



Anisotropy and Crystallite Misalignment in Textured Superconductors

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Abstract

A misalignment of anisotropic crystallites causes small values of anisotropy and decreases the critical current density of textured polycrystalline superconductors. To relate the crystallite misalignment and out-plane anisotropy, the magnetic properties of the textured Bi2223 polycrystalline superconductor were investigated. A distribution of orientation angles of crystallites was determined using different data: scanning electron microscopy images and hysteresis magnetization loops when an external magnetic field was applied at different angles with respect to the texturing plane of the sample. It was demonstrated that the standard deviation of the distribution and the magnetic disorder angle of crystallites in textured samples can be determined from the magnetization data in perpendicular directions. These data may be either the irreversible magnetization measured for two different orientations of the sample or the simultaneously measured magnetization projections parallel and perpendicular to the magnetic field.

Keywords Critical current · Anisotropy · BSCCO · Texture · Distribution function · Magnetization · Hysteresis loop

1 Introduction

The outstanding values of the critical current density in the ab plane $j_{c,ab}$ and the high critical temperatures could bring Bi₂Sr₂CaCu₂O_{8+x} (Bi2212) and Bi₂Sr₂Ca₂Cu₃O_{10+x} (Bi2223) at the top of superconductor applications. However, the strong anisotropy coefficient $\gamma \lesssim 100$ of these materials [1] hinders their rise. The overall critical current density j_c of polycrystalline superconductors is very sensitive to arrangement of crystallites [2]. An alignment is crucially important for textured superconductors and tapes [3–8]. The crystallite misalignment strongly decreases j_c as well as γ . Bi2212 and Bi2223 textured ceramics usually have low γ and low j_c values (e.g., $\gamma \approx 1.7$ – 2.5 and $j_c \approx 0.1$ – 1 kA/cm² at $T = 77.4$ K [9–13]) as compared with single crystals and wires. Optimization of the current carrying capacity in textured superconductors requires information about a

distribution of crystallite orientation angles and reasons of the misalignment.

In anisotropic type-II superconductors, the vortex lattice and magnetization depend on an angle between the external magnetic field and the crystal principal axes [14–17]. The magnetization of textured polycrystalline sample is resulted from collective response of the crowd of crystallites. So, the texture in these samples is essential. The degree of texture is estimated from X-ray diffraction data (θ -2 θ and ω -scans) [7, 18, 19]. In recent work [13], the integral magnetic method was suggested. The magnetic misalignment angle θ^* was introduced to characterize texture in the Bi2223 superconductor. This angle θ^* equals an averaged value of the modulus of crystallite deviation from the texturing plane. In the presented work, new ways are paved to determine the distribution of the crystallite orientation angles and the corresponding magnetic misalignment angle θ^* in the textured superconductors.

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2 Experimental Methods

The textured Bi2223 + Ag ceramics were produced by two step routes [9]. At the first stage, the polycrystalline ceramics were synthesized from Bi₂O₃, SrCO₃, PbO, CuO, CaCO, and Ag powder by the solid-state synthesis. Porosity of the synthesized material is about 60% [20]. At the second stage, the texture

was created: the porous material was impregnated by ethanol, pressed up to 500 MPa, and annealed at 830 °C for 50 h.

Scanning electron microscopy (SEM) was carried out using a Hitachi TM4000Plus microscope. Magnetization was measured using a Lakeshore VSM 8604 vibrating sample magnetometer. Measurements were carried out at 77.4 K for different orientations of the rotated sample relative to the external magnetic field H . The sample had a parallelepiped form and sizes of $10 \times 1 \times 1 \text{ mm}^3$. The wider sides of the sample corresponded to the texturing plane. A schematic for magnetization measurements is shown on Fig. 1. Two magnetization projections were measured: M_x along the H direction (it is a general magnetization) and M_y perpendicular to H .

