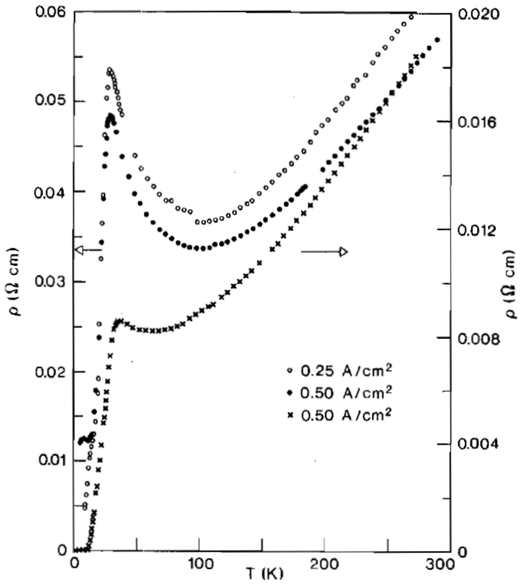

Fig. 5. ρ(T) of La-Ba-Cu-O shows a T_c of 35 K (La214) in 1986 by Bednorz and Müller. [Bednorz J G and Müller K A 1986 *Z. Phys*. B **64** 189]

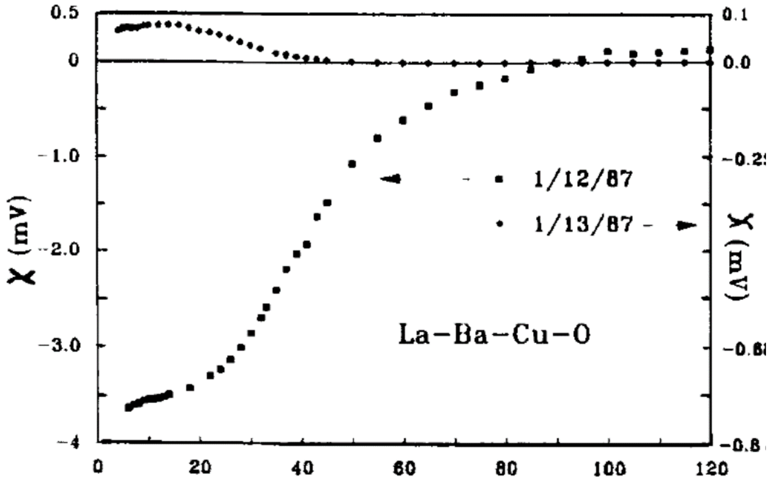

Fig. 6. χ(T) of a mixed-phase La-Ba-Cu-O shows that the superconductivity signal up to 90 K observed on January 12 disappeared on January 13 in 1987 by Chu *et al.* [Chu C W 1988 *AIP Conf. Proc.* **169** 220]

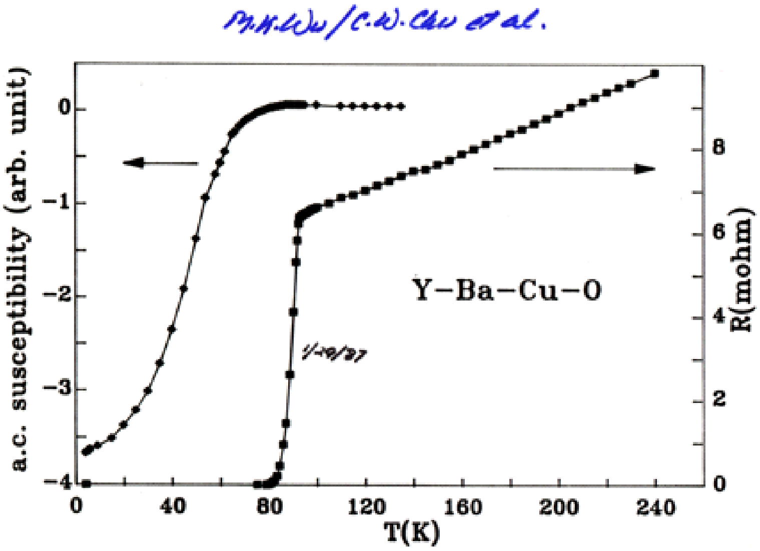

Fig. 7. ρ(T) and χ(T) of Y-Ba-Cu-O shows the T_c above 93 K (Y123) in 1987 by Wu/Chu *et al*. [Wu M K *et al*. 1987 *Phys. Rev. Lett*. **58** 908]

Working with Dave Mao's group at the Geophysical Lab in Washington DC, the phase of the 90 K superconductor was identified as the perovskite-like layered $YBa_2Cu_3O_7$ (123 or YBCO). To test the role of Y in the new superconductor, we partially replaced it with the strongly magnetic Gd but did not detect the expected suppression of T_c. The observation immediately led us to conclude that Y must be electronically isolated from the superconducting electron system and served only as the scaffold to stabilize the structure. A whole series of new 90 K RBCO (R = rare earth) superconductors was subsequently discovered [3]. These discoveries formally inaugurated the era of HTS and became the impetus for the so-called Woodstock of Physics celebration at the 1987 APS March Meeting in New York City. Because of its superior superconducting properties for devices, a large piece of melt-textured YBCO was placed inside the White House National Millennium Time Capsule, which included major discoveries and achievements by Americans across fields such as science, engineering, arts, and literature, in 2000. It is interesting to note that, inside the capsule, next to YBCO was the transistor invented by Bardeen, Brattain, and Shockley. The extensive efforts devoted to realizing the full potential of superconductivity since the beginning of the HTS era have had an immense impact on the development of physics and materials in general beyond superconductivity.

