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Design and Fabrication of High Temperature Thermoelectric Power Measurement Setup.

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Abstract

The designed TEP set up consists of two 250W flat top copper base cylindrical heaters separated by 1 mm. The control loop of these heaters contain K type Cr – Al thermocouples and are controlled by Eurotherm 2604 temperature profile programmer using thyristors with ±0.5 ^{0}C precision. The developed thermoelectric Voltage is sensed by 2604 controller itself by software “iTools” with ± 1 μV precision which also controls heating, cooling rates and dwell time. The above set up has been used to evaluate the transport properties of DVT grown WSe_2 crystals in the temperature range RT to 250 ^{0}C.

Key words: thermoelectric power measurement**,** WSe2 crystals**,** scattering constant, effective mass

1. Introduction

The investigation of the thermoelectric power (TEP) of thermoelectric material can yield valuable information about their electronic and structural properties. Good thermoelectrics can convert heat directly into electrical energy with a reasonable efficiency provided a substantial temperature gradient exists and the material has a high thermoelectric figure of merit (ZT). For efficient high temperature power generation, good thermoelectric materials with large ZT at high temperature are highly desirable. It is essential to evaluate and therefore measure the following key transport parameters: the Seeback coefficient, electrical resistivity, and thermal conductivity. It is often also useful to have data on the Hall coefficient. In any case, the measurement should be done with high accuracy and over a wide range of temperatures. Techniques to measure transport properties at low temperature are described in the literature, [1 – 5]. On the contrary fewer papers are available dealing with high temperature transport property measurements. Almost two decades ago Wood *et al.* [6] developed an apparatus for the Seeback coefficient measurement up to 1900 ^{0}C. A brief mention of the experimental technique also appears only occasionally in research papers dealing with high temperature transport studies [7 - 12]. In this paper we describe an apparatus that was setup in our laboratory to measure the Seeback coefficient at temperatures as high as 800 K.

2. Experimental Details.

A. Description of the apparatus

The longitudinal section of the apparatus is shown in Fig. 1 for the high temperature Seeback coefficient. Basically, it consists of well insulated 250Watt cylindrical heater. The heaters are constructed using nichrom wire and connected to two individual thyristor power supplies which are controlled the Eurotherm 2604 temperature controller. In high temperature thermopower measurement setup one of the key issues is establishing and maintaining a good thermal contact between the sample and the electrical leads which are measuring the emf generated from the sample. Hence, for sample mounting two highly cond-

-ucting copper plates of dimension 35×25×3 mm^3 are brazed on the top of the copper rods inserted into both the heaters and heated with the help of power supply. Holes of ≈ 0.5 mm diameter are drilled into the copper rods just below the two copper plates in order to insert very thin chromal – alumel (type - K) thermocouples.

In order to establish good thermal contact, the sample is mounted on these two copper blocks. For this purpose, we employ a spring loading mechanism made up of stainless steel strips that are situated on the top of the probe, which can be finely adjusted by rotating the knob on the top. In Fig. 1 & Fig. 2 the schematic diagram of the S.S. pressure contacts and sample mounting stage are shown. This ensures that both thermo – emf and temperature difference are measured at the same point and very close to the sample ends. Both the S.S. pressure contacts sense the generated emf due to temperature gradient and store the value in computer memory which is connected via RS232 cable and operated with the help of software 'iTools'.

