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ABSTRACT

This work documents an all-in-one custom setup that allows us to measure the in-plane Seebeck coefficients and electrical conductivities of anisotropic thin film samples close to room temperature. Both pairs, S_{\parallel} and σ_{\parallel} and σ_{\perp} and σ_{\perp} , can be measured using four contacts on the same sample, reducing measurement time and minimizing potential sources of error due to aggregating data from several distinct samples. The setup allows us to measure the electrical conductivity of isotropic samples using the well-known van der Pauw method. For samples with in-plane anisotropy, the two components σ_{\parallel} and σ_{\perp} can be extracted from the same type of measurements by performing additional calculations. Using the same contacts, the Seebeck coefficient along one direction is measured using a differential steady-state method. After rotating the sample by 90°, the orthogonal Seebeck component can be measured. In order to show the generality of the method, we measure different types of samples, from metal references to oriented doped conjugated polymers.

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I. INTRODUCTION

Electronic and heat transport have been shown to depend on spatial direction for many thermoelectric (TE) material systems, including some of the best performing ones. These anisotropic thermoelectric properties are due to a variety of structural asymmetries, such as different properties along the crystallographic directions, or preferential molecular order, and their accurate knowledge is paramount for the understanding of materials and the design of efficient TE generators.

Among the different material classes, abundant and solution processable organic thermoelectrics (OTEs) are potentially cheap and thus a promising thermoelectric option for low-temperature applications. To make full use of their advantages, OTEs are typically used in an in-plane geometry, where the temperature gradient is applied along the substrate direction.^{1,2} Compared to the more traditional out-of-plane geometry, this requires measurement setups that are adapted to the new geometry.³

An added complication is that OTEs are often intrinsically anisotropic materials. This work focuses on in-plane oriented materials in an in-plane geometry since this may allow increasing the TE performance given by the dimensionless figure of merit,

$$ZT = \frac{S^2 \sigma}{\kappa} T,$$
 (1)

where S is the Seebeck coefficient, σ and κ are the electrical and thermal conductivity, respectively, and T is the average temperature.

Being able to reliably access the anisotropic electrical conductivity and Seebeck coefficient would contribute to a deeper understanding of the transport properties of these systems and, moreover, allow the design of improved generators. Characterization of these anisotropic samples is, however, not straightforward. Typically, measurements of *S* and σ require different sample geometries. For example, while parallel line contacts on an unpatterned sample are often used to determine conductivity, the interpretation of Seebeck measurements in the same geometry is nontrivial.⁴ On the other hand, if *S* and σ are measured on different samples, then the question that the nominally identical pairs of samples are in fact not identical due to, for example, a different doping level, thickness, or morphology could be raised. For anisotropic measurements, even more samples would be required, potentially aggravating the situation.

Here we present a custom setup that allows us to sequentially measure both in-plane orthogonal pairs, S_{\parallel} and σ_{\parallel} and S_{\perp} and σ_{\perp} , on a single sample close to room temperature, in air.

II. SETUP

A sketch of the setup is shown in Fig. 1. It mostly comprises cheap off-the-shelf components. The sample is placed on a sample holder comprising two copper blocks, as shown in Fig. 2, one of which serves as a heater and the other as a heat sink, to create a controllable temperature gradient.

The sample is in contact at the corners with four springloaded type T thermocouples (TCs). A Keithley 2400 SourceMeter is used to measure voltage and apply current. Connections between any of the 4 SourceMeter terminals (V^{\pm} , I^{\pm}) and the 8 TC leads (TC^{copper}_{1,2,3,4}, TC^{constantan}_{1,2,3,4}) are made with a custom relay board. Temperature is regulated with a proportional-integralderivative (PID) controller that drives a solid state relay (SSR), which supplies power to a cartridge heater embedded within the copper block. The experiment is controlled, and data are acquired by a Python script running on a Raspberry Pi single board computer.

As for price, the Keithley 2400 SourceMeter, a piece of equipment that most laboratories will probably already have access to, is the most expensive part of the setup. All other components combined cost a fraction of its price.



FIG. 1. Schematic of the experimental setup.