Thus far, superconductivity proven to be stable above 77 K and at ambient pressure has been found to exist only in the perovskite-like layered cuprate compounds. The highest T_cs of the four major cuprate systems at ambient pressure are 98 K for RBCO, 102 K for BSCCO, 120 K for TBCCO, and 133 K for HBCCO; for each of these systems, the highest T_c is exhibited by the member with three CuO_2 layers per formula. A cuprate compound can be considered to consist of two major parts (Fig. 8): the active part, where the supercurrent flows in the CuO_2 layers, and the passive part, which provides the charge carriers for doping. Therefore, doping occurs in the cuprate high-temperature superconductors just like in semiconductors: through modulation doping without introducing adverse scattering of the carriers in the active part.

The superconductivity of a cuprate depends critically on its quasi-two dimensionality, the number of CuO_2 layers per formula, and the carrier density per Cu-ion, n. Due to the nonuniform charge distribution over the conducting slab of CuO_2 layers and the proximity effect, the highest T_c occurs in the three-layer compounds. The T_c varies with the carrier concentration according to the empirical rule by Presland, *i.e.* $T_c \sim T_{c.max}$ [1-82.6 $(n-n_0)^2$] (Fig. 9) [14].

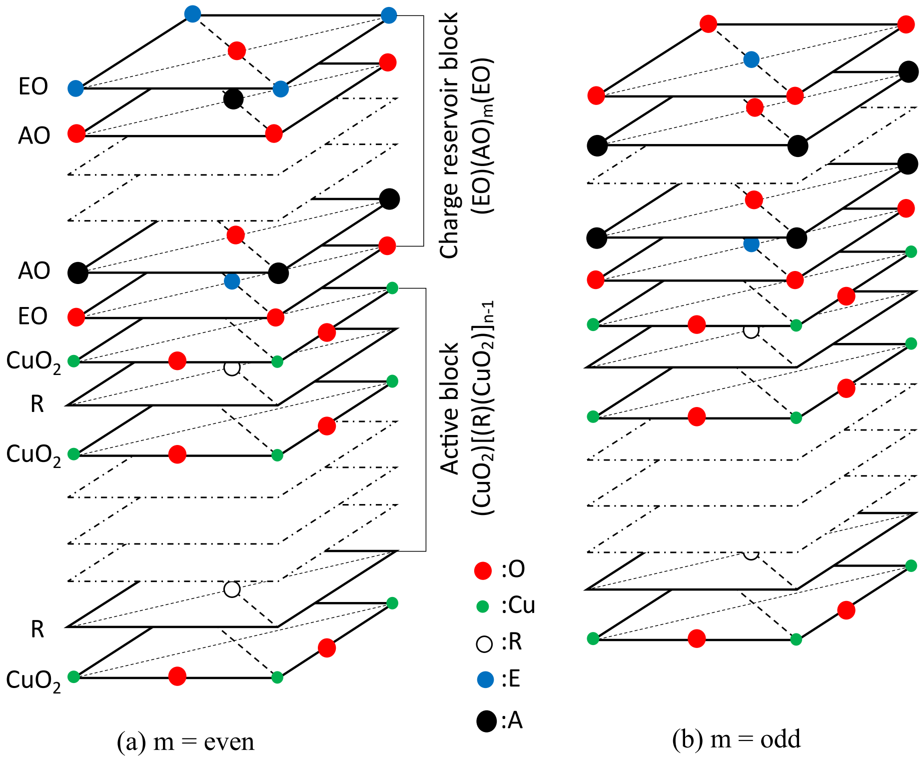

Fig. 8. Schematic layered structure of cuprate HTS. $A_mE_2R_{n-1}Cu_nO_{2n+m+2}$ [A-m(2n-1)n or –02(n-1)n when m = 0] for m = (a) even and (b) odd. [Chu C W 2003 Future High T_c Superconductors (Chapter 5 in Part G Emerging Materials ed D Shaw) *Handbook of Superconducting Materials* ed D Cardwell and D Ginley vol 2 Characterization, Applications and Cryogenics (Bristol: IOP)]

