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PRECISE TEMPERATURE-CONTROLLED COMPLEX OF CRYOGENIC APPARATUS FOR STUDYING CURRENT-VOLTAGE CHARACTERISTICS OF TUNNEL CONTACTS OF SUPERCONDUCTING MATERIALS

Introduction. *The study of current-voltage characteristics (CVC) and their derivatives of tunnel contacts and hybrid heterostructures based on superconducting and ferromagnetic materials under influence microwave radiations and magnet fields in the wide range of temperatures is an actual task for development of element base of spintronics, superconducting electronics (in particular, for superconductive and quantum computers) and ultrasensitive sensors.*

Problem Statement. *One of the modern informative physical methods for studying the properties of tunnel contacts and hybrid heterostructures based on superconducting and ferromagnetic materials is the study of CVC and their derivatives in the low temperature region (mainly liquid helium range) in a magnetic field. This includes the study of magnetoresistance, Hall effect, quantum Hall effect, in particular under the action of spin injection. Today, there is no precision complex of temperature-controlled cryogenic equipment for the study of CVC of tunnel contacts of superconducting materials, which could fully meet the needs of studying the parameters of superconducting materials.*

Purpose. *Development of design and manufacture of precision temperature-controlled complex of cryogenic equipment for research of CVC of tunnel contacts of superconducting materials.*

Results. *A complex of precision temperature-controlled cryogenic system (temperature range 2.0—300 K) has been fabricated for the study of CVC of tunnel contacts of superconducting materials. The complex is based on a liquid-flow helium cryostat with built-in superconducting solenoid (SCS) (magnetic field range 0—2.9 T) and a specialized manipulator for changing the direction of the magnetic field, with a temperature regulator, with a programmable SCS power supply, automated measuring unit of CVC and software to it.*

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Conclusions. *The characteristics of the created cryocomplex are not inferior to the parameters of the best western analogues, and in terms of cost-effectiveness of cryoagent and service use they exceed them.*

Keywords: current-voltage characteristics, tunnel contacts, superconducting heterostructures, and temperature-controlled helium cryosystem.

To create a domestic complex of thermoregulated cryogenic equipment for studying the current-voltage characteristics (CVC) of tunnel contacts of superconducting materials with changeable orientation of the sample with respect to a magnetic field vector and simultaneous supply of an electric field and an optical effect factor to the sample is an urgent problem, because today there are no cryosystems with such a service in the world.

The leading manufacturers of cryogenic equipment with built-in superconducting solenoids (SCS) are *Oxford Instruments plc* (UK), *Cryo Industries of America Inc* (USA), *ABBES Instruments* (USA), *JANIS* Research Company, Inc. (USA), *CryoVac* (Germany), *Cryomagnetics corp* (USA), and *RTI Company* (Russia).

In Ukraine, similar products have been designed by Galkin Donetsk Institute of Physics and Technology of the NAS of Ukraine, Institute of Physics of the NAS of Ukraine (Kyiv), and Verkin Physical and Technical Institute of Low Temperatures of the NAS of Ukraine (Kharkiv). However, the cryostats from these manufacturers do not have such additional options as changeable orientation of the sample with respect to a magnetic field vector and simultaneous supply of strong electric fields to the sample. In addition, in these systems the scanning of magnetic field according to a given program is provided by cryostats equipped with controlled current sources from other manufacturers, which causes inconvenience while installing and operating such cryostats, as each manufacturer commissions and starts up on site only its device and is responsible only for its part of works.

For the time being, there has been no complex of thermoregulated cryogenic equipment for studying the volt-ampere characteristics of tunnel contacts of superconducting materials, which fully meets the needs of studying the parameters of superconducting materials.

Therefore, the creation of a complex of thermoregulated cryogenic equipment for studying the CVC of tunnel contacts of superconducting materials is an urgent task.

The purpose of this research is to develop a single computerized complex of thermoregulated cryogenic equipment, which combines a thermoregulated cryostat with a built-in superconducting solenoid with a field of 0 — 2.9 T, automated temperature controller, controlled current source for programmable scanning of the magnetic field, automated rotation of the sample relative to a magnetic field vector and the simultaneous supply of an electric field and an optical effect factor to the test sample with integrated control modes of operation of all these components from the central computer.

