AUTOMATED SYSTEM FOR INVESTIGATION OF RESISTANCE TEMPERATURE DEPENDENCE OF HIGH TEMPERATURE SUPERCONDUCTORS

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The requirements to an automated system for the temperature dependence investigation of resistance in high temperature superconductors (HTSC) were analyzed. The experimental problems within a real sample are shown. The constructed system and the calibration experiments with a superconducting sample of $YBa_2Cu_3O_{7-\delta}$ were described. The achieved measuring accuracy of temperature was 0.05 K and of voltage 1 μV.

Keywords: resistivity of superconductor, automated measurement

1. INTRODUCTION

Investigations of the temperature dependence of resistance – R(T) and the AC magnetic susceptibility are the most often used methods to diagnosticate superconducting materials (**SCM**). They are based on the two main properties of these materials –the lack of resistivity and the Meissner effect respectively. Both methods are applicable to thin films and poly- and monocrystal samples [1-3]. They can give a lot of information for SCM and for other solid state materials too. For example, the investigation of resistivity in high temperature superconductors (**HTSC**) in normal state gives possibility to search the confirmation of the last hypothesis about the Bose condensation above the critical temperature – T_c and for the pseudo-gap [4].

The R(T) dependence is linear above T_c in zero magnetic field for optimally doped cuprates. The S-shaped super-linear R(T) curve is found for underdoped samples. As the transport properties are closely connected with the scattering mechanisms of the carriers it is obvious the importance of these investigations to find the reason of R(T) linearity.

Receiving of R(T) in a digital form make easier next processing of the experimental data and increases their certainty.

The purpose of this work was to construct an automated system for investigation of R(T) dependence in HTSC. The main problem for the system is the necessity to measure very low values of R in large temperature range. Therefore the requirements for sensitivity and noise immunity are very high.

2. SET-UP AND PARAMETERS OF THE SYSTEM

Scheme of system set-up for investigation of R(T) dependence is shown in Fig.1. The investigated sample together with a thermistor (Pt_{100}) and a heater are situated in a specialized chamber immersed in cryostat with liquid N_2. This construction allows to investigate HTSC with T_c > 77 K and with T_c = 65-77 K using pumping of the liquid N_2 vapour in the cryostat.

