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THE APPARATUS FOR SEEBECK COEFFICIENT MEASUREMENT IN WIDE TEMPERATURE REGION

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The paper describes an apparatus for thermopower measurement. The apparatus allows to measure the Seebeck coefficient in the temperature region of 90 K - 1100 K on samples with resistivity up to 10^{10} Ω.

Measurement of the Seebeck coefficient (thermoelectric power) S gives information about the sign of charge carriers , the electron band structure and the transport mechanism in the sample. The Seebeck effect has also practical use, i.e. conversion of thermal energy to electrical energy. An apparatus was constructed for the Seebeck coefficient measurement on different materials (oxides, layered semiconductors, intercalates). The apparatus allows to measure the Seebeck coefficient in the temperature region of 90 K - 1100 K in two regimes:

1) automatic measurement of S on the samples with resistivity up to 10^5 Ω
2) semiautomatic measurement of S on the samples with resistivity up to 10^{10} Ω.

It is possible to measure samples of to 15 mm height and max. 8 mm diameter.

For the thermopower measurement on the high resistivity samples, the insulation resistance of the probe should be > 10^{13} Ω. Teflon is frequently used as high resistivity insulator material for the construction of sample probe. Upper temperature limits of such probe is, however, about 200 °C. For higher temperatures the ceramic insulator becomes necessary to separate the voltage contact. It brings difficulty with mechanical work-up.

The probe consists of the two brass blocks (Fig. 1). The brass blocks were used because of their good thermal conductivity and relatively high stability

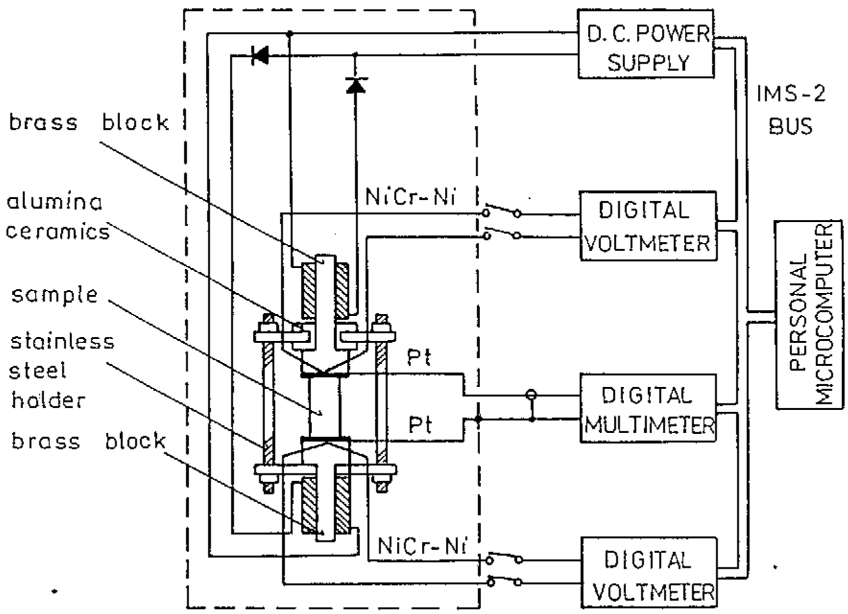

Fig. 1 Schematic diagram of the apparatus for measurements of the Seebeck coefficient

even at elevated temperatures. The sample is placed between these blocks gripped by a stainless steel holder. One of blocks is fixed elastically by a spring placed in the cool region of probe in order to compensate different temperature stretching of sample and stainless steel holder. The blocks are electrically insulated by alumina ceramics. The generated Seebeck voltage is detected by platinum electrodes. Platinum was used for its very low absolute thermopower. Literature data are frequently listed for platinum contacts. These electrodes are wired in through vacuum-tight glass bushings with insulating resistance ≥ 10^{14} Ω and then by coaxial cable. For the voltage measurement on the samples with low resistivity (R < 10^6 Ω) the digital multimeter M1T380 (Metra) is used. The instrument has input impedance ≥ 10^9 Ω and can measure voltage of 100 nV of order. For measurement on the samples with higher resistivity (up to 10^{10} Ω) the electrometer Keithley 610C is used (input impedance ≥ 10^{14} Ω). The temperature of both ends of sample is monitored by nickelchromium-nickel thermocouples (with cool ends 0 °C) digital voltmeters M1T330 (Metra). The thermocouple wires can be disconnected, which is necessary for measurements on high resistivity samples. The temperature gradient on the sample is held by small resistor heaters supplied by a D.C. power supply BS 575 (Tesla). There are two heaters (each of blocks has its own) and, therefore, the Seebeck coefficient can be measured in both directions of temperature gradient. The probe is placed in a quartz tube in order to measure in various atmospheres. The quartz tube can be insulated by metal foils as electrical shield. The probe is cooled by liquid nitrogen for measurements below room temperature. The increase in temperature is maintained by free evaporation of nitrogen (heating rate ~ 1 K/min) below room temperature. Outer resistance furnace controlled by electronic regulator for linear heating rate in wide region (commonly used 1 - 2 K/min) is used above room temperature.

