Experiment SS-X
Conductance Quantization in Gold Nanowires

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Abstract

When a conductor is reduced to a size comparable to the Fermi wavelength of its electrons, the transport of these electrons will be ballistic. This implies a fundamental resistance for such conductors dependent only on fundamental constants. By studying the resistance of nanowires formed when two gold wires break contact, one can make a precision measurement of the value of this macroscopic, yet fundamentally quantum, resistance.

Objectives

1. Build and calibrate a current-to-voltage amplifier circuit that can make fast, low-noise measurements of μA currents.

2. Observe the quantization of nanowire conductance in oscilloscope traces as two gold wires freely vibrate against one another.

3. Use a piezoelectric actuator and a digital storage oscilloscope interfaced with a personal computer to automatically record approximately 1000 traces of current versus time.

4. Characterize and compensate for nonlinearities in an analog-to-digital converter.

5. Analyze data to determine the value of the fundamental unit of conductance G_0, the extent of exact integer quantization in gold nanowires, the resistance of the nanowire leads, the average nanowire lifetimes, and the relative abundances of nanowire atomic structures.

References

[1] J. L. Costa-Krämer, N. García, P. García-Mochales, and P. A. Serena. Nanowire formation in macroscopic metallic contacts: quantum mechanical conductance tapping a table top. *Surface Science*, 342(1-3):L1144 – L1149, 1995.

[2] K. Hansen, E. Lægsgaard, I. Stensgaard, and F. Besenbacher. Quantized conductance in relays. *Phys. Rev. B*, 56(4):2208–2220, Jul 1997.

[3] Henk van Houten and Carlo Beenakker. Quantum point contacts. *Physics Today*, 49(7):22–27, 1996.

1   Introduction

At the macroscale, conductivity is affected by the frequency of atomic defects in materials and thermal dislocations of lattice positions that scatter electrons. On the other hand, electrons in a very short conductor will travel ballisticly along its entire length. In this regime, resistance primarily arises from scattering off of the boundaries of the conductor geometry. If the conductor width is reduced down to the scale of the Fermi wavelength, then quantum-mechanical effects become important. So-called eigenchannels arise which each have a fixed conductance G_0, given by

$$G_0 = \frac{2e^2}{h} \approx \frac{1}{12.9\,\text{k}\Omega}.$$

The number of eigenchannels determines the total resistance of the nanowires. This means that the total conductance of a nanowire is quantized in increments of G_0.

A variety of techniques have been used to generate nanowires with quantum conductance including the mechanically controllable break junction technique (where a thin wire is broken by controlled bending), STM tips contacting surfaces, and holes burned into thin metal films with electron beams. While these techniques can be used to create long-lived, controlled nanowires, two other methods offer extremely simple ways for creating nanowires when the appropriate contact material is used. You will use both of these techniques in this lab: 1) gently tapping two gold wires and 2) using a piezoelectric actuator to slowly move two gold contacts towards and away form each other.

These two techniques rely on the two pieces of gold fusing when they touch and then breaking gradually as they are pulled apart so that, for a brief period, wires several atoms wide exist. Gold is the best metal for using this method without a controlled atmosphere because it is highly non-reactive and maintains an extremely pure surface. Also, its ductility may improve its ability to gradually break and sustain longer-lived nanowires.

Note: This experiment assumes some familiarity with computer-based data analysis software such as MATLAB or GNU R [6]. While none of the required techniques are particularly complicated, you should be prepared to teach yourself how to use this software, as much of the analysis cannot be accomplished with spreadsheet programs like Microsoft Excel. This manual will outline the analysis procedures and provide source code for several utility functions, but it will not walk you through the basic usage of such software, as quality tutorials and reference manuals already exist for that purpose. In short, either you or a friend should be more comfortable using computers than the average student before attempting this lab.

2   Equipment Inventory

- Apparatus containing piezoelectric actuator, gold wires with copper leads, and a moving stage controlled by a microthreaded screw

- PC-based digital storage oscilloscope (Link Instruments DSO-8502 or equivalent)

- PC with DSO software (wdso 8000 or equivalent)

- Digital oscilloscope (Tektronix or comparable)

- Function generator (Wavetek Model 180 or equivalent)

- Power supplies (two Heathkit Models IP-18 or equivalents)

- Solderless breadboard (Digi Designer DD-1 or comparable)

- Multimeter

- BNC voltage probe

- BNC cables

- BNC/alligator clip adapter cable

- Gold wires soldered to long, stiff copper leads

- Operational amplifier (LT1007, 411CN, or equivalent)

- Resistors

- Capacitors

- Shielded copper wires

3   Procedure

After verifying your inventory, you will need to construct an amplifier circuit for visualizing fast, small currents on an oscilloscope. Once you have observed evidence of quantized conductance using rudimentary wire tapping, you will employ a piezoelectric actuator to control repeated nanowire formation, recording traces of conductance vs. time using a digital storage oscilloscope (DSO). Performing statistical analysis on these traces will yield a precision measurement of G_0.

