Quantum phase transition inside the superconducting dome of Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ probed by optical magneto-sensing using NV-centers in diamond

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Unconventional superconductivity often emerges in close proximity to a magnetic instability. Upon suppressing the magnetic transition down to zero temperature by tuning the carrier concentration, pressure, or disorder, the superconducting transition temperature $T_c$ acquires its maximum value. A major challenge is the elucidation of the relationship between the superconducting phase and the strong quantum fluctuations expected near a quantum phase transition (QPT) that is either second order (i.e. a quantum critical point) or weakly first order. While unusual normal state properties, such as non-Fermi liquid behavior of the resistivity, are commonly associated with strong quantum fluctuations, evidence for its presence inside the superconducting dome are much scarcer. In this paper, we use sensitive and minimally invasive optical
magnetometry based on NV-centers in diamond to probe the doping evolution of the $T = 0$ penetration depth in the electron-doped iron-based superconductor Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$. A non-monotonic evolution with a pronounced peak in the vicinity of the putative magnetic QPT is found. This behavior is reminiscent to that previously seen in isovalently-substituted BaFe$_2$(As$_{1-x}$P$_x$)$_2$ compounds, despite the notable differences between these two systems. Whereas the latter is a very clean system that displays nodal superconductivity and a single simultaneous first-order nematic-magnetic transition above, and even somewhat below, $T_c$, the former is a significantly dirtier system with fully gapped superconductivity and split second-order nematic and magnetic transition above $T_c$. Thus our observation that such distinct systems display remarkably similar penetration depth peaks, combined with the theoretical result that a QPT alone does not ensure the existence of a peak, unveils a puzzling and seemingly universal manifestation of quantum fluctuations in the iron pnictides.

**Introduction**

The unconventional superconducting (SC) state of heavy fermions (1–9), cuprates (10–15), organics (16), and iron-based materials (17–23) is located close to the region of the phase diagram where long-range antiferromagnetism disappears, sometimes even overlapping with it. Elucidating the interplay between the putative $T = 0$ antiferromagnetic (AFM) transition – called a quantum phase transition (QPT) – and superconductivity remains one of the main challenges in the field, particularly near a second-order QPT (also known as a quantum critical point, QCP) or a weakly first-order QPT (e.g. a fluctuation-driven first-order transition). In these cases, quantum AFM fluctuations are expected to be strong and reach quite high temperatures. As
such, they are believed to manifest themselves in the non-Fermi liquid behavior of the metallic normal state and also to promote the superconducting instability by providing the glue that binds the Cooper pairs together (24–26). Throughout the manuscript, we use QPT to refer to either a second-order or a weakly first-order QPT. Although both are associated with strong fluctuations, the latter only diverge in the case of a QCP.

Therefore, uncovering the presence of such a QPT is important to understand the mechanism of unconventional superconductivity (9,11). However, most of the probes for strong fluctuations arising from a QPT focus on its possible normal-state manifestations, such as a divergence of the effective electronic mass $m^*$ or non-$T^2$ behavior of the resistivity (19). In contrast, only a few experimental approaches exist which may probe the impact of a QPT directly inside the SC dome. The London penetration depth, $\lambda$, is believed to be one of them. The qualitative argument is that, at zero temperature, both in the clean and dirty limits, $\lambda^{-2}(T = 0) \propto 1/m^*$. Of course, experimentally, the penetration depth is measured at finite temperatures, but since the variation of $\lambda(T)$ is very small up to a significant fraction of $T/T_c$, one can use $\lambda(T \ll T_c)$ instead of $\lambda(0)$. Moreover, as discussed below, the effect of finite temperature would result in the behavior opposite to what we observe.

Taking the proportionality between $\lambda^2$ and $m^*$ at face value, one would expect that the presence of a second-order or weakly first-order QPT inside the SC dome would be manifested as a sharp peak in the $T = 0$ penetration depth. Such a sharp peak in $\lambda$ was indeed observed under the dome of superconductivity in isovalently-substituted BaFe$_2$(As$_{1-x}$P$_x$)$_2$ (P-Ba122) (27, 28), and corroborated by anomalous behaviors of the critical magnetic fields $H_{c2}$ and $H_{c1}$ (21) and of the specific heat (29). Complementary, a behavior typically associated with strong quantum fluctuations was also observed in the normal state of P-Ba122, namely, the linear-in-$T$ resistivity (20, 30). Subsequent theoretical analyses, however, (31–35), revealed that while a sharp enhancement of $\lambda$ is generally expected upon approaching the second-order or weakly first-
order QPT from the pure SC side, due to the build up of critical AFM quantum fluctuations, a peak is not guaranteed to exist at the QPT. This is because $\lambda$ increases inside the AFM phase but does not diverge at the QPT, even if the latter is a second-order transition. Furthermore, the detailed doping dependence of $\lambda$, including the possible peak position, was shown to depend on other non-critical properties, such as disorder and Fermi surface topology.

