

# Measuring the Lower Critical Field of Superconductors Using Nitrogen-Vacancy Centers in Diamond Optical Magnetometry

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The lower critical magnetic field,  $H_{c1}$ , of superconductors is measured by optical magnetometry using ensembles of nitrogen-vacancy centers in diamond. The technique is minimally invasive and allows accurate detection of the vector magnetic field with subgauss sensitivity and submicrometer spatial resolution. These capabilities are used for detailed characterization of the first vortex penetration into superconducting samples from the corners. Aided by the revised calculations of the effective demagnetization factors of actual cuboid-shaped samples, these measurements provide precise determination of  $H_{c1}$  and the related absolute value of the London penetration depth,  $\lambda$ . We apply this method to three well-studied superconductors: optimally doped Ba(Fe<sub>1-x</sub>Cox)<sub>2</sub>As<sub>2</sub>, stoichiometric CaKFe<sub>4</sub>As<sub>4</sub>, and the high- $T_c$  cuprate YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub>. Our results compared well with the values of  $\lambda$  obtained with other techniques, thus adding another noninvasive and sensitive method to measure these important parameters of superconductors.

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## I. INTRODUCTION

Superconductors remain a focus of intense research due to their unusual properties and potential in applications. Cuprates [1] and, more recently, iron-based superconductors [2] are of particular interest due to their high superconducting transition temperature,  $T_c$ , apparently unconventional pairing mechanism [3,4], and rich interplay of magnetism and superconductivity, including their coexistence in the bulk [5–8].

One of the fundamentally important characteristics of a superconductor is the superfluid density, which determines the screening of an external magnetic field and is experimentally evaluated from the absolute value of the London penetration depth  $\lambda(T)$ . Accurate measurements of the lower (also known as the “first”) critical field,  $H_{c1}$ , can be used to obtain  $\lambda$  directly; see Eq. (1). These measurements, however, are not simple. The nonspherical shape of the experimental samples leads to distortion of the magnetic fields at sample edges and necessitates vector-magnetic-field mappings with high spatial resolution of the order of  $\lambda$ , typically in the submicrometer range. This task was approached by use of local probes of magnetic induction, such as miniature Hall probes [9–11], miniature superconducting quantum-interference devices [12] and

magnetic force microscopes [13], with spatial resolution in the micrometer range and sensitivity to a single component of the vector magnetic field.

Among several factors for accurate measurements of  $H_{c1}$ , three are the most important: (i) the “probe” has to be noninvasive so that the local magnetic environment is not disturbed, (ii) it has spatial resolution comparable to  $\lambda$ , and (iii) the demagnetization corrections due to a particular sample geometry or shape should be accounted for properly to facilitate proper determination of  $H_{c1}$  from measured  $H_p$ . Magnetic sensing probes based on nitrogen-vacancy (N-V) centers in diamond satisfy the first two requirements. The magnetic moment of the N-V center itself is of the order of  $\mu_B$ , and thus minimally perturbs the original magnetic state of the measured specimen. Submicrometer spatial resolution can be achieved even with a N-V ensemble (as used here; see Sec. II for details), with a probe of 500-nm diameter and 20-nm thickness [14–18]. Furthermore, the ability to resolve the vector components of the magnetic induction provides better understanding of how the flux enters the sample.

In this work, we present a novel scheme for accurate measurements of  $H_{c1}$  of type-II superconductors using the N-V centers in diamond as an optical probe of local vector magnetic induction. Three different superconductors are measured, including the high- $T_c$  cuprate YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> (YBCO) and iron-based superconductors

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$\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$  and  $\text{CaKFe}_4\text{As}_4$ , to demonstrate the performance of this technique. These materials are subjects of active current research [19,20]. To deduce  $H_{c1}$ , we use modified demagnetization factors derived for realistic three-dimensional geometries and also compare our results with results when demagnetization factors calculated from infinite geometries are used [21].

