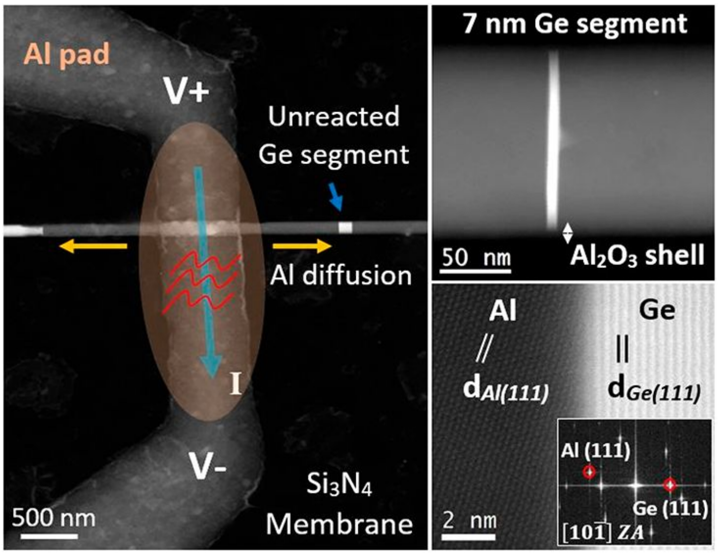
Practical on electrical biasing

Title: Metal propagation in Silicon nanowires by Joule heating

Keywords: in-situ, electrical biasing, nanowires, metal contacts, Joule-heating, silicon nitride membrane.



This practical is about in-situ heating of metals on nanowires (NW) using Joule heating. We pass a current through a metal strip defined on the NW that also serves as an electrical contact. Flowing current through the metal strip, it heats up, and we can initiate a solid-state reaction where the metal starts to propagate into the NW. We will visualize this reaction in real time in the TEM.

Below is a schematic representation of the experiment.

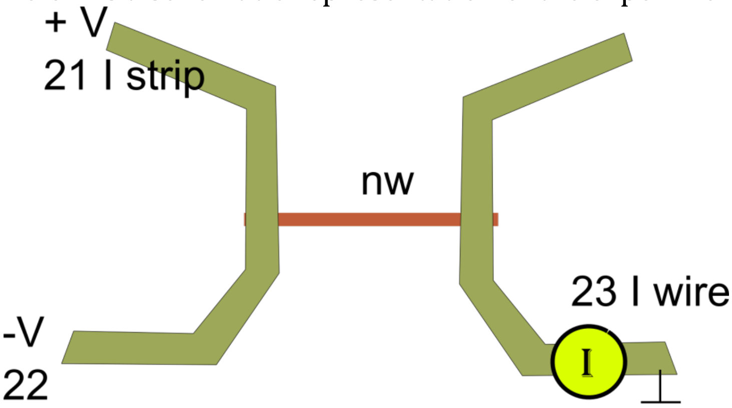


Fig. 1. Description of device and coding of power supply and current meter in program.

This manual takes you step by step through the experiment. Text written in *italic* means that you have to make an action.

We can perform such an experiment with different metals on a NW, for example the following systems: Ge NWs with Ni contacts, Ge NWs with Cu contacts, Si NWs with Pt or Ni contacts. In these cases, an intermetallic, a phase containing both the metal and the Ge is created. In the case of Al on Ge, a pure and monocrystalline Al region is formed. Today we look at Ni on a silicon NW.

1) First, what do we need?
- the contacted NW sample in an in-situ biasing holder
- *check if the metal strip lines are ok and if the tips of the holder are well contacted. We never use a multimeter! and check the resistance between two contacts with a Keithley. Since the stripline is a metal, we should find a resistance on the order of 100 Ohms. It can still work if the resistance is a few kOhm. Because* $P=I^2 R$*, we will heat more if the resistance is lower.*

2) Pole touch, and how it works.
Before we start any electrical in-situ experiment, it's important to know what the TEM does in terms of potential to the sample holder. *Use the multimeter and measure the potential between the inside of the stage with respect to any other metal element on the TEM* (they should all be connected to the same ground of the TEM). *Try to answer the following questions:*
- What do you observe?
- Why?
- What does it imply for your sample that you will expose to an applied potential in the TEM?

