A SUPERCONDUCTIVITY BREAKTHROUGH: FEELING THE PRESSURE TO RELEASE THE PRESSURE

UN HITO SUPERCONDUCTOR: SINTIENDO LA PRESIÓN DE RELAJAR LA PRESIÓN

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The discovery of high-temperature superconductivity in cuprates during the late 1980s triggered an intense race to raise the critical temperature (T_c), driven both by scientific pressure and physical pressure applied via diamond anvil cells. While mercury-based cuprates, particularly $HgBa_2Ca_2Cu_3O_{8+\delta}$ (Hg-1223), held the ambient-pressure record of 134 K for over three decades, achieving higher T_c at ambient conditions remained elusive. This paper reviews the history of pressure-enhanced superconductivity and presents a recent breakthrough: the Pressure-Quench Protocol (PQP). By applying high pressure at room temperature, cooling the sample under pressure, releasing the pressure at cryogenic temperatures (below 20 K), and finally warming to ambient temperature, the high-pressure superconducting phase becomes “kinetically trapped”. This protocol yields an ambient-pressure T_c of approximately 150 K in Hg-1223—the highest ever recorded at ambient pressure. Structural retention is confirmed by synchrotron Xray diffraction, and the method opens new pathways for discovering and stabilizing high-temperature superconducting phases without requiring continuous external pressure.

El descubrimiento de la superconductividad de alta temperatura en los cupratos durante finales de la década de 1980 desencadenó una carrera intensa por elevar la temperatura crítica (T_c), impulsada tanto por la presión científica como por la presión física aplicada mediante celdas de yunque de diamante. Si bien los cupratos basados en mercurio, en particular el $HgBa_2Ca_2Cu_3O_{8+\delta}$ (Hg-1223), mantuvieron el récord de 134 K a presión ambiente durante más de tres décadas, lograr T_c's más altas en condiciones ambientales seguía siendo un desafío. Este artículo revisa la historia de la superconductividad potenciada por presión y presenta un avance reciente: el protocolo de temple por presión (PQP, por sus siglas en inglés). Al aplicar alta presión a temperatura ambiente, enfriar la muestra bajo presión, liberar la presión a temperaturas criogénicas (por debajo de 20 K) y finalmente calentar hasta temperatura ambiente, la fase superconductora de alta presión queda “atrapada cinéticamente”. Este protocolo produce una T_c a presión ambiente de aproximadamente 150 K en Hg-1223—la más alta registrada hasta ahora a presión ambiente. La retención estructural se confirma mediante difracción de rayos X con sincrotrón, y el método abre nuevas vías para descubrir y estabilizar fases superconductoras de alta temperatura sin requerir presión externa continua.

Keywords: Superconductivity, Pressure-Quench Protocol (PQP), Mercury-based cuprates (Hg-1223), Metastable high-pressure phase, Ambient-pressure record critical temperature

The late 1980s and early 1990s were years of extreme pressure for the scientists working in the field of superconductivity.

On the evening of March 18, 1987, during the American Physical Society March Meeting at the Hilton Hotel in New York City, a historic session took place. The discovery of high-temperature superconductivity in the $CuBa_{0.15}La_{1.85}O_4$ “cuprate” in 1986 [1] and in its “cousin” $YBa_2Cu_3O_{7-\delta}$ early in 1987 [2] had triggered an unprecedented frenzy. The session room was full of scientists past midnight; researchers slept in the aisles, and others watched on closed-circuit television outside. This event was immediately dubbed the ”Woodstock of Physics”– making an analogy with the famous three-day music concert celebrated in 1969.

