Laboratory 2: WAFER CLEAN, CLEAVING AND PHOTOLITHOGRAPHY 1

Prior to any processing, the GaAs wafers have to be cleaned to remove any surface oxides and contaminants. In general, III-V wafers undergo a surface etch using BOE followed by rinsing in acetone and IPA. It's important to note that other cleaning procedures that include concentrated HF or dry etching techniques do exist.

A. Buffered Oxide etch (BOE):

Buffered oxide etch (BOE), also known as buffered HF or BHF, is a wet etchant used in microfabrication. Its primary use is in etching thin films of silicon dioxide (SiO_2) or silicon nitride (Si_3N_4). It is a mixture of a buffering agent, such as ammonium fluoride (NH_4F), and hydrofluoric acid (HF). While the solution comes in different concentrations, we would be using a 10:1 volume ratio of 40% NH_4F in water to 49% HF in water. This solution would be pre-mixed for you to use.

B. Rinse:

Wafers are normally rinsed in acetone and iso-propyl alcohol (IPA) to remove any surface contaminants.

Procedure:

1. Take 50 ml of 10:1 BOE solution in a PLASTIC beaker. Caution: Never use a glass beaker for solutions containing HF as it etches glass. Use only plastic tweezers while handing BOE solutions.
2. Place the wafer in the solution for 3 minutes.
3. Rinse the wafer in DI water thoroughly for 2 minutes.
4. Take 50 ml of acetone and IPA in separate glass beakers for rinsing purposes.
5. Rinse the wafer first in acetone for 5 minutes, followed by rinsing in IPA for another 5 minutes.
6. Blow dry wafer with Nitrogen.

C. Cleaving:

We will now have to cleave the 2" wafer into a small piece of approximately 1 x 1 cm for you to continue with processing. This is mainly done to optimize the use of expensive wafers and also have a backup in case something goes wrong with the processing.

1. Place the wafer upside down on tex wipes.
2. Use a diamond scribe to scratch the wafer at the desired edge. It is important to know that you are dealing with a crystal. Consequently, there are only certain planes along which it can be cleaved easily. For the GaAs wafer that you have, the cleaving planes are the ones parallel and perpendicular to the major flat.

3. Gently tap the wafer using the back of a tweezer at the point where you made the scratch. At this point, the wafer should easily cleave with a mirror-like cleaved edge.

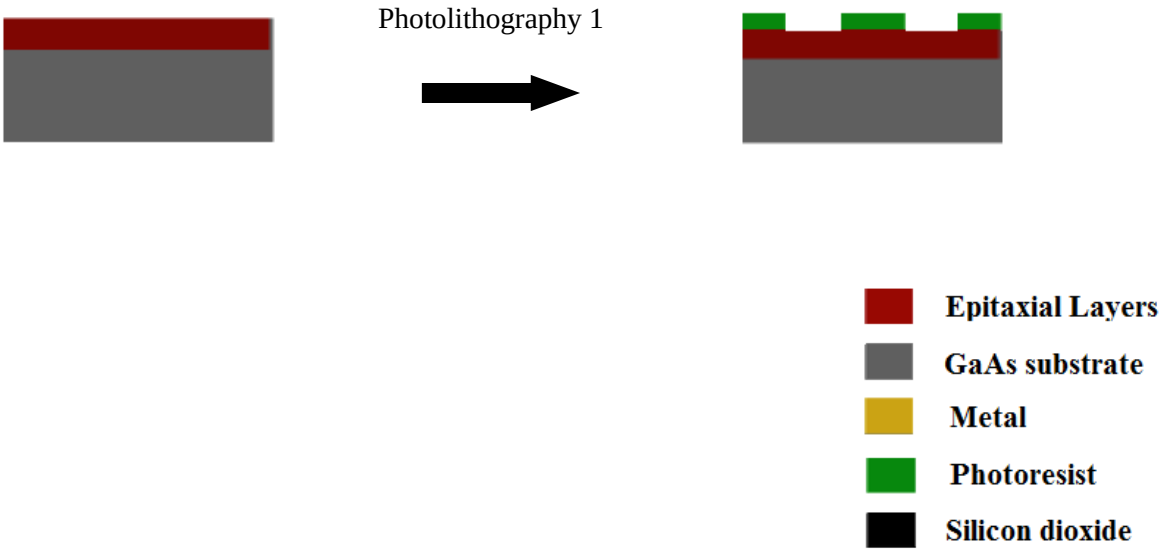
D. Photolithography 1:

The first step of photolithography will define the laser ridges in the wafer. It is often convenient to attach the wafer to a glass slide before continuing with the process. This would prevent any damage to wafer while handling. Use the following recipe to first mount the wafer onto a glass slide.

