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An automated thermoelectric power apparatus using electro-optic relays

A CHAKRAVARTI and R RANGANATHAN

Low Temperature Physics Section, Saha Institute of Nuclear Physics, Sector I, Block AF, Bidhannagar, Calcutta 700 064, India

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Abstract. We report the design and construction of a thermoelectric power apparatus using home-made electro-optic relays with Z-80A microprocessor for automatic data acquisition and control. The advantages of such relays made out of LED-LDR combinations for the measurement of ΔE and ΔT are discussed in detail.

Keywords. Thermoelectric power; cryostat; automation; electro-optic relay; microprocessor.

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1. Introduction

Thermoelectric power (TEP) is a sensitive physical property which describes the processes that occur close to the Fermi surface. It can be defined as

$$S = \lim_{\Delta T \to 0} \frac{\Delta E}{\Delta T}$$

where S is the Seebeck coefficient and ΔE is the differential emf developed across the sample for a small temperature gradient, ΔT. Normally the Seebeck coefficient of a material is measured with respect to that of a reference material (Blatt and Schroeder 1978). In general the TEP measurements are based on two basic techniques, the integral technique and the differential ones (Caskey *et al* 1969). In the integral method the sample is in the form of a long wire whereas in the differential method the samples are rods, pellets or discs. The differential methods of measurement may be divided into two general categories—(i) slowly varying ac technique (Chaikin and Kwak 1975) (ii) the dc technique (Eklund and Mabatah 1977). In the ac technique, a slowly alternating temperature gradient is induced between two quartz blocks. The specimen, whose TEP is to be measured is placed between two gold wires attached to these quartz blocks. In the dc technique, the gradient can either be induced by a heater or it can be a natural gradient. However, some of the common problems encountered during experiments are the contributions from the spurious thermo-emfs, slow drifts, noise pick ups, bad thermal contact between samples and reference materials (e.g. copper), different equilibrium times for the sample and thermocouple which give rise to hysteresis behaviour, the measurement time between ΔE and ΔT, requirement of two nanovoltmeters, data acquisition etc. In this paper, we describe for the first time a method by using home-made electro-optic relays which are controlled by a Z80A microprocessor for the measurement of ΔE and ΔT, particularly for the small temperature gradient ΔT (from $-0{\cdot}5$ K to $+0{\cdot}5$ K) and also for data acquisition, using single (K 181) nanovoltmeter. We show that the common problems as described above can be minimized by using such an inexpensive arrangement.

2. Experimental system

2.1 *The cryostat*

The basic features of the cryostat are essentially the same as that reported in our earlier reference (Chakravarti *et al* 1991). The sample holder assembly is enclosed in a thin walled stainless steel tube ($l = 1075$ mm, $\phi = 39$ mm) which forms the exchange gas space of the cryostat. The sample holder is cooled by allowing helium exchange gas into the exchange gas space, thus placing the sample in thermal contact with the cryogen. To stabilize the temperature, the exchange gas is pumped out to a high vacuum ($\sim 10^{-5}$ torr) and by supplying small currents to the two heaters on the sample holder, the required temperature can be attained.

2.2 *Sample holder*

The details of the sample holder are shown in figure 1. The sample holder is suspended inside the exchange gas space by a stainless steel tube ($l = 952$ mm, $\phi = 10$ mm). It is made out of two pieces of copper. The bottom block has a groove for winding the bottom heater (BHTR—RT resistance 42·2Ω). It is attached to the top block with the help of three cotton fibre rods. The cotton fibre rods serve to isolate the bottom block electrically from the rest of the cryostat and also provide mechanical stability at low temperatures. Due to the threads cut on the rods, it is possible to adjust the gap between the top and bottom copper blocks according to the dimensions of the sample which fits between the top and bottom discs. The maximum gap allowable between the blocks in our set up is 14 mm. A copper disc is attached with low temperature stycast (2850 FT Emerson & Cumming) to the bottom end of the top copper block. This stycast layer serves the purpose of isolating the top copper disc

