

MEASUREMENT OF THE RESISTANCE OF SINGLE CRYSTALS IN A PULSED MAGNETIC FIELD

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A method is described for measuring low electrical resistances of single crystals at low temperatures in pulsed magnetic fields. The method has been used to investigate the galvanomagnetic properties of molybdenum, palladium, rhenium, and beryllium.^[9-11]

1. INTRODUCTION

INVESTIGATION of the galvanomagnetic properties of metals requires measurements of the resistance of pure single-crystal samples in strong magnetic fields at low temperatures.^[1] Electromagnets with field intensity of about 30 kOe are usually employed for this purpose.^[2] Much stronger fields may be obtained by the use of pulse coils.^[3]

As is well known, pulsed fields were first used to study the change of resistance in a magnetic field by P. L. Kapitza. Using a coil with 2.0 turns per centimeter, across which a special generator was short-circuited, he was able to obtain a field of ≈ 350 kOe. To record the change in resistance Kapitza used a loop oscillograph.^[4]

Recently Olsen^[5] and Lüthi^[6] have carried out measurements of the resistance in pulsed magnetic fields of ≈ 250 kOe, obtained by discharging a capacitor bank through a coil with a large number of turns, placed in liquid helium. A similar method of producing pulsed fields was used earlier by Shoenberg^[7] to study the de Haas-van Alphen effect. Since up to now the change of resistance in pulsed magnetic fields has been studied only for polycrystalline samples, and since in Fermi surface studies it is necessary to carry out measurements on single crystals, it was of interest to assemble apparatus capable of carrying out such measurements.

2. CONSTRUCTION OF THE APPARATUS

In the method described here the field was produced by discharging a capacitor bank of $1600 \mu\text{F}$ capacitance through a solenoid placed in liquid helium. The solenoid (Fig. 1) had about 1000 turns/cm. The winding consisted of a copper wire of 0.3

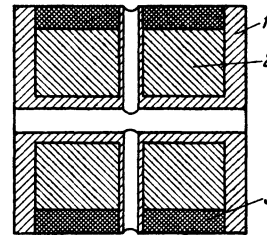


FIG. 1. Pulse coil: 1) Teflon casing; 2) copper winding; 3) belt. The field is directed horizontally; the sample is placed in the vertical aperture.

mm diameter. The bank was charged to 2000 V and in this way fields of up to 180 kOe were obtained; the pulse duration was about 10^{-2} sec. To give the coil mechanical strength the winding was bonded layer by layer with an epoxy resin and reinforced with a belt of nylon fiber. The pulse coil was in the lower part of the cryostat with the coil axis directed horizontally. The sample was placed in the uniform region of the field. The sample holder was closed with a ground-glass joint, making it possible to rotate the sample in the solenoid and thus record the angular variation of the resistance in a magnetic field.

In measuring the resistance of single-crystal samples in strong pulsed magnetic fields one meets with several special difficulties connected, for example, with dynamic stresses acting on the sample, induced emf's, etc. To eliminate the torques acting on the sample, the measuring current was supplied coaxially and the holder itself was used as the coaxial lead. The holder consisted of two coaxial stainless-steel tubes which acted simultaneously as the holder components and the current leads (Fig. 2). Copper wire of 0.05 mm diameter was used for the potential leads. These leads were wrapped round the sample and beyond it were twisted tightly, thus making it possible to reduce

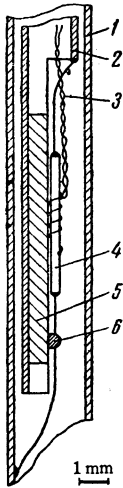


FIG. 2. Sketch of the sample holder:
1) and 2) stainless-steel tubes; 3) potential leads; 4) sample; 5) glass base; 6) BF-2 glue.

the induction interference considerably.

The use of ac gave rise to vibrational loads on the sample. The forces were too small to deform the single crystal, but if the sample was not specially fixed it vibrated in the field, giving rise to considerable interference. A simple and reliable method of preventing this vibration was to fill the shaft of the holder with alcohol.

In order to reduce the direct influence of the strong pulses of the coil leakage field on the instruments, the whole measuring apparatus was placed at a sufficiently large distance from the coil.

