Anomalous capacitance response induced by the superconducting gap in an Au/BiFeO3/La1.84Sr0.16CuO4/LaSrAlO4 heterostructure

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Anomalous capacitance response induced by the superconducting gap in an Au/$BiFeO_3$/$La_{1.84}Sr_{0.16}CuO_4$/$LaSrAlO_4$ heterostructure

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Film capacitor characteristics of Au/$BiFeO_3$/$La_{1.84}Sr_{0.16}CuO_4$ on $LaSrAlO_4$ (001) substrate were studied in the temperature range 10–300 K under magnetic fields up to 7 T. Apparent capacitance anomalies were observed at the superconducting transition temperature T_c of $La_{1.84}Sr_{0.16}CuO_4$. Furthermore, the magnetic field dependences of the dielectric relaxation related activation energy can be well fitted from 10 K to T_c by the superconducting gap versus magnetic field. These results suggest an alternative technique for detecting the superconductivity related features in superconducting film, and also may be useful for future tunable multifunctional devices. © *2013 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4824837]

$BiFeO_3$ (BFO) is the one among the few multiferroic materials with the ferroelectric and magnetic orders above room temperature.[1] In recent years, lead free BFO-based heterostructures have attracted a surge of interest.[2–10] From the applied point of view, it provides a potential platform for designing the next logic and spintronic devices, such as resistive switching behavior[2] and ultraviolet photovoltaic effect.[3] From the fundamental viewpoint, curious interplays of various orders (e.g., ferroelectric, anti-ferromagnetic, ferroelastic, ferromagnetic, etc.) of BFO heterostructures also give rise to a variety of fascinating phenomena, such as magnetoelectric coupling,[4] electric-field induced magnetization reversal,[5] and exchange coupling and bias[6–10] in ferromagnet-multiferroic heterostructures.

More interestingly, superconducting gap induced an anomalous leakage current response in a BFO-superconductor heterostructure,[11] indicating that the superconducting order could be coupled with that of BFO. Moreover, some other BFO-based superconductor heterostructures were reported recently.[12–14] These findings further show the importance of investigating the interfacial interactions between the multiferroic and superconducting layers. However, little attention has been devoted to the couplings between superconductivity and dielectric related behaviors of BFO, especially with the magnetic field effect, in which a well matched dielectric-superconductor interface may lead to interesting phenomena both in theory and experiment.[15–18] In fact, the superconducting transition in electrode layers of $YBa_2Cu_3O_{7-\delta}$ can affect the dielectric properties of a thin film capacitor with $Ba_{0.5}Sr_{0.5}TiO_3$ or $Pb(Zr_{0.53}Ti_{0.47})O_3$ dielectric layer.[19–21] For this observation, the proximity effect[19] or stress change in $YBa_2Cu_3O_{7-\delta}$[20] was suggested. Despite these results, the influence of magnetic fields on the capacitor of BFO-based superconductor heterostructure has not been reported so far. And the dielectric properties of BFO have not been fully studied in the low temperature range yet, although some efforts have been made.[22–24]

In this letter, we investigated the heterostructure between BFO and $La_{1.84}Sr_{0.16}CuO_4$ (LSCO) to reveal the superconducting and magnetic effect on the dielectric characteristics of junction. In particular, LSCO is not only structurally compatible with BFO as an electrode material but also can potentially present a wide range of emergent phenomena through the interfacial couplings.[25,26] Consequently, LSCO is expected to be an excellent candidate for the multiferroic/superconductor epitaxial system. The magnetic effect on the anomalous temperature dependences of capacitance was clearly observed, and the resemblance between activation energy and the superconducting gap was confirmed.

