FKA195: Lab Demo

Thin Film Resistance Measurements

Elementary

According to Ohm's law for circuit theory, the resistance of a material is the applied voltage V divided by the current I drawn across the material across two electrodes.

$$R = V/I \tag{1}$$

Where:
R = resistance (Ohms, Ω)
V = voltage (Volts,V)
I = current (Amperes, A)

This electrical resistance is proportional to the sample's length L and the resistivity ρ and inversely proportional to the sample's cross sectional area S.

$$R = \rho\,(L/S) \tag{2}$$

Where again:
ρ = resistivity ($\Omega\cdot$m). *The resistivity is a property of a material and does not depend on geometry*
S = cross- sectional area (m^2) = **w**idth $\times$ **t**hickness
L = length (m)

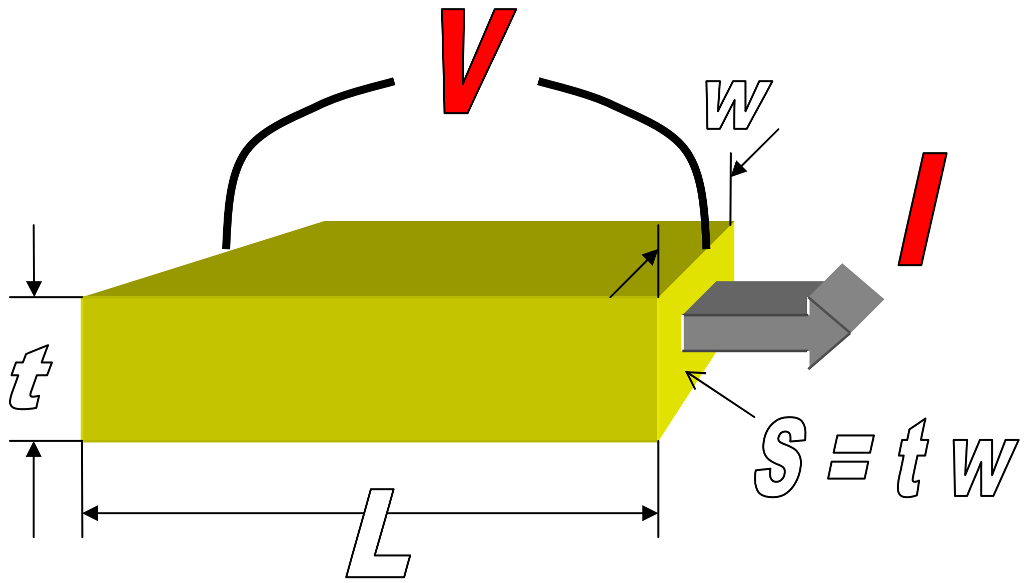


Figure 1. Elementary geometry of resistance measurements.

Elementary (continued)

Parallel and series connections

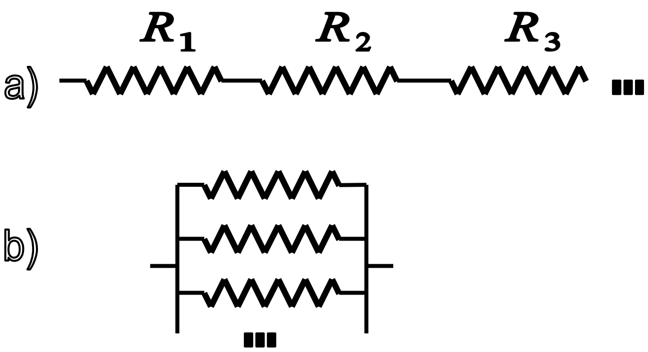


Figure 2. Resistances connected in series (a) and in parallel (b). For N serially connected resistors, $R = R_1 + R_2 + R_3 + \ldots R_i \ldots + R_N$. For N resistors connected in parallel, $1/R = 1/R_1 + 1/R_2 + 1/R_3 + \ldots 1/R_i \ldots + 1/R_N$.