FIG. 2. (a) Sketch and (b) a photograph of the sample holder. A sample supported on glass is placed face-down on the four thermocouples and fixed in place. (c) A sketch detailing the measurement of $R_{12,34}$. (d) A Sketch of a sample with fibers oriented along the x-direction. The Seebeck voltage can be measured either with (e) copper wires or (f) constantan wires, (g) while the temperatures are obtained from the voltages across each thermocouple separately. (h) Four different pairs of corners can be used to measure the Seebeck voltage. For clarity, relays and the reference junction are omitted from the sketches.

A. Sample requirements

The samples have to conform to the requirements of the van der Pauw method. This means they should be homogeneous thin films, with a uniform thickness that is significantly smaller than their length or width, and they cannot contain any holes.⁵ Electrical connection is made at their four corners by relatively small contacts, e.g., small silver paste droplets.

When determining the degree of anisotropy, some additional restrictions apply. The samples have to have a rectangular (or square) shape of known dimensions, and the principal directions of conductivity have to be oriented parallel and perpendicular to the edges.⁶ Finally, if both Seebeck measurements are to be carried out on the same sample, it has to be square shaped. This is because the sample will have to be turned by 90°, which requires the distance between any two adjacent corner contacts to be the same as that between two thermocouples.

B. Method

After bringing the sample in contact to the holder, the resistance between pairs of contacts is measured using the copper TC leads to ensure good ohmic contact. Then, the sheet resistance and the Seebeck coefficient are measured sequentially.

1. Electrical conductivity

The van der Pauw method is used to determine the sheet resistance R_s of the film,⁵ from which the electrical conductivity σ can be obtained if the sample thickness t_z is known,

$$\sigma = \frac{1}{t_z} \frac{1}{R_s}.$$
 (2)

To this end, using the copper leads, a current I_{mn} is supplied between two neighboring contacts m and n, and a voltage V_{op} is measured between the contacts o and p along the opposite edge of the sample, as sketched in Fig. 2(c). For each of the two orthogonal directions xand y, four such measurements are possible, by exchanging positive with negative and current with voltage terminals. Using the average values $R_{x,y}$ of these two groups of measurements,

$$R_x = (R_{12,34} + R_{34,12} + R_{21,43} + R_{43,21} +)/4,$$

$$R_y = (R_{23,41} + R_{41,23} + R_{32,41} + R_{14,32} +)/4,$$
(3)

the sheet resistance R_s can be obtained by solving

$$e^{-\pi \frac{R_x}{R_s}} + e^{-\pi \frac{R_y}{R_s}} = 1.$$
 (4)

In the case of an ideal isotropic square sample, R_x will be equal to R_y . For an anisotropic or rectangular sample, they may differ by orders of magnitude. For anisotropic samples, the basic van der Pauw method only allows us to determine the geometric mean of the conductivity or resistivity,⁶ with no way to determine the components in the general case,

$$R_{\rm s} = \frac{1}{t_z} \sqrt{\rho_{\parallel} \rho_{\perp}} = \frac{1}{t_z \sqrt{\sigma_{\parallel} \sigma_{\perp}}}.$$
 (5)

This bears repeating. This and other simple methods, e.g., collinear four point measurements, are not able to determine the individual components of the resistivity tensor $\rho_{\parallel,\perp}$ but instead measure the geometric mean of both components $\sqrt{\rho_{\parallel}\rho_{\perp}}$, no matter how the probes are oriented or how many measurements are performed.⁷

To determine the parallel and perpendicular components of ρ or σ , a sample that is oriented along these principal directions is necessary. That is, the horizontal and vertical sample edges have to be oriented along the parallel and perpendicular component of the

resistivity tensor [e.g., as sketched in Fig. 2(d)]. When R_x and R_y are measured along either ρ_{\parallel} or ρ_{\perp} , only then $\sqrt{\frac{\rho_x}{\rho_y}} = \sqrt{\frac{\rho_{\parallel}}{\rho_{\perp}}}$ can be obtained from the same van der Pauw measurements, using either of the following equalities:⁶

$$\sqrt{\frac{\rho_{\rm x}}{\rho_{\rm y}}} = -\frac{1}{\pi} \frac{l_y}{l_x} \ln\left(\tanh\left(\frac{\pi}{16}\frac{R_{\rm y}}{R_{\rm s}}\right)\right),\tag{6}$$

$$\sqrt{\frac{\rho_{\rm y}}{\rho_{\rm x}}} = -\frac{1}{\pi} \frac{l_x}{l_y} \ln\left(\tanh\left(\frac{\pi}{16} \frac{R_{\rm x}}{R_{\rm s}}\right)\right). \tag{7}$$

