The role of individual defects on the magnetic screening of HTSC films

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Abstract

The magnetic flux penetration into thin films of high-temperature superconducting YBCO is visualized with high spatial resolution via x-ray microscopy. Therefore superconductors are coated with soft-magnetic CoFeB layers that reproduce the magnetic flux density distribution in an adjacent superconducting film and exhibit at the same time a large XMCD effect. For the first time we present scanning x-ray microscopy in the total electron yield mode using polarized x-rays providing simultaneously structural and magnetic information of the surface with high spatial resolution. Correlating the images of structural and magnetic information the role of individual defects on the magnetic screening capability of the superconductor can be identified.

1. Introduction

The penetration of magnetic flux into superconducting structures in form of quantized magnetic vortices is strongly dependent on the microstructural properties of the superconductor. Vortices feel strong pinning forces at defect structures inside the material where the superconducting properties vary [1–6]. Since the sum of all these pinning forces is directly related to the electric current carrying capability of superconductors these properties are highly interesting for many applications of superconducting materials [7, 8]. It is highly desirable to identify the characteristics of individual defect structures with respect to the flux pinning properties.

The interaction of vortices and defect structures is investigated along two particular routes. First, individual vortices are imaged in particularly prepared material structures such as cleaved single crystals or metallic films of high purity. In these model systems, motion or rearrangement under the influence of changing external fields can be visualized. This allows the description of the interaction between individual defects and vortices [9, 10]. An alternative way of investigation is provided by the visualization of magnetic flux penetration into ‘real’ superconducting structures with high critical current densities that are directly prepared for application. In this case the common magnetic visualization techniques are [11] magneto-optical imaging [12–16], scanning Hall probe microscopy [17–19], scanning SQUID microscopy [20–22], transmission electron microscopy (TEM) [23] or magnetic force microscopy (MFM) [24, 25]. Recently, nitrogen-vacancy imaging has successfully been used for magnetic vortex imaging [26]. All techniques come with their characteristic advantages and disadvantages [27] in terms of spatial resolution, magnetic sensitivity, noise, operation temperature and sample preparation procedures. Among all of these methods only TEM and MFM provide simultaneously magnetic and structural information. However, in case of force microscopy measurements it is difficult to separate topographic and magnetic contributions in the measured signal separation cannot be done free of assumptions. Magnetic electron microscopy (Lorentz microscopy) provides highest spatial resolution of nanometers or below but requires a measurement in transmission geometry. In this case sample preparation significantly modifies the microstructure during preparation.

In this work scanning magnetic x-ray microscopy is used to image magnetic flux penetration into as prepared superconducting structures. The huge advantage of the x-ray microscopy method presented here is the
high spatial resolution of about 100 nm [27] which exceeds the resolution of the methods mentioned above apart from MFM and electron microscopy. In the context of the methods mentioned above x-ray microscopy combines high spatial resolution and reasonable magnetic sensitivity with the ability to investigate applicable superconductors in a wide temperature region. In particular, topographical and magnetic information can be obtained at the same time which allows direct correlation. This feature is in such a direct manner unique among the discussed methods.

The small wavelength of x-rays is a reasonable prerequisite to address submicron resolution with an imaging technique using electromagnetic radiation. To access magnetic information in an element specific manner it is necessary to employ circular polarized x-rays offering x-ray magnetic circular dichroism (XMCD). Since in most superconductors atoms exhibiting a large XMCD effect are absent we deposit a soft-magnetic layer of amorphous CoFeB on top of the superconductor [28]. The 3d transition metals Fe and Co show a strong XMCD effect [29]. When applying an external magnetic field at low temperatures to the superconductor-ferromagnet sample magnetic flux penetrates into the superconductor leading to an inhomogeneous magnetic stray field distribution. The magnetization of the soft-magnetic sensor layer is locally reoriented leading to a spatially varying XMCD effect. This can be used to capture high-resolution images of the magnetic stray field distribution and thus of the magnetic flux penetration into the superconductor [30]. Due to the high Curie temperature of the sensor layer the magnetic domain structure of the sensor layer is stable and allowing measurements up to room temperature [28].

