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TOPICAL REVIEW — Synergetic Extreme Condition User Facility: Breakthroughs and opportunities for the research of physical science

Advanced high-pressure transport measurement system integrated with low temperature and magnetic field

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We briefly introduce a new high-pressure transport measurement system integrated with low temperature and magnetic field that is being established as one of the user experimental stations of the Synergetic Extreme Condition User Facilities in the Huairou District of Beijing, China. To demonstrate the capabilities of the system for condensed matter research, the emergence of some pressure-induced phenomena and physics related to superconductivity found previously is also introduced, and then a perspective for such an advanced high-pressure system is presented.

Keywords: high pressure, low temperature, magnetic field, superconductivity

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1. Introduction

In the research frontiers of condensed matter physics, high pressure is widely adopted as an independent control parameter that can effectively tune crystal and electronic structures and consequently create a variety of new phenomena, which dramatically promote the finding of new materials, through the change of the atomic distance of matter. Consequently, high-pressure research is considered to be a new dimension in condensed matter physics.[1] Remarkably, a great deal of success has been achieved by the application of pressure in the studies on condensed matter, such as converting matter from an insulating or a semiconducting state to a metallic state,[2–7] and from a magnetic state to a nonmagnetic one.[5–9] Pressure-induced superconductivity and enhancement of superconducting transition temperatures (T_C), as well as related phenomena, have been frequently found over a broad range of materials.[7–18] Noticeable are pressure-induced superconducting transitions in compressed elements of sulfur, oxygen, iron, lithium, and black phosphorous[11,19–22] and in complicated compounds of AFe$_2$As$_2$ (where A = Ca, Sr, Ba, or Eu).[23–26] Compelling examples of pressure-enhanced T_C in known superconductors have been observed in the families of copper oxide (cuprate) and iron-based superconductors; for instance, the T_C of mercury-bearing cuprates with a value of 134 K at ambient pressure is pushed up to 164 K at $\sim$ 30 GPa, which holds the highest T_C among all copper oxide superconductors.[27–30] The T_C of LaO$_{1-x}$F$_x$FeAs with a value of 26 K at ambient pressure has been also enhanced to 43 K at $\sim$ 4 GPa.[18,31] The simplest compound superconductor, FeSe, with the T_C value of $\sim$ 8 K at ambient pressure,[32] has been found to jump to 36.7 K at 8.9 GPa.[17]

Meanwhile, pressure-induced phenomena and physics have emerged in the field of superconductivity research. Exciting research progress has been made, such as the observation of superconductivity with a transition temperature of $>$ 200 K in H_3S,[15,33–35] the reemergence of superconductivity in heavy-fermion and alkaline iron selenide superconductors,[8,36] the appearance of a bi-critical point in compressed Ca$_{0.73}$(La$_{0.27}$)FeAs$_2$,[37] and superconductivity in Weyl semimetals.[38–40]

In this article, we first introduce the capabilities of our high-pressure transport measurement system and then present some experimental results achieved by high-pressure research for demonstrating possible applications of the system. Because of the limitation of the article length, it is not possible for us to cover all the important achievements in high-pressure studies on condensed matter in this review; therefore, we focus on some selected works from our research interests.

2. General introduction to the system

The system being established is one of the important equipment of the Synergetic Extreme Condition User Facilities (SECUF). The system integrates three extreme conditions, including high pressure (HP), low temperature (LT), and magnetic field (MF), as described in Fig. 1. The aim of the system established is to provide experimental scientists with a platform to investigate transport properties of condensed matter under extreme conditions.

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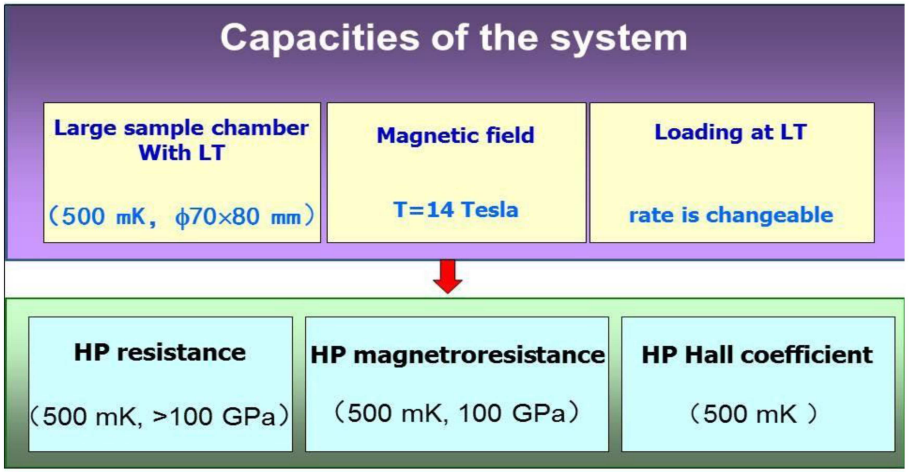


Fig. 1. (color online) Capabilities of the system integrated with extreme conditions of high pressure (HD), low temperature (LT), and magnetic field (MF), as well as the target parameters for the transport measurements of resistance, magnetoresistance, and the Hall coefficient.