3.1   Circuit Assembly

Build the current-to-voltage amplifier circuit described in Figure 1 using the solderless breadboard and circuit components provided with the lab setup. Ignore the "Gold Wires" (R_G) module for now, but mark the corresponding contact points on the breadboard.

The circuit consists of three basic modules: a voltage divider, a current-to-voltage amplifier, and a low-pass filter. The operational amplifier (op-amp) accepts maximum rail voltages of ± 22 V; using rails of ± 10 V should be sufficient. The voltage divider must drop the negative rail voltage to approximately -20 mV. Under these conditions, and with an amplifier gain of approximately 100 kV/A, we would expect an output signal of approximately 150 mV given a load of $1/G_0$. This should be easily visible on an oscilloscope. Finally, the low-pass filter should be chosen to eliminate noise with frequencies above 100 kHz. See the *AEP 1200 Lab Manual* [4] for details regarding resistors, capacitors, filters, and op-amps.

One viable set of components that would achieve these goals is:

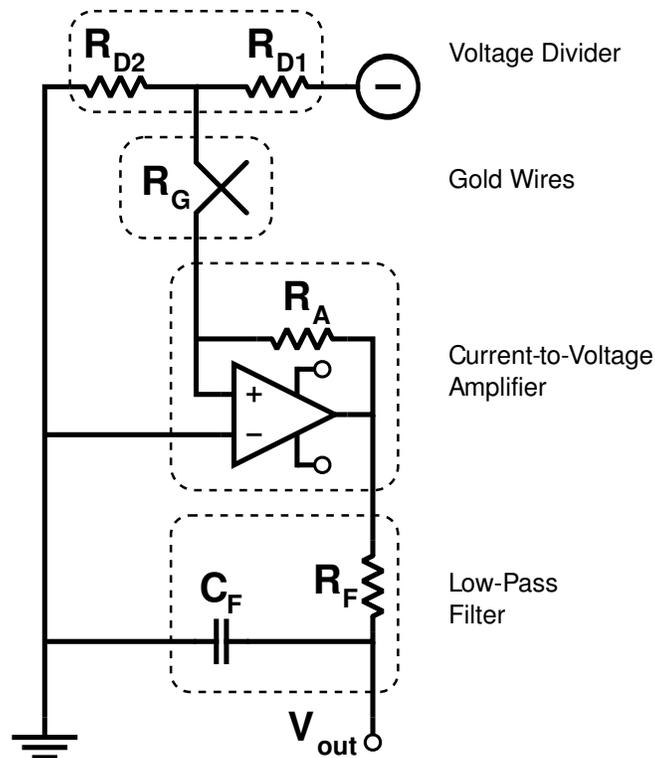- Voltage divider resistor 1: $R_{D1} = 22$ kΩ

Figure 1: Schematic for a current-to-voltage amplifier circuit.

- Voltage divider resistor 2: $R_{D2} = 51\,\Omega$

- Amplifier gain resistor: $R_A = 92\,\text{k}\Omega$

- Low-pass filter resistor: $R_F = 1\,\text{k}\Omega$

- Low-pass filter capacitor: $C_F = 10\,\text{nF}$

- LT1007 operational amplifier with $\pm 10\,\text{V}$ rails

The rail voltages are provided by two power supplies of opposite polarity attached to a common ground, as shown in Figure 2. The current output may need to be raised slightly above the minimum to achieve stability. Be sure not to accidentally ground both outputs of a power supply unit, as doing so may fry the internal transistor.

Connect the digital oscilloscope to the amplifier circuit using a BNC test probe (set to 1X attenuation). Attach the alligator clip to the circiut's ground plane and connect the probe tip to V_{out}. Configure the oscilloscope to use DC coupling for this channel.

