BNC Staff support: Neil Dilley, ndilley@purdue.edu (858) 882-7990

SOP: AJA Ion Mill

Usual Method: load sample via the Load Lock

All users will load samples this way, unless there is specific permission given to vent the main chamber and load the carrier by hand. That is needed only in cases where longer mill time and higher beam energies are used (e.g., milling 1 um of hard material like LiNbO3).

Preparing Sample

- The sample carrier (called the "substrate" by AJA) is TiN coated and has 6 pins along the perimeter, and we will mount samples to that.
- To avoid milling the carrier itself, we recommend covering it with a 6" Si wafer that has a cutout for the sample. Some of these cut out wafers will be provided at the AJA ion mill. See the "Sample Requirements and Preparation" section of Wiki page for AJA about these wafers.
- Three clamps hold the wafer down at the perimeter and are screwed down using the included Allen key for these is provided (see photo).

- For best heat sinking during milling, the sample should be directly bonded to the TiN coated carrier using the white thermal compound (AI Technology, part # CGR7016).
- Press the sample down to get a bond which is completely covering back of sample and also thin but remove excess thermal compound which oozes out from under the sample (scrape with a glass slide or swab). Don't leave air pockets under the sample, but please also conserve this grease, it is $100/can!
- Your sample should be less than 4cm from the middle of the carrier to ensure uniform and high argon milling (see Appendix for milling profile).
- Place the carrier in the cassette with the pins resting in cassette's pockets as shown in photo below.
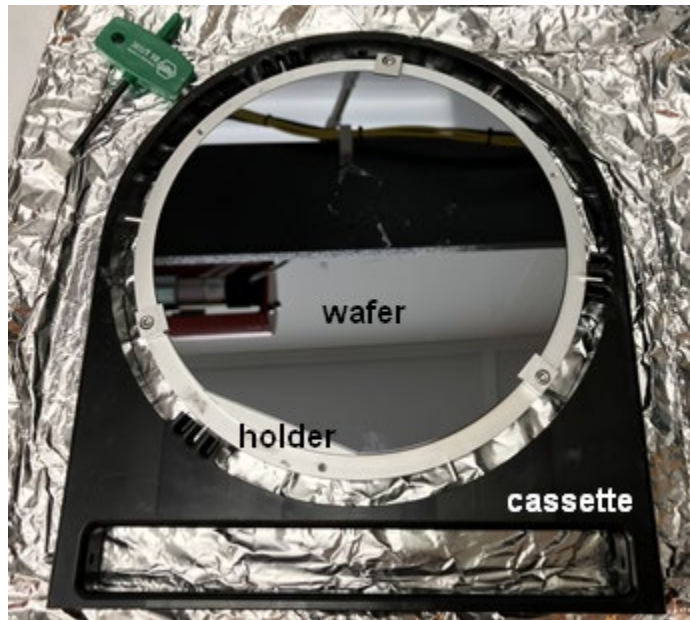
System Startup

- Start your session in iLab: this will provide power to the KRI ion source power supplies.
- Log into the Phase-IIJ software from AJA using the username you were given. Press Enter to get by the initial configuration screen, if it comes up. You will then see the main screen as shown to the right.
- It is very important to use the correct username since permissions for each user are set according to their training. If an incorrect username is used and damage to the instrument occurs, the user and their group is liable for this!
- Ensure that the pressure at the chamber ion gauge is in the low e-7 torr range or lower, as read on the MKS controller. Ion gauge is shown as "HC" (hot cathode) on the controller as is on the first line. Ion gauge may be off, in which case you should ensure that the green LED by the ion gauge reading is lit (cursor UP if not), then press "Sensor On/Off" and wait a couple minutes to let it equilibrate. If pressure is too high, please STOP and contact the staff engineer.
- **IMPORTANT: Make an entry in the e-log at the tablet noting your sample chemistry** and be aware that there are some parameters we need you to record DURING etch for benefit of your processes (RF power, Accel mA, …).
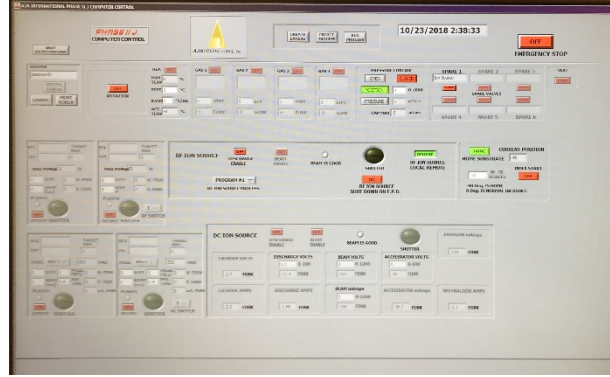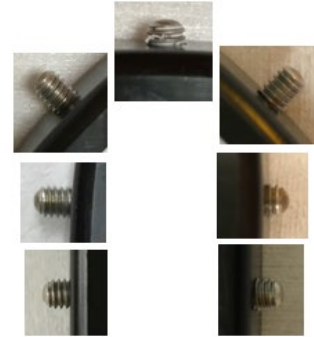
Loading Sample