It is known that a static pressure up to 100 GPa or even higher can only be realized by using a diamond anvil cell (DAC), which is the dominant device for achieving very high pressures in laboratory. Therefore, a DAC is adopted to generate high pressure for the system. In a DAC, the two anvils are placed oppositely, as shown in Fig. 2(a). Because the pressure in high-pressure research means the intensity of pressure, i.e., the force divided by area, the maximum pressure achieved in a DAC depends on the diameter of the culet (the flat area, the most delicate region of the diamond anvil, as shown in Fig. 2(b)). A high pressure can be obtained by reducing the culet to a smaller size. Up till now, it has remained a challenge to prepare the standard four electrodes for high-pressure resistance measurements and the van der Pauw type electrodes for high-pressure Hall measurements on such a tiny culet (Figs. 2(b) and 2(c)).

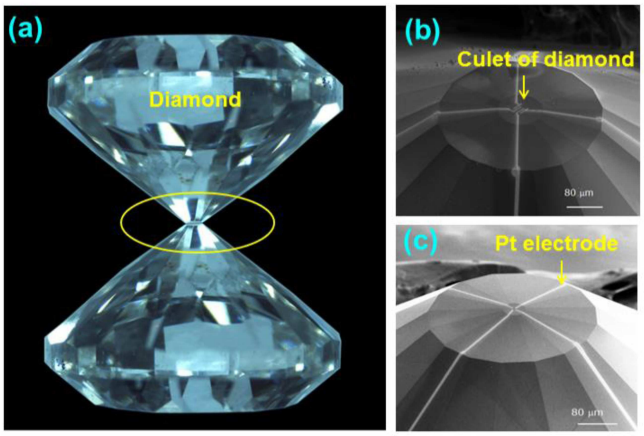


Fig. 2. (color online) (a) Photograph of diamond anvils in a diamond anvil cell. (b) and (c) Scanning electron microcope images of the culet of diamond anvils and the arrangements of the standard four electrodes for resistance measurements and van der Pauw type electrodes for Hall measurements, respectively.

The capacities of the system are shown in Fig. 1. The available pressure for users to conduct electrical resistance and magnetoresistance measurements is $\sim$ 100 GPa (1 GPa = 10^9 Pa). The lowest temperature that can be reached is 500 mK, and a magnetic field up to 14 T can be provided. Although similar equipment with a magnetic field $\sim$ 9 T are available at the Carnegie Institute in USA, the Max Planck Institute in Germany, and Osaka University in Japan, the general parameters of extreme conditions available from this system are among the best in the world. It is believed that this system will provide users with the most advanced high-pressure experimental platform for frontier studies in research fields of condensed matter physics, chemistry, materials science, geophysics, astrophysics, etc.

3. Emergence of phenomena and physics in pressurized condensed matter

Finding exotic phenomena and physics in condensed matter and understanding the underlying physics have long been research frontiers of condensed matter physics, among which superconductivity is a leading research field. The common variables, pressure, temperature, and magnetic field, have been broadly adopted as effective methods to tune crystal and electronic structures, making great contributions to the field of condensed matter physics. The emergence of phenomena in compressed matter leads to deeper insight into the underlying physics. Here, we will introduce some typical results achieved in high pressure studies on conventional and unconventional superconductors.

3.1. Cuprate high-temperature superconductors

The discovery of cuprate high-temperature superconductors impacted science and technology considerably and the excitement in this field has been continuing for the past 30 years. To date, more than two hundreds of cuprate superconductors

have been found,[41] among which $HgBa_2Ca_2Cu_3O_{8+x}$ exhibits the highest T_C up to 134 K at ambient pressure.[28] Many efforts have been made to push T_C higher and the results have been documented in a long list. Pressure was found to play a key role in enhancing T_C in this family. Most impressive is the application of pressure on $HgBa_2Ca_2Cu_3O_{8+x}$, enhancing its T_C to 164 K at $\sim$ 30 GPa,[29,30] as shown in Fig. 3, which remains the record for bulk superconducting materials. Another example that shows pressure-induced enhancement of T_C is found in optimally doped trilayer $Bi_2Sr_2Ca_2Cu_3O_{10+x}$ (Bi-2223), in which its T_C is increased from 108 K at ambient pressure up to 136 K at 36 GPa.[14] It is considered that the natural appearance of an inhomogeneous charge distribution among the inner and outer CuO_2 planes makes the trilayered Bi-2223 to obtain higher T_C under pressure.