3.2 Testing and Calibration

Use a multimeter to measure the actual resistances and capacitances of each component, as well as the voltage output from the power supplies. Using these measurements, predict the
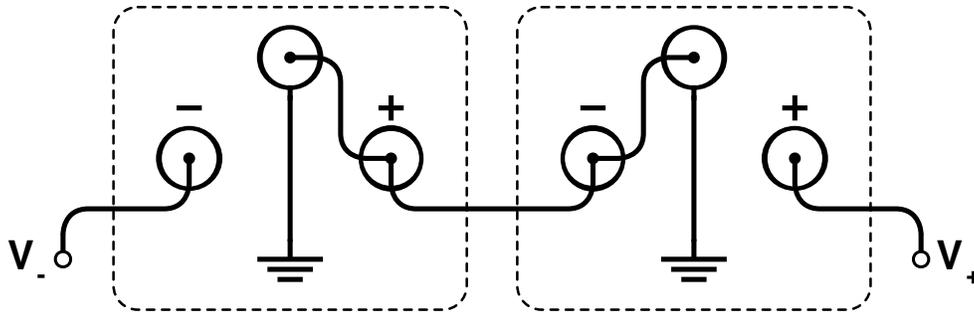
Figure 2: Establishing a common ground for two regulated power supplies of opposite polarity.

quantitative behavior of each of the circuit modules. Test each module in isolation to confirm your measurements and calculations.

While the measured properties of the individual circuit components are sufficient for computing a calibration relation between V_{out} and R_G, you can obtain an independent calibration curve by measuring V_{out} for various known resistances R_G. Loading the circuit with $\approx 10\,\text{k}\Omega$ resistors in place of the "Gold Wires" module will produce calibration points in the vicinity of the expected value of G_0. Perform a linear fit to your measurements to obtain a second calibration relation, and compare it with the relation obtained from circuit theory and the measurements of each component. (When performing the fit, remember that the output is linear with respect to conductance, not resistance.)

3.3 Qualitative Observation

With the circiut calibrated, insert two long, stiff wires with gold tips into the contacts reserved for the "Gold Wires" module. Ensure that the gold tips are clean and that the solder joints are sturdy. When the two wires are put into contact, the resistance of that junction is denoted by R_G. This resistance is effectively zero when the wires are in full contact, but if the junction is broken slowly, there will be a stage during the breaking process in which only a few gold nanowires span the gap, raising R_G into the kΩ range. The current measured by the circuit will be quantized, falling off in steps as each individual nanowire is broken. Therefore, a "stair-step" signal on the oscilloscope will provide evidence of the quantization of conductance.

Because this signal will last less than 1 ms and is not periodic, you must carefully configure the oscilloscope trigger to catch these breaking events. Note the "Run/Stop" button on the oscilloscope; upon catching a quality trace, press this button to halt further collection, allowing you to perform basic measurements on the signal.

Using your calibration curve, compute the expected output voltage given a load of $R_G = 1/G_0 = 12.9\,\text{k}\Omega$. Set the digital oscilloscope to trigger on the falling edge at a value approximately 20% lower. For optimal viewing, your vertical scale should be set to 200 mV/div, and your horizontal scale should be set to 50 µs/div. The trigger should be offset in time so that approximately 400 µs are visible prior to the triggering event. Finally, when performing the experiment, the trigger mode should be set to "Normal."

Now it is time to make nanowires. This part of the lab can be very difficult and requires both patience and practice. The method listed here is intended to help you achieve good data

as quickly as possible, but if you run into difficulty using this method, then modifications and improvements are encouraged.

The gold wires should be soldered to the ends of long copper wires that are stuck into the breadboard. Bend the wires so that they form an X in a plane parallel to the table. The intersection point should be somewhere near the middle of the gold portion of the wires. At the point of intersection the wires should be touching very lightly so that by banging the table, the wires bounce in and out of contact.