In this work we use the magnetic x-ray microscopy method to correlate structural and magnetic information provided by the analysis of superconductor-ferromagnet bilayers under the illumination of polarized x-rays. The analyzed samples consist of bilayers of optimally doped YBa2Cu3O7–δ (YBCO) grown by pulsed laser deposition and amorphous Co82Fe18B10 prepared in an ion beam sputtering process. The evolving surface topography is dominated by the growth islands formed during deposition of the YBCO film (section 2). The magnetic properties of the bilayer are investigated and discussed in terms of which material dominates the magnetic behavior (section 3). The correlation of the surface topography and the magnetic domains via scanning x-ray microscopy (SXM) is shown in section 4 and discussed in section 5.

2. Sample: surface topography

The sample composition of the superconductor and the ferromagnetic sensor layer is sketched in figure 1(a). It consists of a thin film of optimally doped YBCO which is deposited on a single crystalline SrTiO3 (100) substrate by pulsed laser deposition at a temperature of \( T = 750 \, ^\circ C \). The layer thickness is 275 nm. The adjacent amorphous CoFeB layer is deposited by ion beam sputtering at room temperature. Finally, the layers are capped with a thin Al protection layer (not shown in the sketch). The CoFeB layer is 30 nm, the Al layer 2 nm thick. As sketched in figure 1(a), the surface topography of the Stranski-Krastanov-grown YBCO film is adapted by the amorphous CoFeB layer.

An atomic force microscopy (AFM) image shown in figure 1(b) reveals the surface profile of the complete sample stack. Owing to the lattice mismatch of YBCO and STO the superconducting layer exhibits typical Stranski-Krastanov growth that can be identified in the surface topography measurement [31]. The growth islands with typical diameters of several hundreds of nanometers can be identified as light brown structures. Dark brown areas are visible in figure 1(b) which refer to the coalescence regions in between the islands. These
exhibit a smaller surface elevation and a high density of defects. In addition, bright, nearly circular-shaped structures are seen in the AFM representation. These refer to a-axis oriented outgrowths [32, 33].

Figure 1 (c) shows an image obtained by XA contrast at the Co L3-edge of a similar surface area of the same sample. In analogy to figure 1 (b) the growth islands (gray), the coalescence regions (black) and the outgrowths (white) can be identified. The image is obtained using the total electron yield current (TEY), which is a measure for the local absorption in the material. Hence, the elevated structures exhibiting a higher absorption cross section appear brighter. This particular technique is discussed in more detail in combination with figure 3.

Figures 1 (b) and (c) show the surface topography on the same scale. Growth islands and coalescence regions lead to similar structures in both representations [34]. Differences occur when comparing the visual size of the outgrowths, which is due to the fact, that the TEY current enhances the structural edges only. However, the fact that the density of the a-axis oriented objects is nearly the same in both images (we find 1900 outgrowths per mm² for the AFM image and 1700 outgrowths per mm² for the XA image) shows the equivalence of both measurements.

3. Magnetic moment and magnetic flux density

In a bilayer of a superconductor and a ferromagnet the magnetic flux density of the former and the magnetization of the latter interact with each other [28, 35, 36]. In order to separate the magnetic signals from both materials we perform hysteresis measurements using a Quantum Design SQUID magnetometer MPMS-XL at different temperatures. Figure 2 shows magnetic moment hysteresis loops of the bilayer below and above the critical temperature $T_c$ of the superconductor YBCO. Below $T_c$ the magnetic signal is dominated by the signal of the superconductor: figure 2(a) pictures a typical hysteresis loop of a strong pinning YBCO thin film at a temperature of $T = 10$ K. The measurement direction and the orientation of the external field are marked in the sketch. In figure 2(a) the measurement geometry is out-of-plane (c-axis direction) with the external field parallel to the measurement direction. In case of the fully penetrated state the magnetic moment of a superconductor can be converted into the critical current density.

