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Search for broken time-reversal symmetry near the surface of superconducting $YBa_2Cu_3O_{7-\delta}$ films using β-detected nuclear magnetic resonance

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Weak spontaneous magnetic fields are observed near the surface of $YBa_2Cu_3O_{7-\delta}$ films using β-detected nuclear magnetic resonance. Below T_c, the magnetic field distribution in a silver film evaporated onto the superconductor shows additional line broadening, indicating the appearance of small disordered magnetic fields. The line broadening increases linearly with a weak external magnetic field applied parallel to the surface, and is depth independent up to 45 nm from the Ag/$YBa_2Cu_3O_{7-\delta}$ interface. The magnitude of the line broadening extrapolated to zero applied field is less than 0.2 G, and is close to nuclear dipolar broadening in the Ag. This indicates that any fields due to broken time-reversal symmetry are less than 0.2 G.

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I. INTRODUCTION

The highly unconventional electronic properties of high-T_c superconductors (HTSC) give rise to interfacial phenomena that are important fundamentally (e.g., in probing symmetry of the bulk electronic ground state); as well as in applications (e.g., junction-based devices). While significant progress has been made in understanding the transport properties of such interfaces, very little is known about their *magnetic* properties, in part due to the lack of an appropriate local magnetic probe. A particularly unresolved issue is whether the superconducting order parameter (OP) breaks time-reversal symmetry (TRS) near the surface.[1,2] A characteristic feature of TRS breaking (TRSB) is spontaneous magnetization; however, Meissner screening cancels this in the bulk, limiting the associated fields to within the magnetic penetration depth of defects and interfaces.[3] To measure this magnetization directly, one requires a sensitive depth-dependent local magnetic probe. In this paper we use a technique based on depth-controlled β-detected nuclear magnetic resonance (β-NMR) to search for TRSB order near the surface of the high-T_c cuprate superconductor $YBa_2Cu_3O_{7-\delta}$ (YBCO).

In contrast to Ru-based and heavy fermion superconductors,[4,5] there is no evidence for TRSB in the bulk of HTSC cuprates, particularly YBCO,[6] where OP-phase-sensitive measurements have established spin-singlet $d_{x^2-y^2}$-wave order.[7] There are some indications of weak magnetism,[8,9] some of it related to the CuO chains in YBCO,[10] or to vortex cores above the lower critical field H_{c1}.[11] Surface scattering of the Cooper pairs from most surfaces perpendicular to the CuO_2 planes frustrates $d_{x^2-y^2}$-wave order within a few coherence lengths of the interface.[3] This leaves a high density of mobile holes [as evidenced by the zero bias conductance peak (ZBCP) in tunneling spectra] that may condense into a superfluid of different symmetry than the bulk[12] (e.g., s-wave, or TRSB states such as $d_{x^2-y^2} + is$ and $d_{x^2-y^2} + id_{xy}$).[13] Other origins of a TRSB state include frustrated OP near grain boundaries or junctions,[3] the interaction of a self-induced magnetic field caused by the OP distortion with the OP itself,[14] and finite size effects in thin films.[15,16]

Experiments to detect TRSB near surfaces have yielded controversial results. Carmi *et al.* measured a weak spontaneous magnetic field using superconducting quantum interference device (SQUID) magnetometry near the edges of epitaxial c-axis oriented YBCO films below T_c.[17] Spontaneous Zeeman-like splitting of the ZBCP, due to TRSB, has been seen in some tunneling measurements,[18] but not in others.[19] Phase-sensitive measurements, which could also detect spontaneous flux, showed no evidence of a TRSB state.[20] A resolution to this disagreement requires more direct information of interface magnetism in cuprates using a local magnetic probe that can locate the origin and distribution of any such fields on the atomic scale.