Hit the table with your fist or tap the copper portion of one wire with a pencil eraser. (Don't touch the copper wires with your hands because your body acts like an antenna and introduces a lot of unwanted noise.) The oscilloscope signal should jump from 0V to ~ 10V as the wires bounce in and out of contact. With the trigger set to normal, the image of the last falling edge should remain on the oscilloscope screen. You should be looking for a decreasing step function with a step size of approximately 150mV, similar to that shown in Figure 3. If you see a good image on the scope that you would like keep then hit the "Run/Stop" button to stop the scope from replacing the image with another trace. With practice you will get good at timing and capturing images with the run/stop button. While the scope is stopped, you can use the cursors to measure the height and duration of the steps on the screen. To continue taking more traces, just hit the "Run/Stop" button again. See §4.1.1 for additional tips if you have trouble finding these signature traces.
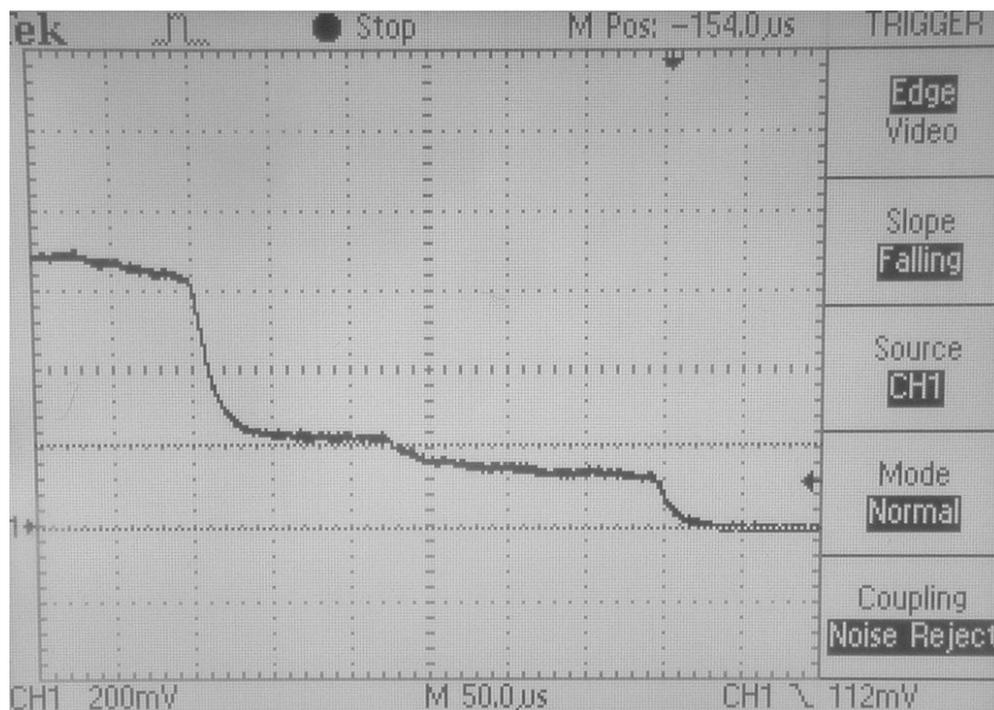

Figure 3: Example of stepped trace indicating quantization.

3.4 Automated Apparatus Assembly

To consistently generate quality traces, you will use an apparatus consisting of a piezoelectric actuator and a traveling stage controlled by a microthreaded screw. Four connections must be made to use the apparatus: the two wires associated with the actuator must be connected to the output of the signal generator, while the two leads to the gold wires are the input and output of the "Gold Wires" module in the circuit. The connection to the signal generator should be made using a BNC adapter cable with alligator clips at one end. The gold wire leads may be inserted directly into the appropriate slots in the breadboard (it does not matter which is input and which is output).

The signal generator should drive the actuator with a triangle wave with an amplitude in the vicinity of $1.5\,$V at a frequency of approximately $0.5\,$Hz. This allows the gold wires to be moved very slowly with respect to one another (creating longer, more stable nanowires) while still allowing sufficiently many traces to be gathered in a reasonable amount of time. The DC offset of the signal generator provides a finer control of the mean separation between the two gold wires when compared to moving the micrometer stage, though the latter can be made more precise by using an Alan wrench.

You may want to split the output of the signal generator and send a copy of it to either the oscilloscope or the DSO. This allows easy amplitude, frequency, and offset measurements that enable repeatability (since the signal generator knobs are not calibrated). When viewed simultaneously with V_{out}, a well-adjusted, zoomed-out trace will resemble Figure 4.
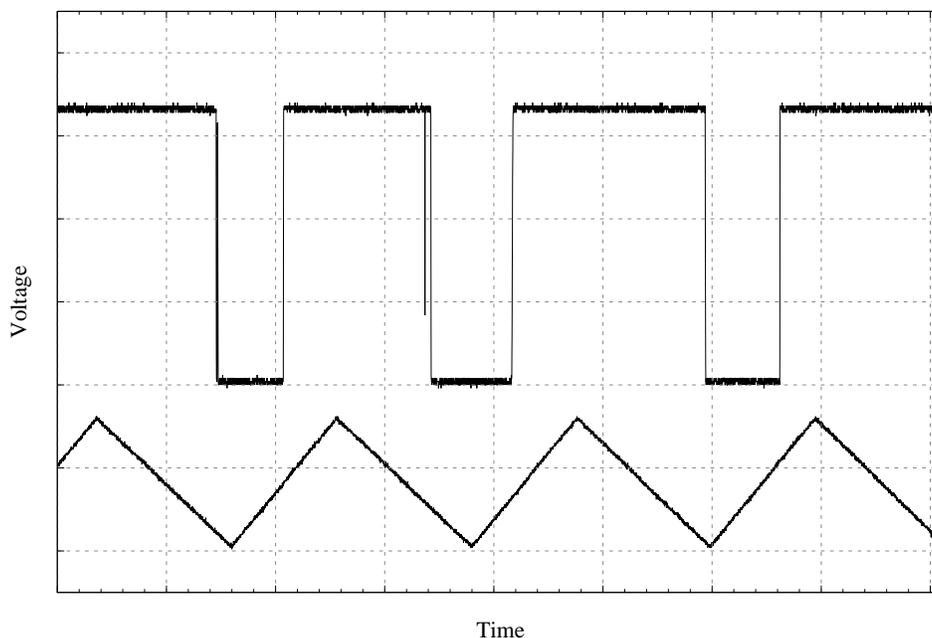

