

Procedure for Producing Alignment Markers on SiO₂/Si Wafers Using Photolithography

DUKE UNIVERSITY
QUANTUM MATERIALS AND DEVICES LAB
SMIF CLEANROOM PROTOCOL

PREPARED BY DAVYD KASPEROVYCH-PUSTOVIT
REVISION 3 - JULY 30, 2025

Contents

1	Cleaning the Wafer	2
2	Spin Coating	2
3	Photolithography	3
4	Developing	3
5	EB Evaporation	3
6	Photoresist Removal	4
7	Expected Quality of Alignment Markers	4
8	Optional: Post Sealing	5

1 Cleaning the Wafer

1. At the spin coat hood in the yellow room, turn on two hotplates. Set one to **115°C** and the other to **135°C**. They will be used later.
2. At the solvent hood with ultrasonic machine, prepare:
 - (a) **Acetone**
 - (b) **IPA**
 - (c) **DI water**
 - (d) Ensure access to **hydrogen gas**.
3. Set the ultrasonic machine knob to **position 5**. If the bath is empty, fill it with **IPA**.
4. Submerge the wafer in a glass dish filled with **acetone**.
5. Place the dish with the wafer into the ultrasonic machine. Start the cleaning cycle and run for **5 minutes**.
6. While cleaning, prepare a second dish with fresh **IPA**.
7. After 5 minutes, remove the dish. Lift the wafer while rinsing with **IPA**, and **immediately** transfer it to the second dish. **Do not let the wafer dry**—dried acetone causes stains.
8. Gently agitate the second dish for **60 s**.
9. Rinse the wafer under running **DI water** for **20 s**.
10. Dry the wafer thoroughly with **nitrogen gas**.
11. Transfer wafer to the yellow room for prebaking.
12. Bake the wafer on the **135°C** hotplate for **3 minutes**. Then let it rest on a clean surface for **2 minutes**.

2 Spin Coating

1. Set the spin coater program to:
 - (a) Recipe 0
 - (b) Step 1: **500 rpm, 5 s** – spread
 - (c) Step 2: **4000 rpm, 30 s** – achieve 0.5 μm thickness
 - (d) Step 3: **0 rpm, 0 s** – end
2. Place the wafer on the chuck. Turn on the vacuum and ensure the wafer is secure (change chuck if needed).
3. Using a pipette, dispense enough **P20 Primer** to evenly coat the wafer.
4. Start the spin cycle. Once complete, turn off the vacuum and transfer the wafer to the **115°C** hotplate. Bake for **60 s**.
5. Let the wafer cool for **60 s**.
6. Repeat the coating steps 2-5 using **S1805 PR** instead of primer.

3 Photolithography

1. Place the wafer at the center of **Photo3**.
2. Set the wafer thickness to **600 μm** .
3. Align the wafer precisely in the **x** and **y** directions.
4. Define the dimensions of the write area.
5. Use the **build job list** function to generate a marker grid.
6. Set the exposure parameters:
 - (a) Mode: **Normal**
 - (b) Resolution: **0.6 μm**
 - (c) Intensity: **60 mJ/cm²**
7. Start the exposure process and wait for completion.

Note:

1. The resolution must be finer than your smallest feature (e.g., use 0.6 μm for a 1 μm feature).
2. Refer to the official documentation for detailed instructions.

4 Developing

1. Submerge the wafer in **MF319** developer for **30 s**.
2. Rinse **immediately** under **DI water** for **30 s**.
3. Dry the wafer using **pressurized nitrogen**.
4. Inspect the developed features under the microscope using **green light**. Only use microscope inside the yellow room.
5. If features are unclear, extend development time in MF319 or adjust the exposure intensity on Photo3.

5 EB Evaporation

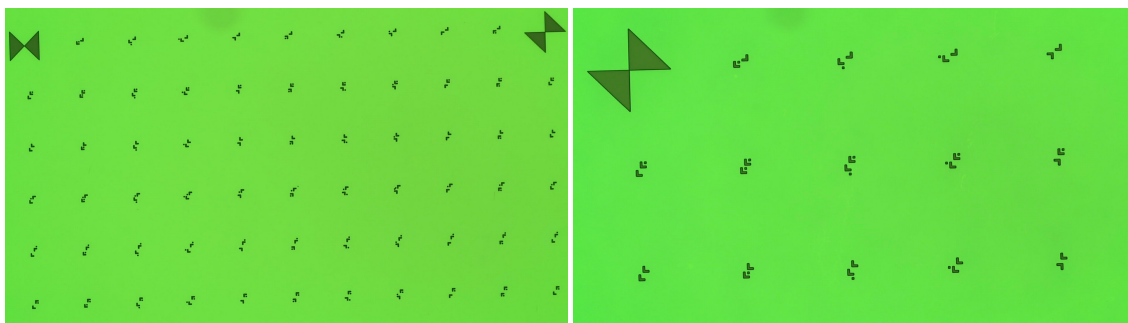
1. Deposit metals in the following order: first **Cr**, then **Au**.
2. Use the following target thicknesses:
 - (a) **Cr**: 5 nm (0.05 kÅ)
 - (b) **Au**: 50 nm (0.5 kÅ)
3. Follow all procedures outlined in the **EVAP 2** manual.
4. The full process takes approximately 2.5 hours.

