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RAPID COMMUNICATION

Direct measurement of the magnetic penetration depth by magnetic force microscopy

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Abstract

We present an experimental approach using magnetic force microscopy for measurements of the absolute value of the magnetic penetration depth λ in superconductors. λ is obtained in a simple and robust way without introducing any tip modeling procedure via direct comparison of the Meissner response curves for a material of interest to those measured for a reference sample. Using a well-characterized Nb film as a reference, we determine the absolute value of λ in a Ba(Fe_{0.92}Co_{0.08})₂As₂ single crystal and a MgB₂ thin film through a comparative experiment. Our apparatus features simultaneous loading of multiple samples, and allows straightforward measurement of the absolute value of λ in superconducting thin film or single-crystal samples.

(Some figures may appear in colour only in the online journal)

1. Introduction

The superconducting coherence length (ξ), magnetic penetration depth (λ), and their anisotropy are fundamental parameters that characterize superconducting materials [1–6]. A number of important properties, such as superconducting critical fields and superconducting fluctuations that affect vortex dynamics, can be obtained if the parameters above are known [1, 6]. The value of ξ , which depends on the Fermi velocity and the condensation energy of the superconducting state, can be estimated from the upper critical field (H_{c2}) using the Ginzburg–Landau theory. λ is related to the density of superconducting electrons [1], and, in contrast to ξ , precise determination of its absolute value is notoriously difficult [1, 2] due to demagnetizing effects, topography-related surface barrier, and inhomogeneity of the

sample. Typically λ is calculated by indirect methods. Several experimental techniques such as tunnel diode oscillator (TDO) [7], temperature dependence of the flux expulsion in the Meissner state [8] or of the reversible magnetization in the mixed state using superconducting quantum interference device (SQUID) magnetometers [9], mutual inductance [10], surface impedance [11], infrared reflectivity [12], muon spin resonance (μ SR) [13, 14], nuclear magnetic resonance (NMR) [15], and magnetic force microscopy (MFM) [16–19] have been employed for measurements of λ in thin films and bulk samples. Each of these methods has its own advantages as well as limitations, and some of them require simulations with multiple fit parameters. For example, TDO measurements depend on the quality and thickness of an Al film deposited on top of a superconductor, which may not be fully reproducible yielding errors in the obtained values of

λ . The temperature dependent Meissner response method is only sensitive for thin films with magnetic field parallel to the surface. The reversible magnetization method works only in clean samples or materials having an extended vortex liquid phase. Mutual inductance and μ SR techniques are limited to thin films and bulk samples, respectively. μ SR measures the second moment of the magnetic field distribution around vortex, consequently details of the vortex structure and the muon's location affect experimental accuracy. Infrared reflectivity allows measurement of the anisotropy λ by polarization of an incident light.

MFM has been widely used for studies of superconductors, particularly for imaging and manipulation of vortices in superconducting thin films and single crystals [20–24]. Recently, MFM was also used as a local probe of the magnetic penetration depth [17–19], where the values of λ were extracted by fitting either an MFM signal from a single vortex [17] (note, this method doesn't work for systems with large values of the magnetic penetration depth [25]) or a Meissner response [18, 19]. These methods require a thorough characterization of the probe tip and a well-defined simulation model (dipole–monopole, monopole–monopole, etc) describing interactions between the MFM tip and a superconducting sample. However, modeling procedures with multiple fitting parameters introduce uncertainties in the resulting λ values. In spite of technical difficulties MFM has certain advantages e.g. localization of measured λ values providing a route to explore anisotropy of λ throughout the sample. In our approach, we obtain local values of λ by directly comparing the Meissner curves for the sample under investigation with those obtained for a reference sample with a well-known λ ; we emphasize that the measurements are done using the same MFM tip during the same cool-down. We observed strong dependence of the Meissner response curves on the shape of the MFM tip. When the tip crashes or even slightly touches the sample surface, Meissner response curves can change significantly. During our experimental procedure we verify that the MFM tip does not change its properties by comparing the Meissner curve from a Nb reference sample before and after the measurement. In this paper we demonstrate the validity of our method by determining λ in a $\text{Ba}(\text{Fe}_{0.92}\text{Co}_{0.08})_2\text{As}_2$ single crystal and a MgB_2 film. We measured λ values at several locations of the samples ($\text{Ba}122$ and MgB_2). The measured λ values at different locations are within a few per cent of each other, well within the reported uncertainty. Our results demonstrate that the same procedure can be used in any single crystal or thin film superconducting samples with a thickness greater than λ (for thinner samples λ can be corrected in a straightforward manner [26, 27]).