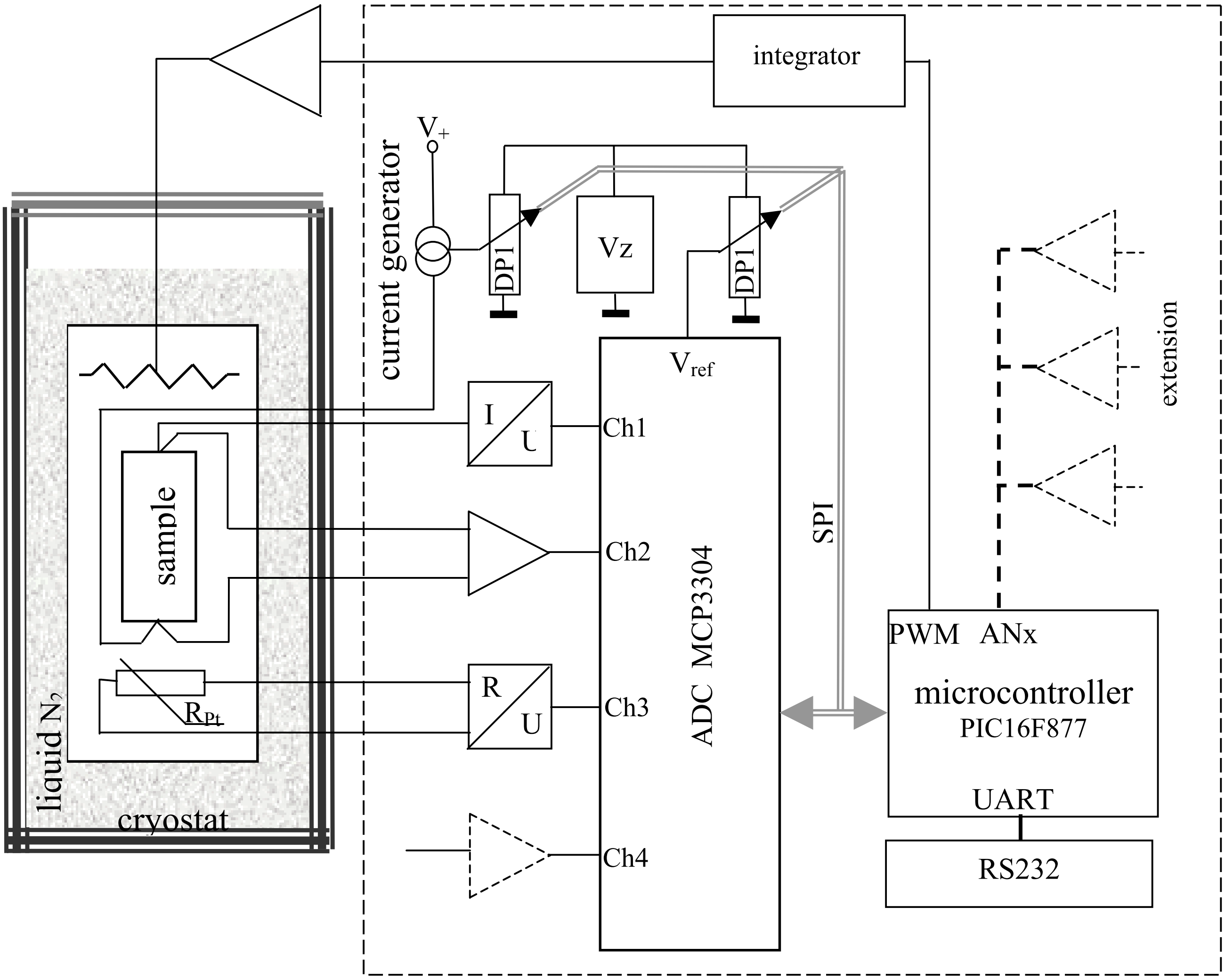

Fig.1.

The 4 probe contact method is used to measure the resistance of the investigated sample. The current through the sample comes from the generator regulated by a digital potentiometer. The generator is commanded by a microcontroller PIC16F877 through a serial peripherial interface. Two ranges with the maximal current of 6 mA and 130 mA are realized. An independent channel is used to measure the real current through the sample.

The voltage group of measuring current through the sample is amplified 1000 times by the high stable amplifier before the analog-to-digital converter (**ADC**). Operational amplifiers (**OA**) AD8574 with automated support of zero (with a voltage asymmetry less than 1 μV) and a temperature drift less than 0,005 μV/°K are used.

Thermistor Pt_{100} is used for temperature measurements. The 3 probe contact method is used but the possibility to change it to 4 probe is considered.

13 bits 4 channels ADC with a differential inputs MCP 3304 is used. It keeps its parameters 0.4-5.0 V in the whole range of the reference voltage. The reference source is based on a programmable precision references LM431. Its temperature dependence is less than 50 ppm/K. Regulated voltage divider, based on the digital potentiometer MCP41010 buffered with OA with automated support of zero AD 8574, is connected after the potentiometric connection between the digital potentiometer and the used OA guarantee that the temperature stability of the OA is commensurable with the instability of the programmable references LM431. As a result the range of the ADC could be changed in the large range.

There are 4 analog input channels yet – one to the MCP 3304 and 3 to DAC built in the microcontroller. They are foreseen to the future development of the system.

Connection to the personal computer is realized through the galvanic modified interface RS 232.

Calibration of the temperature channel is carried out using a set of model resistors ММЭС-Р4831. The temperature range is 25-190 K. The system sensitivity is 0.023 K/div and the integral non-linearity is better than 0.1% in the whole range.

Calibration of the current generator and the measuring channel is realized using digital multimeter with 4000 discretes and automated change of the range. The common integral non-linearity for the both ranges (6 mA and 130 mA) is better than 0.1%.

3. Experimental Problems

Parasite signal caused by thermoelectric power (TEP) due to the temperature gradient in the sample chamber was fixed through the experiments. Sometimes its value was higher than the main signal. It can also change its direction when the

