Brief Reports

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Rotational magnetic processes in a dirty superconductor

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(Received 6 July 1988)

Magnetization-vector (**M**) measurements were made on a thin disk of impure Nb rotated relative to a fixed field **H** at 4.2 K. For all H, **M** is found to be separable into a nonrotating diamagnetic component $\mathbf{M}_d$ plus a penetrating-flux component $\mathbf{M}_p$ that turns rigidly with the sample for small rotation angles. For H below H_{c1} on a hysteresis loop, $\mathbf{M}_p$ diminishes in size but continues to rotate rigidly up to 360°. For H between H_{c1} and H_{c2}, $\mathbf{M}_p$ rotates up to and then remains at some critical angle, indicating a constant frictional torque between $\mathbf{M}_p$ and the sample, which presumably arises from vortex-flux jumps between pinning centers. Moreover, $\mathbf{M}_d$ closely equals $-\mathbf{H}/4\pi$ for H above as well as below H_{c1}.

A recent modification of a vibrating-sample magnetometer has enabled us to measure simultaneously the magnetization components parallel and perpendicular to a fixed magnetic field **H** in the plane of a sample disk rotated about the axis of vibration.[1] Initially, we have used such magnetization-vector (**M**) measurements to study rotational magnetic processes in various spin glasses.[1,2] In the case of Au-Fe, it was learned that the anisotropy field $\mathbf{H}_K$ produced by field-cooling turns rigidly with the sample but only up to some critical angle, thus revealing a constant frictional torque between $\mathbf{H}_K$ and the sample.[2] These results alerted us to the exciting possibility of applying the same method to study analogous rotational processes in superconductors, especially those in which the magnetic flux pinning by impurities and other imperfections is strong enough to produce observable effects. An accurate knowledge of such processes is strategically important for critical-current improvements in the various new classes of high-T_c superconducting oxides. However, for our initial magnetization-vector measurements on a superconductor, we chose to investigate a much simpler material, namely elemental niobium—albeit with a high chemical impurity concentration — and the results of this investigation are reported in this paper.

In a previous rotational magnetic study of type-II superconductors (V and a V-Ti alloy) by Boyer and coworkers,[3] a sample disk was rotated inside two coils mounted orthogonally and the changes of magnetic flux parallel and perpendicular to a fixed field in the disk plane were integrated with respect to some reference state. Though operationally less direct, these experiments are quite analogous to our magnetization-vector measurements performed with a vibrating-sample magnetometer. As we will discuss, many of the results of these earlier experiments are basically similar to ours. However, unlike the previous work, our data analysis contains an essential revealing feature, a decomposition of the measured **M** into two physically different components, which should greatly facilitate theoretical interpretations of the magnetic properties of type-II superconductors.

The Nb sample of our study was a thin disk (5-mm diam, 0.25-mm thick) spark-cut from an ingot produced by arc-melting 60-mesh powder of 99.8% nominal purity. Its magnetization measured after zero-field cooling to 4.2 K is shown in Fig. 1 as a function of increasing and then decreasing field. The two M-vs-H curves, representing respectively the initial magnetization curve (MC) and the upper branch of a hysteresis loop (HL), coincide at nearly zero M in the normal state above $H_{c2}\approx 6.6$ kOe. The initial linear part of MC has a slope of essentially $-1/4\pi$, corresponding to perfect Meissner-effect shielding; the departure from linearity starts at $H_{c1}\approx 0.8$ kOe. The high H_{c2}/H_{c1} ratio (~ 8.2), the low zero-H critical temperature ($T_c\approx 7.4$ K), and the very low resistivity ratio [$\rho(300\text{ K})/\rho(4.2\text{ K})\approx 4.5$] all testify to the high impurity content of our Nb sample material.[4]

The closed-circle points in Fig. 1 represent the starting conditions of the rotational experiments to be discussed here in detail—namely, for $H=0.7$ and 1.0 kOe on the HL and for $H=1.3$ kOe on both the HL and MC, all at 4.2 K. In each experiment, the sample was rotated quasistatically about its disk axis from $\theta=0°$ to 360° and back

