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An ultrafast spectroscopy system for studying dynamic properties of superconductors under high pressure and low temperature conditions

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An ultrafast pump-probe spectroscopy system combined with a cryogenic diamond anvil cell (DAC) instrument is developed to investigate the photo-excitation dynamic properties of condensed materials under low temperature and high pressure (LTHP) conditions. The ultrafast dynamics study is performed on $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212) thin film under LTHP conditions. The superconducting (SC) phase transition has been observed by analyzing the ultrafast dynamics of Bi-2212 as a function of pressure and temperature. Our results suggest that the pump-probe spectroscopy system combined with a cryogenic DAC instrument is an effective method to study the physical mechanism of condensed matter physics at extreme conditions, especially for the SC phase transition.

Keywords: high pressure, ultrafast spectroscopy, superconducting, phase transition

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1. Introduction

High pressure is widely used to adjust the properties of materials due to the changes of atomic interaction and electronic structures.[1] As in the case of superconductors, high pressure could induce the emergence of superconductivity or the increase of the superconducting critical temperature (T_c). For example, the highest T_c record was created in the cuprates $HgBa_2Ca_{m-1}Cu_mO_{2m+2+\delta}$ under ambient pressures, then it was increased up to 164 K at 30 GPa and this record was maintained for nearly two decades.[2] Until recent years, hydrogen-rich compounds attracted widespread attention as a new type of potential high T_c superconductors. In 2014, H_2S was first predicated to transform into the superconducting state with T_c up to 200 K, and it was soon confirmed by different groups successively.[3] After that, various hydrogen-rich compounds presenting high T_c transitions under different high pressures were also found, including La–H, Ba–H, and so on.[4–10]

The main criteria of superconductivity transition include resistance dropping to zero and expelling magnetic fields (Meissner effect). However, due to the extremely small sample size, electrical and magnetic measurement performance is really complex and difficult under ultra-high pressure, usually more than one million atmospheres. Sometimes the extremely weak signals and the poor noise-signal ratio are not good enough to make a convincing judgment, so only a few groups around the world can carry out high-pressure superconductivity research, and there have different statements and opinions on the results of hydride superconductors under high pressure.[11–14]

On the other hand, ultrafast transient spectroscopy measurements have been applied to investigate the electron–phonon interaction, relaxation, and phase transitions of superconductors with several different types, such as cuprates, iron-based, and interface superconductors.[15–24] Fischer *et al.* distinguished the pseudo-gap from the superconducting energy gap accurately by analyzing the relationship between the ultrafast kinetic components of $Y_{1-x}Ca_xBa_2Cu_3O_7$.[15] Mertelj *et al.* found that the pseudo-gap and energy gap of $SmFeAsO_{0.8}F_{0.2}$ may coexist below 50 K.[16] Tian *et al.* observed the superconducting transformation in single-layer FeSe at 68 K through the photoexcited quasiparticles' density and lifetime measurements.[20] The previous studies as mentioned above were conducted at normal pressure with tunable temperature changes, while most ultrafast spectroscopy studies under high pressure were performed at room temperature on a variety of materials like superconductors,[24–27] perovskites,[28,29] Mott insulators, and so on.[30–33] Though it is challenging to combine ultrafast spectroscopy with *in situ* high pressure and cryogenics, it is really beneficial to elucidate the underlying mechanism of superconductivity, especially for the hydride superconductors under high pressure, it may also provide new approaches to understand the relaxation dynamics of quasiparticles, carrier density variation near the Fermi level and electron–phonon interactions, as well as their impact

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on superconducting transitions under high pressure.

In this paper, we designed and assembled an ultrafast spectroscopy system, together with an *in situ* high-pressure micro-cryogenics and a real-time monitoring system, realizing *in situ* LTHP ultrafast dynamic properties investigations. The typical copper oxide superconductor $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212) was selected for the system testing, the transient dynamic properties of Bi-2212 were obtained and a new phase at low temperature was found, which should be assigned to the appearance of superconductivity. The final results reveal that our system is an effective strategy to distinguish the phase transition of condensed matter at extreme conditions, especially for the transition of superconductivity.

