Direct measurement of the temperature dependence of the magnetic penetration depth in Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$ superconductors

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Abstract
The temperature dependence of the in-plane magnetic penetration depth $\lambda_{ab}$ of Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$ single crystals is determined directly from the shielding magnetic susceptibility, measured in the Meissner region with the field parallel to the $ab$ layers. The doping levels studied cover the underdoped, optimally doped and overdoped regimes. At temperatures below 0.5$T_c$ a well-defined power-law behavior $\lambda_{ab}(T) - \lambda_{ab}(0) = AT^n$ (with $n \approx 2.5$) is observed. At lower temperatures ($T < 0.3T_c$) the data are still consistent with $n = 2$ and $A \propto T_c^{-3}$, as predicted by the strong pair-breaking scenario proposed by Gordon et al (2010 Phys. Rev. B 81, 180501(R)). The temperature dependence of the superfluid density $\rho_s \propto \lambda_{ab}^{-2}$ presents a marked positive curvature just below $T_c$, which is a sign of two-gap superconductivity. The analysis of $\rho_s(T)$ in terms of a two-gap model allowed estimation of parameters like the in-band and inter-band couplings, the relative weight of each band, and their dependence on the doping level. A comparison with $\rho_s(T)$ data obtained by using other techniques in compounds with a similar composition is also presented.

Keywords: Fe-based superconductors, penetration depth, pairing symmetry, doping level

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

1. Introduction
Study of the superconducting gap symmetry in Fe-based superconductors (FeSCs) may provide key information on the pairing interaction in these unconventional superconductors. Therefore, works analyzing the differences in the gap structure among the FeSC families, and probing its evolution with the type and concentration of dopants, are at the forefront of the research in these materials [1]. One of the two fundamental lengths in superconductors, the magnetic penetration depth $\lambda$, is directly related (through its temperature dependence) to the superconducting energy gap, and constitutes a useful tool to obtain information about its symmetry [2]. The absolute value and temperature dependence of $\lambda$ have recently been investigated in FeSC by using different experimental techniques, including a tunnel diode resonator (TDR) [3–25], muon-spin rotation ($\mu$SR) [26–32], microwave cavity perturbation [32], two-coil mutual inductance [33], the lower critical field ($H_{c1}$) [24, 34, 35], THz conductivity [36, 37], surface impedance [38–41], and local probes as magnetic force microscopy (MFM) [42, 43], scanning SQUID microscopy (SSS) [42–45], and miniature Hall sensors [24]. Currently

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there is considerable consensus that the low-temperature behavior of $\lambda$ may be described by a power law

$$\lambda(T) = \lambda(0) + AT^n,$$  \hspace{1cm} (1)

where the exponent value depends on the particular FeSC family and, within the same family, on the type and concentration of the dopants. It is also well established that, in the most studied FeSC families (e.g., 111, 112, 1111), when phosphor is used as the pnictide (completely or partially replacing the arsenic), $n$ is close to 1, which is consistent with a nodal superconducting order parameter $\lambda$.

The experimental details are presented in section 2. The low-temperature behavior of $\Delta\lambda_{ab}$ is analyzed in section 3.1. The superfluid density, obtained by using the $\lambda_{ab}(0)$ values in the literature, is analyzed in the full temperature range below $T_c$ in section 3.2. Finally, the concluding remarks are presented in section 4.

2. Experimental details and results

The Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$ crystals were grown by the self-flux method. Their nominal Ni doping levels were $x = 0.0375$ (underdoped, ud), 0.05 (optimally doped, op), and 0.075 (overdoped, ov). The details of the growth procedure and a thorough characterization may be found in [52]. To avoid the complications associated with demagnetizing effects (see below) we used plate-like single crystals (typically $1 \times 1 \times 0.02$ mm$^3$, see table 1) with the FeAs $ab$ layers parallel to their largest faces. They were cleaved from larger crystals by using adhesive tape. The surface irregularities were of the order of 50 nm in depth (as determined by AFM), and the uncertainty in the crystal thickness $L_c \approx 20 \mu$m (the length relevant for the analysis) was below 1%.