It is therefore of general interest to establish whether the sharp peak of $\lambda$ observed in P-Ba122 is particular to this compound or a more universal property of iron-based superconductors. For instance, in other classes of unconventional superconductors, such as cuprates, a sharp peak in $\lambda$ is not observed (36). Indeed, P-Ba122 differs from most iron-pnictide materials in several key aspects. Because the pnictogen atoms have a weaker effect on the impurity scattering as compared to defects in the Fe plane, P-Ba122 is a very clean system, as evident from the observation of quantum oscillations (37). Another important difference is that P-Ba122 has a nodal superconducting gap structure, which, due to the presence of nodal quasiparticles, has a different excitation spectrum than the fully gapped superconductivity more commonly observed in iron pnictides (38, 39).

Previous works on other iron-pnictides compounds, particularly electron-doped BaFe$_2$As$_2$, did not find anomalies in $\lambda$, although the error bars were large and the step in $x$ was coarse (40–42). Particularly in Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ (Co-Ba122), other measurements do indicate a QPT inside the SC phase. These include the microscopic coexistence of nematic and SC phases observed by high resolution x-ray scattering (43) and the coexistence of long-range magnetic order and SC by neutron scattering (44) – although neutron data on a similar electron-doped compound, Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$, were interpreted as evidence for an avoided quantum critical point (45). Other studies on optimally doped compositions report possible strong quantum fluctuations manifested in the normal state properties (46–49), such as elastic constants (47), thermopower (50), resistivity (15), elasto-resistance (23), and nuclear magnetic resonance (51).
In contrast to P-Ba122, Co-Ba122 displays a greater degree of disorder due to the disturbance of the Fe layers by transition metal doping, as it is clear from the large residual resistivities and low RRR \((18)\). Moreover, in Co-Ba122 the AFM transition is split from the nematic one, whereas in P-Ba122, neutron and x-ray scattering experiments suggest that they are simultaneous and first-order \((52)\).

Figure 1: (A) Schematics of the experimental setup. (B) SEM image of the \(x = 0.06\) sample showing a well-defined (001) plane and a sharp edge with the side surface corresponding to (100) tetragonal plane. (C) Optically detected magnetic resonance (ODMR) splitting as a function of temperature measured on warming in a 10 Oe applied magnetic field in the \(x = 0.057\) sample. The two insets show the ODMR spectra below and above \(T_c\). (D) Average of the smaller Zeeman splitting versus the applied magnetic field measured \(T = 4.5\) K approximately 10 \(\mu\)m from the edge inside the sample with \(x = 0.06\).

In this work, we use a novel minimally-invasive high sensitivity optical magnetometer technique based on nitrogen-vacancy (NV) centers in diamond to measure the magnitude of the London penetration depth \(\lambda\) at 4.5 K across the Co-Ba122 phase diagram. The high sensi-
tivity of the NV technique and the precise determination of Co-doping levels via wavelength dispersive spectroscopy (WDS) allow us to clearly identify an anomalous peak in $\lambda(x)$ inside the superconducting dome near optimal doping ($x = 0.057$). This point coincides with the extrapolated location of the AFM/SC boundary, and thus of the QPT, as determined by scattering experiments. This result demonstrates that the occurrence of a sharp peak in $\lambda$ very close to the QPT inside the dome is not limited to clean isovalently-substituted compounds with nodal superconducting gaps, but also occur in the more disordered charge-doped fully-gaped iron pnictides. This suggests that such an anomaly in the penetration depth is a more universal property of iron pnictides despite the theoretical result that a QPT alone is not enough to guarantee such an anomaly, thus shedding new light on the interplay between AFM quantum fluctuations and superconductivity in these systems.