3. MEASUREMENT METHOD

To record signals of 10^{-5} – 10^{-6} V due to the change in the resistance, we used an ac measuring system with resonance amplification (Fig. 3). An

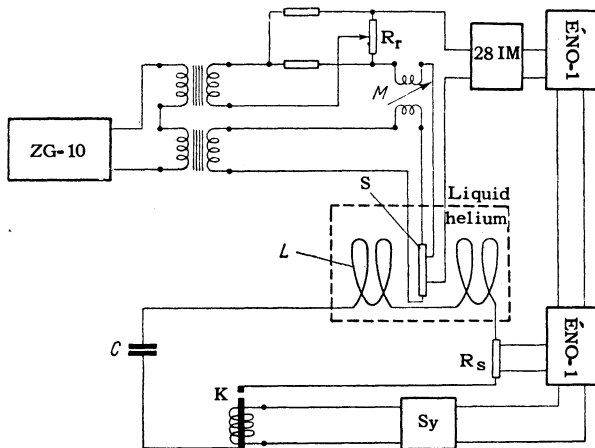


FIG. 3. Block diagram of the measuring circuit: C is the capacitor bank; L is the pulse coil; S is the sample; K is the switch for the coil current; Sy is a device for synchronizing the capacitor discharge through the coil with the horizontal scan of the oscillograph.

audio-frequency ac current (2.5–5 kc) was applied to the sample S. The current in the sample was usually about 0.1–1 A. The signal from the potential leads was fed to a resonance amplifier 28 IM and then to the vertical scan of an ÉNO-1 oscillograph. In series with the measured signal we fed to the amplifier input a voltage which compensated the induction due to the reactive coupling in the supply leads. This induction had the frequency of the measuring current. The active and reactive components of the compensating voltage were taken, respectively, from a slide-wire rheostat R_r and a mutual-inductance M. To avoid electric coupling of the amplifier input to the audio oscillator, the current was supplied to the sample, calibration system and to the compensation system via transformers.

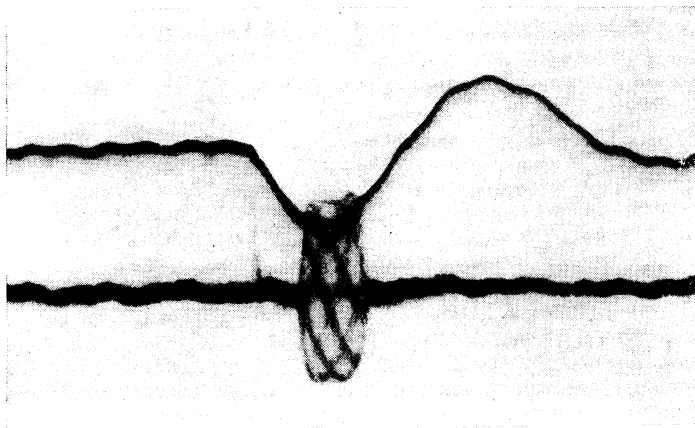
On switching on the measuring current through the sample a signal appeared at the potential leads which consisted of the potential difference across the sample in the absence of the magnetic field and of the reactive induction. By varying the position of the slider on the rheostat R_r and the mutual-inductance coefficient M, this signal can be easily compensated. In this way a "zero" signal was applied to the amplifier input in the absence of a field. The magnitude of this signal determined the sensitivity of the circuit. Usually the "zero" signal amounted to $\approx 2 \times 10^{-7}$ V for a measuring current of ≈ 1 A. This value was somewhat larger than the amplifier noise because of the presence of higher harmonics in the measuring current.

The resistance of the sample changed in a magnetic field and the amplifier input received an unbalance signal proportional to $\Delta\rho(H) = \rho(H) - \rho(0)$. The recording of field and resistance signal was done either on a two-beam oscillograph (Fig. 4¹⁾), or on two single-beam oscillographs with synchronized horizontal scans. Switching on the current through the pulse coil was synchronized with the horizontal scan of the oscillographs, and the current was switched on with an electromagnetic relay. To record the current passing through the coil, the oscillograph showed the potential difference across a standard resistance R_s in the coil circuit. The solenoid constant needed in calculations of the magnetic field intensity was determined with a ballistic circuit.

Analysis of the results of measurements was carried out using oscillogram photographs of the

¹⁾We used here a single-beam instrument with an electronic switch at its input instead of the two-beam oscillograph. In the process of testing the method we carried out measurements on the destruction of the superconductivity.