If the sample length l_x and width l_y are known, then Eq. (5) together with either Eq. (6) or Eq. (7) allows us to determine the individual components $\rho_{\parallel,\perp}$.

2. Seebeck coefficient

Then, the average Seebeck coefficient close to room temperature is determined from the slope of the Seebeck voltage vs the applied temperature difference. To that end, one side of the sample is heated slightly, while the other end is passively cooled by a copper heat sink. Temperatures T at each sample corner are obtained by measuring the voltage across each of the thermocouples. The Seebeck voltage is measured under steady state conditions, as soon as the hot side temperature is stable.⁸ S is measured twice, using just the copper (V_{Cu}) or just the constantan (V_{Co}) leads of a given thermocouple pair, as sketched in Fig. 2.⁹ Because the equipment only allows us to measure a single voltage at a time, the measurements are carried out sequentially in the order $(T_m, T_n, V_{Co}^{mn}, V_{Cu}^{mn}, V_{Cu}^{nm}, V_{Co}^{nm}, T_n, T_m)$ where each value is the average of N readings and V^{mn} denotes the Seebeck voltage measured at corner m with respect to corner n. The pairs of temperatures are averaged, to minimize the effect of any residual thermal drift, and combined with each of the Seebeck voltage measurements, to yield data points in the $\Delta T - V$ diagram.

The same sequence is repeated three more times, to acquire data for all combinations of thermocouples between the two hot and cold contacts [two along the sample edges and two across its diagonal, as sketched in Fig. 2(h)]. Strictly speaking, these measurements are superfluous if the sample is perfectly uniform. However, by using more than one contact on the hot and cold side, additional information on the homogeneity of the sample can be obtained.¹⁰

The procedure is repeated at a different hot side temperature set-point until sufficient data are acquired. Typically, five different hot side temperatures of $T_{\text{hot}} = \{33, 34, 35, 36, \text{ and } 37\}^{\circ}\text{C}$ are used. At the same time, the (uncontrolled) cold side temperature increases slightly from the starting room temperature, as can be seen in Fig. 3(a). The resulting temperature difference is normally of the order of 10 K, and the complete procedure takes about 30 min.

Figure 3 shows an example of the data generated by such a measurement for an isotropic nickel reference sample. Two pairs of $\Delta T - V$ curves are shown. Comparing the normal (V^{mn}) and reverse polarity measurements ($-V^{nm}$) shown in Figs. 3(b) and 3(c), an offset is clearly visible, which is due to stray voltages. The slopes shown in Figs. 3(e) and 3(f) are identical though, which means that those stray voltages do not significantly change during the course of

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FIG. 3. Example data acquired during the measurement of a nickel reference sample: (a) temperature of the hot side, the cold side, per-point average $T = \frac{T_{hot}+T_{cold}}{2}$, and average over the course of the measurement sequence T_{avg} , in red, blue, gray, and dashed, respectively, corresponding Seebeck voltages measured with (b) the cooper leads V_{Cu} and (c) the constantan leads V_{Co} , (d) V_{Cu} plotted vs V_{Co} , (e) V_{Cu} vs ΔT , and (f) V_{Co} vs ΔT . In (b)–(f), the dark squares and light triangles correspond to measurements with the opposite polarity. In (d)–(f), the fitted slopes, the correction due to the thermocouple wires, and the end result are given in the figure legend.

the measurement sequence. The contribution of the thermocouple wires to the measured slopes is accounted for by a simple first order approximation. The Seebeck coefficient of copper $[S_{Cu}(T_{avg})]^{11}$ and constantan $[S_{Co}(T_{avg})]^{12}$ is calculated at the sample temperature averaged over the course of the measurement T_{avg} and added to the measured slope. The results agree very well with each other, as well as with those of the literature (see Fig. 5).

