PRACTICUM III

Fundamental Basics of Modern Technologies

Practical #5

MEASUREMENT OF TRANSPORT PROPERTIES OF EPITAXIAL TiN FILM IN NORMAL AND SUPERCONDUCTING STATES

PURPOSE OF WORK: (1) Experimental study of the temperature dependence of resistance of a sample, measurement of current-voltage characteristics and differential resistance for a sample in the normal and superconducting state. (2) Determination of film parameters: sheet resistance R_s; residual resistance ratio – RRR; the critical temperature of the superconducting transition T_c, the critical current of a sample in the superconducting state. (3) Get acquainted with modern equipment used for transport measurements of thin films.

INTRODUCTION

Thin metal films with thicknesses (d) smaller than 1 μm, are an important component of modern low-temperature electronics.

Characteristics of thin metal films:

Sheet resistance R_s is resistance of a square of a sample. In the ideal case, when the layers are completely homogeneous in terms of structure and thickness, the relation between the sheet resistance R_s and resistivity ρ is the following: $\rho = R_s d$. From this expression it follows that the sheet resistance R_s increases with decreasing thickness of the film.

RRR (residual resistance ratio) is the ratio RRR = R(300K)/R(10K), an experimentally measured parameter that characterizes metal properties of the film. Large RRR value (> 1) is associated with a pure metal.

The critical temperature of the superconducting transition T_c is the temperature upon cooling below the sample becomes a superconductor, with the resistance R=0.

Critical current I_c is the maximum value of direct current (dc) that can flow through a superconductor without energy dissipation. If the current exceeds the critical value, then the some part of the superconductor jumps to the normal (non-superconducting) state. At the normal state, the current flows with the energy dissipation, leading to the heating of the sample.

Retrapping current I_{ret} is the value of the dc current below which the sample regain its superconducting state from the normal state. If it turns out to be less than the critical current I_c, then the current-voltage characteristic of the sample becomes hysteretic.

Description of R(T)-dependence of a thin metal film.

The electrical resistivity of a thin film arises from a variety of scattering mechanisms such as electron-phonon scattering, electron-defect scattering, or the scattering of the electrons at impurities. The famous Matthiessen's rule states that, if these scattering mechanisms are independent from each other, the resistivity of a film is the sum of the individual contributions of different scattering sources:

$$\rho = \underbrace{\rho_{\mathrm{ph}}}_{\rho(T)} + \underbrace{\rho_{\mathrm{defect}} + \rho_{\mathrm{impurity}} + \rho_{\mathrm{boundary}}}_{\rho_{\mathrm{res}}}$$

where ρ_{ph} is the resistivity due to electron-phonon scattering, ρ_{defect} the resistivity due to electron-defect scattering, and $\rho_{impurity}$ the resistivity due to electron-impurity scattering, and $\rho_{boundary}$ the resistivity due to electron scattering at the boundaries of the sample. The three latter resistivities can be summed up to the residual resistivity ρ_{res}, which is the temperature independent.

An alternative formulation of Matthiessen's rule in terms of respective scattering rates has the form:

$$\frac{1}{\tau} = \frac{1}{\tau_{\mathrm{ph}}} + \frac{1}{\tau_{\mathrm{defect}}} + \frac{1}{\tau_{\mathrm{impurity}}} + \frac{1}{\tau_{\mathrm{boundary}}}$$

where the respective scattering times τ indicate the average time between two collisions of an electron.

The typical temperature dependence of the resistivity of a metal is sketched in Figure 1. All metals exhibit the characteristic behavior of decreasing resistivity with decreasing temperature. This is caused by the scattering of electrons at the quantized quasiparticles of the lattice vibration, phonons. The scattering rate τ^{-1}_{ph} for electron-phonon scattering increases proportionally to the number of occupied phonon states. At high temperatures (>200K), this number increases linearly with temperature. Hence, a linear increase of the resistivity is expected in this temperature range:

$\rho_{ph} \propto \tau^{-1}_{ph} \propto T$.

At low temperatures the electrical resistivity of a thin metal film usually does not depend on temperature (see Figure 1). In that regime, the scattering of electrons at defects and impurities dominates.

