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High-Field Galvanomagnetic Properties of Niobium

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The results of high-field galvanomagnetic measurements on a single crystal of niobium with a residual resistance ratio of 4750 are reported. These results resolve most of the uncertainties regarding the Fermi-surface topology of niobium which existed in previous reported data because of limited sample purity.

I. INTRODUCTION

IN our previous paper[1] on the galvanomagnetic properties of niobium and tantalum, several topological features of the niobium Fermi surface were left uncertain because samples of sufficiently high purity were not available. In this paper, we report the results of measurements on a purer sample of niobium (residual resistance ratio of 4750) in magnetic fields up to 105 kOe, which clarify several features of the Fermi surface that had been left in doubt. Specifically we propose answers to the following questions.

(1) Do the $\langle 110\rangle$-directed open orbits exist for all directions of the magnetic field in the $\{110\}$ planes, or do they cease to exist near the $\langle 110\rangle$ axes?

(2) Do two directions of open orbits exist when the field is parallel to a $\langle 110\rangle$ axis?

(3) What are the sizes of the two-dimensional regions of open orbits around the $\langle 100\rangle$ and $\langle 111\rangle$ axes?

(4) What are the values of the Hall coefficients when the field is parallel to the $\langle 100\rangle$ and $\langle 111\rangle$ axes?

II. EXPERIMENTAL DETAILS

The sample preparation was similar to that described in Ref. 1. The niobium single crystal was float-zone refined, degassed at 2300°C in a vacuum of 10^{-9}-mm Hg and then spark-cut into a rectangular parallelepiped 7.1 mm×2.45 mm×2.3 mm with the $[0\bar{1}1]$ axis parallel to within a degree of the current (long) direction. The sample holder, which has been previously described,[2] was modified so that the bottom section had a maximum diameter of $1\frac{1}{32}$ in. All measurements were made at 4.2°K in a superconducting solenoid that could generate fields up to 105 kOe. The dc potentials were detected by a milli-microvoltmeter and recorded on an x-y recorder as a function of either field strength or direction.

III. EXPERIMENTAL RESULTS

Most of the results of our measurements are summarized in Fig. 1. The lines and shaded areas indicate magnetic field directions in which open orbits are observed. These correspond to one- and two-dimensional regions, respectively. The size of the two-dimensional regions is somewhat smaller than in tantalum (compare with Fig. 1 of Ref. 1). The average diameter is 15° around $\langle 100\rangle$ (20° in Ta) and 3° around $\langle 111\rangle$ (7° in Ta).

We are now certain that the lines in the $\{110\}$ planes extend to the $\langle 110\rangle$ axes. Although the height of the $\langle 110\rangle$ ridge is about a factor of 10 smaller between $\langle 110\rangle$ and $\langle 111\rangle$ than between $\langle 111\rangle$ and $\langle 100\rangle$ it can still be seen. This implies that the number of $\langle 110\rangle$-directed open orbits is significantly decreased between $\langle 111\rangle$ and $\langle 110\rangle$ compared to between $\langle 100\rangle$ and $\langle 111\rangle$. The details of this ridge are shown in Fig. 2. This figure was obtained by tipping the field through the $(01\bar{1})$ plane and measuring the maximum signal at various field directions in the plane. The height is defined as the ratio of the maximum voltage to the voltage measured in a general field direction[3] near the $(0\bar{1}1)$ plane, where the magnetoresistance saturated ($\rho_{\text{peak}}/\rho_{\text{sat}}$).

The magnetoresistance saturates with increasing field at the $[011]$ axis, which suggests that this axis is either the center of a two-dimensional region or a

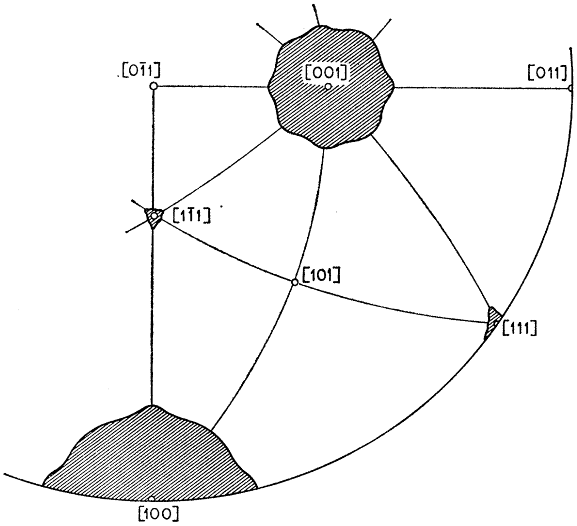

FIG. 1. Stereogram showing the occurance of open orbits in niobium.

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[1] E. Fawcett, W. A. Reed, and R. R. Soden, Phys. Rev. **159**, 533 (1967).

[2] G. F. Brennert, W. A. Reed, and E. Fawcett, Rev. Sci. Instr. 36, 1267 (1965).

[3] A "general field direction" is defined as a field direction which is not the center of a two-dimensional region and where no open orbits exist.

