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To cite this article: S Mahakal *et al* 2020 *J. Phys.: Conf. Ser.* **1579** 012020

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# Simple, Efficient and Economically Viable Techniques for temperature dependant Thermopower data acquisition of Thermoelectric materials

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**Abstract.** A simple, economically viable and precise thermopower measurement techniques have been presented in this paper. Continuous data acquisition and simultaneous control of the instruments are achieved by using LabVIEW software. Set-up is built to measure thermopower of the low temperature thermoelectric material down to 10K. A LabVIEW program is developed to collect thermopower data of few  $\mu\text{V/K}$  to few hundred  $\mu\text{V/K}$ . Thermal stabilization and simple calculations are incorporated to avoid the spurious thermopower. Thermopower measurements, carried out in this set-up are published in reputed journals.

## 1. Introduction

Limitation of conventional energy sources, increasing energy demands and environmental concern enforce the society and the researcher to concentrate on the sustainable energy. Waste heat energy conversion and management by thermoelectric (TE) materials is drawing attention of the scientist. However, worldwide there is resurgence to enhance the efficiency of thermoelectric materials. TE materials are those, which convert heat energy to electrical energy and vice-versa [1]. Efficiency of TE is evaluated by the Figure of merit:  $ZT = \left(\frac{S^2\sigma}{\kappa}\right)T$ , where  $S$ ,  $\sigma$ ,  $\kappa$  are thermopower, electrical conductivity and thermal conductivity respectively. These are interrelated material's property [1, 2]. Optimization of these inherent physical properties may only enhance the efficiency of the TE properties. However, measurements of these properties require dedicated technique.

Thermopower ( $S$ ) is directly related with the electronics energy states of the materials. The nature of energy states and Fermi surface of the materials at a certain temperature are correlated with  $S$  by the following equation [3, 4]:

$$S = - \left( \frac{\pi^2 k_B^2 T}{3|e|} \right) \left[ \frac{d(\ln A(E))}{dE} + \frac{d(\ln l(E))}{dE} \right]_{E=E_F} \quad (1)$$

where,  $k_B$ ,  $e$ ,  $A(E)$ ,  $l(E)$  are Boltzmann constant, electronic charge, area of the Fermi surface and mean free path of electrons respectively. Hence,  $S$  is a complementary tool to estimate the charge carrier,  $\sigma$ ,  $\kappa$  etc. It is noteworthy to mention that sign of measured  $S$  usually represent the nature of the charge



carrier (p or n type) [1]. Further,  $S$  is related with electron-electron and electron-phonon scattering coefficients. In the TE device industry  $S$  help to estimate the efficiency quantitatively. Hence, study of  $S$  is not only interesting from the fundamental point of view but also energy conversion from the waste heat, using TE materials.  $S$  measurement requires a dedicated technique. There are basic two techniques to measure  $S$  i.e. (1) Integral Technique and (2) Differential technique[5].

Integral method requires a voltage measurement between thermocouple leads for a temperature difference. There is wire made by the sample material amid the thermocouple wires (Figure1). Other wire is made with reference material having known  $S$ . Another wire may be connected at the junction to measure temperature of the junction. Let,  $S$  of a sample has to be measured in an ambient temperature (Figure1). Temperature difference  $\Delta T = T - T_0$  is created to carry out the experiment in the ambient temperature  $T_a$ . Now, the voltage  $\Delta V$  (Figure1) may be written by the equation:

$$\Delta V = \int_{T_0}^T (S_x - S_{ref}) dT \quad (2)$$

$T_0$  is the temperature of the cold junction.  $T$  is junction temperature or may be regarded as hot temperature.  $S_{ref}$ ,  $S_x$  represent the  $S$  of reference material and sample material for the temperature difference  $(T - T_0)$ . Hence,  $S_x$  may be written as,

$$S_x(T) = -\frac{d\Delta V}{dT} + S_{ref}(T) \quad (3)$$

In order to carry out the experiment by using integral method, temperature of the junction has to be measured along with  $\Delta V$ .

In the differential method a small temperature difference is created between the two ends of a sample (Figure2). Two thermocouples are used to carry out the temperature and voltage difference measurement between two points. The  $S$  of the sample is:

$$S_x(T_{avg}) = -\frac{\Delta V}{\Delta T} + S_{ref}(T_{avg}) \quad (3)$$

where,  $T_{avg} = (T_1 + T_2)/2$ .