The data from multimeter and both voltmeters are collected by a microcomputer by using IMS-2 bus. The temperature gradient below room temperature is controlled manually. In the measurements above room temperature the temperature gradient can be controlled by a microcomputer. The measurement on the low resistivity samples is fully automatic. In the measurements of high resistivity samples the temperature values are collected by the microcomputer and the voltage value is read from electrometer after disconnecting the thermocouples and manually entered into microcomputer. The Seebeck coefficient is calculated according to the relation $S = \frac{\Delta U}{\Delta T}$, where ΔT is temperature gradient and ΔU is thermoelectric voltage. As the probe is heated on both blocks, the measurement on high resistivity samples with residual voltage can be performed.

The examples of temperature dependence of S measurements are shown in Figs 2 - 4. The temperature gradient is order of tens Kelvin at temperature of liquid nitrogen, falls down when temperature increases to room temperature, and reaches 15 - 20 K above room temperature. The heating rate is 2 K/s. Figure 2 shows results of the measurement on low resistivity sample (sintered pellets of Nd_2CuO_4, $R \sim 10^3$ Ω) up to high temperature (900 K). The temperature dependence measured from nitrogen temperature (pressed pellets PbTe, $R \sim 10^0$ Ω) is shown in Fig. 3. The temperature dependence measurement on a sample with high resistivity ($VOPO_4.2H_2O$, $R \sim 10^8$ Ω) is illustrated in Fig. 4. In the case of high resistivity samples the sputtered platinum contacts on the sample become necessary.

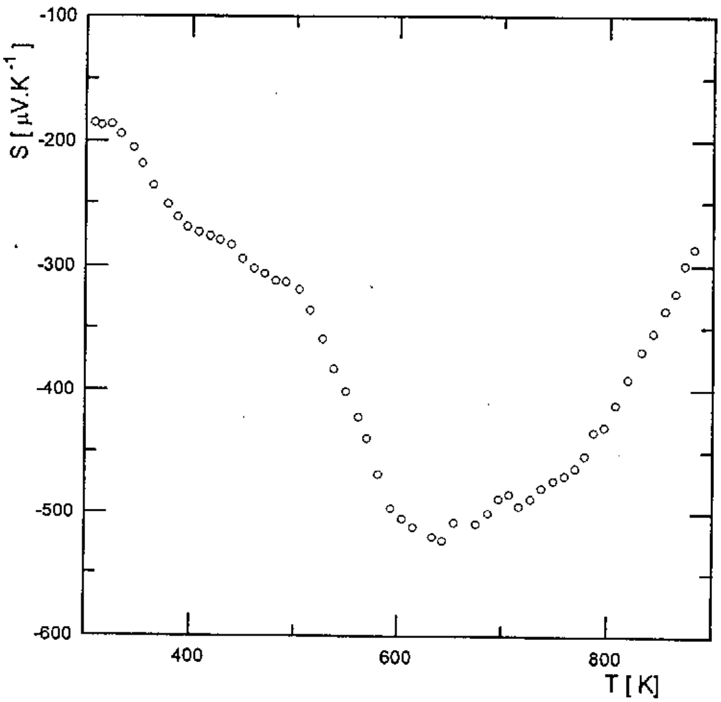

Fig. 2 Temperature dependence of the Seebeck coefficient of sintered pellet of Nd_2CuO_4 annealed in argon (sample size $16 \times 5 \times 1$ mm^3)

All these measurements clearly demonstrate the possibility of our apparatus to measure the Seebeck coefficient in wide teperature region on materials with different resistivities. However, these materials are polycrystalline and their thermopower can be influenced by their preparation method. In order to show the reliability of our measurement apparatus the temperature dependence of S of well defined sample cut from single crystal of Bi_2Te_3 is shown in Fig. 5. This $S(T)$ dependence is compared with the data obtained using a different apparatus in the Department of General and Inorganic Chemistry of University of Pardubice.

