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Authors
Suenaga, M.
Rails, K.M.

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A METHOD OF CRITICAL MAGNETIC FIELD MEASUREMENT FOR HIGH-FIELD SUPERCONDUCTORS IN PULSED MAGNETIC FIELDS

M. Suenaga and K. M. Ralls

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M. Suenaga and K. M. Ralls

Lawrence Radiation Laboratory, Inorganic Materials Research Division
Department of Mineral Technology, College of Engineering
University of California, Berkeley, California

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ABSTRACT

A high magnetic field magnetization technique has been devised which allows rapid measurement of superconducting to normal transition fields ($H_{c2}^*$) for bulk high-field superconductors in pulsed magnetic fields up to 200 kilogauss. Values of $H_{c2}^*$ so obtained are in good agreement with those obtained by resistive techniques. A complete magnetization curve is obtained in the same time that one point could be determined for a resistive critical current density vs field curve. The significance of such a dynamic magnetization curve is discussed.
I. INTRODUCTION

It is desirable in superconductivity research to characterize materials being studied as well as possible. To do so requires the measurement of critical temperature and of critical field (preferably as a function of temperature). The latter poses a special problem when high-field superconductors are being investigated. Conventional solenoids capable of high magnetic field intensities are not commonly available, and those that are available often do not generate the high magnetic fields necessary to study all materials. Superconducting solenoids may provide a partial answer to this problem, but they too have limited magnetic field intensities. A useful apparatus must not destroy itself or the specimen. Pulsed-field techniques have been devised that can be used to measure critical current density ($J_c$) as a function of field and resistive critical field ($H_r$).\(^1\)

If the magnetic field rise time is long enough, the pulsed-field values of $H_r$ agree well with the steady-field values of $H_r$, although electrical "noise" limits the sensitivity to which the onset $H_r$ can be measured for the former. In either steady or pulsed fields a $J_c$ vs $H$ curve must be taken point by point with sufficient time in between points to allow the specimen or the pulsed-field coil to return to thermal equilibrium with its surroundings. Furthermore, resistive techniques require good current contacts and stable voltage contacts. Magnetization measurements eliminate the shortcomings of resistive measurements. Experimental information obtained from magnetization as a function of applied field is essentially equivalent to that obtained from resistive techniques.\(^2\)

For ideal Type II superconductors a completely reversible magnetization curve is expected\(^3\) (Fig. 1). If the demagnetizing coefficient is zero (i.e., the specimen is a long cylinder parallel to the applied field),
the bulk specimen remains completely diamagnetic until the lower critical field, $H_{c1}$, is reached and flux penetration begins. Increasing penetration occurs until the magnetization becomes zero at the upper critical field $H_{c2}$, the point of transition to normal behavior. Actually, for high-field superconductors, the transition occurs at $H_{c2}^*$, the paramagnetically limited upper critical field, where the superconductor magnetization curve has an intercept with the normal state, paramagnetic magnetization curve. Precise magnetization measurements are both laborious and difficult to make, particularly in high magnetic fields. Useful magnetization curves have been obtained in slowly-swept fields or point by point in steady fields. If magnetization vs field could be obtained in pulsed magnetic fields, characterization of superconductors would be greatly simplified. We have devised such a technique for the rapid determination of superconducting transition fields.
II. TECHNIQUE

Pulsed-field magnetization as a function of the applied field for high-field superconductors can be readily displayed as an oscilloscope trace and photographed with high-speed Polaroid film. Two balanced pickup coils and a matched pair of integrators are used to determine the magnetic field intensity and the magnetization. The two coils, which are placed in the most uniform region of pulsed magnetic field, consists of a B coil and a H coil. The B coil fits closely around the superconducting specimen and the H coil surrounds the B coil, with some space separating them. Since magnetization M is proportional to the difference between magnetic induction B and magnetic field intensity H, it is determined by integration of the difference of induced voltages from the B coil and the H coil. In practice, the integrated voltage from (B-H) is connected to the vertical deflection system and the integrated voltage from the H coil is connected to the horizontal deflection system of an oscilloscope. The balance of the pair of coils is maintained with a small carbon rheostat, which is connected to the H coil and adjusted to make the induced voltage difference equal to zero when no specimen is present. The calibration of magnetic fields is made by measuring the maximum voltage on the H scale (horizontal deflection) which corresponds to the known maximum magnetic field for the pulsed-field coil. The magnetization measurement apparatus is shown schematically in Fig. 2. A similar method of measuring magnetization has been applied successfully to ferromagnetic materials.