Additional note: In macroscopic systems, the effect of sample boundaries on the resistivity can normally be neglected. Only at low temperatures and in very clean crystals, where the electron mean free path is of the same order of the sample size, boundary scattering potentially becomes important. In thin films, with the film thickness $d \sim l$, additional scattering mechanisms occur, which increase the resistivity of the system. The two fundamental mechanisms are:

(i) scattering of electrons at external surfaces or interfaces, and

(ii) scattering of electrons at grain boundaries.

As the resistivity is no longer a constant but depends on the dimension of the system these deviations are also called size effects.

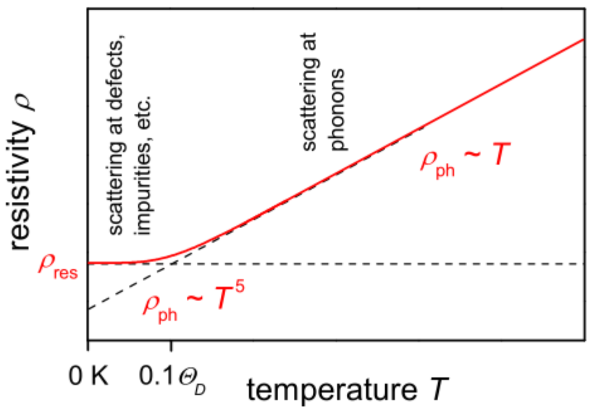


Fig.1 Schematic diagram illustrating the temperature dependence of the electrical resistivity for metallic systems. At high temperatures, the scattering of electrons at phonons dominates, at low temperatures, the scattering at defects and impurities plays the major role.

Description of superconducting properties of a thin metal film

The typical temperature dependence of the resistance sample is shown in Fig.2(a). The resistance falls down to a zero value at some temperature T_c, which is the critical temperature of the superconducting transition. Usually, this temperature is determined as a temperature at which the resistance corresponds to $0.5R_n$, where R_n is the resistance in the normal state, just above the transition. Another important characteristic of a superconducting sample is its critical current, I_c. The critical current can be determined using the current-voltage characteristics (Fig.2(b)). The value of I_c is also a temperature dependent parameter: the lower the temperature, the higher the critical current is.

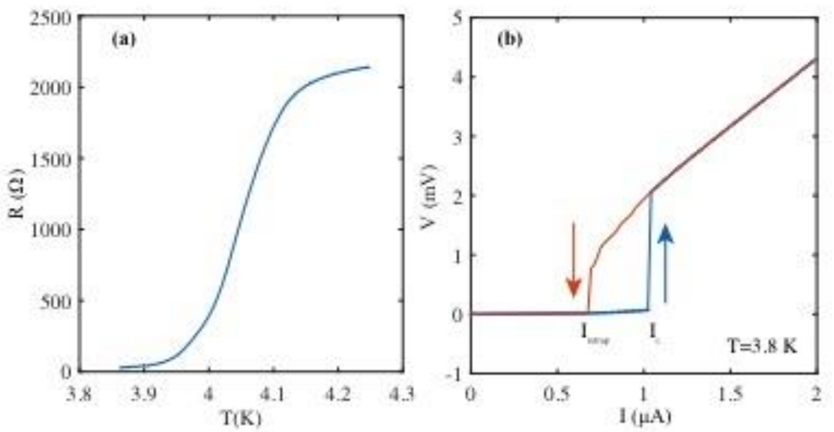


Fig.2 (a) Temperature dependence of the resistance for a superconducting TiN nanobridge. (b) Typical current-voltage characteristics of the TiN nanobridge at the superconducting state. The bath temperature is 3.8 K. The arrows show the direction of increasing (decreasing) of the bias current.

Static and differential resistance in nonlinear circuits

The ratio of voltage to current at a fixed point on the characteristics is usually referred to as *the static resistance*. In linear electrical circuits (Fig.3(a)), the resistance does not change when the current or voltage changes, so $V_1/I_1 = V_2/I_2 = R$.