### A. Lower critical magnetic field

The lower (first) critical field,  $H_{c1}$ , is one of the important fundamental parameters characterizing any type-II superconductor [22]. Above this field, Abrikosov vortices become energetically favorable and start entering the sample from the edges. Importantly,  $H_{c1}$  is related to two fundamental length scales, the London penetration depth,  $\lambda$ , and the coherence length  $\xi$ , as follows [23]:

$$H_{c1} = \frac{\phi_0}{4\pi\lambda^2} \left( \ln \frac{\lambda}{\xi} + 0.497 \right). \quad (1)$$

$\xi$  enters Eq. (1) only as a logarithm and there are other more-direct and more-sensitive ways to determine it experimentally [e.g., from the upper critical field,  $H_{c2} = \phi_0 / (2\pi\xi^2)$ , where  $\phi_0 = 2.07 \times 10^{-15}$  Wb is magnetic flux quantum]. Thus, the London penetration depth  $\lambda$  is often estimated with Eq. (1) if  $H_{c1}$  is experimentally given. In terms of the numerical values, for example, for the  $\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$  iron-based superconductor studied here [5,24],  $\xi \approx 2.3$  nm and  $\lambda \approx 200$  nm, so  $\kappa = \lambda/\xi \approx 87$ , which gives  $H_{c1} \approx 200$  Oe and  $H_{c2} \approx 60$  T. For optimally doped YBCO [25–27],  $\xi \approx 1.6$  nm,  $\lambda \approx 140\text{--}160$  nm,  $\kappa \approx 80\text{--}100$ ,  $H_{c1} \approx 350\text{--}400$  Oe, and  $H_{c2} \approx 120$  T.

In practice, use of Eq. (1) to determine  $H_{c1}$  has two major difficulties: (i) the existence of various surface barriers [28–30] that inhibit the penetration of a magnetic field, hence leading to overestimation of  $H_{c1}$ , and (ii) the distortion of the magnetic field around the actual, finite-size sample that leads to underestimation of  $H_{c1}$ . Therefore, the experimentally detected onset of the magnetic field penetration, denoted here as  $H_p$ , coincides with  $H_{c1}$  only in the case of an infinite slab in a parallel magnetic field and no surface barrier, conditions that are almost impossible to achieve in experiments. However, analysis shows that  $H_p$  is directly proportional to  $H_{c1}$  with the appropriate geometric conversion factor [30,31]. Several previous studies analyzed the situation and now most experimentalists follow the numerical results published by Brandt [30,31], who used approximate nonlinear  $E(j)$  characteristics to estimate the connection between  $H_p$  and  $H_{c1}$ . Here it is important to understand how  $H_p$  is defined.

In Brandt's picture, illustrated in Fig. 1, for samples with a rectangular cross section  $2a \times 2c$  (see Fig. 2) with a magnetic field applied along the  $c$  axis, vortices start forming at the corners [where the local field is highest, Fig. 1(b)] and propagate as nearly straight segments cutting the corners

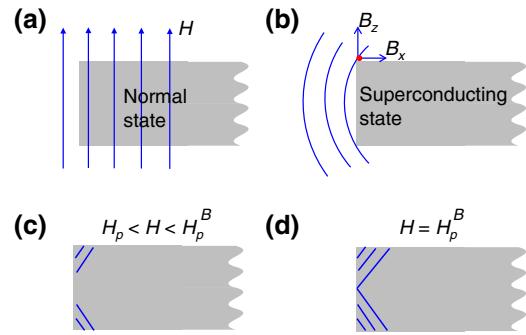


FIG. 1. (a) Applied magnetic field fully penetrating the superconductor in its normal state, (b) total expulsion of the magnetic flux by the superconductor in its superconducting state, (c) magnetic field entering from the corners of the sample at an angle of approximately  $45^\circ$ , and (d) Brandts' scenario when the applied magnetic field is equal to  $H_p^B$ . (See the text for details.)

at approximately  $45^\circ$  [Fig. 1(c)]. When the top and bottom segments meet in the middle of the side [at the “equator,” Fig. 1(d)], the vortex enters the sample completely. At this value of the applied field, which we denote as  $H_p^B$ , the magnetization,  $M(H)$ , reaches maximum amplitude and  $H_p^B \approx H_{c1} \tanh \sqrt{\alpha c/a}$ , where  $\alpha = 0.36$  for an infinite (in the  $b$  direction) strip or  $\alpha = 0.67$  for disks of radius  $a$  [30]. At this field a significant volume of the sample is already occupied by vortices (from the corner cutting) and the local magnetic field at the corners has far exceeded  $H_{c1}$ .

