Experimental setup for the measurement of the thermoelectric power in zero and applied magnetic field

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Abstract

An experimental setup was developed for the measurement of the thermoelectric power (TEP, Seebeck coefficient) in the temperature range from 2 to 350 K and magnetic fields up to 140 kOe. The system was built to fit in a commercial cryostat and is versatile, accurate and automated; using two heaters and two thermometers increases the accuracy of the TEP measurement. High density data of temperature sweeps from 2 to 350 K can be acquired within 16 h and high density data of isothermal field sweeps from 0 to 140 kOe can be obtained within 2 h. Calibrations for the system have been performed on a platinum wire and Bi\textsubscript{2}Sr\textsubscript{2}CaCu\textsubscript{2}O\textsubscript{8+δ} high-\textit{T}_\text{c} superconductors. The measured TEP of phosphor bronze (voltage lead wire) turns out to be very small; the absolute TEP value of the phosphor bronze wire is much less than 0.5 \(\mu\text{V K}^{-1}\) below 80 K. For copper and platinum wires measured against the phosphor bronze wire, the agreement between measured results and the literature data is good. To demonstrate the applied magnetic field response of the system, we report measurements of the TEP on single crystal samples of LaAgSb\textsubscript{2} and CeAgSb\textsubscript{2} in fields up to 140 kOe.

Keywords: thermoelectric power, measurement setup, calibration

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Since its discovery in 1821 by Thomas Johann Seebeck, relatively few studies of the magnetic field dependence of the thermoelectric power (TEP) were carried out, mostly in pure metals [1]. However, over the past few decades, the magnetic field-dependent TEP studies of many materials ranging from magnetic multilayers [2] to high-\textit{T}_\text{c} superconductors [3], to the electron-topological transition [4] and to strongly correlated electron systems [5–7] have provided useful information. Intensive efforts have also been made in the search for highly efficient thermoelectric materials. This being said, the measurement of the intrinsic TEP is particularly difficult even in simple metals such as copper or gold. This is due to the small magnitude of the TEP at low temperatures and its sensitivity to the presence of small concentrations of impurities, where magnetic impurities can enhance the TEP below certain temperatures by means of the Kondo effect [1]. Few experimental details have been given in the literature concerning the measurement setups and the procedure for calibration of lead (as in contacting the sample, not Pb) wires [8–10]. Detailed descriptions of the measurement techniques at low temperatures and high magnetic fields can be found in [9, 10]. In this paper, we describe the development of an experimental setup for the TEP measurement in a Quantum Design (QD), Physical Property Measurement System (PPMS). The PPMS sample puck provides both thermal and electrical contacts to the sample. The merits of this technique are as follows: (i) it is easy to implement using two commercial, Cernox thin-film, resistance cryogenic...
temperature sensors and two strain gauge heaters and (ii) it is easy to control the temperature and the magnetic field of the system using the PPMS platform. Using the PPMS temperature–magnetic field (T–H) environment and the two heaters and two thermometers, an alternating heating method allows for measurements of the TEP of materials over a temperature range from 2 to 350 K and magnetic fields up to 140 kOe. The alternating heating method we use improves the resolution by a factor of 2 and provides a reliable temperature gradient. For the measurement, the sample is mounted directly between the two Cernox thermometers each of which is heated by a strain gauge heater with a constant dc current. An important component of this technique involves the use of phosphor bronze lead wires to reduce the background TEP and magneto-thermoelectric power (MTEP) associated with the lead wires.

2. Experimental setup

In this section, we will describe our specific sample holder (sample stage) and explain the data acquisition process. This measurement setup was designed to fit PPMS cryostat used to control the temperature and the magnetic field of the system. All instruments (current sources, voltmeters, switch system and PPMS) were controlled by National Instruments LabVIEW software. The sample holder can be easily modified and adapted to other cryogenic systems, including those with higher magnetic fields and lower temperatures.