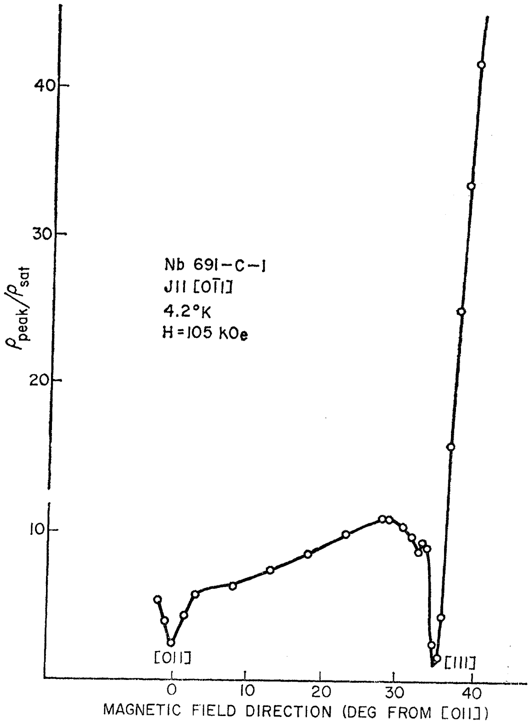

FIG. 2. Height of the open-orbit ridge in the (0$\bar{1}$1) plane between [011] and [111].

field direction that allows two directions of open orbits.[4] However, no two-dimensional region is found, and the Hall voltage is only 30% of the Hall voltage for a general direction at 100 kOe and is not a linear function of the field. We therefore conclude that two directions of open orbits exist when the magnetic field is along the ⟨110⟩ axes. The Hall voltage saturates above 60 kOe instead of being proportional to H^{-2}, but this is probably due to the fact that the Hall voltage has an extremely sharp minimum at ⟨110⟩ and the field was not perfectly along the axis.

The Hall voltage was measured with the field along [100] and [111], and the corresponding Hall coefficients are $R_{\langle 100\rangle}=1.99\times10^{-3}$ and $R_{\langle 111\rangle}=1.36\times10^{-3}$ abohm cm/Oe with an error of 2%. These coefficients correspond to net-carrier densities of $n_{\langle 100\rangle}=0.58$ and $n_{\langle 111\rangle}=0.84$ holes/atom. As is shown in Ref. 1, $R_{\langle 100\rangle}$ is related to the diameter of the arms of the "jungle-gym" measured in the ⟨100⟩-direction (Ref. 1, Figs. 8 and 10). Essentially, the decrease in the carrier density for this field direction compared to a general direction[3] measures the number of electron orbits due to the intersecting arms. This in turn is related to the arm diameter. From our present data we calculate an arm diameter $d_m=0.21(\Gamma H)$, which is approximately 17% larger than the value of $0.18(\Gamma H)$ we reported earlier in the less pure sample. Mattheiss[5] has recently calculated the band of niobium by the augmented plane-wave (APW) method. Figure 3 shows a cross section of the ⟨100⟩ arm passing through the point where the diameter (as well as the extremal area) is a minimum. The dimensions of the arm have been adjusted slightly (increased approximately 10%) to fit the area measured by magnetothermal oscillations.[6,7] The dimension calculated from the Hall data is marked d_m in the figure and is larger than $d_{[100]}$, the dimension measured in the (001) plane. The value of d_m, calculated by Mattheiss and adjusted by him to fit the magnetothermal data, is $0.207(\Gamma H)$, which agrees well with our experimental value.

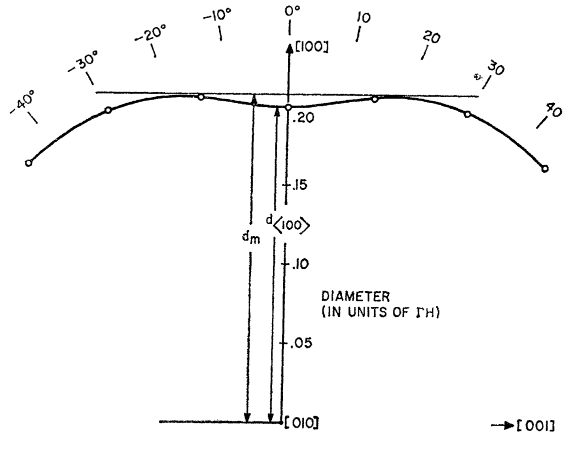

FIG. 3. Cross section of the "jungle-gym" arm at its minimum area (courtesy of L. F. Mattheiss).

IV. CONCLUSION

The galvanomagnetic measurements reported show that the ⟨110⟩-directed open orbits exist for all direction of the field in the {110} planes (except when exactly along ⟨100⟩ and ⟨111⟩) and open orbits exist in two nonintersecting directions when the field is parallel to ⟨110⟩. Hall measurements for the field along ⟨110⟩ give a minimum diameter of the arms of the "jungle gym" to be $0.21(\Gamma H)$.