2. Experiment

The measurements described in this paper were performed in a home-built low-temperature MFM apparatus [28]. We have developed an additional capability of investigating multiple samples (including a reference sample), as shown in figure 1(a), for acquiring a complete set of MFM data for each of the samples within a single cool-down. Each stick–slip

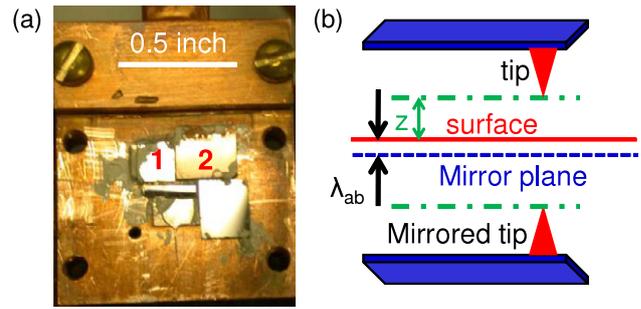


Figure 1. (a) Sample holder with multiple samples. The Nb thin film (300 nm) and the MgB_2 thin film (500 nm) samples are labeled as 1 and 2, respectively. (b) Schematic illustration of the Meissner experiment. The Meissner response force between the probe tip and the sample can be regarded as the force between the real tip and image tip at $2(z + \lambda)$, when $z \gg \lambda$ [18].

positioner has its own built-in optoelectronic encoder which allows repeatable movement with a nanometer-scale precision. First, we record the samples' locations prior to cool-down, when the cryostat is open, and then cool-down the samples. Thermal drift between the room temperature and 4 K is on the order of a few micrometers due to a rigid design of the microscope. We utilize the knowledge of the sample's location acquired at room temperature to navigate the sample at 4 K. The absolute value of λ is obtained by a simple comparison of the Meissner curves for the Nb reference to those obtained in the sample of interest. The Meissner response curve is first measured in a homogeneous reference sample (Nb film) as a function of the tip–sample separation. The origin (zero point) was determined by the frequency setpoint of the feedback in frequency modulation mode. The frequency setpoint is -10 Hz, and the corresponding distance between the tip and the sample is a couple of nanometers. The feedback control of the tip allows us to avoid the crash of the tip. In general, the frequency shift of about -100 Hz during the tip's approach of the sample surface indicates the crash of the tip: this is indicated by a sudden appearance of the external noise during the approach. If the tip crashes, the Meissner curves do not perfectly overlap. In the case of a crash we obtain a new data set in both the Nb reference and the unknown. The tip–sample separation was determined via the calibration of the piezo scanner. Then the cantilever is moved over the sample of interest, and its Meissner response curve is obtained. Direct comparison of these curves (comparative experiments) yields the absolute value of λ in a sample under investigation. The value of λ in the reference sample (Nb film) was verified by a different MFM technique and a SQUID magnetometry measurement [17]. The reference Nb thin film ($T_c \approx 8.8$ K, where resistance drops to zero) has a thickness of 300 nm and was grown by an electron beam deposition. A $\text{Ba}(\text{Fe}_{0.92}\text{Co}_{0.08})_2\text{As}_2$ single crystal ($T_c \approx 22$ K from specific heat capacity measurements) was grown out of FeAs flux [29, 30]. The 500 nm thick MgB_2 film ($T_c \approx 38.3$ K, zero resistance temperature) was grown by reactive evaporation [31]. The MFM measurements were performed