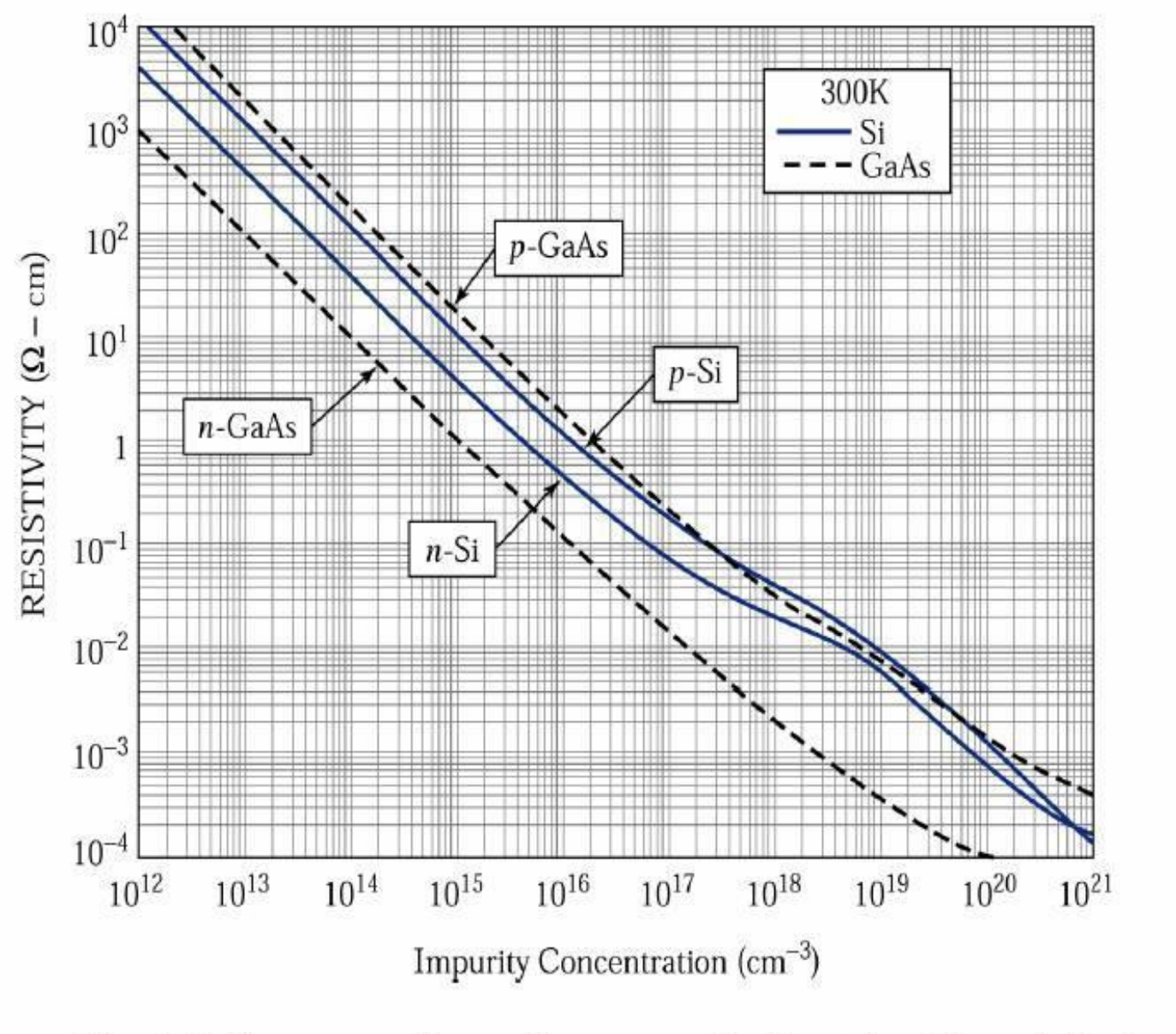
1. Spin coat AZ 5214 on to a glass slide at a speed of 2000 rpm for 20 seconds. (Recipe 9)
2. Place the wafer onto the glass slide and slowly press it down with a cotton swab.
3. Bake on a hot plate at 100 C for 5 minutes.

Once you have mounted the wafer onto the glass slide, let's proceed to the first step of lithography.

4. Spin coat AZ 5214 on the wafer at a speed of 4000 rpm for 40 seconds. (Recipe 1)
5. Bake wafer for 1 min at 100 C.
6. Align on the mask aligner MJB4 using the alignment marks and expose for 3 seconds. The parameters for the exposure include: Align and expose, hard contact and reflected light, with a hold time of 5 seconds and a single exposure cycle.)
7. Take 100 ml of developer solution AZ 300 MIF in a glass beaker.
8. Develop the wafer in the above solution for 45 seconds, shaking vigorously.
9. Rinse the wafer under DI water for 1 minute.
10. Blow dry with Nitrogen and inspect the pattern under an optical microscope.



Use the following figure as a reference while calculating the resistivity for GaAs wafers, as discussed in your lab manual.



Resistivity versus impurity concentration for Si and GaAs.

Laboratory 3: SEMICONDUCTOR ETCH AND SILICON OXIDE DEPOSITION

A. Semiconductor etch "Orange Etch"

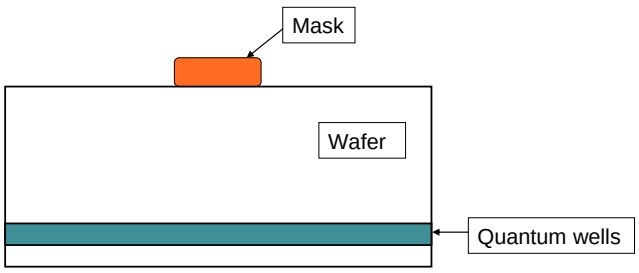
There are different solutions to etch semiconductor III-V wafers. Some of the commonly used etchants are given below:

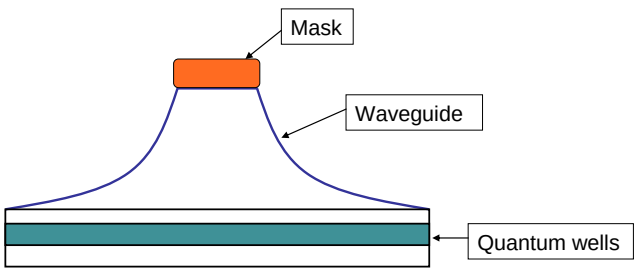
Etchant	Formula	Etches
Acetic Acid	$H_3COOH(l)$	GaAs; Pb; Ti
Hydrochloric Acid	HCl(38%, aq)	Al; Cr; Cu; Fe_2O_3; Ga; GaN; In; Fe; Pb; Ni; NiO, Ni_2O_3; Sn;SnO_2; Ti; Zn
Hydrofluoric Acid	HF(49%, aq)	GaAs; Ni; SiO_2; Ti; Al_2O_3
Nitric Acid	HNO_3(70% aq)	C; Cu; GaAs; In; Fe; Pb; Ni; Ag; Pd; Pt; Sn; Ti; Zn; ZnO
Phosphoric Acid	H_3PO_4(85% aq)	Al; Cu; GaAs; GaN; Fe; Ni; SiN; ZnO
Potassium Hydroxide	KOH(s/aq)	Al; C; Cu; Ag; GaAs; Si; Ti
Sodium Hydroxide	NaOH(s/aq)	Al; Cu; Ag; Ti; Si; GaAs; GaN
Sulfuric Acid	(96%, aq)	C; Cu; GaAs; Fe; Pb; Ni; Ti

Specifically, in our case we use what we call "Orange etch": HBr : HNO_3 : H_2O in 1:1:10 ratio. After mixing, we leave it undisturbed for 1 or 2 days for the solution to stabilize. We will have the solution ready for you prior to the lab.The etch rate is around 1 um/min, but could vary depending upon its age. The etching is isotropic, which means it doesn't have a favored direction. Please note that this solution is photosensitive and degrades in the presence of light. This would mean that the etch rate reduces over time as it is exposed to ambient light conditions.

Procedure:

1. Take about 50 ml of orange etch in a glass beaker.
2. Hold the wafer using PLASTIC tweezers in the solution for the required amount of time. Since we would need to etch the wafer to about 2 microns, let's etch it for 2 minutes.
3. Rinse the wafer thoroughly in DI water and blow dry with nitrogen.
4. Transfer the orange etch back to its container to be reused the next time.



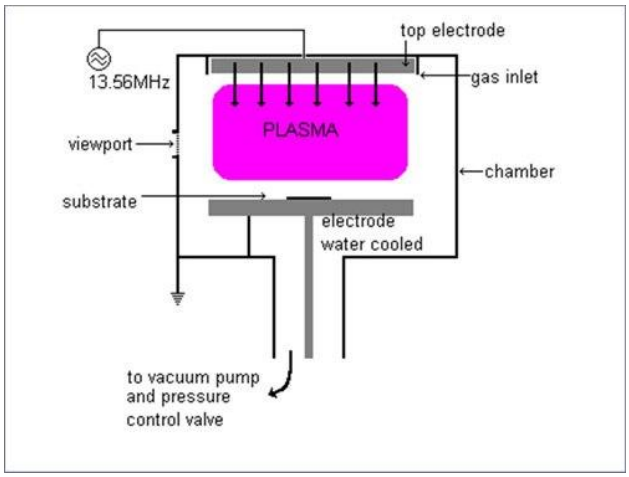


B. Wafer Clean:

Strip the photoresist, in acetone and IPA for 5 minutes each and blow dry with Nitrogen.

Reactive Ion Etching and Plasma enhanced chemical vapor deposition:

The principle of operation for the RIE is the same as that of the PECVD (Plasma enhanced chemical vapor deposition) used to deposit the silicon oxide. In both these processes, a plasma is generally created by RF (AC) frequency or DC discharge between two electrodes, the space between which is filled with the reacting gases. In the plasma, the molecules are ionized, and can easily make chemical reactions, forming the materials to be deposited, or etched as the case may be, depending on the gases used.



C. Hard descum:

Before we deposit silicon oxide on the sample, we have to clean the wafer of any organic photoresist residue. This will be accomplished by doing a process called hard descum on the RIE. This step basically ignites oxygen plasma that can remove any organic material from the wafer. The RIE chamber has O_2, H_2, CF_4, CH_4, SF_6, CHF_3, and Ar. The flow rate for each gas is set by a mass-flow controller (MFC), and the pressure is controlled separately by a butterfly valve between the chamber and the pump. All process in the RIE is done at room temperature.

Procedure:

1. Vent the RIE chamber and load your wafer. (Utilities → Vent)
2. Pump down the chamber (Utilities → Pump chamber) and wait until the pressure reads 1 mtorr.
3. Go to service → Manual mode and use the following parameters for the descum process.

   Oxygen flow rate: 40 sccm.
   Pressure: 200 mtorr

Power: 200W
Time: 5 minutes.

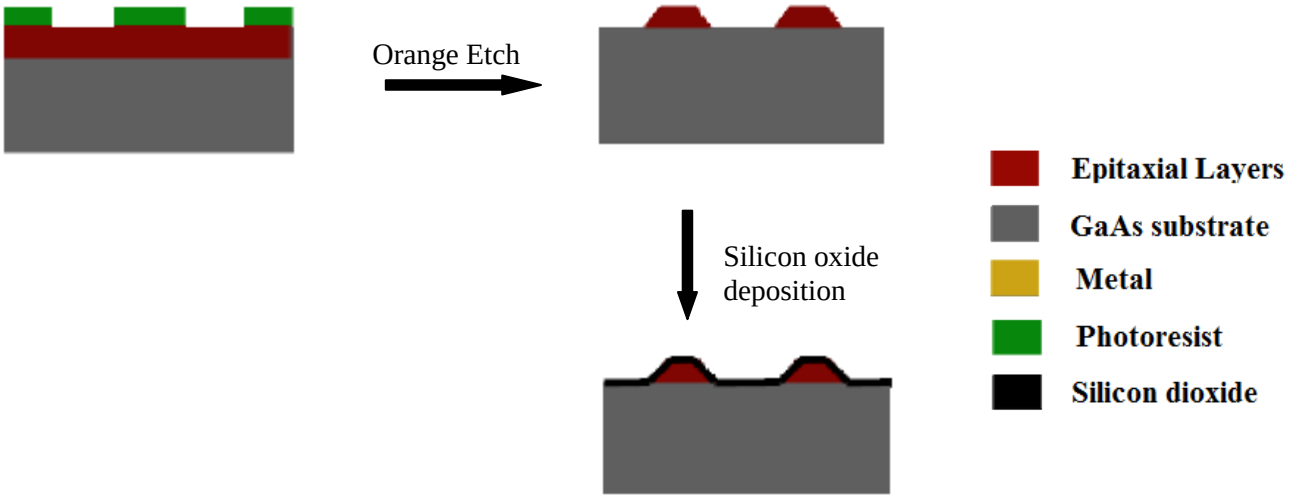
4. Vent the chamber and remove the wafer.
5. Pump down the chamber after doing your process.