2. Experimental setup

The pump-probe spectroscopy system combined with a cryogenic DAC instrument is illustrated in Fig. 1. The femtosecond laser pulse with a wavelength of 800 nm was generated from a laser system (coherent) and split into two parts by a beam splitter. One of them passed through white-light generator to generate the probe laser, in which the 800 nm femtosecond was focused into water by a lens and generated the white light continuum (WLC). In this process, the intensity and the radius of 800 nm femtosecond were modulated by attenuator and aperture, to make sure the WLC covers from 400 nm to 780 nm. The other femtosecond laser passed through the BBO nonlinear crystal to obtain the 400 nm femtosecond, which acted as a pump pulse and excited the sample. The pump and probe pulse were coaxal by dichroscope. Then they together passed through the cryostat and probed the photo-excitation dynamics of the sample. The transmittance change of the probe pulse was collected by a spectrometer and detected by a photomultiplier tube (Zolix, PMTH-S1-CR131) connected to the lock-in amplifier (SR830, DSP). Finally, the data were recorded by a computer. In addition, a Be–Cu DAC and a cryostat made from DAC tools CO., LTD were used to achieve LTHP extreme environment. The vacuum pump (made in Switzerland Fabrication) connected to the cryostats was used to create an adiabatic environment, and a transmission pipe was connected between the cryostat and the liquid nitrogen dewar, with this system we will be able to study materials at temperatures down to 77 K, or down to 10 K, if the liquid helium dewar is used. The whole cryostat was fixed on the three-dimensional motor-driven linear stages, during temperature decreases the position of the sample may change a little bit but can be restored according to the real time monitoring system. The sample temperature was monitored and controlled in real time through the model 22C cryogenic temperature controller. The cryostat also included a pneumatic membrane system as the remote pressure controller, which helped tune the pressure of DAC in the cryostat to avoid any artifacts introduced due to the motion or rotation of the sample when taking it out of the cryostat. Through tuning the pressure controller, we were able to continuously and precisely adjust the pressure on the sample. Moreover, it could ensure precise spatial and temporal overlaps on the sample surface, which was very crucial for the ultrafast pump-probe spectroscopy system.