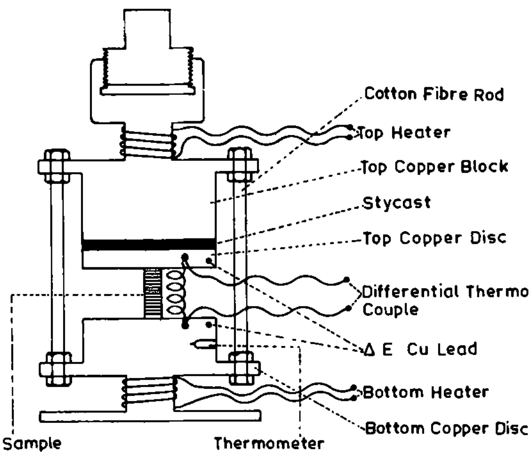

Figure 1. Details of the sample holder.

electrically from the rest of the cryostat while maintaining good thermal contact with the top block. This precaution is required in order to minimize pick up and noise in the microvolt level signals. Another heater (referred to as the top heater—THTR) of room temperature resistance 38·4Ω is wound on the top copper block to control the temperature gradient across the sample. A calibrated platinum resistance thermometer (model PT103 from Lake Shore) is embedded inside a hole with GE 7031 varnish in the bottom copper block to control the temperature to an accuracy better than ±0·05 K using a temperature controller (model 520 from Lake Shore) which drives the BHTR. The THTR is connected to a standard power supply. In order to measure the temperature gradient between the two ends of the sample, a differential thermocouple (calibrated chromel-gold with 0·07 at % iron-chromel) is connected to the two discs near the ends of the sample. The junctions of the thermocouple are embedded in holes in the copper discs (placed as close to the surfaces as possible) with GE 7031 varnish to ensure good thermal contact and electrical insulation. Thermal anchoring has been achieved by winding the chromel wires on the blocks over a layer of GE 7031 varnish. The voltage across the thermocouple is measured by means of a Keithley 181 nanovoltmeter. Two copper wires (SWG 40) are connected directly to the copper blocks very near the surfaces to measure the voltage developed across the sample because of the temperature gradient. All leads were twisted and taken out through cupro-nickel tubes (separately for ΔE, ΔT) to reduce possible pick up.

2.3 *Electro-optic relays*

The signals from the differential thermocouple (ΔT) and from across the sample (ΔE) are both of the order of microvolts, requiring the use of a nanovoltmeter. We decided to use relays to connect the K 181 meter alternately to the ΔT and ΔE leads with a very small time gap between the two measurements. This has been done to enable the use of only one nanovoltmeter rather than two such meters ideally required for such measurements. These relays have to provide extremely large insulation between the input and control signals, since the former is of the order of microvolts whereas the latter is in the volts range. It was found that the CD6004 MOSFET switch and available reed relays had too much cross-talk between signal and control channels (of the order of microvolts). Ordinary relays gave sufficient insulation, but the larger coil current caused gradual heating of the whole assembly, leading to about four or five microvolt thermoelectric signals between the relay contacts. Ultimately, instead of going in for expensive mercury-wetted relays (Berglund and Beairsto 1967) we decided to use a low cost LED-LDR (light emitting diode-light dependent resistance) combination, as shown in figure 2 inset. A small piece of stainless-steel tubing is closed at both ends using nylon stoppers. A LED is connected to the stopper at one end. The other end holds a LDR. With the LED glowing brightly the LDR shows a resistance of about 1 kΩ whereas in the dark its resistance goes above 20 MΩ. Since the input impedance of the Keithley 181 nanovoltmeter is greater than $10^9\,\Omega$ in the 2 mV range, the 1 kΩ on-state resistance is a short for all practical purposes. The 20 MΩ resistance of the off state is also quite small in comparison with $10^9\,\Omega$ and hence the switch should not really be deemed to be open if there is a single connection with the nanovoltmeter. However, if two inputs are present on a terminal of the meter, one with 1 kΩ impedance and the other with 20 MΩ, then it is reasonable to assume