Sheet resistance

Thin films usually have their thickness t (≤ 1 μm) much smaller than all other dimensions, $t \ll w, L$. For the conducting thin films, it is convenient to introduce the so called sheet resistance (or resistance *per square*), $R_{\square}$ (Ω/square). "Square" means the known geometrical figure in its common sense, i.e. a parallelogram having four equal sides and four right angles (width = length, $w = L$). For a given thickness, it does not matter, whether the square is cm- or μm- large. Indeed, Eq. 2 yields: $R = \rho\ (L/wt) = \rho/t = const$ which means that $R_{\square}$ does not depend on w and L for $w = L$.

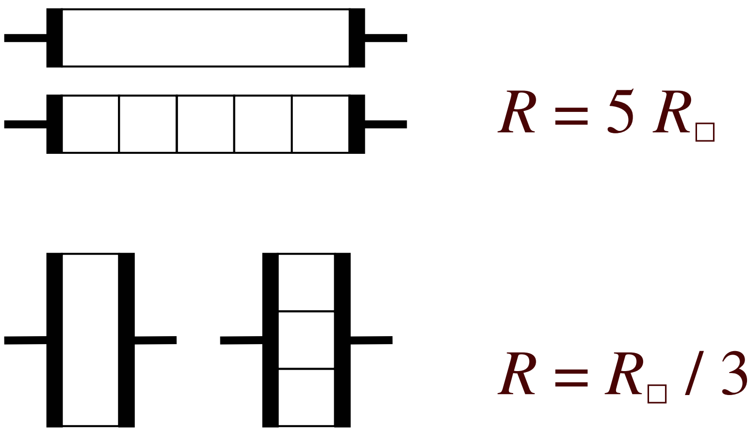


Figure 3. To get the actual resistance of a strip, just count up the number of squares which fit inside the strip. Black bars represent equipotential contact pads with resistances negligibly smaller compared to the resistance of thin film.

Given $R_{\square}$ for a thin film, it is easy to roughly estimate the resistance of the thin film of a simple shape: just count up the number of imaginary squares which fit inside the shape. For M

squares in line with the bias current, the overall resistance will be $MR_\square$, and for those perpendicular to the bias current, the answer will be $R_\square/M$, see Fig. 3.

Elementary (continued)

Simple resistance measurements

1. **Two-probe measurements**

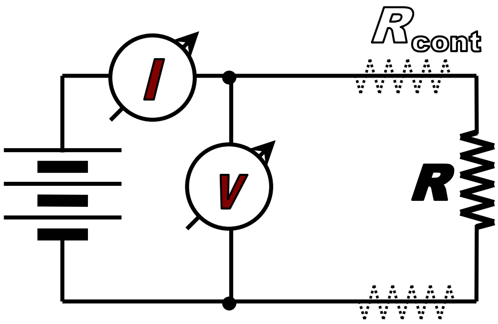


Figure 4. Two-probe resistance measurements. Unknown contact resistance gives error.

In the two-probe measurements, an unknown resistance of connecting wires + contact resistances (together denoted as two R_{cont}) are contributing to the voltage V making the measurements erroneous, $V/I = I(2R_{cont}+R)/I =$ (2Rcont+R) $\neq R$. Only acceptable if $R_{cont} \ll R$.

2. **Four-probe measurements**

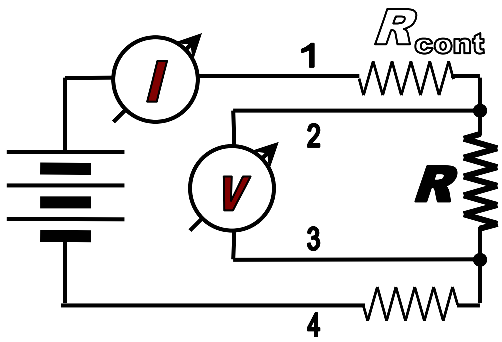


Figure 5. Four-probe resistance measurements. Contact resistance does not contribute to the voltage V. Probes 1 and 4 are usually called "current probes", while 2 and 3 – "voltage probes".

