Topic 3 : Liftoff, HMDS coating , and AZ nLOF 2070 negative resist

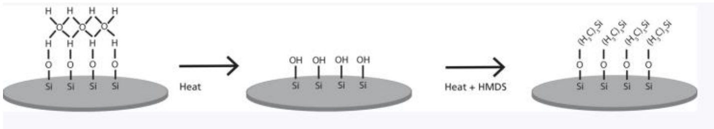
In this laboratory, students will engage in hands-on activities focused on exploring three critical subjects involved in the fabrication of aluminum resistor elements using the liftoff process: first, the liftoff step; second, negative resist; and finally, surface preparation, which includes cleaning and HMDS treatment. Through these subjects, students will learn essential techniques for silicon surface preparation, including cleaning and hexamethyldisilazane (HMDS) coating, as well as measuring contact angles. Students will also apply photoresist using spin coating, perform UV exposure, and execute development procedures. Furthermore, the lab will provide insights into the use of negative photoresist in liftoff processing applications and the use of optical microscopy for characterization.

Organisation:

During the session, you will be divided into small groups, with each group beginning at a different subject. This approach allows for flexible engagement with the subjects. While one student focuses on the negative resist subject, the others can engage in the two remaining subjects. After rotating through all subjects, we will come together to discuss your findings and insights, ensuring that everyone gains a comprehensive understanding of how these subjects interconnect in the liftoff process.

Subject 1: Surface Priming

To enhance photoresist adhesion to the wafer, wafer priming is typically conducted before the spin coating of the resist. This process involves the application of hexamethyldisilazane (HMDS), which can be dispensed in either vapor or liquid form. When applied, HMDS effectively renders the wafer surface hydrophobic, maintaining this property for several days post-treatment. However, it is crucial to avoid over-priming, as this can lead to significantly poor adhesion of the photoresist.



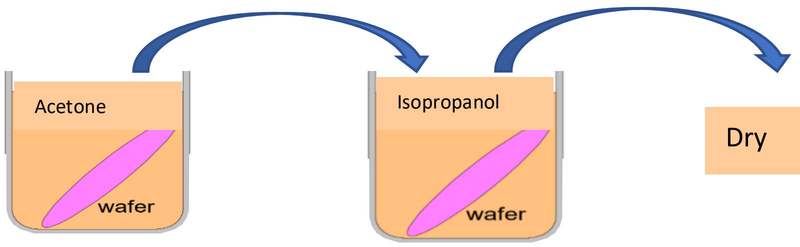
Before priming, surface preparation is very important for proper photoresist adhesion. It is especially important to clean and dry the substrate properly.

❖ Sample Cleaning

To remove contaminants from the wafer surface that include oils and other organic particles, a simple method consists of the following steps:

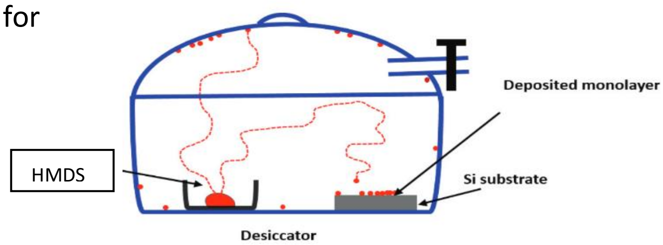
a. In a glass container, cover the surface of the sample with acetone for approximately 5 minutes (The longer you wait, the better the results.).
b. In a separate glass container, place the sample in Isopropyl alcohol (IPA) for few minutes (2-3 min).
c. Remove and Blow Nitrogen (N_2).

Solvents can clean oils and organic residues that appear on silicon surfaces. Unfortunately, solvents themselves (especially acetone) leave their own residues on the sample's surface. This is why a two different solvents method is used.



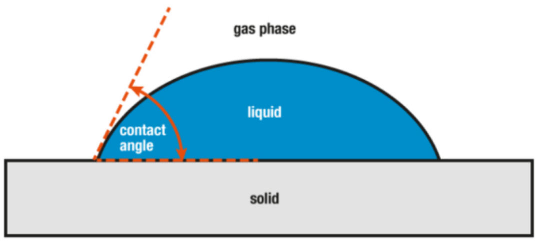
❖ Vapor Priming

- Obtain silicon piece you have already cleaned with acetone and IPA.
- Place the sample inside a desiccator.
- Small amount of HMDS is placed in a small petri dish that is placed in the desiccator.
- Apply a vacuum to the desiccator for few minutes.
- Close the valve of the desiccator.
- Kept the samples under vacuum with silane vapor for 60 min before removing them from the desiccator.