- Check the status of the shutter at the Hiden EPD (left side of system, see photo): if the lever with the black knob is fully counterclockwise (CCW) and the amber LED there is lit, then the shutter is CLOSED and we are OK to proceed. If NOT, then close it by dialing the wheel (shown in photo) to compress the bellows BY 2-3 TURNS BUT NO MORE and move the shutter away from the Hiden detector opening. Then rotate the knob CCW and rotate the wheel back to bring the shutter plate back. The amber LED should be lit.

- Home the substrate in the main chamber using the "HOME" command in Phase-IIJ software. This sets the stage horizontally (angle = -90 deg) so it is ready to receive the carrier via the transfer arm.
- (breakpoint for Advanced Users doing deep etching)
- Raise the transfer arm until it reaches limit switch (amber LED at top of travel will light up)
- Ensure gate valve is closed
- To vent the load lock, shut off breaker labeled "LOAD LOCK" on main AJA console. This will automatically vent it over a few minutes. While the pressure is coming up, release the screw on the clamp which holds the load lock door closed (it will be harder to release when there is air pressure in the load lock). Monitor the pressure on the load lock gauge to see it come up to 760 torr.
- Open load lock door.

- Slide in the sample carrier/cassette assembly all the way
- Lower the transfer arm (down on the joystick at motor controller) to bring hooks into view and use your fingers to rotate the arm so the hooks avoid the pins and cassette parts. Orient the hooks so they are on the RIGHT side of the pins they will grab.
- If the transfer arm does not engage the cassette because it runs into the carrier or cassette or the hooks miss the pins, make sure the set screws are adjusted as shown in photo collage to the right; PLEASE DO NOT ADJUST THE SET SCREWS, instead find the engineer in charge.
- Lower transfer arm until hooks are below pins, rotate transfer arm and raise it so that the pins are captured as shown in the photo. Raise transfer arm until it reaches the upper limit switch.
- Remove the cassette from the load lock.
- Close the load lock door and turn on the load lock breaker.
- Wait until pressure in load lock reaches middle of e-5 torr range. If does NOT reach this pressure range, then vent load lock again, clean the o-ring sealing surface at the door and pump down again.
- Open blinds on view ports and turn on the LED lamp to see inside the chamber better.
- Open the gate valve fully.
- Using joystick, lower the transfer arm until screw on bottom of sample carrier just comes into contact with the threaded hole of the stage in the chamber (it takes a while to move the arm down).
- Engage the carrier by manually turning the screw drive on the top of the transfer arm (it is a normal screw, so turn clockwise as viewed from top).
- As needed, move the transfer arm down to keep the pins away from the bottom of the hooks on transfer arm.
- Continue until the carrier is tight. DO NOT TRY TO OVERTIGHTEN! You will use the reverse method to remove the carrier after done, and if it's too tight then you won't be able to get it loose. You will notice that the rotator stage starts turning if you turn hard enough, so this does also limit the amount of torque you can apply.
- Rotate the transfer arm so YOU ARE SURE the hooks are out of the way, then raise arm all the way to the top of travel (until limit switch reached).
- Close gate valve fully until it clicks.
- Keep the load lock pump running at all times unless you are opening the load lock.
- Wait for the main chamber to pump down into the e-7 torr range before starting to mill.
- In the Phase-IIJ software, call up the process you will be using. See Appendix below for definitions of terms in software (process, layer, program).
- Close the blinds at the view port windows to protect the glass from sputtered material.
- The engineer in charge will train you on specific processes, but here are some general rules which should be built into processes that are provided to you:
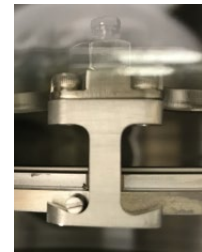
- o Run the discharge alone for 5 minutes before turning on the beam ("beam enable delay").
  - o A Home Holder layer must be placed at the end of the process so that the carrier can be retrieved by the transfer arm.
  - o If at any time you need to stop the milling immediately, press the "Abort Process" button at the top of the screen in the Phase-IIJ software. Please do this only in urgent situations and do not make this a standard way to switch off the beam.
- The Hiden Ion Milling Probe with End Point Detection can be used to either manually monitor an element during milling, or automatically trigger the milling to stop when an element rate count rises or falls. Instructions on its use are in the next section below.

