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Thin Film Thermocouple Fabrication Procedure

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Abstract

This report presents the recipe for fabricating a thin film Ni-Cr thermocouple on a GaAs substrate. Concepts and the technique of using thermocouples for temperature sensing are explained, along with a detailed recipe for their fabrication. Photolithography was performed in Northwestern University's Micro/Nano fabrication facility NUFAB; however, the steps and techniques described can be performed in any microfabrication facility which has the required equipment.

Keywords

Thin Film Thermocouples, Thermocouple Fabrication, Ni-Cr Thermocouples

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1 Introduction

In this report, we present the recipe for fabricating a thin film Ni-Cr thermocouple on a GaAs substrate. First, we describe in brief the concepts and the technique of using thermocouples for temperature sensing [1][2]; then we present a recipe for their fabrication followed by details of the steps involved. Photolithography was performed in Northwestern University's Micro/Nano fabrication facility NUFAB [6]; however, the steps and techniques described can be performed in any microfabrication facility which has the required equipment.

1.1 Thin-Film Thermocouples

Moore scaling without Dennard Scaling has led to increasing power densities in high performance integrated circuits. These higher power densities lead to high operating temperature and thermal gradients in the VLSI chips. These thermal issues impact system reliability and performance. Architectural techniques which react to temperature changes require accurate on-chip temperature sensing of hotspots. In this report, we describe a method of on-chip temperature sensing using the Seebeck Effect in bimetallic thin-film thermocouples [1][2][5][10]. The method is compatible with existing fabrication processes.

The thermocouple metals used in this report are Nickel and Chromium, chosen for their high Seebeck Coefficients. Nickel has a bulk Seebeck coefficient of $-19.5 \mu\text{V}/\text{C}$ and Chromium's bulk Seebeck coefficient is $21.8 \mu\text{V}/\text{C}$. This produces a bulk Seebeck coefficient of $41.8 \mu\text{V}/\text{C}$ for the thermocouple. The thermocouple is fabricated on a semi-insulating GaAs substrate. GaAs was chosen as the semiconductor substrate due to its high resistivity and easy cleaving into thin strips.

The thermocouples function according to the Seebeck effect. The Seebeck effect is found when a temperature difference occurs between two open-circuit junctions of dissimilar conductors and is directly converted into a thermomotive force:

$$V_{\text{HC}} = \alpha (T_{\text{H}} - T_{\text{C}})$$

where V_{HC} represents the Seebeck voltage between the leads, α is the Seebeck coefficient, T_{H} and T_{C} are the temperatures at the hot and cold junction respectively as shown in Figure. 1 [1]. Seebeck voltage is measured between leads at T_0 .

The Seebeck effect has been reported experimentally and investigated theoretically to depend on the thickness of a thin film. [3][4][12-19]. The thickness dependence arises when the film is of order or smaller than the electron mean free path such that boundary scattering starts to compete in magnitude

with bulk scattering. The difference between the thin-film Seebeck coefficient and that of the bulk material is inversely proportional to the metal film thickness.

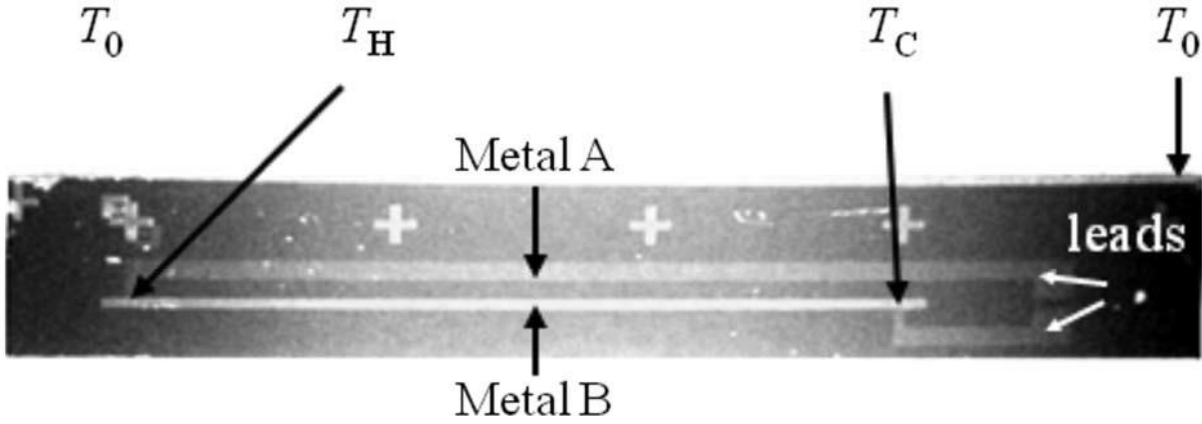


Figure 1: Microscope photograph of 50-nm thin-film thermocouple

The thermocouples are fabricated using the principles of positive photolithography. For this report, we performed photolithography in the NUFAB sodium light room. In this report we use positive photoresist i.e. the exposed photoresist becomes soluble in the developer; although the mask allows negative photolithography as well. The overall structure and the order of fabrication are as follows: first step is the deposition of the NiCr heater layer, then of the Aluminum leads, then the oxide insulation layer, then the Chromium leg of the thermocouple, and lastly the Nickel leg of the thermocouple. After fabrication, the thermocouples are calibrated using small-area resistance temperature dependent thermistors which are epoxied with heat conducting silver epoxy on top of the metal junctions.

The reproducibility of this recipe and the passive nature of thermocouples which require no power, make this technology an attractive solution. The thermocouples can be scaled down and routed along arbitrarily shaped paths in dedicated metal layers on top of a processed integrated circuit using existing CMOS fabrication methods [1].

1.2 Mask for Photolithography

For this recipe, we use the mask shown in Figure 2. It corresponds to the Mask-4 in reference [2]. It is a two-terminal thermocouple mask with an option for negative photolithography on GaAs. The patterns outside the black rectangle are for negative photolithography, and those inside are for positive. The sample size is 1.5cm x 1.2 cm.