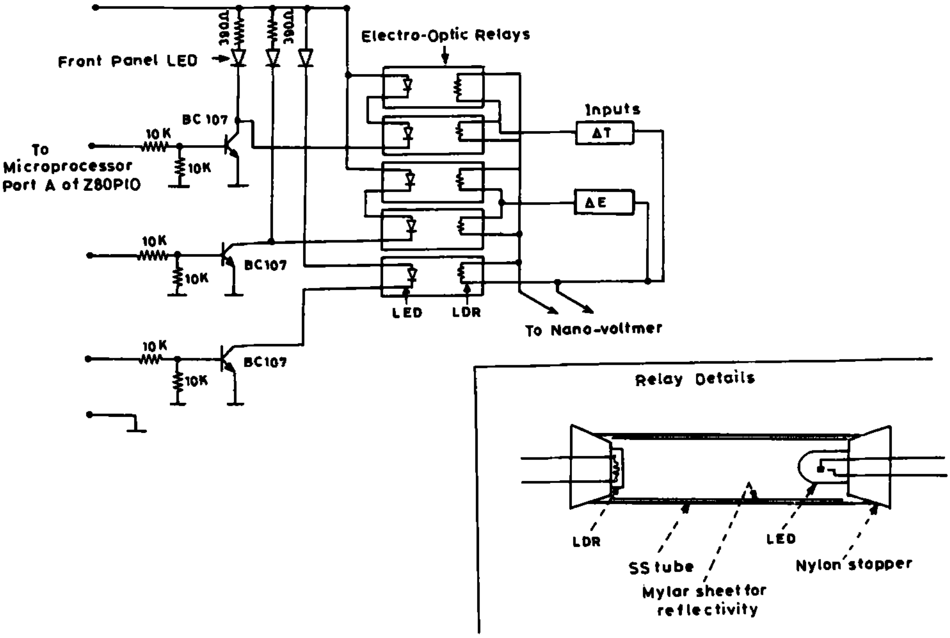

Figure 2. Circuit diagram for electro-optic relays. Inset shows the relay details.

that only the 1 kΩ signal is being measured by the meter, since the 1 kΩ resistance will short any signal coming out through the 20 MΩ path. We wish to emphasize that contact between the signal side and the control side is only through light rays and cross-talk is extremely low. Parallel pairs of these relays have been used on the ΔT and ΔE lines in order to reduce the resistance and also to increase reliability and one more relay is used to short the two input leads of the meter in order to facilitate zeroing. The control lines are connected to Port A of the Z-80 PIO in our MFZ-016 microprocessor trainer via suitable buffer circuits as shown in figure 2. It is thus possible to control the relays via software programmes. The voltage developed across the LDR's is $\sim$250 nV and remain reasonably constant and so are eliminated on taking the slope of the ΔE-ΔT curve.

2.4 *Measurement cycle and microprocessor programme*

A block diagram of the whole set up is given in figure 3. The measurement cycle is as follows:

The measurement is made while warming, so the system is allowed to cool down. The bottom block is now stabilized at the required temperature with the help of the temperature controller. Under this condition the top block will be at a lower temperature and the sign of the ΔT voltage will be negative in accordance with our convention of always measuring 'top block—bottom block'. Now a small current is sent through the top heater and the TEP programme (see figure 4 for flowchart*) is

*The detailed assembly language programme can be obtained from the authors on request.

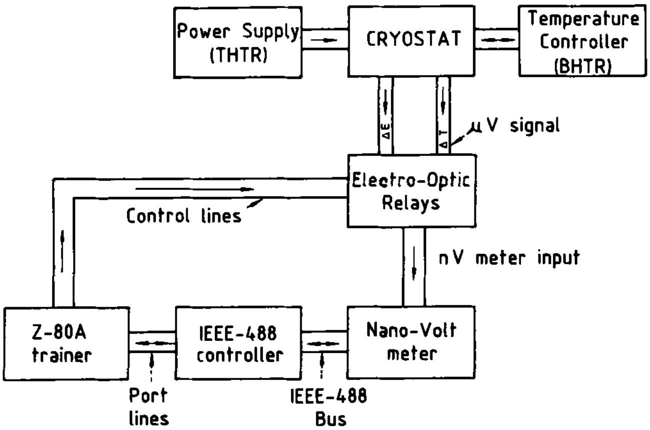

Figure 3. Block diagram of the automatic thermoelectric power experimental set up.