An alternative definition of  $H_p$  is based on the deviation of the local magnetic induction from zero or the total magnetic moment from linear  $M(H)$  behavior. In practice, the local magnetic induction,  $B$ , is measured outside the sample, on its surface close to the sample edge. The external magnetic field expelled by the sample leaks into the sensor,

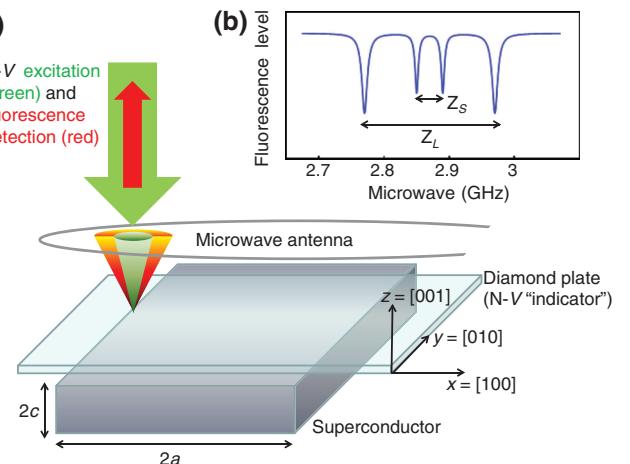


FIG. 2. (a) The key components of the N-V sensing setup (b) ODMR spectrum for a local magnetic field vector with two components,  $\vec{B} = (B_x, 0, B_z)$ . (See the text for details.)

so measured  $B(H)$  is always nonzero, but is still linear in  $H$  and deviates from linearity when vortices start to penetrate the sample from the corners, which can be detected as the onset of the flux penetration field  $H_p$  [11,32]. A similar estimate can be obtained from the  $M(H)$  curves detecting the deviation from linear behavior on application of a magnetic field after cooling in zero field [33]. Another version of this approach is to look for the remnant flux trapped inside the superconductor, which becomes nonzero when a lower critical field is reached in any part of the sample when vortices penetrated and became trapped due to ubiquitous pinnings [34]. In all these scenarios, the lower critical field should be obtained with the appropriate effective demagnetization factor,  $N$ :

$$H_p = H_{c1} (1 + N\chi), \quad (2)$$

where  $\chi$  is the “intrinsic” magnetic susceptibility of the material (i.e., in an “ideal” sample with no demagnetization and surface barriers), which can be taken to be equal to  $-1$  for a robust superconductor at most temperatures below  $T_c$  [for an infinite slab of width  $2w$  in a parallel field,  $\chi = \lambda/w \tanh(w/\lambda) - 1$ , and it is straightforward to check that  $\chi$  is still less than  $-0.995$  even at  $T/T_c = 0.99$ ].

Unfortunately, most previous studies that used local measurements of the onset of magnetic flux penetration obtained with, for example, miniature Hall probes [11,32,34] analyzed the data with Brandt’s formulas for  $H_p^B$  and not with the (more correct in this case)  $H_p$  from Eq. (2).

### B. Effective demagnetizing factors

To use  $H_p$  to determine  $H_{c1}$ , the effective demagnetizing factor,  $N$ , has to be calculated for a specific sample geometry. Strictly speaking,  $N$  is defined only for ellipsoidal samples, which is of little practical use for typical samples of a cuboidal (rectangular-plate) shape. Yet, it is possible to introduce effective demagnetizing factors that were calculated in several previous studies, including the studies by Brandt previously cited, since his estimate of  $H_p^B$  implicitly includes the effective  $N$  [31]. As we recently showed from a full three-dimensional finite-element analysis [21], Brandt provided very accurate expressions for demagnetizing factors in cases of infinite strips or disks of rectangular cross section; see Eq. (7) in Ref. [31]. However, we also found that the effective demagnetizing factors for finite cuboids are quite different from those for infinite two-dimensional (2D) strips and, therefore, the whole method of estimating  $H_{c1}$  from magnetic measurements should be revisited. This is the subject of the present work.

Although we can calculate the effective demagnetization factor with arbitrary precision for a sample of any shape, it is always useful to have simple, but accurate-enough formulas [21]. A good approximation for a  $2a \times 2b \times 2c$

cuboid in a magnetic field along the  $c$  direction is given by [21]

$$N^{-1} = 1 + \frac{3}{4} \frac{c}{a} \left( 1 + \frac{a}{b} \right). \quad (3)$$

Having samples of rectangular cross section is problematic from point of view of the uncertainty in demagnetization effects, but it is advantageous in terms of the absence of surface barriers, because now magnetic flux penetrates from the corners and not parallel to the extended flat surfaces, which is how surface barriers are formed [28]. Moreover, the “geometric barrier” that essentially involves the flux corner penetration described above [30,31] is not relevant if the onset of nonlinearity is detected near the sample edge.