The community felt the pressure to understand and, above all, to raise the T_c –i.e., *the critical temperature* above which superconductivity disappears. Bednorz and Müller’s compound $La_{2-x}Ba_xCuO$, $x = 0.15$ had a T_c of 35 K (−238 °C) at ambient pressure, which was rapidly increased while hydrostatic pressure was applied to the material using a diamond anvil [3]. This observation suggested C. W. Chu and co-workers the creation of the material $YBa_2Cu_3O_{7-\delta}$, that exhibited a critical temperature of 93 K (−180 °C) at ambient pressure. This value of T_c was above liquid nitrogen’s boiling point (77 K), constituting a giant step in terms of “experimental affordability” and potential applications –see Fig. 1.

It can be said that during the late 1980s and early 1990s, the field of superconductivity was driven by pressure: the pressure to find new compounds with higher critical temperatures, and the physical pressure applied by diamond anvil cells. Both served to push higher. *But the holy grail has been always to increase the critical temperature at ambient pressure*. In this article we will present a very recent discovery where a record critical temperature is obtained at ambient pressure. . . by applying hydrostatic pressure!

A SUPERCONDUCTIVITY PRIMER

Superconductivity is a macroscopic quantum state characterized by three fundamental fingerprints. *Zero* *resistance* is the most intuitive: below T_c, the DC electrical resistivity drops to practically zero ohms in high-quality samples, limited only by measurement sensitivity. *The Meissner effect* – the *expulsion* of magnetic flux from the interior of a superconducting sample even when the field does not change in time – distinguishes a superconductor from a perfect conductor. *Flux quantization* is perhaps the most direct evidence of macroscopic quantum coherence: in a superconducting ring or a hollow cylinder, the magnetic flux enclosed is quantized in units of $\Phi_0 = h/2e$, where h is Planck's constant, and e is the charge of the electron. This quantization directly reveals that the charge carriers are electron pairs (called Cooper pairs) instead of single electrons, as in common metals [4]. It also demonstrated that superconductivity is a macroscopic quantum state, along with other phenomena, like Josephson tunneling [5].

Cooper pairs are a crucial ingredient of the microscopic theory of conventional superconductivity, known as the Bardeen-Cooper-Schrieffer (BCS) model [6]. Intuitively, BCS works like this: as an electron moves through the lattice of positive ions, it attracts the ions slightly, creating a local positive charge distortion. A second electron is then attracted to this distortion, forming a bound pair – a Cooper pair– even though both electrons are negatively charged and repel each other in the free space. The lattice vibrations (phonons) act as the glue. This pairing is fragile: the pair size (coherence length) can range from a few to hundreds of nanometers, much larger than the interatomic spacing. But once formed, all Cooper pairs condense into the same quantum ground state, allowing them to flow without resistance. However, Cooper pairs can be destroyed in different ways: by increasing temperature, by increasing an applied magnetic field, or by increasing an applied electric current [4].

Copper pairs can be also destroyed by applying mechanical pressure, but sometimes it can make them stronger. An external pressure applied to a superconductor reduces interatomic distances. Within the framework of the BCS mechanism (valid for conventional superconductors), this may strengthen the electron-phonon coupling by increasing the density of states at the Fermi level, thus increasing the critical temperature. For the case of unconventional superconductors like the cuprates –whose precise pairing mechanism is still unknown– pressure may optimize orbital overlap and charge transfer, implying a higher critical temperature [7].