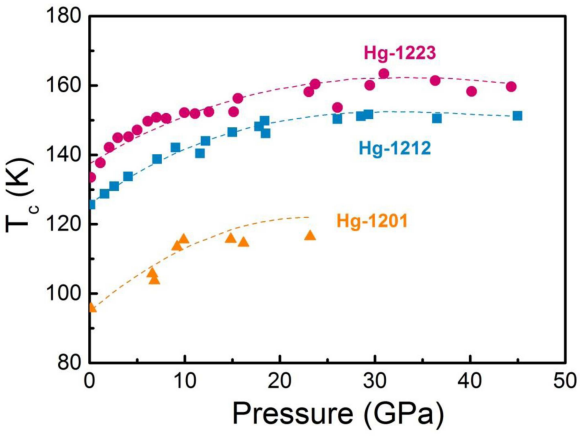


Fig. 3. (color online) Pressure–temperature phase diagram of mercury-bearing cuprate superconductors, showing a T_C up to 164 K. The data are taken from Ref. [29].

3.2. FeAs-based superconductors

The discovery of $LaFeAsO_{1-x}F_x$ superconductors with a T_C of 26 K in 2008[31] brought new excitement to the field of superconductivity and sparked intense research activity in the past 10 years. Many research groups have made great advances in understanding this new kind of high-T_C superconductor with the apparent hope of gaining insight into the underlying superconducting mechanism.[42–48] There are more than 15,000 papers that have been published so far.[48]

Pressure as a 'clean way' plays a prominent role in the search for Fe-based superconductors with higher superconducting transition temperature. High-pressure resistivity measurements of $LaFeAsO_{1-x}F_x$ in a DAC revealed that its T_C can be increased to 43 K at $\sim$ 4 GPa,[18] as shown in Fig. 4. The obtained high pressure result aids the discoveries of REOF-FeAs (where RE = Ce, Nd, Pr, Sm, Gd, or Tb) superconductors with $T_C > 50$ K at ambient pressure through replacing La with smaller RE ions.[49–55]