## II. EXPERIMENT

### A. Optical magnetic sensing using N-V centers in diamond

The vector magnetic induction on the sample surface is measured by optical magnetometry based on N-V centers in diamond. Specifically, the optically detected magnetic resonance (ODMR) of Zeeman-split energy levels in N-V centers, proportional to a local magnetic field, is measured [35]. The magnetosensing using N-V centers has several important advantages for measurements of delicate effects in superconductors: (i) it is minimally invasive (the magnetic moment of the probe itself is on the order of a few Bohr magnetons and hence has a negligible effect on the measured magnetic fields); (ii) it has sufficient spatial resolution (submicrometer spatial mapping can be achieved even with the ensemble mode of N-V sensing); (iii) it is capable of measuring vector magnetic induction [36]. This is particularly important as the detection of flux penetration depends on the location, and magnetic field lines deviate significantly from the direction of the applied field [21].

Measurement protocols, experimental schematics, and deconvolution of the ODMR spectrum into magnetic field components are discussed in detail in our previous work in which the spatial structure of the Meissner state in various superconductors was studied [36]. Here we focus particularly on measurements of the lower critical field,  $H_{c1}$ , and summarize the key experimental details for the completeness.

To measure a local magnetic induction, a magneto-optical “indicator” ( $1.5 \times 1 \times 0.04$  mm $^3$  diamond plate with embedded N-V centers) is placed on top of the superconducting sample with its N-V-active side facing the sample surface. On the “active” side, N-V centers are created within approximately 20 nm from the surface of a single-crystal diamond plate with commercial protocols that involve nitrogen-ion implantation, electron irradiation, and high-temperature annealing in a high vacuum. The

diamond plate has a (100) crystal surface and [100] edges. Therefore, N-V centers are oriented along all four [111] diamond axes, which define the directions of the magnetic field sensing. As a result, possible Zeeman splittings in a random ensemble of N-V centers in (indeed, a single crystal) diamond are given by  $2\gamma_e|\vec{B} \cdot \hat{d}|$ , where  $\gamma_e \approx 2.8$  MHz/G is the gyromagnetic ratio of the N-V-center electronic spin and  $\hat{d}$  is a unit vector along any of the four diamond axes. In a magnetic field along the  $\hat{z}$  direction, that is,  $\vec{B} = (0, 0, B_z)$ , all possible N-V orientations result in the same splitting:

$$Z = \frac{2\gamma_e}{\sqrt{3}} \approx 3.233 \text{ MHz/G}.$$

However, if the magnetic field has two components such that  $\vec{B} = (B_x, 0, B_z)$ , the N-V ensemble will result in two pairs of Zeeman splitting:

$$Z_{L,S} = Z|B_z \pm B_x|,$$

where  $Z_L$  ( $Z_S$ ) refers to larger (smaller) Zeeman splitting. An example of such two pairs of ODMR splitting is shown in Fig. 2(b).

## B. Experimental details

### 1. Experimental setup

The experimental setup is based on an Attocube attoAFM/CFM system and includes a confocal microscope optimized for N-V fluorescence detection inside a helium cryostat with optical parts in vacuum and the sample placed on a temperature-controlled cold stage. A schematic of the experiment is shown in Fig. 2(a). The objective is focused on the N-V centers in an optically transparent diamond plate so that the convolution of the diffraction-limited confocal volume with the N-V distribution essentially leads to a disk-shaped sensing volume of thickness approximately 20 nm and diameter approximately 500 nm. The diamond plate is placed directly on top of a flat sample surface covering the edge and with the N-V-active side facing the sample. More importantly, the superconducting sample edges are carefully aligned with diamond edges so that when the superconductor is in the Meissner state the vector magnetic field at the probing point will have no component along the [010] diamond-crystal direction. This symmetry of placement guarantees only two pairs of Zeeman splitting in the ODMR. A 50× confocal-microscope objective is used both for green-laser excitation and red-fluorescence collection. Microwave radiation with a very small amplitude is applied with a single-turn 50-μm-diameter silver wire.

