

TEM Sample Thinning Procedure for JIB-4700F by using the Bulk Pickup Method

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JEOL Ltd.

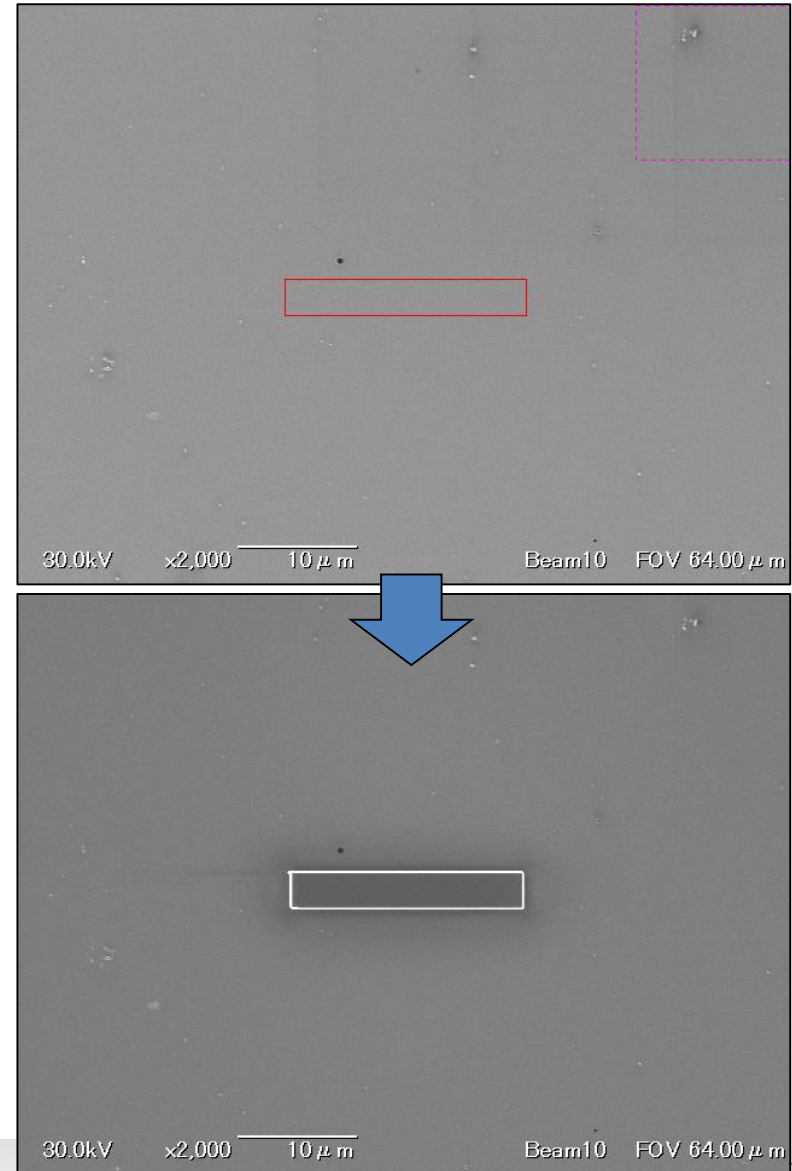
JEOL (UK) Calum Dickinson

Outline

1. Depositing a protective film
2. Rough Milling
3. Bottom Cut
4. Sample Block Separation
5. Pickup and attach to a FIB Grid
6. FIB Grid Milling
7. Thinning Sample
8. Amorphous layer removal with Ga Ion Beam