**Results and Discussion**

The lower critical field, $H_{c1} \propto \lambda^{-2}$, is obtained by detecting the onset of the first penetration of Abrikosov vortices at the sample corners as the applied magnetic field is applied to a sample cooled in zero-field to low temperature. The measurement procedure and experimental schematics of this probing scheme are discussed in detail in our previous works (53, 54) and summarized in *Materials and methods*.

The schematic of the experimental setup is shown in Fig.1(A). The setup consists of a thin diamond plate with one surface activated with NV centers in contact with a cuboid-shaped superconducting sample. The effective demagnetization factor depends on the geometry of the sample (56), therefore it is important to use samples with well defined shapes as determined from screening through a scanning electron microscope (SEM) Fig.1(B). The magnetic induction is measured by monitoring the Zeeman splitting in optically detected magnetic resonance (ODMR). For detection of the superconducting phase transition Fig.1(C), magnetic induction is
Figure 2: (A) Temperature, \( T \), vs. cobalt concentration, \( x \), phase diagram of Ba(Fe\(_{1-x}\)Co\(_x\))\(_2\)As\(_2\) (Co-Ba122) from Refs. (43, 44, 55). Dashed lines show structural, \( T_S(x) \), and antiferromagnetic, \( T_N(x) \), transition lines. Red open squares are the values of \( T_c(x) \) from this work and red solid line is a guide for an eye. (B) Doping dependence of \( H_{c1} \) at 4.5 K across the superconducting dome. A sharp dip is located at \( x = 0.057 \).

measured near the center of the sample, whereas \( H_{c1} \) measurements are performed near the sample’s edge Fig. [D]. The overall measurement protocol is explained in detail in Refs. (53, 54).

Figure 2 shows the temperature-doping phase diagram and \( H_{c1} \) across the superconducting dome. In the underdoped region, coexisting antiferromagnetic and superconducting orders give rise to a rapid increase in \( H_{c1} \), as expected from general theoretical considerations (57). In the overdoped region, a moderate decrease in \( H_{c1} \) is observed, likely due to increasing pair-breaking scattering with larger amount of substitutional disorder and larger superconducting gap anisotropy (39). Most importantly, a distinct peak-shaped anomaly in otherwise smoothly
varying $H_{c1}$ around $x = 0.057$ is clearly observed Fig.2(B). Compared with the phase diagram in Fig.2(A), the anomaly is precisely at the point where the AFM order disappears (by extrapolation to $T = 0$). To rule out any possibility of experimental artifact, we measured two different samples of the same composition around the region of this anomaly and obtained consistent results.

We can also rule out two other features expected to occur as a function of $x$. First, since we are measuring at a fixed $T = 4.5$ K as indicated by a dash line in Fig.3(A), the relative temperature changes as $T/T_c(x)$ where $T_c(x)$ varies between 24 K and 10 K in the studied range. However, London penetration depth increases with the increase of $T/T_c$ and, therefore, cannot result in the peak close to the optimal doping where the reduced temperature is smallest. It is easy to see, using a crude estimate based on the two-fluid model, that even at $T/T_c = 0.5$, the relative change of $\Delta \lambda(0.5T_c)/\lambda(0) \leq 10\%$. Besides, $T_c(x)$ is a smooth slowly changing function. The second anomaly could be due to flux pinning. In our earlier work (58), we showed that critical current density, $j_c(x)$ peaks approaching optimal doping. In our interpretation, this is due to efficient pinning on the structural domains, which become finer, so their density increases and width decreases, both leading to the enhancement of pinning. However, in any model of pinning, critical current is inversely proportional to the London penetration depth (clearly, there is no pinning if $\lambda$ diverges) and, therefore, this mechanism would result in a dip, not the peak in $j_c(x)$. Moreover, the fact that we do not see this supports our assertion that we do not enter the vortex pinning regime at all and only detect the onset of flux penetration.