The Institute of Physics of the NAS of Ukraine has some experience in creating cryosystems for magneto-physical research and accessories to them [1—9]. Based on this experience, a complex of thermoregulated cryogenic equipment has been developed and manufactured for studies of volt-ampere characteristics of tunnel contacts of superconducting materials based on a cryostat with a specialized manipulator (which design is described below), a temperature controller, and a programmable power supply.

The scheme of the cryostat is shown in Fig. 1. Inside the collapsible housing *1* contains a helium tank *2* surrounded by a copper screen *3* cooled by liquid nitrogen poured into the nitrogen tank *4*.

The helium and nitrogen tanks are suspended from the lid *5* on thin-walled tubes made of a material with a low thermal conductivity. The helium tank suspension tubes *6, 7, 8* are used: a) to fix level detector *9* (tube *6*); b) to fix the needle valve

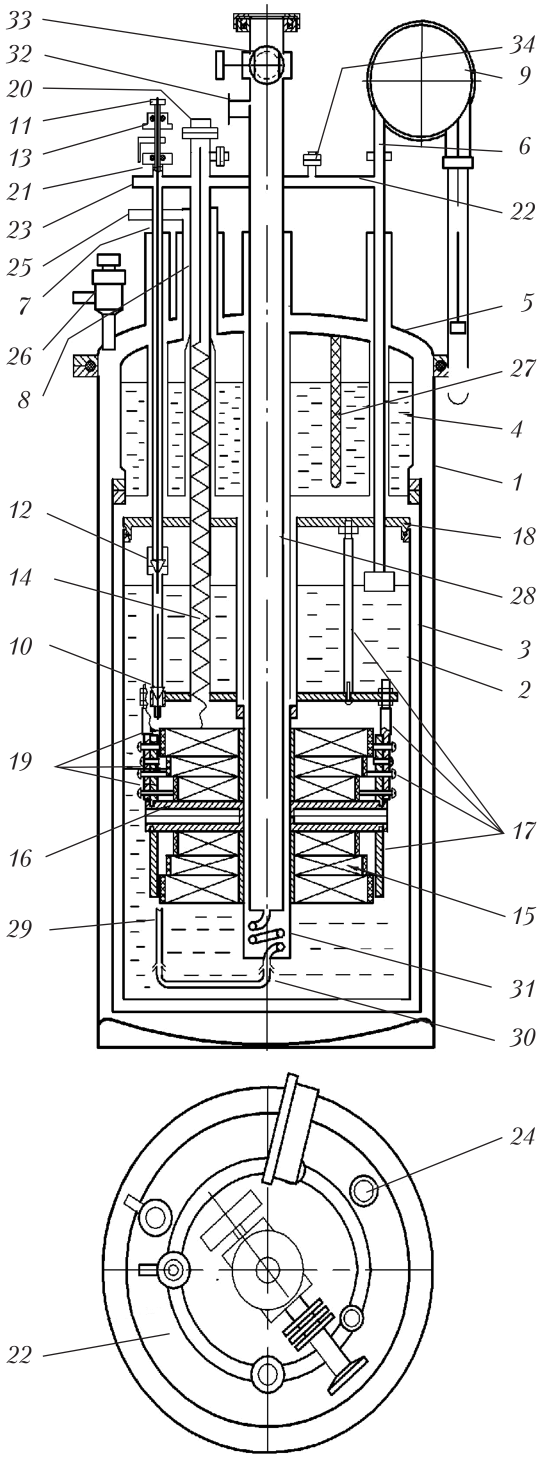

Fig. 1. Cryostat scheme

10 for liquid helium supply, which is controlled by the handle *11*, and the needle valve *12* for gaseous helium supply, which is controlled by handle *13* or to load liquid helium with removed needle valves *10* and *12* (tube 7); c) to fix the power current collector *14* from the SCS (tube 8).

The superconducting solenoid *15* is made in the form of two Helmholtz coils. Each of them consists of three removable sections on which the superconducting wire SKNT-0.33 is wound. The inner sections are fixed on the horizontal axis of the solenoid on the nozzles *16*. The middle and outer sections are fixed on the inner ones. All elements of section *15* through the system of suspensions *17* are attached to the lid *18* of the helium tank, which is sealed to the helium tank *2* with indium gaskets that ensure the necessary tightness. The power current collector *14* is made as a braid of copper wires.

The conductors from each coil of solenoid *15* are soldered through the switching boards *19* to the contacts of the connector *20*. The potential conductors of the solenoid are led to the connector *21*.