2.1. Sample holder

Figures 1(a) and (b) show a schematic diagram of the sample stage built on the PPMS sample puck and a photograph of the actual sample stage. The magnetic field is applied perpendicularly to the plane of the heaters, thermometers and puck platform. Two sample stages are attached to a circular copper heat sink positioned on the 23 mm diameter PPMS sample puck that, when in use, is shielded by a gold-plated copper cap (not shown). We use Cernox sensors (CX-1050-SD package) as thermometers that provide high sensitivity at low temperatures, good sensitivity over a broad range and low magnetic field-induced errors. The dimensions of this package (1.9 × 1.1 × 3.2 mm^3) are large enough to attach a heater and a sample simultaneously to the package surface. Strain gauges (heaters), 0.2 × 1.4 mm^2 and typically R ∼ 120 Ω, are glued to the top of the Cernox thermometers using Stycast 1266 epoxy. In order to ensure thermal isolation, the heat sink (PPMS puck) and the sample stage were separated by a thin (1 mm thickness) G-10 plate. This G-10 plate was glued to the bottom of the Cernox thermometer using the Stycast 1266 epoxy. From several test runs we observed that the two Cernox wires and two heater wires provided enough cooling power to the sample stage since the strain gauge and Cernox each has low thermal mass. Each sample stage, including heater, thermometer and G-10 plate, was glued to the copper heat sink with GE 7301 varnish, so that it could be easily removed by dissolving the GE varnish with ethanol. Because of the constraint of the PPMS sample puck, the distance between two stages can be varied from ∼1.5 mm to ∼6 mm. Large flexibility with respect to the sample size can therefore be gained since the precise configuration of the thermal stage can be easily adjusted. If the sample length is smaller than 1.5 mm, it is hard to establish a temperature difference (ΔT) because both thermal stages are isolated from the heat sink. Typically, samples with the length varying from 2 to 7 mm can be measured. All wires on the measurement cell are thermally anchored to the heat sink. The TEP measurement was made with the PPMS operating in the high vacuum mode with a pressure of ∼10^-5 Torr.

For mounting the samples, and measuring the voltage, two different configurations were tested (figure 1(c)). First, the samples were mounted on the two sample stages with GE varnish. The voltage difference ΔV is measured using a 25 μm diameter copper wire or a phosphor bronze wire attached to the sample using silver epoxy as shown in the top of figure 1(c). Alternatively, the samples were directly mounted to the sample stages using the DuPont 4929N silver paste. The silver paste provides good thermal and electrical contact between the sample and the gold-plated layer on the surface of the Cernox package (bottom of figure 1(c)). The copper wire or the phosphor bronze lead wire is soldered to this gold-plated layer. In this case the voltage difference is obtained by measuring the voltage difference between two sample stages. Since the data were taken in a steady state, by assuming that the temperature of the gold layer is the same as silver paste, the TEP contribution of the sample stage can be ignored. Since the silver paste can be dissolved in hexyl acetate, the sample can be easily detached by carefully adding small amount of this solvent without degrading Stycast or GE varnish. We ran several test measurements to compare thermal coupling between the sample and thermometers by using the silver paste and GE varnish. We found it to be essentially the same for both cases. In general the TEP measurement was performed with the silver paste configuration, because the sample mounting and removal were easier than GE varnish. The GE-varnish configuration is preferred mainly when good electrical contact between the sample and the gold layer of the thermometer with the silver paste cannot be established. For example, when we measure the TEP of the Bi_xSr_yCaCu_2O_8+δ (Bi2212) high-Tc samples for calibration, it was hard to get good electrical contact (see the next section).

2.2. Determination of ∆T, ΔV, Toc, and S

A block diagram of the TEP measurement is shown in figure 1(d). Since the PPMS sample puck provides only 12 wires, they had to be used frugally: six wires in total were used for the two Cernox sensors, which were connected in series, four wires were used for the heaters (two each) and two wires were used for the TEP voltage. The resistance of each Cernox is measured with a Hewlett Packard 34420A nanovoltmeter via a Keithley 7001 switch system with a Keithley 7059 low voltage scanner card. The current was supplied to the Cernox thermometers by a Keithley 220 programmable current source. A temperature difference (∆T) across the sample was established by applying a dc current with two Keithley 220 programmable current sources
Figure 1. (a) Schematic diagram of sample stages. A: strain gauges for heater, B: thermometers (Cernox), C: G-10 for thermal insulation from the heat sink, D: voltage probe wires, E: sample. (b) A photo of the measurement cell. (c) Sample mounting method using GE varnish (top) and silver paste (bottom). (d) Block diagram of the measurement system. The system temperature and the magnetic field are controlled by PPMS. All instruments shown in the block diagram including PPMS are operated by LabVIEW software. The details of the use of the instruments are explained in the text.

alternately through one of the strain gauges at a time, while the voltage difference ($\Delta V$) across the sample was monitored independently with a Hewlett Packard 34420A nanovoltmeter.