Another way to obtain the sample Seebeck coefficient is shown in Fig. 3(d). Here, the Seebeck voltages are plotted against each other (e.g., V_{Cu} vs V_{Co}) instead of against temperature, and S is extracted from that slope using the following equation:⁹

$$S = \frac{S_{\text{Cu}}(T) - S_{\text{Co}}(T)}{1 - \frac{\partial V_{\text{Cu}}}{\partial V_{\text{Co}}}} + S_{\text{Co}}(T).$$
(8)

This way, only the average sample temperature at each data point $T = \frac{T_{\text{hot}}+T_{\text{cold}}}{2}$ has to be known, which may be advantageous in some cases. As seen in Fig. 3(d), this method again agrees well with the others.

Finally, to measure the Seebeck coefficient of an anisotropic sample, the measurement has to be repeated after the sample is rotated by 90° .

C. Hardware

In Sec. II C, the hardware is described in more detail. A Raspberry Pi single board computer controls a Keithley 2400 SourceMeter, which is used to source current and measure voltage. All other parts of the setup are custom built from off-the-shelf components. A schematic of the setup is shown in Fig. 1.

1. Sample holder

The sample holder consists of two $45 \times 20 \times 20 \text{ mm}^3$ machined blocks of copper. From each block protrude two spring-loaded PTFE-sheathed type T thermocouples (RSPro, 0.25 mm/1 mm wire/junction diameter) with exposed junctions and a rated accuracy of ± 0.5 °C. To thermally anchor the thermocouples, they are passed through the entire height of the copper block. The distance between blocks can be adjusted, but is normally fixed such that the thermocouples form a square with a lateral length of 10 mm, on which the sample is placed face down. The copper blocks are insulated from the sample with Kapton tape, and the sample is fixed with copper clamps, as sketched in Fig. 2. Besides the silver paste used to contact the sample corners, no additional thermal grease is used.

2. Heater

One copper block is fitted with a 30 W cartridge heater (RSPro, 40 mm long, \emptyset 6.5 mm). The heater is powered by 230 V mains voltage and controlled by a solid state relay (SSR). The SSR is switched on and off 10 times/s, and the ratio of on- to off-time is controlled by pulse-width modulation (PWM), using the in-built functionality of the Raspberry Pi. As plotted in Fig. 4, one of the thermocouples is continuously polled to give feedback on the sample temperature. Power is adjusted as needed to maintain a constant temperature, using a proportional–integral–derivative (PID) controller implemented in software. During measurements, the sample holder is covered, in order to minimize the effect of any air draft, which might otherwise increase the time necessary for the temperature to stabilize.



3. Relay board

The connections between any of the 2 + 2 SourceMeter current and voltage terminals and the 4 × 2 thermocouple leads are established using a custom-made relay board. Switching is controlled via the 3.3 V GPIO pins of the Raspberry Pi, which are shifted up to 5 V using a SN74HCT245. Four cascaded TPIC6B595 shift registers are used, to control all of the 4 × 4 5 V DPDT latching relays (KEMET EA2-5TNJ). The relays are powered using an external 5 V power source. Besides that, the relay board houses a DS18B20 digital thermometer with a rated accuracy of ±0.5 °C, which is located close to the thermocouple connectors on the PCB. Using the 1-wire protocol, the temperature of the PCB is measured and serves as the reference junction temperature of the thermocouples.

4. Raspberry Pi

The measurements are controlled by a Raspberry Pi 3B single board computer. Using an RPi instead of a regular PC has several advantages. First, it is quite simply cheaper. Second, the RPi provides improved connectivity for no additional cost. The general purpose input output (GPIO) pins are useful to interface with all types of electronics. Here, we use them to switch connections between the thermocouples and the SourceMeter as needed, to read the reference temperature of the thermocouples, and to maintain a stable hot-side temperature during the Seebeck measurement. Besides that, the RPi is connected to the SourceMeter via the general purpose interface bus (GPIB) using a "National Instruments GBIP-USB-HS" universal serial bus (USB) to GPIB adapter.

