

# Logbook: influence of passivation on the nucleation of lithium on copper

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2024-07-05

## About this log

This log includes notes about the experimental work and data analysis of the influence of the degree of passivation of the copper surface before deposition of lithium. The work was performed at the Department of Materials Physics at Chalmers University of Technology between 2024-06-10 and 2024-07-10. The experimental work was performed by Gottfrid Olsson under the supervision and support of Josef Rizell, and is documented chronologically.

For information about mechanical polishing of Cu in CMAL see Appendix A, for information about the assembly of the I-cell (and pressure calculation) see Appendix B, and for information about SEM image sampling see Appendix C. For completeness, here is my Master's Thesis: <https://odr.chalmers.se/items/7f321cb1-4a2e-49e5-9a2c-8b06874e3bd0>.

For experimental data (SEM images, SEM PowerPoints, electrochemical cycling data) and code (SEM image sampling and data analysis), see the GitHub repository <https://github.com/GottfridOlsson/SUMMER-JOB-2024-MATERIALS-PHYSICS>.

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## 2024-06-10: Week 1 – Preparations

2024-06-10

Put syringe filters (Acrodisc 25 mm syringe filters with 0.45 mm WWPTFE membrane, HPLC certified, from PALL) in the oven at 80 °C (will be in there for at least 24 h).

Created a sample table in Excel which will contain all the information about the samples.

2024-06-11

Started cycling of two I-cells<sup>1</sup> built 2024-05-16 with an open-circuit voltage (OCV) around 2.2 V and 2.5 V back then (see the data for OCV the current date), with  $\Delta x = 110.0 \text{ mm}$  and  $\Delta x = 110.3 \text{ mm}$ , respectively. They have since been resting in a plastic bag each, and now are being subjected to a cycling protocol. This was to investigate if the time between the assembly and cycling of the cell had any significant effect on the passivation or on the deposition.

Tried polishing copper (Cu), but the grinding table (Buehler Beta grinder-polisher) did not start. Did some quick electrical troubleshooting, but the problem was apparently not with the supply of electricity to the machine. Spoke to Stefan Gustafsson at CMAL, and he would get back to me at the end of the day with a status update. He did not.

Added DME to the dried sieves.

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<sup>1</sup>For references to things (such as cell configurations) that are not explained here, look first in my Master's thesis.

2024-06-12

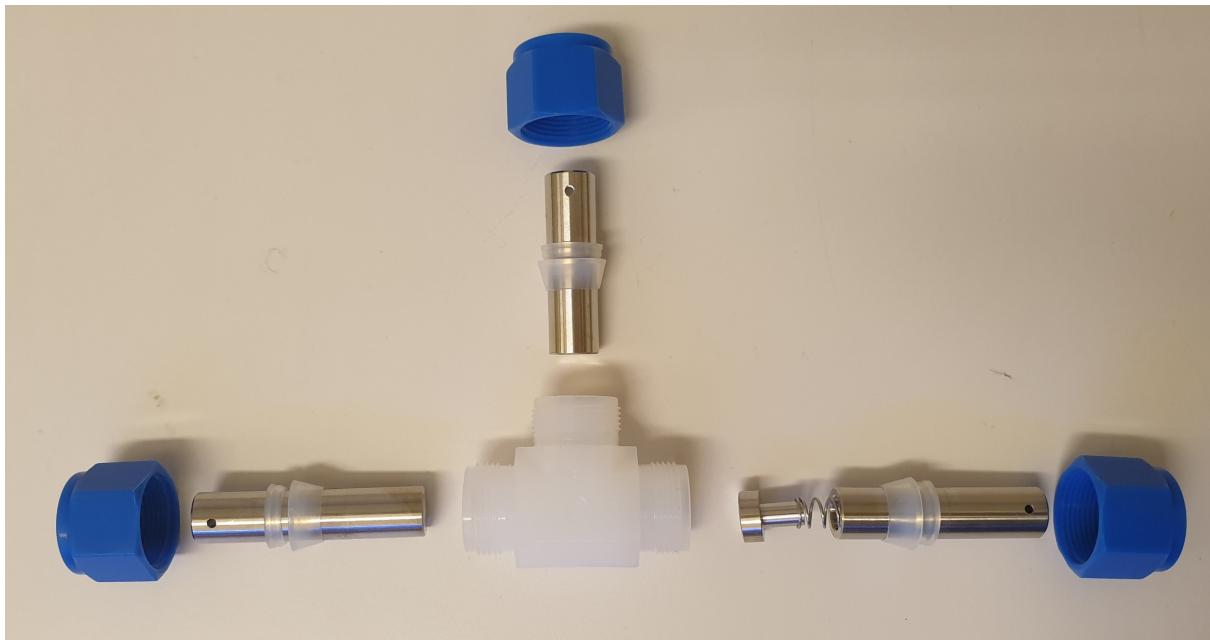
Tried reaching Stefan again, but he was unavailable.

Plotted the passivation in the I-cell for all previous measurements as current as function of time. A rough approximation of the decay of the current is

$$I(t) = \frac{20}{t}$$

where the units of the current is  $\mu\text{A}$  and the time is in min.

Prepared the three-electrode cell with pressure from a spring (TP-cell), see Figure 1.



**Figure 1.** Photograph of the parts of the three-electrode cell with pressure.

The electrolyte was mixed. Specifically, the electrolyte used is lithium bis(fluorosulfonyl)imide (LiFSI) salt in the solvent 1,2-dimethoxyethane (DME). The LiFSI salt was dried in an oven (Büchi B-580) at 80 °C for at least 24 h under vacuum. To remove water molecules from the solvent, the DME was put into a glass vial together with molecular sieves with pore size 3 Å. The molecular sieves had previously been dried in the oven at 220 °C for 24 h under vacuum. The DME soaked the molecular sieves for at least 24 h and the DME was then pushed through a filter (Acrodisc PSF with 0.45  $\mu\text{m}$  pore size) to remove any impurities from the molecular sieves before use.

The wanted concentration is 4 mol L<sup>-1</sup> of LiFSI salt in DME. This concentration is achieved by using a mole ratio of 1.4/1 (DME/LiFSI)<sup>2</sup>. The calculation for the mass of each component is given below. The equation is

$$\frac{\text{mol}_{\text{DME}}}{\text{mol}_{\text{LiFSI}}} = \frac{1.4}{1} \implies \text{mol}_{\text{DME}} = 1.4 \text{ mol}_{\text{LiFSI}}$$

which means that if we take 1 mol of LiFSI we shall take 1.4 mol DME. DME<sup>3</sup> is C<sub>4</sub>H<sub>10</sub>O<sub>2</sub> with molar mass  $M_{\text{DME}} = 90.12 \text{ g mol}^{-1}$  while LiFSI<sup>4</sup> is F<sub>2</sub>LiNO<sub>4</sub>S<sub>2</sub> with  $M_{\text{LiFSI}} = 187.1 \text{ g mol}^{-1}$ . If

<sup>2</sup>Source: <https://doi.org/10.1038/ncomms7362> (supplementary information, Table S1).

<sup>3</sup>Source: <https://pubchem.ncbi.nlm.nih.gov/compound/8071>.