run on the microprocessor trainer. This programme first shorts the inputs of the nanovoltmeter and through the IEEE-488 interface zeroes it. Next the meter is connected to the ΔT terminals, a small delay is executed for stabilization and the ΔT voltage value is read in via the IEEE interface. If the voltage has a larger magnitude than $10\,\mu$V (the sign is negative), which corresponds to a temperature gradient of 0·5 K, the meter is shorted once more and the whole cycle repeats. Due to the gradual heating of the top block (at the rate of about a degree every ten minutes) ΔT becomes just more than $-10\,\mu$V after some time. This ΔT value is then stored in memory and the meter is connected to ΔE and the corresponding value recorded. The next pair of $\Delta T - \Delta E$ values is taken for $\Delta T > -9{\cdot}5\,\mu$V, the programme alternating between zeroing the meter and measuring ΔT till the new condition is satisfied. In this way, forty one pairs of values, from $-10\,\mu$V to $+10\,\mu$V at 500 nV intervals is read in, corresponding to a sweep of $-0{\cdot}5$ K to 0·5 K about the set temperature of the bottom block. Since the separation in time between successive pairs is much larger than the gap between a single measurement of ΔT and ΔE, the ΔE recorded may be considered to correspond to that particular ΔT even though the measurement is not strictly simultaneous. After the data have been taken the microprocessor calculates a straight line least square fit of the $\Delta E - \Delta T$ data, calculates the mean scattering, eliminates the points that are scattered more than two times the root-mean-square scattering, repeats the calculation with the new set of data and stores the slope, intercept, mean scattering and final number of points in a file in the trainer's RAM. The absolute value of the TEP is then calculated by subtracting the relative thermopower (as calculated from the $\Delta E - \Delta T$ slope) from the literature value of the absolute thermopower of copper.

The mean scattering and the number of eliminated points gives an idea of the noise in the data and the intercept gives an idea of the thermal equilibrium attained. In our case, if the intercept goes above 12–15 μV, we consider the data unreliable.

Once this operation (taking about 45 min.) is over, the top heater is switched off and a new temperature is set on the controller for the bottom block. Generally we allow a gradient of about seven to eight degrees to develop before switching on the top heater. In this way the rate of change of gradient as well as the bottom block temperature is sufficiently stabilized by the time actual measurement starts. Attempts

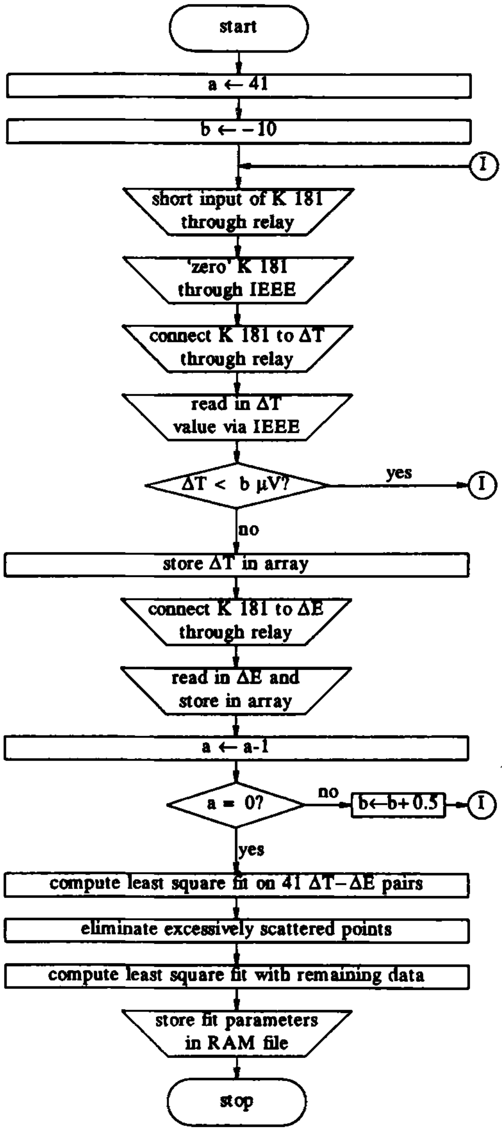

Figure 4. Flow chart for the thermoelectric power measurement cycle.

to reduce the measurement time by starting with lower gradients have led to unacceptably high values of the intercept and erratic results.