In this study, we present direct measurements of the magnetic field near the interface of silver and YBCO films using β-NMR. We measure the field distribution using a highly spin-polarized $^8Li^+$ beam implanted into a thin silver overlayer deposited on YBCO. We find an inhomogeneous broadening of the field distribution below the T_c of YBCO, with the probe ions stopping at an average distance of 8 nm from the Ag/YBCO interface. By extrapolating the line broadening to zero applied field, we find that the mean internal field is very close to experimental resolution determined by the Ag nuclear dipolar fields. In this way we obtain an upper limit of 0.2 G for any spontaneous fields of electronic origin.

II. EXPERIMENT

The experiment was performed using β-NMR of $^8Li^+$ at the ISAC facility at TRIUMF in Vancouver, Canada. For details, see Refs. 21 and 22. Similarly to NMR, to measure the resonance, we apply a field along the spin polarization (here in the plane of the films) $\mathbf{B}_0 = B_0\hat{y}$ (with $5 \leqslant B_0 \leqslant 150$ G) and follow the polarization of $^8\mathrm{Li}^+$ as a function of the frequency ω of a small transverse radio-frequency (rf) field of amplitude $B_1 \sim 1$ G, applied along the $\hat{x}$ axis. The resonance condition is $\omega = \gamma B_{\mathrm{loc}}$, where for $^8\mathrm{Li}^+$, $\gamma = 0.63015$ kHz/G, and B_{loc} is the local field. At this ω, the polarization, initially parallel to the $\hat{y}$ axis, is averaged by precession in the oscillating field. In the absence of dynamic effects, the resulting resonance is generally broadened by any static inhomogeneity in the local magnetic field. Thus, the line shape offers a detailed measurement of the distribution of local magnetic fields in the sampled volume determined by the beamspot (~3 mm in diameter) and the implantation profile (see the forthcoming discussion).

A pulsed rf mode was used in this study. The rf field is applied in 90° pulses randomized in frequency order, instead of the continuous wave (cw) mode commonly used.[22] In randomly pulsed rf (RPRF), one obtains a high signal-to-noise ratio with minimal contribution from both variations in the incoming $^8\mathrm{Li}^+$ rate and cw power broadening. Because of the limited B_1, the RPRF mode is suitable for narrow lines up to a few kHz in width. Figure 1 shows the resonance spectrum at 100 K with a half width at half maximum (HWHM) of approximately $\Delta_0 \approx 0.15$ kHz (or 0.24 G). Similar cw spectra can be at least twice as broad,[21] making it difficult to resolve a small additional broadening.

Major advantages of β-NMR in detecting TRSB are the abilities (i) to implant the probe $^8\mathrm{Li}^+$ at low energy into thin layered structures and (ii) to control the mean implantation depth on the nanometer scale. In this study, $^8\mathrm{Li}^+$ is preferentially implanted into the thin silver overlayer evaporated onto YBCO, instead of the superconductor itself. Stopping the probes in the overlayer eliminates the possibility of the probe perturbing the superconductor. Also, the $^8\mathrm{Li}^+$ nucleus carries a small electric quadrupole moment, so the spectrum in the Ag is free of any quadrupole splittings that are present in noncubic YBCO.[21] Consequently, the resonance of $^8\mathrm{Li}^+$ in Ag, below 1 Tesla, is a single narrow line with a T-independent linewidth attributed to nuclear dipole broadening from the small nuclear moments of ^{107}Ag and ^{109}Ag.[22] From basic magnetostatics, any inhomogeneous fields in the YBCO layer will decay exponentially outside the superconductor as $\exp(-\frac{2\pi}{a}z)$, where a is the length scale of the inhomogeneity.[23–25] Thus we can only detect such fields provided our probe-YBCO stopping distance z is $\lesssim \frac{a}{2\pi}$. Any static field inhomogeneities arising in this way will broaden the intrinsic resonance of the Ag layer.

