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Computer Controlled Thermostat for the Resistivity measurements of the $La_{1-x}Sr_xMnO_3$ thin films

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Introduction

Manganites like $La_{1-x}(Sr, Ca, Ba)_xMnO_3$ are of great interest for last few decades mainly since they exhibit a colossal magnetoresistance (CMR) effect. This has found many applications creating magnetic field sensors and magnetic memory [1]. However, temperature plays even bigger role in the change of the resistance than magnetic field [2]. Hence it is crucial to precisely stabilize the temperature during the measurements of the magnetoresistance (MR). Moreover it is necessary to adjust the temperature to the known values in order to study how it affects the resistivity [3], magnetoresistance and anisotropy [4].

There are many commercially available temperature controllers designed for the industrial or laboratory use of various functionality depending on the cost. Nevertheless in this work we have developed a temperature controlling system that satisfies the needs of setting and keeping the temperature in a small volume between the poles of an electromagnet. Moreover, the system consists mainly of the general purpose equipment which was and is used for the other purposes. In this way the costs were minimized while keeping the optimal performance.

Requirements

Magnetoresistance is defined as [5]

$$MR=\frac{\rho_H-\rho_0}{\rho_0}\times 100\%, \quad (1)$$

here ρ_0 is resistivity at the zero magnetic field and ρ_H is resistivity at a given magnetic field H. The origin of the magnetoresistance lies on the quantum mechanical phenomenon known as *double exchange* which is inherent in manganites. The electron transport through the oxygen ions is greatly enhanced when the Mn^{+3} and Mn^{+4} ions are ordered ferromagnetically, hence the resistivity is lower. In contrast, resistivity is higher when there is no order (paramagnetic state). Obviously, magnetization $\boldsymbol{M}$ is the order parameter which affects resistivity. On the other hand, temperature is a disordering parameter which acts in an opposite way. In general, magnetoresistance can be described using a Brillouin function $\mathcal{B}(H,T)$ [6].

Since the resistance of the sample is both dependent on the magnetic flux density B and temperature T, it is possible to compare its sensitivity to these quantities. Fig. 1 shows magnetic flux density and temperature which affect resistance in the opposite way so, that eventually it is kept constant. The important thing which should be noticed is the slope of this graph. At low temperatures (0 ÷ 15 °C) it is equal 0.085 T/°C (Tesla per degree) but at high temperatures (40 ÷ 50 °C) it almost reaches 1 T/°C.

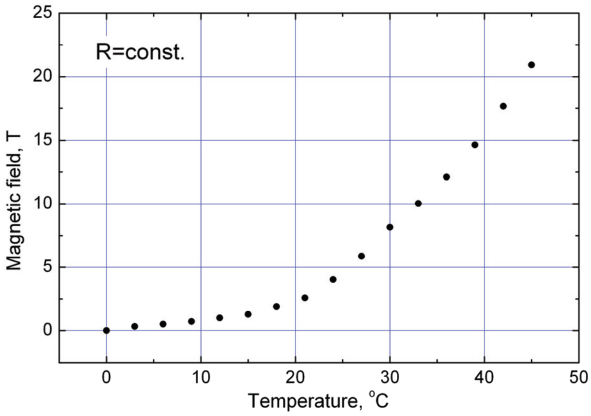

Fig. 1. Graph showing magnetic field corresponding to the same change of resistance as is due to the change of temperature

While measuring the magnetoresistance, changes of the resistance can be wrongly interpreted as they appear due to the magnetic field. The standard deviation of the magnetic field during the measurements is about 5 mT. However in general 1% error is allowed meaning that the magnetic field deviation at 2.5 T might be equal 25 mT. Considering the slope of the graph in Fig. 1, requirements for the temperature stability are lowest at the low temperature – $\Delta T_{max} = 0.06$ °C to make measurements with precision of the magnetic field 5 mT and highest requirements are for the high temperatures, where $\Delta T_{\min} = 0.025$ °C to make measurements with precision of the magnetic field 25 mT.

Measurement methodology

The most known algorithm for temperature stabilization is a PID controller. It was chosen to be used due to its simplicity and good performance [7]. The PID controller algorithm is used to control the power source to adjust heating/cooling taking into account the feedback signal from the temperature sensor. A general purpose laboratory power supply "TTI QL564P" was used as a regulated current source and a multimeter "Tektronix DMM4050" was used as a temperature meter. A Pt 1000 temperature sensor was used as a probe since it is compatible with the multimeter and is not sensitive to the magnetic field. The drawing in Fig. 2 shows the components of the system and how they are interconnected.