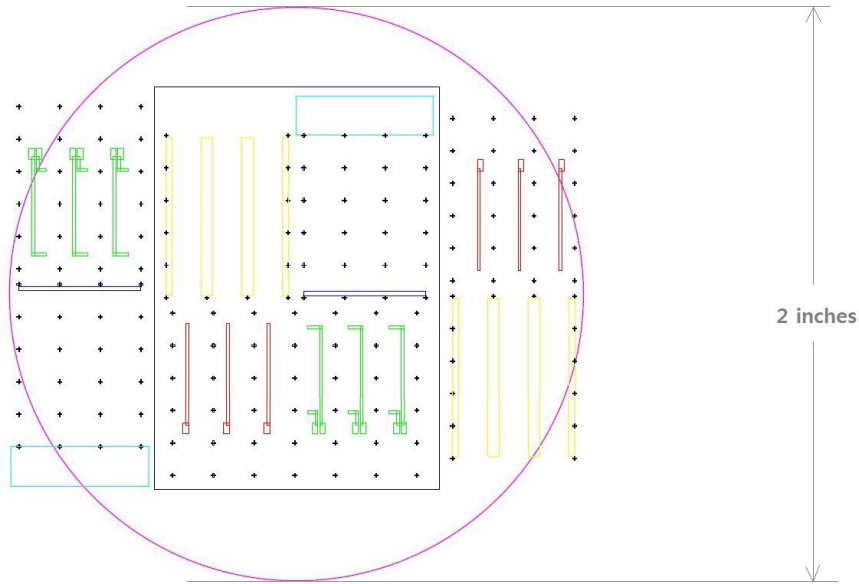


Figure 2: Mask for photolithography

The red rectangle in figure 3 highlights a single device from the batch of three devices shown. It has five layers: a NiChrome heater, aluminum heater leads, SiO₂ insulating layer, chromium leg and a nickel leg of the nickel-chromium thermocouple. Figure 3 shows the overlaid patterns on the mask showing the five different layers in purple(Cr), green(Ni), blue(NiCr), yellow(Al) and cyan(silicon dioxide).

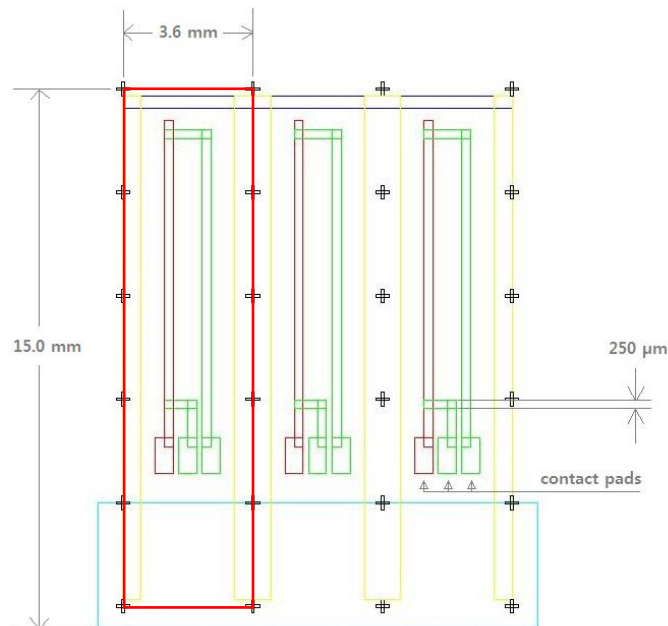


Figure 3: Overlaid patterns on mask

2 Fabrication Procedure Overview

In this section, we list the steps involved in fabricating a Nickel Chromium thermocouple.

2.1 Fabrication steps

- 1) Clean sample.
- 2) Fix sample to glass slide using photoresist.
- 3) Spin photoresist AZ5214E on the sample at 4000RPM for 50 seconds.
- 4) Pre-bake at 91 ° C for 50 seconds.
- 5) Align mask appropriately with sample then expose with UV.
- 6) Develop with AZ400K diluted 1:4 with water for 20-25 seconds.
- 7) Deposit Metal layer or SiO₂ layer through evaporation or sputtering in order*
- 8) Lift-off.

Repeat steps 1- 8, with step 7 in the order and thickness specified below:

*Metal deposition order and thickness:

- 1) NiCr: 100 nm
- 2) Al: 90 nm
- 3) SiO₂: 160 nm (5400 sec, 200W)
- 4) Cr: 50 – 200 nm
- 5) Ni: 50 – 200 nm

Details on these steps are in section 3 of this report.

2.2 Materials required

For fabricating thermocouples:

1. **GaAs substrate (University Wafer):** 4" dia, undoped SI (100), 625 μm, P/P (polished both sides). 20 – 25 device batches per wafer.

2. **Thermistor (US Sensor PPG102C1RD) or thermocouple (Nanmac A13-1):** Two required per sample.
3. **Thermally conductive epoxy (Arctic Alumina Thermal Adhesive):** For heat-sinking.

For testing:

1. **PCB integration:** Electrically conductive epoxy (MG Chemicals 8331S-15G).
2. **DIP integration:** 16-pin DIP.

2.3 Fabrication equipment:

Available in NUFAB/ EPIC:

- **Karl SUSS MA-6 aligner:** It is a front and back side contact lithography printer used for fine line lithography with precision up to 1 micron.
- **AJA sputtering machine:** It deposits metal and dielectric films over a substrate up to 6" in diameter. It is fitted with three 3" diameter sputter guns that are powered by RF, DC, and pulse sources and can be used for single or multi-layer deposition. It is controlled by a computer which provides for recipe generation and process data storage.
- **Spin-coater:** It is used to spin coat photoresists on wafers to evenly distribute them. The spinner can be programmed to ramp up to a specified RPM and for a specific time. The properties of the photoresist and its desired thickness on the wafer determine the RPM and time of spinning.
- **Dektak 8 profilometer:** It measures the feature height of the samples by scanning over it with a stylus.
- **Nikon LV150:** It is an upright reflected light microscope.
- **Lesker Nano38 thermal evaporator:** For depositing metal on the sample.

3 Detailed Fabrication steps

In this section we describe in detail the repeating steps involved in fabricating the Nickel Chromium thermocouple.