The magnetic susceptibility with $H \parallel ab$, $\chi_{ab}$, was measured in several crystals of each composition with a Quantum Design SQUID magnetometer (model MPMS-XL). For this we used a quartz sample holder (0.3 cm in diameter, 22 cm in length) to which the crystals were glued with a minute amount of GE varnish. Two plastic rods at the holder ends ($\sim 0.3$ mm

This technique was previously used to determine the in-plane magnetic penetration depth in a high-$T_c$ cuprate superconductor, see [49]. Some problems arising in the data analysis are commented on in [50].
smaller than the sample space diameter) ensured an alignment better than 0.1°. However, the presence of the Ge varnish may introduce an additional uncertainty in the crystal orientation, which effect will be commented on below. The samples were zero-field cooled (ZFC) by using the ultra-low-field option, which includes a shield for the Earth’s magnetic field and a conventional coil to compensate the superconducting coil’s remnant field down to the 10⁻⁵ Oe level. The magnetic moment \( m \) was measured against temperature (from 2 K up to above \( T_c \)) in the presence of a 5 Oe applied magnetic field, which effect will be commented on below. The samples were studied of the \( (\text{Ni}_{0.479}, \text{Cu}_{0.895}) \) crystals' dimensions in table 1 it may be approximated directly from the \( \chi_{ab} \) layers, the measured magnetic susceptibility would be

\[
\chi_{\| \text{meas}} = \frac{\chi_{\|}}{1 + \chi_{\|}D_{\|}} \cos^2 \alpha + \frac{\chi_{\perp}}{1 + \chi_{\perp}D_{\perp}} \sin^2 \alpha, \tag{4}
\]

where \( D_{\|} \) is the demagnetizing factor for \( H \parallel ab \). From the crystals’ dimensions in table 1 it may be approached as \( D_{\|} \approx \pi L_c / 4L_{ab} \approx 0.015 \) and \( D_{\perp} \approx 1 - 2D_{\|} \approx 0.97 \). As \( \chi_{\perp} \approx -1 \) up to very close to \( T_c \), this expression may be approximated by

\[
\chi_{\||\text{meas}} \approx \chi_{\|} - \frac{\sin^2 \alpha}{1 - D_{\perp}}, \tag{5}
\]

i.e., the difference between the measured magnetic susceptibility and the actual \( \chi_{\|} \) is a temperature independent value (of the order of \( 10^{-2} \) for \( T \approx 1^\circ \)). While it may difficult to determine the absolute value of \( \lambda_{ab} \), it allows determination of its temperature dependence \( \Delta \lambda_{ab}(T) = \lambda_{ab}(T) - \lambda_{ab}(0) \)

| Ni % | Crystal | Dimensions (mm³) | \( T_c \) (K) | \( \Delta T_c \) | \( \chi_{\| \text{meas}}(0) \) |
|------|--------|----------------|-------------|-------------|------------------|
| 3.75 | ud1    | 0.75 × 0.4 × 0.015 | 10.5 | 0.047 | -0.479 |
|      | ud2    | 0.5 × 0.35 × 0.010 | 10.6 | 0.037 | -0.895 |
|      | ud3    | 0.9 × 0.55 × 0.013 | 10.7 | 0.043 | -0.694 |
| 5    | op1    | 1.7 × 0.9 × 0.021  | 19.7 | 0.005 | -0.992 |
|      | op2    | 1.35 × 1.1 × 0.027 | 19.6 | 0.010 | -0.994 |
|      | op3    | 1.2 × 1.0 × 0.020  | 19.7 | 0.005 | -0.992 |
| 7.5  | ov1    | 0.65 × 1.15 × 0.021 | 14.3 | 0.076 | -0.985 |
|      | ov2    | 1.2 × 1.5 × 0.021  | 14.1 | 0.049 | -0.984 |
|      | ov3    | 0.6 × 1.15 × 0.024 | 14.8 | 0.080 | -0.984 |