Some peculiar examples of four-probe technique can be found at http://www.mic.dtu.dk/research/nanotech/nanotech.htm#nanoprobes; http://www.mic.dtu.dk/research/nanotech/nanotech.htm#4p;

Practical schemes

Patterned thin films: nice geometry

In order to measure resistivity of a thin film, as one of the characteristics of the thin-films material, one has to know the geometrical factors. One can use micro-technological thin-film patterning techniques for making slabs or strips with very well defined geometry. A typical pattern can be seen in Fig. 6.

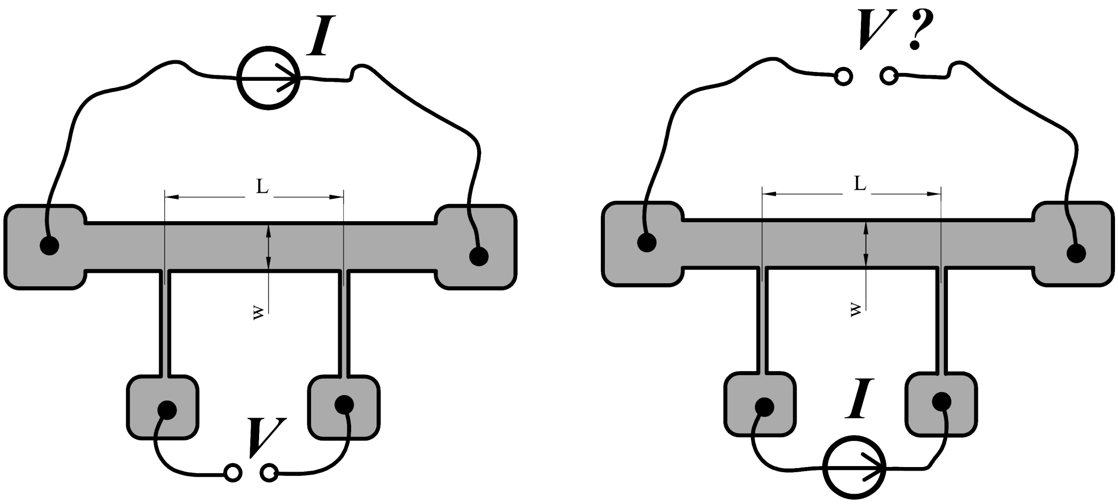


Figure 6. Thin film of thickness *t* patterned into a slab of width *w*. The distance between the voltage contacts is *L.*

Fig. 6 shows a typical slab-shape pattern which can be used for inferring the resistivity from measured *I* and *V*, with help of Eq. 2.

> *Quiz: Would the voltage V change if we exchanged the current and voltage probes, i.e. drove the current through the voltage leads and measured the voltage across the current ones, as illustrated in Fig. 6(right). Why?*

Patterned thin films: arbitrary shapes

In a case of vague geometry or complex shapes, it is difficult to calculate the resistivity from the measured voltage and current. In this case it is convenient to use the so called **van der Pauw method** [3], sketched in Fig. 7. This method uses four isolated contacts on the boundary of a arbitrarily shaped thin film. At least two measurements should be done, according to the geometry sketched in Fig. 7. A total of eight measurements can be done to further reduce effect of thermo-e.m.f. in contacts, with cyclic exchange of current and voltage probes [4].

The resistivity ρ can then be obtained from the following equation:

$$\exp\left(-\frac{\pi t V_{dc}^{ab}}{\rho I}\right)+\exp\left(-\frac{\pi t V_{ca}^{bd}}{\rho I}\right)=1$$

where the upper and lower indices refer to the current and voltage contacts, respectively. Solution of this equation can be represented in the following form:

$$\rho=\frac{\pi t}{\ln 2}\frac{\left(V_{dc}^{ab}+V_{ca}^{bd}\right)}{2I}f(Q),$$

$$Q=\frac{V_{dc}^{ab}}{V_{ca}^{bd}}$$

The geometrical factors *f* can be obtained numerically from the following non-linear equation, see also Fig. 9:

$$\frac{Q-1}{Q+1} = f\ \mathrm{arcosh}\left(\frac{\exp(\ln 2/f)}{2}\right)$$

If $V_{dc}{}^{ab}$ and $V_{ca}{}^{bd}$ are not within 10% of one another, the resistivity cannot be determined accurately.

