Visualization of compensating currents in type-II/1 superconductor via high field cooling

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ABSTRACT

The morphology of vortex lattice domains in bulk type-II/1 superconductors is of central interest for many areas such as fundamental condensed matter physics, engineering science, and the optimization of materials for high transport current superconductivity applications. Here, we present a comprehensive experimental study of a single crystal niobium in the intermediate mixed state and Shubnikov phase with two complementary neutron techniques: high resolution polarized neutron imaging and small-angle neutron scattering. In this way, we were able to identify and visualize the occurrence of compensating currents, the flux line closure, and the freezing of the vortex spacing during the process of field cooling and high field cooling. With the combination of complementary neutron techniques, it was possible to add insights into the quest for the understanding of the flux pinning and nucleation of vortices in type-II/1 superconductors during the process of field cooling and high field cooling.

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In the last decade, the spatial resolution capabilities of neutron imaging have brought additional insights into the investigation of magnetic phenomena in matter.1–3 In particular, the thermodynamics of the phase transition between normal and superconducting states and also the flux pinning and trapping in superconductors due to the their peculiar nature of local magnetic interactions have spurred interest of neutron imaging researchers.

Small-angle neutron scattering (SANS) is a well-known established method, which provides information about the size and shape of structures embedded in a homogeneous matrix. While SANS probes the Abrikosov vortex lattice (VL) and yields information, averaged over the illuminated region, about its morphology and spacing covering length scales from 10 to hundreds of nm, the polarized neutron imaging (PNI) technique allows us to assess complementary information about the magnetic field distributions.27–29

Superconductors are classified by the Ginzburg–Landau parameter κ into type-I (κ < 1/√2) and type-II (κ > 1/√2).30,31 Here, the Ginzburg–Landau parameter is defined as κ = λ_f/λ_L, with λ_f being the London penetration depth and ξ_f the superconducting coherence length. The phase diagram of type-II superconductors is characterized by the Meissner state (MS), which also occurs in type-I superconductors, and the Shubnikov phase (SH), aside from the normal conducting state (NS), as shown in Figs. 1(a) and 1(c). Furthermore, superconductors characterized by κ ≥ 1/√2, such as niobium, are subclassified as type-II/1, while κ ≥ 1/√2 as type-II/2. The vortex lattice of type-II/1 superconductors shows both attractive and repulsive intervortex interaction components, as opposed to type-II/2, which is characterized by a purely repulsive behavior.32 This peculiar behavior of the VL of type-II/1 superconductors can be observed in the intermediate mixed state (IMS), in which a domain structure develops in the form of a mixed
Numerous studies, and they are still a subject of investigations. The origin and thermodynamics of the IMS have been the subject of investigations. The critical field, respectively.

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Recent investigations, based on a neutron multiscale approach, of the IMS state in niobium, focused on the process of field cooling FC. In order to extend the results of the previous investigations, this study is based on the combination of magnetization, flux pinning, and line closure in a type-II/1 superconductor during the process of FC and high field cooling HFC. In order to extend the results of the previous investigations, this study is based on the combination of magnetization, flux pinning, and line closure in a type-II/1 superconductor during the process of FC and high field cooling HFC.

The sample is a low purity single crystal disk of niobium with 12 mm diameter, 2 mm thickness, and a residual resistivity ratio (RRR) of ≈10, which has been characterized by a quantum design physical property measurement system (PPMS). The (111) crystallographic axis is perpendicular to the face of the Nb disk. This kind of low purity specimen can be considered a realistic model material for practical applications of superconductors.

The magnetization curves of the Nb sample are shown in Fig. 2 as a function of the applied magnetic field, perpendicular to the face of the disk, for various temperatures between 4 and 9 K. Such magnetization curves are typical for type-II/1 superconductors: the behavior of the magnetization hysteresis loop is determined by an entrance of the VL into the sample and by trapping the magnetic field. The broad hysteresis loop is characteristic of a strong pinning behavior, a hallmark of superconductors with a low RRR. The demagnetization factor (D) for a disk with the magnetic field applied along the cylindrical axis has been estimated to be ≈0.88, calculated according to

\[
P = \frac{I_s - I_l}{I_s + I_l},
\]

where \(I_s\) and \(I_l\) are the transmitted intensities measured behind a spin analyzer, with parallel (+) and antiparallel (−) alignment to the probed polarization direction. The precession angle \(\phi\) is given by the path integral as follows:

\[
\phi = \omega_L t = \frac{\gamma_n B t}{v} \int B(s) ds = \frac{\gamma_n m_n A}{h} \int B(s) ds,
\]

where \(\omega_L\) is the Larmor frequency, \(t\) is the time a neutron spends in the magnetic field \(B\), \(\gamma_n\) is the gyromagnetic ratio of the neutron, \(v\) is the velocity, \(m_n\) is the neutron mass, \(A\) is the wavelength, and \(h\) is the Planck constant. In order to detect a precession angle, the neutrons have to experience a non-adiabatic transition, which depends on the magnetic field gradient and neutron energy. The adiabatic regime is defined by the following condition:

\[
\frac{1}{B} \left( \frac{dB}{dt} \right) \ll \omega_L.
\]
where $r$ and $h$ are the radius and the thickness of the cylinder. The SANS measurements have been carried out at SANS-I, at PSI, via rocking scans over $\pm 2^\circ$. The data have been corrected for a high temperature background using a background measurement in the normal state. The external magnetic field is applied along the neutron beam direction and perpendicular to the face of the Nb disk. A sixfold pattern is observed for the illuminated area of the sample, corresponding to the central inner part of the Nb disk $\approx 8$ mm in diameter, as expected from the literature. The properties of the VL extracted from the SANS during the FC measurements, depicted in Fig. 3(a), show a plateau in the IMS state and the characteristic $g_{VL}$ behavior in the SH phase for magnetic field $B \approx H_c \approx 45$ mT, in agreement with the literature. Here, $g_{VL}$ is the reciprocal lattice vector, i.e., the inverse of the vortex lattice parameter $a_{VL}$, and can be expressed as:

$$g_{VL} = \frac{4\pi}{\sqrt{3}a_{VL}} = \sqrt{\frac{8\pi^2 B_{out}}{3\Phi_0}},$$

where $\Phi_0$ is the magnetic flux quantum and $B_{out}$ depends on the demagnetization $D$ of the sample. However, in the probed magnetic field range, the sample does not show the characteristic drop at a very low field due to the flux expulsion in the MS, mainly due to pinning and impurities, yellow region in Fig. 3(a). This does not allow us to estimate the value of the field for the IMS-MS phase transition. The field dependence of the integrated scattering intensity in the FC measurements, as illustrated in Fig. 3(b), shows the characteristic peak at the IMS-SH phase transition, in good agreement with the literature. The HFC measurements, on the other hand, show a freezing of $g_{VL}$ at the initial value, i.e., when the superconducting phase is accessed at 80 mT, without exhibiting any discontinuities throughout the SH-IMS phase transition. In a similar manner, the integrated intensity distribution, which is proportional to the magnetic flux structures in the sample, is constant for HFC, as shown in Fig. 3(b), and indicates no nucleation process of vortex domains, in agreement with the results reported by Bykov et al.

The PNI measurements have been carried out at the polarized cold neutron beamline BOA, at PSI. The monochromatic neutron beam is polarized along the vertical axis with an overall polarization of $\approx 90\%$ at $\lambda = 3.5$ Å. A spatial resolution of 130 $\mu$m has been measured with a Siemens star test object. The cryomagnet used for the measurements generates a magnetic field that is aligned along the propagation direction of the neutrons, and it is adiabatically coupled with the incoming neutron spin polarization and the spin analyzer. A complete sequence covering the magnetic field ranging from 20 mT to 80 mT has been acquired for both FC and HFC. In Figs. 4(a)–4(d), a selection of images is shown. Throughout the whole FC procedure, only a weak signal has been detected, visible only at the edge of the disk, as shown in Figs. 4(a) and 4(c). This indicates that during the SH-IMS phase transition in the FC procedure, the neutron polarization is adiabatically coupled, as defined in Eq. (3), with the external magnetic field applied to the sample, which, thus, has no

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**FIG. 2.** PPMS data of the Nb disk. Magnetization as a function of the applied external magnetic field perpendicular to the face of the disk, measured for different temperatures between 4 K and 9 K in the range between $\pm 350$ mT. Here, the initial magnetization curves after zero field cooling (ZFC) and three quarter of a hysteresis loop are shown. The demagnetization factor $D$ is shown in gray as $M = -DH_{ext} = -0.99H_{ext}$.

**FIG. 3.** SANS results. (a) Reciprocal lattice vector $g_{VL}$ measured by SANS as a function of increasing and decreasing applied magnetic fields through FC (blue) and HFC (orange). For a high field, the FC data points are proportional to $\sqrt{B}$, characteristic of the SH phase. (b) Integrated scattering intensity curves. The blue data points have been acquired via independent FC procedures. The orange data points have been measured via the HFC procedure. $H_c \approx 45$ mT defines the boundary between IMS (red) and SH (green). The MS (yellow) has not been probed during the SANS measurements.
perpendicular component to the polarization vector; hence, no spin flipping precession occurs and the polarization is equal to +1, as shown in Fig. 4(a).\(^{29}\) Hence, it serves as the reference point for the comparison of the images acquired during the HFC hysteresis loop. The PNI images of the HFC, on the other hand, show a field dependent behavior emerging at the periphery of the disk, as illustrated in Figs. 4(b)–4(d). This local decrease in polarization during the HFC becomes more pronounced for weaker fields, and it shifts toward larger radii, extending beyond the edge of the Nb disk, as shown in Figs. 4(d) and 4(e). The fact that a polarization of \( P \approx 0 \) % is reached, as illustrated in Fig. 4(e) for the case of HFC at 20 mT, indicates a non-adiabatic transition, according to Eq. (3), due to the strong gradient generated by the swirling of the magnetic field, as depicted in Fig. 1(b).

While the VI cannot be directly resolved in real space by PNI, a comprehensive interpretation of the VI structure can be inferred via the SANS results, which show the characteristic SH-IMS phase transition in FC followed by the subsequent nucleation of an irregular domain structure. In contrast, for HFC, no signs of the SH-IMS phase transition have been observed experimentally, and \( g_{UL} \) and the integrated scattering intensity indicate a freezing of the VL at the initial value of the thermodynamic path, which does not lead to a vortex domain formation, mainly due to pinning. The PNI results do not show a stationary system during the freezing of the VL for HFC, and a clear field dependent ring appears, which protrudes beyond the edge of the disk. Since circulating currents appear only during ZFC, the signal induced by the HFC procedure leads to the compensating currents and the flux line closure as depicted in Fig. 1(b). A similar situation has been observed using the Faraday effect in high T\(_c\) superconductors.\(^{38}\)

In conclusion, we have shown how the combination of the results obtained with two complementary neutron techniques, SANS and PNI, provides additional insights into the thermodynamics of low purity type-II/1 superconductors by characterizing the VI throughout the thermodynamic path and visualizing the occurrence of compensating currents and flux line closure.

See the supplementary material for the SANS diffraction patterns and for the complete dataset of the PNI images.

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**DATA AVAILABILITY**

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

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