Figure 4: Contact breaking in-sync with actuator driver.

3.5   Quantitative Observation

The voltage probe should now be attached to channel A1 of the DSO, which is connected to the PC via a USB link. Start the DSO software and become comfortable with its interface (the function generator provides a good test signal).

The oscilloscope can record traces at four different relative resolutions. These are known as "Memory size" and can be selected under the "Timebase" menu. The lowest resolution ("8K") is sufficient for this experiment, though a size of "32K" works as well. The higher resolutions will limit your collection rate and produce too much data to conveniently manage.

Once you have chosen your "Memory size," select a sampling rate that will provide a total time/acquisition between 0.5 ms and 1 ms. Recommended values are "10 MSa (100 ns)" for the 8K memory size and "100 MSa (10 ns)" for the 32K memory size. You may want to zoom out when trying to acheive a stable signal, however.

The oscilloscope view, controlled by the buttons on the left edge of the screen, should be set similarly to the digital oscilloscope used when making the qualitative observations. As the vertical range is larger, you may want to use a setting of 100 mV/div and an offset of -400 mV. The coupling should remain DC, and the attenuation should be set to "1X," as on the probe. Horizontally, the view should be zoomed out so that the entire trace is visible. Using the recommended resolution settings above, this corresponds to a scale of approximately 50 µs/div, just as with the digital oscilloscope. As the horizontal range is larger, you will see a greater portion of each trace, compensating for the hopefully longer nanowire lifetimes achievable with this setup.

Similarly, set the trigger similarly to how it was set for the digital oscilloscope. It should be offset in time by at least 450 µs, leaving at least 3/4 of the trace prior to the trigger. Unfortunately, the oscilloscope cannot reliably distinguish between a rising and a falling edge, so as many as half of the triggers will be spirrious.

By adjusting the microthreaded screw and the function generator's DC offset and amplitude, you should find a configuration where the two gold wires go in and out of contact with each cycle of the driving signal, as shown in Figure 4. This will require a lot of patience, as the two gold wires may be resistant to fusing or breaking. In general, quick, sharp adjustments of each of the parameters, with occasional deliberate overshoots to shake things loose, are more successful than slow, smooth adjustments. Set the trigger to "Auto" mode while performing these adjustments so you can immediately know whether or not the wires are in contact and thus know which direction to make the adjustments in. Even when you have achieved a stable signal, you may need to smoothly readjust the DC offset over time to maintain it while gathering hundreds of traces.

Once you have a stable signal, you will want to save a collection of its traces. This features of the DSO software is called "datalogging." Select "File," then "Datalog settings" to open up the configuration dialog. Select the "Data(.CSB)" format and provide a file name prefix unique to each data run. Reset the counter before each new run as well. Click "OK" to close the dialog, then, with the trigger in Normal mode, select "File," "Datalog" to begin recording traces. If you need to make any adjustments, pause data logging by selecting "File," "Datalog" again. These files will be saved in the DSO software's directory on the C: drive.