3.1 Initial Setup

- We do photolithography in the NUFAB Sodium light lab clean room.
- Take clean room wipe and place sample box on it. Fix the clean wipe room to the table by placing beakers on the corners. Clean tweezers.
- Fill the Acetone and ISP bottles; keep them accessible.
- Set heater-1 to 100 ° C and heater-2 to 91 ° C.



Figure 4: Initial Setup

- View sample under a microscope and choose a side you will be holding the sample from. Always hold the sample from that side so as not to dirty the other sides.
- Make sure your hand is underneath the sample when moving it at all times to prevent it from falling down.
- Calibrate the film thickness produced by the thermal evaporator and sputtering machine before use by depositing on one-half of a dummy sample of silicon or GaAs; and then measuring it using a surface profilometer.
- If evaporation cannot be performed on the same day after photolithography, leave sample in the cleanroom.

3.2 Cleaning the sample:

- Cleanliness of the sample is of utmost priority, and it might not always be possible to clean the sample thoroughly if it is contaminated. So be careful when handling or moving it.
- Hold the sample with tweezers. Spray acetone on the sample thoroughly. Then quickly before the acetone evaporates, spray ISP on the sample washing off the acetone. Then rinse sample thoroughly with DI water.
- Dry with the N2 gun aiming perpendicular to the sample. Hold the sample so the tweezers are below the sample.
- It is important not to let acetone evaporate from the sample as it leaves a white residue, which can be hard to clean.
- Try moving to the next stage of the fabrication procedure right after cleaning without having to place sample down again.

3.3 Pipette Photoresist:

- We use the photoresist AZ5214E.
- Take a pipette and clean it using the N2 gun, always making sure nothing touches the tip of the pipette. Fix a bulb to the pipette.
- Take photoresist out from the fridge, open cap and tilt it so that the level of the photoresist is visible and near the neck of the bottle as shown in Figure 5. Press bulb, of the pipette outside the bottle, immerse tip of pipette in the bottle, then release bulb slowly and take the pipette out without touching the sides of the sample. Make sure no photoresist enters the bulb of the pipette.
- Twirl the pipette to remove the bubbles.



Figure 5: Pipette Photoresist

3.4 Fix sample to glass slide:

- We fix a glass slide under the sample to have better vacuum when we need to hold the sample in equipments using vacuum chucks. Also with the glass slide, we do not have to touch the sample when moving it or to attach it using other kinds of chucks, and can fix it using the glass slide.
- Put a drop of photoresist on a glass slide, just enough to spread under the entire chip.
- Spread the photoresist on the slide using a pick.
- Put the sample on the photoresist and align to leave a larger area on one side and even distance on the other sides. Put the glass slide on the 91 ° C heater for 6 minutes.
- This fixes the sample on the glass slide.
- The glass slide can be removed by immersing the sample in a beaker filled with acetone.