D. Oxide Deposition - PECVD:

Oxide deposition happens at a high temperature (250 – 300 C). The thickness of the oxide you deposit depends on the maximum electric field that will be applied to the device without causing a breakdown of the dielectric. For our laser, we would be depositing about 3000 A of oxide at a temperature of 250 C. Prior to any oxide deposition, we need to clean the chamber and do a pre-deposition. The chamber would be pre-cleaned and heated before you come to the lab. We would also need a 4" Si carrier wafer to place our laser on for impedance matching purposes.

Procedure:

1. Set the temperature of the chamber to 250 C (Utilities → set standby temperature).
2. Vent the chamber (Utilities → Vent) and place the Silicon carrier wafer on the hot plate.
3. Pump the chamber (Utilities → Pump chamber) and wait until the pressure reads 2 mtorr.
4. Load the process OSiO2 (Process → Load) and run it for 10 minutes. This will deposit silicon oxide everywhere inside the chamber and create a very uniform environment.
5. After the process is completed, vent the chamber and place your wafer at the center of the Si carrier wafer.
6. Pump the chamber and wait until the pressure reads 2 mtorr.
7. Load the same process OSiO2 and run it for 15 minutes. The deposition rate for the given growth conditions is 200 A/min.
8. Vent the chamber and remove the laser and the carrier once the process is done.
9. Pump the chamber down and DO NOT forget to bring down the temperature to 25 C.



Laboratory 4: PHOTOLITHOGRAPHY 2, DRY ETCH, DESCUM

In our last lab, we deposited Silicon oxide to isolate the various laser bars. In this lab, we will be opening windows on top of the ridges to etch the silicon oxide for depositing contacts.

A. Photolithography 2:

1. Mount the wafer on a glass slide as explained in laboratory 2.
2. Spin coat HMDS on to the wafer at a speed of 4000 rpm for 40 seconds. HMDS is an adhesion promoter that helps the photoresist to wet the surface of the semiconductor. We need it in this step because of the uneven ridge surface that we have.
3. Spin coat AZ 5214 at a speed of 4000 rpm for 40 seconds, TWICE.
4. Bake at 100 C for 1min 15 seconds.
5. Align on the mask aligner MJB4 using the alignment marks and expose for 4.5 seconds. The parameters for the exposure include: Align and expose, hard contact and reflected light, with a hold time of 5 seconds and a single exposure cycle.
6. Take 100 ml of developer solution AZ 300 MIF in a glass beaker.
7. Develop the wafer in the above solution for 40 seconds, shaking vigorously.
8. Rinse the wafer under DI water for 1 minute.
9. Blow dry with Nitrogen and inspect the pattern under an optical microscope.

B. Light descum:

We need to clean the top surface of the wafer before etching the silicon oxide. You will now do a clean using an oxygen plasma on the RIE. Care should be taken to not over-do the oxygen plasma clean as it can etch away the photoresist. As explained before, load the sample into the RIE chamber and use the following parameters for the descum:

Oxygen flow rate: 40 sccm.
Pressure: 100 mtorr
Power: 100W
Time: 25 seconds.

C. Dry Etch:

Wait until the pressure reads 0 mtorr on the RIE. Dry etching is a combination of a physical and chemical process. The plasma created removes the material (oxide or nitride) from the wafer, while the gases react with it and make them volatile, thus taking them away from the wafer. Etch rates depend on many parameters including gas flow rates, pressure and the RF power to the system. Use the following parameters for etching silicon oxide:

Flow rates: CF4 : 25 and H2: 6
Pressure: 150 mtorr
Power: 150 W
Time: varies depending on thickness.

For the above recipe, the etch rate is about 196 A / minute. For a nominal 3000 A of oxide deposited, you need an etch time of about 16 minutes. Once the etch has been completed, wait for the pressure to go to 0 mtorr and then vent the chamber.