When we apply a small temperature difference across the sample, the temperatures ($T_1(t)$, $T_2(t)$) and a voltage ($V(t)$) are recorded as a function of time, as illustrated in figure 2. $T_1$ and $T_2$ are the temperatures of the two Cernox thermometers that the sample spans. $t_i$ represents the time just before alternating power to the heaters (e.g. #1 on and #2 off) and $t_f$ indicates the time just before the next power switch (e.g. #1 off and #2 on). As shown in figures 2(c) and (d) in particular, from a linear fit of the measured voltage and temperature as a function of time, $\Delta T$ and $\Delta V$, respectively, the sample temperature $T_{av}$ and the TEP ($S = -\Delta V / \Delta T$) are calculated using the following equations:

$$2\Delta T = (T_{2f} - T_{1f}) + (T_{1i} - T_{2i})$$

$$2\Delta V = V_f - V_i$$

$$T_{av} = \frac{(T_{2f} + T_{1f}) + (T_{2i} + T_{1i})}{4}.$$ 

Since the temperature difference is generated by alternately applying power to one of the heaters, the measured voltage corresponds to $2\Delta V$. Thus, the TEP of the sample is calculated by $S = -2\Delta V / 2\Delta T$. Figure 2 shows the data corresponding to a measurement performed near 55 K on a platinum (Pt) wire sample, using phosphor bronze lead wires. The puck temperature was ramped at the rate of 0.1 K min$^{-1}$. A complete cycle, used to determine $\Delta T$ and $\Delta V$, took a time period ($\tau$) of 50 s. The parameters ($T_{1i}$, $T_{1f}$, $T_{2i}$, $T_{2f}$, $V_i$ and $V_f$) were determined by a linear fit of the data as a function of time as shown in figure 2(c) and (d).

The heater current ($I$) and time period ($\tau$), needed to generate given $\Delta T$, are not easy to estimate a priori, because of the temperature dependence of multiple parameters, such as the thermal conductivity and heat capacity of the sample, sample stage and all electrical wiring of the apparatus. Therefore, the current and measurement time for given $\Delta T$ were determined empirically at several temperatures by applying constant power to one of the heaters. For determining the final temperature and voltage, after switching the power from one heater to the other, the number of data points for a linear fit was selected within constant temperature and voltage region as a function of time. Although it depends on the sample under investigation, typical values of $\tau \sim 45$ s at 2 K and $\tau \sim 150$ s at 300 K for this setup allowed an accurate determination of the final values of $T_f$ and $V_f$. The typical values of the heater current were $I \sim 0.8$ mA to generate $\Delta T \sim 0.2$ K at 2 K and $I \sim 5$ mA to generate $\Delta T \sim 1.0$ K at 300 K.

By utilizing two heaters and an alternating gradient $\Delta T$, we avoid problems associated with offset voltages. $V_i$ and $V_f$ represent the thermal voltages in the circuit, which include spurious voltages and the TEP of lead wires. In fact, for very low values of the TEP, it is often necessary to consider an
Figure 2. Measurement procedure to extract the TEP from data corresponding to the measurement performed near 55 K on Pt-wire versus phosphor bronze wire. Actual time period (τ) between subsequent cycles, used to calculate the TEP, was 50 s. (a) Measured temperatures of both thermometers (T₁ and T₂) and (b) sample voltage (V) as a function of time. Note small (∼0.1 K min⁻¹) drift superimposed on data. (c) One cycle of measurement to determine parameters Δ₁T, Δ₁V: initial temperature Tᵰ, final temperature Tᵦ, initial voltage Vᵰ, final voltage Vᵦ and offset voltage Vᵨ. The solid lines represent the linear fit to the measurement data. The temperature difference for T₁ (T₂) is determined by Δ₁T₁ = Tᵰ₁ − Tᵦ₁ (Δ₁T₂ = Tᵦ₂ − Tᵰ₂) so that 2Δ₁T = Δ₁T₁ + Δ₁T₂. The voltage difference is calculated as 2Δ₁V = Vᵦ − Vᵰ (see the text).