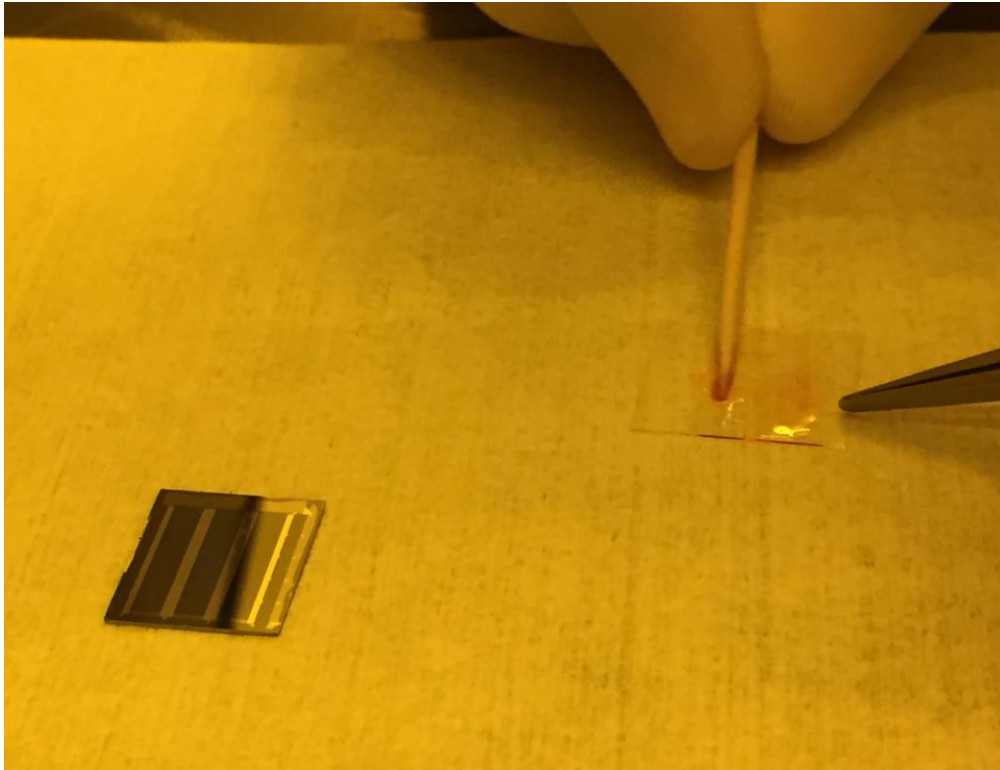


Figure 6: Spread photoresist on slide

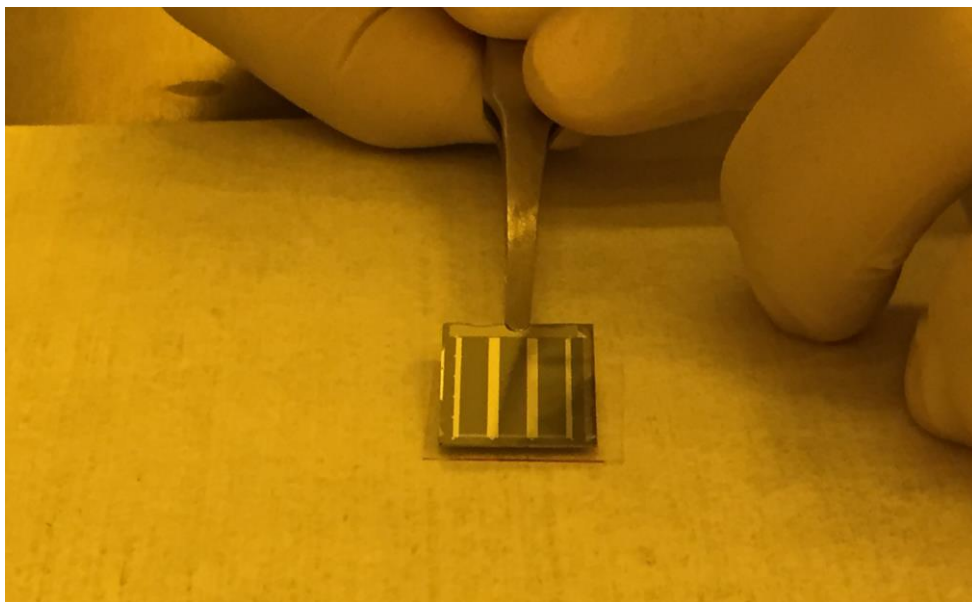


Figure 7: Place sample on the photoresist covered slide

3.5 Spread photoresist using spinner:

- We put photoresist on the sample and then spin it to distribute it evenly.
- Clean sample holder; resembling either Figure 8 or 9; with acetone then dry it with N₂.
- Fix it in the spinner.
- Spread aluminum foil in the spinner as shown in figure 10.
- Place glass slide with sample on the sample holder. Center it so that the center of the sample coincides with the center of the holder.
- Press Vacuum ON by clicking *HOLD*.
- Check to make sure it is fixed properly and centered.
- Then take photoresist in a pipette and start covering the sample with it. Make sure to remove the bubbles first; cover the sample fully and as evenly as possible with the photoresist. Uniformly cover the area of the sample with drops of photoresist and wait for it cover the sample Figure 11. Fill any uncovered areas.
- Be careful sure not to touch the sample with the tip of the pipette.
- Load appropriate program then click start centering (for NUFAB the program is *BOYA4000*). This program rotates the sample at 4000RPM which gives an adequate thickness of photoresist on the sample.
- Check that the sample is centered.
- Close the spinner hood and start the program. The program takes 50 seconds.