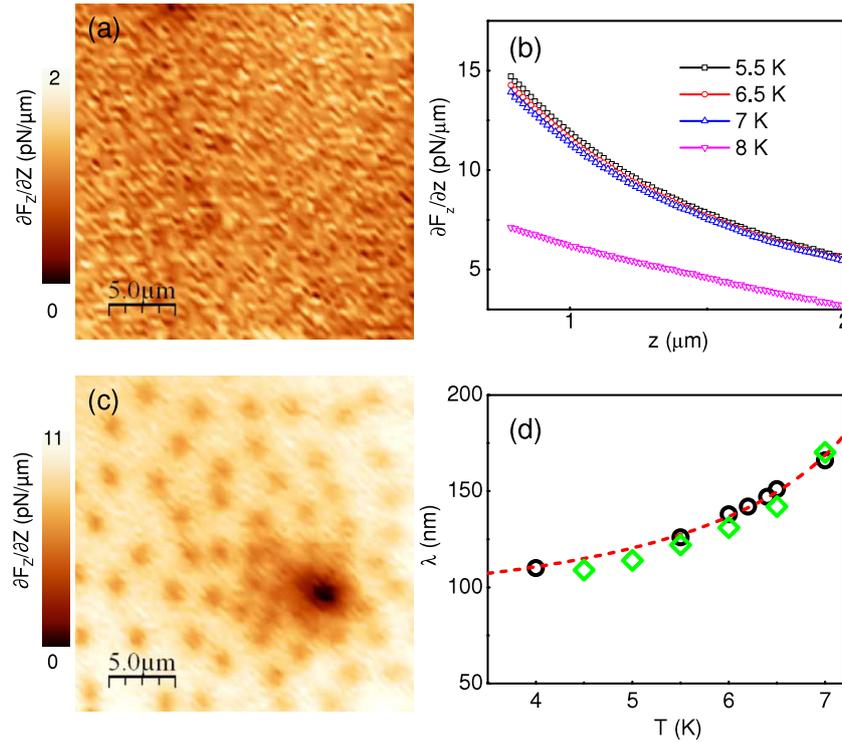


Figure 2. (a) MFM image of the Nb sample at $T = 4 \text{ K}$; no vortices are present in a $25 \mu\text{m} \times 25 \mu\text{m}$ field of view. The unit of the color scale is converted from Hz to $\text{pN } \mu\text{m}^{-1}$ using the relation $df/dz = (2k/f_0)\delta f$, where f_0 is the resonant frequency and k is the spring constant of the cantilever. Uniformity of Meissner response in a $25 \mu\text{m} \times 25 \mu\text{m}$ field of view shows homogeneity of λ . (b) Meissner response curves taken over the scan area shown in (a). (c) The MFM image taken with the same field of view as in (a) after the Meissner response experiment at 8 K ; vortices are nucleated by the magnetic field of the MFM tip. (d) Temperature dependence of the penetration depth $\lambda(T)$ in Nb obtained by overlaying Meissner curves on top of the 4 K reference curve (black circles). Green squares represent SQUID data. The red-dashed curve represents the conventional isotropic BCS model, expressed as $\lambda(T) = \lambda(0)(1 - t^2)^{-1/2}$, where t is T/T_c .

using a high resolution Nanosensors cantilever⁵ that was polarized along the tip axis in a 3 T field of a superconducting magnet. The superconducting samples are zero field-cooled for the Meissner experiment and are field-cooled in a field of few Oersted, applied perpendicular to the film surface and parallel to the probe tip, for imaging of vortices. All samples are electrically grounded to eliminate a possible electric force contribution from a stray charge on the sample to the magnetic Meissner force.

