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Experimental observation of lattice distortions due to a flux line lattice in niobium

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Abstract. A polarized neutron scattering investigation of the flux line lattice in the type-II superconductor niobium is reported. A modulation of the nuclear lattice has been detected, and the magnitude of the first Fourier component of the lattice distortion established relative to the magnitude of the magnetic scattering. This constitutes the first experimental observation of lattice distortions due to the presence of magnetic flux lines within the bulk of a type-II superconductor. Using a simple microscopic model the lattice distortion in niobium is estimated. A new mechanism is suggested for the coupling of the flux line lattice to the crystallographic lattice. The experimental technique opens up the possibility of investigating the microscopic mechanism of flux line - nuclear lattice interactions, in particular the pinning of flux lines within the bulk of a type-II superconductor.

PACS. 74.60.-w Type-II superconductivity – 78.70.Nx Neutron inelastic scattering

1 Introduction

Interest in either the fundamental or applied aspects of superconductivity requires a more detailed understanding of the interaction between the magnetic flux lines in a type-II superconductor. For many applications it is vital to minimize the motion of the flux lines which give rise to dissipation within the system. In particular the critical currents, which are determined by the pinning forces of the magnetic flux line, need to be understood. In order to optimize the application of superconductors identification of the basic interaction between the flux line lattice (FLL) and the nuclear structure will permit not only an understanding of the structure of the flux line lattice but also a determination of its orientation relative to the nuclear lattice.

The interaction between the periodic lattice of flux lines and the nuclear lattice has been investigated using spin polarized neutrons in a small angle scattering experiment. The presence of a FLL gives rise to a distortion of the nuclear lattice. This distortion has been measured and the size of the effect determined for niobium within the intermediate state. The present investigation opens up a completely new area of research addressed at the interaction between the periodic magnetic field modulation (which is coupled to the superconducting order parameter) and the nuclear structure of the lattice.

The first experimental observation of a flux line lattice in a type-II superconductor was carried out using decoration techniques [1]. These experiments confirmed the triangular arrangement of the FLL and established the existence of a FLL as predicted by Abrikosov [2]. More recently, the STM investigation of individual flux lines and their internal structure has opened new possibilities [3] such as the investigation of bound states within the vortex core. However, such investigations are limited to the surface of the material. In contrast, neutron scattering experiments are able to investigate the flux line lattice in the bulk of the superconductor and provide detailed information of their spatial arrangement. de Gennes and Matricon [4] were the first to suggest the investigation of a flux line lattice (FLL) by neutron scattering. Subsequently Cribier *et al.* [5] were able to observe Bragg scattering which originated from the periodic arrangement of flux lines inside a superconductor. These experiments were followed by systematic studies of various aspects of the FLL, such as the motion of flux lines [6]. For a recent review of the subject see Brandt [7]. These investigations have been extended to other groups of materials including high-Tc superconductors [8] and Heavy Fermion systems [9].

The magnetic moment of the neutron interacts with the magnetization density within a sample and if the magnetization density is arranged in a periodic fashion, as is the case for a flux line lattice in a type-II superconductor, the neutrons are Bragg scattered. Due to the large lattice constant of the FLL (with lattice constants of the order of 1000 Å) the Bragg reflections occur at small scattering angles in diffraction experiments.

The neutron scattering from a FLL is magnetic in origin. However, if the nuclear lattice responds with a small