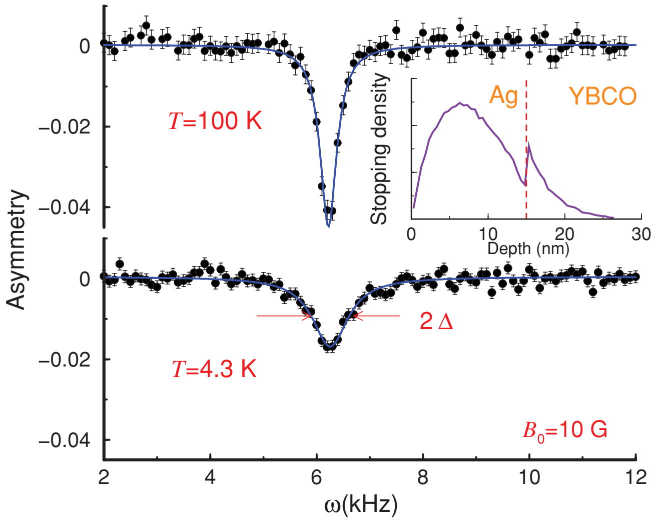

FIG. 1. (Color online) Typical β-NMR spectra taken by implanting 2 keV $^8\mathrm{Li}^+$ into Ag/YBCO(110), in an external field of $B_0 = 10$ G (FC) applied along the surface of the film. Solid lines are fits to a Lorentzian of HWHM Δ. Inset: Simulated implantation profile using TRIM.SP for $^8\mathrm{Li}^+$ of 2 keV in 15 nm of Ag on YBCO (Ref. 26). The $^8\mathrm{Li}^+$ stops at an average depth of 8 nm away from the Ag/YBCO interface.

The measurements presented here were carried out on (110)-, (103)-, and (001)-oriented YBCO films capped with 15 or 50 nm of Ag. The (110) film ($T_c = 86.7$ K) was grown by rf magnetron sputtering on a (110) $\mathrm{SrTiO_3}$ (STO) substrate measuring 8 × 6 mm. The (103) film ($T_c = 84$ K) was grown under similar conditions. Three (001) films were also studied, (i) one with $T_c = 88.7$ K grown on (001) STO under similar conditions as the (110), and (ii) two films ($T_c \sim 88.0$ K) grown by thermal coevaporation on 8 × 10 mm $\mathrm{LaAlO_3}$. The films are epitaxial and atomic force microscopy was used to characterize the surface roughness, which is in the range 6–12 nm. All samples were capped *ex situ* with 15 nm of Ag, except one of the last two, which was capped with 50 nm, by direct current (DC) sputtering 99.99% Ag at room temperature under an Ar pressure of 30 mtorr at a rate of 0.5 Å/s while rotating the sample to ensure uniformity. The $^8\mathrm{Li}^+$ implantation energy was varied so that the probe ions are implanted at average depths ranging from 8 to 43 nm. The inset of Fig. 1 shows the simulated stopping profile of 2 keV $^8\mathrm{Li}^+$ ions in 15 nm of Ag on YBCO using TRIM.SP.[26] Here the average probe-YBCO distance is ~8 nm. At 2 keV, about 20% of $^8\mathrm{Li}^+$ ions stop in the YBCO, yielding no associated NMR signal due to fast spin-lattice relaxation at low fields. To measure the NMR resonance in Ag, a small field is required, and our measurements were taken by field cooling (FC) in a small field B_0 or zero field cooling (ZFC). Residual magnetic fields were reduced to less than 30 mG normal to the surface when FC, or in all directions when ZFC. The samples were aligned parallel to the field with an accuracy of at least 0.5°.

III. RESULTS

Figure 1 shows two resonances at 100 and 4.3 K in Ag/YBCO(110). Above T_c, the resonances are all identical and show negligible differences in amplitude and linewidth, and are indistinguishable from those intrinsic to Ag. Below T_c, the resonance broadens, and therefore is reduced in amplitude. The broadening is symmetric, unlike the field distribution within the bulk of a superconductor in an ordered vortex lattice state.[27] Such a symmetric broadening is typical of a more disordered vortex distribution.[28–30] The HWHM, Δ, of a single Lorentzian fit to both (110) and (001) samples is plotted in Fig. 2. It is nearly T independent above T_c, consistent with the small nuclear dipolar broadening in Ag. It is the same in both samples and comparable to a control sample of Ag grown on an insulating STO substrate under similar conditions (open

