Observation of a Single Crystal of Vortex Lines in a Type-II Superconductor

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A quasiperfect crystal of vortex lines was observed by neutron diffraction. The quality of the crystal allows the direct detection and measurement of the relative intensities at the Bragg peaks [10], [20], and [11] (in the reciprocal space of the two-dimensional lattice) and gives, for the first time, the possibility of mapping the magnetic field of the mixed state.

The samples used are single-crystal slabs of Nb degassed in a high vacuum at 2000°C for 1 week, then chemically electropolished. The [111] axis of the Nb crystal is parallel to $0y$ (Fig. 1); the $0z$ axis is at $9°\pm 1°$ to a $[1\bar{1}0]$ axis. The ratio of the resistivities at 300 and 4.2°K (at 8000 Oe) is 80. The critical current densities are low, 30 to 50 A/cm^2 at 1500 Oe. The dimensions of the slabs are $\Delta x = 10$ mm, $\Delta y = 25$ mm, $\Delta z = 1$ mm. The incident neutron beam, the vertical direction, and the applied magnetic field define the three orthogonal axes $0x'$, $0y'$, and $0z'$ of the lab frame. The magnetic field is always perpendicular to the slab ($0z' = 0z$). During the rotation of the slab around $0z'$, the neutron-illuminated volume is kept almost constant.

The main features of the neutron beam are the same as in that used by Cribier *et al.*[1] The wavelength λ_n is 4.3 Å. The resolution $\Delta\lambda_n/\lambda_n$ has been improved and is about 8%; the angular divergence of both the incident and diffracted beams is $\Delta\theta = \pm 3.5'$.

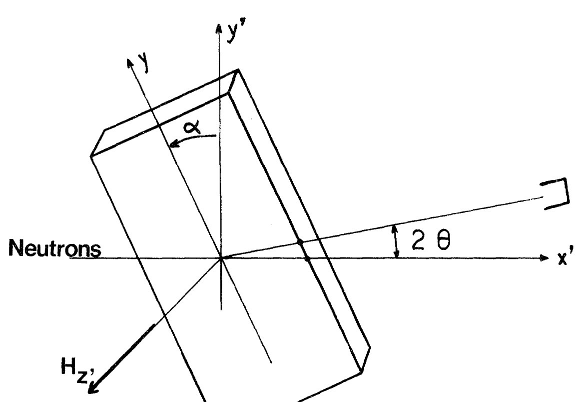

FIG. 1. Geometrical disposition.

We measure the scattered intensity $I(\alpha, 2\theta)$ in a direction making an angle 2θ with $0x'$ (Fig. 1) for a given orientation $\alpha = (0y, 0y')$ of the sample. A Bragg peak at $2\theta = 2\theta_B$ (typically $2\theta_B \sim 10'$ to 20′) on the curve $I(\alpha = \alpha_{ij}, 2\theta)$ indicates an ordering of the crystalline vortex with an interreticular distance $d = \lambda_n/2\theta_B$. A peak on the curve $I(\alpha, 2\theta = 2\theta_B)$, the so-called "rocking curve," indicates a preferential orientation of the vortex lattice. The width of the rocking curve is a classic measure of the quality of a monocrystal.

The most surprising but clearly established result is illustrated in Fig. 2. The full width at half-maximum (FWHM) of the rocking curve of the vortex crystal (V crystal) is $\Delta\alpha = 28'$ (the instrumental width is about 4′). This quasiperfect V crystal was obtained by decreasing the

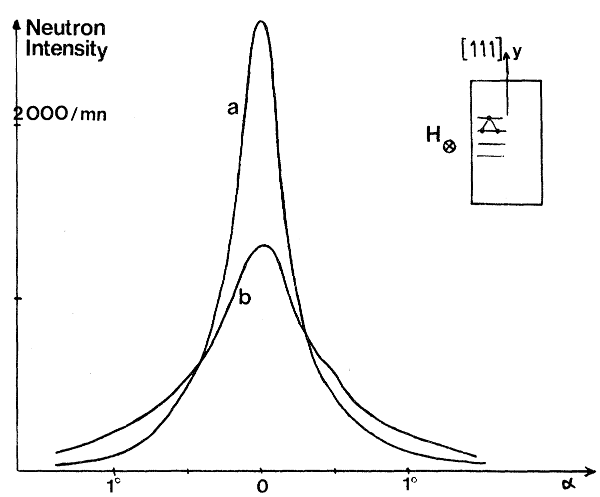

FIG. 2. Rocking curves $I(\alpha, 2\theta = 2\theta_B)$. (a) V crystal created by decreasing field from H_{c2} to 1500 Oe; FWHM $\Delta\alpha = 24'$. (b) V crystal obtained from the first magnetization from 0 to 1500 Oe; FWHM $\Delta\alpha = 48'$.