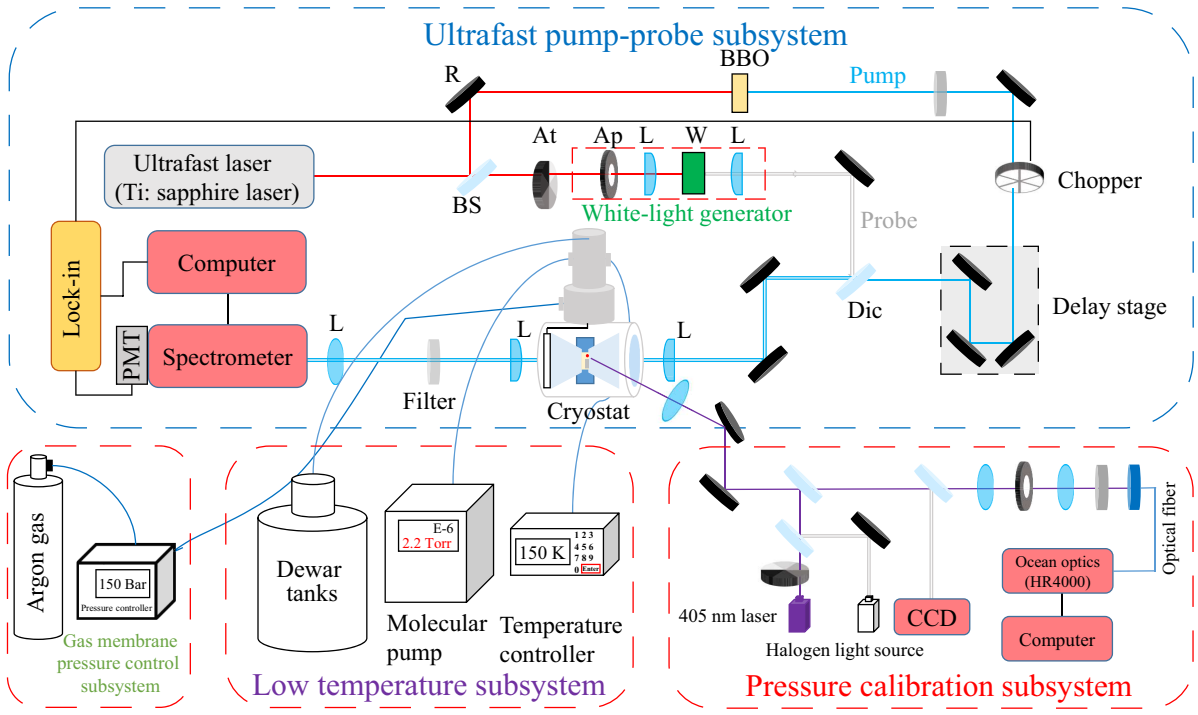


Fig. 1. Schematic experimental setup for the *in situ* high-pressure, low-temperature time-resolved ultrafast optical spectroscopy instrument. BS: beam splitter; BBO: nonlinear crystal; R: reflector; F: filter; At: attenuator; Ap: aperture; L: lens; W: water; Dic: dichroscope.

As the DAC was wrapped within the micro-cryogenics, we used an achromatic lens instead of micro-objective to focus the pump and probe beams onto the sample, and employed an independent micro-telescope to monitor the sample condition in the DAC chamber to avoid any interference from the reflector. This visual image monitor system also contains two components for laser injection and for back reflecting signal collection, which can be used for *in situ* pressure calibration under LTHP conditions. Thus, it can not only monitor the DAC location and manually tune the DAC back to its original position, but also compress and measure the pressure without taking the DAC out of the cryostat. This will effectively exclude potential artifacts caused by the sample spot-to-spot surface fluctuation and greatly enhance the reliability of the pressure-dependent ultrafast dynamics data.

3. Results and discussion

The Bi-2212 is a typical cuprates superconductor. As early as 1990s, Brorson *et al*. performed the first femtosecond pump-probe measurement of some high-temperature superconductors including the Bi-2212.[34] Then there were also some ultrafast dynamics reports on Bi-2212 mainly focusing on its relaxation process, interaction channels or carrier dynamics.[35–37] Cao *et al*. reported the optical detection of QP dynamics in Bi-2212 single crystal and showed an evidence of a strong phonon bottleneck.[38] So we choose the typical superconductor Bi-2212 as a trial sample to test our LTHP ultrafast spectroscopy system. The homogeneous and clear Bi-2212 film was prepared through a sol–gel method by the Xi'an University of Technology.[39] There were seven identified diffraction peaks belonging to Bi-2212 in the x-ray diffraction (XRD) pattern, as shown in Fig. 2(a). Note that the film is pure without any heterophase like Bi-2201 or Bi-2223. All (00l) peaks in the diffraction spectrum are sharp and high, indicating that Bi-2212 thin films have good *c*-axis preferred orientation. The resistance measurement at atmospheric pressure is presented in Fig. 2(b), which shows that the resistance of Bi-2212 film decreases with temperature and superconducting transition occurs at 86.8 K, which is consistent with most results of T_c of Bi-2212.[39,40] The above characterization experiments confirm that Bi-2212 thin films have good quality and they are exact superconductors at low temperatures.

The Bi-2212 thin film is cut into small pieces after characterization and one of them with the proper size is selected to transfer into the DAC's chamber, as shown in the insect photo in Fig. 2. The size of diamond anvil is about 400 μm and that of sample is about 200 μm. Meanwhile, a ruby ball is also placed in the chamber for pressure calibration. After that, the liquid argon is filled into the chamber to serve as the pressure transmission medium. The sealed pressure can be controlled lower than 1.0 GPa and then can be increased to

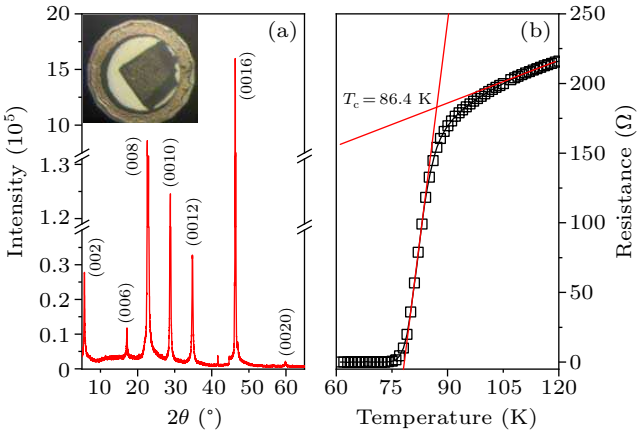


Fig. 2. XRD pattern (a) and *R*–*T* curve (b) of Bi-2212 film at ambient pressure. Inset: the photographs of the sample in the DAC at 1.4 GPa.

