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A Simple System to Measure Superconducting Transition Temperature at High Pressure *

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A simple hydride system is fabricated to measure the superconducting transition temperature T_c under high pressure using a diamond anvil cell (DAC). The system is designed with centrosymetric coils around the diamond that makes it easy to keep balance between the pick-up coil and the inductance coil, while the superconducting states can be modulated with a low-frequency small external magnetic field. Using the device we successfully obtain the T_c evolution as a function of applied pressure up to 10 GPa for $YBa_2Cu_3O_{6+\delta}$ superconductor single crystal.

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The effect of pressure on transition temperature T_c of superconductors is very important in the search for new materials and to understand superconducting mechanisms. Both variation of chemical composition and high pressure can change the T_c of a superconductor. For example, many high T_c superconducting cuprates show positive effects of pressure on T_c.[1,2] The observed T_c increases with pressure in the Hg12$(n-1)n$ homologous series oxides[3] and high-pressure experiments have been prompted on $HgBa_2Ca_2Cu_3O_8$ (Hg-1223) in which the record 160 K T_c was obtained at 30 GPa[4] for Hg 1223. The large positive $|dT_c/dP|$ of high T_c cuprates brings a hope of finding the materials with higher T_c using chemical pressure under ambient condition. However, the unusual pressure effects on T_c provide a new clue to decode the mysterious mechanisms of high-temperature superconductivity (HTS). Pressure-induced superconductivity is also of particular importance for elements. Ashcroft[5] predicted that elemental hydrogen may become a room-temperature superconductor under sufficiently high pressure. It has recently been pointed out that dense, metallic hydrogen-rich compounds in particular, pressurized hydrides of group 14 elements (C, Si, and Ge), are likely candidates for high temperature superconductors.[6,7] Eremets *et al.*[8] reported the transformation of insulating molecular silane to a metal at 50 GPa that further becomes superconducting above 96 GPa.

Usually there are two methods to measure T_c of a superconductor applied with pressure: i.e., either by measuring the electrical resistance or magnetic susceptibility as a function of temperature. In the low-pressure range (below 2 GPa), a piston cylinder cell is usually used to perform transport experiments, in which the sample volume is approximately 0.1–0.01 cm^3 that can be relatively easily manipulated or detected since the sample size is sufficient large. In the high-pressure range (above 2 GPa), a diamond anvil cell (DAC) is usually employed. Given the small dimension of sample (about 0.1 mm in the maximum dimension) in a DAC, it is difficult to measure resistance because of the difficulties in maintaining the insulation layer and preventing the electric conducting wire from both breaking and short circuiting during the loading press. Compared with electrical conductance measurements, the magnetic method has the advantage that one does not need to prepare the electrodes, which is an onerous experience. However, the small volume of sample (10^{-6}–10^{-7} cm^3) in a DAC results in a very weak magnetic signal that can easily be submerged in the background of the cell itself. Several groups have developed new methods to enhance signal-to-noise ratios in magnetic measurement systems.[9−12]

Timofeev *et al.*[13,14] provided a method to measure magnetic signal of a superconductor with a diamond anvil cell. They reduced the background by keeping a fine balance between pick-up coil and compensation coil to pick up the signal from the sample and to increase the sensitivity of the recording system by using an additional alternating low-frequency modulating magnetic field. However, in their design the detection coil and compensation coil of the system are noncentrosymmetric.[15,16] This allows the balance of the two coils to easily shift with temperature, which is an intrinsic difficulty of the Timofeev prototype.

Alternatively, Schilling[17] designed two balanced primary/secondary coil systems located immediately outside the metal gasket. A very low noise level is achieved by appropriate signal compensation and impedance matching as well as both by using a long time constant on the lock-in amplifier during very slow temperature sweeps and by averaging the multiple measurements.[18] However, there is no modulation coil to tune the superconducting state around the critical temperature that prevents from further increasing the signal resolution around T_c.

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In this Letter, referring to the designs of Timofeev as well as Schiling, we report a method of additional modulation from a variable magnetic field to fit a DAC. We design and fabricate a centrosymmetric system of coils instead of the noncentrosymmetric adopted by Timofeev *et al.*

The purpose of using an additional modulation method is that an external magnetic field can suppress the Meissner effect in the superconducting sample. Above T_c, there is no Meissner effect and there is no additional modulation. Below T_c, to suppress the Meissner effect, the external magnetic field must be very large. In fact, our external magnetic field is less than 100 Oe, such an external magnetic field can only suppress the Meissner effect when the temperature is nearly below T_c. When the external magnetic field modulates with a low frequency F_{low}, the sample will vary from the superconducting state to the normal state and then to the superconducting state again. The varying frequency is two times of F_{low} since both positive and external magnetic field can suppress the Meissner effect in the superconducting sample. We can thus detect T_c under various pressure conditions by measuring the modulation peak of sample's ac susceptibility as a function of frequency of $2F_{\text{low}}$ nearly below T_c.