1. Contact Angle measurement

The surface hydrophobicity can be measured by placing a drop of water on the primed surface and measuring the contact angle as shown in figure 1. An optical goniometer (often attached to an optical microscope) is used to measure this angle with reasonable precision and repeatability. The contact angle is affected by various parameters such as temperature, pressure, time, and chemical density. As the surface of the silicon wafer before treatment is hydrophilic, the water contact angle is relatively low, around 40°. After the HMDS treatment, the surface is more hydrophobic and the water contact angle increases significantly between 65° and 80°.



Explanation contact angle

1.1. Measuring contact angle

1. Turn on the microscope and the light.
2. Place your sample on the holder
   - Silicon sample without cleaning
   - Silicon sample with cleaning
   - Silicon sample with cleaning and HMDS treatment
3. Using a micrometer syringe, carefully produce a small droplet. Gently allow the droplet to touch the sample surface, ensuring that it rests evenly on the sample
4. Adjust the illumination to eliminate any reflections, ensuring that the droplet image is clear and well-defined.
5. Focus the USB microscope to achieve optimal sharpness of the image, providing a clear view for analysis.
6. Press the camera icon to snap the drop image.
7. Repeat the experiment at a different position on the sample.
8. Save and transfer the image file then measure the contact angle using an image analysis program, e.g., Image J.

Subject 2: Liftoff of aluminum

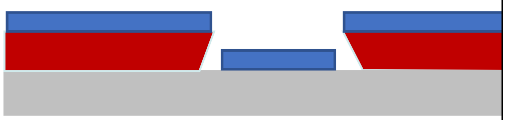
Liftoff processing in microfabrication is an essential technique for creating specific patterns and structures at the micro and nanoscale. This method is particularly valuable when working with materials that are challenging to etch using conventional methods. The process begins by depositing a sacrificial photoresist layer onto a substrate and then defining the desired pattern using precise lithographic techniques. Subsequently, a thin film of the target material, often a metal, is deposited uniformly over the substrate. When the sacrificial photoresist is dissolved with a compatible solvent, such as Acetone, the material on top of the photoresist lifts off, leaving behind the precisely defined microstructures or patterns on the substrate.

Liftoff operating procedure

Your task is to perform the last step of stripping the resist in a solvent until all the metal on the resist is lifted-off.

- Obtain one sample from your TA.
- Place the sample into a small glass container.
- Fill the beaker with enough acetone to cover completely your sample.
- The aluminum will begin to peel-off. Wait until removing completely the photoresist and metal on it. This operation can take time; if the aluminum has not completely lifted off, take a swab and gently wipe the wafer to remove the metal (avoid touching your patterns), or use the ultrasonic bath.
- After finishing, clean your sample with IPA for removing acetone and dry using N_2.
- Inspect your sample under optical microscope.

In this experiment, a sample that has been previously patterned with negative resist followed by metal deposition as shown in the process flow:

Step	Process description	Cross-section after process
01	**Substrate**: Glass wafer • *Teply O_2 plasma treatment,*	
02	*Photolith PR coat* • *1.4 um AZ nLOF 2020 coating in ACS200,*	
03	*Photolith expo+ develop* • *4.7 sec exposure with MJB4 (94mJ/cm^2)* • *Developing in ACS200,*	
04	*Metal deposition* • *20/200 nm Ti/Al in EVA760, 450 mm WD, dep. rate: 5 A/s*	

- *SRD to clean wafers*
- *Teply O2 plasma treatment,*
- *1.4 um AZ nLOF 2020 coating in ACS200,*
- *4.7 sec exposure with MJB4 (94 mJ/cm^2)*
- *Developing in ACS200,*
- *20/200 nm Ti/Al in EVA760, 450 mm WD, dep. rate: 5 A/s*

Subject 3: AZnLOF 2070 negative photoresist

The success of the liftoff operation is highly dependent on the profile of the negative resist. It is crucial for the resist to have an undercut to prevent issues during the liftoff process. An undercut allows for better separation of the resist from the substrate, ensuring that the aluminum layer can be effectively removed without leaving residues or damaging the underlying structures. This careful design of the resist profile is essential for achieving clean and precise fabrication results.