The heterostructure of BFO/LSCO was grown on (001) orientated $LaSrAlO_4$ (LSAO) substrate by magnetron sputtering technique. First, LSCO in thickness of 120 nm was grown on LSAO with the deposition temperature around 740 °C by off-axis dc magnetron sputtering, and then the film was *in-situ* annealed for 25 min in 9 Pa molecular oxygen and slowly cooled down to room temperature. Subsequently, a BFO layer (500 nm) was deposited on the LSCO film at 675 °C, while keeping the oxygen pressure at 5 Pa. Au electrodes were sputtered using a metal shadow mask to study the ferroelectric and dielectric properties. The schematic diagram of measurement circuit is given in the inset of Fig. 1(a). To investigate the true ferroelectric remnant polarization (P_r) characteristics of BFO, the polarization voltage *P-E* hysteresis loops were measured by Radiant Technologies' Precision Premier II in a remanent measuring mode, which is similar to the positive-up-negative-down method in principle.[27] Dielectric properties were investigated using Agilent 4294A precision impedance analyzer (USA) at various frequencies (1 kHz–5 MHz). Magnetization measurement was performed in the Magnetic Property

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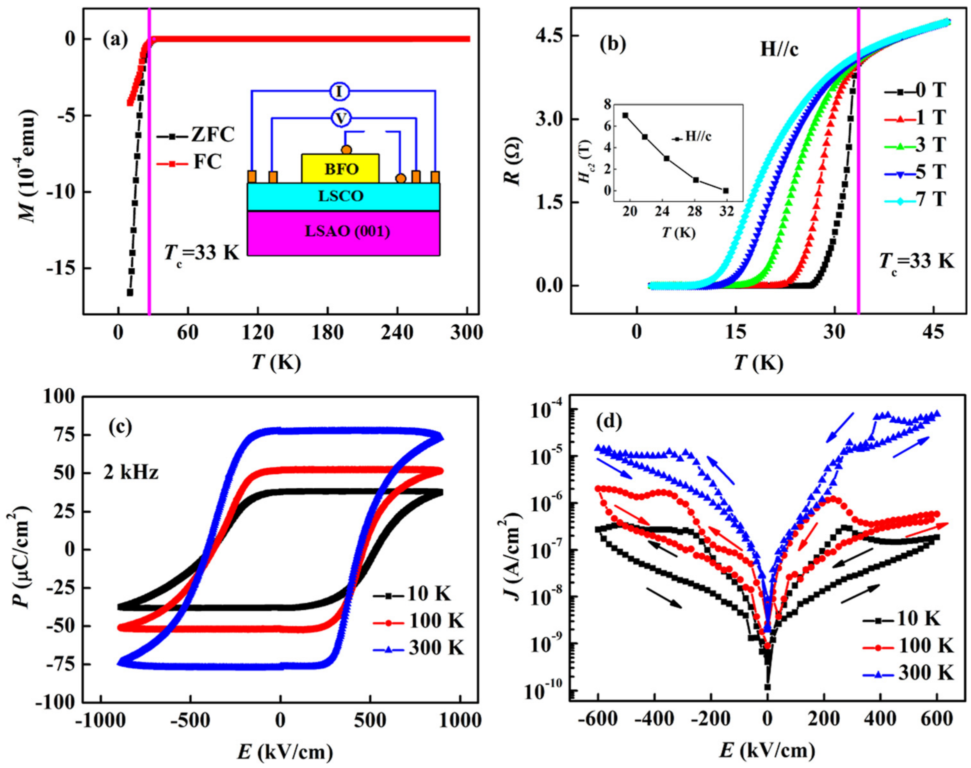

FIG. 1. (a) ZFC and FC magnetization curves as a function of temperature for BFO/LSCO/LSAO heterostructure. The bilayer is oriented with the magnetic field of 50 Oe parallel to the *c*-axis of the films. Superconducting transition is indicated by a magenta line. The inset is the schematic diagram of measurement circuit of the BFO/LSCO/LSAO heterostructure. (b) The broadening of resistive transition for the magnetic fields up to 7 T parallel to the *c*-axis of LSCO. The superconducting transition is indicated by a magenta line. The inset shows the temperature dependences of upper critical field. (c) *P-E* loops of the BFO/LSCO at 10 K, 100 K, and 300 K with the frequency of 2 kHz. (d) Electric field dependences of the current density of the BFO/LSCO at 10 K, 100 K, and 300 K.