3 Experimental Results

SEM images of the sample side, which is perpendicular to the texturing plane, are shown on Fig. 2a, b. As seen, light particles of Ag are surrounded by darker crystallites of Bi2223. The diameter of the silver particles is about 20 μm . The Bi2223 crystallites have a flake-like form with the sizes about $10 \mu\text{m} \times 10 \mu\text{m} \times 1 \mu\text{m}$. The most of crystallites are oriented along the texturing plane. However, some of the crystallites are tilted because they go around the silver particles. We measured the values of the tilt θ of each crystallites on four different SEM images. The obtained distribution of crystallite orientation angles is presented on Fig. 2c. The distribution has a characteristic bell-like form.

The angle dependencies of the magnetization projections at $H=5 \text{ kOe}$, which is much higher than the irreversibility field, are shown on Fig. 3a (α changes from -180° to $+180^\circ$ at this figure). The period of these dependencies is equal to

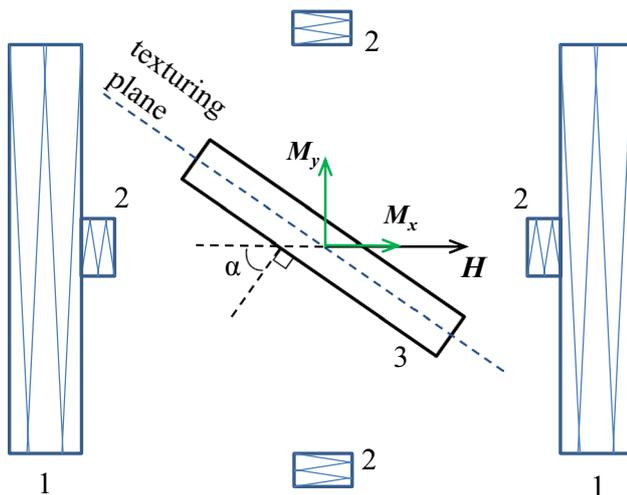


Fig. 1 Schematic for magnetization measurements. The numbers mark the magnet coils (1), the measuring coils (2), and the sample (3)

180° . It can be seen, the positions of $M_x=0$ do not correspond to the extremum positions of M_y .

Figure 3b, c, and d show magnetization hysteresis loops (the M_x and M_y projections) for some values of the angle α between the texturing plane and H . It is seen, the M_y loops are inverted at the angle range $0^\circ < \alpha < +90^\circ$.

Furthermore, we focus on the dependence of the irreversible magnetization M_{irr} on α . The irreversible magnetization is determined from the magnetization hysteresis loops as $M_{\text{irr}}(H) = |M_{\downarrow}(H) - M_{\uparrow}(H)|/2$, where $M_{\uparrow}(H)$ and $M_{\downarrow}(H)$ are the magnetization curves at increasing and decreasing magnetic fields correspondingly. The $M_{\text{irr}}(H)$ dependencies for some values of α are plotted on Fig. 4a.

We designate $M_{\text{irr}}(H=0)$ at some α as $M_{\text{irr},x}(\alpha)$ and $M_{\text{irr},y}(\alpha)$ for the M_x and M_y projections correspondingly. The experimental loops for different α demonstrate that $M_{\text{irr},x}$ decreases in about 2.5 times as α changes from 0° to $+90^\circ$ (or from 0° to -90°). The value of $M_{\text{irr},y}$ is almost zero at $\alpha=0$ and $\alpha=\pm 90^\circ$. The maximal values of $M_{\text{irr},y}(\alpha)$ are achieved at $\alpha=-70^\circ$ and $\alpha=+70^\circ$, for these angles $M_{\text{irr},y} \approx M_{\text{irr},x} \approx 0.6M_{\text{irr},x}(0^\circ)$.

It is convenient to trace three normalized magnetizations $k_{xx0}(\alpha) = M_{\text{irr},x}(\alpha)/M_{\text{irr},x}(0)$, $k_{yx0}(\alpha) = M_{\text{irr},y}(\alpha)/M_{\text{irr},x}(0)$, and $k_{yx}(\alpha) = M_{\text{irr},y}(\alpha)/M_{\text{irr},x}(\alpha) = k_{yx0}(\alpha)/k_{xx0}(\alpha)$. These values obtained from the experimental magnetization hysteresis loops are shown on Fig. 4b–d. A model describing these dependencies is considered at next section.