AZnLOF 2070 is a negative photoresist designed specifically for liftoff processing. The resist produces a negative (undercut) profile to reduce deposition of the material on sidewalls. This design feature facilitates better access for solvents during the liftoff process, allowing for more efficient removal of the film after deposition.

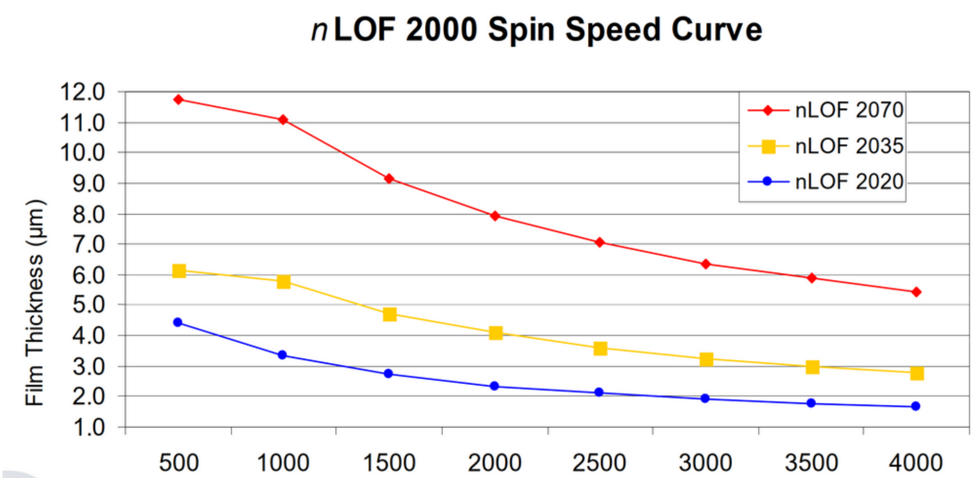
1. Each group will be provided with 4 silicon samples already received HMDS treatment (see surface priming:subject 1)
2. photoresist coating.

- Set the recommended spin program below:

Step	Spin speed (RPM)	Time (s)
1	500	20
2	3000	40

  - Step 1 : to spread the photoresist over the entire sample
  - Setp 2 : to get the desired thickness

From the spin curve (See below), what is the expected thickness of the coated film?



1. Put your sample on the chuck, center the sample and press vacuum.
2. Dispense a small amount of resist on the substrate.
3. Close the lid of the coater and press Run.
4. Soft Bake on the Hotplate at 100°C for 90s.
5. UV exposure for a duration $\mathbf{t_{exp}}$

6. A **post-exposure bake (PEB)** at temperature $\mathbf{T_{PEB}}$ for a duration of $\mathbf{t_{PEB}}$ must be performed for this resist. This step significantly influences the final results of the photoresist profile.
7. During this session, the parameters to be tested are:
   - **Post Exposure Baking time (t_{PEB})**
   - **Post exposure Temperature, (T_{PEB})**
   - **Development time (t_{dev})**
   - **Exposure time (t_{exp})**

8. Prepare sample and mask on their proper holder as follow:
   - Prepare the holder and the photomask for the exposure process
   - Carefully position the wafer on the specific holder.
   - Place the photomask on its holder and secure it with screws so that it does not fall off when turned over.
   - Lower the wafer holder and gently place the wafer on the holder, ensuring it is well centered.
   - Place the mask holder on the four rods and secure it with screws so that the assembly remains stable."
   - Raise the wafer until it is in contact with the mask. Do not apply too much force to avoid breaking the wafer.

9. The parameters for UV exposure and post-exposure bake are listed in the following table

	T_{PEB} (°C)	t_{PEB}(s)	t_{exp} (s) for UV P.250 Lamp	t_{exp} (s) for X-cite lamp
Student 1	100	60	15	18
Student 2	100	90	15	18
Student 3	100	60	20	25
Student 4	110	60	15	18