offset voltage (Vᵨ) in the system and circuit. A common source of the spurious voltage, for example, is the wiring of the system from the voltmeter to the sample space since there is a thermal gradient and several soldering points between various wires. We found that the value of Vᵨ for this setup depended on temperature; it was ∼0.5 μV around 300 K and ∼−1.5 μV around 10 K. If we suppose that Vᵨ is independent of the small ΔT across the sample and has a small temperature dependence as a function of time (adiabatic approximation), Vᵨ can be easily canceled out using two heaters as shown in figure 2(d).

In the early stage of testing this measurement setup, the process of collecting data was checked by measuring the constantan wire (100 μm diameter) against the copper wire (∼20 μm diameter). Since the constantan wire has been known to have a large TEP value compared to the copper wire, the system can be tested without correcting the contribution of the copper wire as shown in figure 3. In this test run, we used the following two protocols. First, a stable temperature method was applied; in this measurement, the sample puck was held at a constant temperature and the TEP of the constantan wire using either one heater or two heaters was measured and found to be basically the same within the error bar of this measurement setup. However, the TEP data for the constantan wire showed a small hysteresis upon cooling and warming between 50 and 260 K with a maximum difference of about 2%. The origin of this hysteresis is not clear, and we expect that it is based on different relaxation times to stabilize the temperatures of the system.

Secondly, we adopted an alternate method which was used to measure the TEP while slowly warming the system temperature at the ramp rate of 0.1 K min⁻¹ below 10 K and of 0.45 K min⁻¹ above 100 K (shown for a measurement of the Pt wire in figure 2(a) for T ∼ 55 K). As the temperature was increased above 10 K, the ramp rate also increased for a certain temperature range, for instance 0.2 K min⁻¹ up to 20 K and 0.3 K min⁻¹ up to 100 K. It is worth noting that if the system temperature slowly warms up, it is necessary to carefully consider the time dependence of the sample temperatures and voltages. In this case we calculated ΔT and ΔV from a linear fit of the data. Continuous measurements while ramping temperature provide a high density of data and reduce the measurement time. In general, it takes 16 h to run from 2 to 350 K. This is in contrast to our finding that the relaxation time
Figure 4. TEP of Pt-wire versus phosphor bronze wire and Pt-wire versus copper wire. Circles and solid line represent the measured data from this work without any corrections. Both references 1 (open squares) and 2 (solid triangles) data are from [1].

To stabilize a sample stage completely under high vacuum at a single temperature is longer than 1 h. Figure 3 shows the TEP of the constantan wire based on these two protocols. In this test run, the agreement between measured results and the reference data is reasonable. The TEP extracted by the second protocol (slow drift of the system temperature) lies between the data taken on warming and cooling using the stable temperature method.

3 System calibration and sample TEP

Since the wires attached to the sample are either copper or phosphor bronze, a second thermal voltage is also generated. The measured TEP is then

\[ S_{\text{measured}} = S_{\text{sample}} - S_{\text{wire}}. \] (1)

Here \( S_{\text{wire}} \) represents the sum of the wire and all system contributions. When measuring an unknown sample, the TEP is the sum of \( S_{\text{wire}} \) and \( S_{\text{measured}} \).