### 2. Temperature variation during measurements

The laser and microwave power used in these experiments are approximately 500 μW and less than 0 dBm,

respectively. While no noticeable effect is observed due to the laser, microwave excitation results in a small change in the base temperature fluctuating between 4.2 and 4.3 K. Therefore, all our experiments are performed at 4.5 K with active temperature control keeping the temperature stable within approximately 10 mK. These fluctuations are much lower than reported in Ref. [37], probably because our microwave-loop-antenna geometry is more optimized for the experiment.

### 3. Spatial resolution

Spatial resolution of the probe is governed by the optical diffraction limit, resulting in lateral resolution of approximately 500 nm. One possibility to increase the lateral resolution is to incorporate super-resolution imaging techniques [38,39]. Another possibility is to use nanoscale scanning N-V probes [40–42]. Magnetic imaging of individual Abrikosov vortices was demonstrated with scanning single N-V probes in Refs. [43,44]. The imaged superconducting materials in those studies were field cooled to the superconducting state in the presence of a weak external background magnetic field to form a well-isolated vortex distribution.

### 4. Integration time

In our  $H_{c1}$  measurements, for each data point (a given position and external magnetic field), the ODMR spectrum is obtained for a 50–100-MHz scan range averaged for ten repetitions. The typical total integration time per data point is 5–10 min. To speed up the experiments, one could use adaptive protocols to modify or optimize the scan range and number of averages according to the previous measurement results. Another possibility is to incorporate real-time lock-in-detection techniques [45].

### 5. Samples

All samples are precharacterized by various thermodynamic and transport techniques (see, e.g., Ref. [46]) and imaged by scanning electron microscopy, and only samples with well-defined surfaces and edges, as shown in Fig. 3(a), are selected for further measurements.

## III. MEASUREMENTS OF THE LOWER CRITICAL FIELD

The experimental protocol for measurements of  $H_{c1}$  is as follows:

(a) The sample is cooled to the target temperature below  $T_c$  in the absence of a magnetic field (zero-field cooling, ZFC). Then a small magnetic field (10 Oe in our case, much smaller than the 200–400 Oe expected for  $H_{c1}$  at low temperatures as discussed in Sec. I) is applied and ODMR signals are recorded at different points