10. development

<table>
<tr><td colspan="3">Step 10-1: Develop the complete sample for 1 min40 sec.
Step 10-2: Rinse in water and dry with N_2.
Step 10-3: Cleave the sample into four pieces. For each part, add an additional time for development as indicated below.
Step 10-4: For each part, repeat steps 1 and 2</td><td>(1) (2)
(3) (4)</td></tr>
<tr><td>(1)</td><td>(2)</td><td>(3)</td><td>(4)</td></tr>
<tr><td>t_{dev} = 1min40s
reference sample</td><td>t_{dev}+20s</td><td>t_{dev}+30s</td><td>t_{dev}+50s</td></tr>
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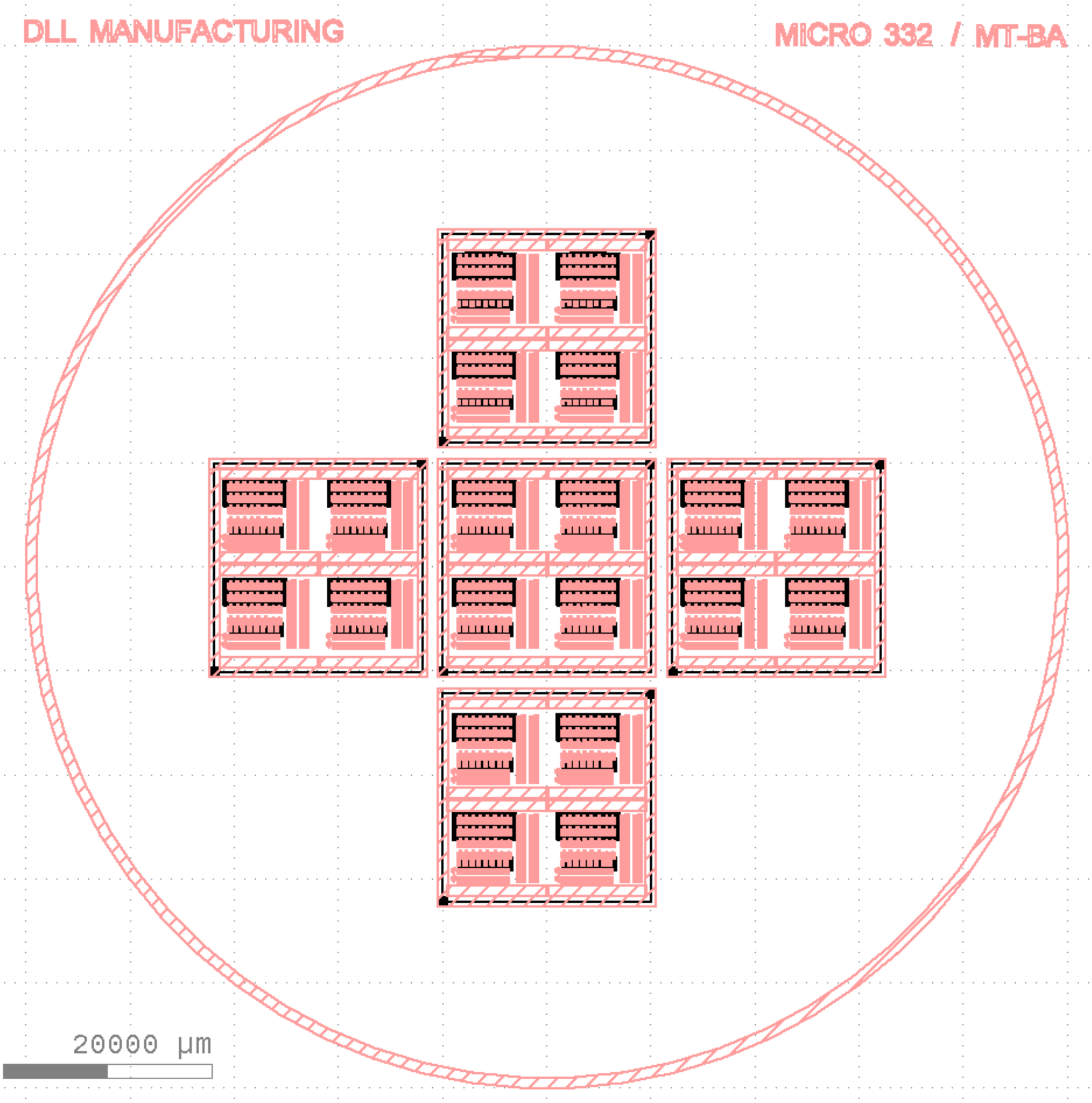
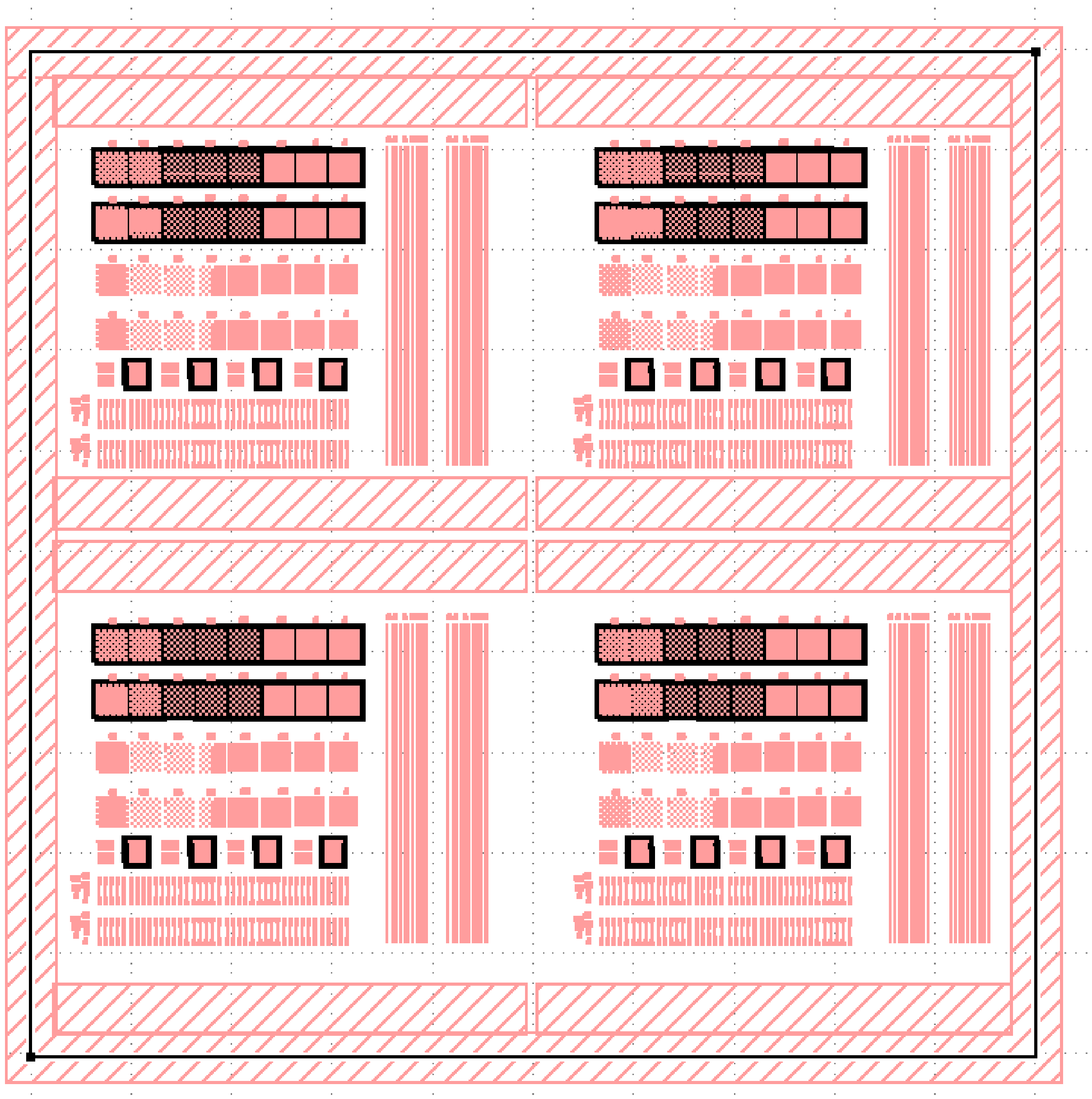
11. Optical microscope observation
    - Each student will examine the samples under the optical microscope, focusing on the same type of feature, either circles or squares, at the same magnification. During this process, students should take measurements of the sizes of the features observed and save the image for further analysis. Each student will have a maximum of 15 minutes at the microscope to ensure that everyone gets their turn without delays.