TABLE I. $2\theta_B$ and α_B for the two-dimensional lattice with $B = 1500$ Oe. The uncertainty in the angles comes only from the experimental setup which is not well adapted to perform precise large angular variations in $\Delta\alpha$.

Reflection	$2\theta_B$ Theoretical values, triangular lattice	$2\theta_B$ Measured	$\Delta\alpha_{ij} = \alpha_{ij} - \alpha_{10}$ Theoretical values, triangular lattice	$\Delta\alpha_{ij}$ Measured
[10]	$2\theta_0$	14′ ±2′	α_{10}	Reference
[20]	$4\theta_0 = 28'$	28′ ±2′	$\Delta\alpha_{20} = \theta_0$	10′ ±4′
[11]	$2\sqrt{3}\,\theta_0 = 24'$	23′ ±2′	$\Delta\alpha_{11} = 30°$	31′ ±1°

magnetic field from $H > H_{c2}$ to $H = 1500$ Oe. The rocking curve is always broader when the field is increased from $H = 0$.

A complete investigation of $I(\alpha, 2\theta)$ reveals that the V crystal is a single crystal The observed width of the Bragg peak is equal to the instrumental width, which proves that the lattice spacing is well defined throughout the sample. This type of single V crystal was observed in five samples with only small differences in quality.

The diffracted neutron intensities by such a V crystal are at least 4 times larger than in the experiments at Cribier *et al.*[1] in spite of the fact that the volume of the sample here is 10 times smaller (0.15 cm^3). Even in this small a V crystal it was possible to observe the Bragg reflections, denoted [10], [20], and [11] in the reciprocal space of the two-dimensional triangular lattice. Table I gives the observed values of α_B and $2\theta_B$. The existence of the triangular lattice is established by these measurements with the same certainty as the direct observation of Trauble and Essmann.[2] Moreover, the lattice here covers a larger area (> 1 cm^2). No diffuse scattering is observed, which proves that the V crystal extends over essentially the whole volume of the Nb crystal.

Table II gives the observed intensities at the maxima of the Bragg peaks, on a relative scale. To a first approximation these numbers can be used for the integrated intensities which are normally taken into account by crystallographers. Initially, the slabs were prepared to study the perfection of the V crystal when a dc current is applied.[3] It is possible to improve the neutron-scattering results by increasing the volume. We are preparing such samples. Nevertheless these first results allow us to test various theoretical models of the field map in pure superconductors (cf. Delrieu[4]). The prediction of Abrikosov with the Ginsburg-Landau equations for the field map are, for example, too small by a factor of 400 to give the peak [11]. The microscopic theory of the nonlocal relation between the magnetic field and the order parameter gives a better agreement with experiment when the magnetization is small compared to the induction (i.e., at $B = 1750$ G).

Improved measurements may give a larger number of coefficients H_{lm} in the two-dimensional Fourier development of the field. These data make it possible to map the local magnetic field at every point of the primitive triangular cell. Previously,[5-7] the field was measured at selected points in the primitive cell using NMR techniques, giving two relations between the Fourier coefficients H_{lm}. Neutron experiments give all the relative values of $|H_{lm}|$. Only the study of these two types of results on the same sample could give the exact magnetic field map.

The orientation of the V crystal in the Nb crystal is shown in the inset of Fig. 2. This orientation is the same in the five slabs. The height of the basic triangle is parallel (to within experimental error ± 2°) to the direction $0y$ which is the [111] axis of the Nb crystal. Cutting some of these slabs, we have carried out many experiments with various shapes of the Nb sample

TABLE II. Relative intensities of Bragg peaks compared with 1.0. These values were obtained for a V crystal created by decreasing the field H, from H higher than H_{c2}. We found that this is the experimental condition to obtain reproducible results.