3) Insert the sample holder in the TEM like a normal holder.
Wait till the vacuum is ok, and go and look for your sample. Do alignments if necessary and select the region you want to image during the in-situ experiment and choose your field of view. Remember that your beam can influence your reaction. So its good to check if lower or higher acceleration voltage makes a difference, calculate the dose you put into your sample, and check if the dose has an influence. Once you are happy with the TEM part of the experiment, blank the beam and prepare the in-situ.

4) Electrical connections
First connect everything, ***except for the sample holder*****.** You can explode the sample for instance by plugging/unplugging a USB connection. So always be very cautious. Exploding the sample is generally the easiest thing to do.
Connect the ground of your experimental setup (the keithleys) to the TEM, we can use the bar at the nitrogen trap. It can be a good idea to test how clean the ground you use at the TEM is, using a multimeter. Your TEM engineer should be able to tell you were the best ground can be found, and you can check the resistance of this ground with respect to metal parts on the TEM, to see which location is convenient to use, or ask the engineer if you can pull a cable from the main ground.
Everything should be connected to the same ground, the ground of the TEM, to avoid ground loops.

5) Preparing the in-situ experiment on the TEM

Synchronize the time of the different computers involved, you might have a system where your camera is already linked with the in-situ data-set. However, if several computers do the different experiments (one for the camera, another one for the heating/electrical biasing) its important that the time for both computers is the same, otherwise it will be difficult to correlate the two data sets later. You will see that there is often a few minutes difference between two computers.

6) The in-situ experiment on the TEM
Now we start to progressively increase the potential drop over the metal strip line, raising and lowering the potential at both its ends symmetrically, see Fig. 1, to progressively increase the current in the strip line. *We start the acquisition of an image series once we observe that the resistance in the metal strip line decreases over time*, since this indicates we are annealing grain bounderies in the metal, and effectively are annealing our contact. Typically we propagate with some mA of current in the stripline. Similar current levels are typically used in commercial chip based heating membranes with a metal spiral. Hopefully, we visualize the metal propagation inside the NW as a darker/brighter contrast with a sharp interface, that is advancing into the NW, due to a difference in mass/thickness. We stop the reaction when we have propagated a certain distance, by lowering the potential over the stripline, decreasing the current.

7) After the in-situ experiment
We can now potentially investigate our structure using complementary TEM analysis, for instance EDX, high-resolution imaging or crystallographic analysis (ASTAR, nanobeam diffraction) to study the reacted regions and the interface. For the FEI compatible in-situ holder, don't forget that your needles will shadow a great deal of your x-rays, making the holder not so suitable for EDX. This problem is greatly reduced in the JEOL version of biasing holders, since the holder is much wider, the sample is therefore much wider, and the contact pins are located further away from the sample region. If you use an FEI system with a DENSSolutions holder, it's better to use a different holder for EDX.
Be wary that your sample after reaction can change when you take it out of the microscope and expose it to air.

Be systematic; make a manual for your in-situ experiment, because it's very easy to forget an obvious detail, making the experiment useless.

Conclusion: electrical in-situ is cool! But its very easy to explode/melt you specimen, and an experiment can be over before you know it.
I hope you enjoyed the practical!

Questions?
In case you have questions later you can email me at martien.den-hertog@neel.cnrs.fr

More reading:
You can find more information and movies of these experiments in the following 2 articles specifically on the Ge Al system, and in the last article specifically

regarding the **(very important but sometimes overlooked) sample preparation**:

K. El hajraoui, E. Robin, C. Zeiner, A. Lugstein, S. Kodjikian, J.L. Rouviere, M.I. den Hertog. *In-situ TEM analysis of copper-germanium nanowire solid-state reaction*, **Nano Letters 19**, 12, 8365-8371 (2019) https://doi.org/10.1021/acs.nanolett.9b01797

M.A. Luong, E. Robin, N. Pauc, P. Gentile, M. Sistani, A. Lugstein, M. Spies, B. Fernandez and M.I. den Hertog. *In-Situ Transmission Electron Microscopy Imaging of Aluminum Diffusion in Germanium Nanowires for the Fabrication of Sub-10 nm Ge Quantum Disks* **ACS Applied Nano Materials 3**, 2, 1891–1899 (2020) https://doi.org/10.1021/acsanm.9b02564

M. Spies, Z. Sadre Momtaz, J. Lähnemann, M.A. Luong, B. Fernandez, T. Fournier, E. Monroy and M.I. den Hertog. *Correlated and in-situ electrical transmission electron microscopy studies and related membrane fabrication*, **Nanotechnology 31**, 472001 (2020) https://dx.doi.org/10.1088/1361-6528/ab99f0