4 Model

Let us consider firstly an anisotropic superconducting plate with strong anisotropy, such that any current circulations along the c axis of the plate (perpendicular to the wider surface that is the ab plane) are negligible. The external magnetic field H induces currents in the ab plane of the plate, and the corresponding magnetization M^* heads along the c axis. The related projection of H on the c axis is $H^* = H \cos \alpha$, here α is the angle between H and c . With the schematic presented above (Fig. 1), two projections of M^* are measured:

$$\begin{aligned} M_x(H) &= M^*(H^*) \cos \alpha, \\ M_y(H) &= M^*(H^*) \sin \alpha \end{aligned} \quad (1)$$

The minus in the expression of M_y takes into account that M_y directs along the y axis for $-90^\circ < \alpha < 0^\circ$ and opposite for $0^\circ < \alpha < +90^\circ$ (Fig. 1). As it follows from Eq. (1):

$$k_{xx0}(\alpha) = |\cos \alpha|, k_{yx0}(\alpha) = |\sin \alpha|, k_{yx}(\alpha) = |\tg \alpha|. \quad (2)$$

The last equality means that $k_{yx}(\alpha) = 1$ and $M_{\text{irr},y} = M_{\text{irr},x}$ at $\alpha = \pm 45^\circ$. However, the data obtained from the magnetization hysteresis loops provide another value for the sample:

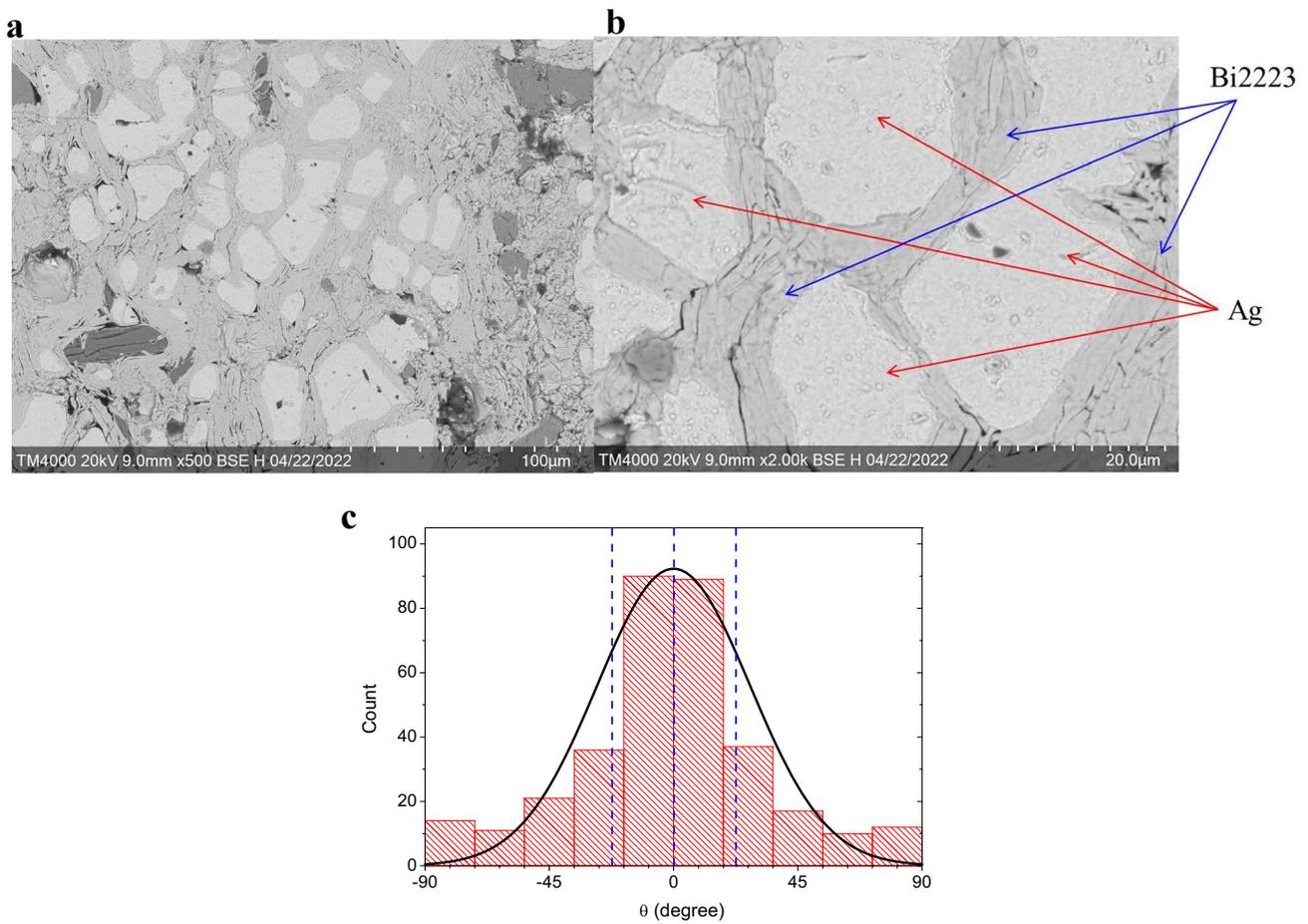


Fig. 2 Crystallites in the textured sample. **a, b** SEM images of the sample side perpendicular to the texturing plane. **c** Distribution of crystallite orientation angles. The bar chart is data obtained from

SEM images. The normal distribution curve is computed (Eq. (3)) for $\sigma=28^\circ$. Vertical dash lines separate the quartiles

$k_{yx}(\pm 45^\circ)=0.73 \pm 0.03$. Indeed, the data points for $k_{yx0}(\alpha)$ and $k_{yx}(\alpha)$ are described by Eq. (2) only for $|\alpha| < 30^\circ$ (Fig. 3c, d). The reason of this divergence is supported to be the crystallite misalignment.

Any crystallite in the textured sample is characterized by the orientation angle θ , which is the angle between

where σ is the standard deviation, and N is a normalizing coefficient. The orientation of any crystallite in the rotated sample is given by the angle $\alpha + \theta$. It should be noted that the orientation $\alpha + \theta$ is equivalent to $\alpha + \theta \pm 180^\circ$. Then, the magnetization projections of the textured sample are determined by

$$M_x(H) = \frac{1}{\pi} \int_{-90^\circ}^{+90^\circ} M^*(H^*) \cos(\alpha + \theta) f(\theta, \sigma) S(\alpha + \theta) d\theta, M_y(H) = -\frac{1}{\pi} \int_{-90^\circ}^{+90^\circ} M^*(H^*) \sin(\alpha + \theta) f(\theta, \sigma) S(\alpha + \theta) d\theta \quad (4)$$

the ab plane of the crystallite and the texturing plane (see Fig. 5), $-90^\circ \leq \theta \leq 90^\circ$. Let us assume that all the crystallites have the same $M^*(H^*)$ dependence, and the distribution of orientation angles is described by the normal distribution function:

$$f(\theta, \sigma) = N/\sigma \exp(-0.5(\theta/\sigma)^2) \quad (3)$$

where $S(\varphi)$ is a function such that $S(\varphi) = 1$ for $|\varphi| \leq 90^\circ$ and $S(\varphi) = -1$ for $|\varphi| > 90^\circ$. This function is needed to account for all upturned crystallites. The crystallites with any value of $\alpha + \theta$ have the same sign of their contribution to M_x . At the same time, the signs of the contribution to M_y are opposite for the crystallites with $\alpha + \theta < 0$ and $\alpha + \theta > 0$ (see Fig. 5).