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distortion to either the modulation of the magnetic field or to the modulation of the superconducting order parameter with a period equal to that of the FLL (*e.g.* due to either magneto- or suprastriction) a small nuclear scattering contribution occurs. Thus the Bragg reflections which arise due to the flux line lattice in type-II superconductors will, in principle, have both a magnetic as well as a nuclear contribution although the nuclear contribution to the scattering is expected to be small. For the homogeneous state of a type-II superconductor and in the absence of a FLL such a nuclear distortion has been observed as a discontinuity at T_c, for example, in thermal expansion measurements of superconductors in a magnetic field. The typical length changes are small and of the order of $\Delta l/l \approx 10^{-5}$–$10^{-7}$. The smallness of the deformation of the nuclear lattice has led to the assumption that the interaction of the magnetic and nuclear lattice is too small to observe using neutron scattering. Although small, the distortions can be observed using spin polarized small angle neutron scattering. This experiment makes use of the interference term occurring in the coherent elastic cross section. If the magnetic (FLL) and nuclear lattice have the same spatial periodicity then the magnetic and nuclear scattering constructively interfere. The magnitude of this scattering is dependent on the direction of the incident neutron polarization and disappears for unpolarized neutrons. Spin dependent polarized neutron scattering has been combined with the small angle scattering technique to investigate the lattice distortions associated with the flux line lattice in niobium. By measuring the flipping ratio, which is defined as the ratio of intensities observed with the neutron spin oriented either parallel or antiparallel to the external magnetic field direction, the interference term can be unambiguously determined. A value of this ratio different from 1 has been observed for neutron scattering from the FLL Bragg peaks. Such a measurement is unique confirmation of the presence of a nonzero nuclear contribution to the Bragg peaks of the FLL.

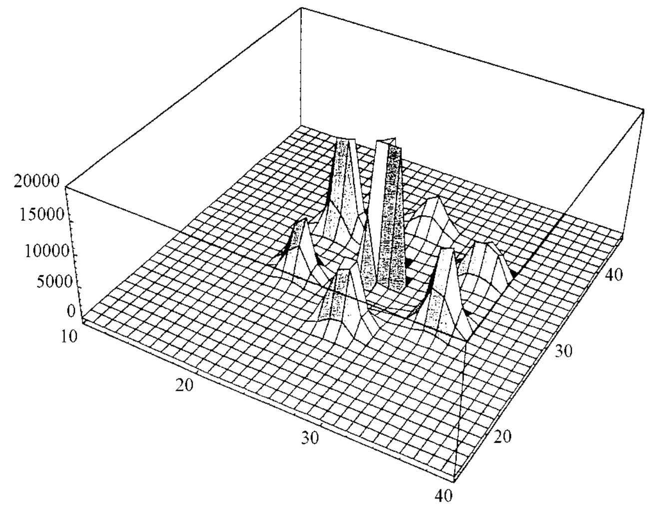

Fig. 1. Spin flip scattering profile for niobium in a field of 0.22 T and at $T = 4.5$ K. The background has been subtracted. A set of six (10)-Bragg FLL reflections is seen centred around the remainder of the direct beam which is situated at the centre of the plot.

2 Experimental

Niobium is a type-II superconductor with a transition temperature of $T_c = 9.2$ K. A disk with a diameter of 12 mm and a thickness of 1 mm was cut from a single crystal with the $\langle 111 \rangle$ axis normal to the disk. For the neutron scattering experiment the disk was oriented inside a horizontal superconducting magnet such that the magnetic field direction, the $\langle 111 \rangle$ axis of the crystal and the incident neutron beam were all parallel to one another. To ensure good resolution a cadmium disk with a hole of diameter 6.3 mm was fixed in front of the sample which enabled the Bragg reflections to be well separated from the direct neutron beam. In this configuration the scattering vector was perpendicular to the magnetic field direction.

The experiment was carried out on the small angle diffractometer D17 located in the guide hall at the ILL. Neutrons with a wavelength of 11 Å were obtained by using a mechanical selector. The spread in wavelength amounted to 10%. A spin polarized neutron beam was obtained by reflection of the incident neutrons off a polarizing neutron mirror. A spin flipper allowed the neutron spin direction to be inverted with respect to the magnetic guide field direction. A neutron spin analyser identical to the polarizer was used to confirm the spin polarization of the neutrons. Using the direct beam flipping ratios in excess of 30 were obtained. For all subsequent measurements the neutron analyser was removed from the scattered beam.

Figure 1 shows the intensity distribution for one spin direction of the neutron and with Bragg reflections present which arise from the FLL within the superconductor. For Bragg reflections with a broad rocking curve, that is for a flux line lattice for which the flux lines are not perfectly straight lines, the Bragg conditions can be fulfilled simultaneously for all Bragg reflections of the first order.