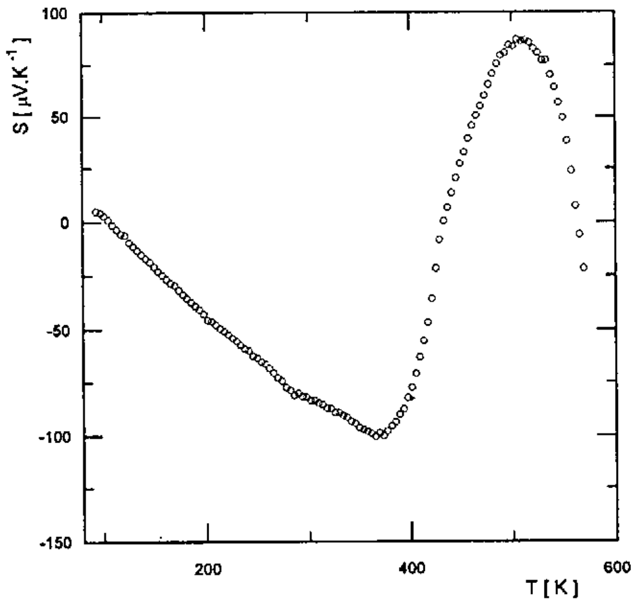

Fig. 3 Temperature dependence of the Seebeck coefficient of pressed pellet of PbTe (sample size 4×2×2 mm^3)

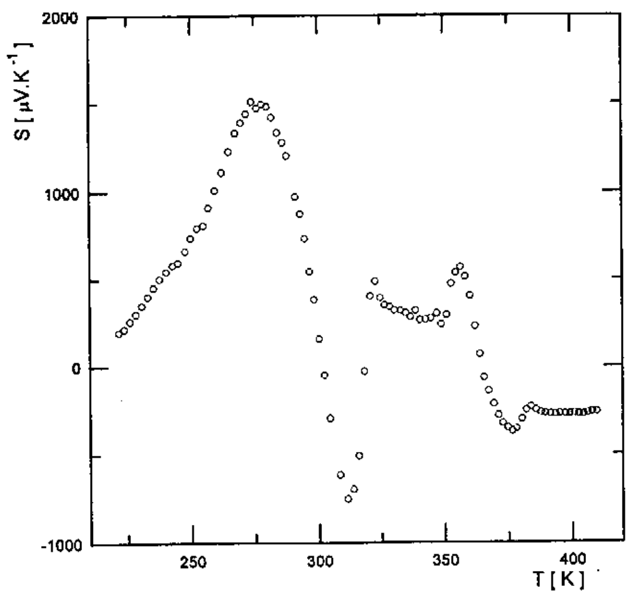

Fig. 4 Temperature dependence of the Seebeck coefficient of pressed pellet of $VOPO_4.2H_2O$ (sample size 8×3×3 mm^3)

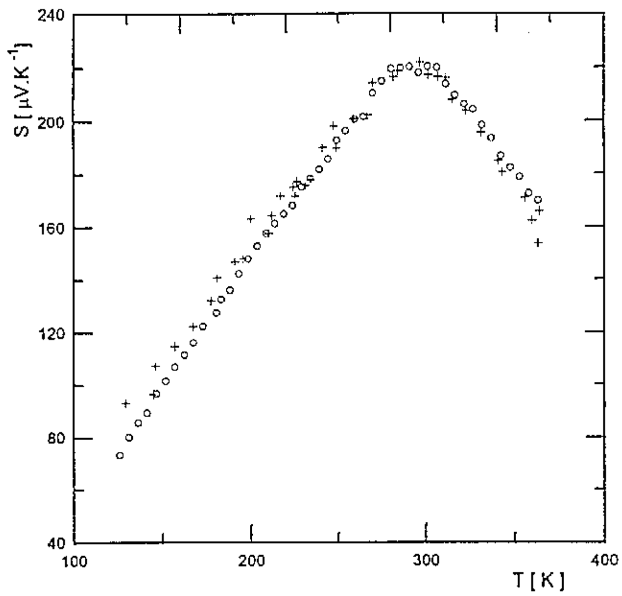

Fig. 5 Temperature dependence of the Seebeck coefficient of the sample cut from Bi_2Te_3 single crystal (circles) is compared with the data obtained by different apparatus (crosses). For detail see text