In the upper part, the suspension tubes of helium tank *2* are connected to each other by collector 22 for the removal of evaporating helium into the main through fitting *23*.

The suspension tube of the nitrogen tank *24* is used to load liquid nitrogen. Nitrogen vapor is discharged through the outer part of tube *8*, cooling the braid of the power collector *14* and removed through fitting *25*.

The vacuum cavity of the cryostat is pumped by a forevacuum pump through vacuum valve *26*. A high vacuum is created by cryopump *27*. In the center of the cryostat housing, there is shaft (loading pipe) *28*. Liquid and gaseous helium is fed to shaft *28* through needle valves *10* and *12,* using tubes *29*, *30,* and coiled pipe *31*. Tube *30* is removable. Helium gas is released through shaft *28* and fitting *32*.

Ball valve *33* is attached to the top of the shaft. It is used to lock the samples when they are replaced, covers the passage channel of the shaft, which allows replacing samples without heating the cryostat. To prevent the cryostat destruction, when the pressure in the helium volume and in the shaft increases, safety valves *34* are installed, the rupture discs of which are calibrated to an operating pressure of 5—7 × 10^4 Pa.

SPS specifications

The SCS constant is a ratio of magnetic field induction B (T) in the center of SCS to current I (A) measured at ambient temperature, at currents in the coil ± I (A) and is equal to 0.15 (T/A). The value of solenoid constant at a helium temperature (4.2 K) is within the limits of measurement error. The SCS constant is measured using *InSb* Hall sensor. Magnetic field induction is determined by the formula:

$$B = 0.15 \times I.$$

The limiting value of supply current at a temperature of 4.2 K is equal to 19.5 A and is the limiting value of the field in the center of the SCS 2.9 T. The limiting current of 19.5 A is supplied to the SCS for 25 min. A power supply module developed at the Institute of Physics of the NAS of Ukraine is used to power the SCS.

The specific feature of configuration of the cryostat-manipulator system is that the thermostat chamber is located on the manipulator.

The operation of the manipulator (Fig. 2) is illustrated by drawings (*a*) functional diagram of device for controlling the stepper motor of the insert; (*b*) vertical section of the insert; (*c*) side view of the insert; (*d*) A view of the insert; (*e*) B—B cross section of the insert.

The functional diagram of the device (Fig. 2, *a*) for controlling the stepper motor consists of manipulator *1* with stepper motor *2*, computer *3* that controls the stepper motor according to a given program through controller *4*, and power supply source *5*.

Manipulator *1* (Fig. 2, *b*) contains a mechanism for rotating substrate *6* with sample *7* around its vertical axis. The mechanism consists of central tube *8*, inside which tube *9* is placed. At the top, tube *9* is rigidly fixed on gear *10* and rotates with it in sleeve *11* rigidly fixed at the top of switch box *12* connected to the upper part of the central tube through sleeve *13*. At the bottom tube *9* is fixed so that it can rotate, in case *14*. From above, tube *9* through gear *10* and gear *15* is kinematically connected to stepper motor *2*, while from below, it is rigidly connected through holder *16* to substrate 6 with sample *7*. Thus, stepper motor *2* rotates substrate *6* with sample *7* around the vertical axis of the insert.

On the upper part of box *12*, platform *17* is fixed. At the platform there are mounted stepper motor 2 with gear *15*, tooth gear *10*, and microswitch *18* (Fig. 2, *c, d*) that determines the initial position of the rotation mechanism of substrate *6* with sample *7*, microswitch 19 that determines the final position of the rotation mechanism, and bracket *20*. At the bottom of tooth gear *10,* there is fixed sleeve *21*, by means of which microswitches 18 and 19 are triggered while the tooth gear is rotating. On the bracket 20, at the top, there is mounted sleeve *22* with tube *23* soldered in it. Sleeve *24* with tube *9* soldered in it rotates around sleeve *22* with tube *23*. On the underside, immovable tube *23* ends above the upper end of sample *7* and is designed for mounting light conductor *25*. On substrate *6*, near sample *7* there are switchboard *26* and spring-loaded clamps *27* and *28* for fixing sample *7* on substrate *6*. In the lower part of the substrate there is coiled electric heater *29*. On case *14,* there are fixed switchboards *30, 31* and *32*, temperature sensor *33* (Fig. 2, *e*) and coiled electric heater *34*. In addition, in the case, there are fixed tubes for supplying cables and conductors extending from the switchboards through case *14* and tube *8* to the connectors on switch box *12*. Tube *35* is designed to lead ultrahigh frequency cable *36* from switchboard *30* to connector *37* on box *12*. Tube 38 is designed to lead a high frequency cable *39* from sensor *40* of the capacitive helium level indicator through switchboard *31* to connector *41* on box 12. Tube *42* is designed to lead conductors from temperature sensor *33* through switchboard *32*, as well as from