* Since only the relative variation of magnetization with magnetic field is of interest, rather than an exact value of magnetization, the separation of the coils is not critical as long as it is large enough to give a reasonable signal for the magnetization.
The pulsed-field magnet, situated in a liquid nitrogen dewar, is capable of generating 200 kG peak field with approximately an 8 msec rise time. The pickup coils are contained within a 3/8" I.D. portion of a liquid helium dewar which fits into the pulsed-field coil. Placement of the H coil around the B coil makes it possible to use a small region of good field homogeneity (deviation ±1 - 2% spherical diameter), necessary for accurate high-field work. This does slightly affect the apparent value of H, however (see Discussion). Specimens are affixed to the end of a 2.7 mm diameter stainless steel tube and inserted into a 3.6 mm diameter thin wall stainless steel tube which extends from the top of the helium dewar to the end of the pickup coils. After thermal equilibration, a complete magnetization curve is produced in the same time as would be required for a single point on a Jc-H curve determined by the resistance method. Removal of one specimen and insertion of another can be accomplished rapidly and with little loss of liquid helium because only a small thermal mass must be cooled each time.
III. EXPERIMENTAL RESULTS

The magnetization technique described above has been used to measure the critical fields \( (H_{c2}^*) \) of niobium carbonitrides. A magnetization curve for a cylindrical NbN specimen (0.25 cm diam. x 2.0 cm long) is shown in Fig. 3a. The critical field is approximately 95 kG, as determined from the intersection of the specimen magnetization curve and the dummy run curve. Hysteresis of the dummy run curve results from imperfect balancing of the coils and imperfect matching of the integrators. The low current density \((112 \text{ A/cm}^2)\) resistive transition for the same specimen (in a transverse magnetic field) gives a virtually identical \(H_{c2}^*\) of 94.5 kG, taken as the end of the resistive transition† (Fig. 4). The voltage rise preceding the end of the transition is caused partly by "flux motion" in the mixed state and partly by the development of normal state regions. The values of \(H_{c2}^*\) determined magnetically and resistively agree with each other to an uncertainty smaller than the absolute uncertainty in magnetic field (approximately 2-3%). The field at which the magnetization exhibits the second maximum corresponds to the field at which resistance shows a minimum (the "peak effect" of \(J_c-H\) curves). For all specimens that display a relatively large change in magnetization near \(H_{c2}^*\), and especially those that show the peak effect, accurate values of \(H_{c2}^*\) can easily be determined. With a material such as NbC, which has a low transition field, \(H_{c2}^*\) can also be accurately determined because the change in magnetization with field is rapid (Fig. 3b).

It is quite difficult, however, to determine the exact value of \(H_{c2}^*\) for high transition materials when there is no peak effect and the variation of

† At sufficiently low search current densities the resistive transition sharpens and approaches the end value measured at higher current densities. Noise puts a lower lower limit on the search current density that will give a detectable voltage.
M with $H$ is very low near the transition. Thus, as specimen perfection is increased and critical current density decreased, the magnetization curve approaches the ideal, reversible magnetization curve,\textsuperscript{2} and an accurate $H_{c2}^{*}$ determination is not possible by this method.
IV. DISCUSSION

Two aspects of this technique require consideration: (1) the geometry of the sensing coils and (2) the significance of the experimental results. In part, the two are related. Placement of the \( H \) coil around the \( B \) coil is somewhat unconventional. This geometry allows optimum centering of both sensing coils and the specimen in the region of the highest field homogeneity. Although the coils can be balanced fairly well (with the external carbon rheostat), it is apparent that as soon as a superconducting specimen is inserted into the \( B \) coil the balance is altered. This is so because the specimen, a core for both coils, is almost perfectly diamagnetic at low fields. Thus, a slight nonlinearity in the \( H \) scale results when the specimen excludes any flux. The deviation of the apparent \( H \) from the true \( H \) (a maximum for a completely diamagnetic specimen) depends upon the area occupied by the specimen relative to the area of the \( H \) coil. If the specimen is \( 1/8" \) diam. and the \( H \) coil is \( 3/8" \) diam., then the maximum deviation gives an apparent field \( 11\% \) less than the true field, i.e., an apparent flux penetration field of 2 kG corresponds to an actual flux penetration field of 2.25 kG.

If the specimen were to remain perfectly diamagnetic to high fields, the error introduced in the determination of \( H \) would be comparable to the volume fraction which the specimen occupies within the \( H \) coil. This can be minimized by separating the \( H \) and \( B \) coils sufficiently. Furthermore, high-field superconductors do not remain perfectly diamagnetic, because flux penetration begins at relatively low fields. This problem could be eliminated if the pulsed-field coil voltage were fed into the horizontal deflection system of the oscilloscope, if a proper phase relationship between the \( H \) and \( M \) signals could be maintained. In any case, the true
value of \( H \) should be obtained at the transition field, where the specimen becomes normal. This is substantiated through a comparison of maximum \( H \) deflection voltage for the calibration curve (no specimen in the B coil) with that for the magnetization run curve. Since the magnetization transition field (Fig. 3a) agrees well with the resistive transition field (Fig. 4) for high-field superconductors, it is apparent that the pulsed-field magnetization technique provides a useful and rapid method for the determination of \( H_{c2}^* \).