The intensity of the scattering was determined for spin up and spin down neutrons, switching the incident neutron spin direction every 15 minutes. The intensity of the FLL scattering was measured at a temperature of 4.5 K and with an applied field strength of $B = 0.22$ T. The determination of the background was carried out at the same temperature as was used for the magnetic flux line measurements, but with an applied field reduced to $B = 0.03$ T, a value smaller than the lower critical field H_{c_1}. A small field is necessary in order to preserve a guide field for the spin polarized neutrons along the whole length of their flight path through the superconducting magnet. For the background measurement the sample was heated to a temperature above 20 K and cooled to 4.5 K in zero field. After the temperature was stabilized the magnetic field of 0.03 T was switched on.

The experimental setup as described above measures the intensity of the Bragg reflections of the FLL both for the neutron spin up and spin down orientation.

The intensities are determined by the Fourier coefficients of the magnetic and the nuclear interaction potential. For the magnetic contribution the interaction potential is given by the modulation of the magnetic induction due to the flux lines in the material. A nuclear distortion accompanying the FLL gives rise to the nuclear contribution. If F_{N} and F_{M} are the nuclear and the magnetic structure amplitudes, respectively, then the intensity of the scattering is determined by

$$\begin{aligned} I_{\uparrow\uparrow} &= C\,|F_{\mathrm{N}} + F_{\mathrm{M}}|^2 \\ I_{\uparrow\downarrow} &= C\,|F_{\mathrm{N}} - F_{\mathrm{M}}|^2 \,. \end{aligned} \tag{1}$$

Here $I_{\uparrow\uparrow}$ and $I_{\uparrow\downarrow}$ are the measured (integrated) Bragg intensities for the case of the magnetic moment of the neutron being oriented either parallel or antiparallel to the magnetic field direction, respectively. The constant C depends on details of the experiment such as the amount of sample in the beam. It is important here to note that C does not depend on the neutron spin direction. The dependence of the nuclear and magnetic scattering is such that the nuclear scattering is independent of the neutron spin direction, while the magnetic structure amplitude changes sign if the neutron spin direction is inverted with respect to the magnetic field. If only magnetic scattering is present ($F_{\mathrm{N}} = 0$) then this change of sign is irrelevant as it is the square of the structure amplitude which determines the scattering intensity. However, if both magnetic as well as nuclear scattering is present, the scattered intensity will depend on the neutron spin orientation.

Thus it is possible to obtain the ratio of these intensities. This ratio is known as the flipping ratio R [10], and it is defined as

$$R = \frac{I_{\uparrow\uparrow}}{I_{\uparrow\downarrow}} = \left|\frac{F_{\mathrm{N}} + F_{\mathrm{M}}}{F_{\mathrm{N}} - F_{\mathrm{M}}}\right|^2 = \left|\frac{1+\gamma}{1-\gamma}\right|^2 \approx 1 + 4\gamma \tag{2}$$

where $\gamma = F_{\mathrm{N}}/F_{\mathrm{M}}$ with $|\gamma| \ll 1$. For this ratio the scaling constant C drops out and R is only a function of the ratios of the nuclear and magnetic structure amplitudes. For $F_{\mathrm{N}} = 0$ the flipping ratio is 1. A deviation of R from 1 is an indication of the presence of an interference term between magnetic and nuclear scattering. Under the assumption that $|F_{\mathrm{N}}| \ll |F_{\mathrm{M}}|$ (which will be shown to be the case for the situation discussed here) $R \approx 1 + 4\gamma$ up to correction terms of order γ^2. For the interpretation of this result it is important to note that the flipping ratio is obtained from an interference term between magnetic and nuclear scattering. Without the interference, *e.g.* for unpolarized neutrons, the intensity is given by

$$I = C\left(|F_{\mathrm{N}}|^2 + |F_{\mathrm{M}}|^2\right). \tag{3}$$

As pointed out in [11] the interference term yields a significant increase in the sensitivity of the experiment compared to experiments which rely on the determination of integrated intensities. Another important advantage of this technique is the absence of a scaling factor. Given the value of the magnetic structure amplitude F_{M}, this allows the absolute value of the nuclear structure amplitude to be determined without further assumptions.