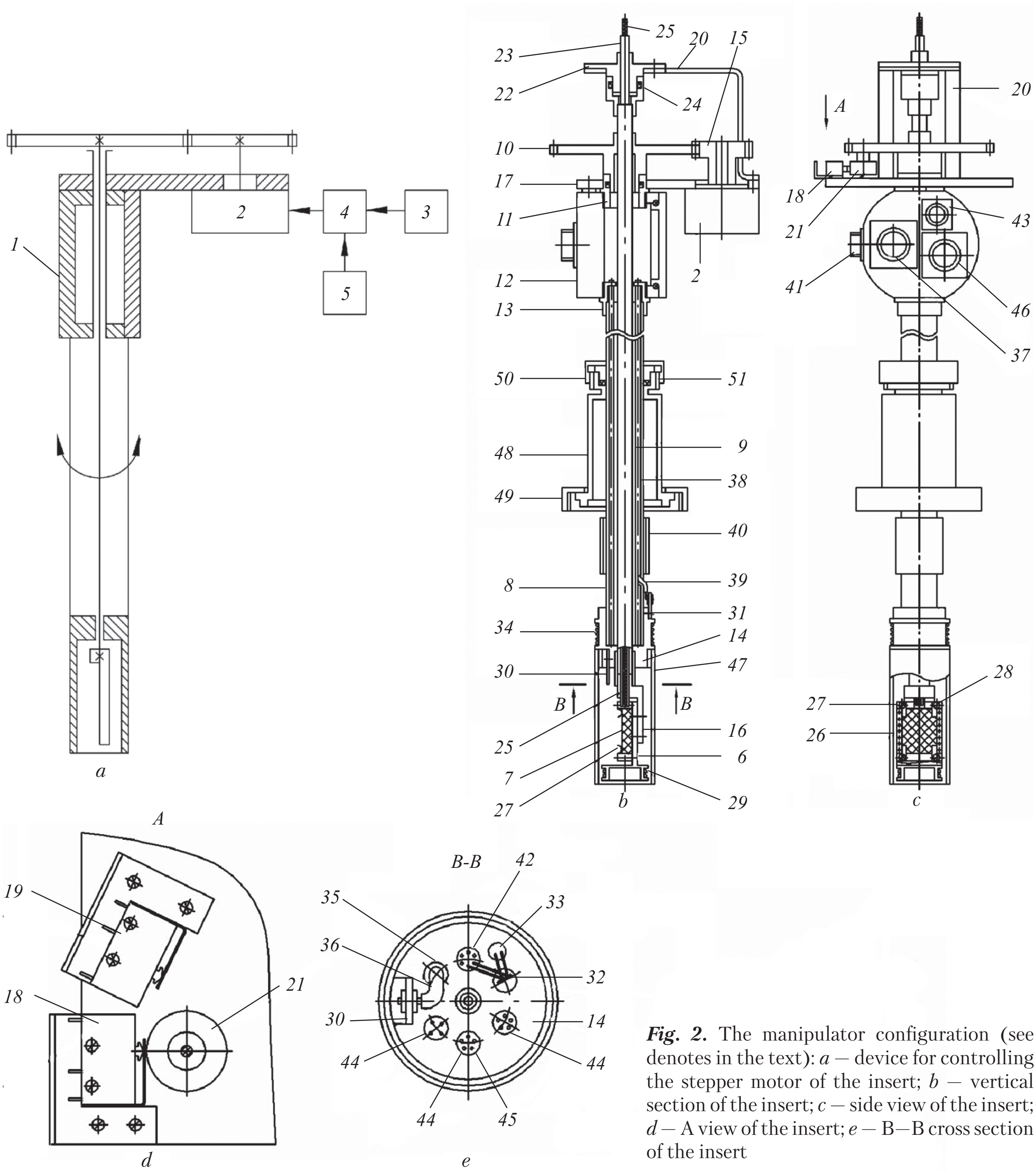

Fig. 2. The manipulator configuration (see denotes in the text): *a* — device for controlling the stepper motor of the insert; *b* — vertical section of the insert; *c* — side view of the insert; *d* — A view of the insert; *e* — B—B cross section of the insert

electric heaters *29* and *34* to connector *43* on box *12*. Tube *44* is designed to lead conductors *45* extending from sample *7* through switching board *26* to connector *46* on box *12*. In the lower part of the case *14*, there is mounted copper screen *47* to reduce a temperature gradient along the sample. The insert has extension *48* for fixing it to the upper branch of the cryostat with the help of cap