FIG. 4. Oscillogram of the destruction of the superconductivity of a Nb-Zr sample at $T = 4.2^\circ\text{K}$. The upper beam recorded the current through the coil and the lower one the voltage signal from the potential electrodes. The superconductivity was destroyed in a field $H = 45 \text{ kOe}$.



type shown in Fig. 5a. The maximum amplitudes of the field, corresponding to the points A_1, A_2, A_3 , etc., (Fig. 5a), were determined. Since the function $\Delta\rho(H)$ is usually non-decreasing, the changes of the resistance corresponding to these values of the field were also determined at the points of the maximum unbalance signal B_1, B_2, B_3 , etc. (Fig. 5b). The exceptions were the cases when the function $\Delta\rho(H)$ was decreasing, for example in the case of a large negative Hall emf (Fig. 6b).

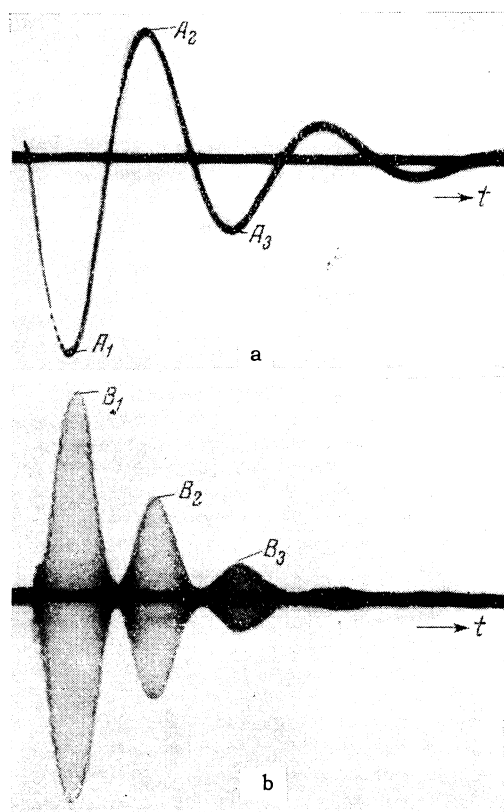


FIG. 5. Oscillogram of resistance measurements on a Pd sample in an oscillating magnetic field: a) oscillogram of the magnetic field variation; b) oscillogram of the unbalance signal proportional to the change in the resistance.

The Hall emf was measured by the usual method of field reversal and for an oscillating discharge the reversal was carried out automatically (Figs. 6a and 6b).

If the function $\Delta\rho(H)$ can become negative, and this may occur in the case of large Hall emf's, or if the resistance has a minimum, it is necessary to know the sign of the signal coming from the potential electrodes. To determine this sign a voltage of the same phase as the current was applied to the horizontal plates of the second oscillograph; the unbalance signal was passed as usual through the amplifier to the vertical plates. The ellipses appearing on the screen at the moment of the field pulse were rotated by a certain angle with respect to the horizontal axis. The sign of this angle determined the sign of the signal from the potential electrodes.

The errors in analysis of the oscillogram photographs, due to the inaccuracy of readings, amounted to about 3%. The systematic error, due to the inaccuracy in the determination of the calibration resistances and the solenoid constant, amounted to about 10%.

Apart from these errors, fundamental errors are possible due to the skin effect and heating of the sample. To reduce the influence of the skin effect it is desirable to use samples which are as small as possible. The transverse dimensions of our samples were usually 0.2–0.5 mm, which is considerably less than the skin-effect depth. In cases when the influence of the skin effect becomes important it may be eliminated by carrying out measurements at different frequencies of the measuring current. The influence of the skin effect due to the oscillatory nature of the magnetic field itself was considerably smaller because the frequency of these oscillations $\Omega \approx (LC)^{-1/2}$ (where L is the inductance of the pulse coil and C is the capacitance of the capacitors) was at least one order of magnitude lower than the frequency of the meas-