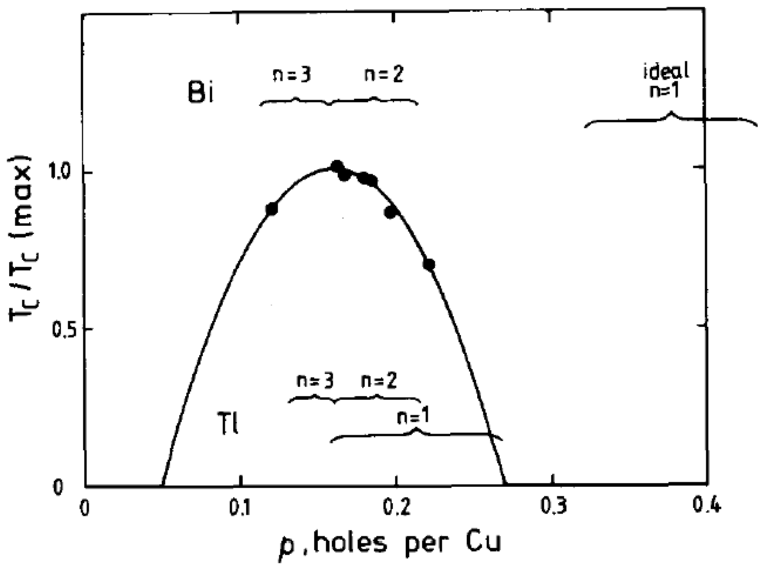

Fig. 9. Schematic phase diagram for cuprate superconductors showing the parabolic superconducting domain for $La_{2-x}Sr_xCuO_4$. [Presland M R *et al*. 1991 *Physica C: Superconductivity* **176** 95]

Major challenges for HTS device applications include the delicate structure of the cuprates and the almost perfect crystallinity needed for these devices to function. Due to the impressive ingenuity of materials scientists and engineers, many of these challenges have been overcome. For example, high-quality RBCO tapes of length have been fabricated for the ITER project by Selvamanickam *et al.* at Houston. The remaining hurdle appears to be lowering the cost.

5. RTS

RTS has long been predicted theoretically by many. Ashcroft [15] and Ginzburg [16] were among the first few who provided more serious discussions of superconductivity with a T_c above 300 K within the BCS framework, $T_c \sim \Theta_c \exp(-1/g)$, where Θ_c is the characteristic temperature of the electron excitation, and g is the attractive interaction. Ashcroft based his discussion on the large Θ_c derived from the large electron-phonon interaction *via* the exchange of phonons in light metallic hydrogen, while Ginzburg proposed an even higher Θ_c derived from the larger electron-electron interaction *via* the exchange of excitons due to the much smaller mass of electrons. There have been sporadic moments of increased activity in the search for high T_c in molecular solids and other materials. However, it was the announcement of achieving a $T_c \sim 203$ K in H_3S under pressure above 150 GPa by Eremets *et al.* in 2015 (Fig. 10) [17] that triggered the current worldwide avalanche of RTS research. The significant achievement that followed was a T_c above 260 K in LaH_{10} under ~ 190 GPa by Hemley *et al.* in 2019 (Fig. 11) [18]. These experiments were carried out in a technically extremely challenging high-pressure environment inside a diamond anvil cell. For further details, we would like to refer interested readers to a recent good review article from China for work done up to 2021 [19]. The reported evidence for RTS generally consists of a sharp resistance drop to a low value (but not to zero) at T_c, the downward shift of T_c in the presence of a magnetic field or when H is replaced by the heavier D isotope, and the diamagnetic shift of the ac magnetic susceptibility or equivalence [17, 18]. Although the above are necessary for, and consistent with, the appearance of superconductivity, they are not sufficient to prove its existence. In addition, the unusual sharpness of the transition and, in parallel, the downward shift of the transition by magnetic field at such a high temperature of nearly 300 K, imply the absence of magnetic flux creep, contradicting our common understanding of the interaction between a superconductor and a magnetic field, especially at such high temperatures. Showing the existence of the Meissner effect in these hydrides will lay all doubts concerning the existence of superconductivity in them to rest, although this would be technically very challenging. One approach that would alleviate this impasse is using the pressure quench process (PQP) that we have recently developed (Fig. 12) to stabilize the high-pressure-induced HTS phase at ambient pressure for rigorous characterization [20]. Interested readers can also find related discussion on what needs to be further studied to confirm "RTS" [21].