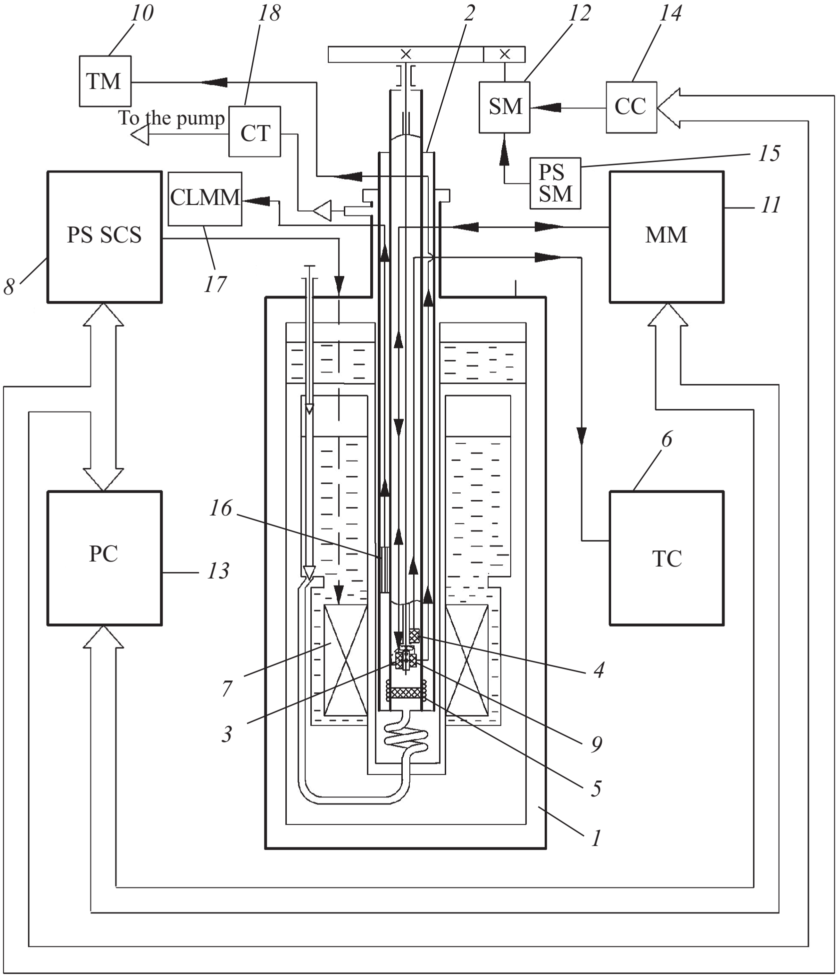

Fig. 3. Functional and structural scheme of the cryocomplex

nut *49*. In the upper part, the extension has stuffing box *50* to enable moving central tube *8* up or down and rotating it around the vertical axis with subsequent fixing of the tube by cap nut *51*.

The insert works as follows: sample *7* is fixed on substrate *6* by clamps *27* and *28* (Fig. 2, *b, c*). Stepper motor *2* through gear *15* and tooth gear *10* rotates tube *9* together with holder *16* and substrate *6* with sample *7*. The angle of sample rotation around the vertical axis of the insert depends on the number of steps of motor *2* set by computer *3* through the controller (Fig 2, *a*) and on the gear ratio of gear *15* and tooth gear *10* (i = 2/9). Given that each step the stepper motor rotates the shaft for an angle of 0.9°, and the gear ratio between gear *15* and tooth gear 10 (i = 2/9), one can determine the angle of rotation of substrate *6* with sample *7* per one motor step: $0.9° \times 2/9 = 0.2°$.