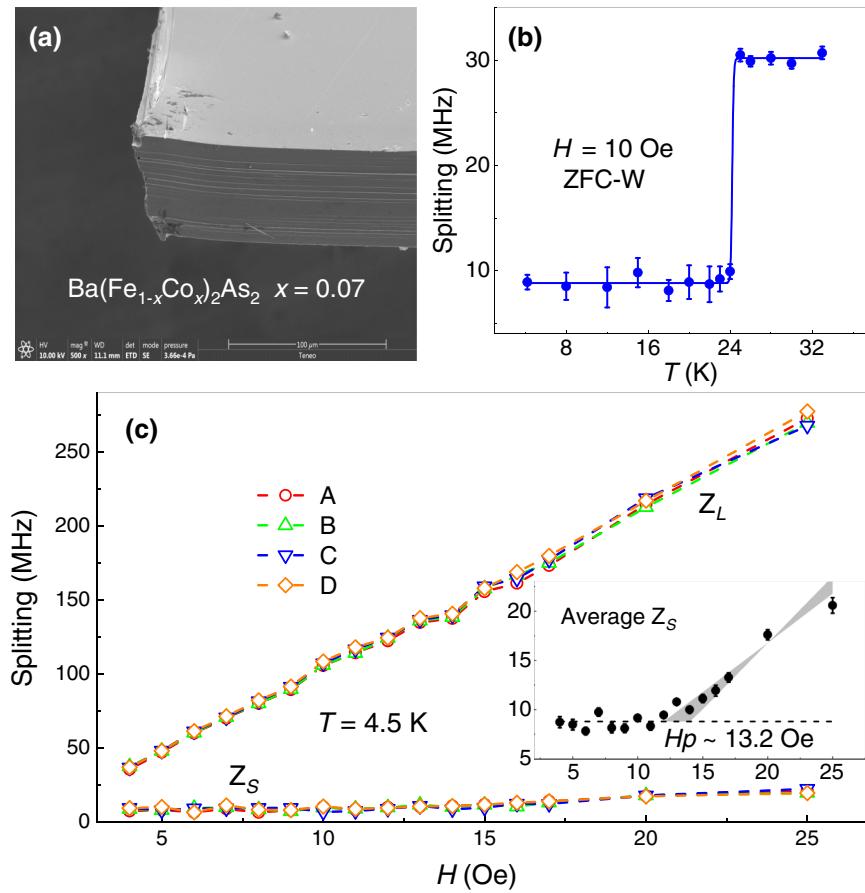


FIG. 3. (a) Scanning-electron-microscope image of the measured single crystal of  $\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$ ,  $x = 0.07$  (b) Detection of the superconducting phase transition at  $T_c \approx 24$  K in the “warming” curve upon zero-field-cool (ZFC-W). Each data point in the plot is obtained from a total integration time of the ODMR of 4 min. Error bars represent the standard errors extracted from the double-Lorentz-function fitting parameters for the dip position (not shown here). (c)  $H_{c1}$  measurements of this sample at 4.5 K. Zeeman splittings measured at four different points (A, B, C, and D) near the edge as a function of increasing magnetic field applied after zero-field cooling (ZFC). The four-point-averaged signal of  $Z_S$  is shown in the inset; a clear “change” at  $H_p = 13.2 \pm 1$  Oe is observed. The shaded area visually captures the spread of measurements after this change, from which the error of  $H_p$  is determined.

along the line perpendicular to the sample edge. Measured ODMR splittings are then converted into the magnetic induction values as described above. This, combined with direct visualization of the sample through a transparent diamond plate, allows accurate determination of the location of the sample edge and provides information about sample homogeneity. The quality of the superconductor is also verified by the sharpness of the transition detected by the ODMR splitting recorded as a function of temperature at any fixed point over the sample; see, for example, Fig. 3(b).

(b) After this initial preparation and edge identification, the magnetic field is removed and the sample is warmed up to above  $T_c$  and then cooled back down to a target temperature, thereby resetting it to the genuine superconducting state with no trapped magnetic field inside. A point inside and over the sample, but close to the edge, is chosen and ODMR spectra are recorded as a function of the external magnetic field, which is applied incrementally in small steps. At each step, the superconducting magnet is switched to a persistent mode to ensure stability of the magnetic field. The deviation from the linear behavior in  $Z_S$  is then detected and recorded as the field of first flux penetration,  $H_p$ .

(c) With use of Eqs. (2), (3), and (1),  $H_{c1}$  and the London penetration depth  $\lambda$  are evaluated. This procedure

is repeated at several locations along the edge to ensure objectivity of the results.

#### IV. RESULTS AND DISCUSSION

To illustrate the method described, we measure  $H_{c1}$  and evaluate the London penetration depth,  $\lambda$ , in three different superconducting materials.

##### A. $\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$ , $x = 0.07$

A well-characterized optimally doped single crystal of  $\text{Ba}(\text{Fe}_{1-x}\text{Co}_x)_2\text{As}_2$ ,  $x = 0.07$ , of cuboidal shape with dimensions  $1.0 \times 1.2 \times 0.05$  mm $^3$  was selected. The scanning-electron-microscope image in Fig. 3(a) shows a well-defined rectangular corner with flat clean surface and straight edges. The superconducting transition temperature,  $T_c \approx 24$  K, determined with a conventional magnetometer, is consistent with our ODMR measurements at the location on the sample surface inside the sample as shown in Fig. 3(b). ODMR splittings at four different locations on the sample surface near the edge are labeled A, B, C, and D in Fig. 3(c). These four points are approximately 5  $\mu\text{m}$  from neighboring points and each point is approximately 10  $\mu\text{m}$  from the edge inside the sample. As discussed above, the two Zeeman splittings  $Z_L$  and  $Z_S$  correspond to linear combinations of horizontal ( $B_x$ ) and

vertical ( $B_z$ ) components of the magnetic induction. Notice excellent reproducibility of the results, indicating homogeneous superconducting properties of our sample. The inset in Fig. 3(c) shows the average (of four points) small splitting signal ( $Z_S$ ). A clear onset of first flux penetration is determined at  $H_p = 13.2 \pm 1$  Oe.