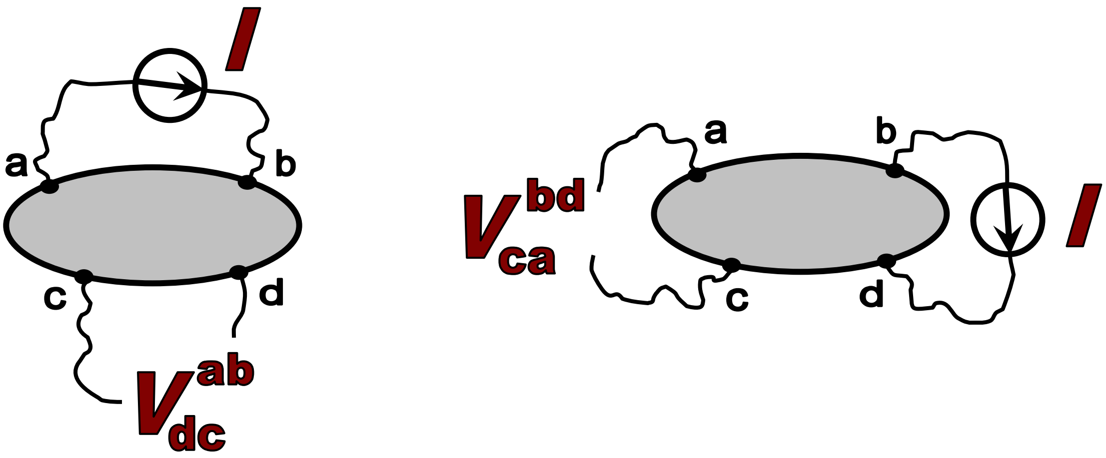


Figure 8. Van der Pauw method of resistivity measurements of a thin film of arbitrary shape.

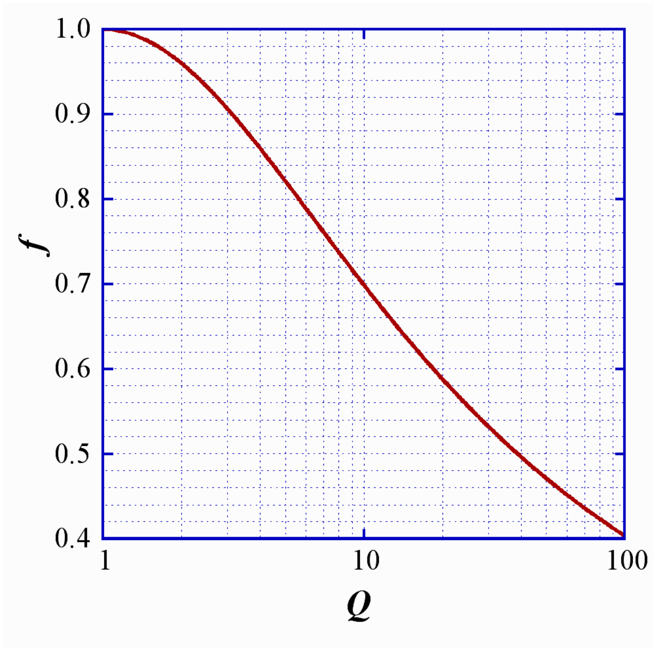


Figure 9. The relationship between f and Q.

Non-patterned thin films: large wafers

Consider a large wafer covered by a conducting thin film of known thickness t. The goal is to infer resistivity from simple four-probe measurements. The practice is to use equally spaced probes placed in a row somewhere in the middle of the wafer.

Provided the separation s between contacts is much smaller (at least 4 times) than the radius of the wafer, and $t < s/2$,