<sup>4</sup>Source: [https://pubchem.ncbi.nlm.nih.gov/compound/Lithium-bis\\_fluorosulfonyl\\_imide](https://pubchem.ncbi.nlm.nih.gov/compound/Lithium-bis_fluorosulfonyl_imide).

we take  $m_{\text{LiFSI}}$  as the mass of LiFSI that means  $m_{\text{LiFSI}}/M_{\text{LiFSI}}$  moles LiFSI, and in the same way if we take  $m_{\text{DME}}$  mass of DME we get  $m_{\text{DME}}/M_{\text{DME}}$  moles DME. Together, and with the sought mole ratio  $r_{\text{DME/LiFSI}}$ , we get in the general case that

$$m_{\text{DME}} = r_{\text{DME/LiFSI}} \frac{M_{\text{DME}}}{M_{\text{LiFSI}}} m_{\text{LiFSI}}$$

which with the numbers above gives

$$m_{\text{DME}} = 1.4 \frac{90.12 \text{ g mol}^{-1}}{187.1 \text{ g mol}^{-1}} m_{\text{LiFSI}} = 0.6743 m_{\text{LiFSI}}.$$

For example, if we want a total electrolyte mass of 5 g we should take  $m_{\text{LiFSI}} = 2.986 \text{ g}$  and  $m_{\text{DME}} = 2.014 \text{ g}$ .

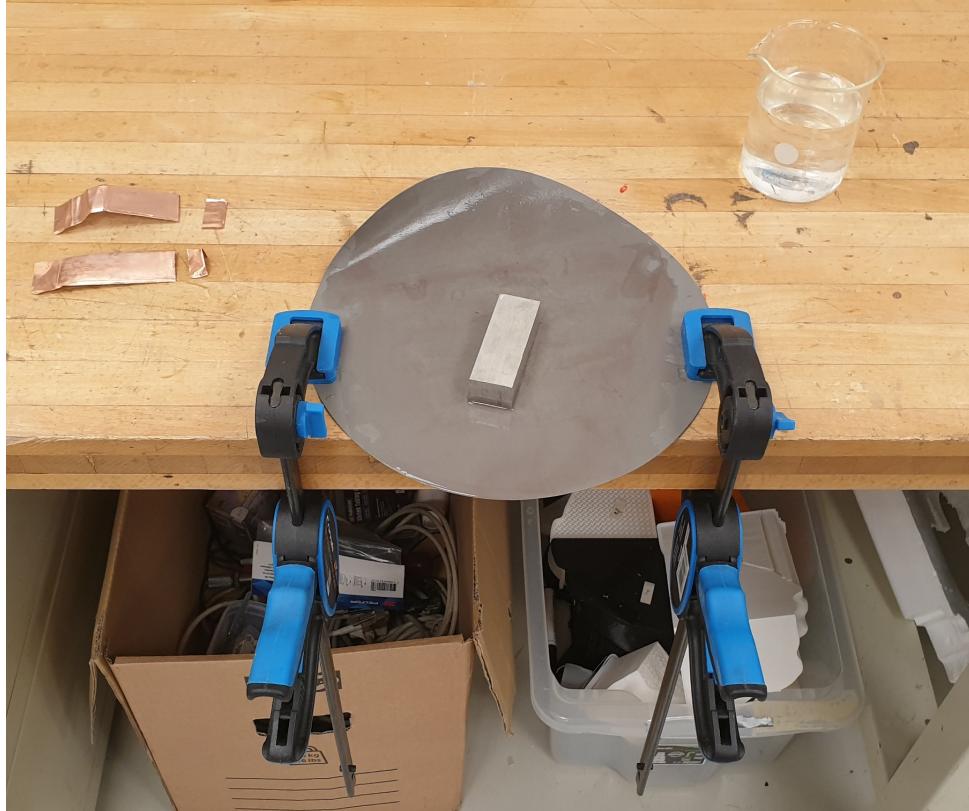
The actual measurements were  $m_{\text{LiFSI}} = 2986.0 \text{ mg}$  and  $m_{\text{DME}} = 2018.8 \text{ mg}$  (measured with a scale with  $\pm 0.1 \text{ mg}$ ). The actual mole ratio becomes

$$r_{\text{DME/LiFSI}} = \frac{m_{\text{DME}}}{m_{\text{LiFSI}}} \frac{M_{\text{LiFSI}}}{M_{\text{DME}}} = \frac{2018.8 \text{ mg}}{2986.0 \text{ mg}} \frac{187.1 \text{ g mol}^{-1}}{90.12 \text{ g mol}^{-1}} = 1.40364$$

which is close to a mole ratio of 1.4 (to two decimal places). The mixed electrolyte is being magnetic stirred (for at least 24 h at room temperature).

2024-06-13

Tried polishing the Cu by hand, see Figure 2. Unfortunately, polishing the Cu by hand seemed to be difficult if one wished to achieve an even polish of a larger piece of Cu. I am certain that it is possible, but this method probably either produces a subpar polish, or small amounts of well polished Cu. In any case, a real grinding table significantly makes the polishing significantly easier and faster.



**Figure 2.** Photograph of the setup used to try to polish the Cu by hand. The grinding paper was held in place by two clamps on the edge of a table. The Cu (waste) strip was held around a metal cuboid whilst polishing with manually applied water. It seemed to work somewhat okay at first, but after having torn the Cu twice I deemed this method unfeasible for producing larger quantities of high-quality (even) polished Cu.

Gave it another go, this time using the setup shown in Figure 3. This setup worked much better, with no tearing of the Cu as well as resulting in okay-ish surface finish. This method is quite bad compared to using instrument at CMAL, both due to the work being manual and with respect to the resulting surface on the Cu. Still, I deemed parts of this polished Cu to be good enough for electrochemical measurements. Pieces of this Cu were punched and cleaned (a note is writing in the sample table).



**Figure 3.** Photograph of the second setup used to try to polish the Cu by hand. This time the Cu was placed onto a flat piece of metal cuboid, and was held in place by two clamps on the edge of a table. The grinding paper was cut and held in place around another metal cuboid using the fingers. Water was applied manually when needed during the polishing (P1200, P2500, and lastly P4000-grade grinding paper). This setup worked significantly better, mostly due to the Cu being fixed and fixed onto a flat(er) surface.

2024-06-14

Built four I-cells and one TP-cells (Swagelok-type three-electrode cells with pressure). Ran cycling protocol on them (see sample table).

See PowerPoint for plots.