It is noteworthy to mention that in general  $S$  is measured in differential method now-a-days. Differential method is realistic approach for accurate  $S$  measurement. In the integral method sample is used in the form of wire. However, it is difficult to prepare wire of all samples. In this article, we have reported  $S$  measurement setup along with program to control the instruments and data acquisition using differential technique.

Details of setup, measurement procedure and key features of program using LabVIEW software for accurate estimation of  $S$  are reported in this article. Differential technique has been employed to carry out the  $S$  measurement. The Setup is built to carry out the experiment with any geometrical shape of sample.  $S$  may be measured at different ambient temperature and in wide range of temperature. Spurious  $S$  value may be incorporated from the contact resistance and temperature measurement. Further, thermal stabilization is very much important during the measurement. Here we present a less expensive, easy to mount sample and accurate  $S$  measurement techniques and setup.

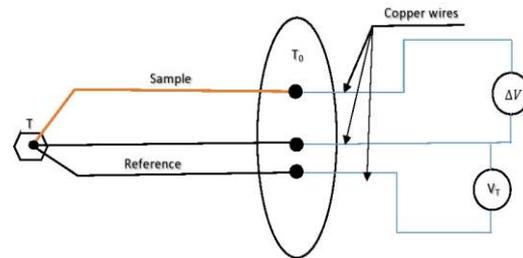


Figure1. Schematic diagram for thermopower measurement using integral method.

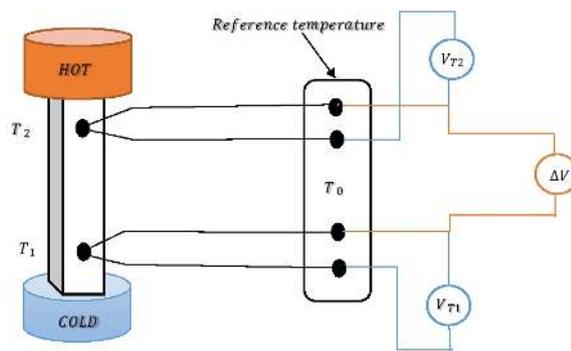


Figure2. Schematic diagram for thermopower measurement using differential method.

## 2. Experimental setup

Simple and conventional techniques are used to build the sample holder.  $S(T)$  measurements were performed in the range of 20 - 300 K with the help of closed cycle cryostat [Make-ColdEdge Technologies, USA]. Automatic data acquisition using LabVIEW software is employed. The measurement as well as data acquisition is fully automated and computer controlled using indigenously developed program. The schematic diagram of the sample holder is presented in Figure 3. There are two copper blocks in the sample holder. The upper copper block ( $U_B$ ) and lower copper block ( $L_B$ ) are joined by two Bakelite screws ( $B_S$ ). Hence, two copper electrodes ( $U_B$  and  $L_B$ ) are electrically isolated but mechanically connected. Sample is kept between  $U_B$  and  $L_B$ . In order to maintain pressure contact between Sample and electrodes, springs are attached to the top of the  $B_S$  at the  $U_B$ . A good electrical contact is required between the electrodes and sample to avoid spurious  $S$ . Small rectangular deeps are cut in the sampler holder for better grip of the sample. Further, ends of the sample are cured with silver paste and kept at the rectangular groove in sample holder. These help to make good electrical contact. Heaters are attached at upper electrode ( $U_{H_C}$ ) and lower electrode ( $L_{H_C}$ ) of the sample holder.  $U_{H_C}$  and  $L_{H_C}$  create and control temperature difference ( $\Delta T$ ) between the two ends of the sample. The program adjusts current in  $U_{H_C}$  and  $L_{H_C}$ . A chromel-AuFe(0.07%) thermocouple ( $T_C$ ) is attached just top of the sample for the precise measurement of  $\Delta T$ . Holes are drilled in the copper electrodes to fix junction of the thermocouple. Junctions of the thermocouples are cured with GE-Varnish for thermal contact with the electrodes without electrical connection. The copper wires  $V_L$  measure thermopower.  $V_L$  is directly soldered with the  $U_B$  and  $L_B$ . It is noteworthy to mention that entire set-up is covered with an aluminum foil to avoid the radiation heat loss and thermal stabilization. A cigarette paper is introduced between  $L_B$  and cold head of the cryostat for electrical insulation but thermally connection.