$$R_{\square} = \pi/\ln 2 \cdot (V/I) \approx 4.532(V/I),$$

and

$$\rho = R_{\square}\, t$$

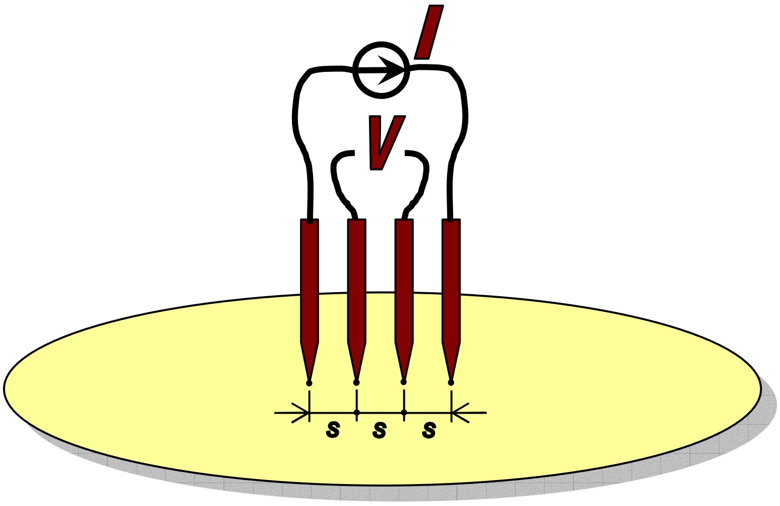


Figure 10. Sheet resistance of a large-area thin film.

One can also show that for the contacts placed in the corners of a square $s \times s$, the corresponding equation will be:

$$R_{\square} =$$

$$= \frac{\pi}{\ln\sqrt{2}} \frac{V}{I} \approx 9.065 \frac{V}{I}$$

For the cases then the wafer size is comparable with s, numerical methods should be used.

Low Impedance Measurements

Offset Voltages

- ### Thermoelectric voltages

Low impedance measurements are associated with low-level voltage measurements. Those can be affected by various parasitic voltages among which thermoelectric voltages are the most common source of errors. These voltages are generated when different parts of a circuit made of dissimilar metals are at different temperatures. The Seebeck coefficients S of various materials with respect to copper are summarized in the table below. To minimize thermoelectric voltages, circuits should be made using one and the same material, say, copper.

Reference material	Material	S (μV/K)
Cu	Cu	<0.2
Cu	Ag	0.3
Cu	Au	0.3
Cu	Pb/Sn	1 - 3
Cu	Fe	11
Cu	W	1
Cu	Kovar	40 - 75
Cu	CuO	1000

Connections must also be kept clean and free of oxides. As is seen from the table, clean Cu-Cu connections have S <0.2 μV/K, while Cu-CuO connections (old oxidized contacts) may have S as high as 1 μV/K!

Minimizing temperature gradients also reduces thermoelectric voltages. All junctions should be in close proximity to each other and to have good thermal anchoring to a common, massive heat sink. Electrical insulators having high thermal conductivity must be used, such as anodized aluminum, beryllium oxide, or sapphire.

Measurements of sources at cryogenic temperatures pose special problems, since the source of signal may be near zero Kelvin while the voltmeter is at ~300K, i.e. there is a very large temperature gradient. Care must be taken as to make sure the wires coming from the room-temperature parts of the measurements setup down to the cryogenic ones are uniform and are taken from one and the same batch (spoon).

To minimize the temperature gradients, equipment should reach thermal equilibrium in a constant ambient temperature. Equipment should also be kept away from direct sunlight, ventilation air-flows, and other sources of heat flow or moving air. Finally, wrapping connections in insulating foam also minimizes temperature gradients caused by air movement.

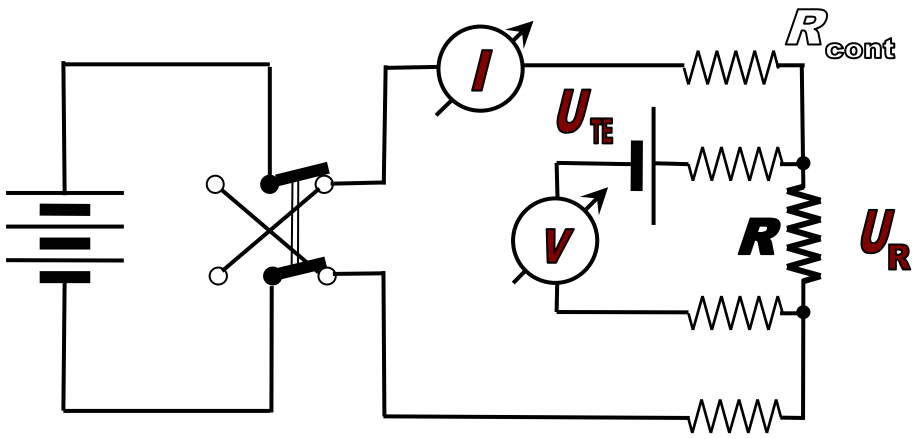


Figure 11. Bias reversal schematic.

