Quantum Design electrical transport user training seminar

part 2: experiment design

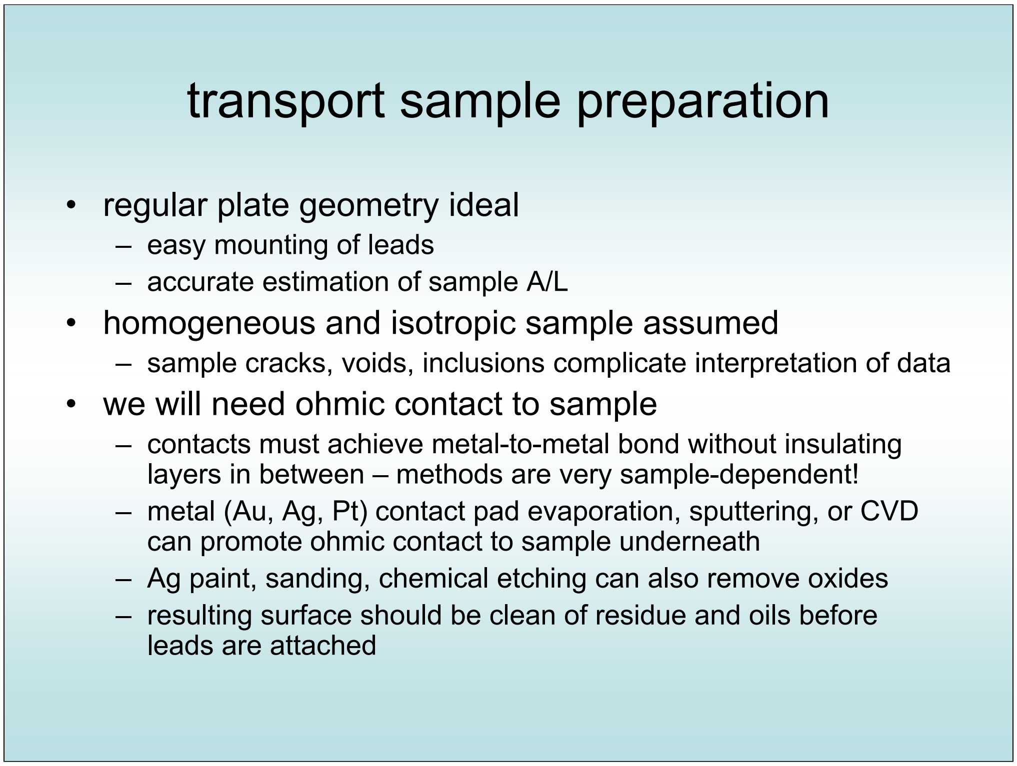
July, 2010
Neil Dilley, Ph.D.
Quantum Design, Inc.

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?

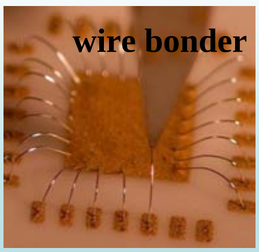
- 3-channel **Resistivity** (Res. ● I-V ● voltmeter)
  - sweet spot: 1 Ω – 1 MΩ
  - measures continuously (<1 sec. per pt.)
  - no diagnostics of data quality or of current source railing
- 2-channel **AC Transport** (Res. ● Hall ● I-V ● crit. curr.)
  - sweet spot: < μΩ to 10 kΩ
  - developed during 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/dI (dI/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



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).

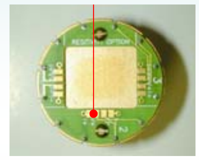
Spring-loaded pins also are sometimes prone to issues as insulating barriers can form between the pin and the sample.

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 ρ 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 MΩ)
  - uses I+ and V- leads ONLY
  - contact resistance can be high, but contacts must still be ohmic

mounting sample to the puck

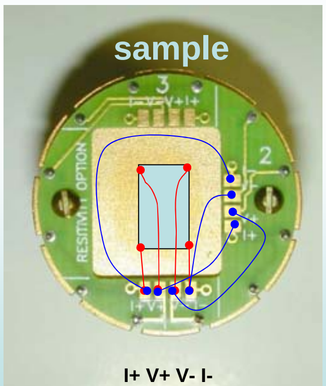
- sample must be insulated from the puck surface
  - Kapton tape
  - cigarette paper + varnish
  - insulating thin film substrate



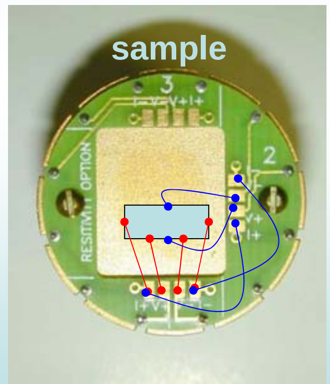
- easiest to solder wires to pads on puck
- ground path from puck:
  - puck / sample chamber / PPMS / vacuum pump / wall
- ALL low-level measurements should be isolated from (noisy) ground

Resistivity (ETO) sample mounting

van der Pauw resistivity
using Ch.1 and Ch.2



Ch.1: resistivity
Ch.2 : Hall



Main point: it is easy to jumper wires from one set of pads to another rather than making multiple connection to the sample.

