

Observing the Hall Effect, Part II: Germanium (Electrical Transport Option) http://education.gdusa.com/experiments.html

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Description: The objective of this educational module is to measure the Hall voltage V_H to determine the Hall coefficient R_H of germanium and determine its carrier concentration (and type) as a function of temperature. It is suggested to perform this module after completing Observing the Hall Effect, Part I: Copper.

Background:

The Hall effect (HE)¹, arises from the sideways deflection of charges in a conductor upon application of a magnetic field. This effect was discovered in 1879 by Edwin Hall and, as described below, provides a method to determine the concentration (*n*) and sign (e.g. electrons or holes) of the charge carriers. The HE is now used extensively in characterizing metals, semiconductors, and ferromagnets². V_H is sufficiently large in semiconductors enabling the development of Hall probes, which are used as compact inexpensive magnetometers.

Germanium is an indirect bandgap semiconductor with a room temperature bandgap $E_G = 0.67 \text{ eV}$. As discussed in the module on Hall effect measurements in Cu, the Hall coefficient R_H is given by the following equation:

$$R_H = V_H t / IB = 1 / ne \tag{1}$$

Recall that when electrons are the charge carriers, R_H is negative and when holes are the charge carriers, R_H is positive. Typical carriers densities in semiconductors are orders of magnitude lower than in metals (typical values range from ~10¹⁴ to 10¹⁸ cm⁻³). Thus, R_H can be orders of magnitude larger in semiconductors in comparison to metals. The correspondingly large Hall voltage ($V_H = R_H IB/t$) enables semiconductors to be used as low-cost magnetic field sensors with numerous applications³. More generally, HE measurements provide a powerful means to characterize semiconductors, an important task given the ubiquity of semiconductor devices in virtually every aspect of modern technology. Hall voltage measurements, in combination with resistivity measurements (with certain caveats – see below), suffice to determine both the carrier density and mobility. The mobility (typically denoted μ) characterizes the drift velocity, that is $v = \mu E$ where v is the drift velocity and E is the applied electric field. Mobility values are typically presented in terms of cm²/Vs. For example in GaAs, the electron mobility at room temperature is ~8500 cm²/Vs, whereas for holes the mobility is ~400 cm²/Vs. In Ge, the numbers are ~3900 and 1900 cm²/Vs for electrons and holes, respectively⁴. The larger the mobility the greater the conductivity since $\sigma = ne\mu$, along with a correspondingly smaller resistivity:

 $\rho = 1/\sigma = 1/\operatorname{ne}\mu \tag{2}$

A glance at Eqns. (1) and (2) shows that determining R_H and ρ allows for the determination of n and μ .

These equations are a simplification, and would be appropriate for a material where either electrons or holes are the dominant carriers. In semiconductors, both electrons and holes can simultaneously contribute to the transport, and they must be treated, initially at least, on equal footing. A basic understanding of this can be obtained from considering Figure 1. The defining characteristic of semiconductors is the existence of a nonzero bandgap with typical values on the order of 1 eV. For an intrinsic semiconductor as depicted in Fig. 1(a) (i.e. one without intentional doping), this leads to a small carrier density that is determined via thermal activation across the gap. Intuitively, the larger the gap, the smaller the carrier density at a given temperature. For intrinsic semiconductors, for each electron thermally excited across the gap into the conduction band, there is a corresponding hole in the valence band, both of which contribute to the transport properties of the material – this is often termed ambipolar transport. Consideration of electrons and holes to the transport leads to the following expression for the Hall voltage:

$$V_{H} = \frac{p\mu_{h}^{2} - n\mu_{e}^{2}}{e(p\mu_{h} - n\mu_{e})^{2}} = \frac{p - n\theta^{2}}{e(p + n\theta)^{2}}$$
(3)

In this equation, p(n) is the hole (electron) carrier density, $\mu_p(\mu_e)$ the corresponding hole (electron) mobility, and $\theta = \mu_e/\mu_h$. Eqn. (3) highlights that the situation is more complicated for ambipolar transport⁵. However, quite often semiconductors are doped with atoms to create extrinsic semiconductors. The addition of donors (e.g. P or As in Ge, see Fig. 1 (b)) adds additional electrons to the conduction band at temperatures sufficient to ionize the donor impurities. Similarly, the addition of acceptors (e.g. B or Al in Ge, see Fig. 1(c)), adds additional holes to the valence band at temperatures sufficient to ionize the donor to ionize the