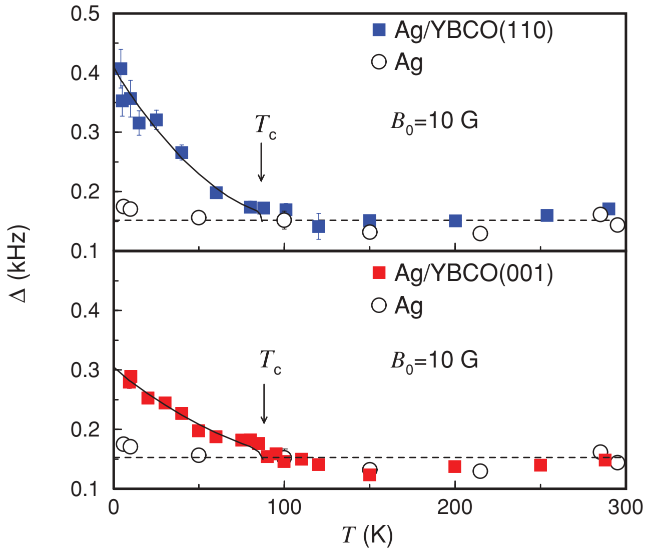

FIG. 2. (Color online) T dependence of the HWHM, Δ, of the resonance of 2 keV ^{8}Li$^+$ implanted into Ag/YBCO(110), Ag/YBCO(001), and Ag. The data on the Ag/YBCO(110) and Ag film were taken using FC, and ZFC for the Ag/YBCO(001). The widths were independent of FC or ZFC. The dashed lines represent the average Ag width Δ_0, the arrows point to the T_c of the YBCO films, and the solid lines are guide to the eye.

circles, Fig. 2). Below T_c, the resonance broadens, signaling the appearance of disordered static magnetic fields in the underlying YBCO.

Δ below T_c is slightly larger in Ag on the (110) film than on the (001) film. In an applied field of 10 G, the additional broadening, $\Delta - \Delta_0$, at $\approx$5 K is already very small, about 0.25 kHz for the (110) film, and 0.15 kHz for the (001) film. This broadening is clearly caused by the superconducting YBCO, since it is absent above T_c and in the Ag film without YBCO. It is, however, not accompanied by a resonance shift (see Fig. 1). The resonance frequency is constant from 300 to 5 K in all films, independent of FC or ZFC. This shows that there is no superconducting proximity effect in the Ag layer in which an induced Meissner shielding of the applied field leads to a diamagnetic resonance shift (e.g., as seen recently in Ag/Nb heterostructures).[31] The temperature-dependent broadening below T_c is not linked to TRSB, as the latter state is expected theoretically to appear at a second transition temperature $T_{c2} \ll T_c$,[3,12,13] consistent with some tunneling measurements.[18] In contrast, the broadening here has an onset close to T_c.

The line broadening versus the external magnetic field in the c-axis sample is displayed in Fig. 3. At 100 K, Δ is field independent, as expected. At 10 K (ZFC), Δ increases linearly with the applied field. This field-dependent broadening is attributed to inhomogeneous penetration of the applied field in the form of flux vortices. Penetration of vortices would not typically occur at these fields well below H_{c1},[32] especially since the demagnetization factor for the field parallel to the film is very small. However, at the interface the flux lines may penetrate more easily due to suppression of the d-wave order near twin or grain boundaries.[33,34] Moreover, surface roughness suppresses the Bean-Livingston surface barrier, and vortices may nucleate at fields $H \leqslant H_{c1}$.[35] Surface vortices have been observed in YBCO crystals in fields as small as 4 G applied parallel to the surface.[36] At such low fields, the vortex spacing d is of the order of few microns.[37] Outside the superconductor, the resulting field inhomogeneity leads to a depth-independent broadening, since $z \ll d$, consistent with our results (inset of Fig. 3).