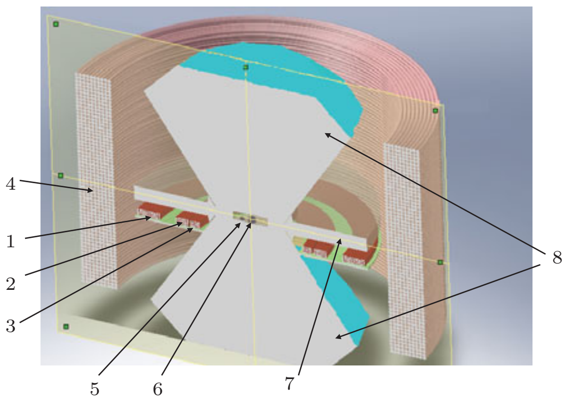

Fig. 1. Schematic view of the setup of the central part of the system. (1) High frequency exciting coil, (2) compensating coil, (3) signal coil, (4) low frequency modulating coil, (5) ruby, (6) sample, (7) metal gasket, (8) diamonds.

Figure 1 shows schematically the diamond anvil cell setup and the centrosymmetric design of the coil system for the magnetic measurements. The measurement method is based on the balanced coils technique with an alternating magnetic field as shown in Fig. 2. The detection coil and compensation coil are made by winding 20 μm diameter Cu wires. After polymerization, the coil has an inner diameter of about 2 mm and is shaped to sit on the lower diamond. To reach the optimal effect, several coils have been tried with the turns between 100 and 500. Increasing the number of turns can increase the sensitivity of the measurement but also increases the diameter of the coils, which reduces the filling factor. The pick-up coil consists of 400 turns of a wire placed around the diamond. The compensation coil and the detection coil are winded by one wire. The compensation coil consists of a few turns on the outside of the detection coil. The compensation coil is wound by hand while monitoring the total signal to obtain a balanced circuit. The modulation coil is made by a 500 μm Cu wire. It consists about one thousand turns of a wire.

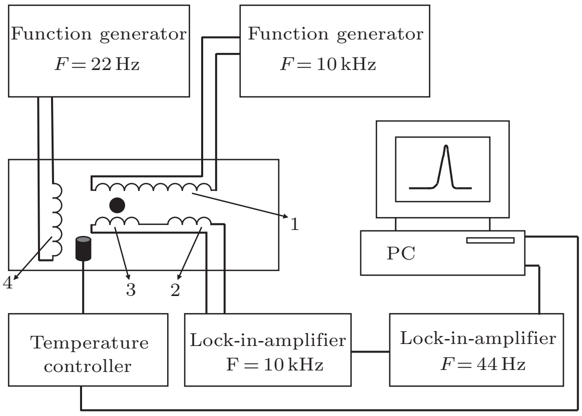

Fig. 2. Logic connections of the whole measuring system. (1) High frequency exciting coil, (2) compensating coil, (3) signal coil, (4) low frequency modulating coil.

Figure 2 presents a block diagram of the circuit. The primary field is generated by a higher frequency function generator. The modulation field is generated by a low frequency function generator. Tests showed that the intensity of the superconducting transition signal is proportional to the magnetic field and the frequency. However, when the field is too high, the transition will become broad and increasing the frequency has the similar effect. Increasing the frequency may cause excessive shielding by eddy currents in the metallic parts of the cell and the gasket. It seems that the overall signal resolution is better when the primary field is about tens of Oe at 10 kHz. To obtain the modulation effect, the frequency of the modulation field must be far away from the frequency of the primary field. We chose the modulation field of about 50 Oe at 22 Hz. The signal from the detection coil and the compensation coil is input to the high frequency lock-in amplifier, and the signal from the out-put of the high frequency lock-in amplifier is feedback to the low frequency lock-in amplifier. The signal and the temperature are recorded by a computer.