Figures 2(b) and (c) picture the same bilayer, now above $T_c$, in out-of-plane (b) and in-plane (c) geometry. Both hysteresis loops show a typical soft-ferromagnetic behavior with the easy axis in-plane. The saturation magnetic moment $M_s$ is identical in both geometries. The magnetic signal of the superconductor has vanished.

Figure 2. SQUID magnetometer magnetization curves of the double layer YBCO/CoFeB below $T_c$ ($T = 10$ K) (a), and above $T_c$ (b) and (c). (d) shows the positive data from (a) to (c) on a logarithmic scale. The magnetic signal from the superconductor is three orders of magnitude higher than from the CoFeB.
Figure 2 (d) combines the data of the positive quadrants of all three hysteresis loops in one diagram. The magnetic moment is depicted on a logarithmic scale in order to show the difference in the absolute values of the magnetic moments. Even if we consider that the superconductor has approximately 9 times the volume of the ferromagnet, the magnetic moment of the YBCO film is three orders of magnitude higher than the magnetic moment of the soft-magnetic CoFeB layer. We have shown that there is hardly any negative influence between the superconductor and the ferromagnet [28].

These results show that at low temperatures the magnetic properties of the bilayer can be described as a superconductor with a small magnetic perturbation. This is a prerequisite for the application of the CoFeB layer as probe for the flux penetration properties of the YBCO film.

4. X-ray microscopy results

We perform magnetic x-ray microscopy based on the XMCD effect in order to visualize the magnetic flux density in the YBCO layer. An indirect measurement via the magnetization distribution in the sensor layer CoFeB is performed. For magnetic contrast the x-ray energy is tuned to the Co L3-edge and the sample is scanned by a focused x-ray beam with circular polarization. The difference between positive and negative circular polarization reveals the XMCD effect which is a direct measure for the local magnetization [37, 38]. The average of the two polarizations results in the pure XA. This is demonstrated by two images taken with the scanning x-ray microscope MAXYMUS at the undulator beamline UE46-PGM2 at the synchrotron BESSY II in Berlin, Germany. Figures 3 (a) and (b) show images taken with positive and negative circular polarization. Their average is depicted in figure 3 (c), the difference in figure 3 (d).

The images in figure 3 show a 10 μm × 20 μm area of the superconductor/ferromagnet bilayer. The edge of the sample is located on the left side of the image. The black region is outside the sample.

The measurement mode used for these nontransparent samples is the TEY mode [39]. It is a measure of the current created by emitted secondary electrons following the resonant excitation process into the 3d states. It probes only the surface near layers of approximately the electron emission length of 2–5 nm [39]. The TEY current is proportional to the local absorption cross section of the material which in turn is highly dependent on the surface topography of the sample. Peaks in the sample surface reveal a higher absorption and therefore a higher current than flat surfaces or even dips or holes in the surface. This is the dominant effect in surface sensitive TEY measurements and it is independent of the polarization direction. Therefore the raw images shown in figures 3 (a) and (b) show the surface topography of the sample. Dark features refer to low lying areas such as the coalescence regions between growth islands and white structures refer to peaks such as outgrowths. Averaging the two raw images enhances the surface contrast as shown in figure 3 (c) which is comparable to the AFM image in figure 1.
The transition probability from the 2p to the 3d states depends on the polarization of the circular polarized light. The resulting contrast is opposed in the two raw images. It can be enhanced by taking the difference between the two images. In the resulting image the surface structure cancels out and the pure magnetic contrast remains. The result of the difference is shown in figure 3(d).