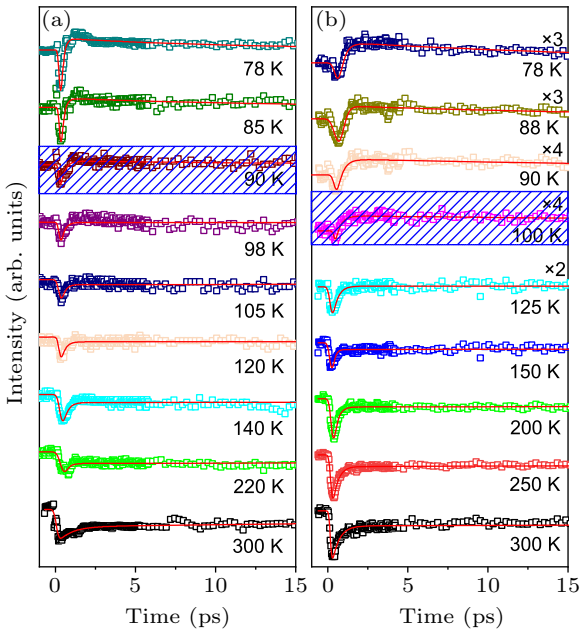


Fig. 3. Temperature-dependent transient curves of Bi-2212 film at (a) 1.4 GPa and (b) 2.2 GPa.

aimed pressure for later spectra measurements. Figure 3 summarized the photo-induced transient curves of Bi-2212 film at different pressures, in which the excitation fluence is fixed in the experiment. Therefore, it is reasonable to speculate that the concentration of photo-generated transient species is also almost invariable with pressure when the excitation intensity is fixed. The decay curves are determined by the relaxation and annihilation of photo-generated species. Figure 3(a) shows the pump-probe curves of Bi-2212 film at 1.4 GPa, which reflect the concentration relaxation process of photo-generated species.[41] According to the previous report, the structure of the Bi-2212 film is still kept invariable before the pressure increases to 37.0 GPa.[40] In this situation, the variance of pressure can influence the relaxation process of photo-generated species in Bi-2212 film, but can not affect the corresponding relaxation mechanism. If the pressure goes higher and the phase transition of Bi-2212 film occurs, in which the photo-generated species in the new phase structure may be different

from that of the initial structure, inducing the TA curves may apparently change. Herein, Fig. 3(a) exhibits that the transient signal relaxes rapidly in the initial time and then follows a slow process, which is similar to the decay curves of superconductor film in the previous report.[20] As the temperature decreases, the signal of the slow component gradually decreases. When the temperature decreases to $\sim$ 90 K, the shape of the decay curves changes completely. A slow positive component appears in the decay curves, suggesting a phase transition may occur and the transition temperature locates at $\sim$ 90 K. In this situation, the spectral signal, the concentration, and the lifetime of photo-generated species in Bi-2212 film may change with phase transition. As the pressure increases to 2.2 GPa as seen in Fig. 3(b), the decay curves of photo-generated species also exhibit a similar evolution to those at 1.4 GPa. Herein, the shape of decay curves also changes at low temperatures, showing that the transition occurs around 100 K. Tian's report on ultrafast dynamics study of single unit cell FeSe has shown similar superconductivity transition showing the photoinduced differential reflectivity changes around T_c.[20] This suggests that the pressure can really affect the relaxation process of photo-generated species but does not change the relaxation mechanism. In other words, increasing environmental pressure may also increase the transition temperature of Bi-2212 film and this tendency is consistent with many theoretical predictions and experimental results.[22,40,42]