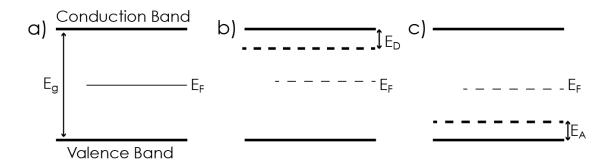


Figure 1: schematic of semiconductor with energy gap E_G . a) For an intrinsic semiconductor (i.e. undoped), the Fermi energy E_F (more properly, the chemical potential) is at $\frac{1}{2}E_G$. b) The addition of donors with binding energy E_D increases the conduction band carrier density (electrons) while doping with acceptors with binding energy E_A , increases the valence band carrier density (holes). Doping leads to a shift of E_F away from midgap.

acceptor impurities. If the dopant density is sufficiently larger that the intrinsic carrier density then Eqn. 3 simplifies. For example, if the acceptor dopant density is such that $p >> n\theta^2$, then Eqn. 3 simplifies to the conventional form (i.e. Eqn. 1, note however, we now have separate symbols – p for holes and n for electrons).

It is important to emphasize that temperature plays a key role because of the thermal excitation of carriers across the gap and because of donor thermal ionization. A given doped semiconductor could exhibit extrinsic behavior at room temperature with a crossover to intrinsic behavior with decreasing temperature because of dopant freeze-out (i.e. below the temperature where there is appreciable dopant ionization). A crossover to intrinsic behavior could also occur at higher temperatures because of thermal excitation of carriers across the gap in excess of the dopant density. Temperature dependent HE measurements enable the determination of this behavior. Excellent discussions of the statistical mechanics of carrier density in intrinsic and extrinsic semiconductors can be found in the notes⁶.

Versalab can be used to measure the Hall effect and the resistivity. However, in this module, we will focus on Hall measurements of p-doped Ge as a function

of temperature. It may be useful to go back and review the Cu Hall Effect module as it contains complementary information.

Notes:

1. See, for example, Chapter 6 of Introduction to Solid State Physics, 7th edition, C. Kittel, Wiley and Sons, New York 1996.

2. http://en.wikipedia.org/wiki/Hall_effect

3. Hall-Effect Sensors: Theory and Application, E. Ramsden, Elsevier, Amsterdam 2006.

4. An excellent online source for the properties of numerous semiconductors can be found at <u>http://www.ioffe.ru/SVA/NSM/Semicond/</u>

5. It is worth mentioning at this point that a more complete analysis of the Hall Effect must treat the statistical nature of the carrier velocity, leading to prefactor in Eqn. 1 or 3 that is \sim 1.

6. Chapter 8 of Introduction to Solid State Physics, 7th edition, C. Kittel, Wiley and Sons, New York 1996; Chapter 28 of Solid State Physics, N. W. Ashcroft and N.D. Mermin, W.B. Saunders, Philadelphia 1976; Chapter 1of Physics of Semiconductor Devices, S.M. Sze, Wiley & Sons, New York 1981.

Student Learning Outcomes:

- Students will learn the basic aspects of sample handling with silver paint.
- Students will learn the technical aspects of performing small signal transport measurements.
- Students will learn fundamental techniques in electronic transport characterization of materials.

Safety Information:

Before attempting to perform any parts of this student experiment, please read the entire contents of: this Educational Module, the VersaLab User's Manual (1300-001), and the Electro-Transport Option Manual (1084-700), and observe all instructions, warnings and cautions. These are provided to help you understand how to safely and properly use the equipment, perform the experiments and reach the best student learning outcomes.

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse, improper operation of the system and the information contained in this Educational Module.

The following Safety warnings apply to this Educational Module. We recommend that you study them carefully and discuss the details with your instructor before starting the work:

WARNING!



Always use Personal Protective Equipment (PPE) during every step of sample preparation. Failure to do so might cause bodily harm.





TOXIC HAZARD!

Acetone is toxic if swallowed. For more information consult the Material Safety Data Sheet available on this website:

http://www.guidechem.com/msds/110-20-3.html

Instructions:

In this section, we will provide details on sample preparation and, subsequently, performing the HE measurement using the VersaLab/ETO. We note that the approach is similar to the Cu Hall effect measurements, but we there we will take a slightly different approach if for no other reason to point out alternatives.

Several items are needed for this experiment, which includes:

- A piece of semiconductor wafer. In the present case, we are using a ptype doped Ge wafer, but other samples that you may have access will work.
- You may need a caliper to measure the semiconductor sample thickness. In our case, the sample wafer is 500 microns thick as measured by the vendor.
- Diamond scribe to assist in cleaving the semiconductor sample
- Cotton swabs and acetone to clean the semiconductor surface
- Silver paste and thin gauge wire
- Kapton tape and apiezon N grease
- Tweezers, toothpick (for spreading H grease)
- Latex or nitrile gloves for sample handling
- ETO transport puck
- Puck wiring test station and ohm meter (to test continuity of silver paste joints)

Prior to performing the HE measurement with the VersaLab/ETO, it is important to prepare the sample as detailed in the following steps:

a.) First, it will be important to obtain an appropriate sized sample that can fit within the sample puck. In our case, we had available a 2" diameter p-type Ge wafer as shown in Figure 2.