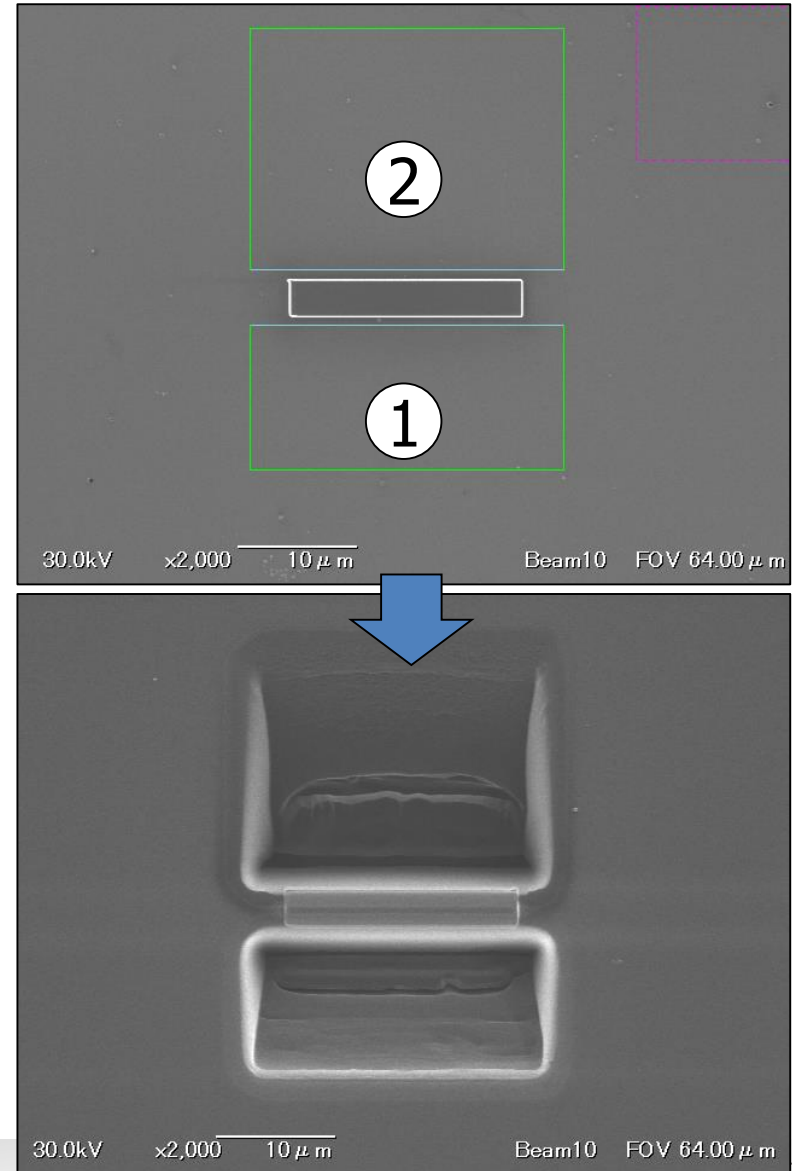
Depositing a protective film

- Tilt=53 °
- Mode: DEPO (Carbon)
- 30kV, 100pA (9) - 300pA (8)
- 20 μ m(x) , 3 μ m(y) Beam 8-9
- Dose=1.5 (thin) - 2.0 (thick)



Bulk cut(Rough milling) (1/2)

- Tilt=53 °
- Mode : RAPID Beam 2-4
- 30kV, 10nA (4) -50nA (2)
- Lower side –①
 - 27 μ m(x), 18 μ m(y)
 - Dose ~ 15
 - Finish: Top
- 上側Upper side –②
 - 27 μ m(x), 25 μ m(y)
 - Dose ~ 10
 - Finish: Bottom