3 Analysis

The experimentally observed scattering intensity for one neutron spin direction is shown in Figure 1 having made the background correction. Summing up only those data points for which the measured intensity exceeds a value of $\sim 1\%$ of the maximum count of the Bragg reflection and excluding the region of the direct beam, the total summed intensity is determined for all Bragg reflections equivalent to the first order (10)-Bragg reflection. For the measurement at $T = 4.5$ K the intensities are evaluated as $I_{\uparrow\uparrow}$ and $I_{\uparrow\downarrow}$. Their ratio is obtained as $R-1 \cong 4\gamma$ resulting in an experimental gamma value of

$$\gamma \cong \frac{R-1}{4} = -0.003740 \pm 0.000893. \tag{4}$$

The determination of the flipping ratio has, in principle, to be corrected for the finite polarization of the neutron beam and the flipping efficiency of the spin flipper. However, in view of the high flipping ratio obtained for the direct beam and the closeness of the experimental FLL flipping ratios to the value of 1 such corrections do not lead to any significant change of the experimental value. Therefore a flipping ratio and flipping efficiency correction have not been applied to the experimental values.

The flipping ratio is clearly seen to deviate from a value of 1 expected for the situation for which no nuclear scattering contribution is present. The error in the experimental value is large, amounting to 24%. This is due to the fact that it is the ratio of two large numbers which determines the value of interest here.

4 Model

In order to estimate the size expected for the nuclear contribution a simple model will be developed. Here it suffices to obtain an order of magnitude estimate of the nuclear modulation contribution to the scattering.

Consider a homogeneous superconductor in which a cylinder of radius ξ is cut out and removed. If the cylinder is driven non-superconducting by a magnetic field its volume will change. Following the analysis as given in [12] the volume change is given by

$$\zeta = \frac{V_{\mathrm{n}} - V_{\mathrm{s}}}{V_{\mathrm{s}}} \tag{5}$$

which is related to the pressure derivative of the critical field. The experimental observation of the length change in niobium [13] indicates that the volume of the normal material is smaller than that of the superconducting phase. If the normal material is re-inserted into the superconductor the matching of the surface of the superconductor and the core necessitates the distortions of both superconducting and normal materials. However, for an order of magnitude estimate of interest here these details will be neglected. Thus one arrives at the situation that the normal core is more dense than the surrounding superconductor. In this approximation a gap ΔR (see Fig. 2)

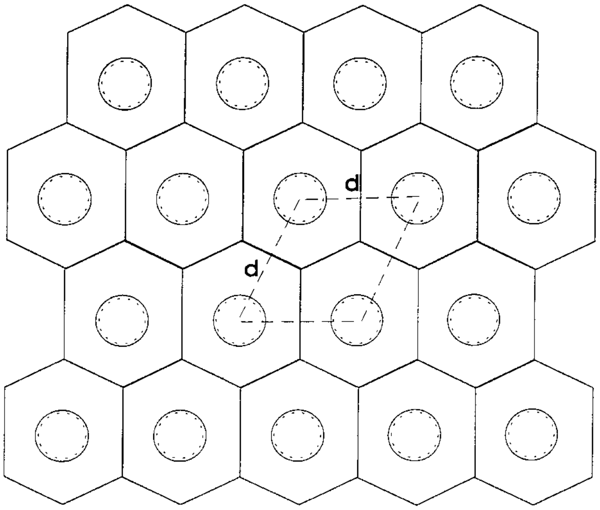

Fig. 2. Two-dimensional flux line lattice. The circle indicate the boundary of the superconducting material, while the dashed circles give the radius (as determined by ξ) of the normal core. The radii are not drawn to scale. The hexagonal FLL unit cell with lattice parameter d is indicated.

develops between the normal and the superconducting material. For niobium the volume change [14] is of the order of $\Delta V/V = -3\times10^{-7}$. For a coherence length of $\xi \approx 350$ Å the gap ΔR is of order 10^{-4} Å. The nuclear scattering density of the normal material $\rho_{\rm n} = \frac{2\pi\hbar^2}{m_{\rm n}}\frac{nb_{\rm Nb}}{V_{\rm n}} = \frac{2\pi\hbar^2}{m_{\rm n}}\frac{nb_{\rm Nb}}{V_{\rm s}+\Delta V}$ changes with respect to the superconducting scattering density $\rho_{\rm s}$ with $\Delta\rho = \rho_{\rm n} - \rho_{\rm s} = -\zeta\rho_{\rm s}$. Here $m_{\rm n}$ is the neutron mass, n is the number of niobium atoms inside the volume V, and $b_{\rm Nb}$ is the nuclear coherent scattering length of niobium, namely 0.7054×10^{-12} [cm] [15]. Thus due to this nuclear distortion the nuclear structure amplitude of a single straight flux line, and using cylindrical coordinates, is given by