Once you have logged sufficiently many traces (perhaps around 1000), you will want to generate a histogram of all of their voltages. Peaks in this histogram should be equally spaced and should correspond to integer multiples of G_0. Note, however, that the data has already been
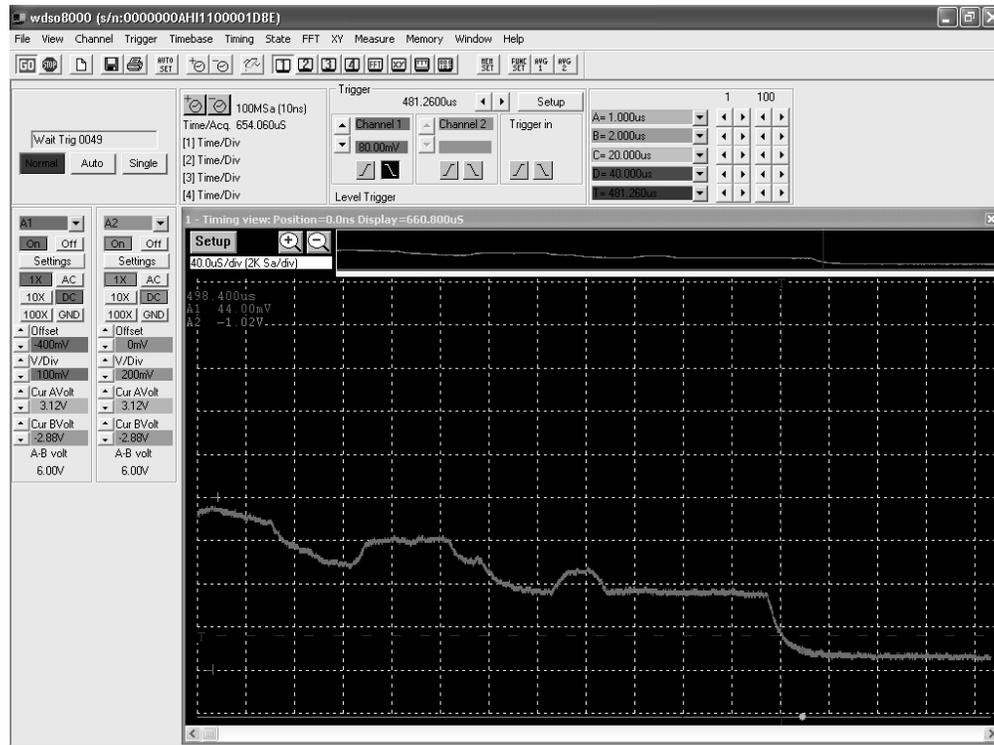
Figure 5: Screenshot of DSO software.

binned by the analog-to-digital converter (ADC). Any rebinning must coincide with the original bin boundaries for the histogram to be meaningful. As the ADC has only 256 bins, it is reasonable to use these as the histogram divisions without performing any rebinning at all.

Not all traces will correspond to slowly breaking nanowires. You will want to remove these spurious traces somehow, preferably with an automated algorithm. One option, implemented in Listing 1, is to perform a linear fit to each trace and require that it have a sufficiently negative slope. This eliminates flat signals as well as triggers on the rising edge.

Measure the peaks in this histogram, estimate the errors in their locations, and compare with the theoretical value of G_0 and integer multiples thereof. You should be able to achieve a precision of better than 5%.

3.6   Measuring ADC Nonlinearity

The DSO employs an 8-bit analog-to-digital converter (ADC) to assign a fixed-precision value to the voltage sampled at each point in time. This effectively divides the visible voltage range into 256 bins into which the voltage at any sampling instant may fall. Ideally, these bins would all be of equal width (neglecting the two overflow bins), allowing the raw oscilloscope output to be histogrammed directly. However, the ADC is not perfectly linear, and bin widths may vary more than 20% with respect to one another. This effectively introduces a large amount of noise into voltage histograms and limits the precision to which one may determine G_0. See Hansen et al. [2] for details.

Fortunately, one can straightforwardly measure the effective bin widths of the ADC using a ramping voltage like the rising or falling edge of a triangle wave. Such a signal should ideally be sampeled equally often in each voltage bin, producing a flat histogram (again neglecting overflow bins). The extent to which any bin's histogram bar exceeds or falls short of the average height corresponds to that bin's relative width. Dividing histogram bins by this width will remove the noise introduced by ADC's nonlinearity. Figure 6 illustrates this process for a 3-bit ADC, where Δ_i is the width of voltage bin i.
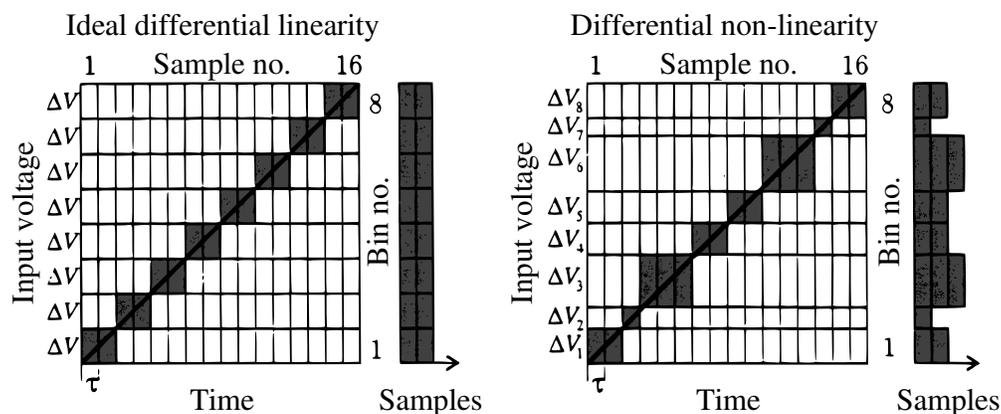