For non-linear loads (Fig.3(b)), the static resistance at each point of the IV-characteristics is different, and it changes when the current or voltage changes:

$r_{1st} = V_1/I_1$; $r_{2st} = V_2/I_2$.

If it is necessary to consider fast processes in a nonlinear circuit, someone usually uses the concept of the *dynamic (differential) resistance*. The differential resistance at any point of the IV-characteristics (Fig. 3(b)) is determined by the tangent at this point:

$r_{1diff} = \delta V_1/\delta I_1$, $r_{2diff} = \delta V_2/\delta I_2$.

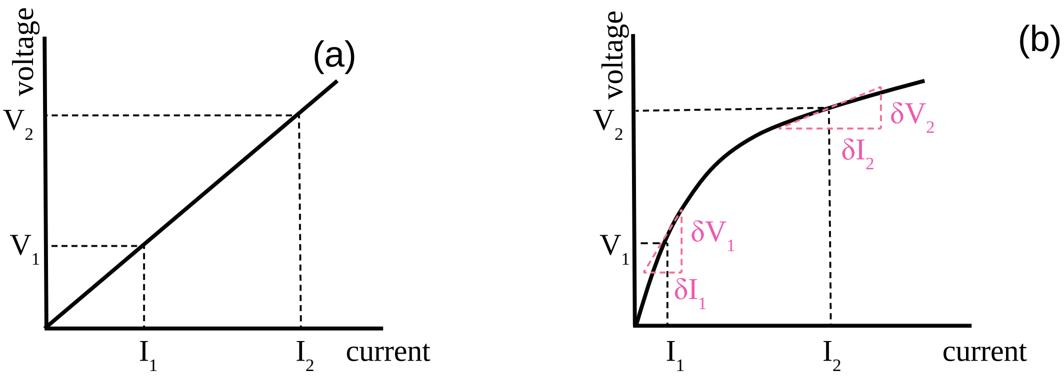


Fig.3 *Schematic diagram illustrating the static and differential resistance in linear (a) and nonlinear (b) circuits.*

EXPERIMENTAL METHODS

The sample, studied in this work**,** is a thin 20-nm thick TiN film. The film is patterned in the form of a Hall bridge, which allows measurements in a 4-probe configuration.

In the four-probe method, the conductive layer has the shape shown in Fig.4, where, unlike the simple 2-probe method, the sample has the additional voltage probes.

The sheet resistance of the film can be calculated using the following formula:

$R_S = w/l\ U/I$

where l - distance between the voltage contacts, w is the width of the sample. The resistivity of the material is determined as $\rho = R_s d$.

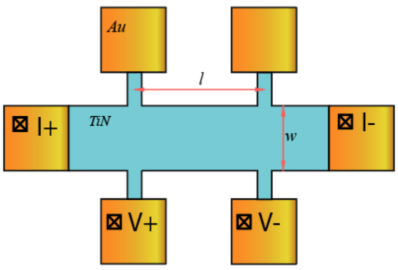


Fig.4. Schematic diagram illustrating a 4-probe connection. d=20nm, w=500um, l=1000um.

Advantages of this method:

- contact resistance problems are eliminated;
- uniform current flow from the left to the right voltage contact.

The experimental setup for measuring static and differential resistance and current-voltage characteristics is shown in Fig.5. For measurements at temperatures down to 4.2 K, dipstick is used which allows work with the Dewar vessel. The sample is mounted to the holder. The experimental setup is made in a way that the sample is located near to the diode thermometer. Each high-resistance wire comes to each contact of the sample that provides a 4-point circuit.

The experiments at low temperatures include three types of measurements:

1. To measure the *temperature dependence of the resistance*, you need to use a LakeShore A370 resistance bridge. The sample should be biased with a small alternating current (10 uA) at low frequency. The diode thermometer is biased by dc current (10 uA), the voltage from the diode thermometer is read by LakeShore 218 temperature monitor and then is converted into Kelvin degrees.

2. To measure *the current-voltage characteristics* at a fixed temperature $V(I)(I_{dc},T_{bath})$, a current source *Yokogawa G200* and voltmeter *Keysight 34461A* are used. The sample should be biased with a dc current I_{dc} from the dc source. To bias the sample by dc current I_{dc} the bias voltage should be applied through the load resistance R_{load} (it's preinstalled inside the handmade Bias-tee), which is greater than the total resistance of the sample with contacts and setup wires.