Reversing Bias

When measuring a small voltage, the error caused by thermoelectric voltages appearing in the contacts can be subtracted by taking two measurements with opposite direction of the bias current.

By subtracting one measured voltage from another, the parasitic thermoelectric voltage cancels out from the result, as is seen from the following equation:

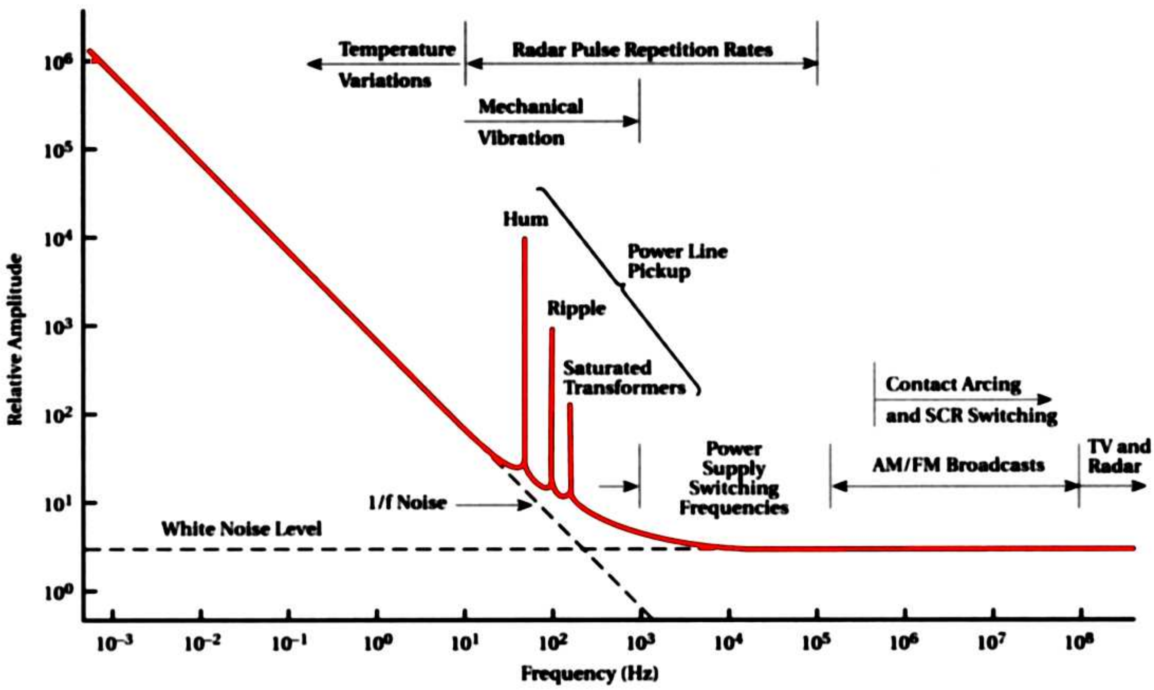
$$\frac{V_+ - V_-}{2|I|} = \frac{(U_R + U_{TE}) - (-U_R + U_{TE})}{2|I|} = \frac{2U_R}{2|I|} = R$$

Notice that this technique cancels out the thermoelectric EMF term U_{TE}, which represents the algebraic sum of all thermoelectric EMFs in the circuit. If the measured voltage is the result of a current source flowing through the unknown resistance, then either the current-reversal method or the offset-compensated ohms method may be used to cancel the thermoelectric EMFs. These methods are described in Section 3.3.2.

- **RFI / EMI**

RFI (Radio Frequency Interference) and EMI (Electromagnetic Interference) are terms for electromagnetic interference to measurements. These cover a wide range of frequencies. Figure 12 shows a typical frequency (*f*) spectrum of these interference signals in comparison with other noise signals such as "l/*f*" and thermal noise.

