

Short instructions for Precision Etching Coating System

WARNINGS

- The samples must be DRIED, without particles which could be pulled by vacuum into the system! The system is more sensitive than FEG SEM! Special care with magnetic particles!
- The voltage should never be 10 keV (except etching)
- The current should not exceed 425 μA !!
- When starting check that the pressure is $< 10^{-3}$ Pa
- Do not touch the inner part of the holder and it is mandatory use of gloves!
- When you click VENT / VAC always wait for the light to turn off and then re-click the button! Look at the LCD monitor (MDP must be 100%)
- Read the manufacturer's instructions!
- Check that the flow of argon 5 bar.

Initial settings

- The device is switched on (green switch)
- Vacuum $< 5 \cdot 10^{-3}$ Pa (if you turn off purging)
- Foreline between 3 and 4 Torr
- The speed of turbo molecular pump is 100% (MDP)
- HT voltage is switched off (no red light)
- HT potentiometer on zero
- Coating ion gun: ON (purging!)
- Etching ion gun: OFF
- Gas: Argon
- Shutter closed (screw below)
- Quartz scale closed (position shield)
- Chamber lighting off

Inserting the sample

- Switch "fixed" is OFF
- Potentiometers for the angle and tilt on the minimum ("rock")
- Switch "rotation" is OFF
- Potentiometer for the rotation speed on the minimum
- Detach the cable for the rotation of the sample
- Move switch "airlock CONTROL" to "OUT"
- When it comes out, click "VENT" (several times)
- Take it out gently (watch the o-ring!)
- Insert the adapter with the samples
- SLIGHTLY push the holder inside (horizontally) and watch the pin
- Click "VAC" (MDP 100%) (several times)
- Move switch "airlock CONTROL" to "IN"
- Connect the cable for the rotation of the sample

Settings on the coater

- Shutter closed (screw facing down)
- detector for measuring the thickness closed - position "SHIELD"
- Turn on the **thickness MONITOR**
 - click "STOP" (reset the P fail) - green light ON
 - click the "FILM NUMBER" and use the buttons up and down, to select the target number:

1 = carbon (C)

2 = chromium (Cr) +LN₂ trap

3 = gold / palladium (Au/Pd)

4 = platinum (Pt)

- click the "START" button (reset the thickness to 0)

The stabilization of ion guns for coating

- Set "HIGH VOLTAGE TIMER" on 30 min
- Click the "START / STOP"
- **Slowly increase** the potentiometer to the voltage for sputtering (6 to 9 keV) and wait for the stable current
- If necessary, change the flows in the ion guns
- Max. current can be 425 μA , optimal around **350 μA** .

Inserting the target for sputtering

- **Turn off HT**
- Select the target and push it into the chamber – the label of the coating material is on top
- When it is inserted **turn on HT!**
- Clean the target only when the system is opened (look at the sheet glued on the wall!)

Sputtering SEM sample

- Optionally activate the tilt and rotation of the sample
- Check that the thickness on the thickness monitor is zero, otherwise press the "START" (0,01kA = 1nm)
- SIMULTANEOUSLY turn SHUTTER AND SHIELD
- When you reach the desired thickness close the SHUTTER. Write the coating time in the notebook.
- Close the SHIELD.
- Reduce the potentiometer "BEAM ENERGY" on 0 kV
- Take the target OUT
- Set the purging of the coating ion guns
- Turn OFF ROCK and ROTATE
- Turn OFF the THICKNESS MONITOR
- Click "START / STOP" if it did not itself turned off. Hold START / STOP to put the timer back to 30 min.

Sputtering TEM sample

- **Insert the sample with the holder (except analytical!)**
- Check that the thickness in the thickness monitor is zero, otherwise press the "START" (0,01kA = 1nm)
- SIMULTANEOUSLY turn SHUTTER AND SHIELD.
- When you reach the desired thickness close the SHUTTER. Write the coating time in the notebook.
- Close the SHIELD.
- Reduce the potentiometer "BEAM ENERGY" on 0 kV
- Take the target OUT
- Set the purging of the coating ion guns
- Turn OFF ROCK and ROTATE
- Turn OFF the THICKNESS MONITOR
- Click "START / STOP" if it did not itself turned off. Hold START / STOP to put the timer back to 30 min.

Etching of the sample

- **One hour before** – set purging!

Short instructions for Precision Etching Coating System

- Etching ion gun ON
- Coating ion gun OFF
- The vacuum should be $< 10^{-3}$ Pa (turn off the gun)
- Shutter closed (screw facing down)
- Targets out.
- Turn the switch for etching gun ON
- Set "HIGH VOLTAGE TIMER" on 30 min
- click the "START / STOP"
- Slowly increase the potentiometer to the desired voltage and wait until it is stable.
- Optionally activated inclination of the sample and the rotation.
- Check the time and turn the SHUTTER.
- When you reach the desired etching time close the SHUTTER and write the time in the notebook
- Reduce the potentiometer "BEAM ENERGY" on 0 kV
- Turn OFF the etching gun. Set the purging of the coating ion guns
- Turn OFF ROCK, ROTATE and TILT
- Click "START / STOP" if it did not itself turned off. Hold START / STOP to put the timer back to 30 min

Taking the sample out

- Detach the cable for the rotation of the sample
- Move switch "airlock CONTROL" to "OUT"
- When the holder comes out click "VENT" (several times)
- Take it out gently (watch the o-ring!)
- Take out the adapter with the samples
- SLIGHTLY push the holder inside (horizontally) and watch the pin
- Click "VAC" (MDP 100%) (several times)
- Move switch "airlock CONTROL" to "IN"
- Connect the cable for the rotation of the sample
- Write in the notebook

Hints:

C: 8-9 keV, L&D 280 -300 μ A, 0,2 - 0,4 $\text{\AA}/\text{s}$,
min 4 nm (2 min) **max 8 nm** (4 min)
Au/Pd: 6-7,5 keV, L&D 200 - 220 μ A, 1,2 - 1,3 $\text{\AA}/\text{s}$,
min 3 nm (25 s) **max 6 nm** (50 s)
Pt: 6-7 keV, L&D 200 - 220 μ A, 0,7 - 0,8 $\text{\AA}/\text{s}$,
min 3 nm (40 s) **max 6 nm** (80 s)
Cr: 6-7 keV, L&D 200 μ A, 0,5 - 0,7 $\text{\AA}/\text{s}$,
min 3 nm (1 min) **max 8 nm** (2,5 min) + LN₂ trap

Cleaning the ion guns (purging)

When the guns have been exposed to bad vacuum, or the PECS has been shut down for a few hours.

Cleaning the guns for coating

- HV voltage OFF
- Ion etching gun OFF
- Right coating gun is OFF
- Left gun ON and set the gas flow so that you have a vacuum of about $1,3 \cdot 10^{-2}$ Pa ($1 \cdot 10^{-4}$ Torr)
- Turn off the left coating gun
- Turn right rifle and adjust the gas flow so that you have a vacuum of about $1,3 \cdot 10^{-2}$ Pa ($1 \cdot 10^{-4}$ Torr)
- Turn both guns (left and right) and leave to act 20 minutes

Cleaning the etching gun

- HV voltage OFF
- Ion etching gun turned ON and set the flow of Ar to about $1,3 \cdot 10^{-2}$ Pa ($1 \cdot 10^{-4}$ Torr)
- Leave it to work for at least 15 minutes
- Right and left the gun OFF