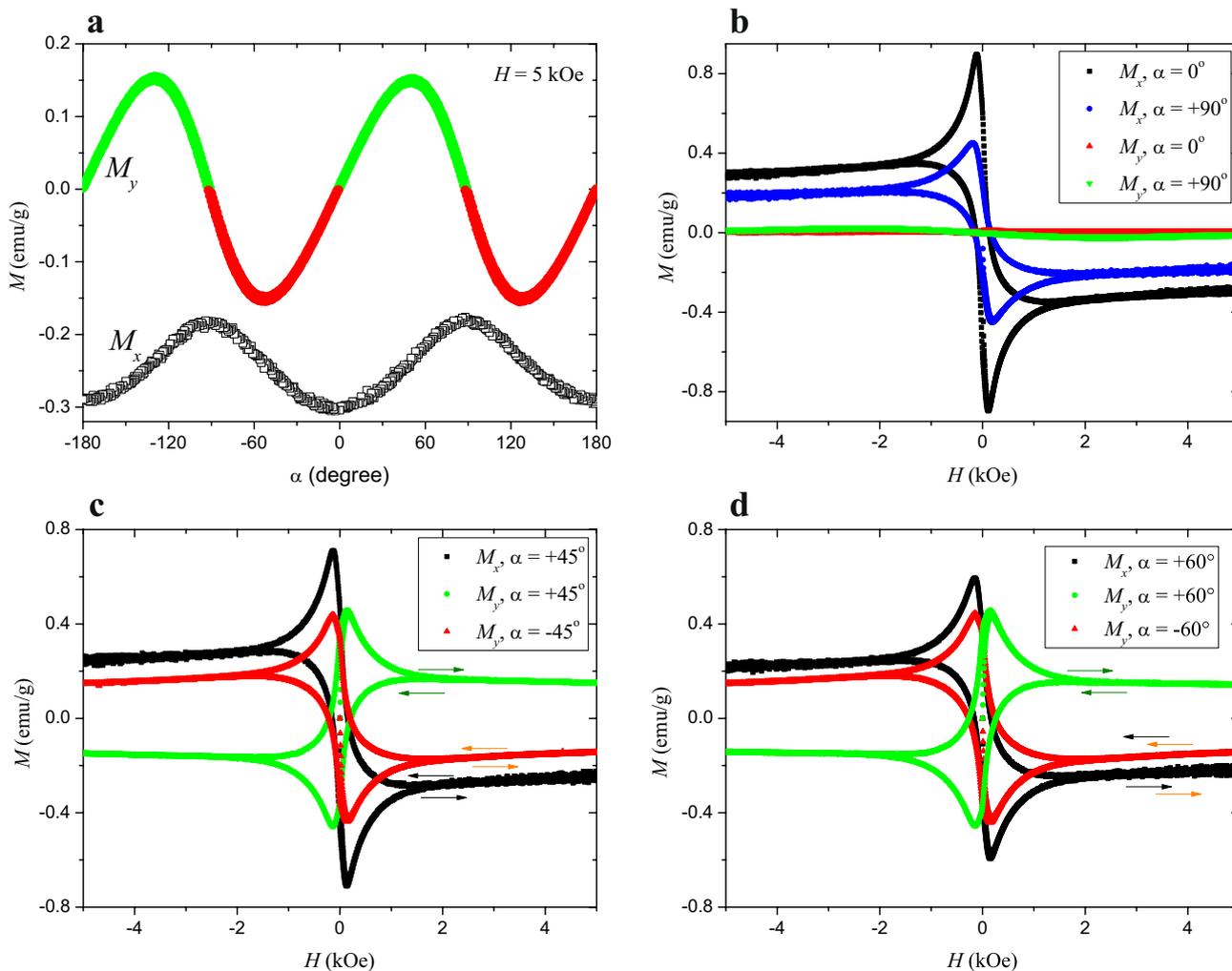


Fig. 3 Experimental magnetization isothermal curves at $T=77.4$ K. **a** Angle dependencies of magnetization projections at $H=5$ kOe. Magnetization hysteresis loops for M_x and M_y projections **b** $\alpha=0^\circ$

and $+90^\circ$, **c** $\alpha = -45^\circ$ and $+45^\circ$, **d** $\alpha = -60^\circ$ and $+60^\circ$. The arrows mark directions of the change of H

