Quantum Design electrical transport user training seminar

part 2: experiment design

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outline of seminar

- which transport option to use?
  - Resistivity, ACT, ETO
- sample preparation
- attaching leads to your sample
- mounting sample on the puck
  - examples for certain measurements
- inserting puck in PPMS
  - grounding considerations
- measurement sequence examples
which transport option to use?

- **3-channel Resistivity** (Res. • I-V • voltmeter)
  - sweet spot: 1 Ω – 1 MΩ
  - measures continuously (<1 sec. per pt.)
  - no diagnostics of data quality or of current source railing

- **2-channel AC Transport** (Res. • Hall • I-V • crit. curr.)
  - sweet spot: < μΩ to 10 kΩ
  - developed during high-Tc (max current=2 A; critical current)
  - data rate not as fast (DSP tasks take >1 sec.)
  - I-V and c.c. are DC measurements (best noise ~50 nV)

- **2-channel ETO** (Res. • dV/dl (dl/dV) • I-V (V-I))
  - sweet spots: μΩ – 10 MΩ (4-wire) and 2 MΩ – 5 GΩ (2-wire)
  - fast measurements again (continuous excitation mode)
  - two independent sources, meters
transport sample preparation

- regular plate geometry ideal
  - easy mounting of leads
  - accurate estimation of sample A/L
- homogeneous and isotropic sample assumed
  - sample cracks, voids, inclusions complicate interpretation of data
- we will need ohmic contact to sample
  - contacts must achieve metal-to-metal bond without insulating layers in between – methods are very sample-dependent!
  - metal (Au, Ag, Pt) contact pad evaporation, sputtering, or CVD can promote ohmic contact to sample underneath
  - Ag paint, sanding, chemical etching can also remove oxides
  - resulting surface should be clean of residue and oils before leads are attached

see Wikipedia article: “ohmic contact”.
attaching leads to the sample

- sample mounting methods – note that PPMS goes up to 400 K!
  - solder: easy, but does not stick to most samples; many solder formulations exist (alloys of In, Pb, Sb, Ag...)
  - ultrasonic soldering (In solder): iron has piezo that breaks oxide barriers
  - wire-bonding: Au or Al 25 micron wires; good contacts when they take; expensive and delicate equipment
  - Ag, Pt or Au paint: volatile matrix evaporates and leaves metal particles; little adhesion force; good electrical contact
  - Ag epoxy (2-part): stronger adhesion, bake sample >100 C
  - pressed In or In-Ga contacts: getting good bond is an art
  - spring-loaded pins: easy to mount/unmount sample but geometry fixed

Ag epoxy is known to become thermally insulating at low T and can sometimes become electrically insulating (it is a mixture of Ag particles and an insulating 2-part epoxy).

Spring-loaded pins also are sometimes prone to issues as insulating barriers can form between the pin and the sample.
placement of leads on sample

• standard 4-wire method
  – 4 distinct contacts along a line on the sample surface
  – current leads I+/I- at ends of sample, V+/V- in between
    • current flow is uniform where V+/V- are located
• van der Pauw method
  – uses 2 measurements to determine $\rho$ directly
  – leads placed on *perimeter* of isotropic, homogeneous and uniform thickness sample
    – see PPMS Resistivity app. note 1076-304
• 2-wire method (only in ETO, for $R>1 \text{M}\Omega$)
  – uses I+ and V- leads ONLY
  – contact resistance can be high, but contacts must still be ohmic
mounting sample to the puck

- sample must be insulated from the puck surface
  - Kapton tape
  - cigarette paper + varnish
  - insulating thin film substrate
- easiest to solder wires to pads on puck
- ground path from puck:
  - puck / sample chamber / PPMS / vacuum pump / wall
- ALL low-level measurements should be isolated from (noisy) ground
Main point: it is easy to jumper wires from one set of pads to another rather than making multiple connection to the sample.

van der Pauw: for more info see PPMS app note 1076-304 on website www.qdusa.com

When one channel is measuring, the other channels are open circuit so they do not interfere.

Another variant: Ch.1 and Ch.2 are van der Pauw, and Ch.3 is Hall.
ACT 5-wire Hall method

- must null voltage at zero field
- requires low R sample
  - 100 Ohm pot
- 4-wire also possible

![Sample](image)
ACT: sample mounting

Ch.1: 5-probe Hall
Ch.2: resistivity

Ch.1: 4-probe Hall
ACT: rotator sample mount

- one set of current leads shared between Ch.1 and Ch.2

Figure 7-2 taken from ACT user manual.
verify/repair contacts on bench

- the most important applications issue with transport measurements!!
- test contacts with ohmmeter (DMM) after sample mounted on puck or rotator board
  - use puck test station P150
  - adapter provided for rotator boards
- is contact resistance $R_{\text{contacts}}$ too high?
  - $R_{\text{measured}} = R_{\text{leads}} + R_{\text{contacts}} + R_{\text{DMMcontact}} + R_{\text{sample}}$
  - $R_{\text{leads}}$ known for a given metal (Ag, Pt, Cu)
  - $R_{\text{DMMcontact}} \approx 0.2 \Omega$
  - $R_{\text{sample}}$ estimated or measured with 4-probe DMM
- methods of lowering $R_{\text{contacts}}$
  - touch up with Ag paint, allow to dry
  - “sparking” (CAUTION!)
    - bipolar current source (~100 mA)
    - switching polarity will create high voltages across resistive junctions, sometimes breaks down oxide barriers

sometimes contacts are fine at 300 K but become insulating at low temperatures. Schottky (semiconductor-metal) barriers are often to blame.
(brief aside: grounding in PPMS)

- dewar singly-connected to ground through pumping line (red)
- experiment cable shield is grounded to electronics, not dewar
- if sample is ESD sensitive, then insert into PPMS by: 1) plug in all experiment cabling to PPMS, 2) turn on electronics but do not start measurements, 3) ground extraction tool (+you) to M6000 chassis during insertion
measurement sequence examples

• (Resistivity) scan excitation example.seq
  – scans on Ch.1 and Ch.2 from – to + currents, first sets up channels
  – Note Bridge Configuration must be ON
• (Resistivity) Ch.1 resistivity example.seq
  – sets up Ch.1 (turns off all others)
  – measures 100 points, each is avg of 4 measurements
  – Note Bridge Configuration “No Action” as bridge already set up
• (ACT) Hall vs Field.seq
  – measures Hall on Ch.1 and Ch.2 from -6 to +6 tesla
  – 103 Hz used for a.c. frequency; 17 Hz also found to be good, must determine best one for your lab by trying different ones (prime numbers, away from line freq. and harmonics)