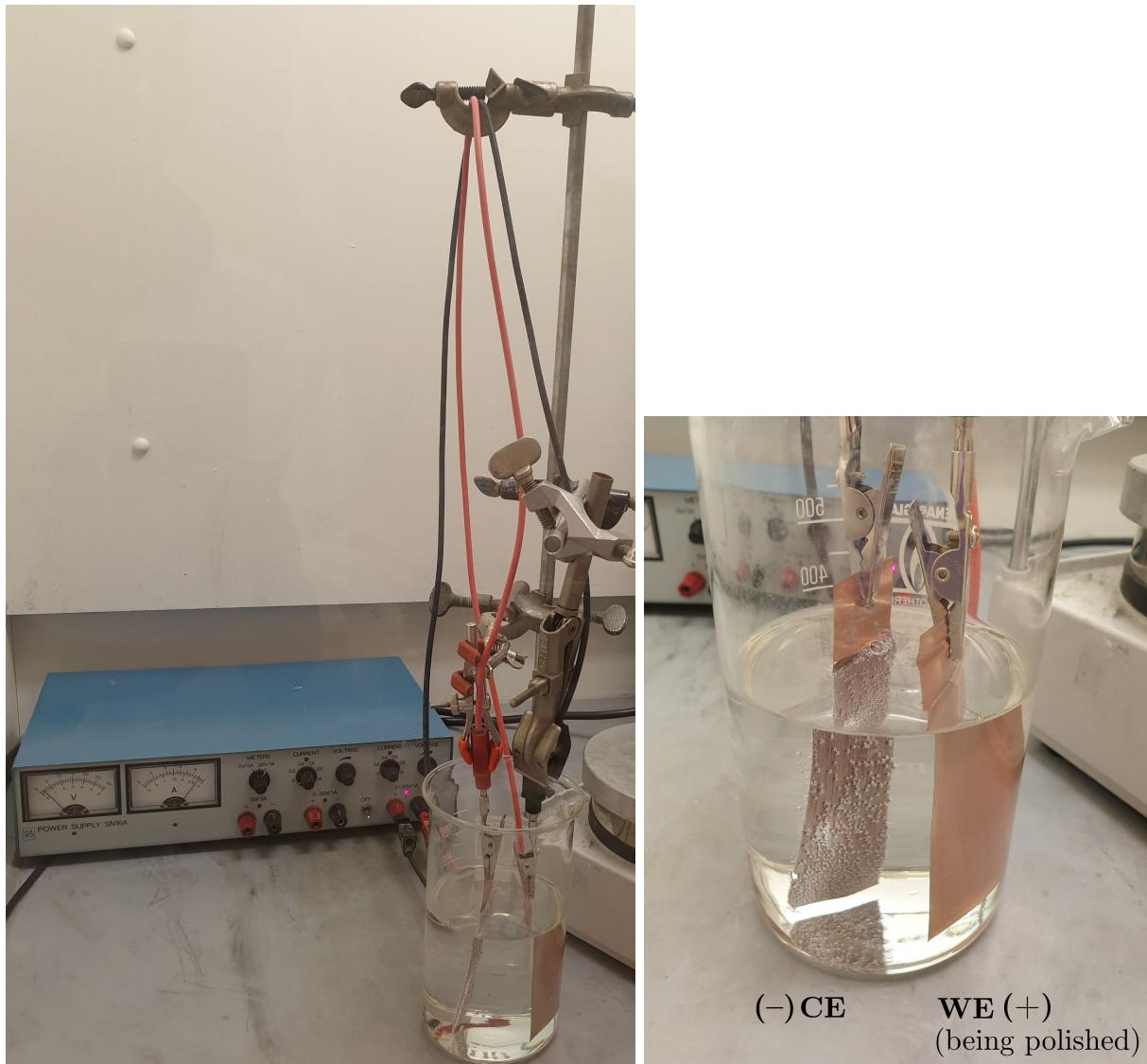
## 2024-06-17: Week 2 – Electropolishing and SEM

2024-06-17

Cleaned another set of four I-cells. By having a total set of eight I-cell parts, we could perform SEM measurements of four samples each two days in a row.

Polished two sheets of Cu at CMAL (the grinding table was now fixed). This was orders of magnitude easier and with better result compared to polishing by hand, and much faster.

In order to ease the thresholding and analysis of the SEM images, an even lower surface roughness is desirable. Therefore, the polished Cu polished to grit paper P400 was further polished by electropolishing (EP). The polished (P4000 grit paper) Cu sheet was cut in two before EP one piece at a time (to fit inside the beaker). The setup (placed in a fume hood) used is shown in Figure 4. The setup consisted of a DC power source connected by "banana" connectors to crocodile clips which were clipped to two pieces of Cu, the counter electrode (CE) and the working electrode (WE). The CE was connected to the negative (-) port of the DC power supply, while the WE was connected to the positive (+). Both pieces of Cu were put into a glass beaker filled with phosphoric acid (85 % concentration in water) such that only the Cu



**Figure 4.** Photographs of the setup during EP of Cu sheet.

were immersed in the acid. The EP was performed by applying a constant 2 V (WE vs. CE; that is  $E_{WE} - E_{CE} = 2$  V) for 10 min per Cu sheet as the WE.

After the EP, each Cu sample was rinsed thoroughly using deionized water, followed by two less thoroughly rinses of first acetone and then isopropanol. When the solvent had evaporated from the Cu surface, the sheet was put into a plastic bag. The P4000-polished Cu and the Cu after EP is shown in Figure 5. The EP sample show a more mirror-like finish, having removed some surface roughness. Though, some "grinding-marks" or "grinding-lines" were still visible. Still, the surface looked clearly improved which hopefully makes the image analysis easier and cleaner.



**Figure 5.** Photograph of the Cu after polishing to P4000 (left) and the Cu after EP (right).

2024-06-18

The EP Cu sheets were punched with diameter 10 mm and then cleaned by immersing each Cu piece in its own small glass beaker (polished side up to avoid scratches) and filled with solvent. The glass beakers were put into a larger glass container filled shallowly with deionized water. Acetone was used first during the ultrasonic cleaning, it was then poured out and replaced by isopropanol during the second ultrasonic cleaning (at room temperature both times). The ultrasonic cleaning was done using FisherBrand ultrasonic processor (120 Watt 20 kHz, model CL-18) at amplitude 70 % for 10 min per solvent. The Cu pieces were then dried (letting the solvent evaporate) and lastly transferred into the argon-filled glovebox.

Everything should be ready for the cell assembly and cycling tomorrow, and then SEM in the afternoon.

2024-06-19

Assembly, cycling and SEM measurement of S06, S07, 08, S09. See sample table and PowerPoint.

2024-06-20

Export of SEM images. Open `.sem` images with MultiPak and export them as `.tif`. Name convention: {date-of-SEM}-{SEM-image-number}-{SampleID}-{magnification(Xnnnn)}.tif. Magnification because the MultiPak software does NOT export the images with the scalebar (for some reason), and the magnification value is needed both in order to add the scalebar using my own Python script and to calculate the imaged area (for nuclei areal density).

## 2024-06-24: Week 3 – More SEM and data analysis

2024-06-24

Washed Cu as done throughout the Master's Thesis; put each Cu in a separate glass beaker, filled with acetone, ultrasonic was for 10 minutes and then another wash but with isopropanol. The difference between this and the previous Cu wash was that the FisherBrand Easy ultrasonic cleaner was used.

2024-06-25

Built and cycled cells with sample ID: S12, S13, S14, and S15 (these use the Cu washed yesterday). SEM in the afternoon.