The value of M^* is independent of α only at $H=0$. It allows us to express the k_{xx0} , k_{yx0} , and k_{yx} :

which were calculated using Eq. (2), correspond to $\sigma=0^\circ$. The best agreement between the data points and computed

$$\begin{aligned}
 k_{xx0} &= \left| \frac{\int_{-90^\circ}^{+90^\circ} \cos(\alpha + \theta) f(\theta, \sigma) S(\alpha + \theta) d\theta}{\int_{-90^\circ}^{+90^\circ} \cos(\theta) f(\theta, \sigma) d\theta} \right|, \\
 k_{yx0} &= \left| \frac{\int_{-90^\circ}^{+90^\circ} \sin(\alpha + \theta) f(\theta, \sigma) S(\alpha + \theta) d\theta}{\int_{-90^\circ}^{+90^\circ} \cos(\alpha) f(\theta, \sigma) d\theta} \right|, \\
 k_{yx} &= \left| \frac{\int_{-90^\circ}^{+90^\circ} \sin(\alpha + \theta) f(\theta, \sigma) S(\alpha + \theta) d\theta}{\int_{-90^\circ}^{+90^\circ} \cos(\alpha + \theta) f(\theta, \sigma) S(\alpha + \theta) d\theta} \right|.
 \end{aligned}
 \tag{5}$$

5 Discussion

We used Eq. (5) to fit the normalized magnetizations $k_{xx0}(\alpha)$, $k_{yx0}(\alpha)$, and $k_{yx}(\alpha)$ on Fig. 4b–d. The single fitting parameter is used to be σ . The dash lines on Fig. 4b–d,

curves (solid lines) was achieved for $\sigma=28^\circ$. It appears that maxima of $k_{xx0}(\alpha)$ and $k_{yx}(\alpha)$ occurs at $|\alpha|>45^\circ$. Also, the normal distribution of Eq. (3) with $\sigma=28^\circ$ describes successfully the bar chart obtained from SEM images (Fig. 2c).