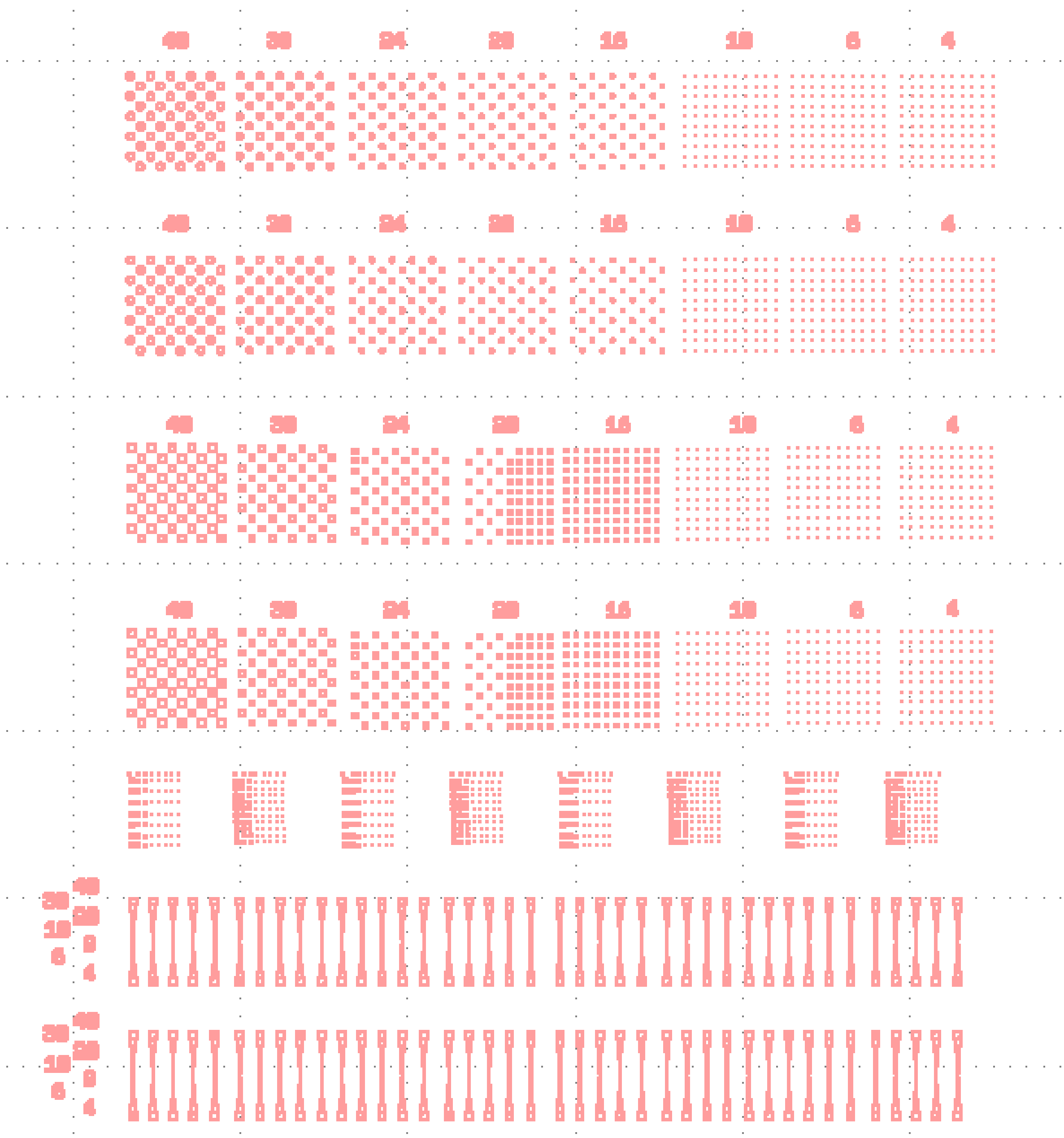
Mechanical cleaving is a technique that uses a diamond scribe to mark the surface of the wafer along a predetermined cleave plane. Using a wafer cleaving pliers, the wafer is carefully bent or twisted to initiate a crack along the marked line, allowing the wafer to be split or broken cleanly. To begin the process, lightly scratch the edge of the silicon (Si) sample without applying any pressure; a typical scratch length is approximately 2 mm. Once scribing is complete, position the wafer on the wafer cleaving pliers tool, aligning the scribe line with the edge on the jaws. By applying gentle pressure, the scribe line should automatically propagate down the wafer, ensuring a smooth cleaving process.