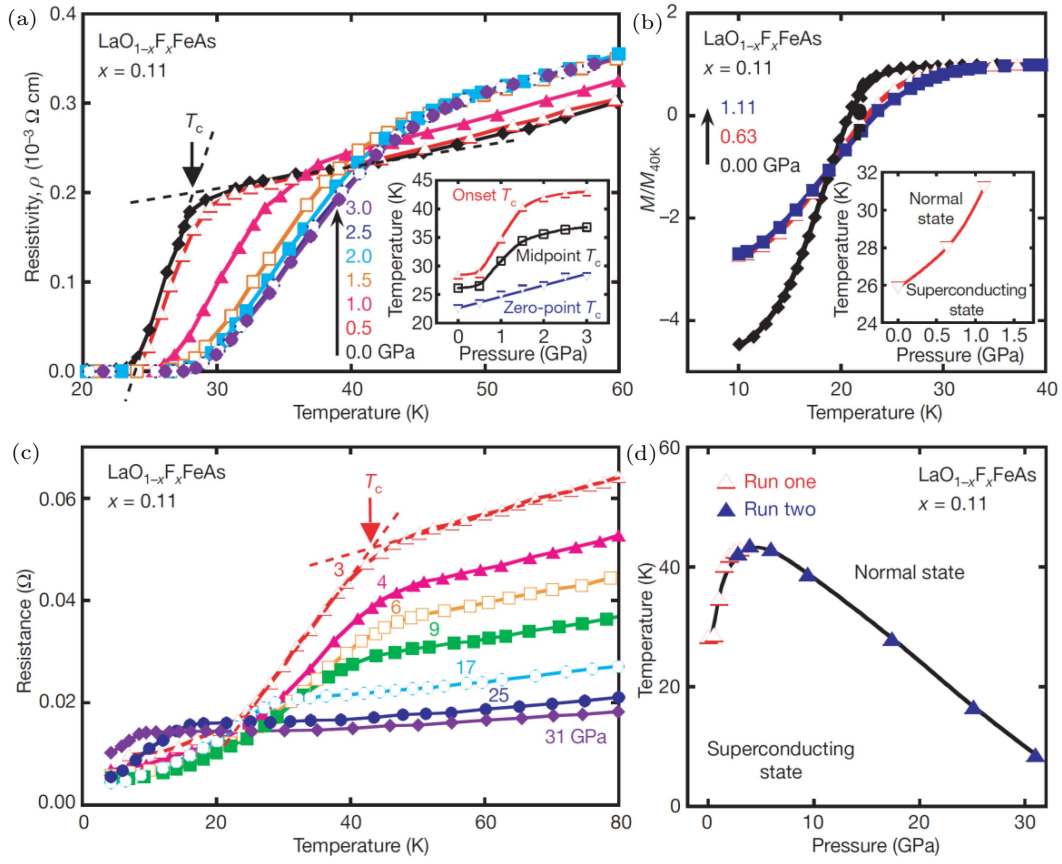


Fig. 4. (color online) Superconducting properties in compressed $LaO_{0.89}F_{0.11}FeAs$. (a) Results obtained from measurements in a piston–cylinder pressure device (b) Magnetic dependence of T_C under pressure. (c) Results obtained from measurements in a DAC, showing pressure-induced enhancement of T_C in the superconductor. (d) Pressure–temperature phase diagram. Reproduced with permission from Ref. [18].

Because parent compounds were found in one of the subsystems of FeAs-based superconductors, whose chemical formula is $A'Fe_2As_2$ (where A' = Ca, Sr, or Ba) and $EuFe_2As_2$,[56–59] this provides an opportunity to identify the effect of pressure in developing superconductivity and the correlation between the magnetic ordered state and the superconducting state in this new type of superconductor. High-pressure studies indicated that the superconductivity of these compounds is very sensitive to the pressure environments.[23–26,60–64] The physics behind this is considered to be associated with pressure-induced lattice distortion.[65–67]

More recently, high-pressure studies on the antiferromagnetic compound $Ca_{0.73}La_{0.27}FeAs_2$ revealed unexpected phenomena: a first-order transition between antiferromagnetic (AFM) and superconducting phases and a bi-critical point at $\sim$ 3 GPa and 26 K,[37] as shown in Fig. 5. These results are expected to provide a valuable experimental foundation for understanding the interplay between the AFM and superconducting phases, a most challenging issue in the field of superconductivity.

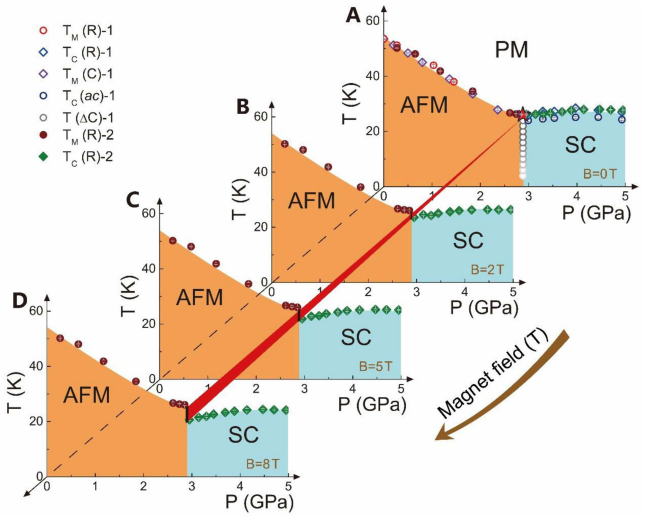


Fig. 5. (color online) Temperature–pressure phase diagrams obtained at different magnetic fields for $Ca_{0.73}La_{0.27}FeAs_2$ single crystals. The acronyms PM, AFM, and SC stand for paramagnetic, antiferromagnetic, and superconducting phases, respectively. The magenta-colored circles and custom diamonds represent the temperature of the AMF phase transition detected from two-run electrical resistance measurements (T_M(R-1) and T_M(R-2)) and heat capacity T_M(C) measurements under hydrostatic pressure conditions. The green diamonds and blue circles stand for the superconducting transition temperature determined from the resistance T_C(R-1) and ac susceptibility T_C(ac) measurements, respectively. The position of the red star denotes the location of the bi-critical point, which is determined by an intersection of extrapolated lines of the pressure-dependent T_M and T_C. The gray circles are the data extracted from the heat capacity results. The radial red line shows the suppressed tendency of T_C by the magnetic field. Reproduced with permission from Ref. [37].