3. Data analysis
3.1. Low-temperature behavior of the in-plane penetration depth
In view of the crystals’ geometry, the relationship between \( \chi_{\|} \) and \( \lambda_{ab} \) may be approximated by [54]

\[
\chi_{\|} = -1 + \frac{2\lambda_{ab}}{L_c} \tanh \frac{L_c}{2\lambda_{ab}}. \tag{3}
\]

This expression would allow determination of the absolute value of \( \lambda_{ab} \) directly from the \( \chi_{\|} \) data in figure 2. However, even a small crystal misalignment may lead to a non-negligible contribution coming from the field component perpendicular to the Fe-layers. Denoting by \( \alpha \) the possible angle between \( H \) and the crystal \( ab \) layers, the measured magnetic susceptibility would be

\[
\chi_{\||\text{meas}} = \frac{\chi_{\|}}{1 + \chi_{\|}D_{\|}} \cos^2 \alpha + \frac{\chi_{\perp}}{1 + \chi_{\perp}D_{\perp}} \sin^2 \alpha, \tag{4}
\]

where \( D_{\|} \) is the demagnetizing factor for \( H \parallel ab \). From the crystals’ dimensions in table 1 it may be approached as \( D_{\|} \approx \pi L_c / 4L_{ab} \approx 0.015 \) and \( D_{\perp} \approx 1 - 2D_{\|} \approx 0.97 \). As \( \chi_{\perp} \approx -1 \) up to very close to \( T_c \), this expression may be approximated by

\[
\chi_{\||\text{meas}} \approx \chi_{\|} - \frac{\sin^2 \alpha}{1 - D_{\perp}}, \tag{5}
\]

i.e., the difference between the measured magnetic susceptibility and the actual \( \chi_{\|} \) is a temperature independent value (of the order of \( 10^{-2} \) for \( T \approx 1^\circ \)). While it may difficult to determine the absolute value of \( \lambda_{ab} \), it allows determination of its temperature dependence \( \Delta \lambda_{ab}(T) = \lambda_{ab}(T) - \lambda_{ab}(0) \)
with accuracy. As the crystal thicknesses $L_c$ are of the order of 20 $\mu$m, and the reported values of $\lambda_{ab}(0)$ are smaller than 1 $\mu$m (see below), it may be safely approximated $\tanh(L_c/2\lambda_{ab}) \approx 1$ up to very close to $T_c$ (typically for $T < 0.9T_c$ it is found that $\lambda_{ab}(T) < 0.2L_c$ and the approximation is accurate within 1%). Then, from equations (3) and (5) it follows that

$$\Delta\lambda_{ab}(T) \approx \frac{L_c}{2} \left[ \chi^{\text{meas}}_\parallel(T) - \chi^{\text{meas}}(0) \right].$$

Taking into account the above mentioned resolution of our measurement system and the geometry of the crystals used, our technique allows us to detect changes in $\lambda_{ab}$ of the order of $\sim$5 nm, slightly larger than the typical resolution of the TDR technique (about 1 nm, see e.g., the figures in [12] with TDR measurements in the same compounds).

Detail of the $\chi^{\text{meas}}_\parallel(T)$ behavior at low temperatures is presented in the insets of figure 2. The low-temperature saturation values were determined by fitting a power law in the temperature region up to $\sim 0.5T_c$ (solid lines). The resulting $\chi^{\text{meas}}(0)$ values are compiled in table 1. Optimally doped and overdoped crystals present $|\chi^{\text{meas}}_\parallel(0)|$ values typically 0.02 larger that the ones expected in view of the $\lambda_{ab}(0)$ values in the literature (260 ± 50 nm in crystals with 5% Ni, and 340 ± 60 nm in crystals with 7.5% Ni) [24]. This is consistent with crystal misalignments of about $\alpha \sim 1.5^\circ$. Crystals with 3.75% Ni present a larger scattering in the $\chi^{\text{meas}}(0)$ values that can hardly be attributed to crystal misalignments: as for this composition $\lambda_{ab}(0) = 450 ± 80$ nm [24], one would have to assume $\alpha \approx 2°-7°$. The scattering in the $\chi^{\text{meas}}_\parallel(0)$ values of UD crystals could then be attributed to a possible presence of non-superconducting domains in the crystals, maybe associated with the proximity of this doping level to the non-superconducting phase.