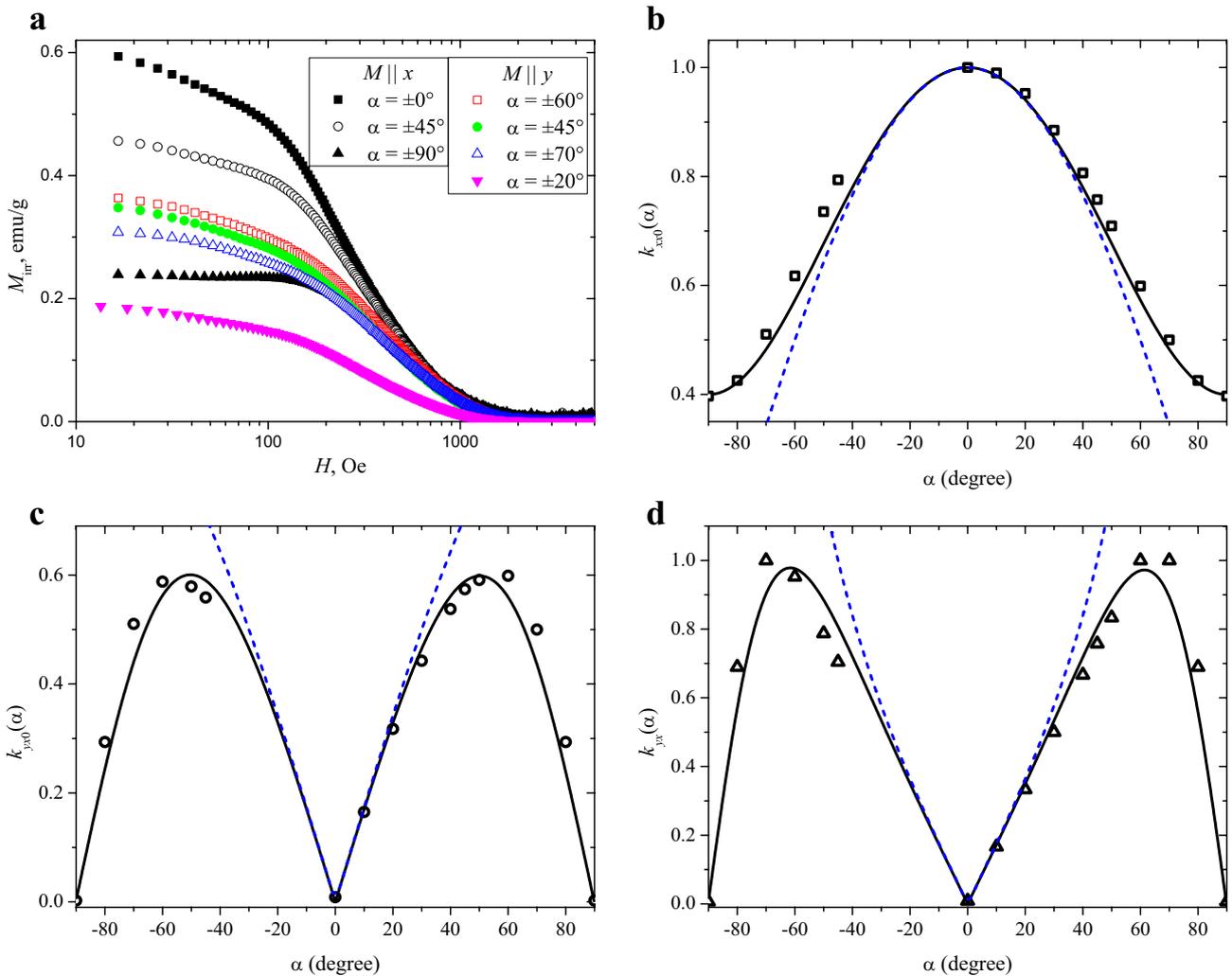


Fig. 4 The irreversible magnetization. **a** The dependence of M_{irr} on the magnetic field H (The axis H is in a logarithmic scale). The normalized values of irreversible magnetization: **b** $k_{xx0} = M_{irr,x}(\alpha)/M_{irr,x}(0)$, **c** $k_{yx0} = M_{irr,y}(\alpha)/M_{irr,x}(0)$, and **d** $k_{yx} = M_{irr,y}(\alpha)/M_{irr,x}(\alpha)$. Symbols are

obtained from experimental magnetization hysteresis loops, dash lines were calculated using Eq. (2). Solid lines were calculated using Eq. (5)

Discrepancy between the data points and the curves for the well-ordered case ($\sigma = 0^\circ$, dashed lines on Fig. 4b, c, d) may be used to estimate the value of σ . It is supported that the value of $k_{xx0}(90^\circ)$ is most convenient for this estimation. It should be noted that the anisotropy coefficient γ , which is used in previous works about the related textured Bi2223 [10, 12, 13], corresponds to $1/k_{xx0}(90^\circ)$. Figure 6 shows the dependence of $k_{xx0}(90^\circ)$ on σ (dash line) that was computed using Eq. (5). The arrows demonstrate the determination of σ for the considered sample ($k_{xx0}(90^\circ) = 0.4$).

The magnetization measurements for two orientations ($\alpha = 0^\circ$ and $\alpha = 90^\circ$) are required to obtain the values of $k_{xx0}(90^\circ)$, γ , and σ [6]. The same result can be attained from $k_{yx}(\alpha)$ using only one orientation $45^\circ \leq |\alpha| < 90^\circ$. An example

of such estimation for $k_{yx}(45^\circ) = 0.73$ is presented on Fig. 6, where the dependence of $k_{yx}(45^\circ)$ on σ was computed using Eq. (5) (solid line). As it is seen on Fig. 6, the values of σ estimated from $k_{xx0}(90^\circ)$ and $k_{yx}(45^\circ)$ coincide with the fitting result $\sigma = 28^\circ$.

The other parameter, the magnetic misalignment angle θ^* , was used to characterize anisotropy in previous works [13, 21]. As it was derived in [13], the value of θ^* is determined from the experimental magnetization loops as $\theta^* = \text{arccot}(M_{irr,x}(0^\circ)/M_{irr,x}(90^\circ)) = \text{arctg}(k_{xx0}(90^\circ))$. We suppose that θ^* corresponds to the boundaries between the quartiles of the orientation angle distribution. Providing $\sigma = 28^\circ$ in Eq. (3), one obtains $\theta^* = 21.5^\circ$ (see left and right vertical dash lines on Fig. 2c). The similar value of θ^* was early estimated for the texture Bi2223 ceramics [13].

