Process parameters for FIB Milling Sapphire - TEM Sample Preperation

Tilt Stage to 54°

Step 1 - Platinum deposition

Milling mode	Width (µm)	Height (µm)	X frequency	Y frequency	Milling current	Time (s)	Gas ID 1	Gas ID 2	Gas wait time	Comments
Deposition mode	22	3	20,000	1	300 pA	480	None	Platinum	3	Can use GIS 1 or GIS 2

Step 2 - Carbon deposition

Milling mode	Width (µm)	Height (µm)	X frequency	Y frequency	Milling current	Time (s)	Gas ID 1	Gas ID 2	Gas wait time	Comments
Deposition mode	22	3	20,000	1	300 pA	480	Carbon	None	3	Carbon is available only on GIS 1

Step 3a - Trapezoidal milling 1

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	20	10	15	60	Al2O3	30	12 nA	0	No	Position the milling area ~0.5µm away from Pt/C deposition; Time ~19:54

Step 3b - Trapezoidal milling 2

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	20	10	15	60	Al2O3	30	12 nA	180	No	Use the flip option and ~5-6µm flip distance to position the milling area ~1µm away from Pt/C deposition; Time ~19:54

Step 4a - Rectangular milling - clean the sample walls - side 1

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	20	2	15	30	Al2O3	0	8 nA	0	No	Start slightly away from the sample; Time ~ 8:52

Step 4b - Rectangular milling - clean the sample walls - side 1

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	20	2	15	30	Al2O3	0	4 nA	0	No	Additional steps might be necessary if a clean surface is not obtained; Time ~ 14:12

Step 4c - Rectangular milling - clean the sample walls - side 2

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	20	2	15	30	Al2O3	0	8 nA	0	No	Use the flip option and ~3-4 µm flip distance to position the milling area ~1µm away from Pt/C deposition ; Time ~ 8:52

Step 4d - Rectangular milling - clean the sample walls - side 2

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	20	2	15	30	Al2O3	0	4 nA	0	No	Additional steps might be necessary if a clean surface is not obtained (can check only in FIB image); Time ~ 14:12

Tilt Stage to 0°

Step 5 - J-cut

Step 5a - bottom

Milling mode	Width (µm)	Height (µm)	-	No. of Layers	-	Time (s)	Milling current	Angle	Track WD	Comments
Mill for time	20	0.75	-	60	-	200	2 nA	180	No	Some adjustment in the position might be necessary for the cut to succeed. Watch for the dark line in the trench cut by trapezoidal cut. 200 - 240 s needed for this operation

Step 5b - Left side

Milling mode	Width (µm)	Height (µm)	-	No. of Layers	-	Time (s)	Milling current	Angle	Track WD	Comments
Mill for time	0.75	0.75	-	60	-	200	2 nA	180	No	Make sure the cut goes below the cut from step 5 a

Step 5c - Right side (partial)

Milling mode	Width (µm)	Height (µm)	-	No. of Layers	-	Time (s)	Milling current	Angle	Track WD	Comments
Mill for time	0.75	0.75	-	60	-	200	2 nA	180	No	Make sure the cut goes below the cut from step 5 a

Step 6 - Insert Omniprobe

Comments

- Disable touch alarm ->check SCM on; Can be accessed from Specimen Current Monitor Menu

Step 6a - Weld Omniprobe to sample

Milling mode	Width (µm)	Height (µm)	X frequency	Y frequency	Milling current	Time (s)	Gas ID 1	Gas ID 2	Gas wait time	Comments
Deposition mode	2.5	2.5	20,000	1	20 pA	300	None	Pt	3	This operation must use GIS 2. It will not work with GIS 1. Check if there is sufficient deposition

Step 5d - Right side (remaining material)

Milling mode	Width (µm)	Height (µm)	-	No. of Layers	-	Time (s)	Milling current	Angle	Track WD	Comments
Mill for time	0.75	0.75	-	60	-	200	2 nA	180	No	Make sure the sample is properly separated