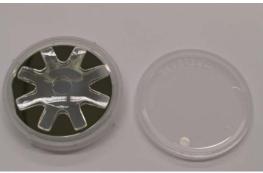


Figure 2: p-type Ge wafer

b.) Using a diamond scribe, score the wafer as shown in Figure 3.

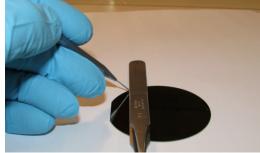


Figure 3: Score the wafer with a diamond scribe

c.) Place the wafer with the scribed line along a straight edge such as a microscope slide and press gently. The wafer should break along the scribed line as shown in Figure 4. Repeat this process along the orthogonal directions until you have a square sample. Other approaches may be more precise depending on the equipment available in your lab (e.g. wafer saw).

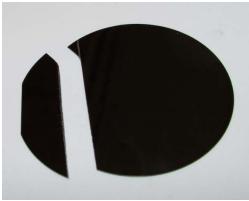


Figure 4: Cleaved Ge wafer

- d.) The sample should be cleaned with acetone prior to mounting on the wafer puck and wiring up.
- e.) For this sample we, instead of soldering we will attach the leads using silver paste (Fig. 5). A sharp tip such as a syringe will be useful to precisely locate the soldering paste and wires. For good adhesion to the wafer, we first used the diamond scribe to gently scratch the area where the silver paste will be applied. The process is quite similar to soldering as described in the Cu Hall Effect module.



Figure 5: Silver paste and syringe for sample contacts

- f.) Let the silver paste dry for ~20 minutes before handling the sample.
- g.) While the silver paste is drying, the puck can be prepared. Cut a small sample of kapton tape (see Figure 6) and apply it to the sample puck (this will serve as electrical insulation similar to the cigarette paper in the Cu HE module).

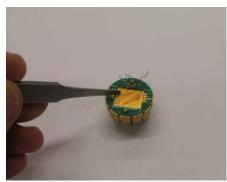


Figure 6: Kapton tape as insulation

- h.) Apply a thin layer of Apiezon N grease to the kapton. This is to adhere the sample to the puck. Importantly, N-type grease is appropriate for going to low temperatures as will be performed later in this experiment.
- i.) Place the sample on the puck and use the silver paste to connect the wires to channel 2. Refer to Figure 7 for the proper contact geometry.

Again, let the sliver paste dry for a sufficient amount of time prior to making measurements. Note: we have used silver paste down to 50K in the VersaLab without any problems and without baking the sample to drive off the volatiles from the silver paste. It appears that drying at room temperature is sufficient.

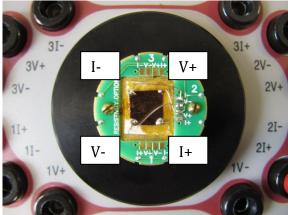


Figure 7: Sample wiring with corners labeled

j.) As shown in Figure 8, the puck wiring test station provides a convenient means to check the continuity of the soldering prior to insertion into the VersaLab instrument. In the Cu experiment, the resistance was less than 1 Ohm. This need not be the case for the semiconductor and as Fig. 8 shows, for our sample the resistance is ~0.9 k Ω .



Figure 8: Checking sample for continuity

k.) At this point the transport puck/Cu foil is ready to be inserted into the VersaLab. This is accomplished using the puck insertion tool as described in the VersaLab user's manual (Figure 1-2). Upon inserting the puck, it is important to make sure the tab on the transport puck is properly aligned. The tab should face towards the front of the VersaLab, and it is possible to feel a slight click. At this point gentle downward pressure will allow for appropriate seating of the sample puck into the sample chamber.

I.) The sample chamber can be sealed by inserting the baffle set (don't forget the o-ring) and Kwik-Flange clamp.

The rest of this experiment will utilize the MultiVu software. Please see Chapter 3 of the VersaLab manual and Chapter 4 of the ETO manual for complete details. Although this procedure is similar for the Cu foil measurement in Part I, there are some differences.