3. Results and discussion

3.1. MFM measurement in the Nb film

As the magnetic tip approaches a superconducting sample, it experiences a Meissner force (Meissner response) induced by the shielding currents in the sample that screen the magnetic field of the tip. The experimental procedure is as follows: first the probe tip is brought close to the reference sample (Nb film, position 1 in panel (a) of figure 1) and the Meissner response is recorded as a function of the tip-sample separation at a certain temperature T . There should be no vortices present in a large field of view of the sample, i.e., the sample should remain in a pure Meissner state. Figure 2(a) shows the Meissner state

with no vortices present in the $25 \mu\text{m} \times 25 \mu\text{m}$ field of view after a small compensation field was applied above T_c of Nb to compensate the remnant stray field of the superconducting magnet.

Figure 2(b) shows several Meissner response curves for the Nb film reference sample between 5.5 and 8 K . The Meissner response force is a function of λ and the tip-sample separation z , $F_M = F(z + \lambda)$ [18, 32]. For a given temperature T , $F_M[z + \lambda(T)] = F_M[z + \lambda(4 \text{ K}) + \delta\lambda]$, where $\delta\lambda = \lambda(T) - \lambda(4 \text{ K})$. Therefore, to determine the λ value at a particular temperature T , $F_M[z + \lambda(T)]$ is shifted along the z axis to coincide with $F_M[z + \lambda(4 \text{ K})]$, a reference curve measured at 4 K , and the value of the shift yields the value of $\delta\lambda(T)$.

The Meissner response curve taken at 8 K shows a behavior very different from the data taken at lower temperatures. Figure 2(c) shows the MFM image acquired at 4 K after the Meissner measurement was performed at 8 K . The scan areas in figures 2(a) and (c) are the same. The vortices seen in figure 2(c) were generated by the field of the probe tip. They cause magnetic field leakage and weaken the Meissner response (see figure 2(b), 8 K curve). Such a behavior is observed when λ is comparable to the film thickness, and the magnetic field from the tip cannot be fully screened [26, 27]. Special care must therefore be taken with the Meissner technique in the vicinity of T_c in thin films since increasing λ may take the sample out of the pure Meissner state.

⁵ A SSS-QMFMR Cantilever, Nanosensors, Inc.

To verify the 4 K value of $\lambda(4\text{ K}) = 110 \pm 10\text{ nm}$ for the Nb reference, measured previously using a different MFM technique [17], we performed measurements of λ using the SQUID magnetometry. The Nb reference sample used in MFM measurements (film $L = 3.2\text{ mm} \times W = 4.2\text{ mm}$) was oriented carefully with \mathbf{H} parallel to the surface along the side L , using a home-built sample holder. By measuring the transverse component m_{\perp} of the magnetic moment \mathbf{m} in the Meissner state (which should be zero in the case of perfect alignment) we confirmed that the mis-orientation between \mathbf{H} and the film surface was $\phi_{\text{mis}} \sim 0.2^{\circ}$. In this configuration, in the Meissner state the component of \mathbf{m} parallel to \mathbf{H} is $m_{\parallel} = (H/4\pi) \times LWd_{\text{eff}}$, where the effective thickness $d_{\text{eff}}(T) = d - 2\lambda(T) \tanh(d/2\lambda(T))$ is smaller than the geometrical thickness due to the field penetration from both surfaces [1, 2]. We measured m_{\parallel} versus H at several T , and from the slopes dm_{\parallel}/dH we extracted $\lambda(T)$. The main source of error in this method is the spurious contribution to m_{\parallel} due to the projection of the Meissner signal arising from the transverse field component, $\sim m_{\perp} \phi_{\text{mis}}$, which is $\sim 10\%$ of m_{\parallel} , thus introducing an error of $\sim 5\%$. However, it does not distort the functional dependence of $\lambda(T)$. The absolute values of $\lambda(T)$ from these SQUID measurements are marked with green squares in figure 2(d) and agree well with both the MFM data (black circles) and the isotropic single-gap BCS model (red-dashed curve).