If the thermopower to be measured is exceedingly small (500 nV/K or less), however, the above measurement procedure is unsatisfactory. This is because on connecting the nanovoltmeter input from ΔT to ΔE, the input voltage has to decay from about 10 μV to about 100 nV. This decay and the stabilization of the meter at these values takes considerable time during which the gradient changes appreciably. Also, the damping and filter functions of the K 181 nanovoltmeter cannot be used if there are large changes in input. To deal with these situations another algorithm has been developed for dual nanovoltmeter measurements, with one meter connected permanently to the

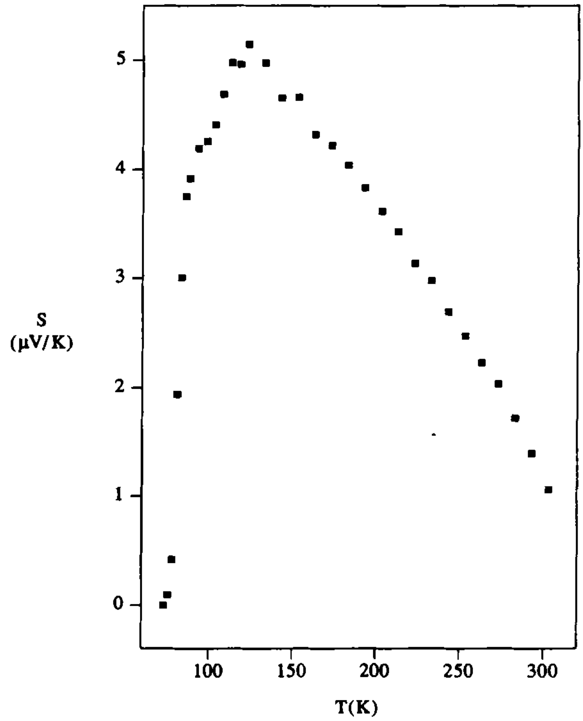

Figure 5. Absolute thermoelectric power of $Bi_2Sr_2Ca_1Cu_2O$ sample. (Each point in the figure is the result of a linear fit of about 41 sets of $\Delta E - \Delta T$ pairs to estimate the relative TEP).

ΔT leads and the other to the ΔE leads with separate IEEE-488 addresses. The system was tested with a specpure nickel sample (6 × 2 mm) and the absolute thermopower was determined within 5% accuracy in the temperature range 56 K to 300 K. The result of a typical measurement run on a high temperature superconductor sample ($Bi_4Sr_4Ca_2Cu_4O_y$) (Jayavel *et al* 1991) is shown in figure 5, where the typical error is $\leqslant 0{\cdot}08\,\mu V$ for the temperature range 73 K to 303 K.

3. Conclusions

We have described here an automated thermo-electric power apparatus for the first time using electro-optic relays controlled via Z-80A microprocessor for switching between ΔT and ΔE and also for data acquisition with extremely low cross talk, by creating the temperature gradient ΔT from $-0{\cdot}5$ K to $0{\cdot}5$ K.

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References

Berglund C N and Beairsto R C 1967 *Rev. Sci. Instrum.* **38** 66
Blatt F J and Schroeder P J 1978 *in Thermoelectricity in metallic conductors* (New York: Plenum Press)
Caskey G R, Sellmyer D J and Rubin L G 1969 *Rev. Sci. Instrum.* **40** 1280
Chaikin P M and Kwak J F 1975 *Rev. Sci. Instrum.* **46** 218
Chakravarti A, Ranganathan R and Raychaudhuri A K 1991 *Pramana – J. Phys.* **36** 231
Eklund P C and Mabatah A K 1977 *Rev. Sci. Instrum.* **48** 775
Jayavel R, Murugakoothan P, Venkateswara Rao C R, Subramanian C, Ramasamy P, Chakravarti A, Ranganathan R and Raychaudhuri A K 1991 *Solid. State Commun.* **79** 421