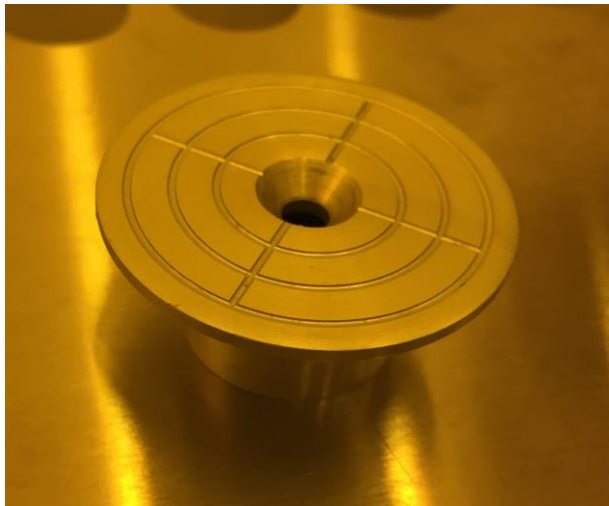


Figure 8: Spinner chuck 1



Figure 9: Spinner chuck 2

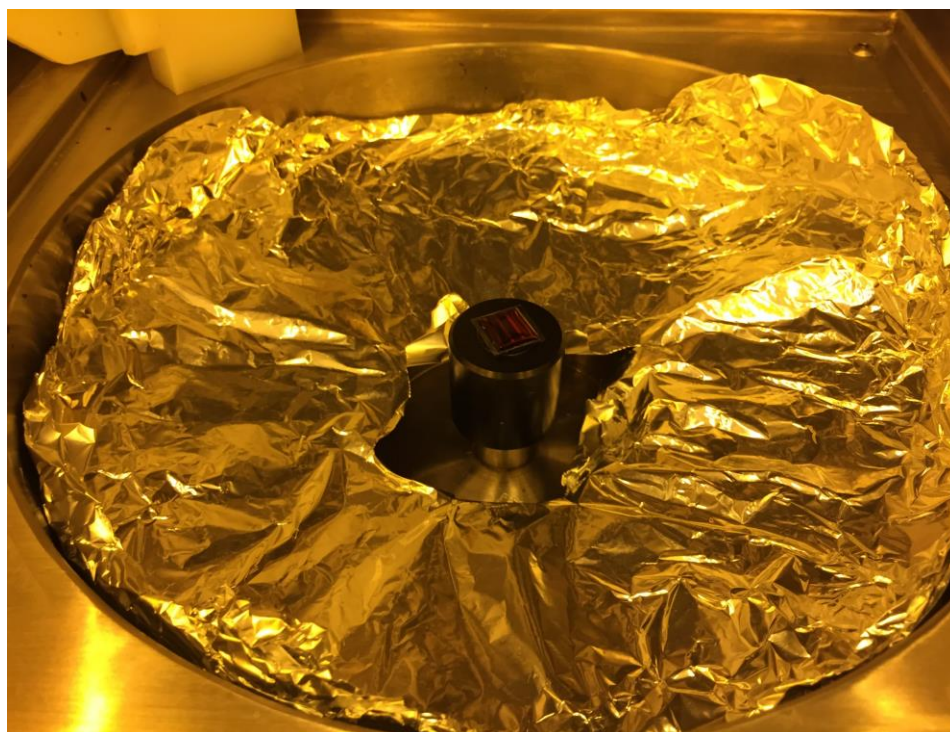


Figure 10: Inside of the Spinner hood

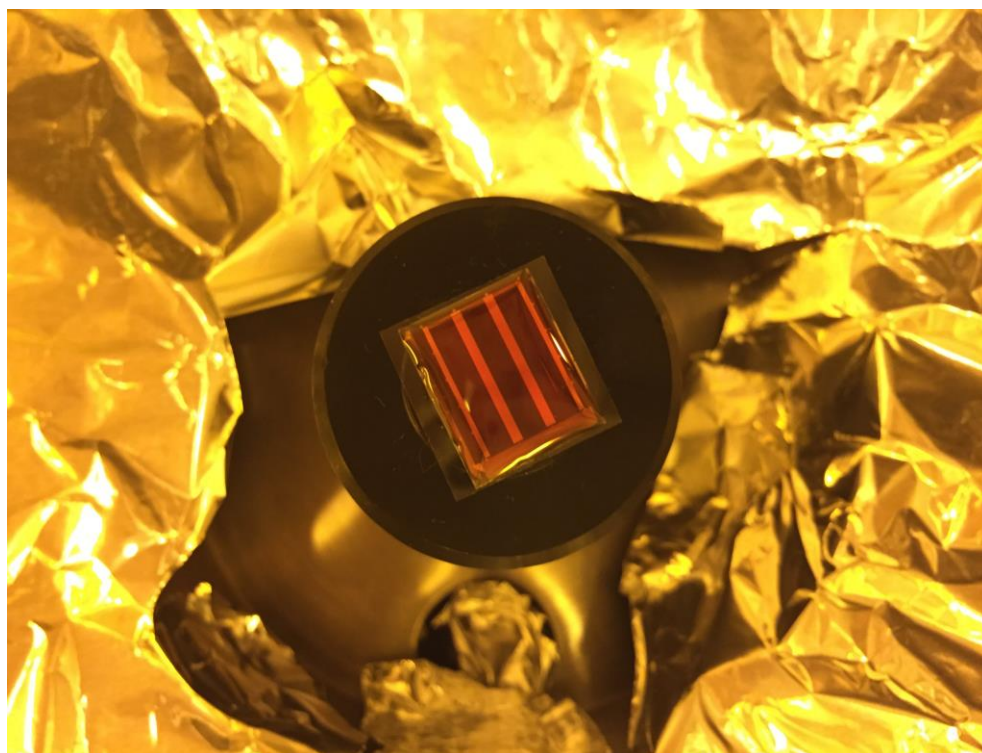


Figure 11: Sample in spinner covered with photoresist

3.6 Prebake the sample

- Put the photoresist covered sample on the heater at 91 ° C for 50 seconds.

3.7 Align mask and sample:

- Now we will align the mask with the sample and expose the sample to UV.
- Turn N2 supply on (perpendicular to the pipe is off).
- Switch the lamp power on, then switch CP ON, then press Start.
- Wait for ~10 minutes till the lamp says “idle”.
- Now turn on the MA6 aligner by slightly turning the ON switch.
- Wait till screen says “load”, then press “Load”.
- Wait till it says “load sample” on the screen.
- Change program to 8 by pressing “edit program”; change options using up/down arrow keys to select option 8, then press “edit program” again.
- Press “edit parameter”, increase exposure time to 4 seconds, change air gap to 80μm, press “edit parameter” again to confirm.
- Now press “Change mask”.
- Put the mask on the tray (orange/chrome side will be up) release the clam, then press “enter”. Vacuum should be ON now. Check that the mask is firmly fixed to the chuck (Figure 13).
- Lift mask tray, invert it keeping one hand under the mask for safety and insert it under the microscope making sure it goes all the way in.
- Press “change mask” again; mask holder should be fixed in place now.
- On screen it should say “Load sample”.
- Drag out sample tray and replace sample holder with one of appropriate size.
- Fix sample on holder with a small drop of photoresist.
- With tray still out Press “load”.
- Press “enter”, check vacuum between sample and chuck, push tray inside.
- At this stage make sure program is not set to flood exposure.
- If system warns of vacuum loss: Check to make sure the sample is secured on the chuck; if you are confident the sample is secure you may ignore the warning and press “enter”.
- Press enter (sample moves up till it touches mask).
- Wait till it says align substrate.
- Turn off the BSA microscope, then press “F1” then “enter”, use arrow keys to move microscope.

- Microfocus knob is on the top.
- If the position of the sample is beyond the reach of the XY planes, we need to unload the sample and move it manually.
- Press “unload” then wait till it says “unload” on the screen, remove sample chuck, move sample on the sample holder appropriately then load it back.
 - Now we can align: **We align by moving the sample, the mask is stationary.**
 - Align using the round knobs on the right and left side.
 - Try to locate a corner of the sample to get an idea of your location w.r.t. the sample.
 - Transverse the corner all the way to its opposite corner. Bring sample within one alignment mark of the mask.
 - Align one alignment mark of the sample with its corresponding mark on the mask.
 - Now move to the next alignment mark horizontally and check if sample needs to be rotated clockwise or anti clockwise. Rotate the knob accordingly.
 - Repeat alignment steps till satisfied.
- Once you are satisfied with the alignment, press “alignment check”.
- Sample moves up and presses against mask. Now you should be able to reasonably focus on both the mask and sample and get a better sense of the current alignment.
- At this stage, with the contact light ON don’t move the sample as it is touching the mask and moving it will cause the mask and sample to scratch.
- If alignment is not satisfactory, press “alignment check” again and realign.
- Once satisfied with the alignment, press “alignment check” again.
- Press “exposure” (sample moves to alignment check position if not there already).
- Wait and look away till sample is exposed.
- Once exposure is finished, pull sample slide all the way out, it clicks.
- Press “change mask”, pull mask tray out and place safely on a side table, press “enter”. Now vacuum is off; place mask back into the container.
- If required: To do flood exposure press “Edit program” then select option 2 - flood exposure, then press “edit parameter” change parameter time to 60s. We do not have to align for this, just press “load” then “expose”.