Nevertheless, the precision of \( H_{c2}^* \) as determined from the magnetization curve is very dependent upon the magnitude of \( M \) just before the transition where \( M \) goes essentially to zero. As a high-field superconductor approaches ideality the magnetization approaches very small values near the transition field, and the value of \( H_{c2}^* \) becomes imprecise. But this is of minor importance for exploratory research where materials are generally nonideal. A more serious problem involves the interpretation of magnetization measured in pulsed fields.

The magnitude of the magnetization itself is reduced by a uniform scale factor according to the area of the specimen, the area of the \( H \) coil and the area of the B coil. Thus, except for the slight non-linearity in apparent \( H \), the magnetization curve is not distorted by pickup coil geometry.

It has been shown that the critical current density \( (J_c) \) of a high-field superconductor is sensitive to pulsed-field rise time. Since the hysteresis in a magnetization curve is related to \( J_c \), it is expected that the magnitude of \( M \) will be sensitive also. Thus, less hysteresis should occur as the apparent \( J_c \) decreases, although induced eddy currents should work to increase the hysteresis. To what extent these balance each other
we do not know. If it were not for the fact that the lowering of \( J_c \) is nonlinear, i.e., the lowering is greater if the steady-field value of \( J_c \) is larger, we would think that semiquantitative comparisons could be made between magnetization curves. Qualitative comparisons can be made. Pulsed-field magnetization curves contain many features found in quasi-steady-field magnetization curves, including features such as the "peak effect" (Fig. 3a) that can be related directly to resistive measurements (Fig. 4). Flux jumps are observed at low fields for some materials (Fig. 3c). It may well be that pulsed-field magnetization curves are closer to steady-field magnetization curves than are the respective resistive counterparts \((J_c-H)\) curves because the induced supercurrents in the former are naturally limited by the response of the material and no excessive joule heating occurs as when an external current is applied. Comparative tests in steady and in pulsed fields will be necessary to evaluate this conjecture. Preliminary experiments on the flux-penetration field (actually the field for the initial magnetization peak) and the magnitude of the magnetization at that peak indicate a sensitivity to pulse rate (or "rise time").

Finally, we wish to point out that very high pulse rates can lead to eddy-current induced transition fields which are significantly lower than the equilibrium transition field \( H_{c2}^{*} \). Such pulse rates would have to be at least \( 10^3 \) G sec\(^{-1} \), that is, an order of magnitude greater than the largest used, if a significant decrease in the apparent \( H_{c2}^{*} \) were to occur.
V. SUMMARY

A pulsed magnetic field magnetization technique has been developed. Two coaxial, common center coils are used, one to detect the rate of change of the applied magnetic field and the other to detect the rate of change of the magnetic induction for a superconducting specimen. The signals are electronically integrated and properly combined so that a magnetization curve is displayed by the cathode ray tube of an oscilloscope. A high-speed photograph provides a permanent record of the curve. The field $H_{c2}^*$ at which the transition to normal behavior occurs can be determined easily and rapidly for high-field superconducting specimens that are imperfect. Increasing perfection of the specimens reduces hysteresis and makes the determination of $H_{c2}^*$ imprecise. The magnitude of the magnetization and the amount of hysteresis at fields below $H_{c2}^*$ may have semiquantitative significance, and hence, the technique may be useful for the rapid evaluation of high-field superconductors.

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8. M. Williams (to be published).


FIGURE CAPTIONS

Fig. 1  Magnetization curve (schematic) for an ideal Type II superconductor.

Fig. 2a  Schematic diagram of $B$ and $H$ sensing coils for measuring magnetization, $M$.

Fig. 2b  Schematic diagram of the critical field measuring apparatus.

Fig. 3a  The magnetization curve for NbN ($a_0 = 4.381 \pm 0.001\text{Å}$). The horizontal scale is 200 mv/cm and the vertical scale is 5 mv/cm.

Fig. 3b  The magnetization curve for NbC ($a_0 = 4.469 \pm 0.001\text{Å}$). The horizontal scale is 50 mv/cm and the vertical scale is 5 mv/cm.

Fig. 3c  Flux jump phenomena in Nb-N (mostly hexagonal with small amount of cubic NbN). The horizontal scale is 20 mv/cm and the vertical scale is 1 mv/cm.

Fig. 4  The critical current density, $J_c$, vs magnetic field, $H$ for NbN ($a_0 = 4.381 \pm 0.001\text{Å}$) showing the critical field, $H_{c2}^*$, and the "peak effect" in the resistive transition.
Fig. 1

Magnetization ($-4\pi M$)

Magnetic field (H)

$H_{C_1}$ $H_{C_2}$

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Fig. 2
Fig. 3b
Fig. 4
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