The low-temperature behavior of $\Delta\lambda_{ab}(T)$ for all crystals studied is presented in figure 3. The data corresponding to crystals with the same composition roughly fall on the same curve, even in the case of the crystals with 3.75% Ni. The solid lines are fits of a power law,

$$\Delta\lambda_{ab}(T) = A \left( \frac{T}{T_c} \right)^n,$$

(7)

to the set of data for each composition up to $T/T_c = 0.5$. The fit qualities are excellent in all the reduced-temperature range, and lead to the amplitudes and exponents shown in figure 4(a). $n$ is about $\sim$2.5 up to the optimal-doping level, and decreases to $\sim$2.3 for $x = 0.075$. It has been proposed that impurity scattering would strongly affect the low-temperature behavior of $\Delta\lambda_{ab}$. In particular, superconductors with a fully gapped order parameter or with a d-wave symmetry would change their exponential/linear temperature dependences in the clean limit to a power law with $n$ approaching 2 in the dirty limit [2]. Our present results, with $n$ values slightly above $n = 2$, would then be consistent with a nodeless order parameter for all doping levels, affected by the presence of impurity scattering.

6 The consistency between the $\Delta\lambda_{ab}(T)$ data in the three underdoped crystals justifies the applicability of equation (3) also in these samples in spite of the fact that, as commented above, they may present a distribution of non-superconducting domains. This may be explained by taking into account that non-superconducting domains in the sample’s interior are completely screened and have little effect on the measured ZFC magnetic susceptibility. The global $|\chi_\parallel|$ reduction observed in some of these samples may then be attributed to the presence of interconnected non-superconducting domains leading to large (of the order of the sample’s thickness) unscreened areas within the sample. Provided that they are much larger than $\lambda_{ab}$, their presence would not appreciably affect the $\chi_\parallel$ temperature dependence given by equation (3).

7 Note that for the underdoped samples the temperature range is restricted to $T > 0.2T_c$, and the conclusions about the low-temperature behavior are less robust than in the optimally doped and overdoped samples.
Figure 3. (a) Low-temperature behavior of the in-plane magnetic penetration depth (in excess of the $T \rightarrow 0$ K value) for all crystals studied. The solid lines are fits to a general power law up to $T/T_c = 0.5$. The dashed lines are fits to a quadratic power law up to $T/T_c = 0.3$. (b) Low-temperature detail in log–log scale for the overdoped crystals. The solid and dashed lines are the same as in (a). The dot-dashed line is a fit to a power law up to $0.3 T_c$ by using the $n$ value found in [13, 12].

It has been calculated that in the dirty limit the amplitude in the quadratic power law should be proportional to $T_c^{-3}$ [46]. A fit to the experimental data by fixing $n = 2$ (dashed lines in figure 3(a)) is still reasonably good up to $T/T_c \approx 0.3$. Also, as is shown in figure 4(b), the resulting amplitude follows the predicted $T_c^{-3}$ dependence, and is close to the values found in several FeSC families including Ba$(Fe_{1-x}Ni_x)_2As_2$ [24, 46]. This reinforces our above conclusion, and suggests that our crystals are close to the limit of strong impurity scattering.