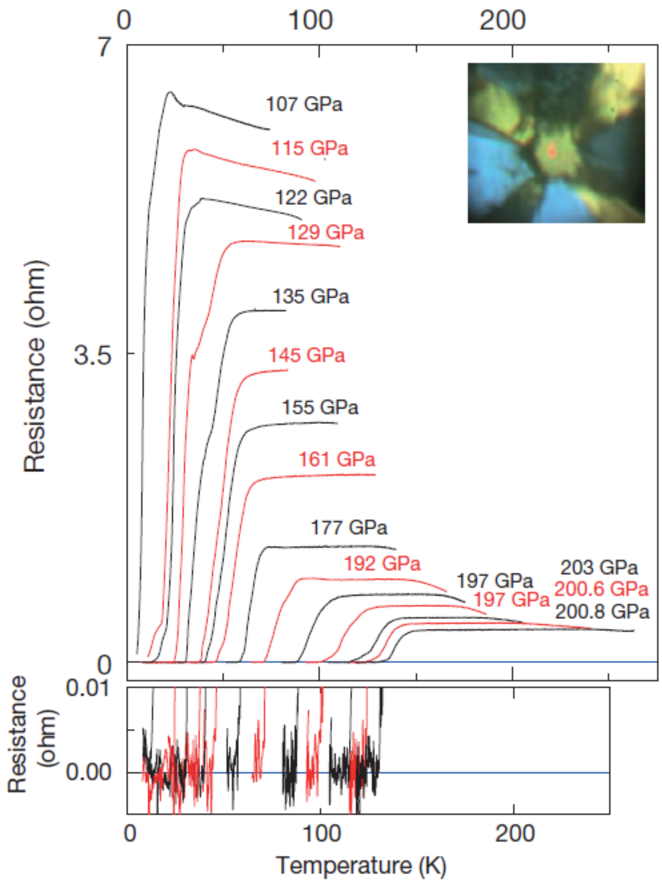

Fig. 10. Temperature dependence of the resistance of sulfur hydride measured at different pressures. [Drozdov A P *et al*. 2015 *Nature* **525** 73]

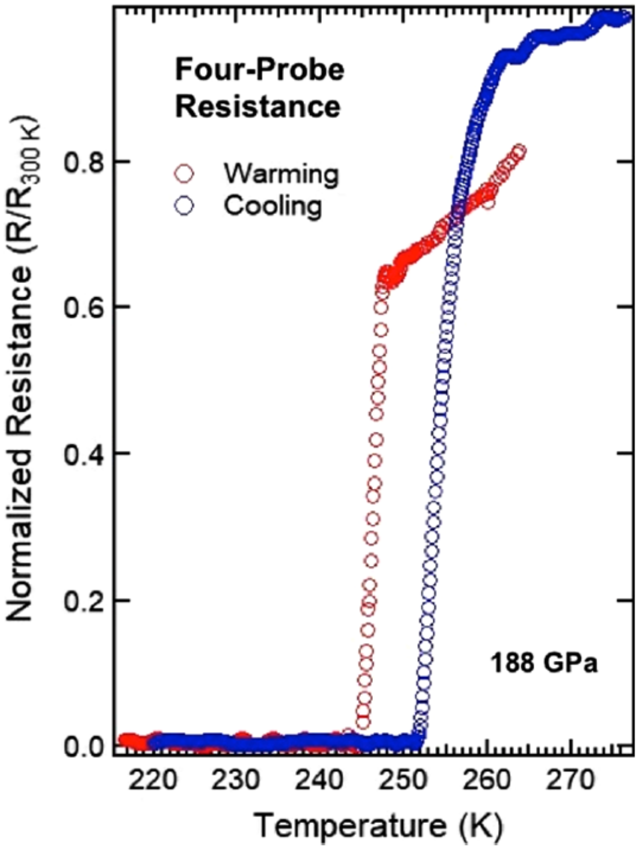

Fig. 11. Normalized electrical resistance of the $LaH_{10\pm x}$ sample. [Somayazulu M *et al*. 2019 *Phys. Rev. Lett.* **122** 027001]

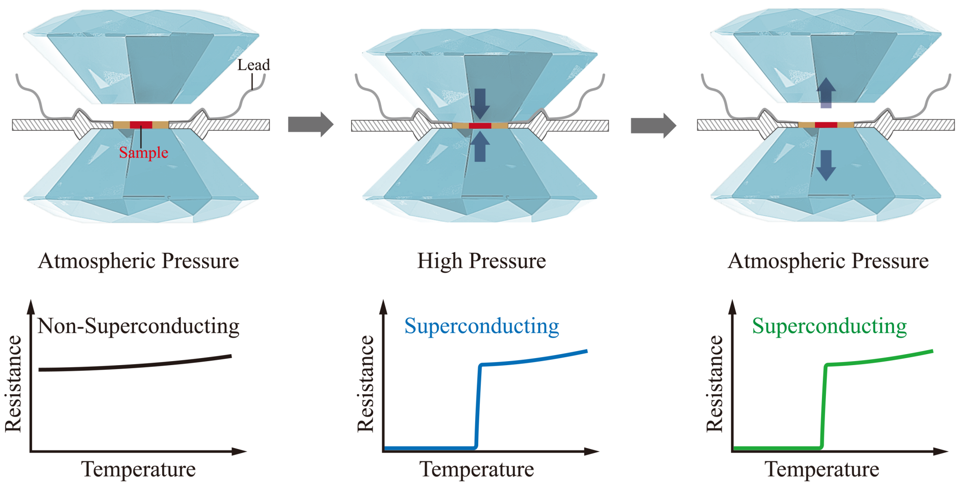

Fig. 12. Schematic diagrams for the main sequence of the pressure quench process. (PQP) [Deng L Z *et al*. 2021 *Proc. Natl. Acad. Sci. U.S.A.* **118** e2108938118]

5.1. Lu-N-H

As mentioned earlier, room-temperature superconductors reported to date have generally been hydrides formed under pressures above 100 GPa. The extreme pressure required poses serious challenges not only to its application but also to unraveling the nature of the physical state of the compound. The paper

“Evidence of near-ambient superconductivity in a N-doped lutetium hydride” [22] by Dias’s group at Rochester thus generated great excitement due to the report of high T_c of ~ 294 K induced by low pressure of 1 GPa, making it a more practical room-temperature superconductor. The published data supporting their claim are briefly summarized in Fig. 13.