SUPERCONDUCTING MATERIALS: A SAGA OF RECORDS

In 1911, Heike Kamerlingh Onnes accidentally found that mercury became superconducting at 4.2 K [8], for which he received the Nobel prize in Physics in 1913 [9]. For the next seven decades, progress was slow, as seen in Fig. 1. For example, Nb_3Ge reached a record critical temperature of 23 K (approximately six times that of mercury) only in 1973. The so-called "conventional superconductors" (represented with black dots in Fig. 1) are well described by the BCS theory and required liquid helium cooling –a rather expensive refrigerant, if compared with liquid nitrogen. The landscape changed dramatically in 1986 when Bednorz and Müller discovered superconductivity at 35 K in $CuBa_{0.15}La_{1.85}O_4$, as mentioned above: not only did it establish a record high critical temperature at ambient pressure; it also broke the tradition of metallic or metallic-alloy superconductors. Moreover, C. W. Chu's group increased the critical temperature in the $CuBa_{0.15}La_{1.85}O_4$ system to 40 K by applying pressure: it is believed that this result made him suspect that the perovskite structure with appropriate atomic substitutions had a serious potential for improvement of superconducting properties beyond those of $CuBa_{0.15}La_{1.85}O_4$. He was proved right by the obtainment of the celebrated $YBa_2Cu_3O_{7-\delta}$ material, with an absolute record critical temperature of 92 K at ambient pressure, in 1987. Besides being an enormous improvement in the critical temperature, crossing the barrier of 77 K meant that the superconducting state could be attained using the relatively inexpensive liquid nitrogen as refrigerant. These achievements kicked off the frantic race for higher critical temperatures starting in 1987.

Other cuprate superconductors with even higher critical temperatures were soon found: all of them have one structural feature in common: the existence of copper-oxygen layers of atoms, along which superconductivity is strong. Fig. 1 shows the members with higher critical temperature of all the main cuprate families by means of red dots (there also exist non-conventional, non-cuprate superconductors with relatively small critical temperatures that are not included in the graph). Now, we will concentrate on a particular cuprate family: the mercury-based materials.

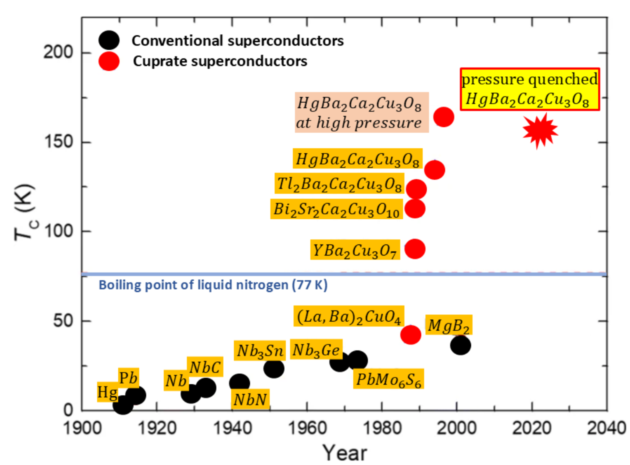

Figure 1. Evolution of the critical temperature in conventional and cuprate superconductors. The chemical formulas shown in the graph for the cuprates are idealized: for example, the actual formula for $HgBa_2Ca_2Cu_3O_8$ is $HgBa_2Ca_2Cu_3O_{8+\delta}$, where $\delta < 1$ represents an imperfection in the oxygen stoichiometry, of paramount importance for obtaining superconductivity.

The path to the mercury-based cuprates began in 1993 at two laboratories nearly simultaneously. In Switzerland, a team at the École Polytechnique Fédérale de Lausanne (EPFL) led by S.N. Putilin and E.V. Antipov were exploring the Hg–Ba–Ca–Cu–O system, a hazardous family many avoided due to the toxicity of mercury. Using a high-pressure synthesis technique to stabilize the structure, they successfully created the first member of a new cuprate family: $HgBa_2Cu_3O_{4+\delta}$. This single-layer compound exhibited a critical temperature of 94 K at normal atmospheric pressure [10]. Almost immediately, a second team led by A. Schilling at ETH Zürich independently confirmed the discovery and reported an even more dramatic result: *a sample of formula* $HgBa_2Cu_3O_{4+\delta}$ *showed superconductivity approximately at* 134 *K, an ambient-pressure record that has survived for more than three decades* [11].