The angle of sample rotation X can be set by the number of steps N of the computer-controlled motor:

$$N = X/0.2.$$

For instance, to rotate the sample for angle $X = 180°$ the required number of steps N of the motor is:

$$N = 180°/0.2° = 900 \text{ steps.}$$

The maximum rotation angle depends on the angular position of microswitch *19* and is equal to 320°.

The functional and structural scheme of the cryocomplex is shown in Fig. 3.

The cryocomplex consists of:

a) the circuit of thermoregulation and temperature stabilization, which consists of cryostat *1*, manipulator *2* with test sample *3*, temperature sensor DTS-100 *4*, electric heater *5*, temperature controller (TC) *6*, and personal computer (PC) *13*;

b) the circuit of regulation and stabilization of the magnetic field, which consists of cryostat *1* with built-in superconducting solenoid *7*, power supply (PS SCS) *8*, manipulator *2* with *InSb* Hall sensor *9*, teslameter (TM) *10*, and PC *13*;

c) the module for automatic control of the sample slope angle, which consists of manipulator *2* rotating the rod together with the holder and sample *3* around the vertical axis with the help of stepper motor (SM) *12* that is connected by mans of electrical and information networks to PC *13* via process controller (CC) *14*, and power supply of the stepper motor (PS SM) *15*;

d) the measuring system for electrophysical measurements, which consists of measuring module (MM) *11* connected to sample *3* on manipulator *2* by the electrical network and to PC *13* by the information network;

e) the capacitive level meter of liquid helium in the cryostat shaft, which consists of a capacitive sensor (CS) *16* and capacitive level meter module (CLMM) *17*;

f) the optical channel in the manipulator, which consists of tube *23* in which light guide *25* is installed to supply the optical effect factor from the outer part of the manipulator to the sample in its lower part;

g) the channel for supplying electromagnetic radiation of ultrahigh frequency (UHF) to the sample in the manipulator, which consists of tube *35* through which UHF cable *36* comes from connector *37* on switch box *12* in the upper part of the manipulator to switchboard *30* in its lower part (Fig. 2, *b, e*).

Since the operation and principle of operation of such components of the complex as the temperature regulator, SCS power supply, etc. have been already described in detail in [7—9], below we explain in detail only the CVC measuring module developed by the authors, which consists of DC source and software.

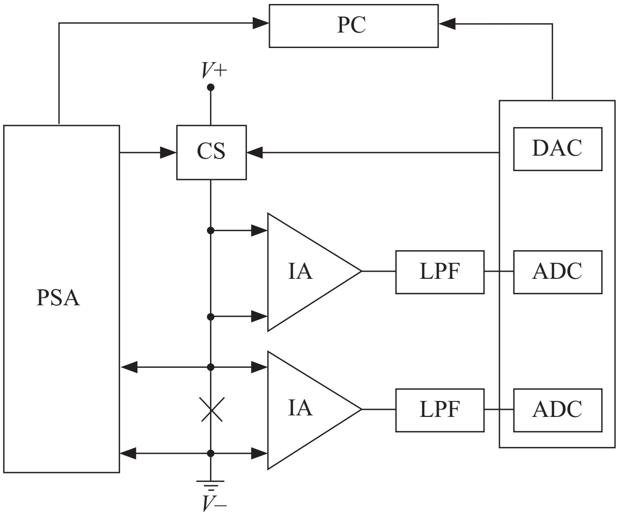

Fig. 4. Block diagram of the system for simultaneous measurement of direct and differential CVC: PC — personal computer; PSA — phase-sensitive amplifier; CS — current source; *R* — shunt resistance; *X* — sample; *V*+, *V*– — voltage sources; IA — instrumentation amplifier; LPF — low pass filter; ADC — analog-to-digital converter; DAC — digital-to-analog converter

Fig. 4 shows a block diagram of measuring equipment that allows simultaneous measurements of direct and differential volt-ampere characteristics of the samples [10]. The current in the circuit is set by a current source (CS) powered by a 24V battery (U +/—). This power supply significantly reduces noise and enables galvanic separation of the sample. The CS receives a DC voltage from a digital-to-analog converter (DAC) and an alternating sinusoidal reference voltage from a phase-sensitive amplifier (PSA). These voltages are added and converted into current with some proportionality factor according to the selected current change range. The PS has four ranges of current change — ± 20; ± 5; ± 1.25; and ± 0.3 mA. The range is selected by the switch. Information about the selected range is sent to the personal computer in the form of a digital code. As a result, the PS generates a sinusoidal current