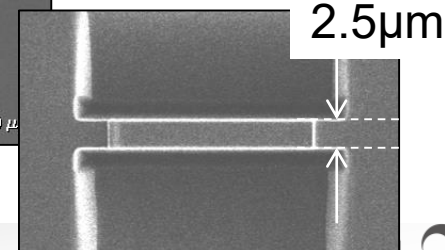
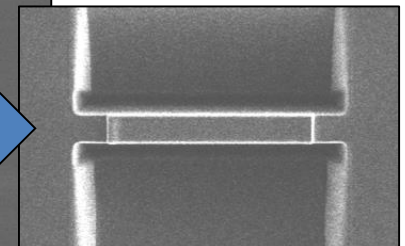
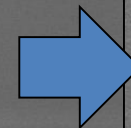
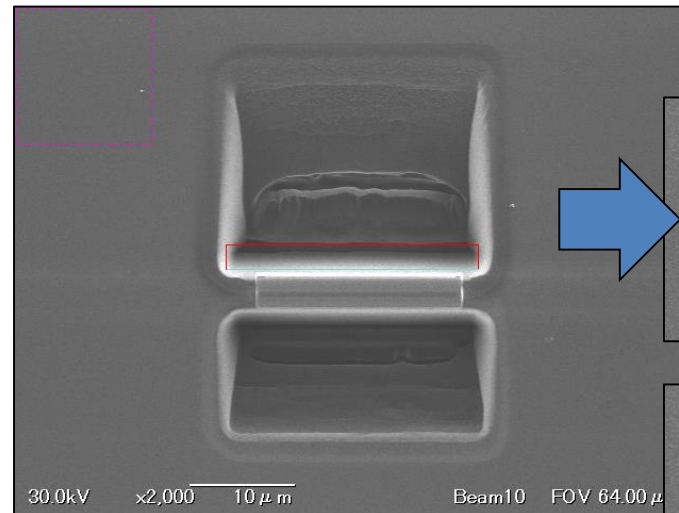
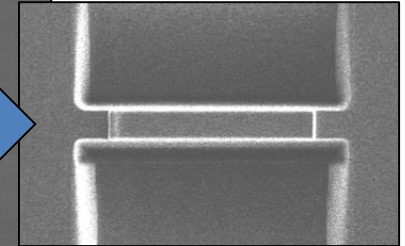
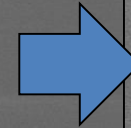
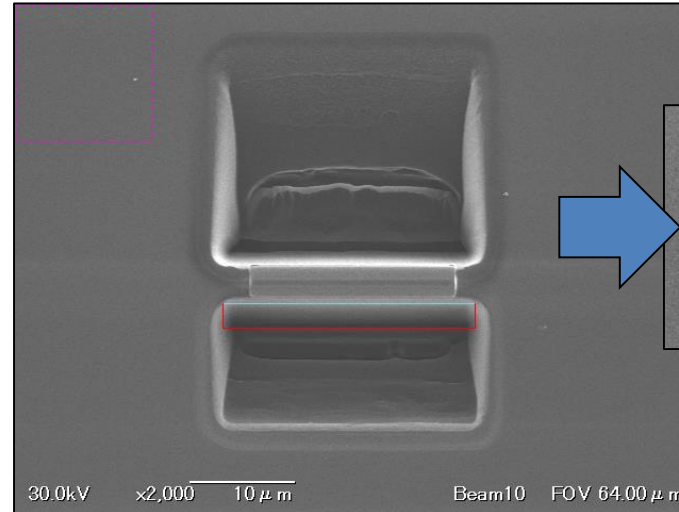
Bulk cut(2/2)

Making about 2.5um thickness piece

- Tilt= $53 \pm 1.5^\circ$
- Mode: MILL
- 30kV, 3nA (5) - 10nA (4)
- Lower side(54.5°)
 - $24\mu\text{m}(\text{x})$, $\sim 4.5\mu\text{m}(\text{y})$
 - Dose ~ 12
 - Finish: Top
- 上側Upper side(51.5°)
 - $24\mu\text{m}(\text{x})$, $\sim 3.8\mu\text{m}(\text{y})$
 - Dose ~ 8
 - Finish: Bottom
- Tilt= 53°

Measure the thickness at Tilt= 53° degree.