In our diamond anvil cell, the top diamond is stationary while the bottom one is moveable. At room temperature we push the bottom diamond toward the top one to apply the pressure by a screw. The diamond culets are about 0.8 mm in diameter. The gasket is made from 250-μm-thick stainless steel that is pre-indented to a thickness of about 70 μm. A 300 μm diameter hole is drilled on the pre-indented gasket. The pressure transmitting medium is silicon oil that will generate a fine quasi-hydrostatic pressure environment at 10 GPa level. The sample has the size of about $130 \times 130 \times 30\,\mu\text{m}^3$. Several small ruby chips are put near the sample. Pressure is determined by the shift in the wavelength of fluorescence from ruby at room temperature. For magnetic susceptibility experiments the cell is placed in a chamber in a cryostat. Temperature is measured with a standard low temperature RhFe thermometer and the overall accuracy is better than ±0.3 K. The measurements are performed in both cooling and heating the cell at a rate of about 0.5 K min^{-1}. Hysteresis between heating and cooling curves is less than 0.4 K.

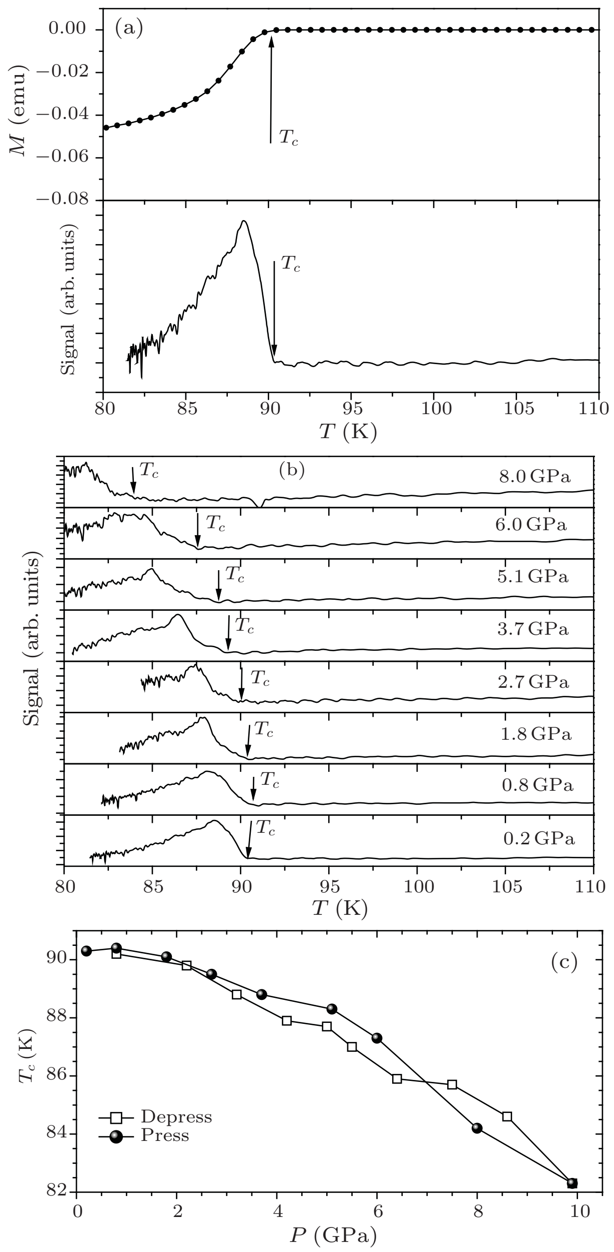

Fig. 3. (a) Superconductivity of a Y-123 superconductor detected using the present system showing a sharp transition. (b) Superconducting transition of Y-123 at various pressures measured using the present system. (c) Evolution of T_c as a function of pressure.

To conduct precise magnetic susceptibility measurements, the whole cell including the gasket has to be made of no-magnet material, like Be–Cu. In these systems since the detection coil and compensation coil systems are centrosymmetric, the background will not increase very much even the gasket is made of stainless steel (T301). In fact, we use a stainless steel gasket that is of the diameter about 3 mm and the background only increase less than one time. Increasing the filling factor can improve the stability of the system over temperature. We reduce as much as possible the diameter of the inner coil to improve the filling factor. We brought all the coil leads separately out of the cell to limit the effect of the variation of magnetization.

Using the system we have successfully measured $T_c(P)$ on $YBa_2Cu_3O_{7-x}$ single crystal as shown in Fig. 3(a). The sample selected clearly shows a peak at 90 K due to superconducting transition at ambient in the cell, which is consistent with the measurement using commercial PPMS. Increasing the pressure up to 10 GPa, T_c of the sample shifts to the low temperature with the a rate of 0.6 kGPa^{-1} as shown in Figs. 3(b) and 3(c). This result is in good agreement with the previous report.[19] The result indicates that this simple system is valid to conduct easy measurements of effects of pressure on T_c for superconductors.

In summary, we have presented here a simple device that allows a relatively easy measurement of pressure dependence of the critical temperature of superconductors, over a wide temperature range. The sensitivity of the method permits a fairly sharp transition with applied pressure.

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