5. Software

The RPi runs the Raspbian operating system, which does not support GPIB out of the box. However, with the rpi-source and linux-gpib packages, support can be added easily enough, allowing us to use an adapter like the GBIP-USB-HS. A Python script is used to control the experiment. In particular, the script takes care of

- sending commands to and receiving data from the SourceMeter via GPIB, using the PyVISA and PyVISA-py Python packages and
- maintaining a stable hot-side temperature using a PID controller implemented in software.

III. VALIDATION

We validated the setup by measuring several types of samples.

- Electrical conductivity measurements were compared to commercial equipment.
- Measurements of the Seebeck coefficient were verified using three different metal references: copper, nickel, and constantan.
- Finally, we used the setup to measure the properties of anisotropic samples of rubbed PBTTT.^{13,14}

A. Isotropic reference samples

1. Electrical conductivity

Measurements of isotropic electrical conductivity were crosschecked with measurements of the same samples, using a commercial Ecopia HMS 5000 Hall measurement system, which is also based on the van der Pauw method.⁵ Results agreed to within 2% for resistances ranging from a few Ω to hundreds of k Ω (data not shown).

2. Seebeck coefficient

Figure 5(a) shows literature values for the Seebeck coefficients of copper,¹¹ nickel,^{11,15–19} and constantan^{12,16,20} vs temperature taken from the literature. These same values were also used to correct for the contribution of the thermocouple, leading to the measured Seebeck coefficient. The functional dependence of $S_{\rm Cu}(T)$ was taken from a study by Burkov *et al.*,¹¹ while $S_{\rm Co}(T) = S_{\rm Cu}(T) - \frac{\partial V_{\rm Sppe}T(T)}{\partial T}$ was calculated from the temperature dependence of type T thermocouples (which are made from copper and constantan) published by NIST,¹² corrected for the copper lead. Figures 5(b)–5(d) show results on metal references of copper, nickel, and constantan, respectively. Each plot contains four sets of data. One each measured with copper or constantan leads, and one determined from $\frac{V_{\rm Co}}{V_{\rm Cu}}$ (or its inverse), as well as the average of all three

methods. As can be seen from Fig. 5, all measurements agree well with the values given in the literature, with minor deviations of the order of $\pm 1 \mu V K^{-1}$.

We are quite content with the results, given that the setup was not initially designed to measure the temperature dependence of S, since the applied temperature differences ΔT are comparatively large, and the average sample temperature is not separately controlled. Instead, the values of S(T) reported in Fig. 5 were obtained by gradually increasing ΔT . For example, the initial data point corresponds to a measurement with a controlled hot side temperature of $T_{\text{hot}} = \{33, 34, 35, 36, \text{ and } 37\}^{\circ}C$ and a resulting (uncontrolled) cold side temperature of $T_{cold} = [23...24]^{\circ}C$. The resulting value of S was then plotted vs the averaged sample temperature; in this case, $T_{\text{avg}} = 29.25 \,^{\circ}$ C. For each subsequent data point, T_{hot} was increased by 5 K, and $T_{\rm avg}$ increased accordingly by about 3 K. While this ad hoc method of varying ΔT may be used to estimate the general temperature dependence of S(T), it is limited to temperatures close to room temperature and cannot compete with the accuracy of more complex devices that control T_{avg} and ΔT separately.



FIG. 5. Reference Seebeck measurements: (a) literature values for S of copper, nickel, and constantan. Measured Seebeck coefficient of (b) copper, (c) nickel, and (d) constantan determined using the copper thermocouple leads, using the constantan leads, or from $\frac{\partial V_{Cu}}{\partial V_{Co}}$, as well as the average of the three methods.

Finally, we use the developed setup to measure the thermoelectric properties of anisotropic samples.