12. Keep the samples in a box for SEM observation and select the ones you prefer to observe (two or three).

Mask design:



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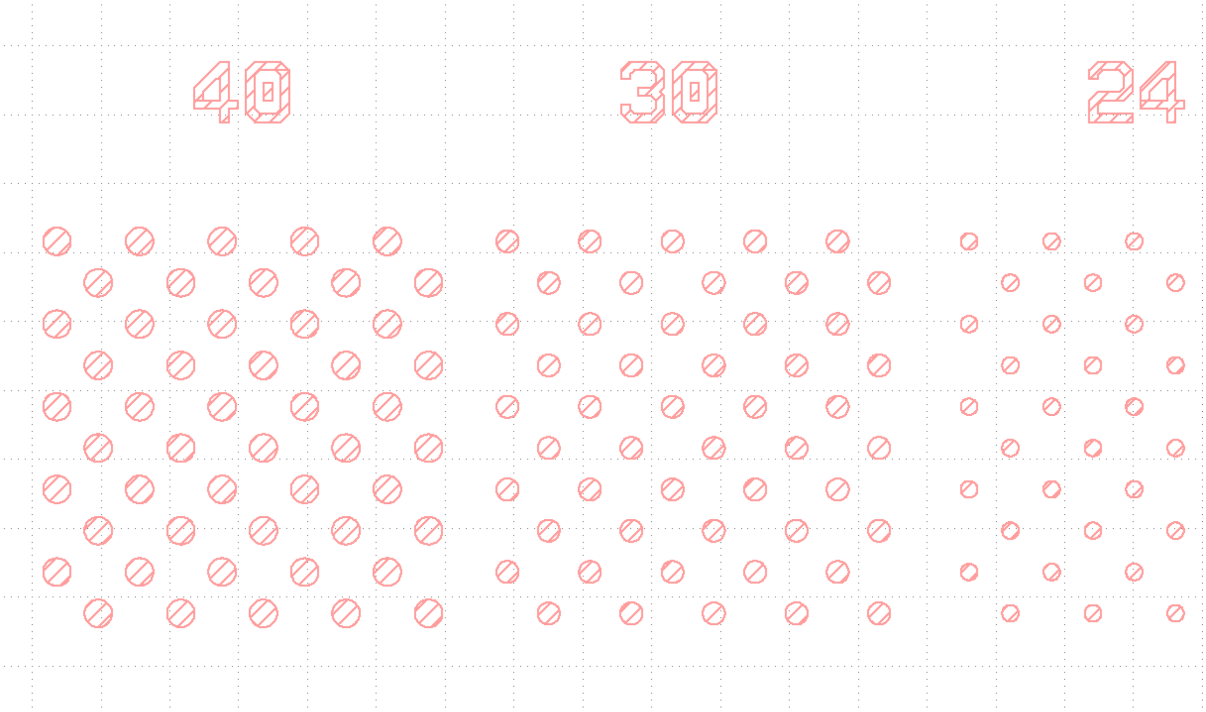