Our results are consistent with recent TDR measurements in crystals with similar compositions [24]. They are also coherent with the results of Kim et al [18], which showed that in optimally doped Ba$(Fe_{1-x}Ni_x)_2As_2$ $n$ diminishes from $n = 2.5$ to $n \sim 2$ when defects are progressively introduced into the crystals through heavy-ion irradiation. However, there is a notable difference from the TDR measurements by Martin et al [12, 13] who found that $n$ falls significantly below 2 in similarly overdoped crystals from the 122 family (BaFe$_{2-x}$M$_x$As$_2$ with M = Pd, Co, Co+Cu, and also Ni). This result led these authors to suggest that the superconducting gap is not universal even within the same 122 family, and that in the overdoped regime it may become highly anisotropic and nodal. In agreement with this proposal, measurements of the fluctuation-induced magnetoresistance above $T_c$ in crystals from the same batches as the ones used in the present work [55] showed that the superconducting anisotropy factor increases with $x$ from $\gamma \approx 2$ at optimal doping up to $\gamma \approx 10$ at high doping levels (7.5% Ni). In addition, studies of the low-temperature specific heat in Ba$(Fe_{1-x}Co_x)_2As_2$ [56] and of point-contact Andreev reflection in Ba$(Fe_{1-x}Ni_x)_2As_2$ [57] also suggest the possible presence of nodes in the overdoped region. However, as is shown in the detailed log–log representation of figure 3(b), exponents below $n = 2$ are clearly out of the experimental uncertainty in all our overdoped crystals.

3.2. Temperature dependence of the superfluid density

A more complete analysis of the superconducting gap structure may be made through the temperature dependence of the normalized superfluid density in the complete temperature range below $T_c$. It may be obtained through

$$\rho_s(T)/\rho_s(0) = \left(1 + \frac{\Delta \lambda_{ab}(T)}{\lambda_{ab}(0)}\right)^{-2}. \quad (8)$$
In this expression we used the $\lambda_{ab}(0)$ versus $T_c$ dependence for the Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$ system derived in [24] from local Hall magnetometry and, independently, from specific heat measurements. It leads to $\lambda_{ab}(0) = 450 \pm 80$ nm for 3.75% Ni, $\lambda_{ab}(0) = 260 \pm 50$ nm for 5% Ni, and $\lambda_{ab}(0) = 340 \pm 60$ nm for 7.5% Ni. These values are consistent with the ones obtained in [58] from optical reflectometry, and with the ones for Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ with equivalent electron concentrations obtained in [16] from TDR. The resulting $\rho_s(T) / \rho_s(0)$ is presented in figure 5 where, for comparison, the results for single-band s-wave and d-wave superconductors are also included. In contrast to these conventional scenarios, the superfluid density presents a notable positive curvature for temperatures just below $T_c$ for all studied doping levels, which is a sign of two-gap superconductivity [59]. This curvature is more pronounced in the overdoped and underdoped crystals, although the differences are close to the uncertainties in the $\lambda_{ab}(0)$ values (a representative example for one of the optimally doped crystals is shown as a shaded area in figure 5(b)).

A quantitative analysis of our $\rho_s(T) / \rho_s(0)$ data is presented in figure 6 in the framework of a self-consistent isotropic s-wave two-gap model (the so-called gamma model) [59]. This model depends on parameters like the in-band ($\lambda_{11}$ and $\lambda_{22}$) and inter-band ($\lambda_{12}$) couplings, the relative density of states ($n_1$ and $n_2 = 1 - n_1$), and the parameter $\gamma$ determining the partial contribution to the superfluid density from each band, $\rho_s = \gamma \rho_{s1} + (1 - \gamma) \rho_{s2}$. This clean s-wave model should not work at low temperatures, where we observe a power-law behavior, but it is expected to provide a reasonable description at higher temperatures [14]. To limit the number of fitting parameters, we have considered that both bands have the same partial density of states, $n_1 = n_2 = 0.5$, while $\lambda_{11}$ was set to give the correct $T_c$, assuming a Debye temperature of 150 K [60]. The lines in the main panel of figure 6 are the fits to the $\rho_s(T) / \rho_s(0)$ data for temperatures above ~0.2$T_c$. The resulting fitting parameters are presented in table 2, and the corresponding temperature-dependent superconducting gaps, $\Delta_1(T)$ and $\Delta_2(T)$, are presented in the insets. In other FeSCs $\Delta_1 / \Delta_2 \approx 2$ and, in view of the small $\gamma$ value, the main contribution to $\rho_s$ comes from the band with a smaller gap [14, 19, 21]. However, the existence of the larger gap and a small inter-band coupling is needed to account for the high $T_c$.