Removing Sample

- When done with the milling, make sure the carrier is homed and rotation stopped.
- Verify that the load lock pressure is middle e-5 torr or lower.
- Open load lock gate valve FULLY.
- Open blinds on view ports and turn on the LED lamp to see inside the chamber better.
- Lower transfer arm with joystick…
- …all the way down to the pins, but this time put the hooks on the OPPOSITE side of the pins since we will be unscrewing the carrier, see photo.
- Unscrew the carrier slowly, raising the transfer arm as needed to keep the pin in the middle of the hook as shown in photo. DO NOT PULL ON THE PINS WHILE THE CARRIER IS SCREWED IN.
- Unscrew carrier until it comes out of the main threads and "clicks" down, indicating it is unthreaded.
- Lift the carrier in the transfer arm all the way up to the top limit switch (amber LED will light).
- Close the load lock gate valve until it CLICKS.
- Turn off the load lock pump breaker and let it vent.
- Open load lock door and place the cassette inside to receive the carrier.
- Lower the carrier with the joystick and rotate it so the three available pins fall in the slots of cassette.
- Lower the transfer arm to get the hooks below pins, rotate the hooks out of the way and lift up the arm to the limit switch.
- Remove carrier/cassette, close the load lock door and pump it down again by turning on the load lock breaker.
- Ensure load lock pumps down to 5e-5 torr, and if not then contact the engineer.
- When removing a sample from the carrier, note that the thermal grease can harden during milling so you must slide it carefully to release the bond, then try to get under it. Might be easier to remove the masking wafer first.
- To get the thermal grease off the back of sample: wipe with an alcohol-wetted swab, it removes easily. Sonicate in IPA if needed.
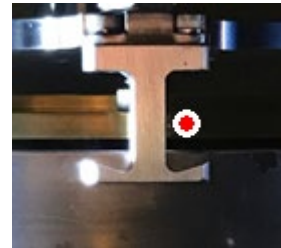
Ending Session

- Copy any data files (e.g., the experiment files for EPD) to your USB drive.
- Make sure the chamber pressure is in the e-7 torr range after process is done (if the ion gauge is off press "sensor on/off" button).
- LOGOUT of Phase-IIJ software, so the next user see the login screen.
- Clean up the work area to remove any trash and samples (hopefully these are not the same!)
- Stop you iLab session, which will cut power to the ion source power supplies.

Hiden End Point Detection (EPD)

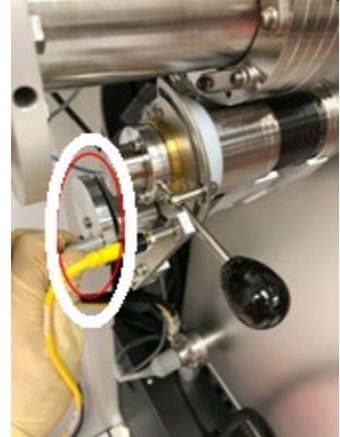Contact at Hiden for EPD/SIMS support: Luke Levin-Pompetski **(734) 542-6666** *or* **(888)-964-4336**

Before you begin, note that here are two methods of using the EPD:

- *Passive*: monitors the species coming off the sample and requires the user to stop the milling manually (abort process) based on rising/falling counts of a species; this is useful in process development and I think it will be the usual method we use. To use this method:
    - o In Hiden MASsoft program: erase any parameters that appear in the Events box of your template so that the template is not dependent on the analog "source shutter open" signal from AJA system.
    - o In Phase-IIJ: the "shut down on EPD" toggle should be OFF since MASsoft will not be sending a shutdown signal.
- *Active*: an EPD condition is set and the MASsoft program sends a signal to AJA to stop the process. For instance, you can trigger on a 50% fall in cobalt signal. Please see MASsoft Professional Manual Addendum on Wiki for details of EPD conditions you can set. This should be done only if you have a well-established milling recipe and an EPD behavior that you have tested. To use this method:
    - o In MASsoft: create a template based on an existing active EPD experiment file: *CoFeB PMA stack_active EPD.exp*
    - o In Phase-IIJ: Set "shut down on EPD" to ON in your milling layer. The layer will end upon an EPD signal OR the end of "mill time" interval (this ensures that the milling doesn't go on indefinitely). So, set the mill time to longer than expected to reach the endpoint.
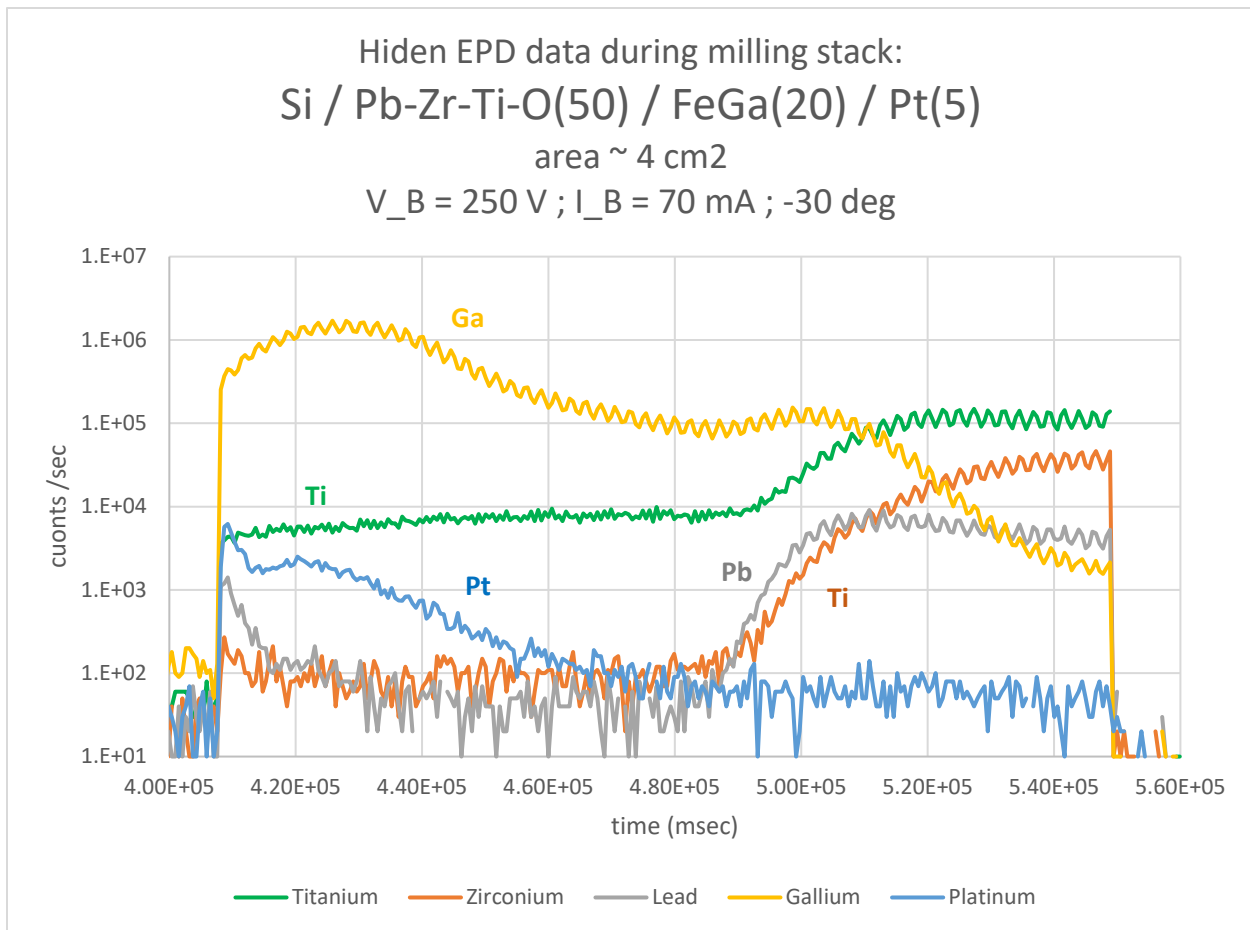
SOP for EPD

- open the shutter at the Hiden: dial the wheel (shown in photo below) to compress the bellows BY 2-3 TURNS BUT NO MORE. This moves the shutter away from Hiden aperture. Then rotate the shutter rod clockwise (viewed from outside); this puts the opening in the shutter plate in front of the Hiden aperture. Finally, dial back the wheel to bring shutter plate closer to the aperture (the amber LED will NOT be lit when the shutter is open).
- pressure gauge at Hiden should be reading in the e-8 torr range when the ion source is off; if not, contact the engineer in charge.
- turn on Hiden MSIU power and wait for it to go through its startup (1 minute).
- start MASsoft program.

- In MASsoft, open template file: check that the ion species being detected are appropriate to your sample; check the endpoint condition (active) or ensure that the events box is empty (passive).
- In the bar at the top of the window for this experiment, it should state "available", meaning that the scan is ready to be run. If not, stop any milling and quit/restart MASsoft.
- MASsoft: if a scan view window is not already opening with the template, open a single scan view that plots all the species being scanned. (Ctrl-click each scan line and Gtrl+G to make new plot). To get desired plot colors for each species, set up view using Ctrl-T.
- It is highly recommended in the plot to right-click the Y-axis and select Y-axis… > Log scale so that smaller signals can be seen. Some elements SEM counts are 100-1000x higher than others!
    - SEM = secondary electron multiplier: this is the detector for EPD.
- MASsoft: press the toolbar Go button ▶ (green arrow) , and a dialog comes up where you select the name of .exp file where you will save this data. Then:
    - Passive mode: EPD scan will start immediately (after a few seconds of setup)
    - Active mode: EPD scan starts once the source shutter is opened and beam is turned on.
- Phase-IIJ: run process, which should start discharge and warm up (5 minutes), then turn on the beam.
- check pressure at Hiden, needs to be in range 1e-6 to 5e-6 torr (there is a protection shutoff trip point in Hiden at 8e-6 torr).
- **make sure counts for each are <1e7 c/sec** (1e5 is a good target). If higher than this, stop milling by pressing "Abort Process" at top of screen in Phase-IIJ software and contact engineer.
- If using active MASsoft template, monitor to see that EPD triggers milling to stop.
- If using passive template, you need to stop EPD scanning and milling independently.
- when done milling, close the Hiden shutter by reversing steps at the beginning of this section so that the amber LED is again lit, indicating the shutter is fully closed.
- Close MASsoft program.
- copy your experiment datafiles (all 6 of them) to USB drive, if desired. You can export data in MasSoft using File > Export > File .CSV. The .CSV will have plottable data for each species detected, in time step of milliseconds.
- turn off Hiden MSIU power switch.