3.3. $A_{1-x}Fe_{2-y}Se_2$ (where A = K, Rb, Tl/K, or Tl/Rb) superconductors

An FeSe superconductor with a T_C of 8 K was discovered by Wu's group in 2008.[32] Surprisingly, the T_C can be increased to $\sim$ 37 K at 8.9 GPa by application of pressure.[17] The pressure-induced dramatic enhancement of T_C in FeSe was considered to be attributed to the pressure-driven increment of spin fluctuation.[68]

In 2011, alkaline iron selenide superconductors, $A_{1-x}Fe_{2-y}Se_2$ (where A = K, Rb, Tl/K, or Tl/Rb), with a transition temperature of $\sim$ 32 K were discovered,[69–72] adding a new subsystem to the family of Fe-based superconductors. The remarkable feature of these superconductors is that the superconducting (SC) phase coexists with an antiferromagnetic insulating phase (245 phase) in the microstructure, which is different from that of the other known FeAs-based superconductors.[31,46,51,73–75] Unexpectedly, high-pressure measurements on $A_{1-x}Fe_{2-y}Se_2$ (where A = K, Rb, Tl/K, or Tl/Rb) revealed that the pressure induces a second superconducting phase with a T_C as high as 48.7 K after the first superconducting phase is suppressed by pressure,[36] as shown in Fig. 6. This is the first example of a pressure-induced reemergence of superconductivity among all high-temperature superconductors. It was proposed that these two superconducting phases may originate from different microphysical mechanisms. Further studies demonstrated that such a pressure-induced reemergence of superconductivity is associated with a quantum criticality.[76]

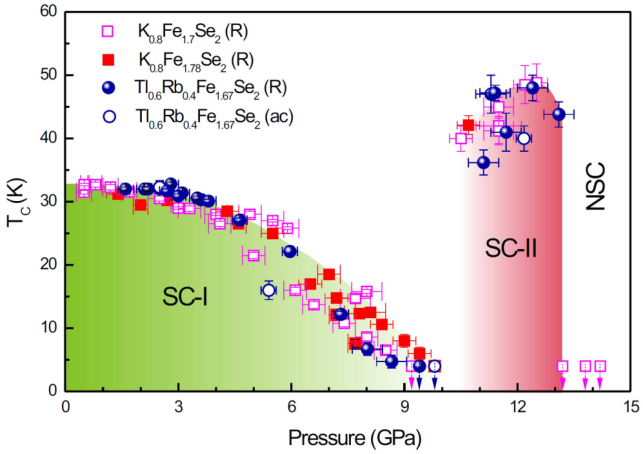


Fig. 6. (color online) Pressure–superconducting transition temperature (T_C) phase diagram for $Tl_{0.6}Rb_{0.4}Fe_{1.67}Se_2$, $K_{0.8}Fe_{1.7}Se_2$ and $K_{0.8}Fe_{1.78}Se_2$. The symbols represent the pressure–temperature conditions at which T_C values were observed from the resistive and alternating current susceptibility measurements; symbols with downward arrows represent the absence of superconductivity to the lowest temperature (4 K). All $Tl_{0.6}Rb_{0.4}Fe_{1.67}Se_2$, $K_{0.8}Fe_{1.7}Se_2$, and $K_{0.8}Fe_{1.78}Se_2$ samples show two superconducting regions (SC-I and SC-II) separated by a critical pressure at $\sim$ 10 GPa. NSC is the nonsuperconducting region at pressure $>$ 13.2 GPa. The maximum T_C is found to be 48.7 K in $K_{0.8}Fe_{1.7}Se_2$ at a pressure of 12.5 GPa. At higher pressures $>$ 13.2 GPa, the samples are nonsuperconducting. Error bars are one standard deviation. Reproduced with permission from Ref. [36].

Owing to the coexistence of the antiferromagnetic insulating and superconducting phases in these superconductors, pressure plays a unique role in revealing its intrinsic physics. To uncover the puzzling issues on the role played by the an-

tiferromagnetic insulating phase in developing the ambient-pressure superconducting phase, comprehensive experimental measurements on the compound with a pure antiferromagnetic insulating phase and mixed phases (antiferromagnetic insulating phase plus superconducting phase) were performed. The results revealed two important issues: 1. the antiferromagnetic insulating phase is not the parent phase of the SC-II phase, and 2. it is a crucial phase for stabilizing the SC-I phase.[4,77,78]

3.4. Heavy-fermion superconductors

The discovery of the first heavy-fermion superconductor, $CeCu_2Si_2$, by Steglich in 1979 not only imitated a new field for studies of unconventional superconductivity but posed a fundamental challenge for understanding the superconducting mechanism in a unified way.[79] Up to now, more than 40 heavy-fermion superconductors have been found.[80,81] Because most non-superconducting heavy-fermion compounds are antiferromagnetic, heavy-fermion superconductors belong to the family of unconventional superconductors.[8,81–85]