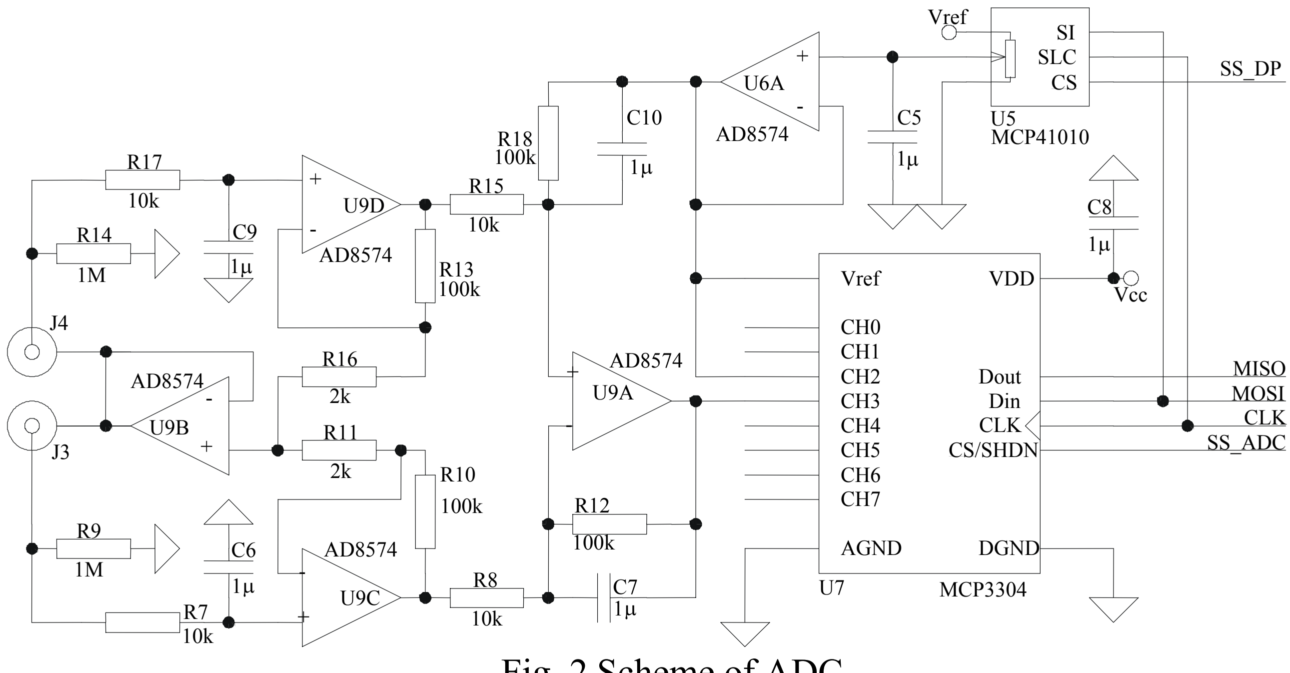

Fig. 2.Scheme of ADC

experimental conditions are changed (e.g. sample change). Because of that, the scheme of preamplifier and its connection with ADC were changed to ensure better scale matching when the sign of measuring voltage was changed. Second and third channels of ADC are used in differential mode (see fig.2) where the shift of the instrumental amplifier is connected to the reference voltage of ADC. So a very good matching of zero at the instrumental amplifier input with the zero of the converter characteristic of ADC was obtained.

When the value of the reference voltage is chosen to be 2.5 mV the value for the range of the measuring channel is ±2.5 mV and the value of 1 discrete is 0.061 μV. At the upper limit value of 400 mV for the reference voltage the range becomes ±400 μV and the discreteness is 0.1 μV.

4. Operating Modes

The system can work in 2 operating modes:

1. Measurement of a voltage drop through the sample – U at I = const to determine T_c of the HTSC sample.
2. Measurement of a voltage drop through the sample – U at T = const and current step-up to determine I_c of the HTSC sample.

The resistance of HTSC in the first mode has been determined on the base of 2 measurements – at minimal current through the sample and at preliminary specified current (1-5 mA usually) at which the experiment is carried out. The real value of resistivity is a ratio of the difference between the measured voltage to the difference between currents through it. In this way the experimental error due to the TEP is eliminated. The present value of temperature is determined at every measurement and saves with other received data.

In the second mode the current through the sample is changed smoothly from zero to preliminary chosen value (above 100 mA usually) and the voltage through it is registered. The purpose is to determine the current value which caused 10 μV change of voltage through the sample from the beginning current point. This is the criteria for determination of I_c [5]. The measurement is short time-delay and the sample temperature changes negligible (by Joule heat due to measuring current). Parasitic TEP doesn't change its value therefore the measurement can be carried out without any correction of its value. The sample temperature is registered through the experiments and its value saves together with the values of the voltage drop and measuring current through the sample.

5. Experimental Results

Polycrystal sample of $YBa_2Cu_3O_{7-\delta}$ was investigated using two modes described above. The experiment for I_c = const was carried out in two steps: 1) At first the sample was cooled slowly to 77 K and 2) After that the heat transfer gas in the sample chamber was pumped out and the sample was lifted on a suitable level above the liquid nitrogen to be heated under control. The results of these 2 steps are shown in fig. 3. The superconducting transition is well seen in both figures. Transition temperature is $T_c = 91.5$ K that is in good agreement with our previous results from ac susceptibility measurements for this superconductor [6].