Example EPD data below:

Hiden EPD data during milling stack:
Si / Pb-Zr-Ti-O(50) / FeGa(20) / Pt(5)
area ~ 4 cm2
V_B = 250 V ; I_B = 70 mA ; -30 deg

Important note about etching oxides: the presence of oxygen in the chamber, due mainly to milled oxide material, will lead to an "Oxygen Effect" and to greatly enhanced signals for most metals. This is because the highly electronegative O2 will grab an electron from the metal M making it M+, so there will be many more M+ ions around than usual. Note that most of the milled material comes off as unionized atoms or molecules, so it is possible to greatly distort the picture if suddenly a much higher fraction of the milled species are positively ionized.

For example, if milling Ta on top of SiO2, the Ta signal will be artificially boosted when we get to SiO2 so you will think that you're still milling Ta but instead you've amplified the trailing drop off in Ta signal because the oxygen is ionizing much more of the remaining Ta to Ta+. In this case, we wanted to stop before the Ta layer was etched so a solution was to look for the falling edge of the previous layer (Ru) and etch a little longer to make sure we are down to the Ta layer.

Appendix

AJA service support: Joe Cronin (781) 545-7365
KRI (ion source) service support: Richie Nethery (970) 232-3448 x350

First, some definitions:

- **CARRIER**: the plate which clamps your wafer; our term for what AJA calls the SUBSTRATE in their software
- **LAYER**: the basic commands for the software. In the layer you will specify things like:
  - if carrier rotation is on/off
  - the angle of the carrier
  - how long to run the discharge before turning on the beam
  - whether you're using End Point Detection to stop the milling
  - whether to leave beam or discharge on at end of layer
  - you can also make a layer in which Ar flows through the neutralizer to cool off filament and nothing else
- **PROCESS**: layers can be strung together into a process, and this is the executable which is run by the software. Typically process examples are: milling + home holder, or mill + cooling (repeated as needed) + home holder
- **PROGRAM**: ion source controller runs according to one of 4 programs which have preset and approved values of beam voltage, beam current, RF power, etc. These programs should NOT be changed by users.

Below are the programs that were put into the KRI Auto Controller on 11/8/2018 with help from Joe Cronin of AJA. Please **DO NOT CHANGE THESE PROGRAMS!** The parameters are chosen to satisfy conditions in tables 3-1 and 3-2 from KRI ion source manual, posted below.

Note to engineer on setting the RF forward power: the preset below should be set to the value at which it ultimately stabilizes for the given flow/voltage/current parameters, otherwise the beam current will be unstable until this RF power reaches its stable value (this value is reached by adjusting the RF frequency to minimize the reflected RF power).

Program #	
Flow [sccm]	
V_beam [V]	V_acc [V]
I_beam [mA]	I_acc [mA]
RF forward power [W]	
Typ. Etch rate for Si [nm/min]	

# 1	
13	
250	38
35	1000
80	

# 2	
13	
250	38
70	1000
135	
9	

# 3	
16	
600	90
225	1000
310	
53	

# 4	
16	
600	90
72	1000
101	

Old programs, initially loaded on tool during install:

#1	
11	
150	26
30	1000
79	

#4	
20	
800	120
330	1000
440	

Conversion: Gas 1 flow to chamber pressure

14 sccm = 1.2 e-4 T
40 sccm = 5 e-4 T – this is the maximum flow/pressure we should use!!