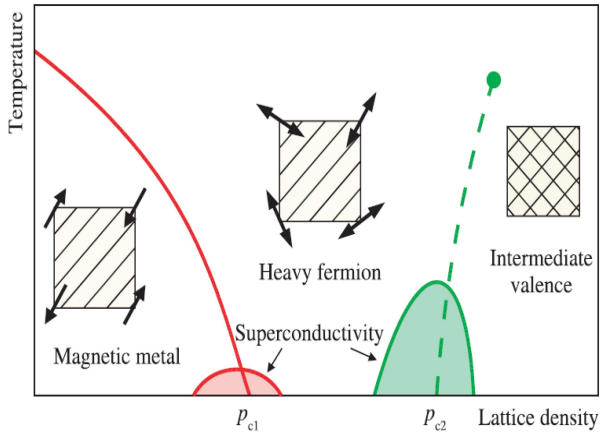


Fig. 7. (color online) Schematic phase diagram of Ce-based narrow-band metals. At low density, the Ce 4f orbitals are singly occupied and carry a static moment (arrows), subject to long-range antiferromagnetic order (red line). Increasing the lattice density (e.g., by hydrostatic pressure) raises the hybridization between more extended orbitals (single-hatched region) and the 4f states, leading to dynamical frustration (Kondo spin-flips; double arrows), which in turn suppresses long-range order and induces an anomalous normal state. In the simplest case, at some distance from the quantum critical point at P_{c1}, where magnetic order is fully suppressed, this state can be modeled as a high-effective-carrier-mass Fermi liquid. Sufficiently close to P_{c1}, magnetically mediated pairing may induce unconventional superconductivity (red dome). Further increasing the hybridization between localized and extended orbitals leads eventually to a less correlated, metallic state (intermediate valence region), in which the 4f electrons delocalize through stronger hybridization with ligand states and occupy wider energy bands at the Fermi energy (cross-hatched region). The transition from the heavy fermion to the intermediate valence configuration may proceed through a first-order phase transition that involves a collapse of the unit cell volume with no change in lattice symmetry (green dashed line), driven by the gain in cohesive energy associated with the wide-band 4f electron configuration. If the critical endpoint of the first-order line is low enough, the system may become sufficiently soft at low temperatures to allow a superconducting state to form around the quantum phase transition associated with the volume collapse, at P_{c2} (green dome). Reproduced with permission from Ref. [8].

A general consensus held for unconventional superconductors is that the superconductivity develops in the vicinity of a suppressed AFM order state, thus, pressure can be used as an effective control parameter to suppress the AFM phase and develop superconductivity.[8,23,37] Based on a number of experimental results, it is believed that antiferromagnetic spin fluctuation is the driving force for the development of the superconducting transition. In addition, valence instability is considered to be another key factor for the development of superconductivity in heavy-fermion compounds.[86] A famous example is the observation of pressure-induced reemergence of superconductivity in $CeCu_2Si_2$, whose pressure–T_C phase diagram exhibits two superconducting domes,[8] as shown in Fig. 7. The accepted explanations are that the antiferromagnetic spin fluctuation is responsible for the first superconducting phase while the valence fluctuation is related to the emergence of the second superconducting phase.[8,87]

Recently, progress has been made in the pressurized non-centrosymmetric $CeTX_3$ (where T = Co, Rh, or Ir and X = Si or Ge) family. Though ambient-pressure unit-cell volumes and critical pressures for superconductivity vary substantially across the series, all family members reach a maximum superconducting transition temperature (T_C^{max}) at a common critical cell volume (V_{crit}), and the T_C^{max} at V_{crit} is found to increase with increasing spin–orbit coupling strength of the d electrons.[88] These correlations show that substantial Kondo and spin–orbit couplings favor superconductivity in this family.

3.5. High-entropy alloy superconductors

High-entropy alloys (HEAs) are a new class of materials that are made from multiple transition-metal elements in equimolar or near-equimolar ratios.[89,90] The elements in HEAs arrange themselves randomly on the crystallographic positions of a simple lattice.[91–94] In many respects, HEAs exhibit excellent properties, including excellent specific strength,[95] ultrahigh fracture toughness at cryogenic temperatures,[96,97] and superior mechanical performance at high temperatures,[98] implying that HEAs have great potential for application. Significantly, superconductivity has been found in HEAs, making them more attractive in the field of materials science.[99,100]

Intriguingly, one of the HEA superconductors, $(TaNb)_{0.67}(HfZrTi)_{0.33}$, displays extraordinarily robust zero-resistance superconductivity under pressure as high as 190 GPa, a pressure that falls within that of the outer core of the earth [101], as shown in Fig. 8. These results revealed by high-pressure studies indicate that this HEA superconductor is a promising candidate for applications under extreme conditions.