D. Wafer Clean:

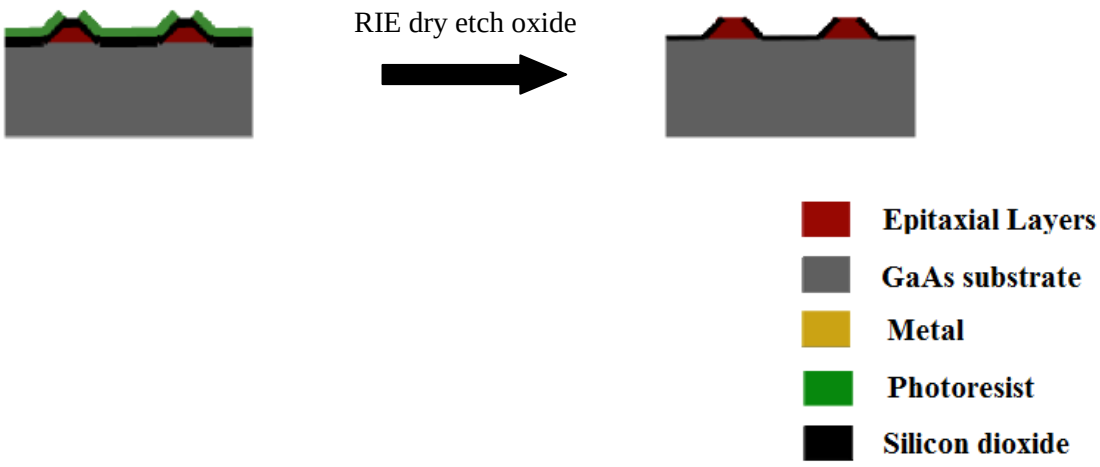
Strip the photoresist, in acetone and IPA for 5 minutes each and blow dry with Nitrogen.

E. Hard descum:

To clean the wafer of any remaining organic photoresist residue, load the wafer in the RIE chamber and do a hard descum using the following parameters:

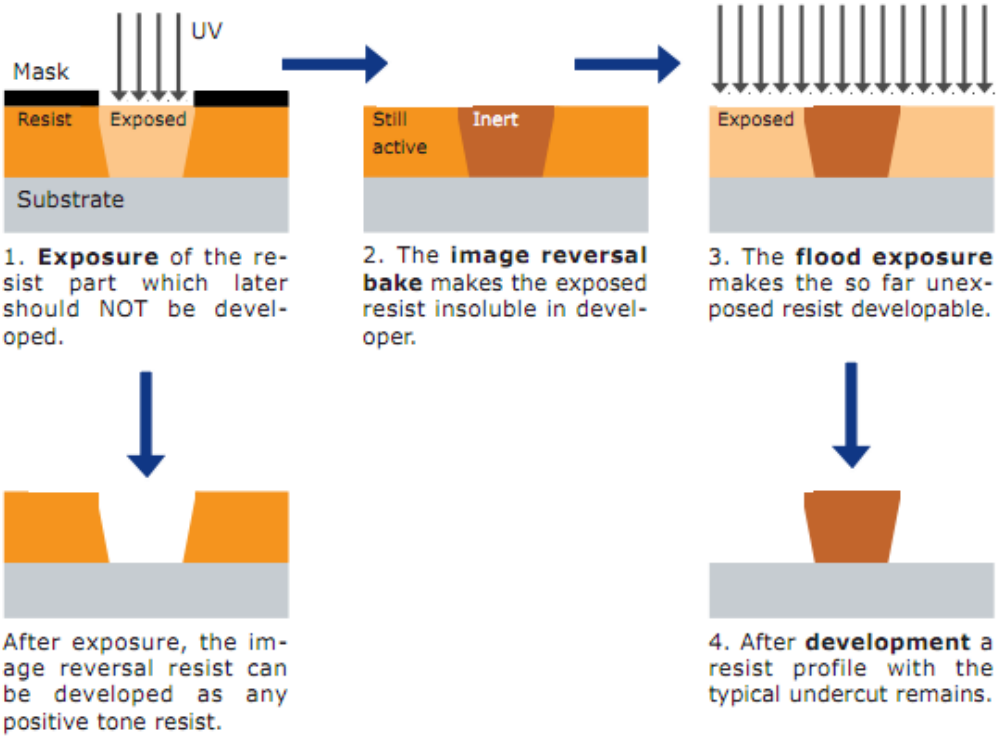
Oxygen flow rate: 40 sccm.
Pressure: 200 mtorr
Power: 200W
Time: 5 minutes.

Vent the chamber and take out your sample.



Laboratory 5: PHOTOLITHOGRAPHY 3, METAL DEPOSITION, LIFT OFF

The final lithography step is to define windows for depositing metal contacts. This lithography step makes use of a process called image reversal. This process is necessary to lift-off metal after deposition. This process reverses the action of positive resist so negative images can be formed with the same resolution and processing ease that a positive resist allows. Also, image reversal allows variations of the slope of the photoresist sidewall for higher resolution and/or lift-off profiles. The process is clearly explained in the following schematic:



A. Photolithography 3:

1. Mount the wafer on a glass slide as explained in laboratory 2.
2. Spin coat HMDS on to the wafer at a speed of 4000 rpm for 40 seconds. HMDS is an adhesion promoter that helps the photoresist to wet the surface of the semiconductor. We need it in this step because of the uneven ridge surface that we have.
3. Spin coat AZ 5214 at a speed of 4000 rpm for 40 seconds.
4. Bake at 100 C for 1min.
5. Align on the mask aligner MJB4 using the alignment marks and expose for 3 seconds. The parameters for the exposure include: Align and expose, hard contact and reflected light, with a hold time of 5 seconds and a single exposure cycle.
6. Hard bake wafer at 119 C for 1 minute.
7. Flood expose for 20 seconds on the MJB4.

8. Take 100 ml of developer solution AZ 300 MIF in a glass beaker.
9. Develop the wafer in the above solution for 40 seconds, shaking vigorously.
10. Rinse the wafer under DI water for 1 minute.
11. Blow dry with Nitrogen and inspect the pattern under an optical microscope.

B. Wafer Clean:

Prior to depositing metal contacts, do a BOE clean of the wafer to remove any surface oxides form the top of the ridges, as described in lab 4.