Reflection	Relative intensity 1250 G	1500 G	1750 G
[10]	1	1	1
[20]	(2.5 ± 0.5)%	1.3%	(0.6 ± 0.1)%
[11]	(2.9 ± 0.2)%	(3.2 ± 0.5)%	3%

(triangular, rectangular, square, trapezoidal) and with various orientations of the [111] axis relative to the small faces of the slabs $\Delta z\,\Delta y$ which are parallel to the applied field. In all of these experiments the [111] axis determines the preferential orientation of the V lattice. However, the quality of the V crystal is best when the sample edges are aligned with the crystallographic lines of high density of the V lattice (as shown in the inset of Fig. 2). This indicates the effect of the faces parallel to the field on the preferential orientation of the V crystal along the anisotropy axis of the Nb crystal. This long-range influence arises as a consequence of the rigidity of the vortex lattice. The details of the experiments will be described later.[8]

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[1]D. Cribier, B. Jacrot, L. Madhav Rao, and R. Farnoux, in *Progress in Low Temperature Physics*, edited by C. J. Gorter (North-Holland, Amsterdam, 1967), Vol. 5, p. 161.

[2]H. Trauble and U. Essmann, Phys. Status Solidi 18, 813 (1966).

[3]Y. Simon and P. Thorel, Phys. Lett. 35A, 450 (1971).

[4]J. M. Delrieu, J. Low Temp. Phys. 6, 197 (1971).

[5]W. Fite and A. G. Redfield, Phys. Rev. Lett. 17, 381 (1966).

[6]J. M. Delrieu and J. M. Winter, Solid State Commun. 4, 545 (1966).

[7]A. Kung, Phys. Rev. Lett. 15, 1006 (1970).

[8]P. Thorel, R. Kahn, Y. Simon, and D. Cribier, to be published.

Anisotropic Microstructure in Evaporated Amorphous Germanium Films*

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Direct evidence for anisotropic microstructure in evaporated amorphous germanium films has been obtained from small-angle x-ray scattering. Low-density regions have approximate linear dimensions of 22 and 46 Å in the film plane and 2200 Å normal to the film plane for the 7-μm-thick films studied. Their volume fraction is only 1 to 2%, if they are assumed to be voids.

Evaporated amorphous Ge and Si films are commonly less dense than crystalline films; the reported density deficits range from 0 to 30% and apparently depend on deposition conditions, as do many other properties of amorphous films.[1] This Letter presents results of small-angle x-ray scattering measurements which clarify the origin of such density deficits. Earlier electron[2] and x-ray[3] scattering measurements indicated that most of the density deficit and its variation were associated with submicroscopic voids, rather than with the intrinsic amorphous structure. However, spherical or randomly oriented voids were assumed in interpreting the measurements.[2,3] The present study indicates that evaporated amorphous germanium films (of ~7 μm thickness) have anisotropic microstructures consisting of rodlike low-density regions oriented perpendicular to the film plane and that submicroscopic voids account for a much smaller part of the density deficit than previously thought. Galeener[4,5] recently interpreted anomalous structure in uv dielectric constants of 1000-Å evaporated amorphous Ge films by postulating cracklike oriented voids. Donovan and Heinemann[6] interpreted features in high-resolution electron micrographs of 100-Å films as voids of the type proposed by Galeener. The present results, on 7-μm-thick films, indicate rodlike rather than cracklike voids.

The geometry used in the scattering measurements[7] and the dependence of observed scattered intensity on both φ and K are shown in Fig. 1. Scattering is anisotropic for $K<0.5$ Å^{-1}. Anisotropy is sharpest in the high-angle tail of the small-angle scattering, i.e., half-widths $\varphi_{1/2}$ are smallest here. The half-widths increase as the scattering angle is reduced. Absolute, slit-length–corrected intensities[8] for $\varphi=0$ are shown in Fig. 2 (squares) as $\log I$ versus K^2.

These data were obtained for films which had been floated off their substrates in warm water or hydrofluoric acid. The large-angle x-ray scattering and radial distribution functions for these films are similar to those published elsewhere.[2,3,9] Anisotropic small-angle scattering similar to