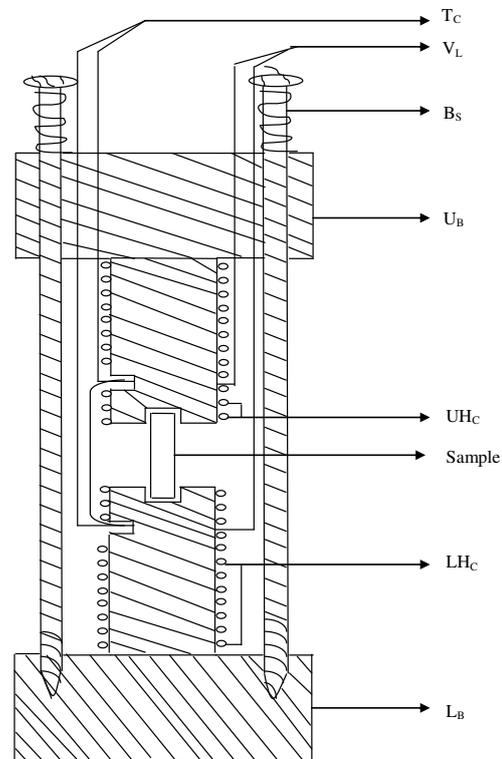


Figure 3. Schematic diagram for Thermopower measurement setup

## 3. Experimental Procedure

$S$  is measured during the heating cycle. The sample space or the inside of the cryostat is evacuated to a vacuum up to  $10^{-2}$  mbar at room temperature by a rotary pump. The sample holder is cooled down to 10K. Temperature controller (Make: Lakeshore, Model: 331) controls ambient temperature by Silicon diode sensor. Heater attached at cold head increases the ambient temperature. Hence, temperature of  $L_B$  remains higher than  $U_B$  during increase in ambient temperature. LabVIEW program sends appropriate current to  $U_{H_C}$  by current Source (Make: Keithley, Model: 6221) to maintain thermal equilibrium between two ends of the sample. Thermal stabilization is achieved by the temperature controller and current source. Multimeter (Make: Keithley, Model: 2000) records voltage of  $T_C$ . The voltage vs. temperature difference data of  $T_C$  is fitted with polynomial and used to convert voltage into ( $\Delta T$ ). After the thermal stabilization, program instruct current source for sending current to  $U_{H_C}$  to build preferred  $\Delta T$ . After that, Current source is automatically switched off. Now,  $U_B$  cools in natural

way. It is noteworthy to mention that voltage difference ( $\Delta V$ ) is built up between two electrodes and changes according to  $\Delta T$ . Voltmeter (Make: HP, Model: 3441) measures  $\Delta V$  data during cooling of  $U_B$  and simultaneously  $\Delta V$  and  $\Delta T$  are recorded. The set of ( $\Delta V$ ) vs.  $\Delta T$  data fitted with straight line equation:

$$y = mx + c \quad (4)$$

where  $y$ ,  $x$ ,  $c$  represent  $\Delta V$ ,  $\Delta T$ , and any arbitrary constant. Hence, the slope  $m = \frac{dy}{dx}$  give the value of thermopwer,  $S$ . Next ambient temperature is set by the program and measures  $S$  automatically. The flow chart for the computer control of automatic data acquisition is shown in figure 4.