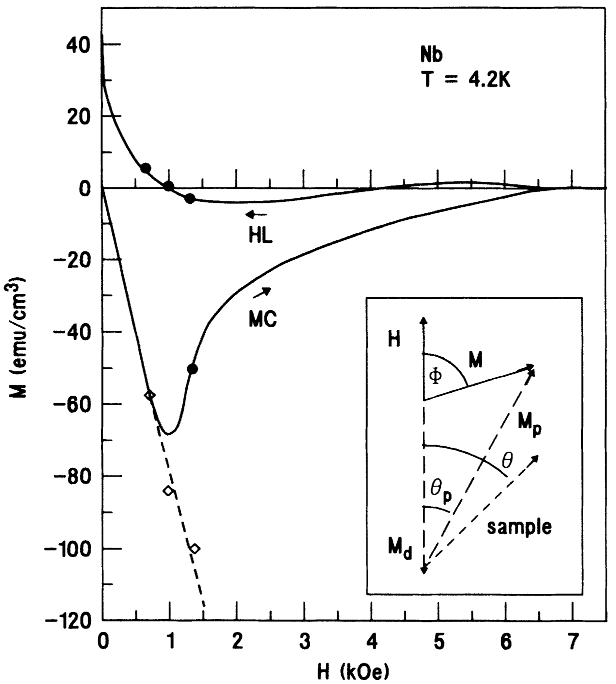

FIG. 1. Magnetization of Nb sample zero-field cooled to 4.2 K as function of increasing field (along magnetization curve MC) and then of decreasing field (along hysteresis loop HL). Closed circles represent starting conditions for rotational experiments described in text and later figures. Open diamonds show deduced values of M_d compared to dashed line of slope $-\frac{1}{4}\pi$. (Inset: vector diagram showing separation of measured **M** into $\mathbf{M}_d$ and $\mathbf{M}_p$ for sample-rotation angle θ, as described in text.)

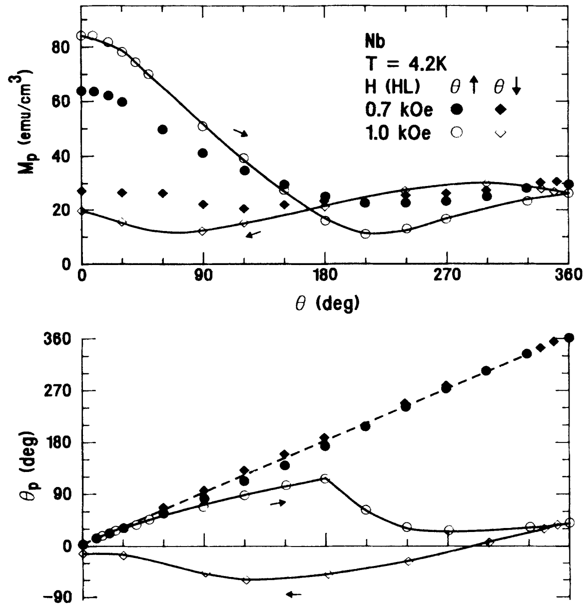

FIG. 2. M_p and θ_p vs increasing and then decreasing θ for a Nb sample at 4.2 K and $H=0.7$ and 1.0 kOe starting on hysteresis loop HL. Dashed line for $\theta_p=\theta$ represents rigid rotation of $\mathbf{M}_p$.

to $\theta=0°$ relative to the fixed **H**, and the components of **M** parallel and perpendicular to **H** in the disk plane were measured at each rotational step. For small θ (typically below 10°), the measured **M** at angle ϕ relative to **H** was assumed to consist of a nonrotating diamagnetic component $\mathbf{M}_d$ and a penetrating-flux component $\mathbf{M}_p$ that turns rigidly with the sample. Thus, with reference to Fig. 1 (inset), the angle θ_p between $\mathbf{M}_p$ and **H** was taken to equal initially the sample rotation angle θ. Our second and last assumption was that the $\mathbf{M}_d$ determined at small θ stays constant in magnitude during all subsequent changes of θ, thus allowing us to determine the evolution in the magnitude and direction of $\mathbf{M}_p$.