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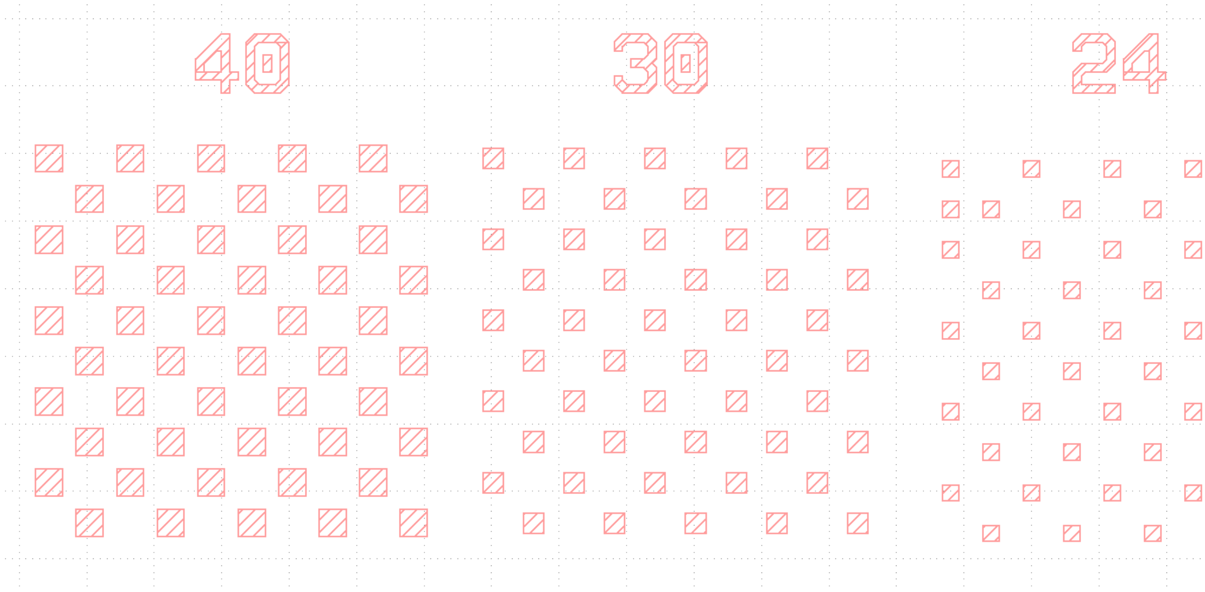
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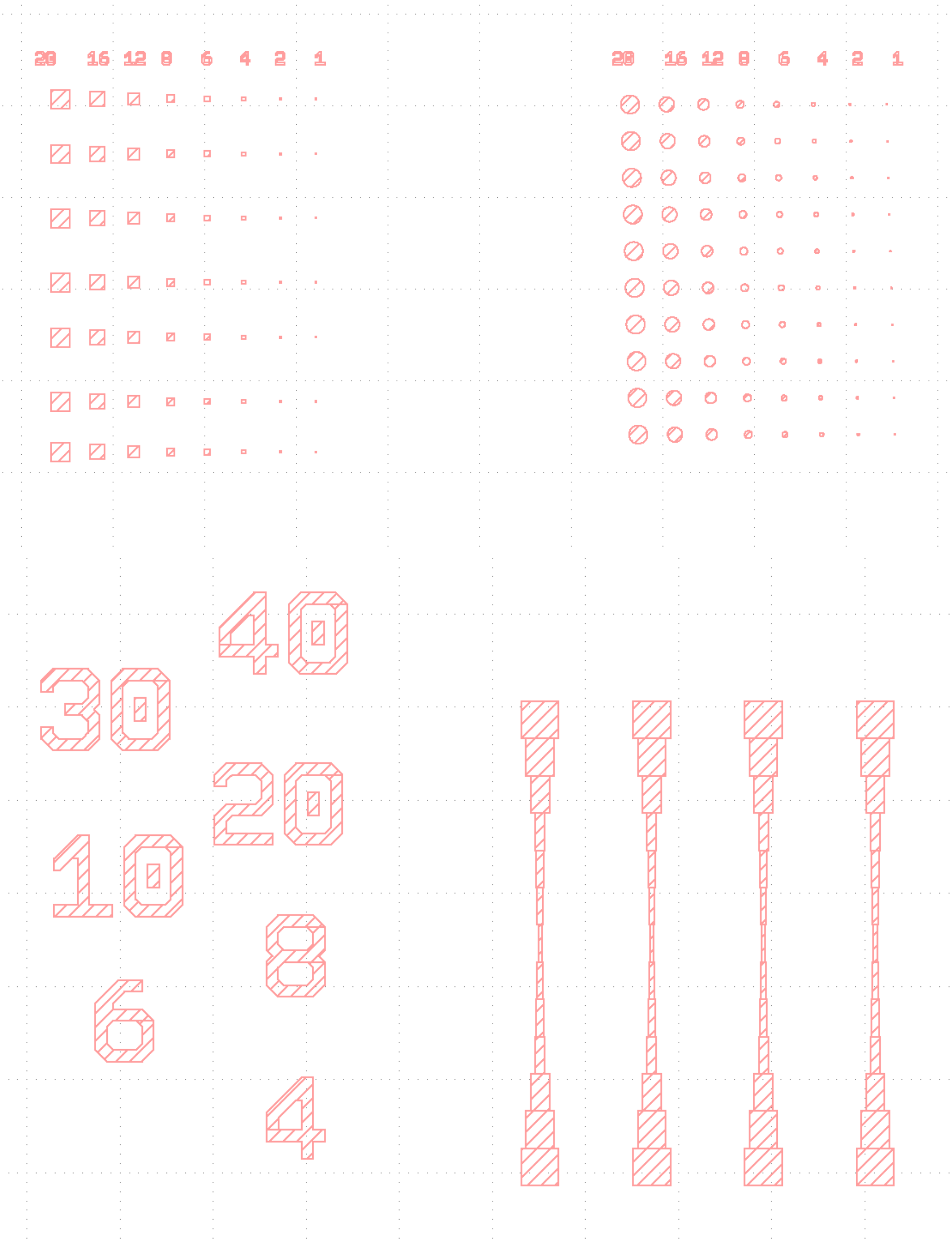

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