RFI or EMI can be caused by sources such as mobile phones, TV or radio signals, or come from computer screens or other digital equipment. It can also be caused by impulse sources, as in the case of high-voltage arcing. In all cases, the sensitive measurement can be distorted if precautions are not properly taken. RFI/EMI signals may lead to a parasitic d.c. reading offset due to DC rectification or an input amplifier overload.



from ref.4

The most obvious precaution is to keep all instruments, cables, and a sample far from the interference source. Shielding the leads and the sample (DUT) often reduces interference effects to an acceptable level. The shields should be connected to input LO, but if the RFI/EMI is also earth-ground based, connecting shields to LO may not reduce the interference. A special screening room may be necessary in some cases to attenuate the RFI signal sufficiently.

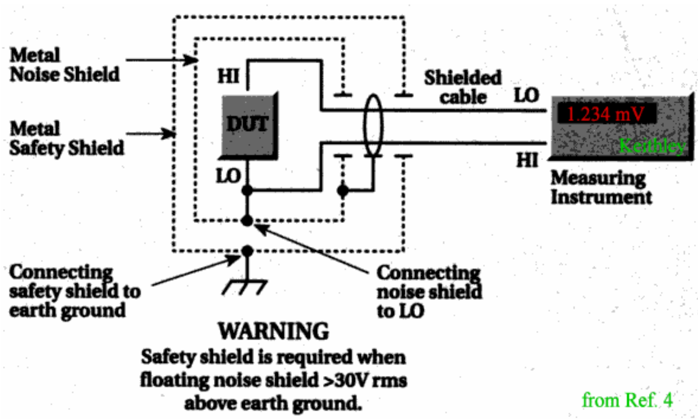


Figure 12. Shielding from RFI/EMI signals (from Ref.4)

- **Johnson noise**

Johnson or thermal noise represents the ultimate resolution in an electrical measurement. This noise is associated with the thermal energy of electrons at finite temperatures. All voltage sources have internal resistance, so all voltage sources develop Johnson noise. The noise voltage across a resistance can be calculated from the following equation:

$$V_J = (4k_B\, T\, B\, R)^{1/2}$$

where V_J is the rms noise voltage, $k_B = 1.38 \times 10^{-23}$ J/K is the Boltzmann's constant, T is the absolute temperature of the resistor R (or an internal resistance of the signal source), B is the noise bandwidth, see Figure 13.

Johnson noise may be reduced by lowering the temperature of the source resistance and by decreasing the bandwidth of the measurement. Cooling the sample from room temperature (293K) to 77K decreases the voltage noise by approximately a factor of 2.

The bandwidth can be reduced by filtering and integration over multiple measurement cycles. The measurement time is then becoming longer.