To understand the observed ODMR splittings, we consider Brandt's results of flux corner cutting and entering in the form of Abrikosov vortices at an angle of approximately  $45^\circ$  with respect to the corner. Therefore, the normal-to-the-sample-surface  $z$  component (along the applied field) and the longitudinal  $x$  component of the magnetic induction are approximately equal and proportional to the applied field. This linear relation continues with increasing applied field until the first flux penetration field,  $H_p$ , is reached. At this point, the angle of the magnetic flux at the sample edges deviates from  $45^\circ$ , trending more toward the  $\hat{z}$  direction. This scenario can be phenomenologically modeled by our representing the magnetic induction components as  $B_{z,x} = DH \pm \delta$  and  $\delta = 0 + \alpha\theta(H - H_p)(H - H_p)^n$ , where  $D$  is an effective demagnetization factor and  $\theta(H)$  is a Heaviside step function. Because the larger splitting  $Z_L$  and the smaller splitting  $Z_S$  are proportional to the sum and difference of  $B_{z,x}$  components, respectively, the change at  $H_p$  is reflected clearly in  $Z_S$  but not in  $Z_L$ . The Zeeman splittings observed in Fig. 3(c) can be understood with this model for the parameters:  $D = 3.5$ ,  $H_p = 13.2$ ,  $\alpha = 0.6$ , and  $n = 1$ . Hence, this provides experimental confirmation for Brandt's description of flux corner cutting and entering at an angle of approximately  $45^\circ$  with respect to the sides.

From the experimental value of  $H_p$  and the effective demagnetization factor for this particular sample,  $N = 0.9168$ , we obtain using Eq. (2),  $H_{c1} = 158 \pm 12$  Oe. Using Eq. (1) and taking  $\xi \approx 2.3$  nm, we obtain the final result,  $\lambda = 226 \pm 10$  nm. This estimate for the penetration depth is comparable with the values obtained from other techniques such as muon spin relaxation (224 nm) [47] and magnetic force microscopy (245 nm) [48]. The agreement is quite remarkable and gives confidence in the validity of the technique developed. Table I summarizes all these estimates. Estimates obtained with Brandt's formulas are also given for comparison.

## B. CaKFe<sub>4</sub>As<sub>4</sub>

The cuboid single crystal of stoichiometric CaKFe<sub>4</sub>As<sub>4</sub> with dimensions of  $1.01 \times 0.99 \times 0.01$  mm<sup>3</sup> is studied. The inset in Fig. 4(a) shows a sharp superconducting phase transition at  $T_c \approx 34$  K. The average ODMR splitting,  $Z_S$ , near the sample edge as a function of the applied magnetic field clearly shows a break associated with the magnetic flux penetration at  $H_p = 8.3 \pm 1.1$  Oe. The error here is determined visually by the shaded region that spans all measurement points. With use of Eqs. (2) and (3), this