2024-06-26

Based on previous calculations during my Master's Thesis<sup>5</sup>, I have now derived an expression of the imaged area  $A$  of a SEM image from the magnification  $M$ . The expression is

$$A(M) = \left( \frac{99840}{M + 6.6} \right)^2$$

where  $A$  is in  $\mu\text{m}^2$  and  $M$  is the 'number of magnification' (written as e.g. X131 or X5000 on each exported SEM image, so here  $M$  is 131 or 5000). This equation assumes that the exported images from the MultiPak software is 512 px by 512 px.

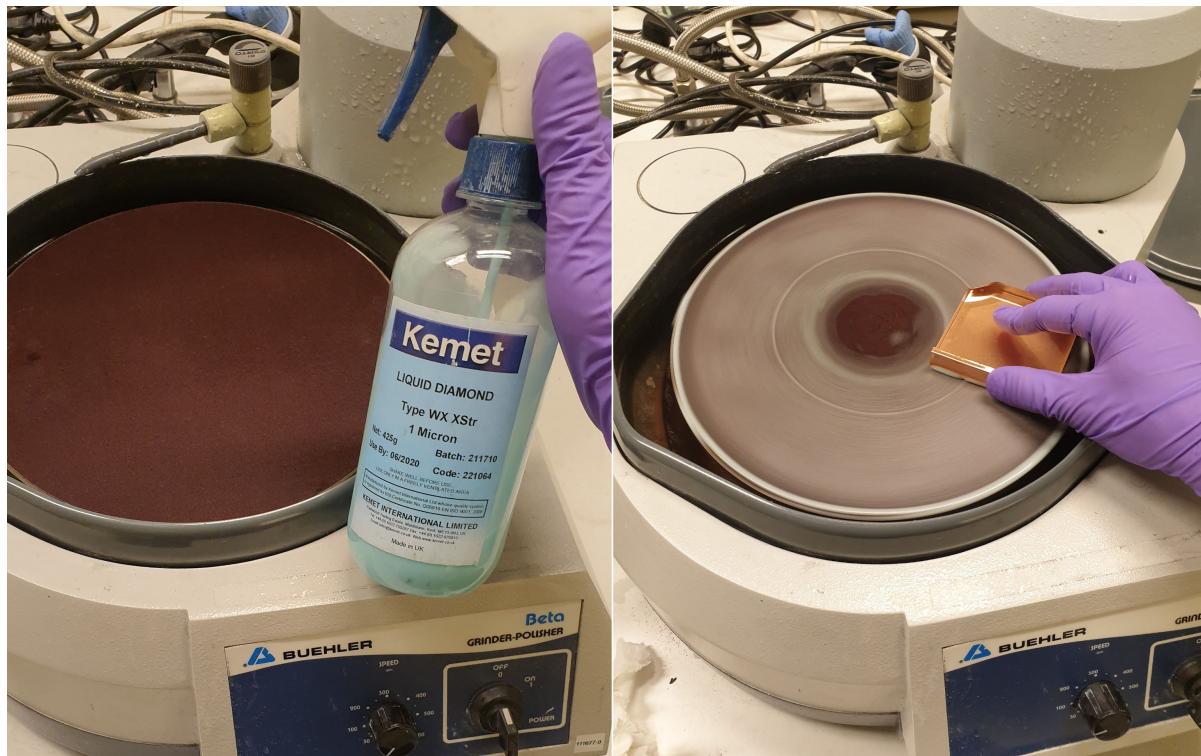
Going to do polishing of Cu using P1200, P2500, P4000, then slurry of particle size 1 microns, and finally slurry of 0.25 microns. This is based on this source <https://www.riogrande.com/knowledge-hub/articles/grit-to-micron-comparison-chart/>, which says that P1200 is comparable to 9 microns and P4000 about 3 microns. Then I extrapolated and guessed that 1 microns followed by 0.25 microns should be fine. Josef also polished his samples to 0.25 microns.

I used things that were already at CMAL for this finer polishing. The polishing was carried out in the same way for the slurry as with the grinding paper. The difference being that a soft sheet with a magnetic backside was place on the turntable instead of a grinding paper, and the slurry was applied (sprayed using a spray bottle) on top of the soft sheet. After the P4000 grinding paper, a soft sheet (MD-Chem, porous neoprene, 250 mm diameter) and 1 micron slurry (Kemet, liquid diamond, type WX XStr) was used to polish the Cu, see Figure 6. The Cu was then washed with deionized water and blown dry using nitrogen gas ( $N_2$ ). Then the soft sheet was washed using water, and another soft sheet (MD-Nap, short synthetic nap, 250 mm diameter) was applied with 0.25 micron slurry (Kemet, liquid diamond, type WX XStr). After polishing and washing with deionized water again, the Cu was blown dry using  $N_2$  and put into a new plastic bag before the punching and washing (as described in the Master's Thesis).

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<sup>5</sup>Where I derived that the length in micrometer per pixel,  $L_{\text{muPerPixel}}$ , as function of the magnification  $M$  was

$$L_{\text{muPerPixel}}(M) = \frac{195}{M + 6.6} \mu\text{m px}^{-1}.$$



**Figure 6.** Photograph of soft sheet on the turntable and the 1 micron slurry used during polishing of the Cu.

2024-06-27

S16, S17, S18, and S19 SEM-measurement. The acquisition method for SEM images is explained in Appendix C.

2024-06-28

Data analysis. Intended workflow:

1. Ilastik (software): train the AI to separate the Li from the Cu. Output: segmented images with one 'color' for Li and one 'color' for Cu.
2. Python or ImageJ: use watershed to identify individual particles from the segmented images, and then do some particle analysis to get 1) number of particles, and 2) area per particle.
3. Python: perform statistical analysis of data from 1) and 2) for each sample.

Ilastik software: version 1.4.0.post1.<sup>6</sup> Guide for pixel identification: <https://www.ilastik.org/documentation/pixelclassification/pixelclassification>. Ilastik cannot open .tif images for some reason. Converting to .tiff using: <https://www.freeconvert.com/tif-to-tiff/download>.

Exporting using 'Simple Segmentation' as Source, with export image settings .tiff as format and all Transformations boxes unticked (no conversion to data type, no renormalization [because that did not do anything], and no transpose of axis order).

Wrote a macro in ImageJ for analysis and tested it. Seems to work fine. Wrote code in Python to do the statistical analysis (putting all the data from different analyzed SEM images together for each sample).

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<sup>6</sup><https://www.ilastik.org/download>.

## 2024-07-01: Week 4 – Analysis, reproducibility, and SEM

2024-07-01

Tried out the data analysis workflow.