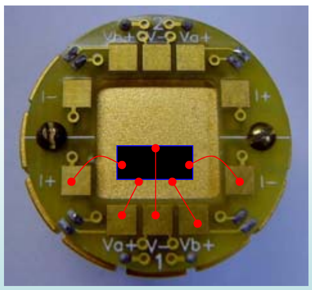
van der Pauw: for more info see PPMS app note 1076-304 on website www.qdusa.com

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

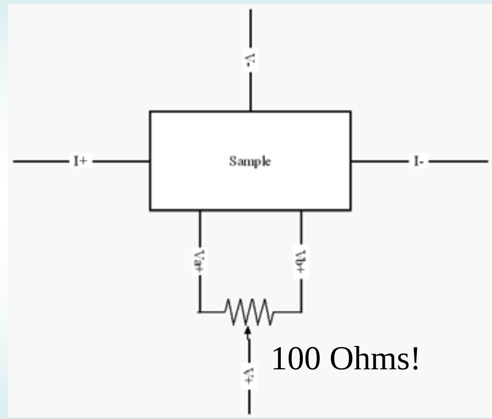
Another variant: Ch.1 and Ch.2 are van der Pauw, and Ch.3 is Hall.

ACT 5-wire Hall method

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

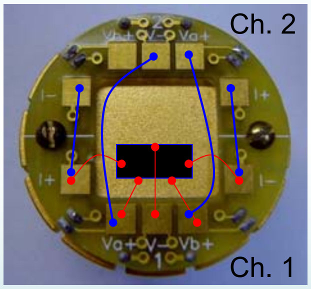


sample



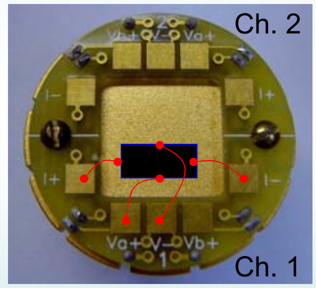
ACT: sample mounting

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



sample

Ch.1: 4-probe Hall



sample

ACT: rotator sample mount

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

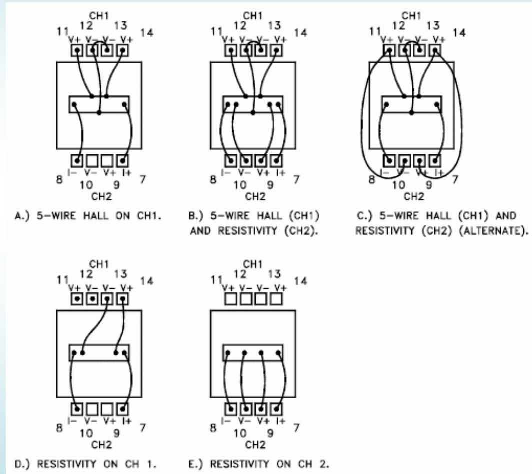


Figure 7-2. Sample-Wiring Diagrams for the Horizontal Rotator Option

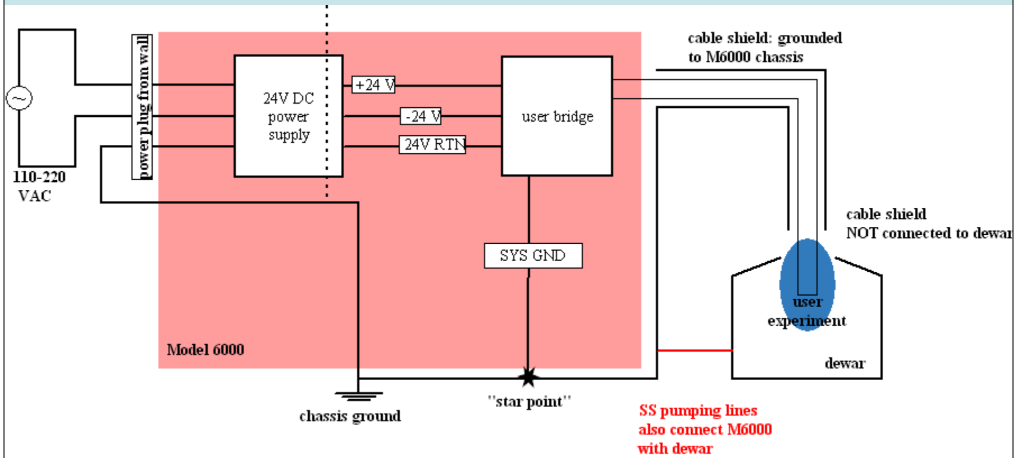
Figure 7-2 taken from ACT user manual.

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 $R_{contacts}$ too high?
    - $R_{measured} = R_{leads} + R_{contacts} + R_{DMMcontact} + R_{sample}$
    - R_{leads} known for a given metal (Ag,Pt,Cu)
    - $R_{DMMcontact} \sim 0.2\ \Omega$
    - R_{sample} estimated or measured with 4-probe DMM
- methods of lowering $R_{contacts}$
  - 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)