(The max. voltage output of Yokogawa G200 is 32 V, into Bias-tee R_{load}(dc)=2.16 kOhm, I_{dc}(max)=14.8 mA). To measure voltage across the sample it would be better to use *preamplifier SR560* with voltage gain=10. Connect A and B outputs from Bias-tee to A and B inputs of the preamplifier, and choose the regime A-B, dc, low pass filter 100 kHz. After amplification, the voltage drop across the sample is measured with the voltmeter, so, connect preamplifier 50 Ohm-output to the voltmeter.

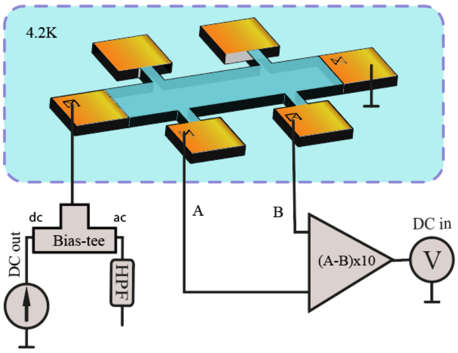


Fig.5. The experimental setup for measuring the current-voltage characteristics.

3. To measure the dependence of the differential resistance on current at a fixed temperature $dV/dI(I_{dc},T_{bath})$, the initial setup should be the same as in p.2. Also the ac(alternating current) bias voltage from lock-in output (sine out, regime internal) should be applied through the load resistance R_{load} (it's preinstalled inside the Bias-tee), (For example, voltage output of lock-in SR830 is 0.5 V, into Bias-tee R_{load}(dc)=203 kOhm, I_{dc}(ac)=2.4 uA). The ac voltage drop across the sample will be also measured after preamplifier with the Lock-in SR830 (input A, regime A). The sample should be biased additionally with a dc current I_{dc} from a dc source Yokogawa G200 (p.2). To feed in at the same time ac and dc current you need to use a bias-tee.

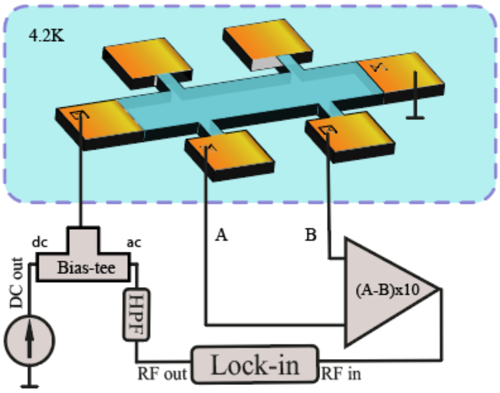


Fig.6. The experimental setup for measuring the current-voltage characteristics and differential resistance.

Bibliography

1. L. I. Maissel and R. Glang, Handbook of Thin Film Technology, McGraw-Hill,New York, 1970
2. A.F. Ioffe, A.R. Regel. Prog. Semicond. 4, 237 (1960).

Equipment

1. Sample (see above)
2. Dewar vessel, dip-stick (homemade)
3. Handmade Bias-tee of DC and RF bias
4. Resistance bridge Lakeshore A370

http://research.physics.illinois.edu/bezryadin/labprotocol/lakeshore_370_Manual.pdf

5. Lock-in SR830

https://www.thinksrs.com/downloads/pdfs/manuals/SR830m.pdf

6. Temperature monitor Lakeshore 218

https://www.lakeshore.com/docs/default-source/product-downloads/218_manual.pdf?sfvrsn=1ea170a7_1