Figure 12: Karl Suss MA6 mask aligner

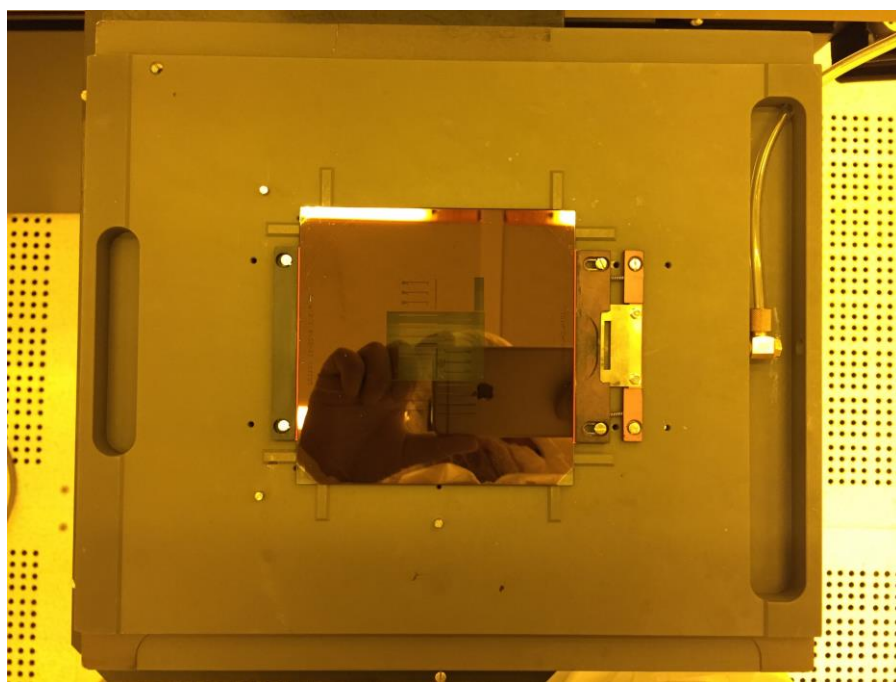


Figure 13: Mask attached to chuck

3.8 Developing

- In this stage the part of the photoresist on the sample which was exposed to UV is removed.
- We work in the acid develop hood which has storage space and disposal facility for developers and their waste.
- We use the developer AZ400k diluted 1:4
- Hold the sample and immerse it in the developer and agitate for 20 seconds.
- Immediately after developing, immerse the sample in DI water.
- Dry with N2 gun.



Figure 14: Developing setup

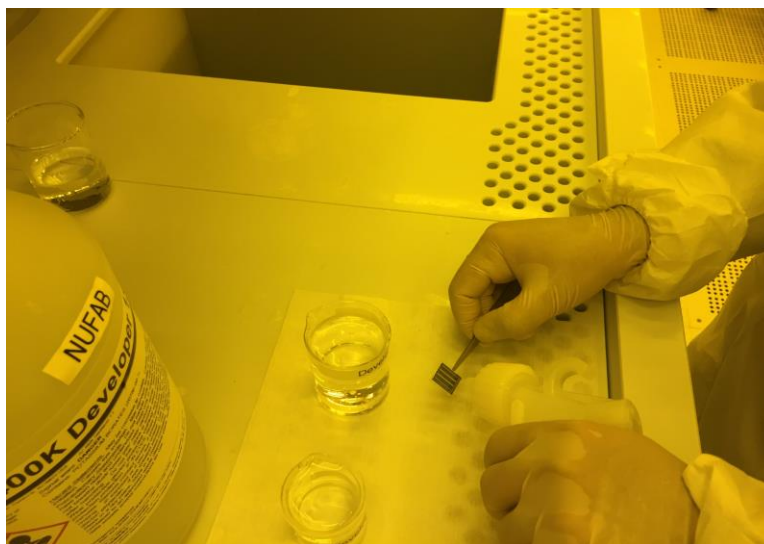


Figure 15: Dry sample with N2 gun

3.9 Metal deposition through evaporation

Reference [7]

System Startup:

- Open the Nitrogen tank valve to the right of the evaporator (100 psi)
- In the Vacuum screen, select Start PC Vent on the right-hand side. When the system is done venting you can select OK on the pop-up window, and open the chamber door. Optional: Under the Deposition menu select Source Shutter 1 on the left. This will move the shutter out of the way if you are adding metal.
- Remove the appropriate metal boat with a hex screw, weigh it, then add metal, weigh the boat again and place it back in the chamber. Note how much the metal you added weighed. The boats are in order from left to right, 1-3. Boat 1 is for Chromium and Boat 3 is for Gold. If you are coating a different metal, please use the center boat holder (Boat 2). If you need to add metal, you should add approximately 10 pellets.
- Remove the round sample holder at the top of the chamber and place your sample into one of the metal clips. Figure 17.
- Place the holder with your sample back in the chamber and make sure it clicks in place.
- Close the chamber door. If you opened the source shutter, close it by selecting source shutter 1 on the Deposition menu.
- Select Start PC Pump on the right-hand side of the screen.
- Select the Sigma menu. If the Sigma software is not currently running in the task bar, you will need to open the software by selecting Sigma Launch 242 on the left-hand side of the screen (select Off to turn it ON). Click OK instead of entering a username and password.
- Open the Sigma software. Select Edit then Films. Here you can select an existing film, or create a new film. If you need to create a new film, please see Ben or Kate.
- Select the appropriate film and then Close Form on the left-hand side of the screen.
- Select Edit and Process. Select the appropriate process/film.
- Under the Layer tab select the film you are using and select the appropriate thickness.
- Under the Source Sensor tab choose the appropriate material. This menu is also where the tooling factor is set. There should just be one tooling factor set, and that is for sensor 1. The max power should never be above 65%. Slew rate 100.00 16. Under the Error menu only crystal fail enabled should be checked.

Coating:

- Go back to the KJLC software.
- Under the Deposition menu, make sure the Shutter Source 1 is closed, and also select the appropriate Boat/Source by clicking on it to highlight it.
- Turn on the Power Supply by clicking ON. Enter a power supply of 30% and get the dark glasses to watch for the metal melting in the boat. After a few minutes if the metal is not melting, you can increase the power supply by increments of 5% and waiting to see if the metal is melted. Au should melt at 30% or 35%.
- When the metal is melted you are ready to turn on the process you created and start coating. To do this you need to enter the Sigma portion of the KJLC software, and make sure that the Signal process name in green type matches the sigma control request film in white type.
- Select Sigma start process ON, and the system will run through the process and apply the desired coating.
- When coating is finished, in the Deposition menu turn the Power Supply Off, and the Boat power Off. Under the Vacuum select PC Vent.
- When the system is done venting click OK. Wait to remove samples as the system is very hot.
- Remove your sample, and remove the boat you used. You can move the source shutter to do this.
- When the boat is placed back in the chamber and the source shutter is closed you can pump the chamber down by selecting Start PC Pump.
- Leave the chamber pumped down.