Our results for M_p and θ_p at $H=0.7$ kOe (HL), as deduced under the foregoing assumptions (with $M_d=-57.7$ emu/cm^3), are shown plotted versus θ in Fig. 2 (closed symbols). We see that M_p decreases steadily as θ is raised to about 180° and then remains small (with minor undulations) for the rest of the rotational cycle, while θ_p follows the variation of θ fairly closely over the entire cycle. Thus, despite its reduced size, $\mathbf{M}_p$ continues to rotate rigidly with the sample, which is consistent with the rigid rotation of a constant magnetization component observed previously[3] for $H<H_{c1}$ on a hysteresis loop. (For $H<H_{c1}$ on the initial magnetization curve, we find that M_p is zero, as expected, with M_d the same as above.)

When H exceeds H_{c1}, it is found that $\mathbf{M}_p$ no longer rotates rigidly with the sample up to 360°. Instead, at a lower θ (which decreases rapidly from 360° with increasing H), θ_p suddenly begins to decrease, reaching small values as θ approaches 360°, and then becomes negative as θ is lowered to 0°. This peculiar behavior is exemplified by our results for $H=1.0$ kOe (HL), where $M_d + -83.4$ emu/cm^3, and M_p and θ_p are plotted versus θ in Fig. 2 (open symbols). The variations of M_p are similar to those for $H=0.7$ kOe (but with more exaggerated undulations), whereas θ_p reaches only $\sim 110°$ (at $\theta \approx 180°$) before it starts to descend rapidly and eventually become negative.

This peculiar behavior of the HL state proves to be transitional in that at higher H (but below H_{c2}) the variations of M_p and θ_p with θ settle into a relatively simple hysteretic pattern. As typified by our results for $H=1.3$ kOe (HL) displayed in Fig. 3 (closed symbols), M_p descends with increasing θ until it reaches and then stays at a fairly constant value for all subsequent changes of θ. Meanwhile, θ_p rises to a plateau value where it remains up to $\theta=360°$ and then decreases to a negative plateau value of the same magnitude as θ is lowered to 0°. Thus, for both directions of rotation, M_p turns with the sample but only up to some critical angle (θ_{pc}) relative to **H**, demonstrating that the torque exerted on $\mathbf{M}_p$ by **H** is balanced macroscopically by a constant frictional torque between $\mathbf{M}_p$ and the sample. Since $\theta_{pc} \approx 50°$, $H=1.3$ kOe, and the plateau value of M_p is ~ 59 emu/cm^3, the size of this frictional torque, $HM_p \sin\theta_{pc} \approx 5.9\times 10^4$ erg/cm^3 per radian of rotation. Microscopically, this quantity may be regarded as the average energy loss associated with the unpinning and repinning of the penetrating flux (consisting of vortices) as it progresses past the imperfections in

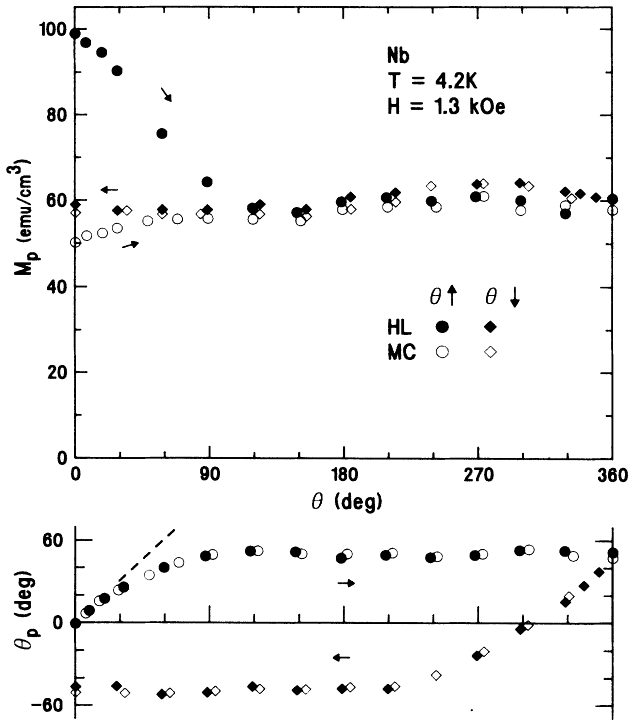

FIG. 3. M_p and θ_p vs increasing and then decreasing θ for Nb sample at 4.2 K and $H = 1.3$ kOe starting on hysteresis loop HL and on magnetization curve MC. Dashed line for $\theta_p = \theta$ represents rigid rotation of $\mathbf{M}_p$.