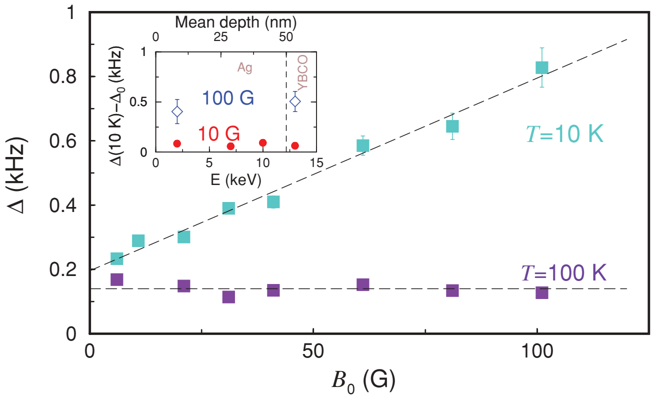

FIG. 3. (Color online) HWHM of the resonance in Ag/YBCO(001) at 10 K taken after ZFC. The dashed lines are linear fits. Inset: energy and depth dependence of the excess HWHM at 10 K [Δ(10 K) $- \Delta_0$] in the 50 nm Ag/YBCO(001) sample at applied fields 10 and 100 G. The resonance at all energies is due to the fraction of ^{8}Li$^+$ landing in Ag film.

IV. DISCUSSION

The T dependence of the linewidth in Fig. 2 is consistent with line broadening due to vortices beneath the surface since one would expect such vortex penetration close to T_c, and that it would increase as the temperature falls due to the decreasing magnetic penetration depth in the superconductor.[38,39] Field inhomogeneities are also expected from local variations of the shielding current density due to surface roughness as well as twin and grain boundaries.[40] This inhomogeneity will be enhanced by increasing the field or decreasing the temperature below T_c, due to a higher current density with pronounced local variations. This would lead to a temperature and field-dependent broadening, as observed in Figs. 2 and 3. It is possible that flux penetration and current density inhomogeneities near the surface could be related to the apparent dead layer seen in HTSC and other superconductors using low-energy μSR.[41,42] Further experiments on atomically flat surfaces may help elucidate the origin of the magnetic field inhomogeneities reported here. However, the field-dependent source of the line broadening is not central to the current study.

The main result of this study is the zero field extrapolation of the broadening at low temperature, which is an estimate of the TRSB fields. The broadening at 10 K extrapolates to $\Delta_{B=0} \approx 0.2$ kHz in (001) and (103), and 0.3 kHz in (110) (not shown). This $\Delta_{B=0}$ is marginally higher than the normal state broadening, $\Delta_0 \approx 0.15$ kHz. Thus, the net internal field in the superconducting state extrapolated to zero applied field, $\Delta_{B=0} - \Delta_0$, is less than 0.15 kHz (or $\sim$0.2 G) in all orientations. Part of the difference is due to the slight broadening of the Ag resonance upon cooling from 100 to

5 K, as seen in Fig. 2 (open circles). Thus, the additional broadening at zero field, $\Delta_{B=0} - \Delta_0$, is an estimate of the spontaneous magnetic field at the Ag/YBCO interface, and has an upper limit of 0.2 G. This extrapolated value is close to our experimental resolution determined by the Ag nuclear moments. These additional fields at low temperature are clearly much weaker than predicted by tunneling experiments in which much stronger spontaneous fields are predicted to cause the ZBCP splitting.[18]

V. CONCLUSION

In conclusion, we have conducted a depth-resolved β-NMR study of the field distribution near the interface of Ag and YBCO films. In all films we find additional broadening of the NMR resonance below T_c, signaling the appearance of disordered internal static fields in YBCO. We established an upper limit of 0.2 G for TRSB fields at low temperature. This rules out any straightforward interpretation based on the TRSB state that was suggested by tunneling measurements to be characterized by a much larger spontaneous magnetic field.[18,19] We have shown that such a putative state must be consistent with a small upper limit on the width of the magnetic field distribution in the adjacent Ag. We have also demonstrated that β-NMR can be used as a sensitive magnetic probe of spontaneous magnetic fields near an interface.

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