3.2. Measurements of the absolute values of λ in a $\text{Ba}(\text{Fe}_{0.92}\text{Co}_{0.08})_2\text{As}_2$ single crystal

In recent years iron-based pnictide superconductors have drawn a great deal of attention since these systems exhibit superconducting properties which are intermediate to conventional BCS superconductors and high- T_c cuprates. Magnetic penetration depth of a $\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$ system (BFCA), the so-called 122 family, was investigated using variety of techniques including TDO [33], μSR [34], and MFM [19]. The Stanford group utilized an MFM technique and reported absolute values of λ in this system as a function of doping level. The authors used fitting algorithms to approximate the tip magnetization and calculate the values of λ [19]. We applied our direct (comparative) technique to measurements of λ in the very same system ($\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$ ($x = 0.08$)) to demonstrate the validity of our approach. Figure 3 shows Meissner curves as a function of the tip-sample separation z obtained from BFCA (blue squares) and a Nb reference (red circles), respectively. The slow decay of a frequency shift in BFCA sample compared to that in the Nb reference indicates greater values of λ in the BFCA sample. The expression for the Meissner response force gradient that MFM detects directly in Nb (assuming monopole-monopole interaction between the tip and the sample) can be written as follows: $(\partial f/\partial z)_{\text{Meissner}}^{\text{Nb}} = \frac{A\Phi_0}{(z+\lambda_{ab}^{\text{Nb}})^3}$, where A is a prefactor that reflects the sensor's geometry and the magnetic moment, Φ_0 is a single magnetic flux quantum, z is the tip-sample separation, and λ_{ab}^{Nb} is the in-plane magnetic penetration depth since the shielding current runs within the basal plane. The Meissner force

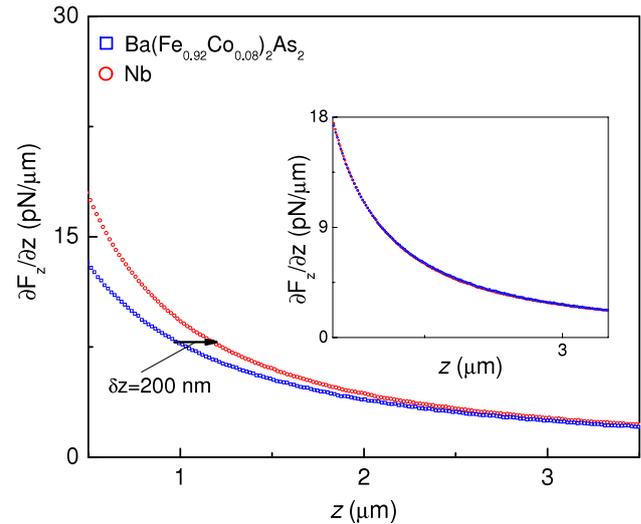


Figure 3. Meissner response curves obtained from (a) the Nb reference, (b) a $\text{Ba}(\text{Fe}_{0.92}\text{Co}_{0.08})_2\text{As}_2$ single crystal at 4 K. Different slopes of the Meissner curves obtained from each sample indicate a systematic change of λ . Inset: the Meissner curve of $\text{Ba}(\text{Fe}_{0.92}\text{Co}_{0.08})_2\text{As}_2$ is shifted by $z = 200\text{ nm}$ along the z axis to overlay the Meissner curve of the Nb reference sample. The difference of the penetration depths $\Delta\lambda = 200\text{ nm}$ can be obtained from the value of the shift along the z axis.

gradient in BFCA has the same functional form but different λ^{BFCA} : $(\partial f/\partial z)_{\text{Meissner}}^{\text{BFCA}} = \frac{A\Phi_0}{(z+\lambda^{\text{BFCA}})^3}$. Two Meissner curves become identical $[(\partial f/\partial z)_{\text{Meissner}}^{\text{BFCA}} = (\partial f/\partial z)_{\text{Meissner}}^{\text{Nb}}]$ when the tip lift z compensates for differences $\lambda^{\text{BFCA}} - \lambda^{\text{Nb}} = \delta\lambda$. In other words, by shifting the Meissner curve for BFCA along the z axis to overlay the Meissner curve measured for Nb, the $\delta\lambda$ value can be extracted. By adding the shifted value $\delta z = \delta\lambda = 200\text{ nm}$ to the $\lambda^{\text{Nb}}(110\text{ nm})$ one obtains $\lambda^{\text{BFCA}} = 310 \pm 30\text{ nm}$. This value is close to the one reported previously [19]. The inset in figure 3 demonstrates that two Meissner curves overlay each other very well after shifting the BFCA curve along the z axis by 200 nm.