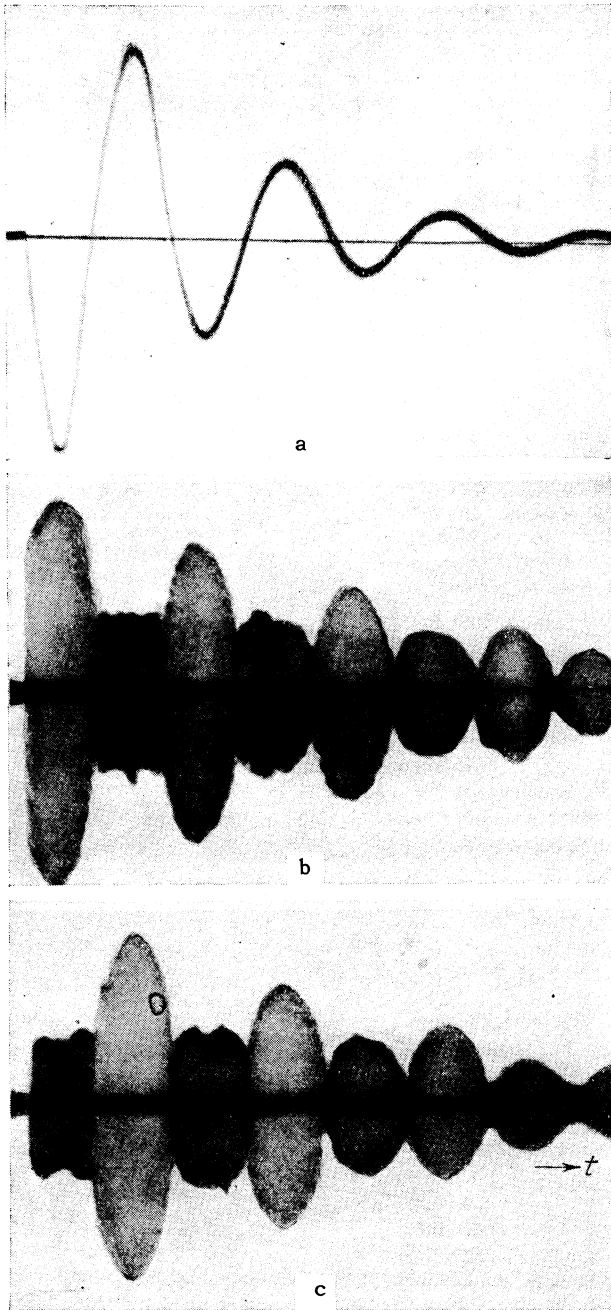


FIG. 6. Oscillograms of the change in resistance and Hall emf for an Re sample: a) oscillogram of the magnetic field variation; b) and c) oscillograms of the signals from the potential electrodes, proportional to the change in the resistance and Hall emf. The direction of the field for the oscillogram b was opposite to the direction for the oscillogram c; therefore the same direction of the field corresponds to the odd peaks in the oscillogram b and the even peaks in the oscillogram c. The other peaks represent the opposite direction of the field. The difference is due to the Hall emf.

uring current, and if necessary it could have been easily reduced.

As for the errors due to sample heating, it should be noted that the heating due to the eddy currents as well as that due to the measuring current are proportional to the electrical conductivity. However, from order-of-magnitude estimates for samples with transverse dimensions of several tenths of a millimeter and having an electrical conductivity $\sigma \approx 10^8 \Omega^{-1}\text{-cm}^{-1}$ the heating should not exceed 1–2 deg C. These estimates were well confirmed by our experiments on the destruction of the superconductivity and by the results obtained by Trořnar.^[8] The influence of sample heating and the skin effect may be considerable only in the case of very pure materials. However, the present method was developed for measurements on not very pure samples, for which the usual fields of ≈ 30 kOe are not strong effective fields ($H_{\text{eff}} = H\rho(300^\circ\text{K})/\rho(4.2^\circ\text{K})$).

Using this method the present authors carried out studies of the galvanomagnetic properties of molybdenum, palladium, beryllium, and rhenium in strong magnetic fields; the results have been published earlier.^[9,10,11]

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¹ Lifshitz, Azbel', and Kaganov, JETP 31, 63 (1957), Soviet Phys. JETP 4, 41 (1957).

² N. E. Alekseevskiĭ and Yu. P. Gařdukov, JETP 36, 447 (1959), Soviet Phys. JETP 9, 311 (1959).

³ G. M. Strakhovskiĭ and N. V. Kravtsov, UFN 70, 693 (1960), Soviet Phys. Uspekhi 3, 260 (1960).

⁴ P. L. Kapitza, Proc. Roy. Soc. (London) A123, 292 (1929).

⁵ J. L. Olsen, Helv. Phys. Acta 26, 798 (1953).

⁶ B. Lũthi, Helv. Phys. Acta 33, 161 (1960).

⁷ D. Schoenberg, Nature 170, 569 (1952).

⁸ E. Trořnar, Candidate's Dissertation, Moscow State University, 1961.

⁹ Alekseevskiĭ, Egorov, Karstens, and Kazak, JETP 43, 731 (1962), Soviet Phys. JETP 16, 519 (1963).

¹⁰ Alekseevskiĭ, Egorov, and Kazak, JETP 44, 1116 (1963), Soviet Phys. JETP 17, 752 (1963).

¹¹ N. E. Alekseevskiĭ and V. S. Egorov, JETP 45, 388 (1963), Soviet Phys. JETP 18, 268 (1964).

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