C. E-beam evaporation:

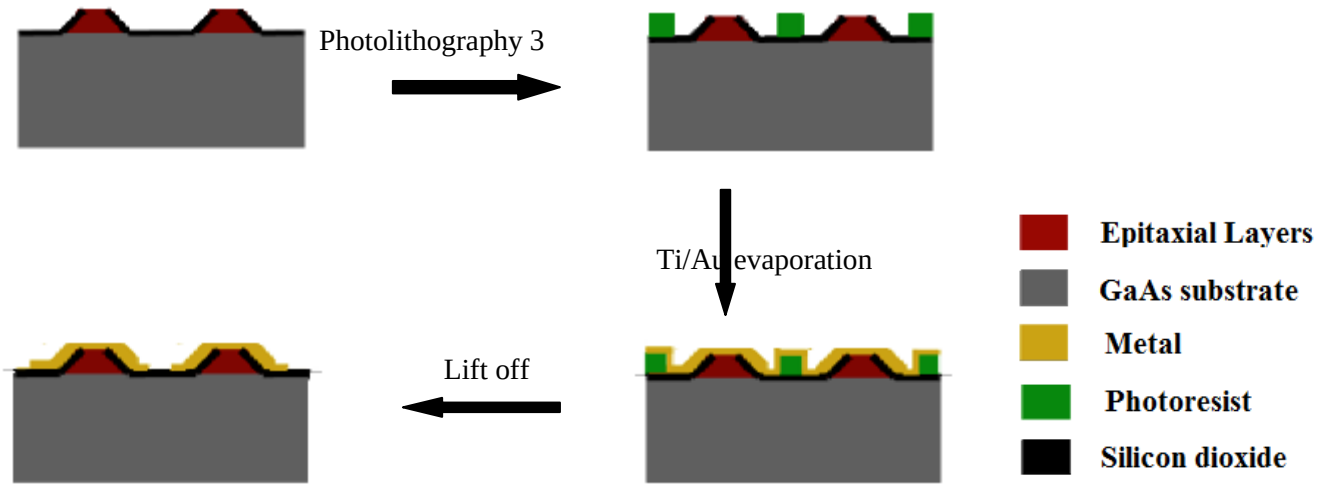
Load the sample into the e-beam evaporation. In this process, a highly energetic electron beam is used to melt metal which then gets evaporated on to the sample. Evaporation is a line-of-sight process and therefore do not cover the side-walls. To adequately cover the side walls with gold, we need to do angle evaporation. For top contacts, we deposit 200 A of Titanium and 2000 A of Gold. We require Titanium because gold does not adhere well to semiconductor surfaces.

Detailed instruction on how to use the evaporator will be given during the lab. For reference purposes, you can have a look at the instruction poster online: http://www.princeton.edu/mnfl/the-tool-list/innovative-systems/.

D. Lift off:

This process removes the metal from unwanted regions by dissolving the photoresist that is underneath the metal.

1. Heat photoresist stripper 1165 on a petri dish to about 95 degrees.
2. Keep the wafer on this solution for about 5 minutes.
3. Transfer the wafer to a petridish containing acetone and rinse it thoroughly, make sure the lift off pattern has been successful.
4. Rinse the wafer with IPA and blow dry with Nitrogen.



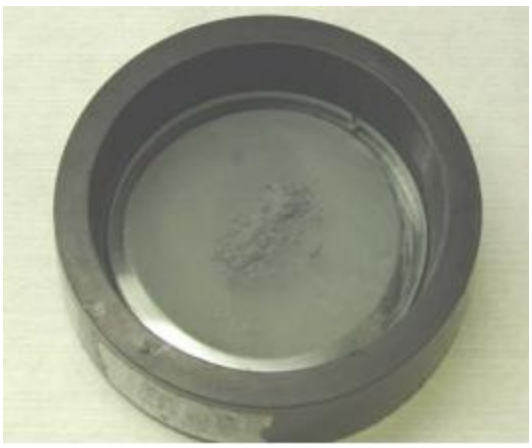
Laboratory 6: LAPPING, BACK CONTACT EVAPORATION

A. Lapping:

We need to thin down the wafer to about 160 microns so as to facility conduction through the device. Lapping can be done using different procedures. The most commonly used techniques include chemical lapping and mechanical lapping. Chemical lapping uses a combination of bromine and methanol to chemically etch the III-V substrate. This process is much faster and gives a mirror-like finish compared to mechanical lapping. However, this method does not come highly recommended because of toxicity issues with using bromine.

Mechanical polishing involves grinding the wafer against particles of different sizes and mechanically etch the surface. While there are different grain sizes available for getting mirror polish, lapping requires only the silicon carbide grit. Use the following procedure for lapping purposes.

1. Turn on the hot plate to a setting between 4 and 5, and place the steel chuck on it.
2. Peel of some crystal wax using a razor blade and put it on top of the steel chuck.
3. As the crystal wax melts, carefully place your wafer face down on the chuck and press down with a wooden stick to flatten.
4. Remove it from the hot plate, and once the wax has solidified, rinse the chuck with water and remove the excess wax around the wafer with acetone.
5. Measure the samples with a micrometer - the thickness should be less than 30 microns difference between any two corners.
6. Place 1 teaspoon SiC 600 grit in the black puck, add 8 drops of water, and shake to suspend.
7. Hold and steel chuck and slowly grind it against the SiC suspension in circular sweeps. It is important to apply uniform pressure on the entire wafer so as not make the surface uneven.
8. Periodically check the thickness of the wafer and stop as soon as you reach an average thickness of 180 – 200 microns throughout the wafer.
9. Heat the steel chuck until the wax melts and carefully remove the wafer from it. Remember, the wafer is very thin now, so handle carefully.
10. Rinse the wafer with acetone and IPA.