7. Current/Voltage source Yokogawa GS200

https://cdn.tmi.yokogawa.com/1/6218/files/IMGS210-01EN.pdf

8. Voltmeter Keysight 34461A

https://literature.cdn.keysight.com/litweb/pdf/34460-90901.pdf

9. Preamplifier SR560

http://research.physics.illinois.edu/bezryadin/labprotocol/SR560Manual.pdf

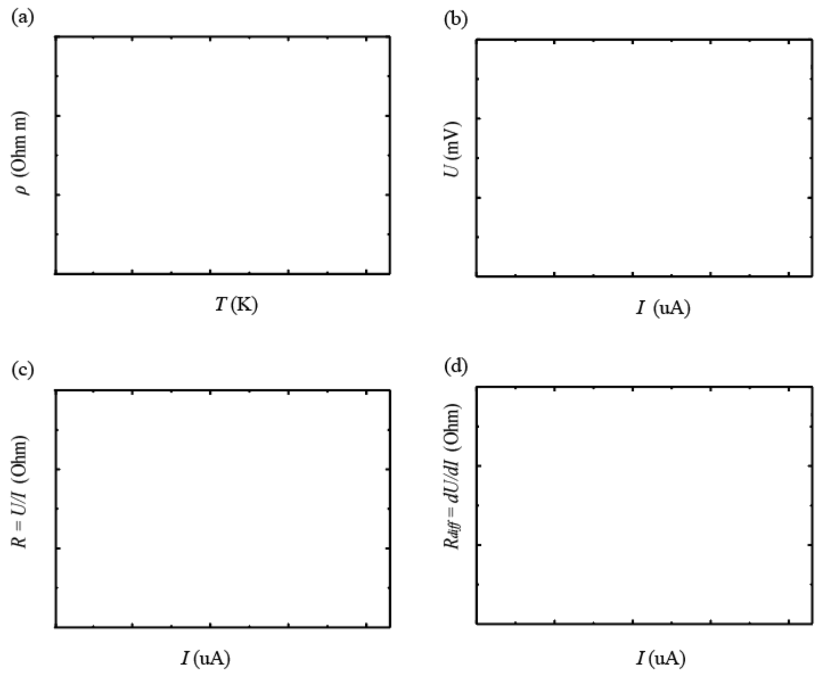
MEASUREMENT PROCEDURE

1) Assemble the electrical circuit of the setup.
2) Measure the R(T)-dependence from 300K to 4.2K.
3) Set the base temperature in the range from 8 to 30 K. Obtain information about the step of changing the current from the laboratory assistant, apply voltage to the circuit through a resistor (turn on the current source), write the value of R_{diff} and U.
4) Set the base temperature to 4.2 K, do step 3.

PRESENTATION of the RESULTS

Plot the graphs in any scientific graphing and data analysis software (Origin, Matlab, etc).
Using the graphs determine the parameters of the Sample (Table 1).

- Plot the dependence of resistivity on temperature on a linear scale **(a)**
- Plot the voltage vs. bias current on a linear scale at T = 10 K and T = 4.2 K **(b)**
- Plot the dependence of the static resistance on the bias current on a linear scale at T = 10K and T = 4.2K **(c)**
- Plot the dependence of the differential resistance on the bias current on a linear scale at T=10K and T = 4.2K **(d)**
- Add the data in Table 1.



R_s(300K)	R_s(10K)	RRR	ρ(10K) (Ω m)	T_C (K)	I_C(4.2K) (uA)	Iret(4.2K) (uA)
..	..	..	..			

PRECEDING DISCUSSION QUESTIONS

1. 4-point (quasi-four-point) measurement scheme, 2-point measurement scheme. What is difference between these schemes?
2. How does the resistance bridge operate?
3. How does the synchronous phase detector Lock-in SR830 measure the static resistance and differential resistance?
4. What is the difference between static and differential resistances in the normal state?
5. 6. What is the difference between static and differential resistances in the superconducting state?
6. What is the “superconducting state”? How it is formed according to the BCS-theory?

DEFENDING DISCUSSION QUESTIONS

1. Clean metal films. The Ioffe-Regel parameter.
2. Temperature dependence of resistance for metals. Electron-phonon contribution to resistance. Residual resistance. The Mattissen rule.
3. Thin films. Surface Resistance, RRR.
4. Superconductivity. Critical temperature. Critical current.
5. What is the difference between static and differential resistances in the normal state?
6. What is the difference between static and differential resistances in the superconducting state?