$$F_{\rm N}^{(\rm core)}(\mathbf{k}) = \frac{m_{\rm n}}{2\pi\hbar^2}\int_0^{2\pi}{\rm d}\theta\int_0^{\xi} r\,{\rm d}r\,\frac{2}{3}\Delta\rho\,{\rm e}^{-i\mathbf{k}\cdot\mathbf{r}}. \quad (6)$$

The factor of 2/3 arises due to the fact that the volume change only occurs in directions perpendicular to the axis of the flux line, the radius of which is determined by the superconducting coherence length ξ. It is this modulation of the nuclear scattering density $\Delta\rho$ with respect to the average density $\rho_{\rm s}$ of the superconductor which gives rise to the nuclear contribution to the scattering. For niobium a value of $\Delta\rho = +3\times10^{-7}\ \rho_{\rm s}$ is used. Evaluating (6) one obtains

$$F_{\rm N}^{(\rm core)} = -2\pi\frac{b_{\rm Nb}}{V_{\rm Nb}}\,\zeta\,\frac{\xi}{k}J_1(k\xi). \quad (7)$$

For the experimental conditions used ($k \approx 6\times10^{-3}$ [Å^{-1}], $\xi = 350$ Å) the argument x of the Bessel function $J_1(x)$ takes a value of $x = k\xi \approx 2.1$, yielding $J_1(k\xi) \approx 0.5$. For these parameters the value of the Bessel function is close to a local maximum, and therefore the above value can be considered an upper limit.

A second contribution arises due to the gap between superconductor and normal material. This contribution is negative compared to the core amplitude, thus reducing the size of the nuclear contribution. However, for an order of magnitude estimate it suffices to only consider the core contribution $F_{\rm N}^{(\rm core)}$.

The result for a single flux line may be generalized to the case of a flux line lattice. Consider the configuration as shown in Figure 2 for which the single flux lines are arranged periodically. Using a simple superposition the average nuclear structure amplitude per niobium atom $\bar{F}_{\rm N}$ can be calculated. The nuclear scattering is normalized to one Nb-atom by multiplying the nuclear structure amplitude by $V_{\rm Nb}/A_{\rm cell}$ where $A_{\rm cell}$ is the area of the unit cell of the FLL. For a triangular lattice have $A_{\rm cell} = \frac{\sqrt{3}}{2}d^2$ where d is the lattice constant of the FLL. This results in an average nuclear structure amplitude of

$$\bar{F}_{\rm N} = -\frac{4\pi}{\sqrt{3}}b_{\rm Nb}\,\zeta\,\left(\frac{\xi}{d}\right)^2\left(\frac{J_1(\xi k)}{\xi k}\right). \quad (8)$$

Thus an upper estimate of the average nuclear structure amplitude per niobium atom is obtained as

$$\bar{F}_{\rm N} \approx 6\times10^{-8}b_{\rm Nb}. \quad (9)$$

For the determination of the flipping ratio, or γ, the nuclear scattering amplitude has to be compared to the scattering amplitude of the magnetic scattering. For the case of the superposition of isolated flux lines in a regular lattice the magnetic structure amplitude has been evaluated by de Gennes [16] in some detail, and the derivation will not be repeated here. For the Bragg reflection defined by the wavevector $\mathbf{k}$ the magnetic structure amplitude is given by

$$F_{\rm M}(\mathbf{k}) = +\frac{m_{\rm n}}{2\pi\hbar^2}\int_{\rm cell}{\rm d}\mathbf{r}\,\mu_{\rm n}\mathbf{B}(\mathbf{r})\,{\rm e}^{-i\mathbf{k}\cdot\mathbf{r}}. \quad (10)$$