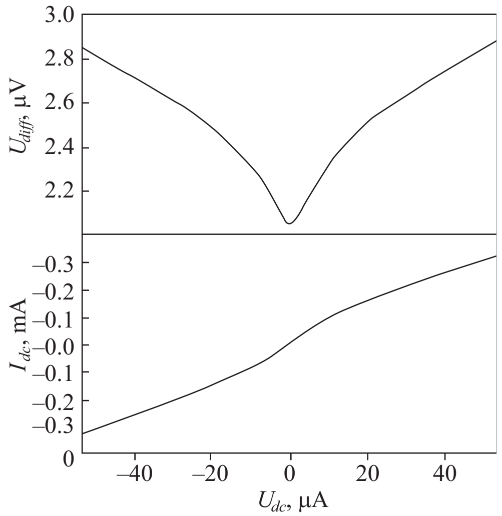

Fig. 5. Differential and direct CVC of hybrid contact MoRe-Si (W) -MoRe

of amplitude I_{ac} with a DC bias I_{dc}. The total current through the sample I_x is determined by the formula $I_x = I_{dc} + I_{ac}\sin(\omega t)$, where ω is frequency of the reference voltage from the PSA. For minimal influence of the variable component, the inequality $I_{dc} >> I_{ac}$ is fulfilled, except for the case of $I_{dc} \approx 0$. Each current range corresponds to its shunt resistor, the voltage of which is proportional to the current through the sample. The voltage from the shunt resistor passes to the instrumentation amplifier (IA) and then, through the low-pass filter (LPF), to the analog-to-digital converter (ADC). This is the way how the channel for measuring the direct current component I_{dc} is realized. The DC voltage component U_{dc} is measured by a similar channel. LPF is used in both channels to eliminate the influence of the AC voltage and current component on the DC component. Data from the two ADC channels are sent to the personal computer (PC).

The voltage from the sample also comes to the LPF that measures a variable component that is proportional to the differential resistance ($U_{ac} \sim R_{dif}$) of the sample. Thus, the values of DC and AC voltage components, as well as the current values are sent to the PC. The measurements are controlled by the program that changes the current value with a certain step and measures the current and voltage at each point. The minimum current step depends on the range and is equal to 300 nA for the used hexadecimal DAC in the range ± 20 mA and to 4.6 pA in the range ± 0.3 mA. It should be noted that the minimum step is much smaller than the noise in real measuring contacts. The

Specifications of the Cryosystem

Parameter	Value
Temperature adjustment range, K	2.0÷80÷300
Cryoagents	Liquid helium, nitrogen
Liquid helium consumption:	
For cooling the cryostat with SCS, l	At most 25
For keeping temperature at 4,2 K, l/h	At most 0.25
For keeping temperature at 1.6 K, l/h	At most 0.7
Nonstop operation at 4,2 K without refilling with cryoagents, h, at least	5
Volume of liquid nitrogen tank, cm^3	2800
Volume of liquid helium tank, cm^3	5500
Diameter of cryostat load channel, mm	19
Weight:	
cryostat, kg	22
manipulator no.1, kg	2
Electric heater mounted on the manipulator	
resistance, ohm	100
material	Nichrome, Ø 0.12; L = 1000 mm
voltage supplied to the electric heater mounted on the manipulator, V	0—40
Operating pressure of collapsible membranes of protective valves, Pa	5—7 × 10^4
The solenoid system:	
magnetic field induction in the center of SCS, T	2.9
field non-uniformity, %	1—1.5
SCS constant, T/A	0.15
Limit current, A	19.5
The system for measuring direct and differential CVC	
sensitivity of the direct channel, μV	0.3
sensitivity of the differential channel, μV	0.05
number of current ranges	4
minimum current step, pA	4.6
bit size of DAC and ADC channels	16
maximum speed of ADC measurements, measurements / s	10^6

number of measurements on the two ADC channels reaches 10^6 / s. Such a high speed allows additional filtering by calculating the average value of current and voltage during measurements at one point. The amplitude of the variable component is measured in parallel.

The program collects data for one cycle of current scanning, builds dependencies in any combination of three values of I_{dc}, U_{dc}, and R_{dif}, allows comparing up to five measured curves with each other and saves data as text files containing three columns of fixed numbers for further processing.