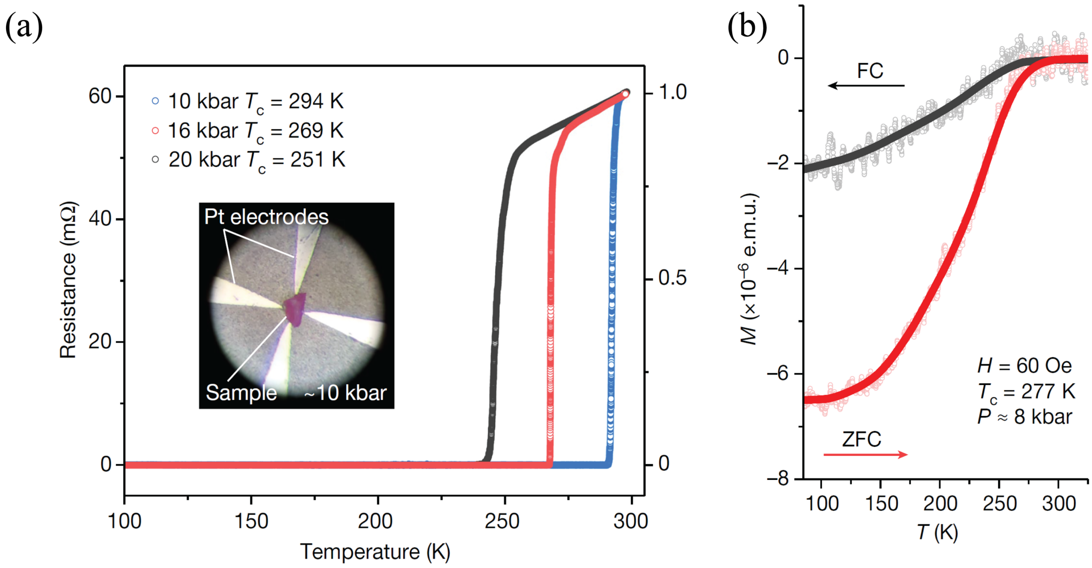

Fig. 13. Evidence for superconductivity in Lu-N-H: (a) Resistance as a function of temperature at different pressures. (b) DC magnetization as a function of temperature under 8 kbar. [Dasenbrock-Gammon N *et al*. 2023 *Nature* **615** 244]

The Rochester paper emphasized the importance of the change of color observed in the sample under pressure to the occurrence of superconductivity under pressure. After hydrogenation treatment of Lu at different temperatures up to 300 K in a flowing hydrogen gas, our measurement of the resistance variation with temperature shown in Fig. 14a indicated that the sample exhibits metallic behavior with highly correlated characteristics. The magnetic susceptibility at 0.1 T increases slowly with decreasing temperature but rises rapidly below ~ 40 K (Fig. 14b), perhaps due to magnetic impurities. We obtained LuH_2 from China and carried out a series of resistive and optical experiments, The temperature-dependent resistance at different pressures is displayed in Fig. 14c. Under pressure up to 86 GPa, the color changes reversibly with the change of resistance of the sample at 300 K, as shown in Fig. 15. The data obtained for our sample indicate that no sign of superconductivity was detected, although the color changes with pressure reported by Dias’s group were duplicated. It should be noted that the detailed sample conditions here may be very different from those for the Rochester paper.

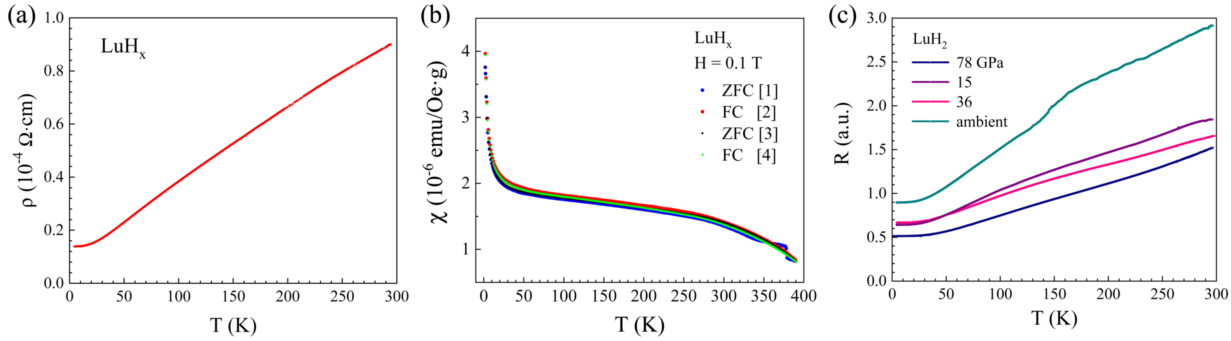

Fig. 14. (a) Resistivity of LuH_x as a function of temperature up to 300 K. (b) DC susceptibility of LuH_x as a function of temperature up to 400 K under 0.1 T. (d) Resistance of LuH_2 as a function of temperature up to 300 K under different pressures.