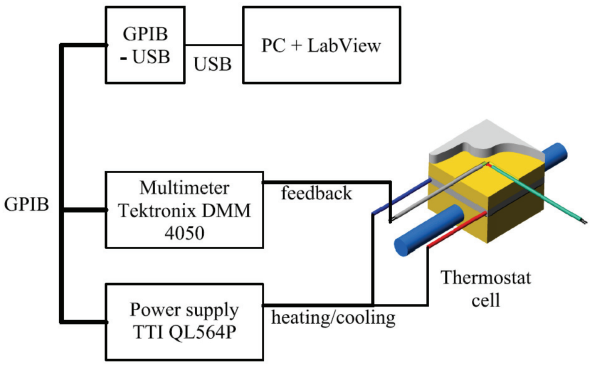

Fig. 2. Schematic diagram of the temperature stabilizing system

It is required that the temperature could be adjusted in a range of 0 ÷ 50 °C. It means that heating and cooling must be incorporated in the design. A simple way to do this is to use a Peltier element. A conventional Peltier element is a device which enables cooling one side and heating another when the current is passed. Reverse effect occurs when the current direction is reversed. A single Peltier device can create temperature difference of up to 70 °C. Keeping one side at a constant temperature, the other side can be heated above or cooled below the room temperature. An efficient way to keep the temperature of the one side constant is to use water flow. Air cooled system was also considered, but a heat sink and a fan would take too much space. Moreover, water cooling system is already present at the electromagnet; and is easy to connect to it.

A thermostat cell as shown in Fig. 3 was made. It consists of the brass block with a cavity (9) and water inlets (1, 2), a Peltier element (10), another brass plate on the other side (11) with the cavities for the temperature probe and the sample (4, 5) and the insulating material – styrofoam on the top. All parts were glued together and fixed at the experiment site – between the poles of the electromagnet. Dimensions of the thermostat cell were chosen to fit in the gap between the poles, which is about 31mm. The cell can be inserted between the poles in different orientations, depending on which direction of the magnetic field is needed. The temperature probe and the sample are very close to each other to ensure that their temperatures are equal.

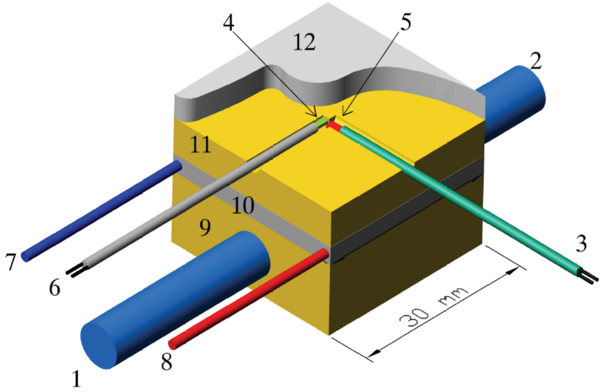

Fig. 3. A drawing of the thermostat cell. 1, 2 – water supply; 3- cable of the sample; 4 – temperature probe; 5 – sample; 6 – cable of the temperature probe; 7, 8 – Peltier power leads; 9 – brass chamber; 10 – Peltier element; 11 - brass plate; 12 – insulating material

Automation using LabView

LabView is a graphical programming environment which offers straight through communication with the hardware devices and a user friendly graphical interface [8]. The main function of the program is to ensure the temperature stabilization which consists of: communication with the power source and the multimeter, calculation of the current using the PID algorithm, applying different setting and options. Another purpose is visualization of the current temperature, so that user can decide when the temperature has stabilized.

The value called *error* ε which is the difference between the desired temperature and the current temperature is calculated in each cycle. It is a main variable in the PID algorithm. Moreover, time spent in each loop dt is calculated. These two variables and the *P*, *I*, *D* constants are used for the calculation of the current using a classical PID formula

$$I = P\varepsilon + I\int \varepsilon dt + D\frac{d\varepsilon}{dt}. \qquad (2)$$

The program consists of several parts that run in the closed loop, as depicted in Fig. 4. First, the user supplied parameters and constants are collected. User is able to change some basic settings, as: the set point temperature, GPIB addresses and models of the power supply and the meter, probe type (Pt1000 or Pt100), heating or cooling mode. More advanced settings are the *P*, *I* and *D* coefficients used in the PID algorithm, maximum current allowed, ramping speed. All these parameters are available at the "front panel" of the software (Fig. 5).

Then, the temperature value is read from the meter and it is used for the ramping procedure. Ramping is a gradual change of the set point value from the current temperature to the desired value. The purpose of the ramping is to minimize the first overshoot, which is a common problem in the PID algorithm. Ramping speed in °C/s is set separately for the increasing temperature and decreasing temperature because the cooling and heating speed is different. Finally, when the actual set point reached the desired value, it is kept constant.