- m.) Activate the ETO option. Under the utilities tab, select activate option. Under the "Available Options" select "Electrical Transport" and then click the activate button. This will activate the instrument and pull up the ETO console.
- n.) On the ETO console, click the "Datafile" tab and choose a sample name and location for your data to be saved. There are default settings. Click "Change" to alter the name and location of the datafile.
- o.) While not required for this measurement, it is useful to know how to pump down the sample chamber. Under the "Instrument" tab, select "Chamber". This will pull up a dialog box. Under "Control" select "Purge/Seal" to initiate putting the sample under vacuum. The left hand side of the dialog box will show the status. This will reduce the sample vacuum to a few torr. To obtain high vacuum, click the "HiVac" tab.
- p.) The next step is to write a sequence to perform the required measurement. Under the "file" tab, click new sequence. This will open a new sequence file (e.g. Sequence1.seq) and the sequence command bar. Double click "Scan Field" to open another dialog box to enter the desired parameters. For example, you might choose an initial field of 10000 Oe and a final field of -10000 Oe in steps of 1000 Oe. The maximum rate that can be chosen is 300 Oe/sec. Once your selections are made, click "ok" to insert the command line into the sequence file.
- q.) Returning to the sequence command bar, click "Electical Transport" and then double click "ETO Resistance". This will pull up the "AC Resistance Measurement" box. Since we have wired up the sample on channel two, select "Enable Measurement" for Channel 2. For the excitation, choose 1mA and 21.3 Hz. Select autorange, and for the measurement configuration choose a one second averaging time and 5 for the number of measurements. Upon clicking "ok", these selections will be inserted into the sequence.
- r.) This constitutes a complete, albeit short, measurement sequence to measure the HE on copper. The sequence can be saved by selecting the "file" tab and "save as" to select a file name and location.
- s.) To initiate the measurement sequence, click the "play" button near the top of the MultiVu software interface. This will start the data acquisition with the data being save under the filename you selected in step o.) above.
- t.) At the bottom of the MultiVu software, the status of the temperature, field, and vacuum can be monitored.

- u.) To monitor the data collection in real time click the "Datafile" tab on the ETO console and select "View". This will plot the data in real time. The default is to show the time stamp on the x-axis, and to plot the resistance and phase angle of both channels one and two. To change this right click on the plot and choose "data selection" to bring up a dialog box to change the view. Change the x-axis to plot field and deselect the boxes for channel one and the phase angle of channel 2. The result is that the Hall resistance will be plotted as a function of the field. Right click on the plot and choose "autoscale" to see the complete data set.
- v.) At the end of the measurement sequence, you should obtain a plot of the Hall resistance as a function of field.
- w.)To return to the zero field condition select the "Instrument" tab and choose "field." This will open a dialog box. Enter a setpoint of 0 Oe and click "set". This will ramp the field down to zero. After venting the sample (if under vacuum), the sample can be removed.
- x.) The measurement you performed was at room temperature. As per the discussion above, we expect the carrier concentration to change as a function of temperature. We will manually cool the sample to 150K (i.e. not using a sequence). To do this, right click in the temperature box in the lower left corner of MultiVu. This will pull up the dialog box. Enter 150K, with a cooling rate of 10K/min and push set. Wait for the temperature to stabilize.
- y.) Repeat the measurement at this temperature. This process could be repeated at several temperatures. It is also possible to write a sequence to fully automate this process.
- z.) Finally, when you are finished set the temperature back to 300K and reduce the field to 0 Oe.

Questions / Analysis:

Some of the following questions are specific to the data that was obtained, while others are of a more open-ended or comparative nature.

1. From your data, determine the Hall coefficient R_H for germanium. Are the carriers electrons or holes? Determine the carrier density of your sample. Make certain to include the error bars for your data, along with a brief description of your error analysis.

2. In semiconductors, it can be a challenge to make a good ohmic contact. Instead, a Schottky barrier can result. Investigate and discuss the origin of Schottky barriers. Identify some approaches that have been used to eliminate Schottky barriers and achieve a good ohmic contact. How would you use the VersaLab to determine if you have an ohmic or Schottky contact? Do you expect having a non-ohmic contact to strongly affect the Hall measurement results?

3. Measure the resistivity of your sample and use this, along with the Hall measurement, to obtain and estimate of the mobility and carrier density. Discuss

any approximations that you make. Look in the literature to determine the most accurate techniques available for such measurements (note: you need not implement these more accurate techniques).

4. Write a sequence to automate the Hall Effect measurements as a function of temperature. Measure your sample at ten or more temperatures and use this data to make a plot of the carrier density versus temperature. Qualitatively describe the temperature dependence of your sample. Is there evidence for carrier freeze out (i.e. do you go to temperatures sufficiently low that you don't ionize the impurities?) Some of these questions can be very sample dependent so you may need to think carefully about your results with regards to the room temperature dopant density, etc.

5. Is there a difference in slope between the 300K data and 150K data? That is, is this data in line with your intuitive expectations for what should occur upon cooling?

6. Either derive or find a derivation of Equation 3 for the Hall voltage when electron and holes both contribute to the transport.