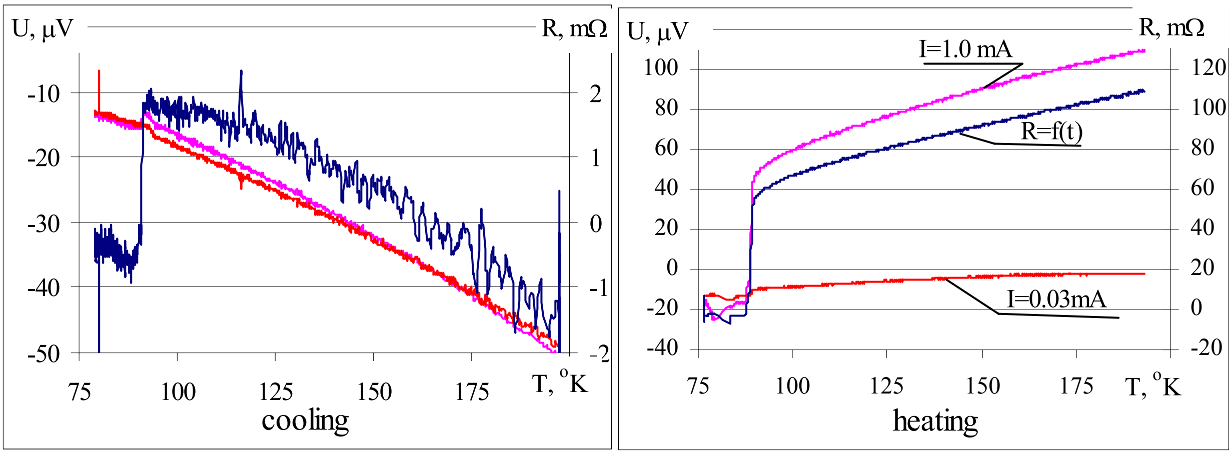

Fig.3. Voltage drop dependence from sample temperature

It is interesting that the noises are too larger when the sample is cooled. Therefore it is recommendable to carry out this investigation when the sample is heated. The difference in the values of U in both cases is a result of the different pressure of the heat transfer gas in the sample chamber. As said above it is pumped out when the sample is heated and the pressure becomes lower than through the cooling process.

The sample investigation with linearly increasing current at T = const was done for values up to 130 mA. The results are shown in fig.4. It is well seen that the resistance is almost constant which means that the sample is in superconducting state. The voltage drop increases almost linear from 2 to 12 µV and according to the criteria said above (see [5]) the critical current I_c = 130 mA.

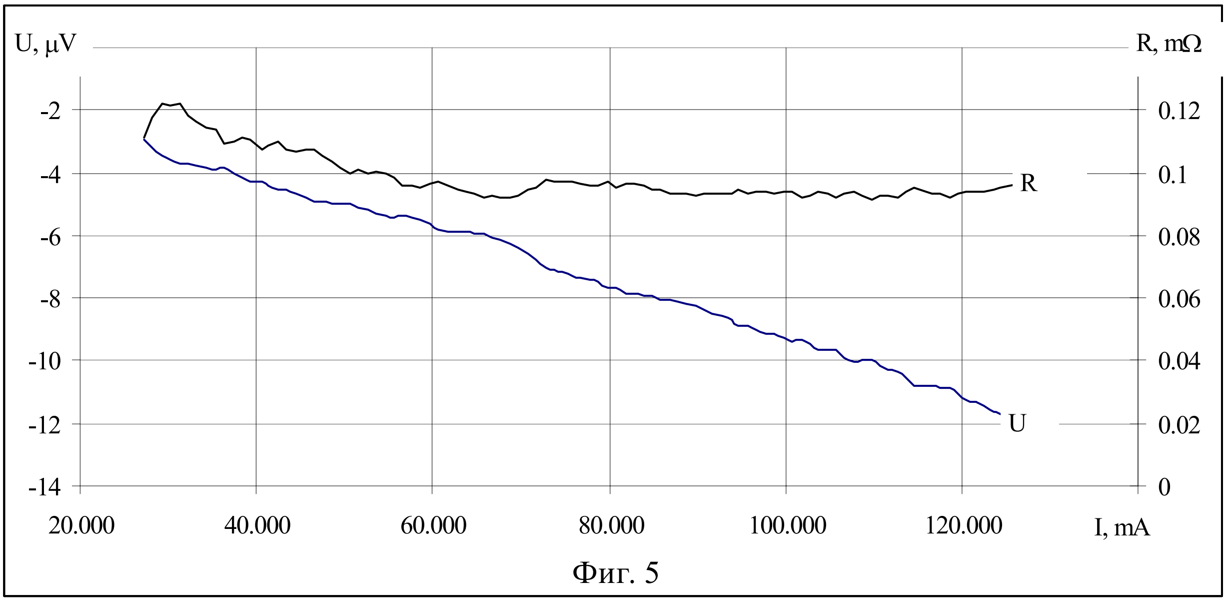

Fig.4. Voltage drop and sample resistivity dependence from current supply at T = 77.4 K

6. CONCLUSION

The designed system for investigation of HTSC gives the possibility to determine quickly and easily some of their most important parameters – T_c and I_c. It can be used to investigate the R(T) dependence of other solid state materials at 25-190 ^{o}K using a suitable cryostat. Therefore it will be very useful in the field of Cryogenics and some other branches of the Solid State Physics.

7. ACKNOWLEDGEMENTS

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