Figure 6: Example of differential nonlinearity in a 3-bit ADC (adapted from Hansen et al. [2]).

Note that the DSO uses a different ADC for each input channel, and that the differential nonlinearities may depend on the oscilloscope settings in use. Therefore, the settings used when measuring the nonlinearity should be identical to those under which the data to be adjusted was taken. It is probably most convenient to make these measurements immediately after a data run, thus producing a nonlinearity characterization for each data set acquired.

To measure the ADC nonlinearity using the ramp method, detatch the voltage probe from DSO channel A1 and attach in its place the output of the function generator. Configure the trigger to fire when the signal crosses the center of the oscilloscope view. Set the function generator to produce a triangle wave, choosing the amplitude, frequency, and DC offset so that the wave will exceed the voltage scale a small distance from both ends (that is, roughly 90% of the trace will be a clean ramp signal while the first and last 5% will be saturated). Note that the output of the function generator may not be symmetric, and since the trigger will likely fire on both the ascending and decending slopes, be sure that the signal saturates the oscilloscope in both cases.

Once you have obtained a stable ramping signal, modify the "Datalog" name, reset its counter, and collect enough traces to achieve a Poisson uncertainty of < 1% in each bin. Create a combined histogram of this dataset, and discard the lowest and highest (overflow) channels. Divide each bin height by the mean bin height. The resulting histogram is a plot of the relative width of each bin. Given a histogram of a set of data taken under the same conditions, divide each bin by its relative width to remove the noise introduced by the differential nonlinearity.

4   Notes

4.1   Finding a Stable Signal

4.1.1   Qualitative Observations

Finding evidence of quantum conductance can be very difficult and frustrating, but once you find a method for tapping wires that works, it is fairly repeatable. Here are a few hints that might help if you are just seeing noise on the oscilloscope:

- The gold wires should be touching, but barely exerting a force on each other so that they can be easily broken apart from oscillations. Push down lightly on the top wire with the eraser on your pencil and let go. You want the wires to bounce up and down together in phase so that it unclear to your naked eye whether or not the wires actually ever break contact. In reality, the wires aren't perfectly in phase and do slightly break apart at the extrema, but with a much smaller amplitude.

- You may also want to try pushing the wires so that they slide against each other as they oscillate up and down. Make sure you try different parts of the gold wires too (the tip, the middle, close to the solder point).

- Every time the wires break contact, the oscilloscope displays a new trace on the screen. When the oscillations die down and the wires are in constant contact, the scope stays fixed on the last trace. Most of the time you may find that this transition is abrupt, meaning that the scope goes rapidly from many traces per second to a single constant trace. We have had the most success when there is a more gradual transition to a constant trace. If you tap the wires in such a way that the traces become gradually less frequent to the point where you have approximately 2-3 traces per second, then you are close to the optimal tapping mode. Also keep your eye open for flat lines around 120 mV which may just be long single steps. If you see a lot of these, then zoom out the time axis to encompass more steps that could be getting cut off. You can also expand your view by adjusting the vertical and horizontal position of your signal. You are only interested in what happens prior to the final falling edge, so the image can be moved to the bottom right of the screen.

4.2   Data Manipulation and Analysis

4.2.1   Data Compression and Transfer

When using the "datalog" capabilities of the DSO software, a file will be written for each trace captured. Using a "memory size" of "32K," a collection of 1000 traces will be over 1 GiB in size. This makes it difficult to transfer the files to another computer for analysis. Fortunately, compressing (zipping) the files can reduce the collection's size to 100 MiB and elliminates the hassle of handling thousands of files at once.

Unfortunately, the ZIP capabilities built into Microsoft Windows are completely incapable of processing this much data in a timely manner. Therefore, the lab PC is equipped with a copy of the "7-Zip" file archival software. In addition to creating ZIP files that can be extracted on any personal computer, it can also automatically split an archive into multiple parts that will fit on

Figure 7: Example of gold wire tips supported on long lever arms.

smaller USB thumb drives. Consult the application's "Help" document or the project's web site (`http://www.7-zip.org`) for more information.