The tables below (taken from RFICP 140 Ion Source Manual) are critical for setting program values and were used in designing programs 1-4 on the Auto Controller. USERS SHOULD NOT WRITE NEW PROGRAMS WITHOUT THE ASSISTANCE OF STAFF. How to use them: in Table 3-2 **select the beam voltage you want**. The beam voltage is the kinetic energy of the Ar atoms that will be striking the surface, *and every material has some minimum energy requirement in order to knock material off*. We use 250 V (program #2) for thin CoFeB/Ta/MgO stacks, while 600 V (program #3) is used for thick LiNbO3 films. If you're above this critical beam voltage, then etch rate is determined by the ion beam current. Table 3-2 tells you the maximum beam current that you can use for a given voltage, and also tells you the recommended accel V and RF power to set. Note that when the beam turns on, the RF power will adjust (could be higher or lower than set point) in order to give us the requested beam current.
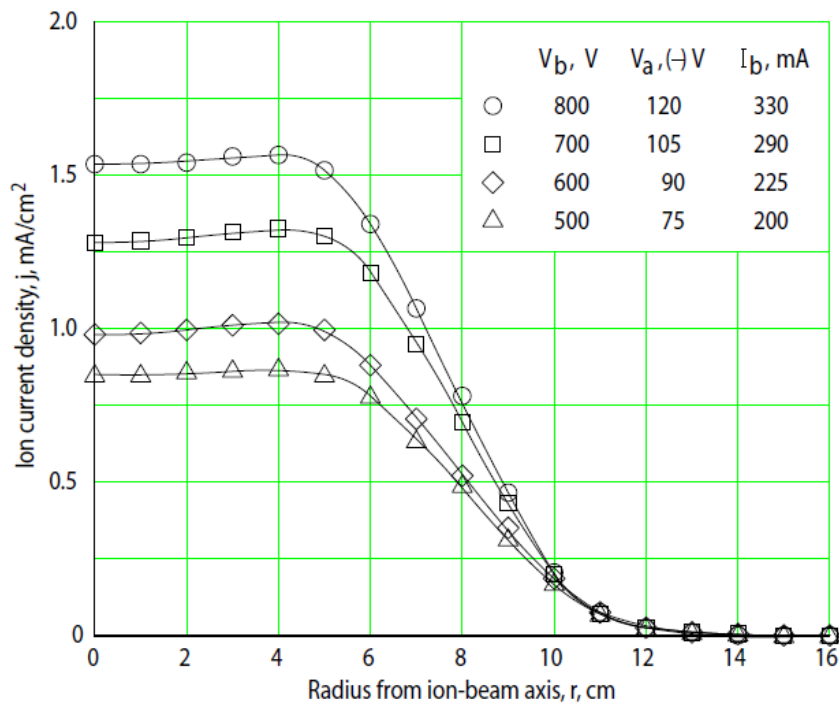
Table 3-2 Maximum argon ion beam current