3.3. Measurement of the absolute value of λ in a MgB_2 film

We used the same approach to measure the absolute value of λ in a MgB_2 thin film sample. The Meissner response curve taken at 4 K in the Nb was used as a reference Meissner curve and compared to the Meissner response curve measured in MgB_2 . The offset between these two curves, shown in figure 4(d), yields the absolute value of λ at 4 K. The $\delta\lambda$ between the Nb and MgB_2 curves at 4 K equals 90 nm yielding $\lambda(4\text{ K}) = 200 \pm 30\text{ nm}$ in MgB_2 . Our experimental error is 10%–15%, and depends on the magnitude of λ and the system noise level. Absolute values of λ in MgB_2 measured with various techniques range from 40 to 210 nm [35–41]. The large variation of λ in MgB_2 may be due to inclusion of impurities, such as C, N, and Al, which replace either Mg or B and significantly affect the electronic structure of the system due to the two-band nature of MgB_2 . The origin of the impurities-induced large value of λ and its temperature dependence obtained from the Meissner response curves in

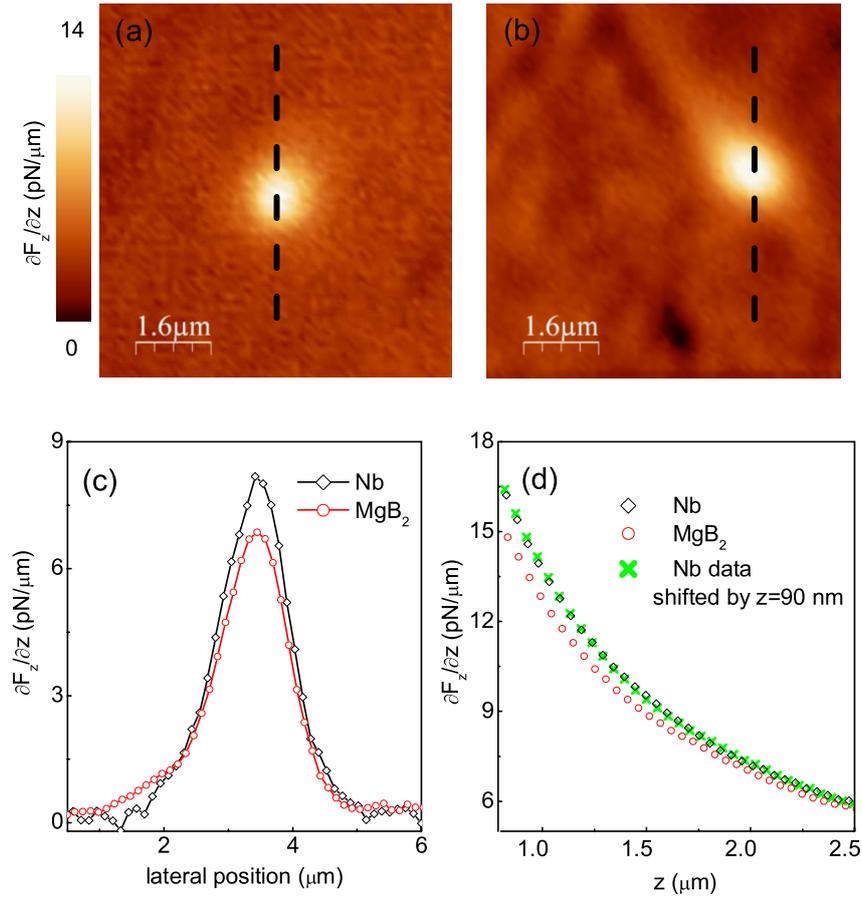


Figure 4. Single-vortex images in (a) the Nb thin film and (b) the MgB₂ thin film samples at $T = 4$ K. Both images were taken with a tip-lift height of 400 nm. (c) The single-vortex profiles taken along the dotted lines in (a) and (b). (d) Meissner response curves taken at 4 K in Nb (black square) and MgB₂ (red circle) with the same experimental condition. The green-crossed marks represent that the red circle (MgB₂), after shifted by 90 nm along the z axis, is overlaid over the black square (Nb) to show the validity of our approach.

the MgB₂ film taken at different temperatures (not shown) are described elsewhere [42].

We also imaged individual vortices in both Nb and MgB₂. Direct comparison of the vortex profiles provides additional information on the magnitude of λ . The maximum force gradient at the center of a vortex, $\max(\partial f/\partial z)$ (MFM is sensitive to a force gradient), is proportional to $(z + \lambda_{ab})^{-3}$ for a monopole–monopole model of the tip–vortex interaction [23, 24, 43]. In this model, the larger value of λ results in a smaller force gradient at the center of a vortex. Figures 4(a) and (b) show well-isolated vortices acquired in the Nb reference and the MgB₂ sample at 4 K. Direct comparison of vortex profiles (see figure 4(c)), taken along the dotted lines in figures 4(a) and (b), shows that the force gradient in the MgB₂ sample is smaller than that in the Nb sample. This indicates a larger value of λ in our MgB₂ film. It is worth noting that the difference of the gradient forces between the Nb and MgB₂ based on a