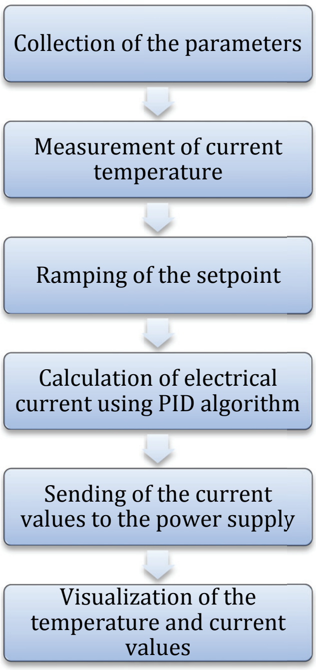

Fig. 4. Program algorithm flowchart

The cycle period dt was chosen to be much shorter than the thermal time constants of the thermostat, but long enough to average the temperature readings. Stability of the system is very sensitive to the noise in the feedback circuit due to the differential component. The temperature readings are averaged at the meter device during 1 second before they are sent to the PC. Thus, optimal stability is reached without increasing the time needed to reach the temperature set point.

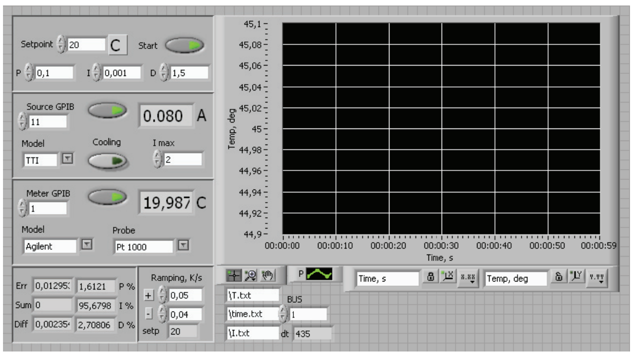

Fig. 5. The „front panel" of the controlling program written in the LabView programming environment. The graph window is used to visualize the change of the temperature over time

Communication with the external devices was accomplished by standard commands specified in the manufacturer manual. GPIB – USB adaptor was used to interconnect all the instruments and then connect to the PC via single USB cable.

Results and discussion

Temperature change from 15 to 30 °C is shown in Fig. 6. First part takes around 100 seconds to reach the desired temperature. Here the ramping procedure is used to gradually increase the set point. The result is that the temperature changes almost linearly. Then, the set point is kept constant. The first overshoot here is equal 2.4 °C. It is proportional to the change of the temperature. It is not relevant to avoid it, since it does not affect the overall stabilization time. Stabilization up to ±0.02 °C takes about 300 seconds if there are no significant perturbations, i.e. change of the sample. The time is shorter if the change in temperature is smaller. Natural airflow in the room does not affect the temperature inside the cell.

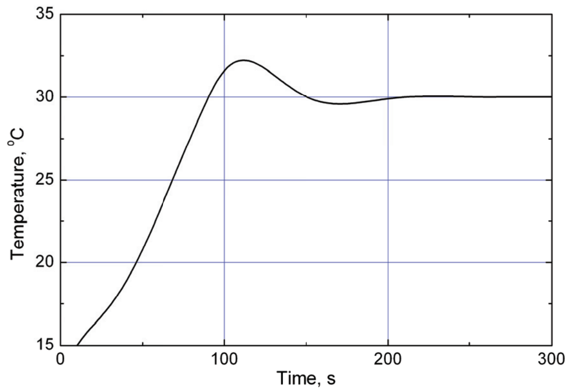

Fig. 6. Temperature change and stabilization graph. Initial temperature was 15 °C, temperature set was 30 °C

Fig. 7 shows manganite resistance dependence on magnetic field measured at four different temperatures. Noise due to the temperature drift is lower than the thickness of the lines. Therefore the curves look very smooth. Moreover, measurements of the same sample agree when repeated after some time.

