PHYSICAL REVIEW LETTERS

VOLUME 8

JUNE 15, 1962

NUMBER 12

OBSERVED SIGN REVERSAL OF A MAGNETIC FIELD PENETRATING A SUPERCONDUCTOR

K. E. Drangeid and R. Sommerhalder

International Business Machines Corporation, Zurich Research Laboratory, Zurich, Switzerland (Received May 14, 1962)

Measurements of the attenuation of a magnetic field in superconducting tin have revealed the sign reversal predicted by nonlocal theory.¹ These experiments give a straightforward demonstration of a nonlocal Meissner effect in tin. While this demonstration comes too late to be of primary importance, it suggests that the form of the penetration curve can be investigated in further experiments, leading to more detailed information on the integral kernel in the theoretical expression relating the density of supercurrents and the magnetic vector potential.

In this experiment, tin films were evaporated onto the outside surface of a rotating glass substrate to obtain long hollow cylindrical films. They were then cut from the vacuum system and cooled down to liquid helium temperatures. A magnetic field H_a at frequency $\nu = 1.1 \times 10^5$ cycles/ sec was applied parallel to the axis of the cylinder; the magnetic field H_i which penetrated through the films into the interior of the hollow body was detected. (Although the sign reversal is in fact a dc effect, it should be frequency-independent below microwave frequencies. The choice of ν was dictated by instrumental considerations.)

The equipment² was designed according to the results of our numerical calculations.^{2,3} A de-tailed account of the experimental work will be published later; it is, therefore, only briefly described here.

The current for the exciting field was produced by a sine wave generator; a maximum field strength of H_a = 30 Oe was available. The (strongly attenuated) magnetic field which penetrates the superconducting film was detected by a pickup coil placed inside the superconducting cylinder. This signal, tuned with an external capacity and amplified in a tuned amplifier, was compared with the drive current in the exciting coil on a dual beam cathode-ray scope with external triggering. Both the amplitudes and the phases of the two voltages could then be compared.

Figure 1 shows a plot of measured reciprocal field attenuation ratios H_a/H_i versus temperature T for two films. In the case of film I (31 000 Å thick), the field attenuation ratios are detectable only down to 3.4°K. At lower temperatures, H_i becomes immeasurably small; the plateau below 3.4°K, corresponding to $H_a/H_i = 1 \times 10^9$, reflects the limitation of our equipment. Film II had a thickness of 18 700 Å and a residual resistance ratio of r = 0.03. Measured reciprocal field attenuation ratios for this film are appreciably smaller than 1×10^9 at all temperatures measured, and thereby well within the demonstrated sensitivity of the equipment.

Figure 2 shows oscilloscope photographs of the exciting current and the corresponding pickup voltage taken on film II at $T = 3.41^{\circ}$ K and $T = 2.88^{\circ}$ K, respectively. Taking the current signals as reference, the 180° shift in the corresponding voltage signals is at once evident.

An analysis of the phase shift between exciting current and pickup voltage involves knowledge of the absolute phase shift of the apparatus. It turns out that the external and attenuated magnetic fields are in phase at $T = 3.41^{\circ}$ K and 180° out of phase at $T = 2.88^{\circ}$ K.



FIG. 1. Reciprocal field attenuation ratio H_a/H_i as a function of temperature T. o-Film I, 31000 Å; Δ -Film II, 18700 Å.

Finally, we wish to stress some important points on techniques of film preparation and measurement.

Our films (diameter 2 cm, length 15 cm) were evaporated at a pressure of $p \approx 10^{-9}$ mm Hg at a deposition rate of several 100 Å/sec on polished Pyrex glass substrates at a temperature between -100°C and room temperature. However, tin films evaporated under such clean conditions generally become rather grainy,⁴ and films so prepared by us showed appreciable leakage in the superconducting state. We found this difficulty could be avoided by precoating the substrates with SnO₂ and then never annealing them at a temperature higher than room temperature.

In addition, small holes of the order of 10^{-2} -mm diameter (as may be produced by dust) give rise to leakage signals⁵; such films were discarded.

The earth's magnetic field was compensated to better than 10^{-2} Oe in order to reduce effects of frozen-in flux.



FIG. 2. Signals representing the pickup voltage and exciting current photographed from the cathode-ray scope (Film II): (a) $T = 3.41^{\circ}$ K; (b) $T = 2.88^{\circ}$ K.



FIG. 3. Reciprocal field attenuation ratio $H_a/H_i(0)$ and phase shift $\varphi(\Delta)$ between H_a and H_i as a function of exciting field H_a (Film II at 2.88°K). Some high-field runs on this sample were also taken while cooling down to 2.88°K. It is possible that the detailed shape of this curve might have been affected thereby; the general form of the curve is, however, representative.

Externally applied fields must not exceed a few percent of the critical field H_c .⁶ Otherwise, normal conducting enclosures are formed in the film, and pronounced nonlinearity of the field attenuation is observed (Fig. 3) which may considerably affect the observation of the sign reversal or even mask it completely.