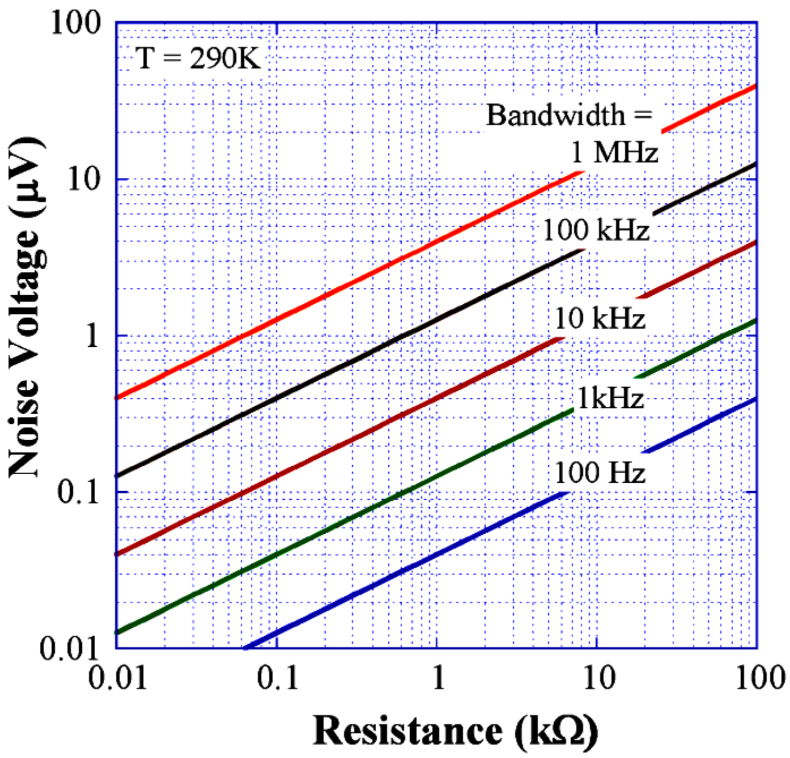


Figure 13. Thermal noise voltage as a function of resistance and bandwidth

High Source-Resistance Measurements

In case when resistance of a sample is becoming high and comparable with a leakage resistance of connecting cables, an error arises due to a shunting effect.

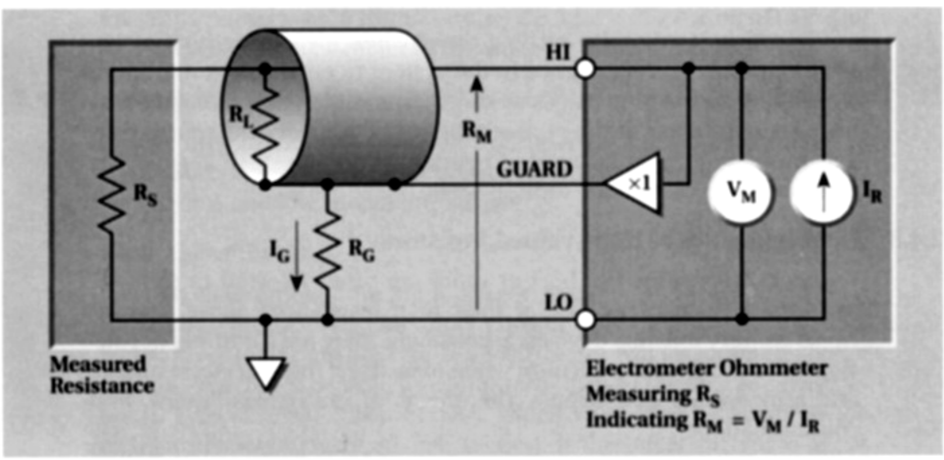


Figure 14. Guarding cable shield to eliminate leakage resistance. (from Ref. 1&4)

This shunting effect can be effectively eliminated by driving the cable shield with a unity-gain amplifier, as shown in Fig. 14. The voltage across the shunting (leakage) resistor R_L, and, accordingly, the current through it becomes zero. This means that the whole bias current flows through the source resistance allowing accurate measurements of the latter to be done. The leakage current (I_G) through the cable-to-ground leakage resistance (R_G) does not affect the measurements, although it can be much larger than the main bias current.

Measurements of Temperature Dependence of a Normal Metal- and YBaCuO- thin films

Two thin films are going to be measured during the demonstration: a normal-metal thin film (Cu, Au, or Al) and a thin epitaxial film of high-temperature superconductor (YBaCuO), both the room-temperature resistances and their temperature dependencies.

A task will be to calculate the sheet resistance for each film and the resistivity of each material (at different temperatures). Questions may be asked regarding, say, differences between two- and four- probe measurements, their advantages and drawbacks, ways of eliminating parasitic voltages, ways of improving accuracy in measuring and calculation of resistivity, etc.

References:

1) High Resistance Measurements (http://www.keithley.com/servlet/Data?id=6584)

2) Reducing Resistance Measurement Uncertainty: DC Current Reversals vs. Classic Offset Compensation :(http://www.keithley.com/servlet/Data?id=4644)

3) L.J. van der Pauw, *Philips Res. Rep.* **13**, 1 (1958); *Ibid.,* **16**, 187 (1961); *Philips Tech. Rev.* **20**, 220 (1959);

4) *Low Level Measurements, 5th edition*. Keithley knowledge tutorial book. http://www.keithley.com.