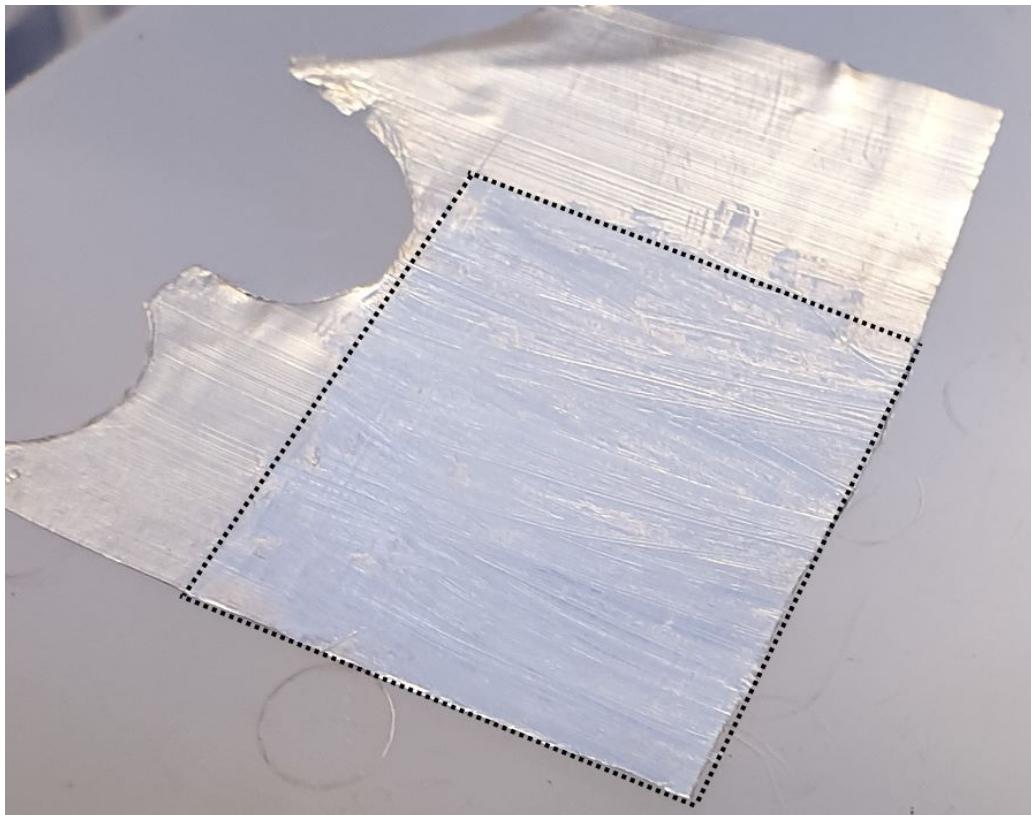
The workflow is as follows:

1. Take SEM images with the instrument. This produces images in the format `.sem`.
2. Export the images (one by one) by opening them in the MultiPak software (which is the only way to read the `.sem` images as far as I can tell) and exporting them as `.tif`. During this step, include the magnification in the file name as this information is not included otherwise in the image.
3. Use Ilastik software to segment the SEM images.
  - Since the Ilastik software does not want to open the exported `.tif` images, convert them to, for example, `.tiff` using: <https://www.freeconvert.com/tif-to-tiff>. Yes, this is cumbersome. Yes, there is probably a better way of doing this.
  - Open the images in Ilastik.
  - Train (or use an already trained program).
  - Perform the segmentation.
  - Export using "Simple Segmentation" as the "Source", with export image settings `.tiff` as format and all Transformations boxes unticked (no conversion to data type, no renormalization [because that did not do anything], and no transpose of axis order).
4. Run the macro for image analysis in ImageJ.
  - Edit the macro to include the folders of the images you want to perform the analysis on. Save the macro.
  - Open ImageJ, and run the macro (under "Plugins", "Macros", "Run").
  - Check that the analyzed images seems to make sense (is not obviously badly analyzed).
5. Run the Python script "SEM-images-statistical-analysis.py"
  - Open and edit the script to include the folders of the images you want to analyze.
  - Run the script. It will produce a `.csv` file with the combined results of all analyzed images for each sample. This includes the radius for each analyzed particle, the number of analyzed images, total number of analyzed particles, total imaged area, and the calculated areal density. The radius for each analyzed particle is included because one can then read the `.csv` file and plot the data in any program.

The workflow seems to work fine for images from S18. However, since the AI was trained on smaller, crystalline, white Li particles, it had trouble to correctly identify (and close) larger "flatter", and darker, Li particles. This lead to poor segmentation and therefore analysis, as seen from the images from ImageJ where the particles had been 'identified' (poorly, that is).

2024-07-04

Cells built and cycled, followed by a SEM session. Tried punching Li with diameter 12 mm, and also "polish" the Li before punching. Figure 7 shows the Li which had been polished by scraping a scalpel at a 45-degree angle to the Li in long strokes (first in one direction and then in a direction orthogonal to the first to completely cover the area).



**Figure 7.** Polished Li (marked area) by scraping the Li surface with a scalpel held at an angle.

## 2024-07-08: Week 5 – 3D-printing

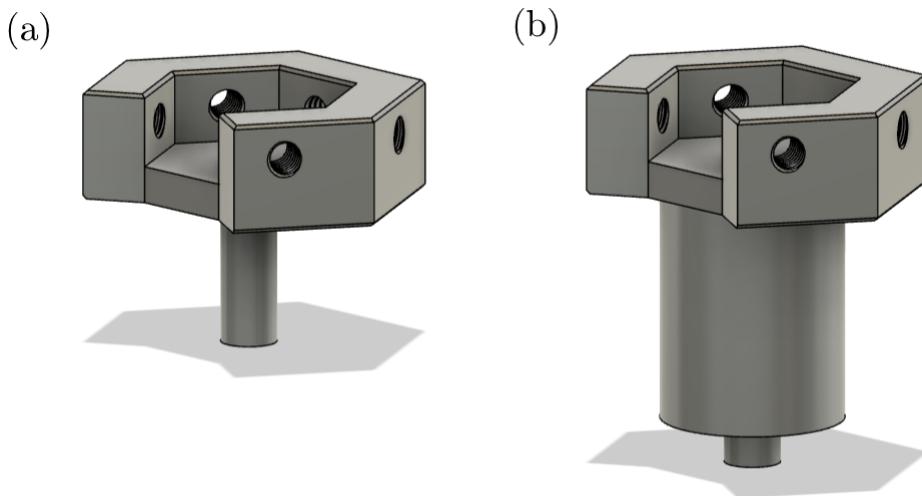
2024-07-08

3D-printing of hexagonal plates for polishing. Printed on 3D Teamet's SLA resin printer (Form3+) in the laser cutter room. Used an adaptive layer height during print with Black V4 resin.

2024-07-09

Cleaned the 3D-prings by first snipping away the support materials with a pair of pliers, and removed the resin on the surface of each part by first drying them with a paper towel and then sonicating them in alcohol for 2 minutes twice (drying them of using paper towels in between). The parts were then cured (hardened by exposure to UV light) at 60 °C for 60 min.

Altered the holder to have four holes for screws around the hex nut on the cell, and picked the diameter of 3.10 mm in Fusion 360. This resulted previously in a diameter of 3.13 mm (after curing) which was the one with the best fit according to Josef's friend. Also created one holder with a foot which can be mounted directly onto the turntable, without the need for the PEEK holder they have at the synchrotron facility. This is shown in Figure 8.



**Figure 8.** Screen capture of 3D-models in Fusion 360, with (a) the new holder with four screws (and with a pin at the bottom to put into another PEEK holder), and (b) the new holder with four screws and with its own "foot" (that can be placed directly into the turntable).