Step 7 - TEM grid preparatory milling

Milling mode	Width (µm)	Height (µm)	-	No. of Layers	-	Time (s)	Milling current	Angle	Track WD	Comments
Mill for time	14	2	-	60	-	200	2 nA	180	No	This is to create a small pocket in the TEM grid to seat the sample; Width should be ~2-3 µm larger than sample width

Step 8 - Weld TEM lamella to grid

Milling mode	Width (µm)	Height (µm)	X frequency	Y frequency	Milling current	Time (s)	Gas ID 1	Gas ID 2	Gas wait time	Comments
Deposition mode	2.5	2.5	20,000	1	20 pA	300	None	Pt	3	This operation must use GIS 2. It will not work with GIS 1. Check if there is sufficient deposition; reposition and repeat as necessary (2-4 times)

Step 9 - Detach Omniprobe

Milling mode	Width (µm)	Height (µm)	-	No. of Layers	-	Time (s)	Milling current	Angle	Track WD	Comments
Mill for time	0.75	0.75	-	60	-	120	2 nA	180	No	Make sure the Omniprobe is detached from the sample; reposition and repeat if necessary

- Re-enable touch alarm -> uncheck SCM on; Can be accessed from Specimen Current Monitor Menu

Tilt Stage to 54°

Step 10a - Side 1 thinning (1 of 4)

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	17	1.5	10	1	Al2O3	0	2 nA	0	No	Start from slightly outside the sample; May need to be repeated if the sample is too thick

Step 10b - Side 1 thinning (2 of 4)

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	16	1	10	1	Al2O3	0	300 pA	0	No	May need to be repeated if the sample is too thick

Step 10c - Side 1 thinning (3of 4)

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	15	0.6	15	1	Silicon	0	140 pA	0	No	

Tilt Stage to 55.5°

Step 10d - Side 1 thinning (4 of 4)

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	14	0.5	15	1	Silicon	0	50 pA	0	No	

Stage Delta Rotation by 180°

Step 11a - Side 1 thinning (1 of 4)

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	17	0.7	10	1	Al2O3	0	2 nA	0	No	Start from slightly outside the sample; May need to be repeated if the sample is too thick

Step 11b - Side 1 thinning (2 of 4)

Milling mode	Width (µm)	Height (µm)	Depth (µm)	No. of layers	Material	Slope (°)	Milling current	Angle	Track WD	Comments
Mill for depth	16	0.4	15	1	Silicon	0	300 pA	0	No	If not already switched, must use SE2 detector from this stage

Step 11c - Side 2 thinning (3of 4)										
Milling mode	**Width (µm)**	**Height (µm)**	**Depth (µm)**	**No. of layers**	**Material**	**Slope (°)**	**Milling current**	**Angle**	**Track WD**	**Comments**
Mill for depth	15	0.2	15	1	Silicon	0	140 pA	0	No	

Step 11d - Side 1 thinning (4 of 4)										
Milling mode	**Width (µm)**	**Height (µm)**	**Depth (µm)**	**No. of layers**	**Material**	**Slope (°)**	**Milling current**	**Angle**	**Track WD**	**Comments**
Mill for depth	14	0.3	15	1	Silicon	0	50 pA	0	No	Select the entire sample as the height of the box; Watch for electron transparency

TEM sample preperation is complete!
Tilt stage to 0°

General Comments

- All milling/deposition done at 30 kV
- Red line of the rectangles/trapezium should face the sample area
- Disable touch alarm when using Omniprobe (check SCM on), re-enable touch alarm (uncheck SCM on) after detaching Omniprobe (Specimen Current Monitor Menu)
- Each time the GIS is inserted, check the positioning on the FIB image
- Constantly check the emission current (should be 2µA)
- If the FIB shuts off after deposition, outgas the Pt (when outgassing, make sure column chamber valve is closed (Airlock menu)); Outgas option can be accessed from Gas Injection System Menu
- If no deposition is happening, Pt could be empty - notify the staff