Figure 16: Inside evaporator

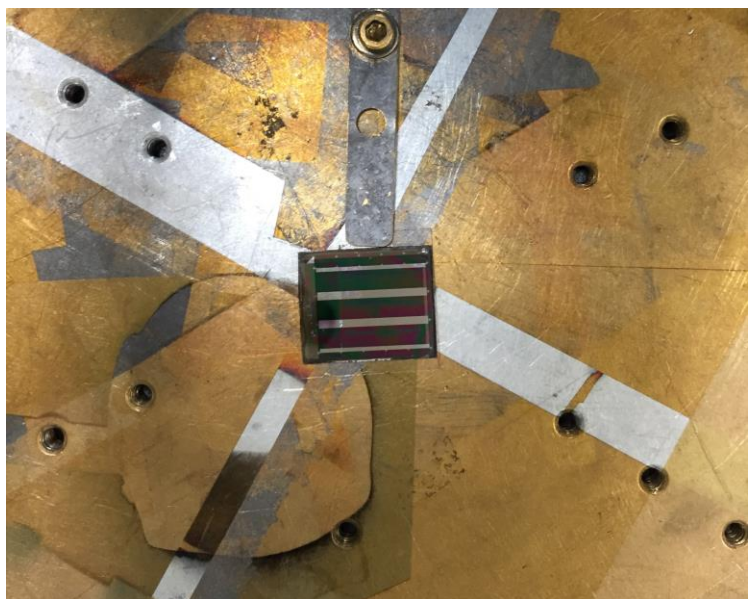


Fig 17: Evaporator chuck

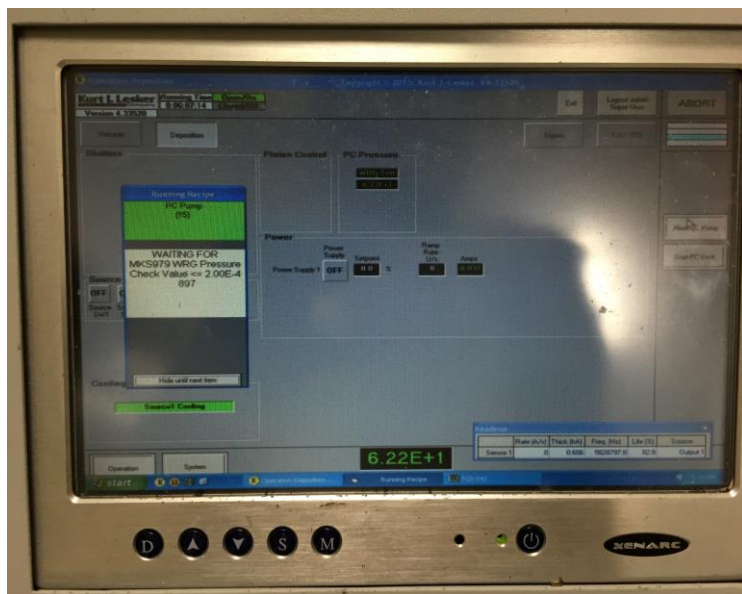


Fig 18: Lesker Nano38 GUI

3.10 HCl dip

- After the deposition of the Chromium layer, surface oxidation of Chromium may occur before Nickel is deposited. If chromium oxide is formed at the junction where Cr and Ni meet the surface might lose its smoothness and the junction will become resistive.
- To prevent this we perform a Hydrochloric (HCl) acid dip of the sample.
- Dipping the sample in HCl causes the proton from HCl to bond with the oxygen atoms from the chromium oxide thereby removing the oxidized layer from the junction.
- Dip the sample in the acid for 5 seconds.
- Blow-dry the sample using the nitrogen gun right afterward.
- Use extreme precaution when handling the acid.
- Dispose of the acid following the lab's guidelines.

3.11 Sputtering

Refer [9]