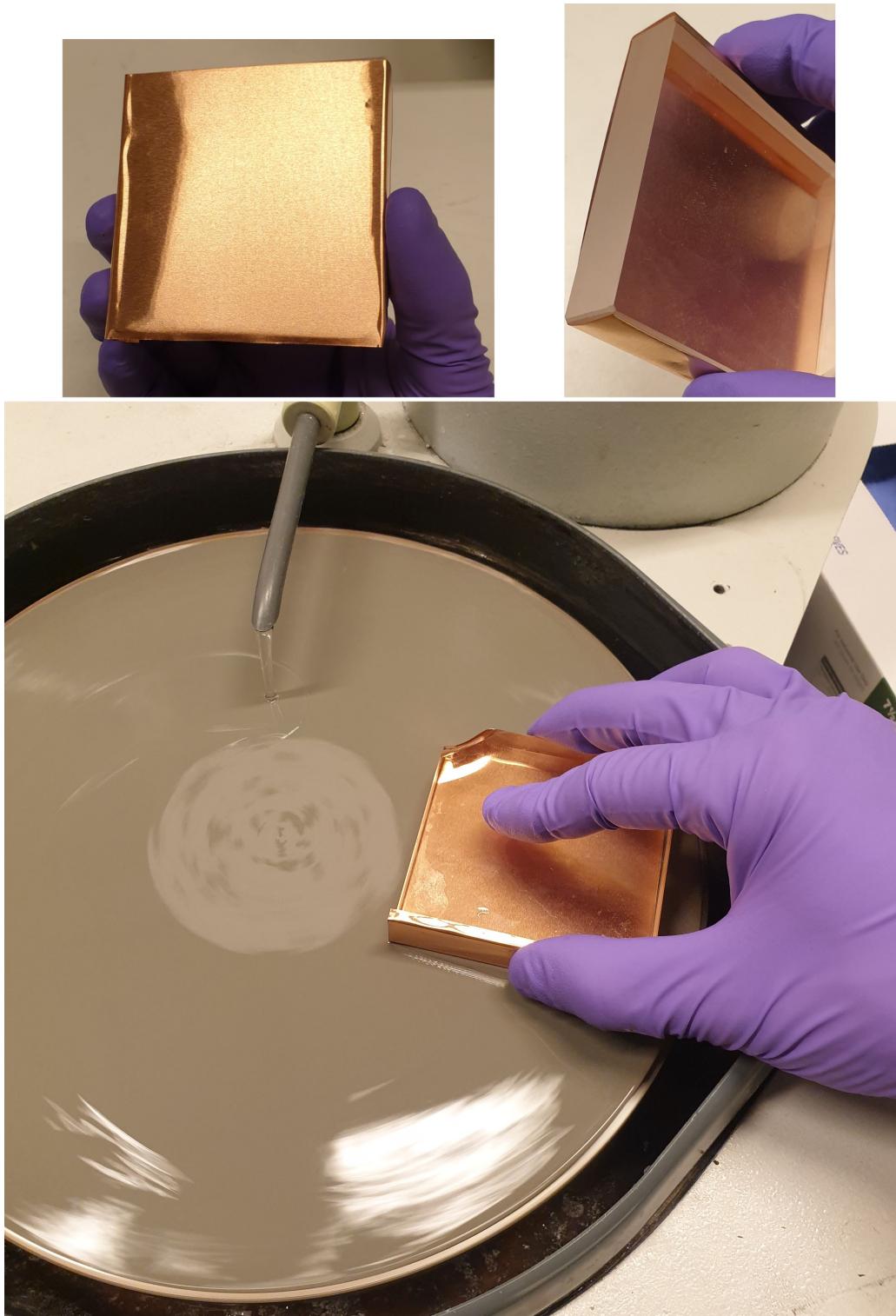
Started with a print of nine (a)-holders that should be done before the end of the day, and will start another print of (a)- and (b)-holders.

2024-07-10

Took care of the 3D-prints the same way as described yesterday. The only exception is that the cure was performed at 60 °C, the same, but this time for 40 min. I used the same cure-time as the first time the holders were printed to get the same amount of expansion.

## A Mechanical polishing of Cu

Figure 9 shows the polishing of one 70 mm by 70 mm (dimensions of the glass piece) Cu sheet on the turntable. First, two edges of the Cu sheet were bent around the edges of a flat piece of square glass. A grinding paper was then placed center on the turntable (Buehler Beta grinder-polisher) and the water was turned on. During polishing, the grinding paper rotated while the Cu sheet was held in place by applying pressure on the edges of the glass using the fingers. An even surface



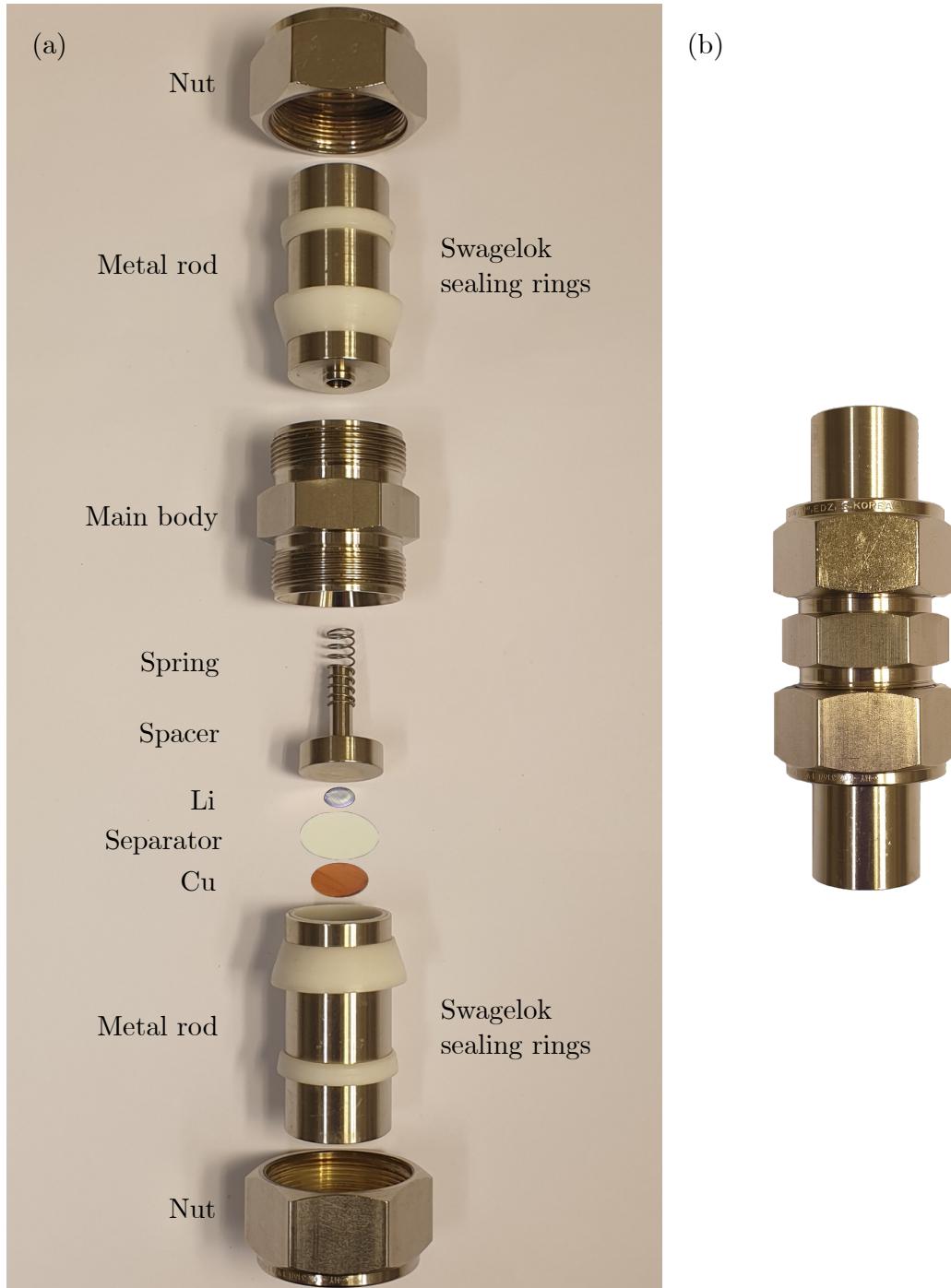
**Figure 9.** Polishing of a Cu sheet using a turntable. See text for details.