4.2.2   Data Formatting and Importing

If you are performing analysis on a UNIX-based system, you may use `awk` and some simple shell scripting to reformat the datalog's .CSB files into something that can be easily imported by your chosen analysis tool. See Listing 2. If you will be performing your analysis using `R`, the functions in the appendix will import the files for you. See Listing 1.

Note that the columns in the CSB files are *not* properly aligned. Fortunately, constant offsets in time have no effect, but you cannot construct a mapping from ADC bins to voltages by comparing the second and third columns.

4.2.3   The Sieve

The recommended sieve of requiring a sufficiently negative average slope is only one of many possibilities. You may exercise your creativity in thinking of other ways to more accurately select only stepped traces. For example, you might consider recording the driving signal too and requiring that its value be near its minimum.

4.2.4   Data Export

If you would prefer to do your data analysis or plotting using a program like Microsoft Excel (not recommended), you will still need to perform some preliminary data reduction, as Excel cannot handle this quantity of data itself. If you use `R` to perform this reduction (perhaps using the utility functions in Listing 1), you can export the resulting histograms using the command: `write.table(var, file="filename")`, where `var` is the histogram vector you wish to export and `filename` is the name of the file you want to write this data to (it should probably end in `.txt`).

5   Additional References

[4] Joel Brock (instructor). *AEP 1200 Lab Manual: Introduction to Nanoscience & Nanoengineering.* Cornell University, Ithaca, NY, Fall 2008.

[5] Anil Khurana. Ballistic electron transport through a narrow channel is quantized. *Physics Today*, 41(11):21–23, 1988.

[6] R Development Core Team. *R: A Language and Environment for Statistical Computing.* R Foundation for Statistical Computing, Vienna, Austria, 2008. ISBN 3-900051-07-0.

[7] Varlei Rodrigues, Tobias Fuhrer, and Daniel Ugarte. Signature of atomic structure in the quantum conductance of gold nanowires. *Phys. Rev. Lett.*, 85(19):4124–4127, Nov 2000.

A   Source Code

Listing 1: Utility functions for creating histograms from datalogs using GNU R. Results can be exported using `write.table`.

```
## The following routines assume that the current directory contains a
## collection of DSO datalog files in CSB format and that data was
## taken on channel A1.

## Given a vector of raw DSO data values (integers between 0 and 255),
## compute the natural histogram (a 1-to-1 correspondance between ADC
## bins and histogram bins).
ssx_dsohist <- function(v)
{
    h <- rep.int(0, 256)
    for (i in v)
    {
        h[256 - i] <- h[256 - i] + 1
    }
    h
}
```

```
## Given a datalog prefix and the range of its file numbers, compute
## the natural histogram for the entire dataset.
ssx_batchhist <- function(prefix, low, high, suffix=".CSB")
{
    h <- rep.int(0, 256)
    for (i in low:high)
    {
        h <- h + ssx_dsohist(read.csv(
                sprintf("%s%06d%s", prefix, i, suffix),
                header=FALSE, skip=21)$V2)
    }
    h
}


## Given a datalog prefix and the range of its file numbers, compute
## the natural histogram for the subset of traces with a sufficiently
## negative slope.
ssx_sievehist <- function(prefix, low, high, suffix=".CSB")
{
    h <- rep.int(0, 256)
    for (i in low:high)
    {
        dat <- read.csv(sprintf("%s%06d%s", prefix, i, suffix),
                header=FALSE, skip=21)
        lb <- 9
        ub <- length(dat$V1) - 8
        tmp <- data.frame(t = dat$V1[lb:ub], v = dat$V3[lb:ub])
        slope <- lm(v ~ t, tmp)$coefficients[2]
        if (slope < -1e-7)
        {
            h <- h + ssx_dsohist(dat$V2)
        }
    }
    h
}
```

Listing 2: Bash script to extract data columns from CSB files. Output is suitable for use with tools like `gnuplot` or `read.table` in GNU R.

```
#!/bin/bash
# Extract Time and ChA1 Voltage columns from all .CSB files in the
# current directory, appending .txt to the file names
for fname in *.CSB; do
    tail -n+22 "${fname}" | awk -F ',' '{print $1,$2,$3}' \
            > "${fname}.txt"
done
```

B   Revision History

Version	Date	Author(s)	Changes
0.0		plm23	Experiment conception
0.1	2008-12-17	mfh25, mbl33	Preliminary edition
1.0b	2009-05-06	cdm89	First edition