1. Rubbed PBTTT

When heated above their glass transition temperature, polymer chains can be aligned along a preferential direction. In one particularly successful method, the film is rubbed with a velvet cloth.^{13,14} Orientation is induced at the surface and progressively propagates throughout the thickness of the film. This was shown to lead to impressive increases in electrical conductivity and the Seebeck coefficient along the rubbing direction.¹⁴

Here, both rubbing¹³ and doping²¹ of the polymer films was performed according to methods reported previously for PBTTT. Thin films were blade coated from 20 mg/ml solutions of PBTTT in chlorobenzene, using a speed of 30 mm/s and a temperature of 110 °C. Then, the polymer films were heated on a hotplate at 180 °C and rubbed with a thin polytetrafluoroethylene (PTFE) membrane attached to a steel blade using a speed of approximately 1 cm/s. Some of the rubbed polymer films were kept at 180 °C for 15 min and then allowed to cool down to room temperature. In other samples, the degree of orientation



FIG. 6. Oriented films of PBTTT: (a) absorbance and (b) the Seebeck coefficient vs electrical conductivity for films of rubbed PBTTT for two different degrees of anisotropy and three different degrees of doping. Dark and light colors correspond to properties measured perpendicular and parallel to the rubbing direction. For $\lambda > 800$ nm, the polarizer starts to absorb and distort the measurement.

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	$\sigma(\Omega^{-1}\mathrm{cm}^{-1})$	$S(\mu VK^{-1})$	$\overline{\sigma(\Omega^{-1}\mathrm{cm}^{-1})}$	$S(\mu V K^{-1})$
	21	19.4(6)	280	28.9(4)
180°	15	23.6(3)	210	32.3(5)
	1.1	41.4(1)	16	50.0(4)
270°	230	28.1(2)	380	29.0(3)
	210	32.1(4)	350	29.3(6)
	97	43.6(1)	180	41.7(3)

was reduced by heating them above the melting temperature, i.e., to 270 °C. In order to avoid possible polymer degradation, both rubbing and annealing processes were carried out under a nitrogen atmosphere. For doping, polymer films were immersed for 10 min at 40 °C in a co-dissolution of 0.3 wt/wt. % 2,3,5,6-tetrafluoro-7,7,8,8-tetracyanoquinodimethane (F4TCNQ) and 3 wt/wt. % bis(trifluoromethane)sulfonimide lithium salt (Li-TFSI), using ethyl acetate as a solvent.

Figure 6(a) shows the absorbance of two representative samples annealed at 180 °C and 270 °C, while Fig. 6(b) and Table I summarize the thermoelectric measurements on samples at different doping levels. Similar to what was reported by Vijayakumar *et al.*,¹⁴ we observe significant optical anisotropy as well as $\sigma_{\parallel} > \sigma_{\perp}$ and $S_{\parallel} > S_{\perp}$ for the highly oriented samples, which means that the thermoelectric power factor $S^2 \sigma$ parallel to the rubbing direction is superior to that in the perpendicular direction. In our case, $\sigma_{\parallel} \approx 13 \times \sigma_{\perp}$ while $S_{\parallel} \approx S_{\perp} + 9 \,\mu$ V K⁻¹. The other samples have only a little residual orientation, as evidenced by the relatively small change in absorbance, $\sigma_{\parallel} \approx 1.5 \times \sigma_{\perp}$, and no significant difference in the Seebeck coefficient. The somewhat unexpected fact that the maximum electrical conductivities of both kinds of samples are similar hints at a difference in dopant uptake and will be the topic of further research.

IV. CONCLUSIONS

In summary, we presented a cheap, custom setup to measure the components of in-plane electrical conductivity and the Seebeck coefficient of anisotropic thin films. Because a single sample is sufficient to perform all measurements, errors associated with sample variability can be avoided. We validate the setup by measuring known metal reference samples and demonstrate its usefulness by characterizing anisotropic films of oriented PBTTT. Further in-depth work on films of rubbed PBTTT and uniaxially aligned carbon nanotubes are forthcoming.

AUTHORS' CONTRIBUTIONS

B.D. contributed toward the conceptualization, software, investigation, and writing of the original draft; O.Z.-A. contributed toward investigation, review, and editing; and M.C.-Q. contributed toward supervision, review, and editing.

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DATA AVAILABILITY

The data that support the findings of this study, as well as the Python script used to control the setup, are available from the corresponding author upon reasonable request.

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