$\mu_{\rm n} = -1.91\mu_{\rm N}\sigma_{\rm n}$ is the magnetic moment of the neutron, $\mu_{\rm N}$ the nuclear magneton, $\sigma_{\rm n}$ the neutron Pauli-spin operator with eigenvalues ±1. For the scattering geometry used in the present experiment, the scattering vector and the direction of the magnetic field are at right angles. Therefore the direction dependence simplifies, and the magnetic structure amplitude can be considered as a scalar function rather than a vector. Evaluation of the integral (see [16]) results in a magnetic structure amplitude proportional to $F_{\rm M} \sim -\frac{\gamma}{1+k^2\lambda^2}\cdot\frac{\boldsymbol{\sigma}\cdot\mathbf{B}}{|\boldsymbol{\sigma}\cdot\mathbf{B}|}$. Here λ is the penetration depth of the superconductor (according to [17] $\lambda \approx 450$ Å). The resulting scattering length per Nb-atom, in units of $b_{\rm Nb}$, is $\bar{F}_{\rm M} = 1.57\times10^{-2}\,b_{\rm Nb}$.

Thus on the basis of this simple model a γ value is expected which is of the order of $\gamma \cong 10^{-6}$. A comparison with the experimentally observed value of the flipping ratio, which is of the order of 10^{-3} shows that the disagreement is substantial, amounting to approximately three orders of magnitude. Certainly such a discrepancy can not be remedied by small alterations of parameters used in the calculation. It is pointed out that the volume effect is commonly used in the literature to model the coupling between the FLL and the nuclear lattice. This process, on its own, is clearly not consistent with the present spin polarized neutron scattering investigation, and a different mechanism has to be at work here.

In order to identify the physics which could give rise to the relatively large value of the flipping ratio, it has to be recognized that the present experimental situation investigates an inhomogeneous system for which, within the simple model discussed above, superconducting and non-superconducting materials coexist next to one another. This coexistence necessitates a shift of chemical potential μ of the material within the non-superconducting core of the flux line. Such a shift can be achieved by a lattice distortion. According to band structure estimates the value of the derivative of the chemical potential with respect to the lattice parameter a takes a value of $\mathrm{d}\mu/\mathrm{d}a = -89[\mathrm{eV}/\mathrm{\AA}]$. An upper limit for the shift of μ is given by the size of the gap of the superconductor. With a value of $\Delta\mu \approx 10$ K this process yields an order of magnitude value for the change in lattice parameter of $\Delta a/a \approx 10^{-5}$. This lattice distortion is two orders of magnitude larger than the value due to the volume effect. It is therefore argued here that it is more likely an electronic process caused by a shift of the chemical potential which is the dominant process in determining the size of the nuclear lattice distortion due to the presence of the FLL within the Shubnikov phase of type-II-superconductors. This process gives rise to a self trapping of electrons inside the normal core of the flux line and to the observed lattice distortion.

5 Discussion

The investigation reported here provides clear experimental evidence demonstrating that firstly, a nuclear distortion exists around flux lines in a type-II superconductor, and secondly that this effect is experimentally measurable. This is the first unambiguous experimental observation of this kind within the bulk of the material. Despite the smallness of the effect the experimental sensitivity of a spin polarized neutron scattering experiment is sufficient for the experimental determination of distortions of magnitude 10^{-5}.

The experimental technique as presented above is a novel tool in the investigation of superconductors. It allows a more detailed study of the flux lines and their interaction with the nuclear lattice. In this respect the experimental technique has relevance for the investigation of pinning of flux lines, a point of substantial practical importance.

The present investigation has demonstrated the feasability of the investigation of FLL with spin polarized neutrons. In particular the observation of the effect in Nb illustrates that the sensitivity is sufficient for such experiments. Due to its mechanical hardness niobium is not the most favourable material for carrying out such an investigation. The observation however of a flipping ratio, even for this material, is a clear demonstration of the capabilities of such investigations.

The magnitude of the observed flipping ratio clearly indicates that the size difference between superconducting and non-superconducting material, commonly used for the description of FLL-nuclear lattice coupling, is insufficient for explaining the present observation. A new mechanism, based on the self trapping of electrons within the core of the flux line, is put forward as a possible mechanism, by which the nuclear lattice responds to the presence of a flux line.

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