Sometime later, the race to unlock that potential was taken up by C.W. Chu's group at the University of Houston. Knowing that pressure was a powerful enhancing tool, from decades of experience, they synthesized the mercury compound $HgBa_2Ca_2Cu_3O_8$, placed a tiny sample inside a diamond anvil cell and squeezed it to pressures as high as 45 GPa. The effect was remarkable: the atomic layers were forced closer together, optimizing the electronic interactions, and the critical temperature soared to 164 K under pressure, shattering the previous record. This is the highest confirmed critical temperature ever achieved in a cuprate superconductor [12].

The value of the record critical temperature (under pressure) increased in 2015, when the compound H_2S (hydrogen sulfide), under a pressure of 155 GPa, showed a critical temperature of 203 K [13]. This is considered a metallic superconductor, but it is not included in Fig. 1. Finally a compound with a clathrate structure, formed by a network of ten hydrogen atoms with a lanthanum atom at its center (LaH_{10}), subjected to a pressure of 170 GPa, showed superconductivity at temperatures below 250 K (−23 °C, almost ambient temperature!) [14].

This search for room-temperature superconducting materials has not been free of controversy. A team led by Ranga Dias at the University of Rochester published an article in 2020 claiming that a carbon–sulfur hydride-based superconductor, when subjected to a pressure of 267 GPa, exhibited superconductivity at 15 °C. However, after numerous doubts were raised by several researchers in the field, the article was retracted by *Nature* in 2022, against the authors' wishes, who stood by their claim of having made a valid discovery. In 2023, the same team reported (again to *Nature*) an even more striking result: superconductivity at 21 °C and a pressure of 1 GPa in the compound lutetium–nitrogen hydride (N-Lu-H). That same year, the study was retracted by the journal, in an even more concerned turn, as some of the co-authors requested the retraction.

Beyond this case, in 2023 a South Korean team presented online a copper-lead-phosphorus-oxygen alloy (known as LK-99) that was supposedly a superconductor at ambient temperature and pressure, generating excitement and debate on social media. However, the scientific community's failure to replicate the result completely refuted the finding, and it is now known that the anomalies observed in LK-99 were due to the presence of copper sulfide impurities in the sample. We personally owe a lot to the mercury-based cuprates: due to their high critical temperature, our research group was able to experimentally study superconducting phenomena within a relatively wide temperature range, although liquid nitrogen was the only refrigerant available to us. We were able to systematically measure the hysteresis and relaxation effects typical of ceramic superconductors, demonstrating that polycrystals of nominal composition $HgBa_2Cu_3O_{8+\delta}$ behave as a collection of strongly superconducting grains connected by "weak links" that experience Josephson tunneling [15–18].

QUENCHING THE PRESSURE

While most of the scientists in the superconducting community feel today much less pressure than commonly experienced during the late 1980's and early 1990's, some of the protagonists of that era have kept the feeling alive. That is arguably the case with C. W. Chu.

In a very recent article, his group successfully solved a longstanding problem: how to preserve a highpressure superconducting phase after the applied pressure is released. They achieved it through a method called Pressure-Quench Protocol (PQP) that was applied to polycrystalline samples of $HgBa_2Ca_2Cu_3O_{8+\delta}$ (Hg-1223) [19], It consists of four basic steps, during which the temperature dependence of the sample's resistivity was measured. Here we explain the PQP process:

Step 1 – High-pressure conditioning at room temperature. A Hg-1223 polycrystalline sample is loaded into a diamond anvil chamber with a pressure medium (neon or Daphne oil). The pressure is increased to a value known to optimize the of this cuprate, [12].

Step 2 – Cooling under pressure. While maintaining a high pressure, the anvil chamber is cooled down, and the resistance of the sample is verified in real time using the four-probe method. The black curve in Fig. 2 illustrated the evolution of the resistance for a particular experiment, shown for temperatures above 100 K: a wide transition from the normal state at room temperature (at the right of the curve) to the superconducting state (to the left of the curve) can be seen. The sample was actually taken all the way down to a temperature of 4.2 K, not shown in the graph.

