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Citation: Chin. Phys. B . 2018, 27(7): 077101. doi: 10.1088/1674-1056/27/7/077101

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# Quantum oscillation measurements in high magnetic field and ultra-low temperature

Pu Wang(王瀑)<sup>1,2</sup>, Gang Li(李岗)<sup>1,2</sup>, and Jian-Lin Luo(雒建林)<sup>1,2,†</sup>

<sup>1</sup>Beijing National Laboratory for Condensed Matter Physics, Institute of Physics, Chinese Academy of Sciences, Beijing 100190, China

<sup>2</sup>School of Physical Sciences, University of Chinese Academy of Sciences, Beijing 100190, China

(Received 6 March 2018; revised manuscript received 25 April 2018; published online 1 June 2018)

The physical properties of a solid are determined by the electrons near the Fermi energy and their low-lying excitations. Thus, it is crucially important to obtain the band structure near the Fermi energy of a material to understand many novel phenomena that occur, such as high- $T_c$  superconductivity, density waves, and Dirac-type excitations. One important way to determine the Fermi surface topology of a material is from its quantum oscillations in an external magnetic field. In this article, we provide a brief introduction to the substation at the Synergetic Extreme Condition User Facility (SECUF), with a focus on quantum oscillation measurements, including our motivation, the structure of and the challenges in building the substation, and perspectives.

**Keywords:** Quantum oscillation measurements, Fermi surface, superconducting magnet

**PACS:** 71.18.+y, 07.20.Mc, 07.55.Db

**DOI:** 10.1088/1674-1056/27/7/077101

## 1. Scientific background

A magnetic field can interact with both charge and magnetic moment. It is an important thermodynamic parameter in tuning properties of a material and has been widely employed in the research of condensed matter physics, chemistry, and biology. Interest in the science at high magnetic fields is continuous and worldwide.<sup>[1–3]</sup> Two high-magnetic-field facilities have recently been built in China: the quasi-static field lab at Hefei and the pulse field lab at Wuhan. The effort in the operation of such facilities can be approximately divided into two parts. One was to develop magnets with various field profiles with respect to strength, dimension, and time scale, and the other was to accommodate experimental techniques that use magnets while combined with other parameters like pressure, cryogenic temperature, and external probes like electromagnetic radiation or neutrons.

In the research discipline of solid-state physics, magnetic fields have been used for decades and played a significant role in the discovery of new phenomena. For example, the quantum Hall effect and the fractional quantum Hall effect were discovered in two-dimensional (2D) electron gases in sufficiently high magnetic fields. A magnetic field  $> 10$  T along the trigonal axis of three-dimensional (3D) Bismuth crystal brings the material into the quantum limit yielding a new collective state.<sup>[4]</sup> In addition, a magnetic field induces reentrant superconductivity in f-electron materials like URhGe<sup>[5]</sup> and organic compounds.<sup>[6]</sup> At the same time, magnetic field helps advance our understanding of the microscopic origin of various macro-

scopic properties. For example, the evolution of the upper critical field of a superconductor with temperature and field tilt angle provides information about the superconducting pairing strength and its anisotropy, and a magnetic field-temperature phase diagram of a superconductor with other tuning parameters shows which collective modes exist and whether they are collaborative or competitive.

The properties of materials which consists of atoms with a periodical arrangement in real space are characterized by the electronic band structure. If the highest occupied band(s) in momentum space is(are) not completely filled and there is an energy separating the occupied states from the empty states, the material is metallic, the constant energy contour is a Fermi surface, and the corresponding energy is the Fermi energy. It is very important to experimentally determine the Fermi surface because charge carriers close to the Fermi energy contribute the most to the transport properties of a conductor, as the band structure calculation and prediction always require examination.

When a metal is placed in an external magnetic field, its electron energy levels are quantized into discrete Landau levels. The interval and the degeneracy of the Landau levels vary with the external magnetic field, causing the associated density of states to change with the magnetic field. The properties of a metal oscillate with  $B$  as consecutive Landau levels pass through the extremal cross section of a Fermi surface that is perpendicular to the external magnetic field. This is the magnetic quantum oscillation phenomenon.<sup>[7,8]</sup> This oscillation is periodic in inverse magnet field, and the period, or frequency,

<sup>†</sup>Corresponding author. E-mail: [jllo@iphy.ac.cn](mailto:jllo@iphy.ac.cn)

is solely determined by the area of the extremal cross section of the Fermi surface and a few elementary constants. Thus, by mapping the quantum oscillation frequencies of a crystalline sample with respect to the tilt angle of the magnetic field, it is possible to construct the shape and size of the corresponding Fermi surfaces. The oscillations can be measured experimentally via various physical quantities, including, but not limited to, magnetoresistance (the Shubnikov-de Haas effect), magnetization (the de Haas-van Alphen effect), magnetorestriction, heat capacity, and thermoconductivity.