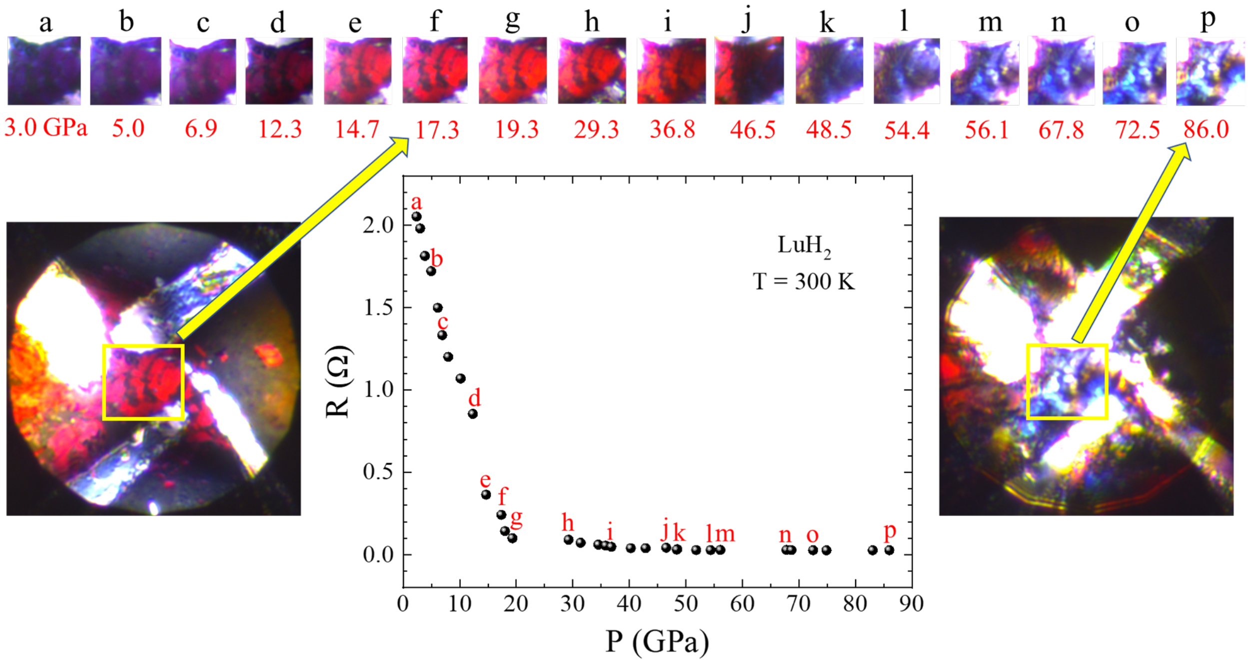

Fig. 15. Optical micrographs and resistance of the LuH_2 sample under different pressures at room temperature.

5.2. LK-99

Very recently, the report "Superconductor $Pb_{10-x}Cu_x(PO_4)_6O$ showing levitation at room temperature and atmospheric pressure and mechanism" and a related report on this LK-99 compound by Lee *et al*. [23, 24] appeared on the arXiv preprint server on July 22, 2023, like a bomb dropped on the recently agitated world of superconductivity. They showed: (i) a video of levitation of a piece of their sample over a magnet at room temperature (Fig. 16a); (ii) large diamagnetic susceptibility up to 350 K (Fig. 16b); (iii) a sharp diamagnetic transition near 323 K (Fig. 16b); (iv) a sharp drop in resistance at ~ 400 K (Fig. 16c); and (v) sharp transitions in the I-V characteristics at different temperatures up to ~ 400 K (Fig. 16d). Since the experimental conditions are available to many researchers worldwide, coupled with the significance of the discovery, we trust many must have joined the search.

All the above reported observations, (i)-(v), together appear to be consistent with the suggested detection of RTS near 400 K in LK-99 by Lee *et al*. However, after a careful examination of their results and comparison with our results [25] on samples prepared according to their recipe, many serious

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questions arise as discussed below. For example, the magnetic levitation shown in Fig. 16a and supplemented by Figs. 16b-d was initially considered by many to be the most substantial piece of supporting evidence for RTS claimed by Lee *et al*., as also highlighted in their paper title mentioned above. Unfortunately, a careful examination by us of their video in (i) suggests that the sample cannot be a superconductor, but rather a magnetized weak magnet, since the video showed the sample could be pushed around and even flipped over above the magnet. The diamagnetism of the sample shown in (ii) is also unrealistically large. The value of our sample is two orders of magnitude smaller than graphite, and not strong enough to lift the sample either. For a thin slab of graphite, complete stable levitation is possible but only when the sample is light enough and the field profile is properly shaped, as we have shown using a setup provided by T. C. Chiang (Fig. 17a). Additionally, their sample resistance never drops to zero, and thus the current-induced sharp voltage increase in the I-V measurements cannot be associated with a superconducting transition. Their X-ray diffraction data clearly show an impurity phase of Cu_2S, which displays a transition from the beta-to-gamma phase or a high-to-low resistance transition on cooling (Fig. 17b). The increasing currents needed to induce the voltage jumps in the I-V characteristics at the apparent decreasing temperatures are due to the electrical current heating of the sample, which causes the gamma-to-beta phase transition, rather than a superconducting-to-normal transition, as demonstrated by our data (Figs. 17c and 17d). In conclusion, all reported evidence for RTS in LK-99 thus far appears to be explained in terms of effects other than superconductivity.