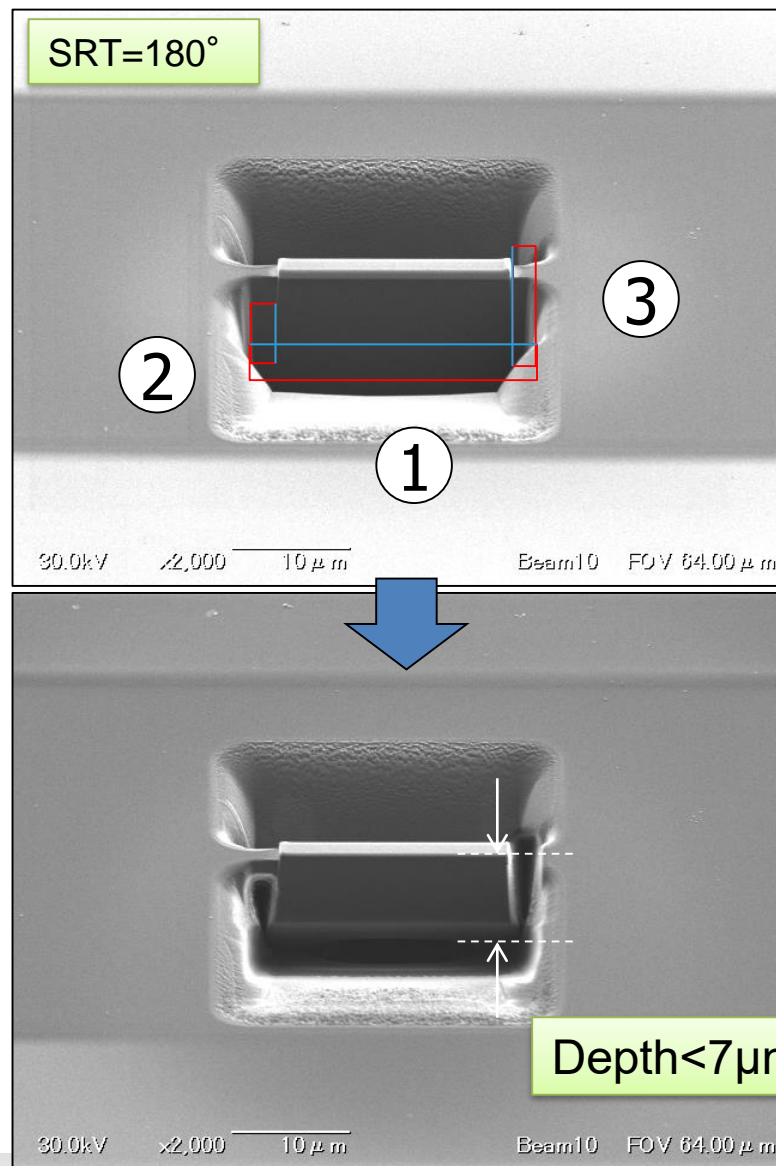
Beam 4-5



Bottom cut

- Tilt=0° (Scan Rotation=180°)
- Mode: MILL
- 30kV, 3nA (5) -10nA (4)
- Right side –①
 - 2 μ m(x) ,10 μ m(y)
 - Dose~15
 - Stop it, When the Beam penetrates
- Left side –②
 - 2 μ m(x) ,7 μ m(y)
 - Dose~15
 - Stop it, When the Beam penetrates
- Lower side –③
 - 22 μ m(x) ,3.5 μ m(y)
 - Dose~50
 - Stop it, When the Beam penetrates