Quantum oscillation measurements provide information not only about the Fermi surface size, but also about the charge carriers. For a standard 3D Fermi liquid, the first harmonic of the oscillatory part is given by the Lifshitz-Kosevich formula:<sup>[9]</sup>

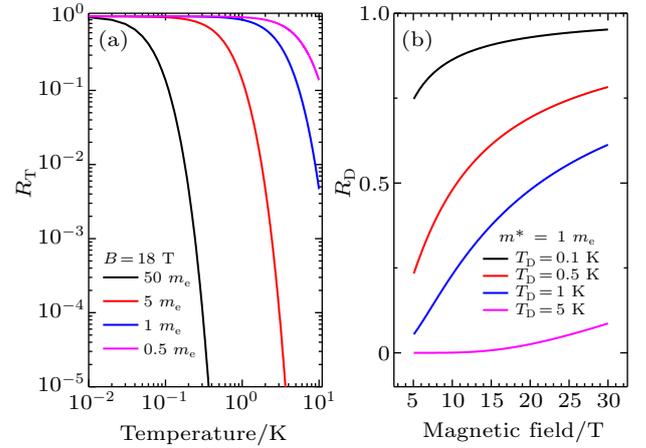
$$M_{\text{OSC}} \propto CB^{3/2}R_{\text{D}}R_{\text{T}}R_{\text{S}} \sin\left(\frac{2\pi F}{B} + \varphi\right),$$

where  $F$  is the oscillation frequency and  $R_{\text{D}}$ ,  $R_{\text{T}}$ , and  $R_{\text{S}}$  are the damping factors from impurity scattering, temperature, and spin splitting, respectively.  $R_{\text{T}} = X/\sinh X$ , where  $X = (2\pi^2 k_{\text{B}} m^* T)/(\hbar e B)$  with  $m^*$  being the averaged effective mass of electrons along the extremal cross section. Therefore, by tracking the change in oscillation amplitude with temperature at a given field tilt angle, an effective mass that takes both electron-electron and electron-phonon interactions into account can be extracted.

In addition to high-quality single crystals, low temperature and a high magnetic field are usually required for quantum oscillation experiments. As illustrated in Fig. 1(a), for charge carriers with a large effective mass, the temperature must be much lower than the He-4 temperature before the oscillation amplitude decreases by decades. Figure 1(b) shows that even with a unit effective mass, if the scattering rate is high, the oscillation amplitude increases to a non-negligible value only at high magnetic fields. Furthermore, because the oscillation part of a measurable quantity is often small compared to the overall background, highly sensitive experimental techniques with a sufficiently large dynamic range are required.

Compared to other Fermi surface-probing experimental techniques, especially angle-resolved photoemission spectroscopy (ARPES) based on the photoelectric effect, quantum oscillation has its own advantages. First, it is a thermodynamic probe that usually detects the properties of the sample bulk, but not limited to layers close to sample surface, as photons used in ARPES have a penetration depth. Second, a quantum oscillation experiment can reach areas in the magnetic field-temperature phase space that are principally or technically difficult for ARPES to reach. Thus, quantum oscillation is an irreplaceable technique for researching novel materials

in condensed matter physics. A few examples are discussed below.