simple point tip model [$\partial f/\partial z \propto (z + \lambda_{ab})^{-3}$] should be a factor of two but that of the gradient forces in figure 4(c) is about 15%. This is because the force gradient equation used here is a simplified form, appropriate for an ideal situation of a point tip model; it indicates the magnitude of relative forces for different values of λ . In reality, one should include the tip

geometry effect, which significantly complicates the equation and requires extensive simulations. One of the central points in this paper is that we can avoid the question of the tip geometry effect altogether by using a comparative method between the reference (Nb) and the unknown sample. It should be noted that the relatively high tip–sample separation (400 nm tip lift) and the tip geometry are responsible for the broadening of the vortex force profiles in both Nb and MgB₂ samples in figure 4(c). It is worth noting that an MFM image reflects the Meissner responses of an MFM tip at each location, indicating a local variation of λ , and thus information of inhomogeneity is present in the image. The MFM image of MgB₂ exhibits inhomogeneity due to impurities on a micrometer scale while that of Nb shows homogeneity [42].

4. Conclusion

In conclusion, we have developed an experimental method and apparatus to determine the absolute value of the magnetic penetration depth λ in superconducting samples by comparing their Meissner response curves to those acquired for a homogeneous Nb reference film. We used this method to obtain the absolute value of $\lambda(4\text{ K}) = 310 \pm 30$ nm in a Ba(Fe_{0.92}Co_{0.08})₂As₂ single crystal, consistent with the value

reported by the Stanford group. We also measured $\lambda(4\text{ K}) = 200 \pm 30\text{ nm}$ in a MgB_2 film. Our MFM apparatus allows us to simultaneously load and investigate multiple samples (over ten samples can be studied at once, providing an opportunity to explore the complete phase diagram of a superconducting system), and most importantly to use the same cantilever tip for both Nb reference and the samples under investigation in a single cool-down. This capability enables *in situ* calibration of the MFM tip on a known homogeneous Nb sample and does not introduce any additional uncertainties due to modeling of the tip geometry and the resulting tip field. The validity of our approach is established by comparing the MFM and SQUID magnetometer measurements of the temperature dependence of λ in the Nb reference film. Our experimental approach opens the possibility of measuring the absolute value of $\lambda(T)$ in film and bulk superconducting samples.

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