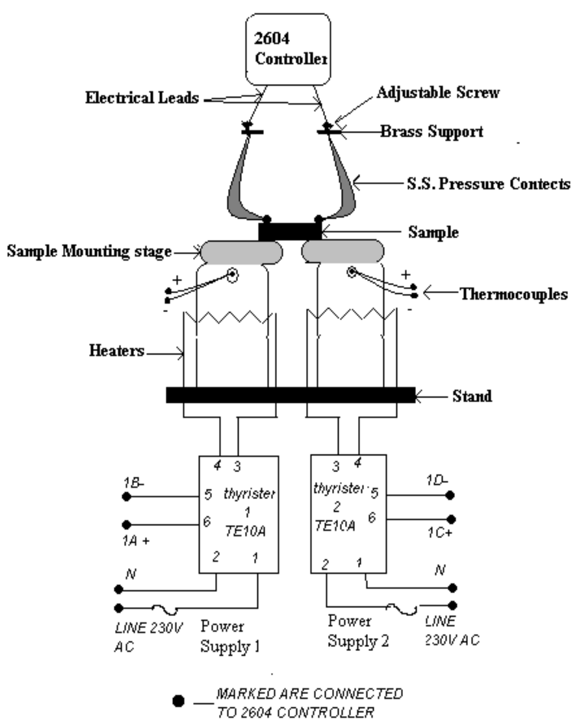

Fig. 1 Experimental setup for TEP measurement with Pressure contacts.

1. Temperature Measurement and Control

Accurate temperature setting, its control and measurement are critical for investigation of the Seeback coefficient. One needs to know not only the absolute temperature at which the measurement is taken but also the temperature along the length of the sample – an accurately determined gradient when studying the Seebeck effect.

Temperature of the heaters and hence both the copper plates are controlled by a Eurotherm 2604 temperature controller that sets and maintain the operational point to within $\pm 0.5^0$C over the range of temperature extending up to 800 K. This type of thermocouple remains stable and is resistant to oxidation at least up to 1000 ^{0}C. Since the entire system is placed in a vacuum chamber with pressure of 10^{-3} Torr, the influence of the thermal convection is eliminated and there is negligible temperature drift during the measurement. Also the same metal wires for both voltage leads are brought out of the vacuum chamber using a specially designed feed through to eliminate any artifacts due to dissimilar junction (not shown in figure). Here the TEP measurement is facilitated by using a computer system which can be controlled by the software 'iTools'. Fig. 3 is a block diagram of the equipment setup. The sample mounting stage is attached with the heaters and both heaters are placed in vacuum chamber. Two thyristors, which can be controlled individually by temperature controller Eurotherm 2604, are used to provide ac current for the heaters. The voltage across thermocouple and voltage across the sample are sensed by the same 2604 (multi purpose) controller. All electrical connections of the controller are interfaced with computer via RS232 cable.

2. Experimental Procedure

The sample with the area of at least 2×2 mm^2 is required for our TEP experiment. The sample mounting stage is first cleaned with acetone. Sample is than placed on the copper plates. Two ends of sample are connected to the electrical leads with the two taper ended S.S pressure contacts. (See Fig. 1 & 2)

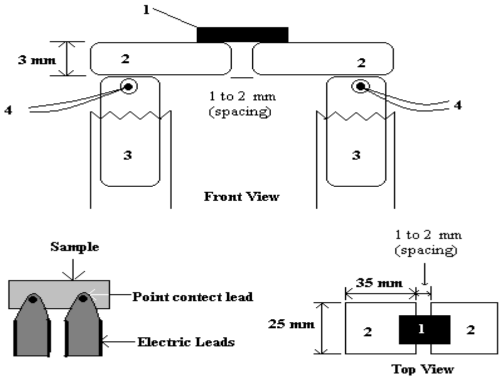

Fig. 2 Sample mounting Stage {1-Sample, 2-Copper plate, 3-Heater, 4-thermo couple}

The sample is positioned by placing its ends on the copper blocks and pressed with the taper ended strips and pressure is adjusted by the screw. The measurements are taken in a temperature range of 300 K to 800 K. The temperature difference (ΔT) between the ends of the sample can be kept 5 K to 10 K. The current for the heater is regulated by the controller (connected with the computer) to ensure the desired value of $\Delta \tilde{T}$ When the emf is generated for particular temperature gradient, it is sensed by the controller. The computer program chooses the correct value for S (Seeback coefficient) based on the value of ΔT. This is derived by the equation $S = \Delta \varsigma / \Delta T$. Here we connect the hot probe to the negative of the controller and cold probe to the positive of the controller. Hence n – type samples yield a negative Seeback voltage V. During the measurement, an S-T curve is plotted on the computer screen along with the numerical display of the latest pair of data (S, T) with the resolution of 1μV. The obtained data can be stored by selecting data logging facility of the software. The S-T curve as seen by OPC Scope of the software and software flow chart are shown in Fig.4 (a) and 4(b) respectively**.**

3. Results and Discussion.

To test our apparatus we have measured the thermopower of WSe_2 crystal grown by direct vapor transport technique. These crystals were found in the form of thin platelets having silvery opaque appearance [14, 18].