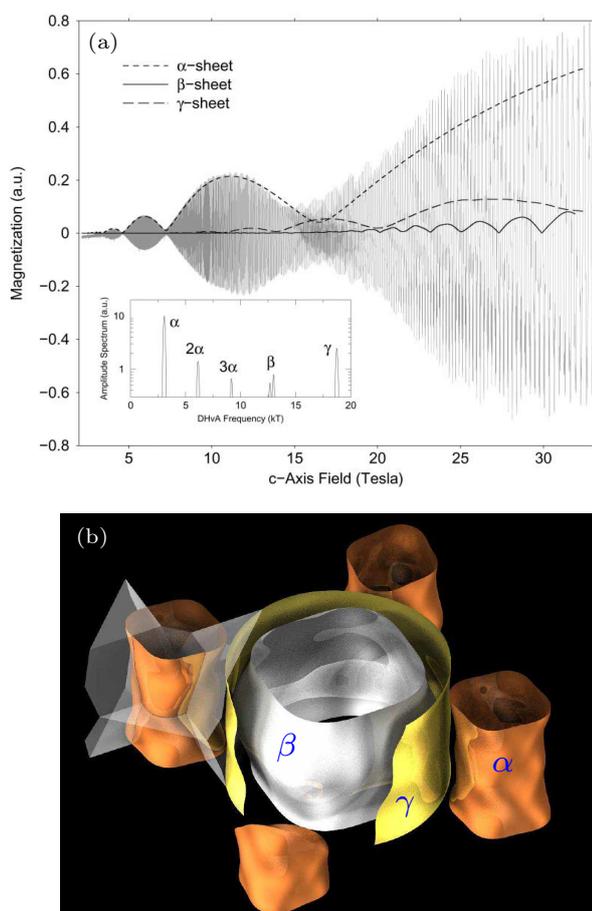


**Fig. 1.** (color online) Change of the temperature damping factor  $R_{\text{T}}$  and the impurity scattering factor  $R_{\text{D}}$  with input parameters. (a)  $R_{\text{T}}$  as a function of temperature with a magnetic field of 18 T and four different effective masses. (b)  $R_{\text{D}}$  as a function of the magnetic field with an effective mass fixed at 1 and different Dingle temperatures  $T_{\text{D}}$ , which is inversely proportional to scattering rate.

In the study of high-temperature superconducting cuprates on the hole-doping side, ARPES showed that the overdoped region has a large circle-like quasi-2D Fermi surface at the normal state, with proper counting of carrier density. However, on the underdoped side, ARPES revealed a so-called “Fermi arc”, which is not closed, in the nodal direction.<sup>[10]</sup> How the overdoped region evolves into the underdoped region and whether there is a closed Fermi surface at the underdoped side are open questions. Quantum oscillation in underdoped cuprates was first observed in the Hall resistance of  $\text{YBa}_2\text{Cu}_3\text{O}_{6.5}$  using a field higher than 40 T in 2007<sup>[11]</sup> and corresponds to a small, electron-type closed Fermi surface. Since then, with the refinement of the quality of crystals, quantum oscillation has been observed at the underdoped side with different doping levels using different probes, including magnetoresistance, torque, heat capacity, and surface impedance, with a total of three closely located frequencies identified.<sup>[12]</sup> Although there is no consensus on whether this Fermi surface arises from a charge-ordered normal state, this knowledge is an important step toward understanding underdoped cuprates.

In the study of the normal metallic state of  $\text{Sr}_2\text{RuO}_4$ , structurally analogous to cuprates but with a line node in its superconducting gap function, early quantum oscillation measurements confirmed its overall band structure by local-density approximation (LDA) calculations. On the other hand, ARPES experiments initially suffered from surface reconstruction and did not succeed in mapping out the Fermi surface topology, especially along the nodal direction of the Brillouin zone, where the three Fermi surfaces almost touch, until

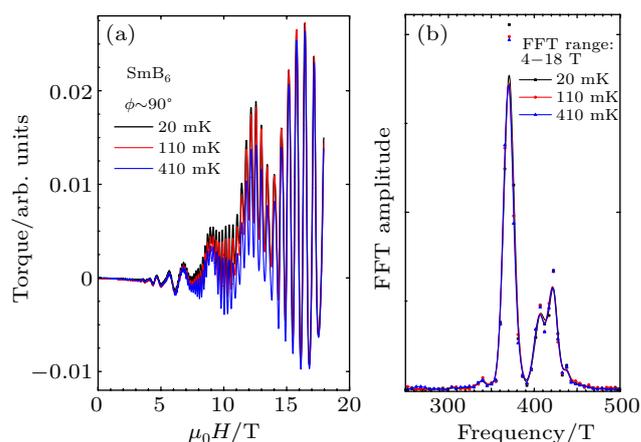
later when different surface preparation and excitation photon energy were used. Figure 2 shows the quantum oscillation data of  $\text{Sr}_2\text{RuO}_4$  obtained by magnetization with all three fundamental frequencies extracted. More recent quantum oscillation data provide not only the correct in-plane Fermi surface, but also detailed interplanar dispersions,<sup>[14]</sup> which could be very important in understanding superconductivity in this compound.