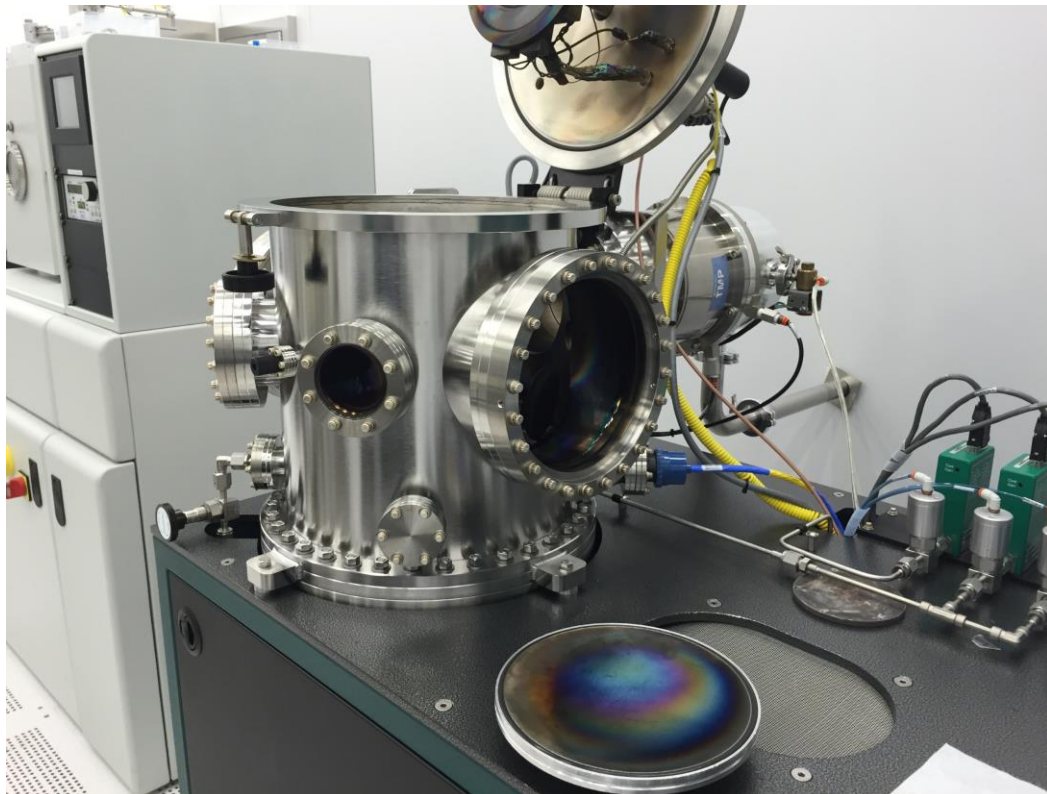


Figure 19: AJA Orion Sputtering Chamber

- We deposit the SiO_2 layer using the sputtering machine.

- Select 4" or 6" substrate holder keeping in mind that 4" holder can be heated up to 850°C while 6" holder can be heated only up to 400° C.
- Mount the substrates using screws and clips.
- Vent the chamber. All gases and plasma must be off before attempting to vent the chamber as displayed on laptop screen.
- When the Convectron reads about 760 Torr (atmosphere), the substrate transfer door can be opened and the manual vent valve should be closed.
- Transfer the Substrate to the Chamber.
- Open the top lid of the chamber and replace the target with SiO₂.
- Pump Down the chamber to 10⁻⁷ torr.
- Deposition of the metal or dielectric can be done in a manual mode or by running a recipe. We will use recipe BOYA which gives us the required thickness of 160nm for SiO₂.
- Once the recipe ends, vent the chamber, remove the substrate and leave the chamber in pump down state.



Figure 20: AJA Orion Sputter system

3.12 Lift off:

- This is the last stage of a layer's deposition process. In this stage, the metal deposited over the photoresist is removed leaving behind the desired pattern.
- Clean beakers with acetone and ISP.
- Fill a flat beaker with acetone.
- Pick sample with tweezers and place in the acetone filled beaker.
- Wait for the photoresist to dissolve leaving behind the sample.
- You may use the ultrasound cleaners to speed up the process.
- The sample is separated from the glass slide at this stage.
- Check under microscope to make sure the lift off is complete. You may do a precursory check even with the sample still under acetone.
- Remove the sample from the acetone beaker then clean it following steps in section 3.2.
- Check sample under microscope to inspect deposition.

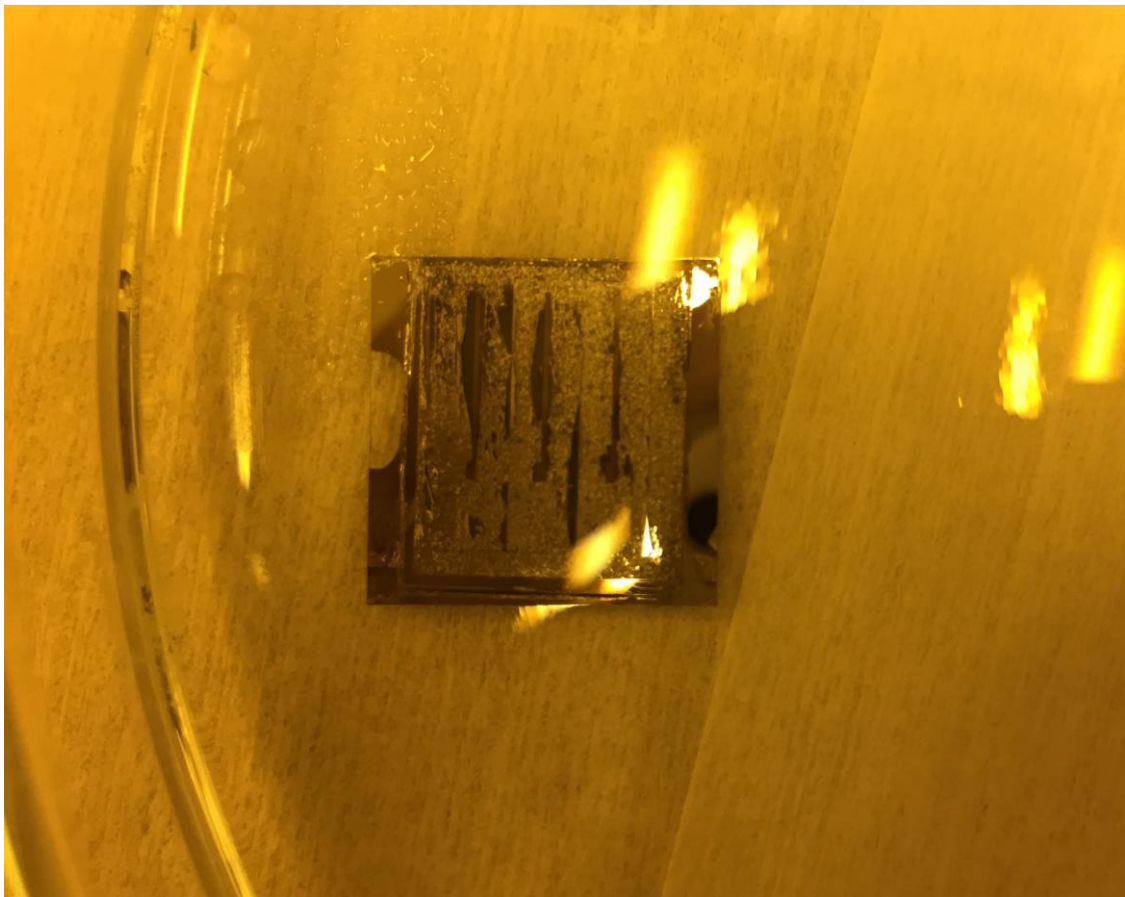


Figure 21: Lift off

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