To pin down the position of the peak in $\lambda$, Fig.3(A) zooms the temperature-doping phase diagram obtained in Refs. (43, 44) near the optimal doping region. The commensurate (C) antiferromagnetic order evolves into incommensurate (IC) at the very edge of the AMF phase, as expected theoretically (59). $\lambda$ deduced from our $H_{c1}$ measurements in the corresponding region is shown in Fig.3(B). An anomalous increase in $\lambda$ is clearly visible near the composition where
Figure 3: (A) Detailed phase diagram of Co-Ba122 in the region of structural and magnetic transition lines entering the “dome of superconductivity”. The locations of commensurate, C, and incommensurate, IC, antiferromagnetic orders are adopted from Refs. (43, 44). The horizontal dashed line shows measurement temperature of 4.5 K used in this work. (B) London penetration depth $\lambda$ (4.5K) as a function of cobalt concentration, $x$. The peak in $\lambda$ at $x = 0.057$ coincides precisely with the linear extrapolation of the back-bent $T_N(x)$ under the dome to $T = 0$.

AFM order becomes incommensurate and eventually disappears. Note that the extrapolated position of the AFM-QPT inside the SC dome is not the same as if the extrapolation was done above the SC dome. The reason is the back-bending of the AFM transition line, which is observed by neutron scattering (43, 44) and attributed to the competition between AFM and SC (60).

A direct comparison of Co-Ba122 and P-Ba122 reveals striking similarities. It turns out that a simple re-scaling of the phosphorus composition (divided by a factor of 5.3) results in a
good match of all principal transition lines as shown in Fig. 4(A), where $T_N(x)$ lines were not changed between two compounds. When plotted in the re-scaled phosphorus $y$–axis, as shown in Fig. 4(B), the behavior of $\lambda(x)$ near optimal doping is remarkably similar in both compounds with similar peak values of $\sim 300$ nm. This is astounding considering how different the behavior is deep in the overdoped region. In the normal superconductor, $\lambda(0)$ depends only on the normal state properties in the clean limit and increases with disorder. Therefore, the increase of $\lambda(x)$ observed in overdoped Co-Ba122 may be attributed to a significant increase of the scattering rate due to charge doping and increasing gap anisotropy, whereas isoivalent, hence cleaner, P-Ba122 remains flat. On the underdoped side, both compositions show a steep increase of $\lambda(x)$ (for P-Ba122, see MFM measurements in Ref. (28)) due to coexisting magnetic order.

It is surprising that both compounds display such a similar behavior for the penetration depth near the putative QPT. Surprising, not only because of how different their disorder level and gap structure are (clean and nodal for P-Ba122, dirty and nodeless for Co-Ba122), but also because of the different characters of their AFM and nematic transitions. While in P-Ba122 they collapse into a single first-order transition line well before crossing the SC dome, in Co-Ba122 two separate, second-order transition lines cross the superconducting dome and continue to exist separately down to $T/T_c \approx 0.5$. For the lower temperature, the fate of these transitions is not well understood, at least experimentally. Interestingly, a recent theoretical analysis argued that, despite crossing the dome as two separate transitions, the nematic and magnetic transitions merge at a temperature $T_{merge} < T_c$ and continue as a single weakly first-order transition line down to $T = 0$ (62). This would imply a single QPT may exist in both P-Ba122 and Co-Ba122.

Importantly, even if a second-order QPT exists within the superconducting dome, be it AFM or nematic, theoretical analyses show that its critical fluctuations are expected to cause an enhancement of $\lambda$, without a divergence, upon approaching the QPT from the non-AFM side, but not necessarily a peak (31–35). This makes it even more surprising our observation that a peak
in $\lambda$ exists and nearly coincides with the extrapolated AFM-QPT in two compounds as different as P-Ba122 and Co-Ba122. Interestingly, disorder has also been proposed to be an important ingredient to trigger a peak in $\lambda$, either by promoting a SC-AFM microemulsion with frustrated Josephson couplings between SC grains (34), or by tuning the delicate balance between the competing SC and AFM orders (35). While the residual resistivity ratios of P-Ba122 and
Co-Ba122 are dramatically different, indicating that the latter is much dirtier than the former, recent NMR measurements reported evidence of significantly inhomogeneous dynamics in both compounds (63).