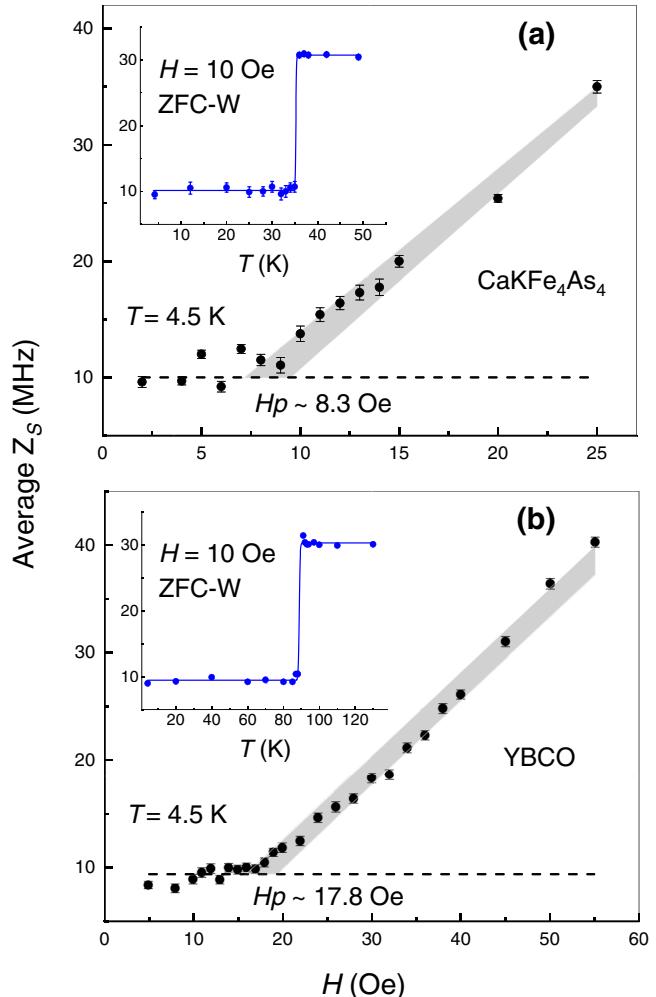


FIG. 4. Measurements of the field of first flux penetration,  $H_p$ , in single crystals of (a) CaKFe<sub>4</sub>As<sub>4</sub> and (b) YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub> . The insets show superconducting phase transitions at  $T_c \approx 34$  K and 88 K, respectively. ZFC-W, zero-field-cool warming.

results in the estimation of  $H_{c1} = 394 \pm 52$  Oe. Using Eq. (1) and  $\xi \approx 2.15$  nm [46], we estimate  $\lambda = 141 \pm 11$  nm. This result was used to calculate the superfluid density in Ref. [46], which is consistent with isotropic two-gap  $s_\pm$  pairing state.

## C. YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>

To look at a very different system, we also measure a single crystal of a well-known cuprate superconductor, YBCO. The sample dimensions are  $0.5 \times 0.85 \times 0.017$  mm<sup>3</sup>. The inset in Fig. 4(b) shows a sharp superconducting phase transition at  $T_c \approx 88$  K. The clear break associated with the magnetic field of first flux penetration in the average  $Z_S$  versus  $H$  plot is observed at  $H_p = 17.8 \pm 1.6$  Oe. With use of Eqs. (2) and (3), this leads to estimation of  $H_{c1} = 344 \pm 31$  Oe. Using Eq. (1) and coherence length  $\xi \approx 1.6$  nm [25,27], we estimate  $\lambda \approx 156 \pm 8$  nm. All

TABLE I. Estimates for  $H_{c1}$  and  $\lambda$ . Here “2D” refers to values obtained with Brandt’s formulas.

Superconductor	$T_c$ (K)	$H_{c1}^{2D}$ (G)	$\lambda^{2D}$ (nm)	$H_{c1}$ (G)	$\lambda$ (nm)	$\lambda$ (nm) from the literature
Ba(Fe <sub>1-x</sub> Co <sub>x</sub> ) <sub>2</sub> As <sub>2</sub> , $x = 0.07$	24.3	102 ± 8	288 ± 12	158 ± 12	226 ± 10	270, 245, 224 [47–49]
CaKFe <sub>4</sub> As <sub>4</sub>	34	139 ± 18	251 ± 18	394 ± 52	141 ± 11	208, 187 [50]
YBCO	88.3	163 ± 15	236 ± 12	344 ± 31	156 ± 8	146, 160, 155, 149 [26, 51–53]

estimates, including values obtained with Brandt’s formulas and from other techniques, are summarized in Table I. Once again, good agreement is seen between our estimates and the values reported in the literature obtained with other techniques, such as muon spin relaxation (155 nm) [52], microwave-cavity perturbation (160 nm) [51], and use of a tunnel-diode resonator (140 nm) [26].

## V. CONCLUSIONS

In summary we use N-V centers in diamond for sensing of the optical vector magnetic field at low temperatures to measure the lower critical field,  $H_{c1}$ , in type-II superconductors. The minimally invasive nature and optical-diffraction-limited small size of the probe make a N-V sensor ideal for this purpose. The capability of resolving vector components provides a unique advantage, which allows direct verification of Brandt’s model of magnetic flux penetration that proceeds via corner cutting by vortices at an angle of approximately 45° with respect to the edges. We apply this technique to three different superconductors: optimally doped Ba(Fe<sub>1-x</sub>Co<sub>x</sub>)<sub>2</sub>As<sub>2</sub>,  $x = 0.07$ , stoichiometric CaKFe<sub>4</sub>As<sub>4</sub>, and the high- $T_c$  cuprate YBCO. The London penetration depths evaluated from the  $H_{c1}$  values obtained are in good agreement with values in the literature. Our approach is a very useful noninvasive way to estimate the absolute value of London penetration depth  $\lambda(T)$ , needed to obtain temperature-dependent superfluid density, a quantity directly comparable to theoretical calculations.

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