Measurement Systems (MPMS, Quantum Design). The resistance measurements of LSCO were carried out by a standard four-terminal method in a temperature range of 10–45 K. The temperature and applied magnetic fields (up to 7 T) during measurements of resistances and dielectric properties were controlled by a Physical Property Measurement System (PPMS, Quantum Design). The magnetic field H was taken to be parallel to the c-axis of the heterostructure.

The X-ray diffraction patterns (not shown here) of LSCO thin film and BFO/LSCO heterostructure indicated that LSCO and BFO crystallized perfectly with a (00*l*) crystalline orientation. All peaks of BFO/LSCO correspond to LSCO and BFO phases.

The superconducting transition temperature T_c of the heterostructure is about 33 K, which is determined from the temperature dependences of magnetization field in 50 Oe under zero field cooling (ZFC) and field cooling (FC) processes, and also can be determined from the temperature dependent resistance of LSCO under magnetic fields (see Figs. 1(a) and 1(b)). The reduced temperature of zero resistance may be related to the relatively lower oxygen pressure during the process of sputtering the second layer of BFO.[28] The inset of Fig. 1(b) shows the temperature dependences of the upper critical field H_{c2} determined from the resistance drops to 90% of the normal state resistance.[29] Within the weak-coupling BCS theory, the H_{c2} at $T = 0$ K can be determined by the Werthamer-Helfand-Hohenberg (WHH) equation,[30]

$$H_{c2}(0) = -0.693[(\mathrm{d}H_{c2}/\mathrm{d}T)]_{T_c} T_c. \quad (1)$$

The $H_{c2}(0)$ can be estimated to be about 40 T with $H//c$, which is consistent with that previously reported for LSCO film.[31]

The ferroelectric hysteresis loops of Au/BFO/LSCO capacitor with different temperatures at the frequency of $f = 2$ kHz in Fig. 1(c) show a square shape, and yield a large remnant polarization P_r value of 75 μC/cm^2 at 300 K, which is much higher than the value $\sim$7.3 μC/cm^2 reported earlier for an epitaxial film of BFO.[32] It has been found that the BFO usually exhibits high leakage current, which would limit the wide applications of the material.[33] Fig. 1(d) depicts the current density versus electric field at 10 K, 100 K, and 300 K, where the arrows indicate the voltage sweeping directions. With an electric field of $\pm$ 200 kV/cm at 300 K, the leakage current density is about 10^{-6} A/cm^2, which is much lower than the prior reports.[33,34] The Schottky contacts of Au/BFO may help to suppress the leakage current.[35] Both the lower leakage current density and the good ferroelectric hysteresis loops indicate the high quality of our sample.

Figure 2 illustrates the frequency dependences of real (C') and imaginary (C'') parts of capacitance for Au/BFO/LSCO at the selected temperatures with magnetic fields up to 7 T. Apparently, C' is almost constant at low frequency, and then drastically decreases at high frequency, while C'' increases at low frequency, and then decreases with further increasing frequency at all selected temperatures. More interestingly, with increasing magnetic field, C'obviously decreases and the peak frequency of C'' shifts to low values at the selected temperatures of 15 K and 20 K. Nevertheless, both the C' and C'' have no response to magnetic fields at 40 K and 60 K.