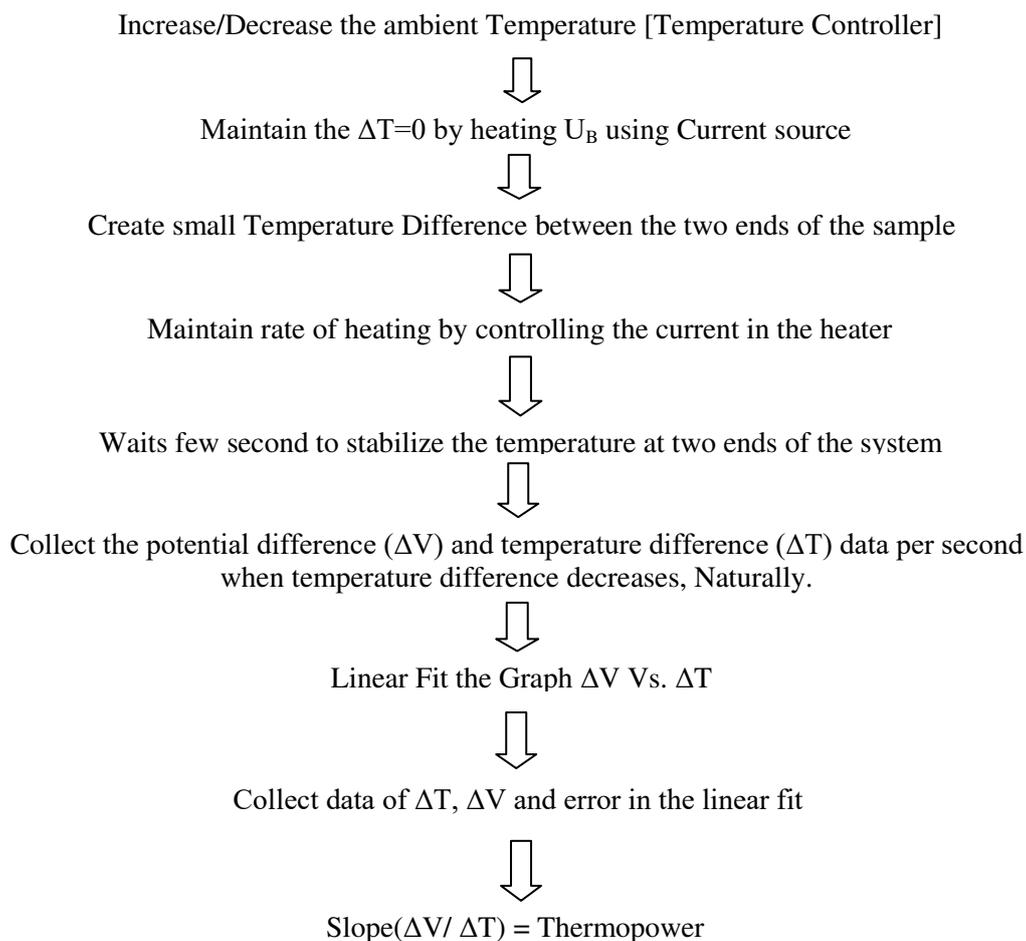


Figure4. Flowchart of the computer program used to carry out the Thermopower measurement.

Experimental results obtained by this setup are published in many reputed international journals. Thermoelectric material viz.,  $\text{Bi}_{1-x}\text{Sb}_x$ ,  $\text{Sb}_2\text{Te}_3$  samples are extensively studied using this setup [6, 7]. This setup investigate  $S$  for wide range of temperature. It measures  $S$  from 10K to 300K. Electron-electron (e-e) and electron-phonon interaction (e-ph) coefficient are extracted for  $\text{Bi}_{1-x}\text{Sb}_x$  using the equation (Figure5) [6]:

$$S = AT + BT^3 \quad (5)$$

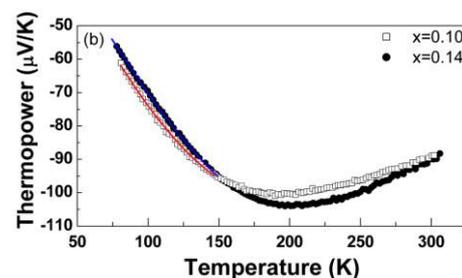


Figure5. Thermal variation of  $S$ . Solid line indicates the best fit with equation 5 [6]

Where A, B represent e-e and e-ph respectively. Further, Band gap is also calculated from the S(T). Das et.al. studied and fitted with a model of S value of  $\text{Sb}_2\text{Te}_{3-x}\text{Se}_x$  [7]. It is noteworthy to mention that negative value of S for Bi-Sb alloy and positive value for  $\text{Sb}_2\text{Te}_3$  corroborated with experimental nature of charge carrier. These are typical example for the quality of data produces by the setup.

S measures in this setup using differential technique. Thermal stabilization and measurement technique is important for accurate value. However, control measurements require almost twelve hour to collect data in the temperature range 10K to 300K in the interval of 3K. The setup and the program have potential enough to characterize the TE as well as transport properties in detail.

### Acknowledgement

We acknowledge the support of Science and Engineering Research Board, Government of India in the form of sanctioning research project, File Nos. EEO/2018/001224

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