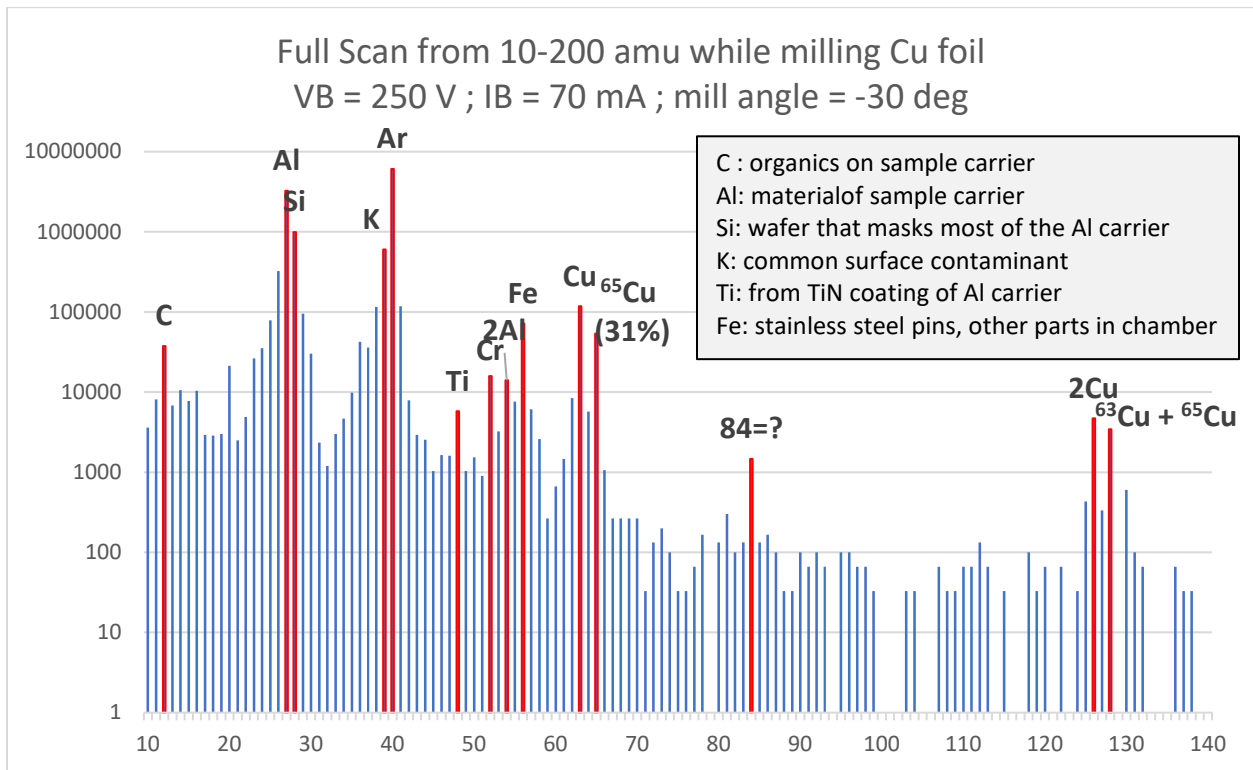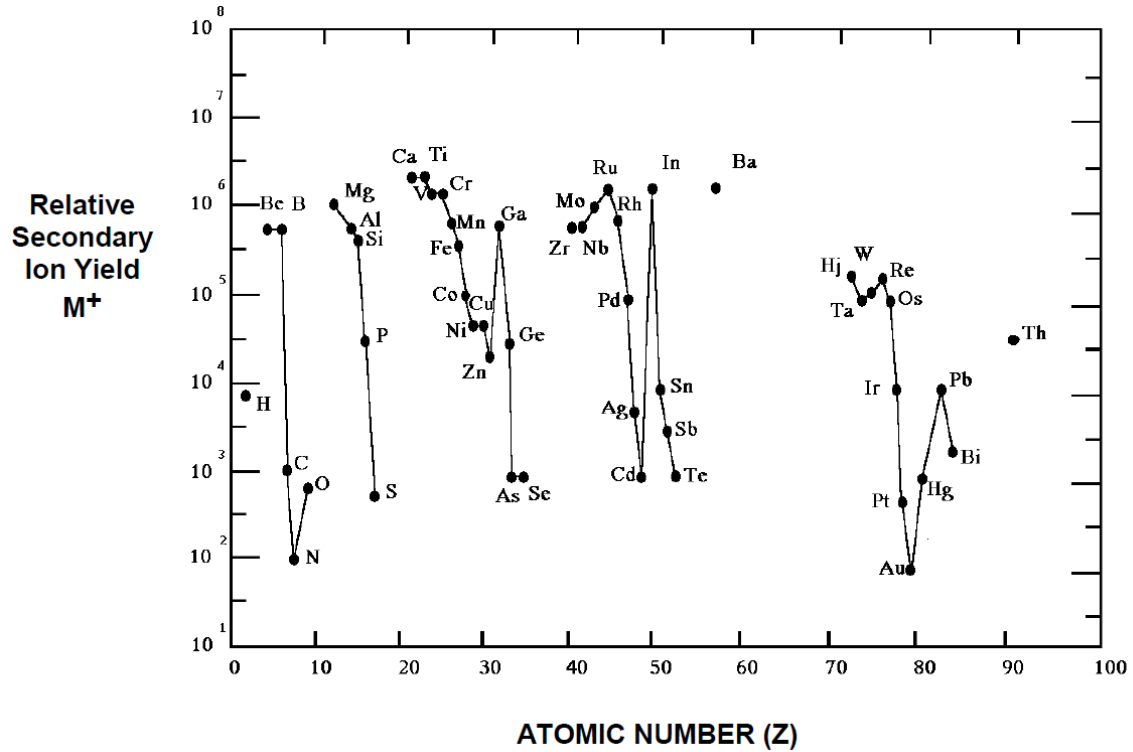| Beam, V	Accel,(-)V	Ion beam current, mA	Approx. rf power, W
100	15	15	70
200	30	45	100
300	45	110	170
400	60	150	220
500	75	200	280
600	90	225	310
700	105	290	390
800	120	330	440
900	135	440	600
1000	150	490	670
1100	165	550	760
1200	180	620	870

Start by selecting a beam voltage. Milling rate increases with beam voltage, but so does substrate heating, so we typically use 250 V for thin layers (up to 50nm) and 600 V for thick layers (~um).

Table 3-1 Argon gas flow in sccm.

Pressure, Torr	Ion beam current, mA							
	100	200	300	400	500	600	700	800
0.5×10^{-4}	18	22	26	31	37	43	50	56
1.0×10^{-4}	16	20	24	29	35	41	47	54
1.5×10^{-4}	14	18	22	27	33	39	45	52
2.0×10^{-4}	12	16	20	25	31	37	43	50
2.5×10^{-4}	10	14	18	23	29	35	41	48
3.0×10^{-4}	8	12	16	21	27	33	39	45
3.5×10^{-4}	6	10	14	19	24	31	37	43

	V_b, V	V_a, (-)V	I_b, mA
○	800	120	330
□	700	105	290
◇	600	90	225
△	500	75	200

Ion current density, j, mA/cm^2 — Radius from ion-beam axis, r, cm

Put your sample within 4cm of the center of the carrier. This ensures a maximal and uniform etch of your sample and also best sensitivity of the Hiden EPD to sample material.