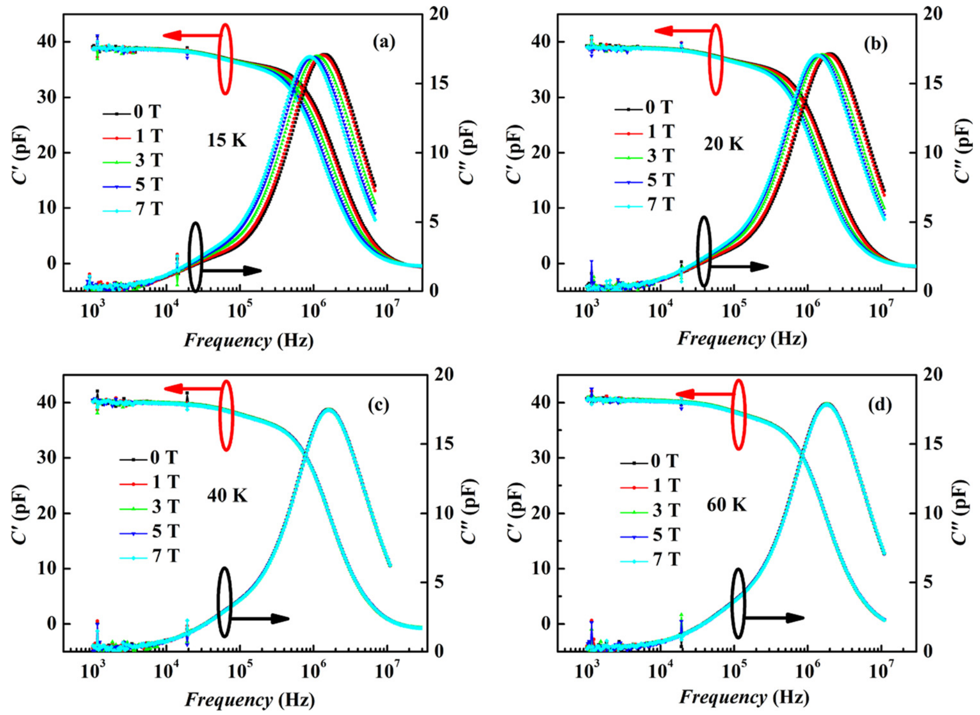

FIG. 2. Frequency dependencies of real and imaginary parts of capacitances of BFO/LSCO at (a) 15 K, (b) 20 K, (c) 40 K, and (d) 60 K in magnetic fields up to 7 T.

Figures 3(a) and 3(b) present the temperature and magnetic field dependences of the C' and C'' at frequency of 1000 kHz, respectively. Both C' and C'' present the same with increasing magnetic fields above T_c. However, as the temperature approaches the T_c of LSCO, both C' and C'' show the sharp change and can be affected greatly by the magnetic field. The fascinating jumps of the capacitance only appear at a relatively high frequency as shown in Fig. 3(c), and the contrast between Figs. 3(a) and 3(d) also confirms this point, which is similar to that reported in $Pb(Zi,Ti)O_3/YBa_2Cu_3O_7$ heterostructure.[21] In fact, the discontinuity of capacitance with the magnetic field at the T_c was predicted by the Ginzburg-Landau approach previously.[15,17]

The capacitance variations in magnetic fields can be characterized by a *MC* parameter defined as $MC = 100 \times |C'(H) - C'(0)|/C'(0)$, where $C'(H)$ and $C'(0)$ are real parts of capacitance with and without magnetic fields, respectively. Fig. 4 shows the frequency and temperature dependences of *MC* for the sample in different magnetic fields. It can be noted that the amplitude of the *MC* effect for $H = 7$ T amounts to nearly 60% at 20 K, the *MC* effect can be achieved in a wide frequency range from 200 kHz to 2 MHz, see Fig. 4(a), and only appears near the T_c of LSCO, see Fig. 4(b). It should be noted that the *MC* effect is much higher than that reported in a single phase BFO system.[36] The shift of C'' frequencies with magnetic fields indicates that magnetic fields can affect the dielectric relaxation. It is known that one of the most important outcomes of the dielectric relaxation is the quantitative information of relaxation time $\tau(T)$, which can be obtained from the equation of $\tau(T) = 1/2\pi f_{max}$, where f_{max} is the frequency of the peak of C''. From Fig. 5(a), we can see that the variations of τ show different behaviors at different temperature ranges. Below T_c, the τ decreases with decreasing temperatures and follows