polish was achieved by using the fingers to apply pressure on top of the glass in spots that at first received less grinding. Once an even surface polish was achieved for a given grinding paper, the next grinding paper was used. The following silicon carbide grinding papers from Beuhler were used: P1200, P2500, and P4000, in order of polishing from coarser to finer. After the finest grinding paper, the Cu was blown dry using nitrogen gas, put in a new plastic bag and sealed. Then followed the punching and cleaning of electrodes, as described in subsection 4.1.1. in my Master's Thesis (<https://odr.chalmers.se/items/7f321cb1-4a2e-49e5-9a2c-8b06874e3bd0>).

## B Assembly of the I-cell and pressure calculation

Described below is the assembly of the I-cell (what I call the metal two-electrode Swagelok-type cell) and the calculation of the applied pressure due to the compressed spring.

Figure 10 shows the parts of the I-cell. I will refer to the parts as the "lower" and "upper" as its position with respect to the main body in Figure 10. For example, the upper metal rod is not the same as the lower metal rod.



**Figure 10.** (a) Parts of the I-cell and (b) the assembled cell.

First, a nut with its Swagelok sealing rings is threaded onto the main body. Then, the lower metal rod is inserted and the nut tightened fast by hand. This results in Figure 11. Since all



**Figure 11.** The lower metal rod is fastened to the main body and one nut.

parts of the cell are metal, at this stage it is crucial to confirm that the lower metal rod and the main body is electronically isolated. Using a multimeter on the resistance setting, the resistance between the lower metal rod and the main body is measured. I recommend first measuring the resistance with the multimeter probes both touching first The main body (there should be very little resistance), and then both touching the lower metal rod (there should be very little resistance). Now, probing the lower metal rod with one multimeter probe and the main body using the other multimeter probe (there should be a very high resistance, maybe the multimeter says "-1" or something, indicating that the resistance is too large to be measured). However you do it, make sure there is no electronic contact between the lower metal rod and the main body.

Placing the assembled parts thus far into the green holder shown in Figure 12 makes the rest of the assembly easier and minimizes the risk of short-circuiting. Next, a piece of Cu is placed polished side up in the lower metal rod followed by a separator. Then the Cu and separator is centered in the cell. I do this by rotating the cell where it sits in the hole in the green holder, constantly looking at how the Cu and separator "wobbles" around the center. Then I use the tweezers to carefully push the Cu and separator in towards the center until I can rotate the cell with minimal "wobble".

Next, Li is cut and punched and placed on the spacer (that goes into the hole in the lower metal rod). Using the polypropylene (PP) sheet, the Li is centered by eye on the spacer and then flattened by pushing the PP sheet down on it. After the electrolyte is added as centered on the Cu as possible, the spacer with the Li is put down into the lower metal rod and some pressure is applied for a second and then released. Then follows the spring on the spacer, and the upper nut is placed and threaded onto the main body with its Swagelok sealing rings. The only part left is the upper metal rod (on which I usually write "Li" on to know what end is which). Before putting in the upper metal rod, I prepare the calipers by setting its measurement to 110.2 mm.

I aim for the I-cell to have 110.0 mm final total length ( $\Delta x$ ) and since the cell usually becomes a little bit compressed when tightening the upper nut, I compensate by a longer length in the calipers. When I'm ready, the last metal rod is put down such that it rests on the spring. Then I pick up the whole cell and the calipers (holding them vertically in the air), carefully place the lower metal rod onto the lower jaw of the caliper (that has isolating tape on it), compress the upper metal rod into the cell until it is below the upper jaw of the caliper (which also has isolating tape on it) and then release. It looks sort of like in Figure 13 Finally, while the cell is in between the caliper jaws, the upper nut is tightened until it fixes the upper metal rod in place at which point the caliper is removed. Then the cell is tightened one final time by holding one hand on each nut and tightening as hard as I can manage. The cell is labeled and put into a large plastic bag (as to prevent an accidental short-circuit).

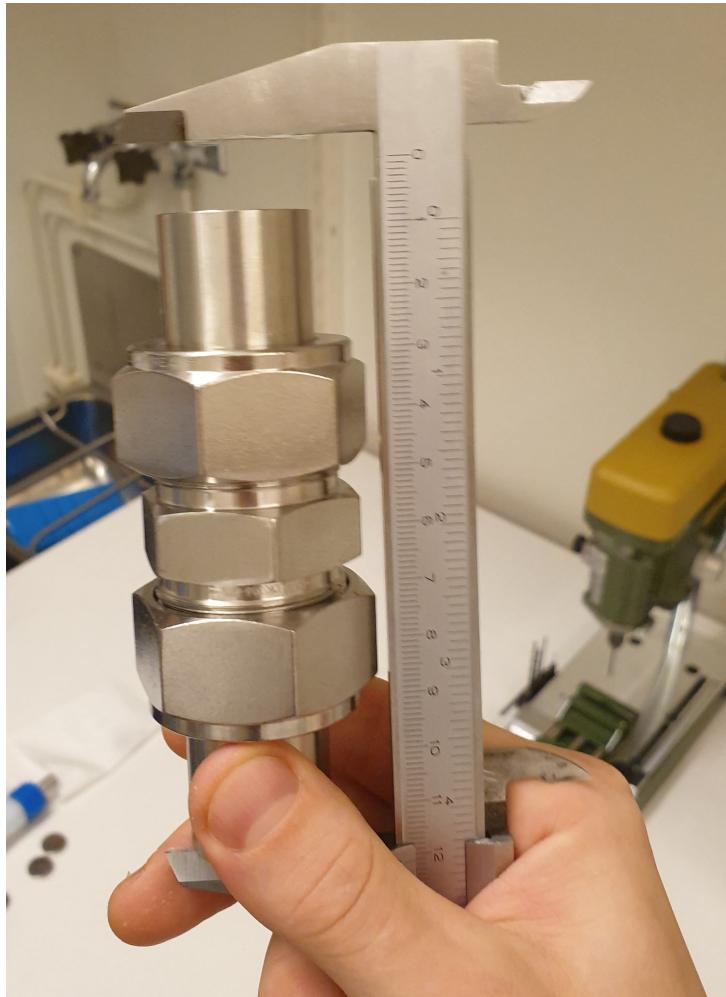


**Figure 12.** Half-way through the assembly. Placing the lower part of the cell into the green holder makes the assembly process easier.