The TEP of copper is strongly dependent on magnetic impurities below 100 K due to the Kondo effect [1] and therefore no reliable (or universal) reference data set is available for low temperatures. On the other hand, a superconducting material is a suitable reference because \( S = 0 \) in the superconducting state. In the present study, Pt-wire and Bi2212 high-\( T_c \) superconductors were each, separately, mounted between the two sample stages and calibration measurements were performed. These were sufficient for determining the lead wire contribution \( S_{\text{wire}} \). For the high temperature region, the pure Pt-wire (≈50 \( \mu \)m diameter) was used as a reference. Figure 4 shows the TEP of the Pt-wire versus copper wire and Pt-wire versus phosphor bronze wire. The result of the Pt-wire versus phosphor bronze wire is in good agreement with the absolute TEP value of Pt [1] which implies that the absolute TEP value of the phosphor bronze wire is negligible. Note that below 100 K, the Pt-wire manifests slightly different TEP responses depending on the heat treatment (annealing) of the wire. At low temperatures, we employed two superconducting Bi2212 compounds with \( T_c \) about \( \sim 82 \) K and \( \sim 92 \) K, where the different \( T_c \) values may be due to the heating of the sample in air. The results of the TEP measurement for Bi2212 against copper and phosphor bronze wires are shown in figure 5. In this calibration measurement, the samples were mounted on the two sample stages with GE varnish. The copper and phosphor bronze wires were attached to the sample using silver epoxy (top configuration of figure 1(c)). Here we used Bright Brushing Gold to attach the wire to the Bi2212 because using only silver epoxy provided a poor electrical contact, usually on the order of \( 10^3 \) \( \Omega \). After painting on the Bright Brushing Gold, the sample was heated up to \( 400 \) °C quickly, held for 5 min and air quenched to room temperature, where the contact resistance was reduced to below 100 \( \Omega \).

The absolute TEP of copper and phosphor bronze wires we measured and that of copper, from the literature, are shown in figure 6. Because \( S = 0 \) in the superconducting state, the observed TEP is the absolute TEP of copper and phosphor bronze wires. From figure 6(a) it is dramatically clear that the absolute TEP value of the phosphor bronze wire is very small, \( S \ll 0.5 \mu \text{V K}^{-1} \), up to 80 K. For the copper wire, the agreement between measured results and the literature data is reasonable. The inset of figure 6(b) shows the low temperature TEP of the copper wire. For the copper wire measured against phosphor bronze, no correction was added. These data indicate a fairly good agreement with the data taken from figure 5(b). The estimated uncertainty for the copper wire is about 0.3 \( \mu \text{V K}^{-1} \). In addition to the subtraction errors, we believe that this disagreement is due to a difference in quality of the copper wire in [1] and that used in this measurement.

As an aside, it should be noted that the low temperature, oscillatory behavior of the Bi2212 sample for \( H > 0 \) (figure 5) is reproducible. Although similar behavior was observed in the Nernst signal and associated with the plastic flow of the vortices [11], the origin of this phenomena is still somewhat unclear.

Previous TEP measurements at low temperatures and in high magnetic fields have had to take into account the significant contribution of the background voltage. By using well-known elemental metal wires of copper or gold and superconducting materials, these background contributions can be accounted for, correcting the background contribution. For small single crystals, an alternating ac current technique, utilizing a thermocouple, has been used to measure the TEP under high magnetic fields for a wide range of temperatures [9, 10]. Although the thermocouple wire provides a good sensitivity for relative temperatures, an accurate determination of \( \Delta T \) in high magnetic fields becomes difficult and large efforts are needed to calibrate the field dependence of the thermocouple wire.