Step 3 – Pressure release at low temperature. This is the critical innovation: instead of releasing pressure at room temperature (which would cause the lattice to relax to the ambientpressure structure), the anvil chamber is slowly decompressed back to 1 atmosphere while the sample remains below 20 K. This çold decompression"kinetically "traps" the highpressure crystal structure because atomic diffusion (the "reordering of atoms") is frozen. For the sample illustrated in Fig. 2, the decompression took place at 4.2 K (not seen in the graph).

Step 4 – Warming to ambient temperature. After full pressure release, the anvil chamber is warmed to 300 K. The retained high-pressure phase remains metastable. The red curve in Fig. 2 shows the superconducting-to-normal transition as temperature was increased after releasing the pressure at 4.2 K. The resulting critical temperature indicated by a vertical arrow in the graph (that should be more properly called onset critical temperature [18]) is approximately 150 K at 1 atm– the highest ambientpressure ever recorded, breaking the previous record of 134 K [11]. Notice that the figure does not show a superconducting resistivity very near zero ohms, but the data is shown for relatively high temperatures; superconductivity was assessed also using other techniques –especially AC susceptibility.

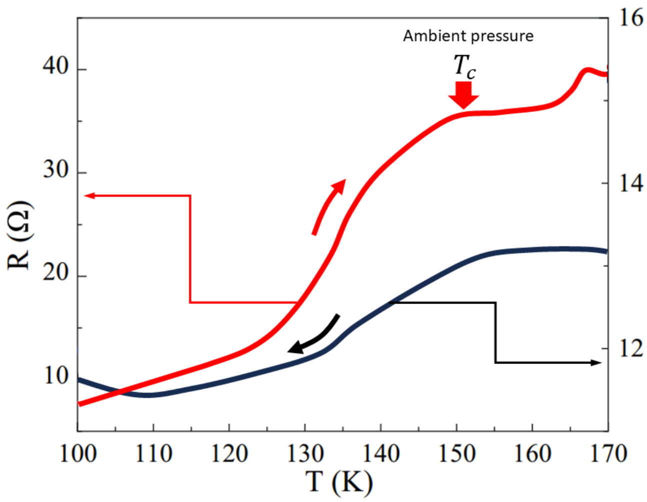

Figure 2. Tracking the sample's resistance during the Pressure-Quench Protocol in real time. The resistivity vs. temperature curves shown correspond to an experiment performed for one $HgBa_2Ca_2Cu_3O_8$ is $HgBa_2Ca_2Cu_3O_{8+\delta}$ polycrystalline sample. The horizontal arrows indicate the vertical axis corresponding to each curve (Sketch inspired by Fig.2E of [19]).

Of course, establishing a critical temperature record is a very serious matter, so the authors performed several further studies to crosscheck the results. Structural retention was confirmed by synchrotron Xray diffraction. Rietveld refinement showed that copperoxygen bond lengths and apical oxygen positions remain locked in the compressed geometry. Phonon calculations indicated that this geometry increases the electronic density of states at the Fermi level, thereby enhancing the pairing interaction. In addition to these "intrinsic" reasons to explain the improvement in superconducting properties, "extrinsic" factors seem to crucially contribute: the authors speculate that sample imperfections may help "trapping" the strongly superconducting phase after releasing the pressure [19]. The complexity of sample imperfections at many levels and their effects on the superconducting properties have indeed haunted us for decades [15–18], so it is not surprising for us that the precise role of the defects in the process is far from clear in [19]. In any case, the PQP constitutes a breakthrough in the field of superconducting materials.

In an informal encounter with C. W. Chu in 2000, one of us (E.A.) asked him "Professor, where do you think the discovery of room-temperature superconductivity will occur?". He thought for a couple of seconds, and said "Probably in an unknown, little laboratory somewhere in the world". After learning about his own group's Pressure-Quenched Protocol, one may suspect that, after all, Prof. Chu was not exactly right.

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