**Conclusions**

In conclusion, we measured $\lambda$ across the superconducting dome of Co-Ba122 using minimally-invasive and high-sensitivity optical magnetometry based on the NV centers in diamond. The measurements revealed a sharp peak in $\lambda$ in this quite disordered, charge-doped system. This peak coincides with the quantum phase transition (QPT) point found by extrapolation to $T = 0$ of the antiferromagnetic (AFM) phase boundary inside the dome of superconductivity. This result shows that the coincidence of the $\lambda$ peak with the QPT is not limited to clean isovalently-substituted compounds with nodal superconducting gaps, but also exists in electron-doped compositions with fully-gaped superconductivity, suggesting that this may be a more universal and ubiquitous manifestation of a QPT in iron-based superconductors. This puzzling observation raises important theoretical questions regarding the interplay between SC and AFM, as one does not expect that a QPT will generally lead to a peak in lambda (31–35). This result also suggests that this quantum phase transition inside the dome is very robust with respect to disorder. Whether the same manifestation is featured in other unconventional superconductors remains to be determined. For instance, while some kind of anomaly in $\lambda(x)$ is observed in the MFM measurements in hole-doped pnictide Ba$_{1-x}$K$_x$Fe$_2$As$_2$ (K-Ba122) (64), more precisely tuned and closer spaced compositions are needed to clearly resolve it (or not). Moreover, the current data available in the cuprates suggest the opposite behavior, with $\lambda$ dipping, not peaking, at the putative QPT (36). Considering how few parameters can be used to probe QPT in superconducting state, further detailed investigations are clearly warranted to establish a full and objective picture.
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Materials and methods

Sample preparation

High quality single crystals of BaCo122 were grown by using self-flux solution growth technique as described in Ref. (55). Cobalt concentration was measured by using wavelength dispersive spectroscopy (WDS). Crystals were first cleaved with razor blade into thin plates typically 50 µm thick and two shiny cleavage surfaces corresponding to (001) plane of the tetragonal structure. Cuboid samples with four sharp edges were further cleaved from the platelets along (100) and (010) tetragonal directions, see Fig. I(B). Side surfaces of the cleave are of high enough quality to make optical reflectance measurements (65) despite notable slab structure. Quality of the edges between (001) top and (100) side surfaces was controlled by SEM imaging. Only those samples were selected which had well-defined sharp edges as shown in Fig. I(B) and even (001) surfaces so that the sensor is in direct contact with the sample.
Experimental setup and determination of $\lambda$

The nitrogen-vacancy centers are embedded in a 40 $\mu$m thick electronic-grade single crystalline diamond plate (purchased from Element-Six) with [100] surface. NV centers are activated only in one side at approximately 20 nm deep from the surface. This diamond plate is placed directly onto a flat surface of a superconducting sample such that the surface containing NV-centers is in direct contact with the sample. The low-temperature measurement setup is based on Attocube AFM/CFM combo. Sensor preparation, measurement protocols and experimental setup are explained in detail in Ref. (54).

For the $H_{c1}$ measurements, the sample is cooled down to 4.5 K in zero magnetic field and then magnetic field is applied along the $z$—direction (crystallographic $c$—axis), perpendicular to the sample flat face. The confocal objective is focused on the NV centers at a spot right at the edge (inside) of the sample and optically-detected magnetic resonance (ODMR) splitting (proportional to the local magnetic induction) is measured. When the applied field is increased, above a field of first vortex penetration, $H_p$, Abrikosov vortices enter the sample cutting the sharp corners and the deviation of the signal from otherwise linear behavior is detected. We note that this field is different from the field of flux penetration calculated by Brandt (66) at which vortex segments meet at the center and the whole vortex is pushed into the sample interior by the Lorentz force resulting in a significant change in $M(H)$ dependence. In our case, the detected field corresponds to $H_{c1}$ amplified by the demagnetization correction. Specifically, using recently calculated effective demagnetization factors for $2a \times 2b \times 2c$ cuboid-shaped samples (56),

$$N = \frac{1}{1 + \frac{3c}{4a}(1 + \frac{a}{b})},$$

the lower critical field, $H_{c1}$, and, consequently, $\lambda$, are deduced from the measured $H_p$. 

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\[ H_{c1} = \frac{H_p}{1 - N} = \frac{\Phi_0}{4\pi \lambda^2} \left[ \ln \left( \frac{\lambda}{\xi} \right) + 0.5 \right], \]

where \( \Phi_0 \) is flux quantum and \( \xi \) is coherence length and numerical factor 0.5 is from the revised calculations of \( H_{c1} \) by C. R. Hu (67). Recently, Yip et al. used a similar approach to obtain critical fields in a single crystal of BaFe\(_2\)(As\(_{0.59}\)P\(_{0.41}\))\(_2\) (68).

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