In order to exclude the difficulties due to the magnetothermoelectric power (MTEP) measurement based primarily on the field dependence of \( S_{\text{wire}} \) and thermometer calibrations, we selected phosphor bronze wire and Cernox. Whereas the TEP of the copper (Cu) wire is not small and shows a field dependence, phosphor bronze wire provides essentially
zero TEP over a wide temperature range and is almost temperature and field independent [12] as shown in figures 5 and 6. Therefore, in this measurement setup, the magnetic field dependence of the TEP of samples, including the quantum oscillation (de Haas–van Alphen oscillation) at low temperatures, can be reliably measured. To demonstrate the versatility and reliability of this technique, two research samples (as opposed to wires of Cu or Pt) were measured; the TEP data are shown in figure 7 as a function of the temperature and figure 8 as a function of the applied magnetic field. LaAgSb2 has been observed to have a charge density wave order at ∼210 K and ∼185 K [13, 14], and CeAgSb2 was characterized as a ferromagnetic Kondo lattice compound with the Curie temperature $T_c = 9.8$ K [13]. In both compounds de Haas–van Alphen (dHvA) oscillations at low temperatures have been observed [15]. These single crystals were grown by excess Sb flux [13]. Samples were prepared with dimensions about 0.8 × 0.2 × 2.5 mm$^3$ for LaAgSb2 and 0.8 × 0.2 × 3 mm$^3$ for CeAgSb2. Zero-field measurement of resistivity and the TEP of both materials are presented in figure 7. The resistivity data are consistent with the earlier study and the TEP has clear features at the same transition temperatures. For $H \parallel c$ at 2.3 K with $\Delta T = 0.2$ K, for both materials, dHvA-type oscillations were observed in the TEP as a function of field, $S(H)$, shown in figure 8. Fourier analysis (fast Fourier transform) of the $S(H)$ data reveals peaks in the spectrum. The observed frequencies match those obtained from resistivity and magnetization [15]. The detailed data analysis will be published elsewhere [16]. So as to provide a clear sense of how readily $S(T, H)$ data can be acquired using this technique, it should be noted that the temperature dependence of the TEP was taken over ∼14 h and the field dependence was taken at the ramp rate of 25 Oe s$^{-1}$ (∼2 h).

The accuracy of this technique was estimated by using the measurement of the Pt and Cu wires. The estimated uncertainty of this system over all temperature ranges falls within a maximum of ±1 μV K$^{-1}$, and the relative accuracy is within a maximum of 10%. In the high temperature region, roughly above 100 K, the main uncertainty originates from inaccurate determination of the $\Delta T$ due to the relatively low sensitivity of the Cernox. The absolute and relative temperatures of Cernox were observed within a resolution of 4 mK at low temperatures, and the relative error at
high temperatures falls within $\sim$200 mK. For materials having low thermal conductivity, the error may be larger due to the temperature difference between the sample and the thermometer. For materials having small TEP, less than $0.5 \mu V K^{-1}$, the error can also be larger due to noise. More contributions to the error need to be considered for TEP measurements in the magnetic field. For instance, due to the heat conducting environment which is mainly caused by the induced current by applying magnetic fields ($d\Phi /dt$), it is very important to make sure that the ramp rate of the magnetic field should be slow enough to avoid additional heating and reduce the induced voltage due to the open loop. Alternatively, the TEP can be measured stepping the magnetic field with the magnet in persistent mode for each value of the field.

4. Summary of technical parameters and reference information

- Operation range: temperature range from 2 to 350 K and magnetic fields up to 140 kOe.
- Sample dimension: the length of the sample is between 1.5 and 7 mm.
- $\Delta T$: from 0.1 to 2.5 K, depending on the temperature and the absolute TEP value of the sample.
- Ramp rate of the system temperature: it can be varied up to 1 K min$^{-1}$. For example, in the calibration measurement, it was selected from 0.1 K min$^{-1}$ up to 10 K, 0.35 K min$^{-1}$ up to 100 K and 0.45 K min$^{-1}$ above 100 K.
- Estimated accuracy: maximum of $\pm 1 \mu V K^{-1}$ and 10% depending on the temperature and the sample. The limit of accuracy is mainly imposed by the limitations in the thermometry and the thermal contact between the sample and the thermal stage. If the absolute TEP of the sample is smaller than $0.5 \mu V K^{-1}$ the fluctuation of the sample voltage was observed.
- Copper wire: 0.025 mm diameter, Puratronic, 99.995% (metals basis), Alfa Aesar. Detected impurity elements are Fe, Ag, O and S (as provided by the supplier).
- Phosphor–bronze wire: Cu$_{0.94}$Sn$_{0.06}$ alloy, 0.025 mm diameter, GoodFellow.
- Platinum wire: 0.05 mm diameter, 99.95% (metals basis), Alfa Aesar.
- Silver epoxy: H20E, Epotek.
• Strain gauge: FLG-02-23, 0.2 × 1.4 mm² grid made from the Cu–Ni alloy and 3.5 × 2.5 mm² thin epoxy backing, Tokyo Sokki Kenkyujo Co., Ltd.
• Silver paste: DuPont 4929N silver paint, DuPont, Inc.
• Stycast 1266: Emerson & Cuming, Inc.

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