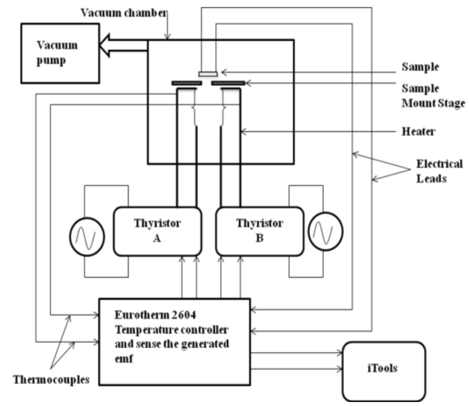

Fig. 3 Block Diagram of the equipment setup

For the TEP measurement, the crystals having dimensions $6\times5\times0.03$ mm^3 were used. The temperature difference between the two junctions was kept constant at 5 K till the temperature of the hot junction increased from 300 to 573 K. The variation of the Seeback Voltage with inverse of temperature is shown in Fig. 5. The sign of TEP is found to be positive and remains so over the entire temperature range indicating p-type conductivity of the crystals. It is observed that TEP increases with increasing temperature, confirming the typical semiconducting behavior of WSe_2.We also calculate the value of scattering parameter which gives the value of 1.3 shows that the ionized impurity scattering dominates the charge transport mechanism in WSe_2. Using the TEP value we have also calculated the effective mass of holes of value 4.917×10^{-31} and find that the effective density of state of value 3.5036×10^{27} m^{-3}. All these parameters are in good agreement with the values reported in literature [Refs. 13 – 19].

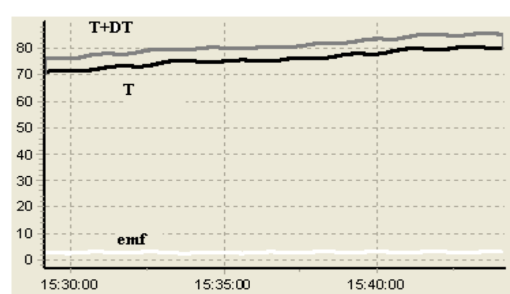

Fig. 4(a) S-T curve as observed through OPC Scope of iTools software

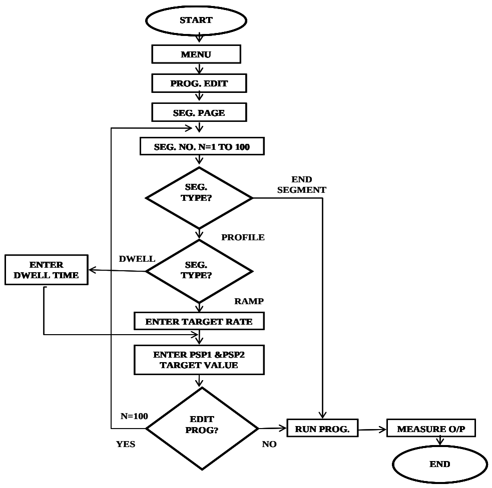

Fig. 4(b) Software flow chart.

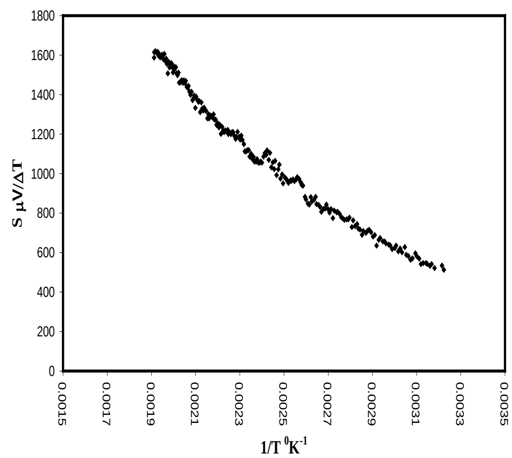

Fig. 5 variation of the Seeback Voltage with inverse of temperature

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