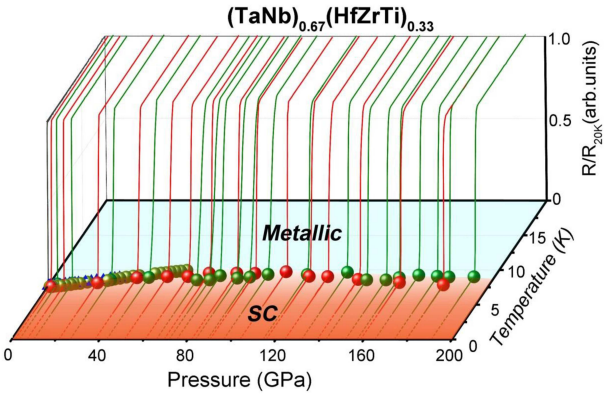


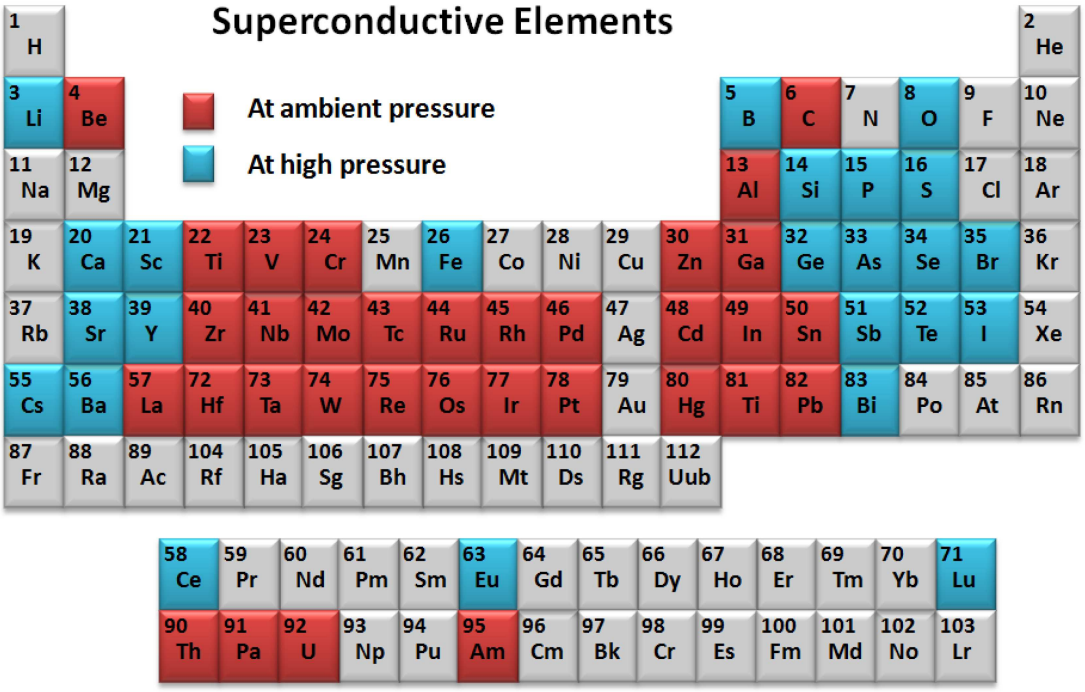
Fig. 8. (color online) Phase diagram of superconducting transition temperature versus applied pressure up to 190.6 GPa for the HEAs, combined with plots of the corresponding resistance vs. temperature. Blue triangles and dark-yellow balls in the diagram represent the zero-resistance superconducting transition temperature T_C obtained from measurements at pressure < 60 GPa, while the red and green balls stand for zero-resistance T_C from the measurements at pressures up to 179.2 and 190.6 GPa, respectively. Reproduced with permission from Ref. [101].

3.6. Element superconductors

Superconductivity was first discovered in element Hg by Onnes,[102] initiating the research field of superconductivity. Later on, numerous element superconductors were discovered. Up to now, thirty three elements are found to be superconducting at ambient pressure.[103,104] By application of pressure, twenty four non-superconducting elements become superconductors,[105,106] as described in the periodic table (Table 1).

Remarkably, pressure can also enhance the superconducting transition temperature for the ambient-pressure element superconductors. Examples can be found in compressed zirconium (with its T_C increasing from 0.55 K at ambient pressure to ~ 11 K at 30 GPa[107]) and vanadium (with its T_C increasing linearly with pressure and reaching ~ 17 K at 120 GPa[108]).

Table 1. Periodic table displaying element superconductors at ambient pressure (highlighted in red) and superconductors (highlighted in blue) at high pressure.