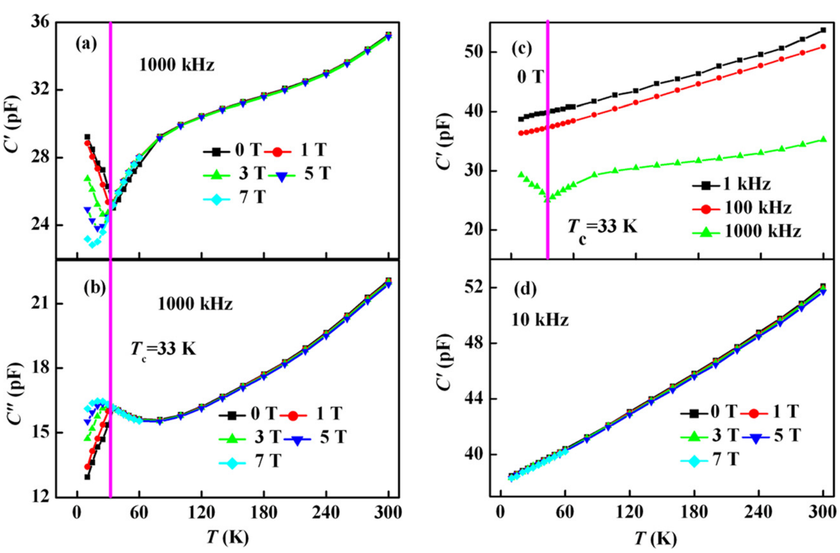

FIG. 3. (a) Temperature dependencies of real part of capacitances of BFO/LSCO at 1000 kHz in magnetic fields up to 7 T. (b) Temperature dependencies of imaginary part of capacitances of BFO/LSCO at 1000 kHz in magnetic fields up to 7 T. (c) Temperature dependencies of real part of capacitances of BFO/LSCO at 1 kHz, 100 kHz, and 1000 kHz without magnetic field. (d) Temperature dependencies of real parts of capacitances of BFO/LSCO at 10 kHz in magnetic fields up to 7 T. The temperature of superconducting transition is indicated by a magenta line.

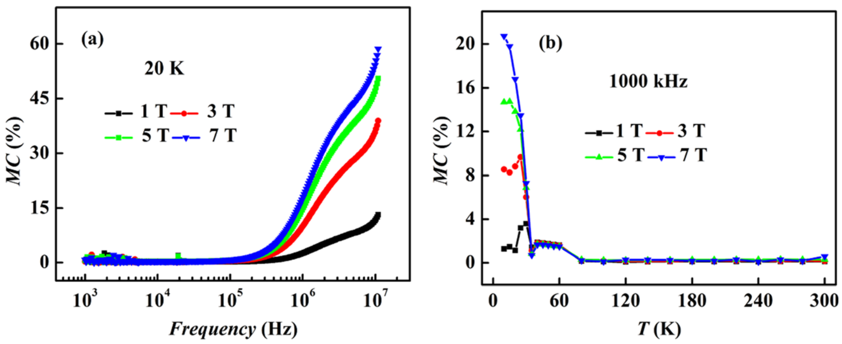

FIG. 4. (a) Frequency dependencies of the *MC* effect at 20 K. (b) Temperature dependencies of the *MC* effect at a frequency of 1000 kHz in magnetic fields up to 7 T.

the extended Arrhenius law $\tau = \tau_0 \exp[-E_a/k_B T]$, where E_a is the activation energy, k_B is the Boltzmann constant, and the τ_0 is the relaxation time at the critical temperature. In turn, the $E_a(H)$ could be obtained as shown in Fig. 5(b). Most surprisingly, the obtained activation energy E_a is about 10.6 meV in $H = 0$ T, which is close to the superconducting gap of $\Delta_0 = 10.3$ meV in LSCO given by Raman spectra[37] and scanning tunneling microscope.[38] This suggests that the variations of C' and C'' are closely related to the superconducting gap of LSCO. Indeed, similar anomalies, caused by the evolution of the superconducting gap, have been observed in other superconductor-based heterostructures.[25,26,39] To reveal the effect of the superconducting transition on the BFO/LSCO interface more deeply, the superconducting gap as the function of magnetic field should be further investigated and compared with the activation energy.