**Fig. 2.** (color online) (a) Quantum oscillations of  $\text{Sr}_2\text{RuO}_4$  measured by torque magnetization in a resistive magnet up to 33 T (from Ref. [13]). (b) Representation of corresponding Fermi surfaces.

In recent years, characterizing solid-state matter by the topology of its band structure has been more widely recognized and much progress has been made. There is experimental confirmation of new theoretical developments, including topological insulators, Dirac semimetals, and Weyl semimetals. For topological insulators, quantum oscillation experiments have been employed to identify the 2D Fermi surface of the surface state. In addition, via Landau level indexing, a nonzero Berry phase is used as the criterion for Dirac-type linear dispersion. When theorist expanded their consideration of topology protection to systems with strong electron-electron interactions, a new class called topological Kondo insulator is proposed, with  $\text{SmB}_6$  as a representative.  $\text{SmB}_6$  has been studied for decades but its resistivity saturation at low temper-

ature is still not well understood. Various origins of the in-gap conducting state have been suggested, with the topological protected surface state being the latest. Li *et al.*<sup>[15]</sup> used torque magnetometry to study the magnetization of  $\text{SmB}_6$  up to 45 T and observed quantum oscillation with three different frequencies. By tracking the angular dependence of oscillation frequencies, they are suggested to be arising from two-dimensional Fermi surfaces having the same symmetry as the crystal surface, thus serves as an important validation of the topological Kondo insulator proposal. Part of the data taken at sub-1 K temperature is shown in Fig. 3.



**Fig. 3.** (color online) (a) Magnetic torque of  $\text{SmB}_6$  measured in a superconducting magnet at dilution refrigerator temperature. (b) Corresponding fast Fourier transform (FFT) spectra. Oscillation amplitude is only slightly temperature dependent in the measured temperature range.

Because of the important information that quantum oscillation experiments can provide, researchers need an integrated low-temperature, high-magnetic-field environment equipped with sensitive probes to carry out such work. Commercially available superconducting magnets usually go up to only 22 T, limited by the NbTi and Nb<sub>3</sub>Sn superconducting wires used. In addition, the cost of the setup and maintenance of the magnets is high for an individual lab. There are a few large-scale high-magnetic-field facilities worldwide that can provide a higher magnetic field, either quasi-static or in pulses. For DC magnet represented by the National High Magnetic Field Laboratory (NHMFL) at Tallahassee, Florida, US, a huge amount of electricity, measured by power in tens of MW, is needed to energize the magnet to bring field up to 36 T in a resistive magnet, and 45 T in a resistive and superconducting hybrid magnet. Due to its uniqueness, the available magnet times are always fully fulfilled by different experimental proposals. On the other hand, because of the short time of a magnetic field pulse, usually measured in milliseconds, it is challenging to utilize all the probes for quantum oscillation in dc fields at a pulsed magnetic field facility. Thus, an all-superconducting magnet working in quasi-static mode, with a magnetic field strength comparable to that of a resistive magnet but with a

much lower operational cost and equipped with sub-1 K refrigerators, is desired. For almost a decade, the NHMFL has been working on such a system that incorporates a low-temperature superconducting wire outsert coil and a high-temperature superconducting tape insert coil. Recently, the system reached 32 T, so deployment for use is expected soon.<sup>[16]</sup> However, the system is planned for general-purpose use.

## 2. Structure of the quantum oscillation substation

At the Synergetic Extreme Condition User Facility (SECUF), a substation with an all-superconducting hybrid magnet and dilution refrigerator, focusing on quantum oscillation experiments, is planned and now under construction. The substation comprises (1) the magnet, (2) the low-temperature environment, (3) sample stage, and (4) measurement equipment. A schematic of the substation is shown in Fig. 4.

### 2.1. Magnet

An all-superconducting hybrid magnet comprising both a low-temperature superconducting (LTS) outsert coil and a high-temperature superconducting (HTS) insert coil is being developed and will be built by institute of electric engineering(IEE), CAS. The initial goal is to generate a 26 T magnetic field by combining a 15 T field from a Nb<sub>3</sub>Sn/NbTi LTS coil and an 11 T field from a Rare earth-Barium-Copper-Oxygen (ReBCO) tape (REBCO) coil, with a 35-mm clear cold bore. IEE has successfully tested a prototype and is now working on technically challenging issues like magnet quenching detection and protection. In addition, because the substation is designed to be a user system, optimization of cryogenic service and the operation of the magnet power supply are also being considered.