In figure 3(d) the magnetic domains represent the magnetic flux density of the superconductor. The stray field of the superconductor magnetizes the CoFeB sensor layer and imprints a magnetic domain structure, which is then measured. The superconductor has been prepared at 12 K by applying and subsequently removing a perpendicular external field of 8.8 mT. Calculations from magneto-optical measurement show that the magnetic stray field created by the YBCO film has an in-plane component of 200 Oe which is an order of magnitude larger than the coercivity of the CoFeB layer (figure 2(c)). As a consequence the magnetic flux has partially penetrated the superconductor starting from the rim of the sample. The magnetic flux is represented by the dark domains. Bright domains refer to areas that have not been penetrated at this particular external field. The measurement has been performed at room temperature. Since the remanent state of the sensor layer is close to its saturation and the temperature dependence is negligible up to 300 K, the magnetic domains at room temperature resemble those of low temperatures [28]. This has been shown by magneto-optical imaging on larger length scales and is extrapolated to the submicron scale which has not been accessible at very low temperatures yet.

Figures 3(c) and (d) show the x-ray absorption (XA) image and the XMCD image constructed by averaging and taking the difference of two raw images. In figure 4 these two images are overlayed in order to correlate the surface structure and the magnetic domains.

Figures 4(a) and (g) show these two images: the XMCD image and the XA signal. From the XMCD image the domain walls are extracted and marked by a red line in figure 4(b). The contrast of the XA image is altered to enhance the bright structures in figure 4(e) and the dark structures in figure 4(f). Both images are now overlayed with the red line marking the domain walls. This is depicted in figures 4(c) and (d).

In figure 4(c) the outgrowths (white) are visible mostly in the middle of the domains, not in the vicinity of a domain wall. In contrast, figure 4(d) shows the domains walls spanning the shortest distance between the coalescence regions (black). In order to investigate this further, figure 5 shows a magnification of figure 4(d).

In figure 5(a) the overlay between the XA image and the domain walls is pictured in color. The growth islands are shown in red, the coalescence regions in black and the outgrowths in yellow. The domain walls are marked in white. Figure 5(b) depicts an enlargement at the indicated region. The domain walls follow the coalescence regions. In between the coalescence regions the domain walls span across the growth islands along the shortest distance.
distance. This shows that the growth islands themselves do not exhibit a significant pinning force. To show the correlation in more detail two vertical profile plots are taken and marked with arrows in the image of figure 5(a). The profiles show the surface topography and the white lines mark the spot where the domain walls are crossed. In most cases (16 of 18) the domain walls lie inside or directly aside of a coalescence region (marked with a check).

We conclude that the domain walls follow the coalescence regions of the superconductor. Since the domain walls of the sensor layer represent the flux front of the superconductor the flux front is pinned by the defects of the defect-rich coalescence regions. It is expected that the coalescence regions offer a higher pinning force due to the reduced layer thickness and high defect density. This therefore favors the progression of the flux front along these coalescence regions. For the indirect x-ray microscopy method via a sensor layer this is strong evidence that the magnetic domains measured at room temperature in the sensor layer resemble the position of the low temperature flux front in the superconductor.

5. Conclusion

Magnetic flux penetration into thin superconducting films is investigated by SXM in the TEY mode. This recently developed method allows the characterization of the magnetic flux density distribution and the surface topography at the same time at exactly the same spot on a length scale of a few hundred nanometers. These properties are unique among the magnetic characterization techniques providing spatially resolved data. The correlation of the flux density distribution in the superconductor with its defect structure is analyzed without using any assumptions or models. In particular the role of peak a-axis outgrowths and dislocation-rich coalescence regions of growth islands are determined with respect to the interaction with the penetrating magnetic flux front. We can show that misoriented outgrowths do not influence the position of the magnetic flux front at all. The flux front on the other hand spans the shortest distance between coalescence regions. We conclude that the flux front of the superconductor is mainly pinned at these thin, defect-rich coalescence regions. For the size of the pinning defects and the domain walls the method of SXM is a powerful tool since it is independent of any special sample preparation or surface smoothness. It is possible to directly investigate real, strong pinning systems.

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