Fig. 5 shows the direct and differential CVC of hybrid three-layer contact measured by the designed module (Fig. 4). The contact is made of two thin films (~ 100 nm) of a superconducting *MoRe* alloy, which are separated by a layer of silicon containing tungsten nanoinclusions with a particle size of several tens nanometers. The measurements are carried out at a temperature of 4.2 K, the temperature value is recorded by a diode temperature sensor *DTS-100*. The study is conducted in the Earth's magnetic field that is considered quite small, so it does not cause a noticeable change in the CVC of the studied heterostructures.

Software

The software developed for the personal computer allows controlling the real time measurements of direct and differential volt-ampere characteristics using E-502 module. The computer communicates with the E-502 module via USB or LAN connection. In the program settings there are options to connect the selected ADC channels of the E-502 module to the channels of current, voltage, and differential voltage measurements, to select the number of averaging points, time of point measurement, to set the current range and scanning algorithms from smaller to larger or cyclic scanning, to set fixed value of current, and to measure the noise level for each channel. The program allows simultaneous viewing the four measured CVC, comparing them, zooming the graphs, and saving data to disc as a tabular text file. The used low-pass filters and averaging algorithms for several measured points make it possible to reduce the noise level to ± 0.2 μV per measured CVC. The obtained specifications of the cryosystem are given in the table below.

Hence, a unique innovative complex of precision thermoregulated cryogenic equipment has been developed. It is used for studying the CVC of tunnel contacts of superconducting materials and enables changing the sample orientation relative to a magnetic field vector with simultaneous supply of an electric field and an optical effect factor to the sample. This complex is as good as the best foreign counterparts in terms of capabilities and surpasses them in terms of cost effectiveness (cryoagent consumption and maintenance costs).

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ПРЕЦИЗІЙНИЙ ТЕРМОРЕГУЛЬОВАНИЙ КОМПЛЕКС КРІОГЕННОЇ АПАРАТУРИ ДЛЯ ДОСЛІДЖЕННЯ ВОЛЬТ-АМПЕРНИХ ХАРАКТЕРИСТИК ТУНЕЛЬНИХ КОНТАКТІВ НАДПРОВІДНИХ МАТЕРІАЛІВ

Вступ. Дослідження вольт-амперних характеристик (ВАХ) та їхніх похідних тунельних контактів та гібридних гетероструктур на базі надпровідних та феромагнітних матеріалів під дією надвисокочастотного (НВЧ) випромінювання та магнітних полів в широкому діапазоні температур є актуальним завданням для розроблення елементної бази спінтроніки, надпровідникової електроніки (зокрема для надпровідних і квантових комп'ютерів) та надчутливих сенсорів.

Проблематика. Одним із сучасних інформативних фізичних методів досліджень властивостей тунельних контактів та гібридних гетероструктур на базі надпровідних та феромагнітних матеріалів є дослідження ВАХ та їхніх похідних в області низьких температур (переважно діапазон рідкого гелію) в магнітному полі. Сюди входить дослідження магнітоопору, ефекту Холла, квантового ефекту Холла, зокрема й під дією спінової інжекції. На сьогодні не існує прецизійного комплексу терморегульованої кріогенної апаратури для досліджень ВАХ тунельних контактів надпровідних матеріалів, який би повністю міг задовольнити потреби вивчення параметрів надпровідних матеріалів.

Мета. Розроблення конструкції та виготовлення прецизійного терморегульованого комплексу кріогенної апаратури для досліджень ВАХ тунельних контактів надпровідних матеріалів.

Результати. Виготовлено комплекс прецизійної терморегульованої кріогенної системи (діапазон температур 2,0–300 К) для дослідження ВАХ тунельних контактів надпровідних матеріалів. Комплекс створено на базі гелієвого кріостата рідинно-проточного типу з вбудованим надпровідним соленоїдом (НПС) (діапазон магнітного поля 0–2,9 Тл) та спеціалізованим маніпулятором для зміни напрямку магнітного поля, з регулятором температури, з програмованим блоком живлення НПС, автоматизованим блоком вимірювання ВАХ та програмним забезпеченням до нього.

Висновки. Характеристики створеного кріокомплексу не поступаються параметрам кращих західних аналогів, а за показниками економічності використання кріоагенту та сервісу перевищують їх.

Ключові слова: вольт-амперні характеристики, тунельні контакти, надпровідні гетероструктури, терморегульована гелієва кріосистема.