6 Photoresist Removal

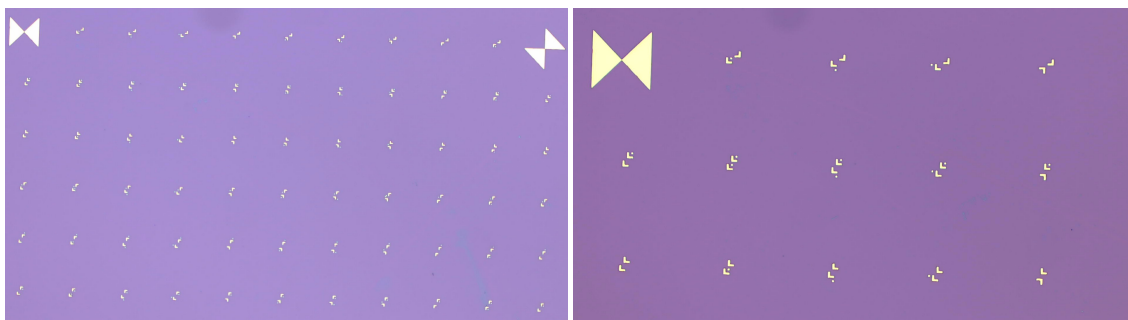
1. Pour **1165 PR remover** into a clean glass dish.
2. Submerge the wafer and soak for **8 hours**.
IMPORTANT: After this step, the wafer must remain fully submerged in a solution at all times!
3. After soaking, move the dish to the solvent hood with the ultrasonic machine.
4. Set the ultrasonic machine knob to **position 2**. Note, higher settings increase the risk of small features detaching.
5. Place the dish with the wafer into the machine and run the cleaning cycle for **3 minutes**.
6. While cleaning, prepare a second dish with fresh **IPA**.
7. After 3 minutes, remove the dish. While rinsing the wafer with **IPA**, carefully lift it and **immediately** transfer it to a clean dish.
8. Inspect the wafer under a **stereo microscope**, checking for any remaining gold dust on the surface.
9. If gold dust is still present, repeat the rinse with **IPA** and transfer the wafer to a new clean dish. Continue until the wafer is completely free of gold dust.
10. Rinse wafer with **DI water**.
11. Dry the wafer using **pressurized nitrogen**.
12. Inspect the wafer to ensure complete photoresist removal.

7 Expected Quality of Alignment Markers

1. After Step 4 - Developing in MF319



2. After Step 6 - Photoresist Removal



8 Optional: Post Sealing

After complete removal of photoresist (PR), the next step may involve dicing the wafer. This process poses a risk of contaminating wafer features with dust and other particles. To protect the surface, the wafer is coated with a temporary layer of resist that serves as a protective seal.

This layer prevents particle adhesion and can be easily removed after dicing using the standard PR removal procedure.

The procedure is outlined below.

1. At the spin coat hood in the yellow room, set up a hotplate to **180°C**. It will be used later.
2. At the solvent hood with ultrasonic machine, prepare:
 - (a) **Acetone**
 - (b) **IPA**
 - (c) **DI water**
 - (d) Ensure access to **hydrogen gas**.
3. Set the ultrasonic machine knob to **position 2**. If the bath is empty, fill it with **IPA**.
4. Submerge the wafer in a glass dish filled with **acetone**.
5. Place the dish with the wafer into the ultrasonic machine. Start the cleaning cycle and run for **5 minutes**.
6. While cleaning, prepare a second dish with fresh **IPA**.
7. After 5 minutes, remove the dish. Lift the wafer while rinsing with **IPA**, and **immediately** transfer it to the second dish. **Do not let the wafer dry**—dried acetone causes stains.
8. Gently agitate the second dish for **60 s**.
9. Rinse the wafer under running **DI water** for **20 s**.
10. Dry the wafer thoroughly with **nitrogen gas**.
11. Transfer wafer to the yellow room for prebaking.
12. Bake the wafer on the **180°C** hotplate for **2 minutes**. Then, let it rest on a clean surface for **2 minutes**.
13. Set the spin coater program to:
 - (a) Recipe 0
 - (b) Step 1: **500 rpm, 5 s**
 - (c) Step 2: **3000 rpm, 40 s**
 - (d) Step 3: **0 rpm, 0 s**
14. Place the wafer on the chuck. Turn on the vacuum and ensure the wafer is secure (change chuck if needed).
15. Using a pipette, dispense enough **PMMA 950A4** resist to evenly coat the wafer.
16. Start the spin cycle. Once complete, turn off the vacuum and transfer the wafer to the **180°C** hotplate and bake for **2 minutes**.
17. Let wafer cool for **2 minutes**.
18. Inspect the wafer under the microscope.

References

- **Photo3 Manual:** https://smif.pratt.duke.edu/sites/smif.pratt.duke.edu/files/2025/PHOT03_SOP_rev-2025-05.pdf
- **EB Evaporation:** <https://smif.pratt.duke.edu/sites/smif.pratt.duke.edu/files/2024/EVAP2%20operating%20Procedure%2012.pdf>
- **Nomarski Microscope Manual:** https://smif.pratt.duke.edu/sites/smif.pratt.duke.edu/files/operating/Microscope%20Camera%20operation_0.pdf
- **PMMA Double Layer Process:**
<https://smif.pratt.duke.edu/sites/smif.pratt.duke.edu/files/operating/EBL%20Resist%20Processes%206.pdf>