There is applied pressure between the electrodes due to the spring being compressed, exerting a force on the spacer towards the lower metal rod. The pressure in the cell from the spring is given by

$$P = \frac{F}{A} = \frac{k\Delta x}{A}$$

where  $F$  is the force due to the spring, with spring constant  $k$ , being compressed a distance  $\Delta x$  from equilibrium acting on an area  $A$ . The full length of the I-cells – where the spring is not compressed ( $\Delta x = 0$ ) – was measured to be 120.0 mm (measured with calipers) and since the Cu is about 0.1 mm and the Li roll has a measured thickness of 0.2 mm (measured using a micrometer)



**Figure 13.** During the final steps of the assembly, the I-cell is placed between the jaws (with isolating tape on them) of the caliper. In the photograph, I am holding the cell and caliper with the right hand. During assembly in the glove box, the cell is held with the left hand, the caliper in the right hand and the upper nut tightened using fingers of the left hand. (This was not possible to do while taking a photograph). (There should not be a gap between the cell and caliper jaw either).

screw), the total length of the assembled I-cell without any pressure is  $L_0 = 120.3 \text{ mm}$  (ignoring the separator thickness of  $0.025 \text{ mm}$  which is smaller than the uncertainty in  $L_0$ ). Next, the spring constant was measured to be  $k = (520 \pm 14) \text{ N m}^{-1}$  with data from 11 measured springs (the plastic bag of springs measured are marked with this data). So, all in all the pressure due to the spring can be calculated by measuring the assembled cell length  $L$  (from which  $\Delta x = |L_0 - L|$ ) is given by

$$P = \frac{k|L_0 - L|}{\pi (d/2)^2} = \frac{4}{\pi} d^{-2} k |L_0 - L|$$

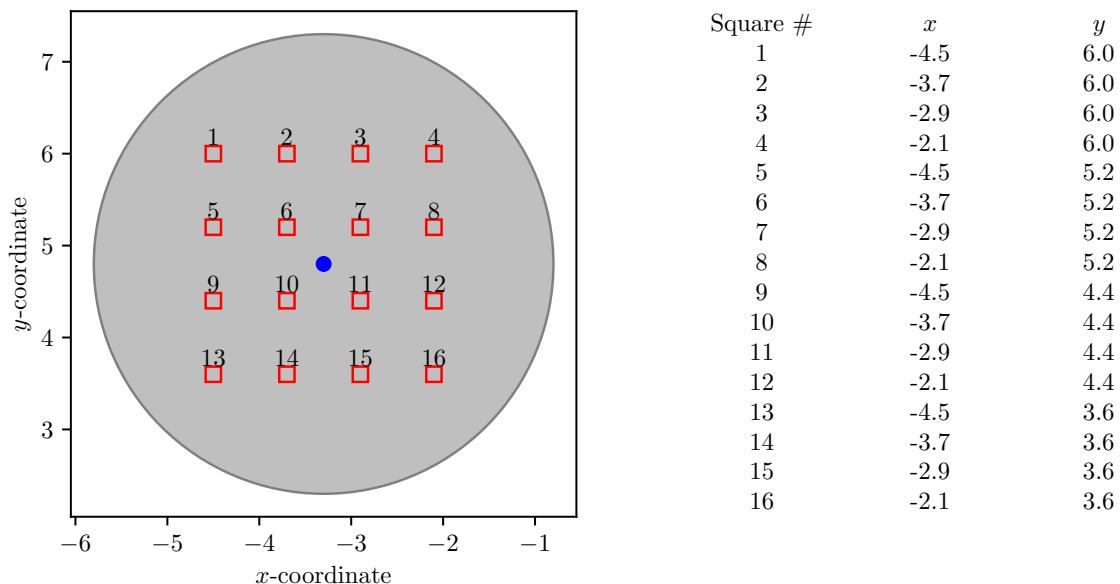
where we assume a circular area with diameter  $d$  for the pressure to be calculated on. Putting in our measured quantities, we get

$$P(L, d) = \frac{520 \text{ N m}^{-1} |120.3 \text{ mm} - L|}{\pi (d/2)^2} = 662 \text{ N m}^{-1} \frac{120.3 \text{ mm} - L}{d^2}$$

which gives the applied pressure from the total length  $L$  of the assembled I-cell and the circular disk of interest (Li or Cu) with diameter  $d$  on which the pressure wishes to be calculated.

## C Acquisition of SEM images in a grid for analysis

The acquisition of SEM images for image analysis is described below. I reasoned that for good statistics, *at least* 100 particles should be analyzed per sample (Cu surface). To aid the analysis, the particles should be images with good resolution. An experience-based guess is that about 20 to 40 particles per SEM image gives sufficient resolution, while still having relatively many particles. With around 30 particles per image, one could take ten SEM images to reach 300 particles per sample. The SEM images should be representative over the deposited area. I therefore suggest that the SEM images are taken in a grid on the Cu surface, for example in a 4 by 4 grid as shown in Figure 14. For reproducibility, the grid should be centered on the Li deposition, and the images be taken with some distance  $d$  (in either the  $x$ - or  $y$ -direction) apart. In Figure 14,  $d$  has been chosen to be 0.8 mm which seemingly samples the SEM images in locations 'far' apart in the Li deposition, while keeping all the sampled parts well within the Li deposition. A too small value of  $d$  will not necessarily give a good average of the particle size distribution over the deposited area, and a too large  $d$  could result in the edge positions (square 1, 4, 13, and 16) being outside the deposited area of Li. With about 30 particles per image and 16 SEM images, the total number of particles per sample would be about 400.



**Figure 14.** Illustration of the positions (red squares) around the center (blue dot) of the deposited Li (gray circle). SEM images are taken at each of the numbered red squares.

Some comments about Figure 14. The SEM instrument can provide the  $x$ - and  $y$ -coordinates of where currently imaged position (in absolute and relative coordinates). After deposition, there will be a Li circle 5 mm in diameter on the Cu (10 mm in diameter), which is illustrated as a gray circle. To find the center of this circle, I have manually looked for the top edge, moved down until I reached the bottom edge of the Li deposition and taken the average to get  $y_0$ . Doing the same but with the left and right edge, I get  $x_0$ . Then, I move to the position of the manually determined center  $(x_0, y_0)$  and use relative coordinates in the SEM to move between the grid positions. I wrote a script in Python<sup>7</sup> which returns the relative movements for this sampling given a center point  $(x_0, y_0)$  and the value of  $d$ .

<sup>7</sup>See: <https://github.com/GottfridOlsson/SUMMER-JOB-2024-MATERIALS-PHYSICS>.