We acknowledge the important help of H. Müller and H. Seitz in these experiments. We also take the opportunity of thanking Professor P. Grassmann and Professor J. L. Olsen of the Institut fuer kalorische Apparate und Kaeltetechnik ETH, Zurich, Switzerland, for their continuous good will in making liquid helium available to us.

¹A. B. Pippard, Proc. Roy. Soc. (London) <u>A216</u>, 547 (1953).

²K. E. Drangeid, M. C. Gutzwiller, R. Sommerhalder, and H. Thomas, Proceedings of the Symposium on the Electric and Magnetic Properties of Thin Metallic Layers, Louvain, September, 1961 (to be published).

³R. Sommerhalder and H. Thomas, Helv. Phys. Acta <u>34</u>, 29 (1961) and <u>34</u>, 265 (1961). ⁴H. L. Caswell, J. Appl. Phys. <u>32</u>, 105 (1961).

⁵H. Kaden, <u>Die elektromagnetische Schirmung in der</u> <u>Fernmeldeund Hochfrequenztechnik</u> (Springer-Verlag, Berlin, 1950), p. 169.

⁶R. Jaggi and R. Sommerhalder, Helv. Phys. Acta <u>33</u>, 1 (1960).

STRUCTURE OF THE γ FORM OF SOLID He^{4†}

A. F. Schuch and R. L. Mills

Los Alamos Scientific Laboratory, University of California, Los Alamos, New Mexico (Received April 26, 1962)

In the course of measuring sound velocities in compressed He⁴, Vignos and Fairbank¹ discovered a new solid phase (designated the γ phase), which occurred in a narrow crescent-shaped band just above melting pressures, between 1.45 and 1.78°K. They observed that the new γ phase had a larger molar volume and entropy than the adjacent α phase, which was known to have the hexagonal close-packed (hcp) structure. Reasoning by analogy with the case of solid He³ which undergoes a similar transition,^{2,3} they thought it likely that the new phase in He^4 was body-centered cubic (bcc). For solid He³ the transition between the hcp and bcc structures produces a volume change, ΔV_{tr} , of ~ 0.12 cc/mole near the melting curve.² Assuming that the same structures apply to the new He⁴ transition, Schuch⁴ used the Pauling⁵ relation between bond length and coordination number to derive $\Delta V_{tr} = 0.187 \text{ cc/mole for } \text{He}^4 \text{ at } 28.9 \text{ atm and}$ 1.723°K. This value agrees closely with recent accurate measurements⁶ of ΔV_{tr} and provides evidence that the γ phase is bcc. Further indication for this structure comes from a sound velocity study⁷ made on mixtures of He^3 and He^4 . As the concentration of He^3 was raised from 0 to 100%, the P-T coordinates of the transition shifted smoothly upward to those known for the hcp-bcc transition in pure He³. The present experiment was aimed at a direct determination of the structure of the He⁴ γ phase by x-ray diffraction.

The very narrow pressure and temperature limits which define the γ phase of solid He⁴ made it necessary to modify the cryostat and cell arrangement used previously.⁸ The sample was contained in a Be cell (0.8-mm bore by 0.4-mm wall) which could be oscillated through an angle of forty degrees. The cell was surrounded at each end by a small container of liquid He which oscillated with the cell and which was kept supplied with liquid from the main stationary Dewar through flexible metal bellows. A copper radiation shield, soldered to the main He Dewar, surrounded the cell assembly. This shield had windows of 0.00035-in. thick Ni foil for transmitting and filtering the x rays. Surrounding it was another copper shield cooled to liquid N₂ temperature and equipped with thin (0.0003-in. thick) Al windows. A steel capillary filling tube was joined to the bottom end of the Be cell by a conical compression closure. The tube was coiled inside the He radiation shield to reduce thermal conduction to the cell. It was estimated that the portion of the Be cell exposed to x rays ran $\sim 0.04 \text{K}^{\circ}$ warmer than the He bath. Pressure in the cell inlet capillary was measured to ± 1 psi with a 0-1000 psi Heise gauge previously calibrated with a free-piston gauge. High-purity He was obtained from the Amarillo Station of the Bureau of Mines. It was further purified by passing it through a charcoal trap cooled with liquid N₂. The photographs were taken with a $4 - \times 5$ -inch flat plate camera placed 5 cm from the sample. The radiation used was $\operatorname{Cu} K_{\alpha}$ filtered, as mentioned, and collimated by a 0.040-in. aperture.

The sample was usually prepared by maintaining He at a suitably high pressure in the cell during cooling, although solid could be formed by cooling first and then raising the pressure above the freezing point. As was the case with the earlier work on He³ and He⁴, the x-ray reflections were single spots and not continuous rings, indicating that the solid was present in the form of large crystals and not as a powder.

It was found that solid He^4 does indeed undergo a structure change in the region described by Vignos and Fairbank.¹ The diffraction spots from the hexagonal cell were replaced by a set which could be indexed as the 110 and 200 reflections from a cubic body-centered cell. With respect to the 110



FIG. 2. Signals representing the pickup voltage and exciting current photographed from the cathode-ray scope (Film II): (a) $T = 3.41^{\circ}$ K; (b) $T = 2.88^{\circ}$ K.