### 2.2. Low-temperature environment

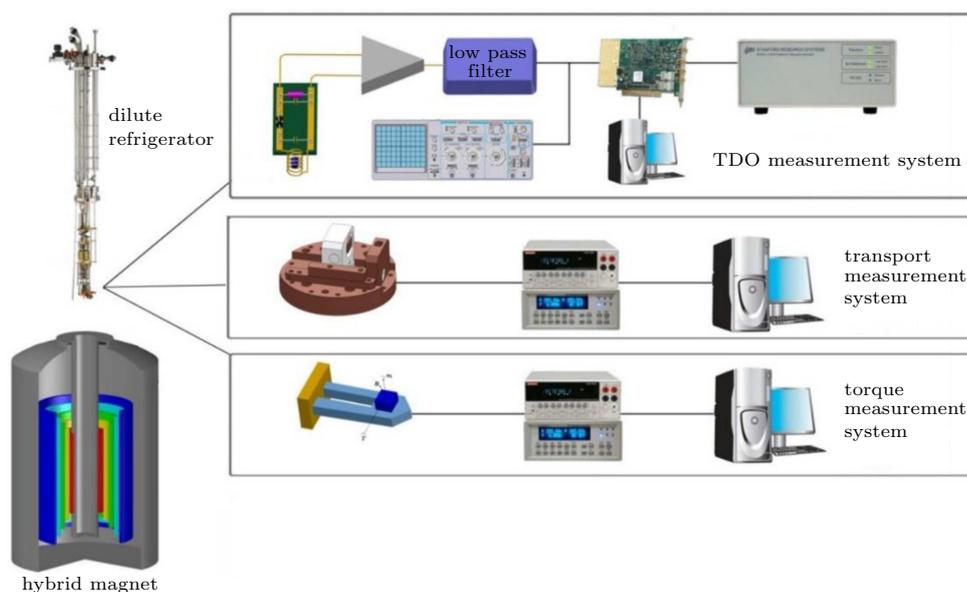
A customized top-loading dilution refrigerator will be fitted into the magnet Dewar with a working condition base temperature targeted at 30 mK for samples in liquid/vapor. In addition to the restricted bore size of the magnet, magnetic levitation of He-3 at high field and strong field gradient requires careful consideration of the structure and layout of the dilution refrigerator insert. Thermometry at dilution refrigerator temperature and under a high magnetic field will also be tested.

### 2.3. Sample stage and rotator

To carry out angle-dependent measurements, the ability to control and measure the angle between a crystal sample and the magnetic field *in situ* is critical. Work needs to be performed at very low temperatures without much friction and cannot be affected by a strong magnetic field. A customized pulling structure is suitable for this requirement; special materials and very high-precision machining are required. Although available space is limited by the tail size of the dilution refrigerator unit, we are confident that at least an 8 × 8-mm<sup>2</sup> area on a rotating stage can be provided for sample or device.

### 2.4. Measurement equipment

To detect quantum oscillation signals in either transport or magnetization channels, the substation will be equipped with highly sensitive electronics for both dc and ac measurements, with a frequency up to megahertz. Both analog and digital electronics will be utilized, with commonality and expandability in consideration. The capability to perform magnetoresistance, Hall resistance, tunneling diode oscillator (TDO), and capacitive magnetic torque measurements will be provided first.



**Fig. 4.** (color online) Schematic of the quantum oscillation substation. Separate probes fit into the dilution refrigerator insert and are used for different types of measurements, i.e., tunneling diode oscillator (TDO) measurement, electrical transport measurement, and torque magnetometry.

### 3. Summary and perspectives

In this article, we showed the importance of experimentally determined fermiology using quantum oscillation measurements and the need for an environment that integrates ultra-low temperature and a high magnetic field. In addition, we presented the structure of the quantum oscillation substation of SECUF at Huairou, Beijing. Developing a user-friendly, all-superconducting hybrid 26 T magnet is technically challenging, but the knowledge in both technique and engineering accumulated during the process will certainly enhance our capabilities in high-magnetic-field science. Upon completion, the substation will provide researchers with easy access to otherwise difficult-to-combine experimental conditions, and let them focus on the study of the electronic structure of unconventional and novel superconductors, heavy-fermion systems, and topologically nontrivial compounds. Because the substation is close to other stations where spectroscopic probes are readily available, a collaborative thorough investigation of systems is expected. Furthermore, the substation will serve as a platform for the development and implementation of new experimental probes and the successful testing of techniques that can be used at other research centers.

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