A recent letter by Alekseevskii *et al.*[8] reports galvanomagnetic measurements on niobium crystals with resistance ratios from 400–1000. They do not report the two-dimensional region around ⟨111⟩ and measure an arm diameter $d_m=0.15(\Gamma H)$. This low value for d_m

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[4] According to the successful theory of I. M. Lifshitz, M. Ya Azbel', and M. I. Kaganov {Zh. Eksperim. i Teor. Fiz. 35, 1251 (1958) [English transl.: Soviet Phys.—JETP 8, 875 (1959)]}, for a field direction that permits two directions of nonintersecting open orbits, the magnetoresistance saturates with increasing field and the Hall coefficient is proportional to H^{-2}.

[5] L. F. Mattheiss, Bull. Am. Phys. Soc. 13, 508 (1968); and (private communication).

[6] M. H. Halloran, F. S. L. Hsu, and J. E. Kunzler, Bull. Am. Phys. Soc. 13, 59 (1968).

[7] J. E. Graebner, J. H. Condon, F. S. L. Hsu, and J. E. Kunzler, Bull. Am. Phys. Soc. 13, 508 (1968); and (private communication).

[8] N. E. Alekseevskii, K. H. Bertel, A. V. Dubrovin, and K. E. Karstens, Zh. Eksperim. i Teor. Fiz., Pis'ma v Redaktsiyu 6, 637 (1967) [English transl.: Soviet Phys.—JETP Letters 6, 132 (1968)].

is probably a result of the field not being accurately aligned along the [100] axis and the relatively low purity of their samples. They also report anisotropy of the resistance in fields of 2–4 kOe and attribute it to either anisotropy of the impurity or defect distribution in the sample, or anisotropy of the superconducting energy gap. Measurements by Reed *et al.*[9] on the anisotropy of H_2 in niobium and NbTa alloys show that this anisotropy is not due to the superconducting energy-gap anisotropy and is probably due to either the defect distribution or some nonuniformity of the surface.

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[9] W. A. Reed, E. Fawcett, P. P. M. Meincke, P. C. Hohenberg, N. R. Werthamer, in *Proceedings of the Tenth International Conference on Low-Temperature Physics, Moscow, 1966*, edited by M. P. Malkov (Proizvodstrenno-Izdatel'skii Kombinat, VINITI, Moscow, 1967), Vol. IIA, p. 368.

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Steady-State, ac-Temperature Calorimetry*

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A steady-state technique for measuring heat capacity using ac heating is described. Heat is applied sinusoidally in time to a sample coupled thermally to a reservoir; the resultant equilibrium temperature of the sample contains a term that is both inversely proportional to the heat capacity and measurable with high precision. The effects of various corrections that must be applied to the data are considered in detail. Measurements of the absolute magnitude of the heat capacity of indium and the field dependence of the heat capacity of beryllium have been made and are used to illustrate the power of the method. The observed quantum oscillations in the heat capacity of beryllium are in agreement with predictions based on other measurements.

INTRODUCTION

LOW-TEMPERATURE calorimetry often suffers from the transient nature of the traditional measurements—a characteristic that makes difficult the use of any of the now highly developed signal-averaging techniques to extract the wanted signal from noise. This limitation is of little importance in the measurement of the absolute value of heat capacity where other considerations, mainly thermometer calibration, already limit the accuracy of the measurements. In the determination of small changes in the heat capacity, however, the signal-to-noise ratio can become the limiting factor.

Additional disadvantages of traditional techniques stem from the necessity of thermal isolation of the sample from its surroundings. A sample must be quite large to minimize the effects of stray heat leaks. Helium exchange gas or a complicated heat switch may be required to obtain sufficient isolation during a heat-capacity measurement and yet cool down the sample in a reasonable length of time. A sample suspension system trades off mechanical stability to accomplish thermal isolation, possibly making the apparatus sensitive to vibrations.

To alleviate the problems discussed above, a steady-state calorimetry technique employing ac heating has been developed.[1–3] This method makes possible a much more precise measurement of heat-capacity changes as a function of an external parameter, although the absolute accuracy of the heat capacity data is no better than that obtainable with the traditional methods. The sample can be quite small. Neither exchange gas nor a heat switch is required. The suspension system can be quite rigid if the sample is of a reasonable size. The sample is connected thermally to a heat reservoir and so rapidly returns to thermal equilibrium after experiencing any extraneous heat input. Finally, a continuous read-out of the heat capacity is possible.

In the following sections the general theory of the method is presented and measurements are reported on indium and beryllium illustrating the power of the technique.

THEORY

A. Sample of Infinite Thermal Conductivity

Consider a system, as in Fig. 1, consisting of a heater, thermometer, and sample, each assumed to have infinite thermal conductivity and heat capacities C_h,

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[1] P. Sullivan and G. Seidel, Ann. Acad. Sci. Fennicae **A210**, 58 (1966).

[2] P. Sullivan and G. Seidel, Phys. Letters **25A**, 229 (1967).

[3] P. Sullivan, thesis, Brown University (unpublished).