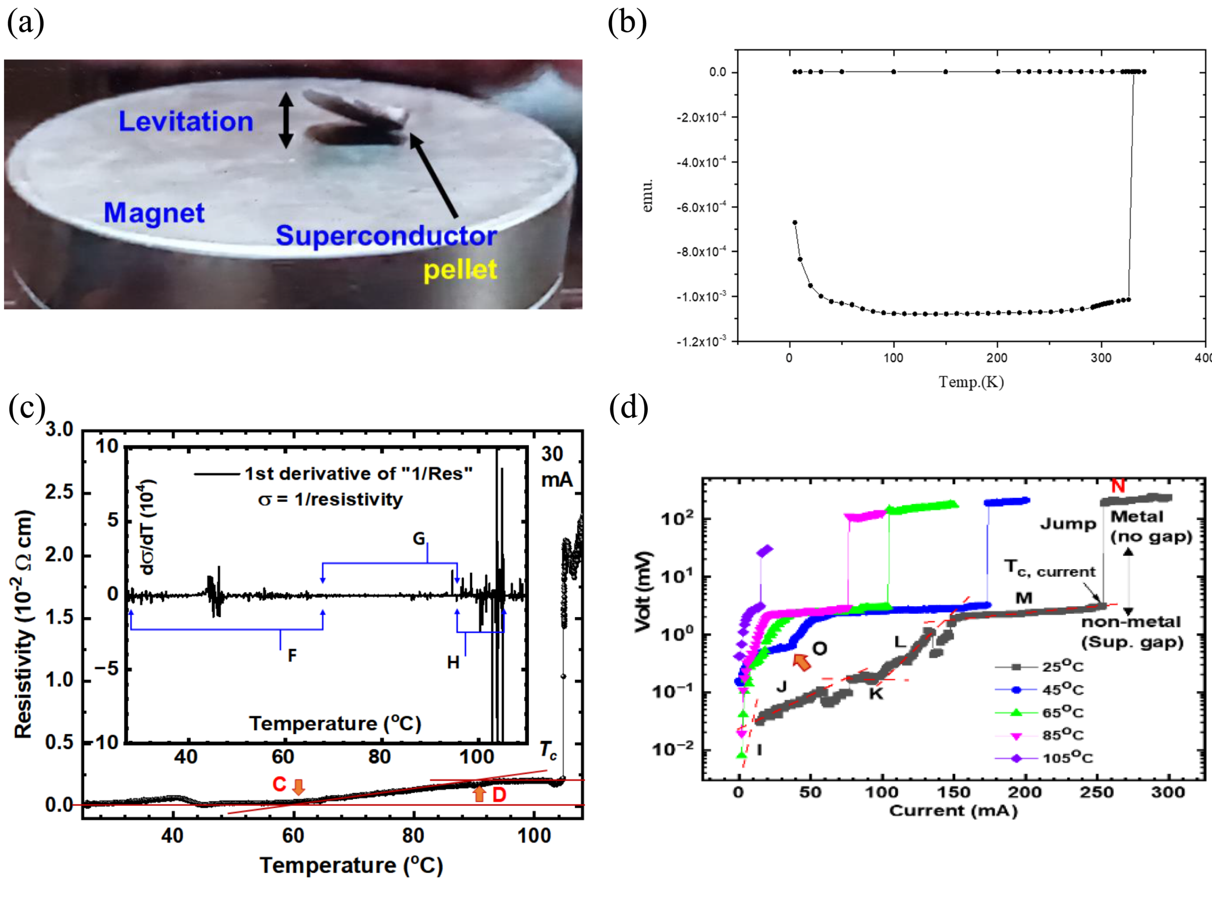

Fig. 16. (a) Photo of levitation phenomenon for a LK-99 sample. (b) Temperature-dependent diamagnetic susceptibility of a LK-99 sample. (c) Temperature-dependent resistivity of a LK-99 sample. Inset: d(1/resistivity)/dT. (d) Temperature dependence of an I-V y-axis log curve, obtained through a method measuring voltage with applying current. [Lee S *et al*. 2023 *Preprint* arXiv:2307.12037; Lee S *et al*. 2023 *J. Korean Cryst. Growth Cryst*. **33** 61]