To the best of our knowledge, there are no $\rho_s(T)$ data for Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$ to compare with. However, some works have studied the very similar Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$. In the TDR measurements by Gordon et al in these compounds [16], an analogous positive curvature was also observed near $T_c$ (see the pink dotted line in figure 5(b) for the optimal-doping level). In the same work a similar dependence of $\rho_s(T) / \rho_s(0)$ on the doping level was also found, which was attributed to an enhanced gap anisotropy when departing from the optimal doping. However, authors using other techniques have obtained significantly different results in the same compounds [28, 32, 33, 41, 43]. As an example, in figure 5(b) we compare the data in the literature for optimally doped Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ with our data for Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$. As may be seen, in the data from [28, 32, 33, 43] the positive curvature is much less pronounced or even is not observed, while in the most recent data [41] it is larger. The notable differences observed by using the same technique in crystals coming from different batches are also significant (see [2, 16]); this was attributed to differences in the impurity scattering between the samples used.

**Table 2.** Parameters arising in the analysis of the superfluid density in the framework of the two-gap gamma model. See the main text for details.

| Ni % | $n_1$ | $\lambda_{11}$ | $\lambda_{12}$ | $\gamma$ |
|------|-------|----------------|---------------|---------|
| 3.75 | 0.5   | 0.68           | 0.32          | 0.120   |
| 5    | 0.5   | 0.84           | 0.49          | 0.148   |
| 7.5  | 0.5   | 0.75           | 0.35          | 0.148   |

![Figure 5](image-url)
24], but contrasts with the results of Martin et al [18, 27] over other experimental procedures in the same compounds [12, 13]. This is in agreement with results obtained by using a nodeless order parameter in the limit of strong impurity scattering. This is roughly proportional to $T$ almost independent of the doping level and an amplitude $\lambda(T)$, and $\Delta_2(T)$, are presented in the insets. For details, see the main text.

4. Conclusions

We have shown that measurements of the shielding magnetic susceptibility in the Meissner region allow a direct and reliable determination of the temperature dependence of the in-plane magnetic penetration depth in easily exfoliable Fe-based superconductors. By using high-quality Ba(Fe$_{1-x}$Ni$_x$)$_2$As$_2$ single crystals we studied the evolution of $\Delta_{ab}(T)$, $\lambda_{ab}(T)$ with the two-gap gamma model of Kogan et al [59] (solid lines). We only include data verifying $\lambda_{ab}(T) < 0.2L_c$, for which equation (6) is correct within 1%. The shaded areas represent the uncertainties in the $\lambda_{ab}(0)$ values used. The resulting temperature-dependent superconducting gaps, $\Delta_1(T)$ and $\Delta_2(T)$, are presented in the insets. For details, see the main text.

When combined with the $\lambda_{ab}(0)$ values in the literature, our measurements also allowed us to study the temperature dependence of the superfluid density $\rho_s \propto \lambda_{ab}^{-2}$ in the full temperature range below $T_c$. We find a marked positive curvature of $\rho_s$ in a wide temperature region below $T_c$ (slightly increasing for doping levels away from the optimal one) which is interpreted in the framework of a self-consistent isotropic $s$-wave two-gap model (the so-called gamma model) [59]. These results agree with the TDR measurements of Gordon et al [16] in the very similar Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$ system, but present notable differences with other works in the same compound [2, 8, 10, 11, 12]. More measurements are needed in order to determine the evolution of the gap symmetry with the doping level and across the different FeSC families.

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