the rotating sample while maintaining a constant mean orientation relative to **H**. Since this energy loss is a measure of the maximum pinning forces, it determines the upper limit of the electric current that can be carried without dissipation.[5]

For comparison, we performed the rotational experiment for $H = 1.3$ kOe on the MC as well as on the HL, as indicated earlier. In both cases, the value deduced for M_d was -99.9 emu/cm^3. Our results for M_p and θ_p vs θ in the MC case are also displayed in Fig. 3 (open symbols) and, aside from M_p starting from below rather than above its plateau value, the variations of M_p and θ_p are essentially identical to those in the HL case. Thus, as was observed previously,[3] the two cases result in the same steady-state behavior after repeated sample rotation. The close similarity of the two cases is found to continue at higher H, but with a rising plateau value of M_p and a decreasing θ_{pc}, such that the frictional torque grows steadily as H increases towards H_{c2}.

Another striking feature of our results concerns M_d, the diamagnetic component of the magnetization, whose values for the cases discussed are plotted versus H in Fig. 1. In each case, the deduced M_d value lies very close to a line (shown dashed) of slope $-1/4\pi$, corresponding to perfect Meissner-effect shielding. It is particularly noteworthy that this situation obtains even for H much larger than H_{c1}. To our knowledge, this rather remarkable basic property, which is inaccessible by conventional (nonrotational) magnetic measurements, has not been anticipated theoretically.

Except for the frictional torque, which could have been determined alternatively by direct magnetic-torque measurements,[6] all our findings reported here derive uniquely from the capability of magnetization-vector measurements. However, the advantage of this technique that we have exploited (but was previously neglected[3]) depends crucially on the separating out of the initial rigid rotation of $\mathbf{M}_p$, which is experimentally difficult if the rotations with the sample are rigid only for very small angles. Fortunately, in our demonstration study of impure Nb, the flux-pinning forces were strong enough to allow unambiguous separations of $\mathbf{M}_p$ from the nonrotating $\mathbf{M}_d$. In general, for superconducting materials of sufficient imperfection, the magnetization-vector technique holds considerable promise for future magnetic characterizations.

We are grateful to Dr. N. Rivier for stimulating discussions of dirty superconductors and to H. P. Goeckner for his help in the sample preparation. This work was supported at the University of Illinois at Chicago by the National Science Foundation (Grant No. DMR84-06898) and at the Argonne National Laboratory by the U. S. Department of Energy (BES-Materials Science, Contract No. W-31-109-Eng-38).

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[3]R. Boyer and M. A. R. LeBlanc, Solid State Commun. **24**, 261 (1977); R. Boyer, G. Fillion, and M. A. R. LeBlanc, J. Appl. Phys. **51**, 1692 (1980).

[4]Besides some metallic impurities, our Nb sample undoubtedly contains considerable oxygen (originally on the surface of the powder before melting). If oxygen is the major impurity, our measured values of H_{c2}/H_{c1}, etc., suggest that its concentration is between 1 and 2 at.%, with reference to C. C. Koch, J. O. Scarbrough, and D. M. Kroeger, Phys. Rev. B **9**, 888 (1974).

[5]M. Tinkham, *Introduction to Superconductivity* (McGraw-Hill, New York, 1975), Chap. 5; J. R. Clem and A. Perez-Gonzalez, Phys. Rev. B **30**, 5041 (1984), and references therein.

[6]Magnetic torque measurements on the high-T_c superconductor, $La_{1.85}Sr_{0.15}CuO_4$, have been performed by C. Giovannella, G. Collin, and I. A. Campbell [J. Phys. (Paris) **48**, 1835 (1987)], who report "rigid" and "viscous" rotations of the penetrating flux. The "viscous" rotations consist of nearly instantaneous changes (which we have been calling "frictional") followed by slower relaxation effects (which we have observed to be small and have thus ignored in our initial study).