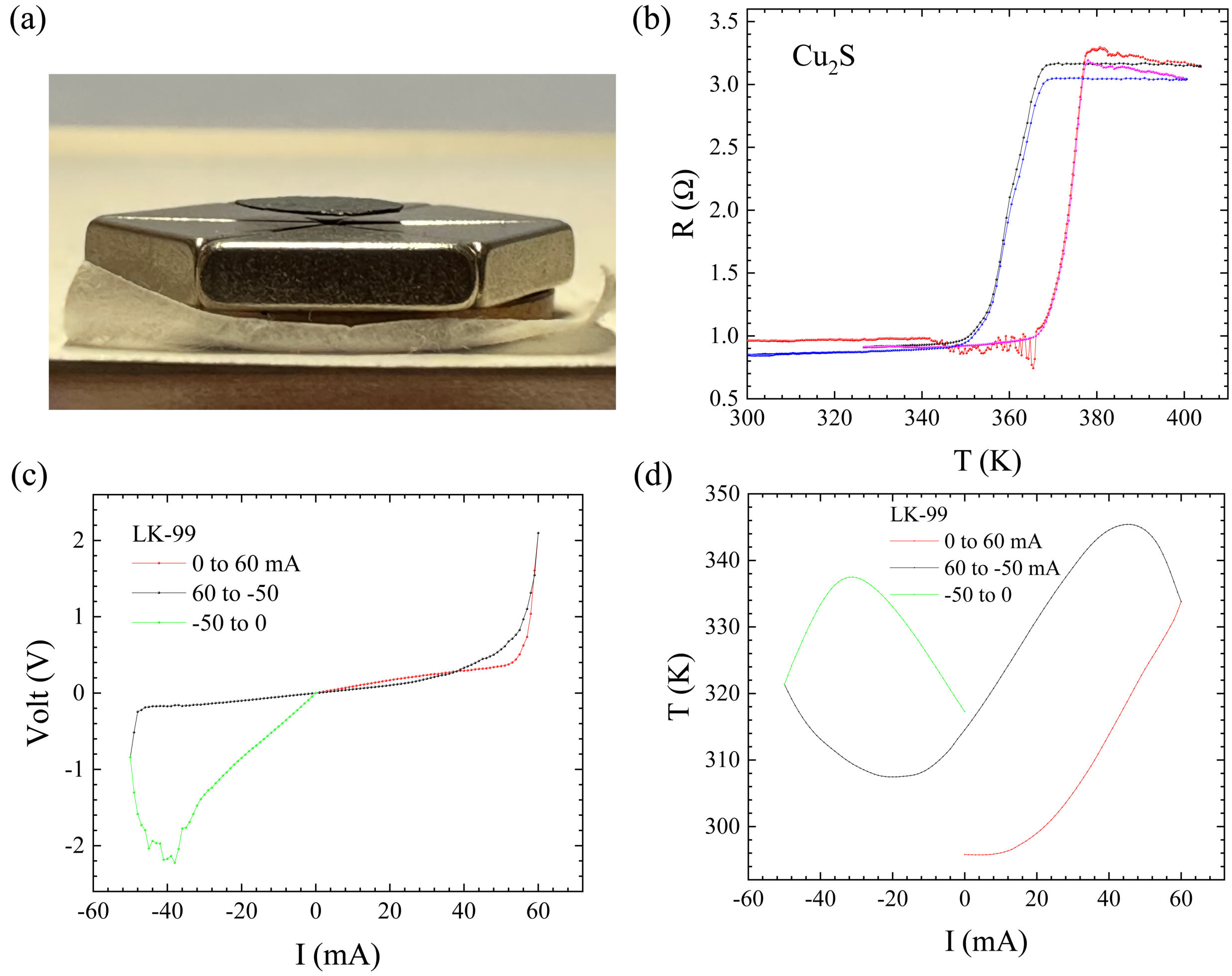

Fig. 17. (a) Photo of levitation phenomenon for a thin slab of graphite. (b) Resistance of Cu_2S as a function of temperature, demonstrating a structural phase transition at ~ 360 K. (c) Voltage *vs*. current for a LK-99 sample measured at room temperature. (d) Temperature monitored by a temperature sensor in contact with LK-99 *vs*. current. (c) and (d) demonstrate that the voltage change was due to the sample being heated by the current. [Habamahoro T *et al*. 2023 *Preprint* arXiv:2311.03558]

6. Conclusion

In our presentation, we have first briefly reviewed the long history of the search for higher T_c in the field of superconductivity, particularly for the stable intermetallic (LTS, with T_c up to 23 K) and cuprate (HTS, with T_c up to 164 K) superconductors. Great progress has been made in all areas of superconductivity research, from basic to applied, over the last century. Accompanying the advancements made in LTS and HTS science and technology over the last few decades, a solid and rigorous experimental framework concerning the search, development, and even authentication of new discoveries has been established. All these can serve as valuable references in the infancy of research into RTS, the science and even very existence of which are yet to be revealed. As a token addition in this paper, we have also presented and commented on our preliminary data on Lu-N-H and LK-99, the two most discussed "RTS" compounds in the last few months, for reference. Although our results regarding superconductivity in these compounds were unfortunately negative, we believe that while old wishes may be dashed, new hopes will arise and the lure of room-temperature superconductivity will remain too strong for many of us to resist.

7. References

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