Actually, the increase in pressure may broaden the electronic cloud distribution of transient species, which may promote the interaction among transient species and accelerate their annihilation through collision recombination.[42] Therefore, the collision recombination at high pressure may be responsible for this rapid component. On the other hand, considering the structure and atom component of Bi-2212, the electron coupled with phonon (e–ph) may be responsible for this rapid component of transient species, according to the previous report.[20] Besides the rapid collision recombination of transient species, a slow component also appears in the photoexcitation decay curves, which may be assigned to the contribution of across-gap quasiparticle recombination according to the previous report.[20] Herein the relaxation of across-gap quasiparticle recombination is affected by the phonon-induced pair breaking for the condensed matter like Bi-2212 and can be used to distinguish the phase transition induced by temperature. In this situation, the lifetime and the amplitude of the dynamic component as mentioned above can be extracted from experimental data through fitting a bi-exponential function, i.e. $\Delta A/A = A_{\text{rapid}} \exp(-t/\tau_{\text{rapid}}) + A_{\text{slow}} \exp(-t/\tau_{\text{slow}}) + A_0$, and the fitting curves are presented in Fig. 3. In this situation, all the A_{rapid}, τ_{rapid}, A_{slow}, τ_{slow} depending on the temperatures can be obtained. Note that A_0 is assigned to the background level owing to the noise.

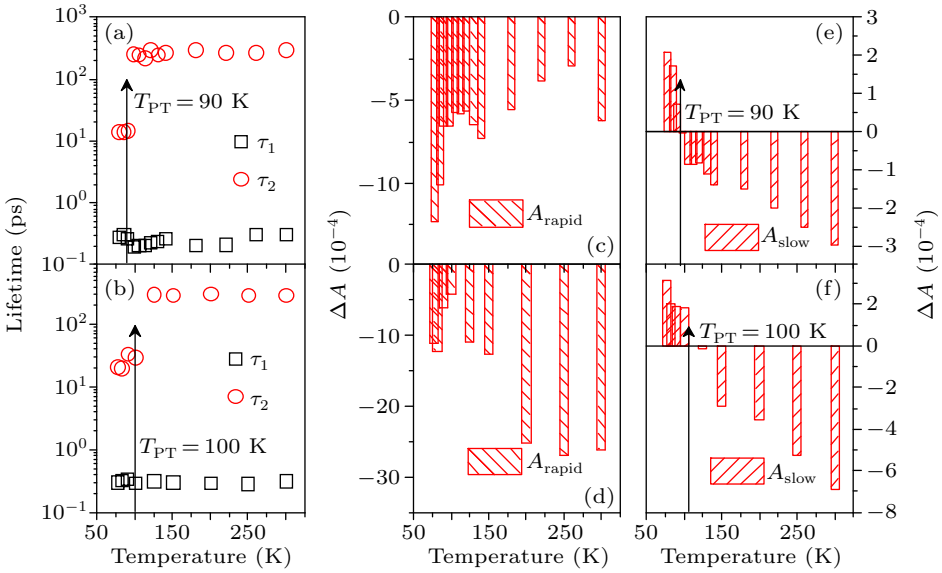


Fig. 4. Temperature-dependent lifetime of the fast and slow components at (a) 1.4 GPa and (b) 2.2 GPa. Panels (c) A_{rapid} and (e) A_{slow} are the rapid and slow components at 1.4 GPa. Panels (d) A_{rapid} and (f) A_{slow} are the rapid and slow components at 2.2 GPa. T_{PT} is the temperature of phase transition.

As seen in Fig. 4(a), the lifetime of the rapid component is fixed at about 0.4 ps and its amplitude at different temperatures is all located in the negative region, which is consistent with the fitted results in the previous report as seen in Fig. 4(c). The lifetime of the slow component at different temperatures is about 300 ps above 90 K. As the temperature decreases, the lifetime suddenly decreases to $\sim$ 20 ps and keeps shortening with the temperature decreasing. Meanwhile, its amplitude also distributes from the negative region to the positive region as seen in Fig. 4(e), the transition temperature is 90 K and it is consistent with that in Fig. 4(a). As the pressure increases to 2.2 GPa, the fitting results are also extracted from