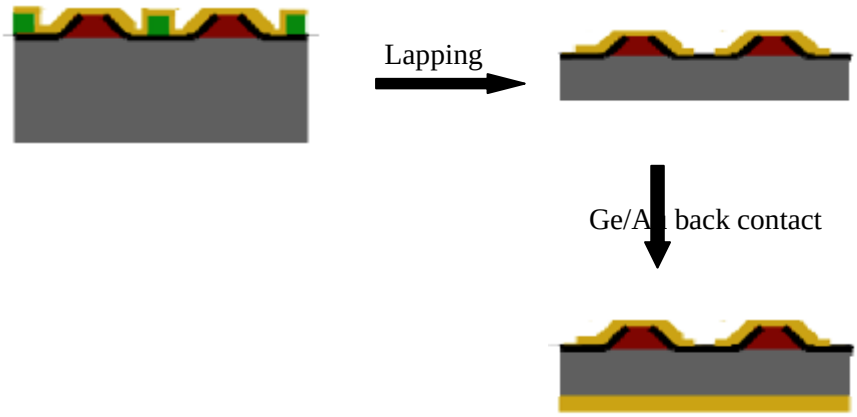
B. Hard Descum:

Prior to back contact deposition, clean the backside of the wafer by doing a hard descum on the RIE system following the procedure given in lab 4.

C. Back Contact Evaporation:

As we did in the last lab, load the sample into the evaporator and pump down the chamber. For back side contacts, we will be deposition 150 A of Germanium and 3000 A of Gold.

Once the evaporation is done, the wafer is ready to be mounted, cleaved and wire bonded, which we will be doing in the next lab.



Laboratory 7: CLEAVING, MOUNTING, WIRE BONDING

A. Cleaving:

You have to cleave the wafer into laser bars of different cavity length. Cavity length plays an important role in the operation of lasers. A shorter cavity would mean a lower input current while at the same time increasing the losses. Thus, there is a tradeoff between low input power and compensating losses in the cavity. In this lab, we will be cleaving the wafer into standard bars of 3 mm cavity length.

1. Place the wafer on an adhesive tape.
2. Mount it on the sample stage such that the laser bars are parallel to the direction of motion of the scribe.
3. Switch on the vacuum.
4. Slowly, bring down the scribe a little inside edge of the wafer where you want to cleave and scratch the wafer.
5. Drag the scribe to the edge and lift it. Repeat this process for about 8 times until you've made a visible mark on the wafer. You should careful not to scratch the wafer at different positions each time you go back.
6. Remove the wafer from the stage and place it upside down on a soft surface.
7. Using super fine tweezers, gently tap the area where you made the mark with the scribe to cleave the sample.
8. Upon cleaving, carefully remove the sample from the adhesive tape and proceed to mounting.

B. Mounting:

The wafer is mounted on to a copper block using Indium paste. Follow the procedure given below to mount the wafer and the gold pads.

1. Use minimum quantity of Indium paste to attach the wafer to the copper block and press it lightly with a cotton swab.
2. Make sure the cleaved edge of the wafer exactly aligns with the edge of the copper block. If the wafer comes out of the copper block, heat extraction would be poor and will lead to device failure. If the wafer is well inside the copper block, the light coming out of the laser would be instantly scattered before it reaches the detector.
3. Set the hot plate at 120 C.
4. Clamp this set up of the wafer and the copper block onto the hot plate and slowly use the needles to press down the wafer.
5. Increase the temperature to 190 C and stay there for about 2 minutes.
6. With the wafer still clamped in, reduce the temperature to 120 C.

7. As the temperature drops to below 130 C, take the copper block out and attach the gold pads using the paste.
8. Heat this to 120 C while holding down the gold pads with wooden sticks.
9. After about 2 minutes, remove the copper block from the hot plate and allow it to cool down to room temperature.

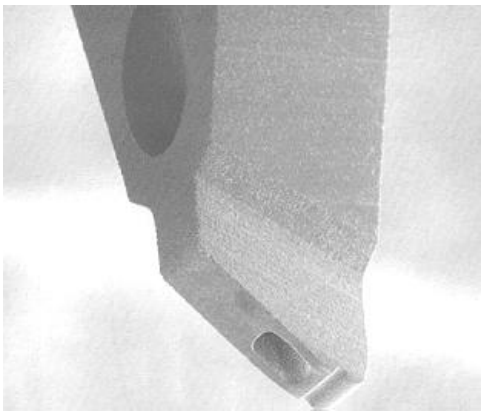
C. Wire Bonding:

Once the wafer is mounted along with the gold pads onto a copper block, you have to make contacts to your device from the pads. This process is wire bonding. This involves connecting a thin Gold wire (25 microns in diameter) from the device of interest to the gold pads. This wire bonding can be accomplished in many ways – thermocompression bonding, thermosonic bonding and ultrasonic bonding.