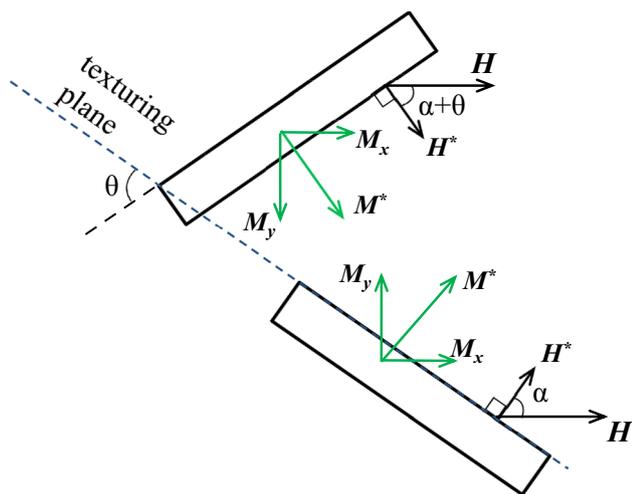


Fig. 5 Anisotropic superconducting crystallites in magnetic fields. The upper crystallite is rotated at some angle $\theta > 0$ relative to the texturing plane. The lower crystallite has $\theta = 0$

In paper [19], the Lotgering factor (LF) as an index of crystallographic orientation was correlated with σ . The value of $\sigma = 28^\circ$ corresponds LF about 0.6. However, the higher value of LF ≈ 0.98 obtained from X-ray diffraction data was reported earlier for the same material [9]. We claim that this value was overrated because just a surface of the textured sample was inspected. It is probably that the texture goes down in the sample inner. It may be suspected that the technique [9], which was used to obtain the textured samples, does not align well crystallites into the whole sample. Really, the SEM images from the inner (Fig. 2a, b) demonstrate a non-ideal arrangement of the crystallites.

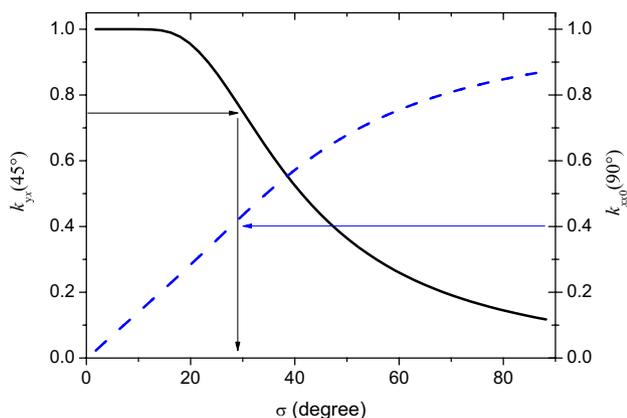


Fig. 6 The normalized irreversible magnetization for different values of the standard deviation. The values of $k_{xz0}(90^\circ)$ (dash line) and $k_{yx}(45^\circ)$ (solid line) are computed using Eq. (5). The arrows demonstrate the determination of σ for the investigated sample

6 Conclusion

The anisotropic magnetization of the textured polycrystalline Bi2223 superconductor was investigated. The magnetization projections parallel and perpendicular to the magnetic field were simultaneously measured. The low value of anisotropy ($\gamma = 2.5$) was confirmed to be the result of the crystallite misalignment. It was established that the distribution of the crystallite misalignment angles can be obtained from microscopic images and from magnetization data for different orientations of the sample and the external magnetic field. Assuming the normal distribution of the crystallite misalignment angles, the standard deviation can be estimated from the magnetization data for the parallel ($\alpha = 90^\circ$) and perpendicular ($\alpha = 0^\circ$) orientations or from the M_x and M_y projections for the single orientation (preferably $\alpha = 45^\circ$).

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Data Availability The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Conflict of Interest The authors declare no competing interests.

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