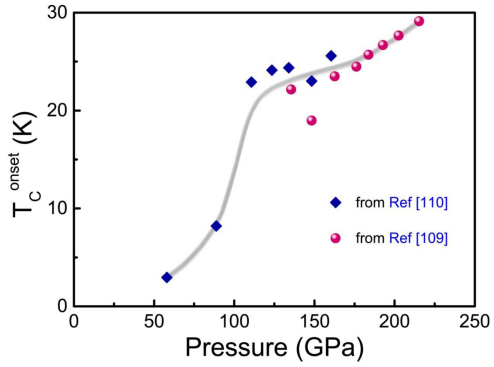


Fig. 9. (color online) Pressure dependence of T_C for element Ca. The data are taken from Refs. [109] and [110]

The highest T_C value (29 K) in elements is found in pressurized calcium, as shown in Fig. 9.[109,110]

Because elements have a pure phase, they are ideal candidates to investigate fundamental physics. Recently, progress has been made in single-crystal black phosphorus.[111,112] Complementary measurements of high-pressure resistance, the Hall effect, and the crystal structure on black phosphorus reveal that hole carriers play an important role in developing superconductivity and enhancing T_C in a single cubic superconductivity in a rhombohedral superconducting phase,[113] as shown in Fig. 10.

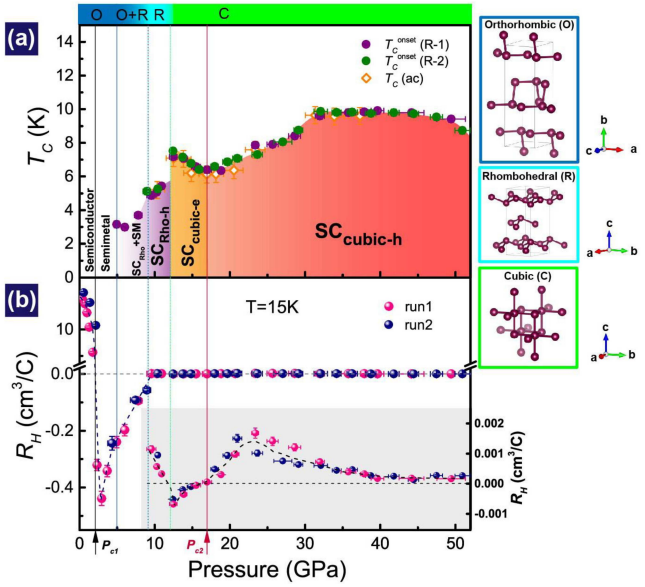


Fig. 10. (color online) Structural, superconducting, and Hall coefficient (R_H) phase diagram of black phosphorus at different pressures. (a) Pressure–temperature phase diagram combined with structural phase information. Here SM and SC_{Rho-h} represent semimetal and superconducting phases with rhombohedral structures and dominant hole carriers, respectively. $SC_{cubic-e}$ and $SC_{cubic-h}$ stand for the superconducting phase with cubic structure and dominance of electron carriers and hole carriers, respectively. T_C^{onset}(R-1) and T_C^{onset}(R-2) represent the onset temperature of superconducting transitions determined from resistance measurements for run 1 and run 2. T_C(ac) represents the superconducting transition temperature determined by ac susceptibility measurements. (b) Pressure dependent R_H measured at 15 K. The inset displays the detailed changes in R_H at the structural phase-transition boundaries. The pink and blue solid points represent Hall coefficients obtained from two independent runs. P_{c1} and P_{c2} stand for the critical pressures of the Lifshitz transitions. Reproduced with permission from Ref. [113].

4. Summary and perspectives

Some impressive progress in the field of superconductivity research has been achieved through high-pressure studies in the past decades, including pressure-induced enhancement of superconducting transition temperatures in cuprates and FeAs-based superconductors, pressure-induced superconductivity in H_2S, and the discovery of Fe-based and element superconductors. Furthermore, many other interesting discoveries have also been made, such as pressure-induced reemergence of superconductivity in heavy-fermion and FeSe-based superconductors, a bi-critical point in an FeAs-based superconductor, and the robustness of superconductivity in a HEA superconductor.

The system being established at SECUF, which integrates the extreme conditions of high pressure, low temperature, and a magnetic field, can perform *in situ* high-pressure (up to 100 GPa) resistance, magnetoresistance, and Hall measurements under low temperature (down to 500 mK) and magnetic field (up to 14 T). With such a powerful high-pressure system, together with other SECUF equipment, it can be expected that an unprecedented opportunity, never available before, for users to find phenomena and physics in condensed matter will be provided. In particular, SECUF will be built at the same campus as the Beijing high-energy synchrotron radiation facilities, at which beamlines for high-pressure x-ray diffraction, absorption, and inelastic scattering measurements will be established. Therefore, it is expected that the complementary high-pressure studies of the 'in-house' instruments and online synchrotron facilities will offer the combined advantage in achieving breakthroughs in frontier research fields of condensed matter physics, materials science, and even chemistry, geophysics, and astrophysics.

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