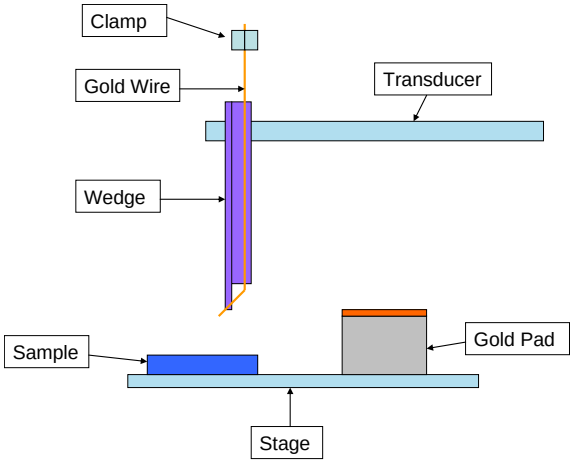
We would be using a thermosonic bonding procedure. This involves the use of force, time, ultrasonics and heat to join two materials. The gold wire is pressed against the hot surface (at 150 deg. C or less) at low force and vibrated for a limited period of time to achieve the bond. Ultrasonic energy, when applied to metal to be bonded, renders it temporarily soft and plastic. This causes the metal to flow under pressure. The acoustic energy frees molecules and dislocates them from their pinned positions which allows the metal to flow under the low compressive forces of the bond. Thus heat at the bond site becomes a byproduct of the bonding process.The friction of the wire breaks up and sweeps aside some contaminants in the weld area exposing clean metallic surfaces which promote the metallurgical bonds. It is important, however, to begin with a clean surface to avoid difficulties or failures in bonding.

1. Switch on the main power to the bonder machine, the lamps, the ultrasonic power source and the heater.
2. Carefully clamp the mounted copper block on to the sample stage, making sure it is vertically aligned with the bonding needle.
3. Check the power levels and the bond number.
4. The first bond should always be on a pad. Gently lower the lever onto the gold pad until you hear a clicking sound.
5. Now, carefully take the level to your device of interest and pat it gently until you hear the clicking sound. The bond is now complete and you can make another bond starting again from step 3.

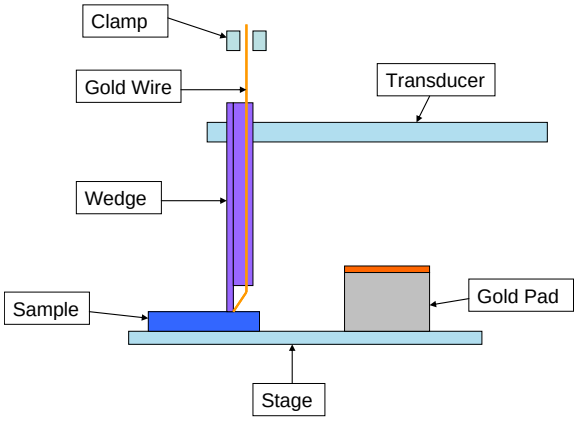
A schematic of the wire bonding process is given below:



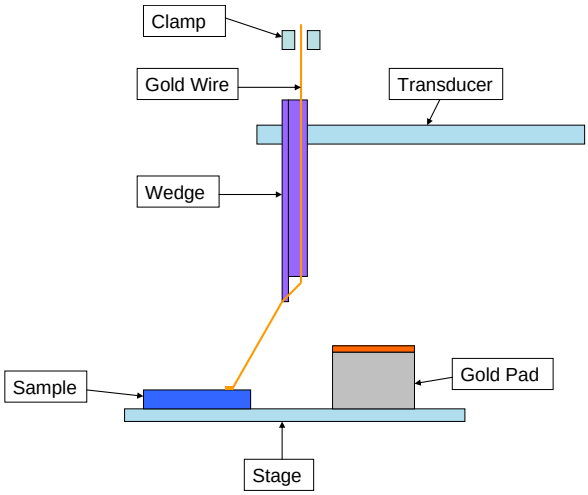
Wedge head structure



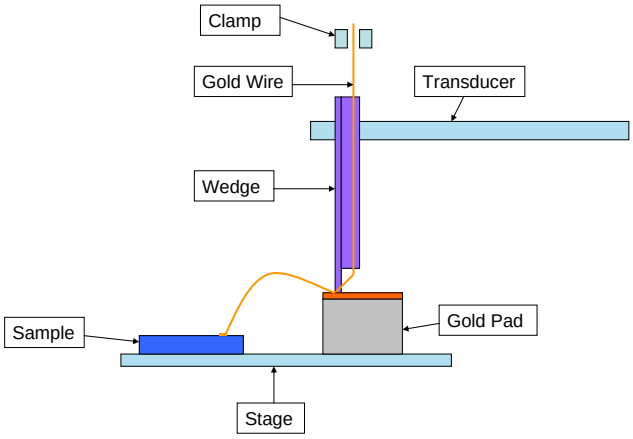
Step 1. Bring wedge near the sample. Clamp is closed at this point, gold wire sticking out from the tip.



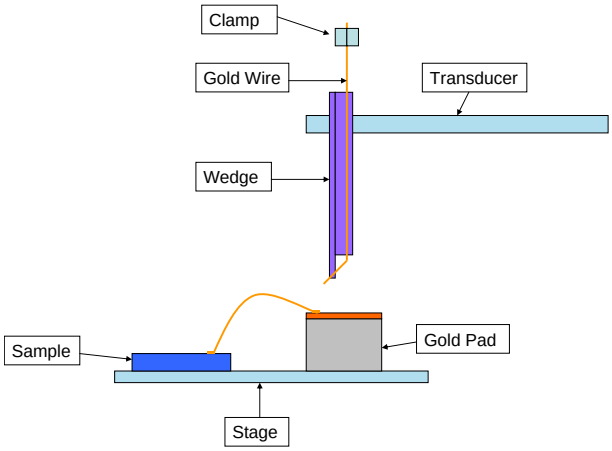
Step 2. Let the tip touch the sample, gently. Ultrasonic wave will be automatically applied to the wire. Clamp opens now.



Step 3. Lift the wedge to a sufficient height. Wire will be pulled out. Move the wedge to the gold pad.



Step 4. Perform the second bond. Curvature is automatically formed in the wire.



Step 5. Clamp is closed after second bond. Lift the wedge again, and the wire will break automatically.