Actually, the zero superconducting gap Δ_0 is nearly proportional to T_c, and the empirical relation between temperature and upper critical field can be formulated as $H_{c2}(T)/H_{c2}(0) = 1 - (T/T_c)^2$. Therefore, the zero temperature superconducting gap as a function of magnetic field can be approximately expressed as

$$\Delta_0(H) = \Delta_0[1 - H/H_{c2}(0)]^{1/2}, \qquad (2)$$

where $H_{c2}(0)$ is the upper critical field at zero temperature, $\Delta_0(H)$ and $\Delta_0(0)$ are the zero temperature superconducting gap with and without magnetic fields, respectively. The values of $\Delta_0(H)$ reduce rapidly with increasing magnetic fields, and could be fitted well by using Eq. (2), with the fitted parameters of $\Delta_0 = 10.62$ meV and $H_{c2}(0) = 40.47$ T. Both the magnitude and the magnetic fields evolution of E_a are similar to the superconducting gap Δ, which further confirms that the capacitance anomaly in the Au/BFO/LSCO case originates from the superconducting transition of LSCO and indicates that the activation energy E_a denotes the value of the superconducting gap of LSCO. Indeed, negative magnetodielectric responses occur because magnetic field suppresses the excitation from a singly occupied state to another state.[40] According to Maxwell-Wagner capacitor model,[41] the effective capacitance response mainly composed of the bulk BFO and the interface between LSCO and BFO. From the above analysis, dielectric-superconducting couplings may occur near the interface at T_c due to the opening of the superconducting gap, causing charge injection from the electrode to the dielectric or vice versa (charge depletion). Therefore, the relative contribution of interface occurred consequently. While for temperatures below T_c, τ displayed almost a twofold reduction magnitude with decreasing temperature. It should be noted that the increase of τ with increasing magnetic fields implies the release of the relaxation process in magnetic fields. So the dielectric relaxation may be related to the superconducting gap opening. Meanwhile, the formation of cooper pairs below T_c and other multiferroic-superconductor couplings effect may also affect the behavior of the interface[15,17]between BFO and LSCO. Moreover, the magnetic field dependences impedances of the heterostructure display the same (not shown here) with a wide frequency range, which suggests that the magnetoresistance effect[42] plays a less important role in our results. It is also important to rule out the strain effect. It has been proposed that phase symmetries could be tunable for BFO on LSAO.[43–45] However, the misfit strain will be relieved at film thickness of ~50 nm.[45] More importantly, the jumps of the capacitance only appear at T_c. The detailed origin of the changes in C' and C'' characteristics under magnetic fields below T_c deserves further investigation. An in-depth study of anomalous capacitance effect in a LSCO/BFO/LSCO junction may add more to the rich functionalities of superconducting/multiferroic heterostructures.

In sum, the magnetic field and temperature dependences of the dielectric spectroscopy were investigated systemically

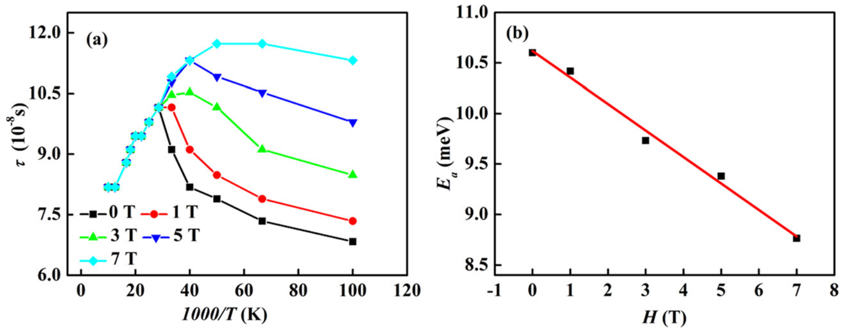

FIG. 5. (a) Temperature dependencies of relaxation time τ in different magnetic fields. (b) Magnetic field dependencies of the activation energy E_a. The red line is the fitting result using Eq. (2).

in artificially engineered epitaxial multiferroic BFO and superconductor LSCO heterostructure. The superconducting transition of LSCO leads to pronounced changes of C' and C'', and the obvious deviation of the peak of τ from its original track indicates the opening of the superconducting gap. The E_a as a function of magnetic field exhibits the same behavior as the superconducting gap. This might give an easy and effective means to detect the energy gap of high temperature superconductors and encourage future studies on multiferroic-superconductor heterostructures to explore the interactions across the interfaces and to construct advanced devices with tunable functionalities.

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