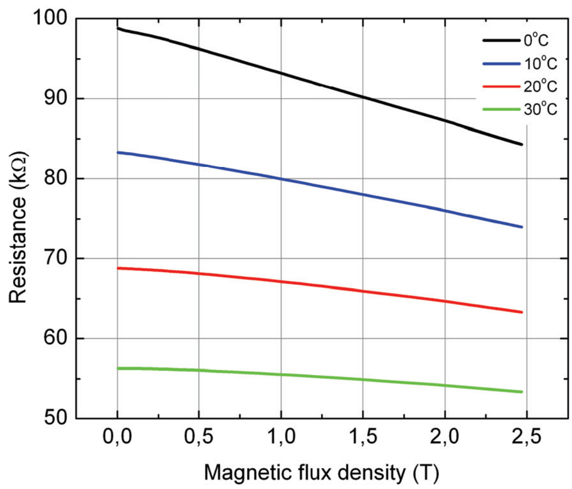

Fig. 7. Resistance dependence on magnetic field measured at several temperatures

In Fig. 8 the resistance dependence on temperature is shown. Resistance was measured in the range of temperatures of 0 ÷ 40 °C. Various preparation procedures were made between the measurements, such as annealing of the contacts at different temperatures, heating in different temperatures and humidity. Such procedures affect the resistivity of the sample, the resistance temperature coefficient and magnetoresistance. The change of the resistance of the order of less than 1% in the whole range of temperatures is observed.

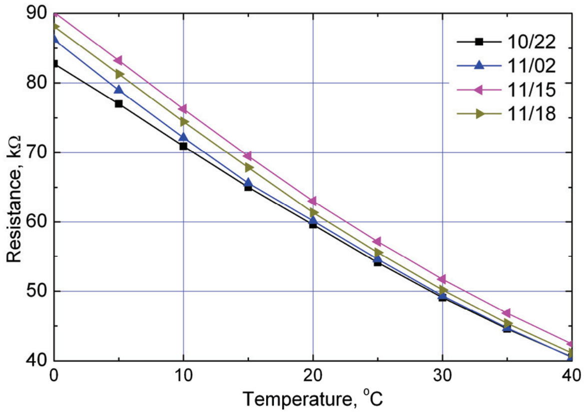

Fig. 8. Resistance dependence on the temperature measured using the temperature stabilizing system. Several measurements of the same sample were made after different preparation procedures

Acknowledgments

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T. Stankevic, V. Stankevic, D. Pavilonis, N. Zurauskiene, J. Novickij, S. Tolvaisiene. Computer Controlled Thermostat for the Resistivity measurements of the $La_{1-x}Sr_xMnO_3$ thin films // Electronics and Electrical Engineering. – Kaunas: Technologija, 2012. – No. 3(119). – P. 53–56.

The temperature stabilization system for the magnetoresistance measurements of the $La_{1-x}Sr_xMnO_3$ manganites is described in this paper. The thermostat cell with the Peltier heating/cooling element was manufactured specially to be placed between the poles of the electromagnet. The heat sink attached to the rear side of the Peltier element is cooled by the flowing tap water. Platinum film temperature probe was used for the temperature feedback signal. Universal multimeter “Tektronix DMM 4050” was used as a temperature meter and a regulated laboratory power supply “TTI QL 564P” was used to supply the current through the Peltier element. Both instruments were controlled by the computer software via the USB and GPIB interfaces. The software implementing a PID algorithm was written in the LabView graphical programming interface. The results show that the temperature of the sample can be changed in 2-3 minutes depending on the temperature step and is kept constant with precision of ±0.02 °C. Ill. 8, bibl. 8 (in English; abstracts in English and Lithuanian).

T. Stankevič, V. Stankevič, D. Pavilonis, N. Žurauskienė, J. Novickij, S. Tolvaišienė. Kompiuterinė temperatūros stabilizavimo sistema plonų sluoksnių $La_{1-x}Sr_xMnO_3$ manganitų varžai matuoti // Elektronika ir elektrotechnika. – Kaunas: Technologija, 2012. – Nr. 3(119). – P. 53–56.

Sistema susideda iš termostato kameros, kuri įtaisoma tarp elektromagneto polių. Kamera susideda iš Peltjė elemento, kuris gali kaitinti arba šaldyti bandymų objektą. Pratekantis vanduo palaiko pastovią Peltjė elemento kitos pusės temperatūrą. Grįžtamajam ryšiui naudojamas platininis temperatūros jutiklis prijungtas prie universalaus matuoklio „Tektronix DMM 4050“. Peltjė elementas maitinamas iš universalaus laboratorinio reguliuojamo srovės šaltinio TTI QL 564P. Abu prietaisai sujungti su kompiuteriu per GPIB sąsają ir per GPIB-USB sąsajų konverterį. Naudojant *LabView* programavimo aplinką buvo sukurta programa, naudojanti PID algoritmą bei valdanti prijungtus prietaisus. Programa užtikrina pasirinktos temperatūros stabilizavimą. Matavimų rezultatai parodė, kad norima temperatūra pasiekiama per kelias minutes priklausomai nuo temperatūrų skirtumo. Ji palaikoma su ±0,02 °C paklaida. Toks tikslumas yra pakankamas manganitų sluoksnų varžai matuoti magnetiniame lauke. Il. 8, bibl. 8 (anglų kalba; santraukos anglų ir lietuvių k.).