decay curves and summarized in Figs. 4(b), 4(d), and 4(f). It is interesting to find that they are much similar to those at 1.4 GPa, but the transition temperature increases up to around 100 K, after the pressure increases from 1.4 GPa to 2.2 GPa. Meanwhile, the lifetime of the slow component decreases from 300 ps to $\sim$ 30 ps after transition and also keeps shortening as the temperature decreases. Meanwhile, the amplitude of the slow component at 2.2 GPa also follows a similar tendency to that at 1.4 GPa. The similar fitted results suggest that the new phase of low temperature at 1.4 GPa is the same as that of low temperature at 2.2 GPa, reflecting that the increase of pressure can increase the transition temperature according to the photo-excitation relaxation curve based on the TA technique. Referring to previous ultrafast transient spectroscopy studies both the lifetime and the amplitude of dynamic component changes strongly suggest a superconducting transition,[17,20,38,40,43] the Bi-2212 film owns apparent superconductivity as mentioned above, it is reasonable to speculate that the phase transition of Bi-2212 at low temperature is assigned to the superconductivity phase, since the similar transient spectral phenomenon related to phase transition of superconductivity is observed based on our system. However, the superconductivity of condensed matter at low temperatures and high pressure is much more complex and further research work is undertaken and will be conducted in the future.

Furthermore, we can directly obtain the e–ph coupling constant λ from the quasiparticle lifetime. The relaxation dynamics is determined by the e–ph coupling with $\lambda\langle\omega^2\rangle = (\pi/3)(k_{\rm B}T_{\rm e}/\hbar\tau_{\rm e-ph})$, where $\tau_{\rm e-ph}$ can be directly measured by our pump-probe spectroscopy [i.e., $\tau_{\rm fast} = 0.4$ ps, as seen in Fig. 4(a)], $\lambda\langle\omega^2\rangle$ is the second moment of the Eliashberg function, ω is the phonon frequency, $k_{\rm B}$ is the Boltzmann constant and $T_{\rm e}$ is the electron temperature.[44] By taking electronic heat capacity coefficient $\kappa = 30$ mJ/(mol·K^2)[45] and $T_{\rm L} = 78$ K we can estimate $T_{\rm e} = 138$ K using the relation[46]

$$T_{\rm e} = \left\langle \sqrt{T_{\rm L}^2 + \frac{2(1-R)F}{\kappa_{\rm v} l_{\rm s}} {\rm e}^{-z/l_{\rm s}}} \right\rangle, \tag{1}$$

where $l_{\rm s}$ is the optical penetration depth, z is the distance from the surface, F is the pump fluence [i.e., $F = 1.47$ μJ/cm^2 in this work] and $\kappa_{\rm v} = \kappa\rho_{\rm n}$ is the electronic heat capacity coefficient for unit volume, with $\rho_{\rm n}$ being the mole density. We can derive $\lambda\langle\omega^2\rangle = 4.7\times10^{27}$ Hz^2 by combining the information above. Extracting the coherent oscillation by the 78 K data in Fig. 3(a) to obtain its frequency which is 1 THz, we obtain that $\lambda = 1.2$. This value is much higher than that of FeSe film ($\lambda = 0.38$), while comparing with bulk Bi-2212, given by angle-resolved photoemission studies the value of λ is around 2–3,[47] our calculation is reasonable and we suppose the e–ph coupling plays a certain role in the superconducting mechanism.

4. Conclusion

We design a new ultrafast spectroscopy combined with a cryogenic diamond anvil cell (DAC) instrument which can probe the phase transition of Bi-2212 at low temperature and high pressure based on the transient dynamic curves. Since the Bi-2212 owns superconductivity at low temperature and its transient dynamic curves are sensitive to the phase transition at low temperature, we speculate that the phase transition occurring in Bi-2212 at low temperature and high pressure should be assigned to the appearance of conductivity. Moreover, the critical temperature may increase with the pressure. In a word, our system can help people to distinguish the phase transition of condensed matter at extreme conditions and investigate the role of pressure in the superconductivity based on carrier dynamics, in comparison with the experimental system with extreme experimental conditions at home and abroad, which offers a new perspective to analyze the mechanism of superconductivity behavior.

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