ESRF

Experiment title:
X-ray detection of vortices in type-II superconductors

Experiment number:
HE 100

Beamline:
ID20

Date of Experiment:
from: June 16, 97 to: June 22, 97

Date of Report:
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Shifts:
17

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Report:

A type-II superconductor in an applied magnetic field will trap magnetic flux into a (more or less) regular array of vortex lines. This is well known, has been extensively studied in the past, and is still today an active field, particularly regarding high temperature superconductors. Due to its inherent magnetic character, it is not surprising that x-ray scattering has not yet played a role in this field: the diffuse and extended character of the magnetic fields together with the extremely small magnetic cross-section for photons would seem to make the use of x-rays futile. However, a recent series of papers [1-3] have shown that the vortex state of a type-II superconductor induces a local charge modulation as well: basically, the vortex core traps some electric charge due to different electronic states of the superconducting and normal (core) regions. This experiment attempted to detect such a charge modulation in a detwinned 2×2×0.025 mm^3 $YBa_2Cu_3O_{6.77}$ high purity single crystal.

The experiment was conducted on ID20 using an incident energy of 8 keV. A horizontal cryomagnet was used with a field of 3.4 Tesla. At this field and x-ray wavelength, the first order vortex reflections are at a scattering angle of about 0.5 degrees. Therefore, we installed a 4 m long flight tube and a 2D gas detector centered at q = 0. This was the first time a SAXS experiment was attempted on ID20. Therefore the SAXS quality of the beamline components were unknown. The sample transmission was 24.22 %, and the cryostat transmission was 39.15 %. The insertion of the cryomagnet in the beam increased the background by a factor of 12.11, and was therefore the major limiting factor in this experiment. The present theories [1-3] essentially predicts that a line of charge be trapped within the vortex core on the scale of the coherence length ξ. Conservative estimates of the amount of trapped charge for YBCO ranges between $1\text{-}6\times10^{-3}$e/Å. Assuming a perfect vortex lattice in an applied field of 3.4 Tesla, the first order reflection was estimated to have a reflectivity of the order of 10^{-15}. With the operational mode of the ring during the experiment (16 bunch), and the beam spot size of 0.1× 0.1 mm, the incident flux was ≈ 10^{12} ph/sec, implying one scattered photon from the vortex lattice every 16 minutes! The only hope for success was therefore that the charge modulation is significantly higher than predicted. More flux could have been obtained with a larger beam size, but the huge background from the cryomagnet caused the 2D PSD gas detector to saturate for larger incident fluxes.

In order to compensate the high background and very low reflectivity of the vortex lattice, a "fast" signal averaging procedure was used to acquire the difference in intensity between high temperature (T = 60 K) and low temperature (T = 20 K) states of the sample. This was achieved by mounting the crystal in a specially designed aluminum sample holder whose temperature was controlled separately from that of the sample stick itself. The crystal was held in a single crystal Si frame which had only a weak thermal connection to the main Al cylinder of the sample holder. Two heaters were installed: one on the Si frame, the other on the Al cylinder. By switching the heater power from the Si frame to the Al cylinder on could rapidly cool the sample from 63 to 13 K in 1 minute, whilst maintaining a constant heat load on the sample stick (which was held at roughly constant temperature of 10-16 K). Thus a difference pattern could be measured within several minutes without modifying the overall thermal profile within the cryostat, minimizing any movement of the sample due to thermal expansion.

Since the vortex lattice orientation is determined by the magnetic field and not the crystal, it was necessary to calibrate the magentic field orientation to a very high degree of accuracy (ca. 40 mdeg both horizontally and vertically) in order to satisfy the Bragg condition. This was acheived by suspending a tiny collimator at the sample position within the cryomagnet. This collimator consisted of to single crystal Si plates held separate by 20 micron thick kapton foils. In a magnetic field, the collimator rotates to align itself parallel to the field and by scanning the magnet orientation and measuring the straight through beam intensity, one was able to determine when the field was parallel to the incoming beam.

As it turned out, the SAXS from the Kapton windows of the cryomagnet was sufficiently high to saturate the 2D gas detector, so that it became necessary to use a large beam-stop and look for second order reflections only. This was done by signal averaging 5 minute runs during 3 days (9 shifts). No hint of any signal arising from the vortex lattice was detectable.

The final 3 shifts were used to characterize the sample at room temperature. Without the cryomagnet, the background was significantly smaller, revealing high intensity, sample-position sensitive streaks which are yet to be understood. Images of these streaks can be found on the WEB at http://www.ill.fr/YellowBook/IN15/vortex/Vortex97.html

This experiment has shown that the signal averaging technique works quite well, and that the field can be properly aligned using a very fine magnetic collimater, but that one needs to use a cryomagnet which does not introduce anything in the beam; in other words one which can be directly connected to the front-end vacuum. This would lower the background by a factor of 12 and allow one to use a higher incident flux. It would also be worthwhile to try other superconducting systems whose charge modulation may be less, but whose SAXS background is significantly smaller.

[1] D. Khomskii, Phys. Rev. Lett. 75, 1384 (1995); D. Khomskii, J. of Superconductivity 9, 7 (1996).
[2] G. Blatter et al., Phys. Rev. Lett. 77, 566 (1996).
[3] Nobuhiko Hayashi, Masanori Ichioka, and Kazushige Machida, (to be published).