Relative Secondary Ion Yield M^+ vs. ATOMIC NUMBER (Z)

Full Scan from 10-200 amu while milling Cu foil
VB = 250 V ; IB = 70 mA ; mill angle = -30 deg

C : organics on sample carrier
Al: materialof sample carrier
Si: wafer that masks most of the Al carrier
K: common surface contaminant
Ti: from TiN coating of Al carrier
Fe: stainless steel pins, other parts in chamber

from "Applications of Broad-Beam Ion Sources: An Introduction"
Kaufman & Robinson, Inc. (2011)
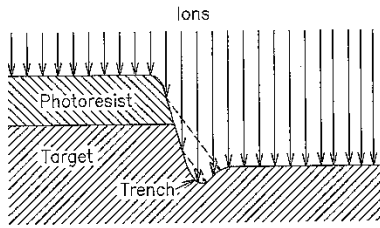
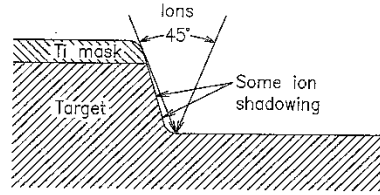Fig. 6-13. Trench formed in GaAs by ions reflected from sloped surface. (After ref. 23.)
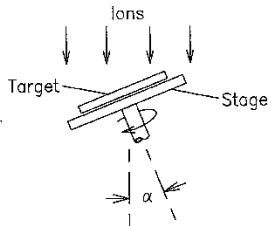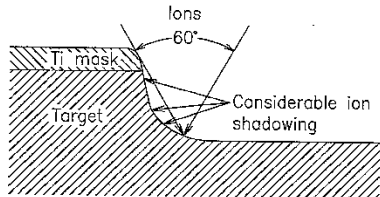

Fig. 6-14. Etching with a rotating, tilted stage. Tilt angle = α.

6-24
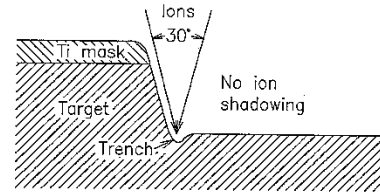
(a) Tilt angle, α, is 15°. Trench is still present.

(b) Tilt angle, α, is 22.5°. Well-defined corner.

(c) Tilt angle, α, is 30°. Corner is rounded.

Fig. 6-15. Effect of tilt angle on the etch-profile shape in GaAs. See Fig. 14 for tilt-angle definition. (After ref. 23.)
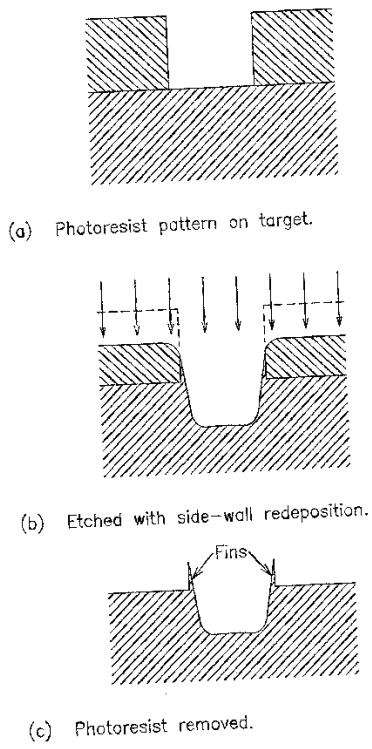
6-25

(a) Photoresist pattern on target.

(b) Etched with side-wall redeposition.

Fins

(c) Photoresist removed.

Fig. 6-16. Etched photoresist pattern, with redeposition on side walls of photoresist.

6-26

(a) Etched with side-wall redeposition.

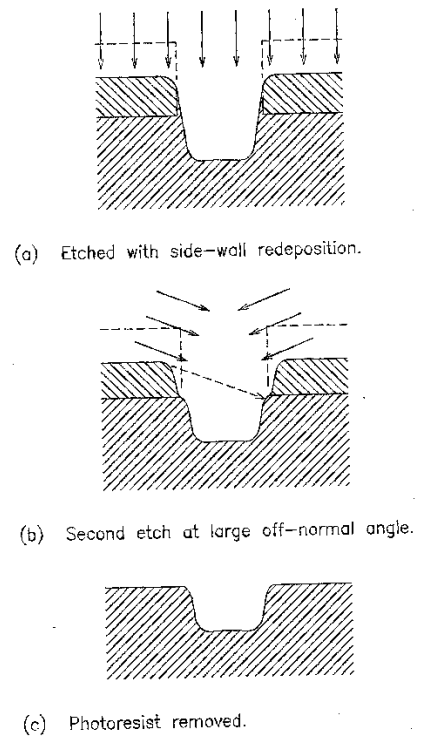(b) Second etch at large off-normal angle.

(c) Photoresist removed.

Fig. 6-17. Etched photoresist pattern, with two-step etch.

6-27