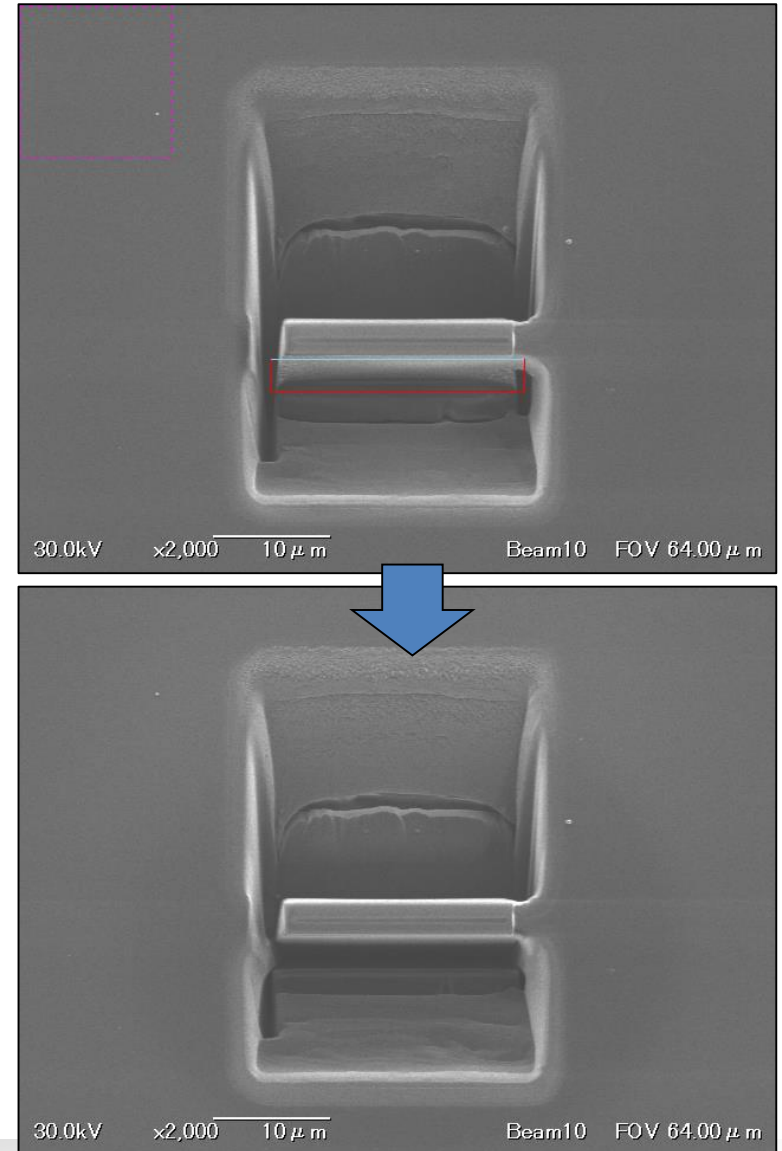
Beam 4-5



Removing redeposition

- Tilt=53+1.5°
- Mode : MILL Beam 4-5
- 30kV, 3nA (5) -10nA (4)
- Lower side(54.5°)
 - 23.5μm(x) , ~2.5μm(y)
 - Dose ~10

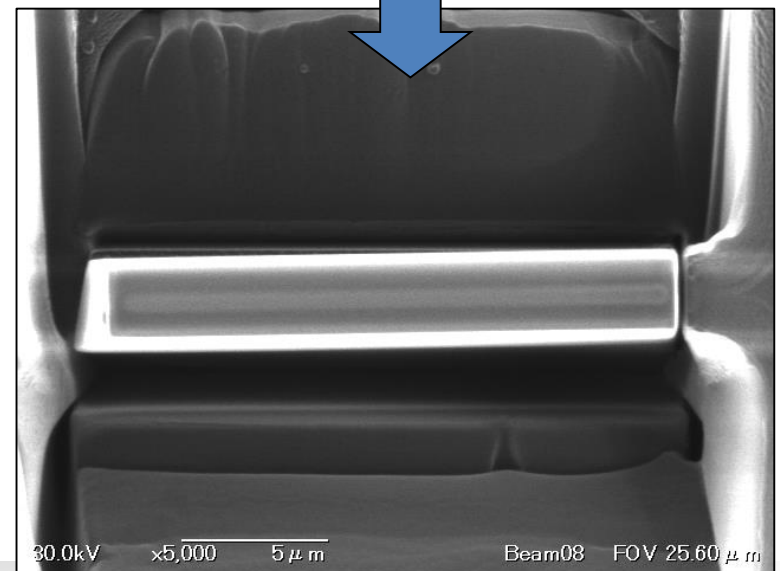
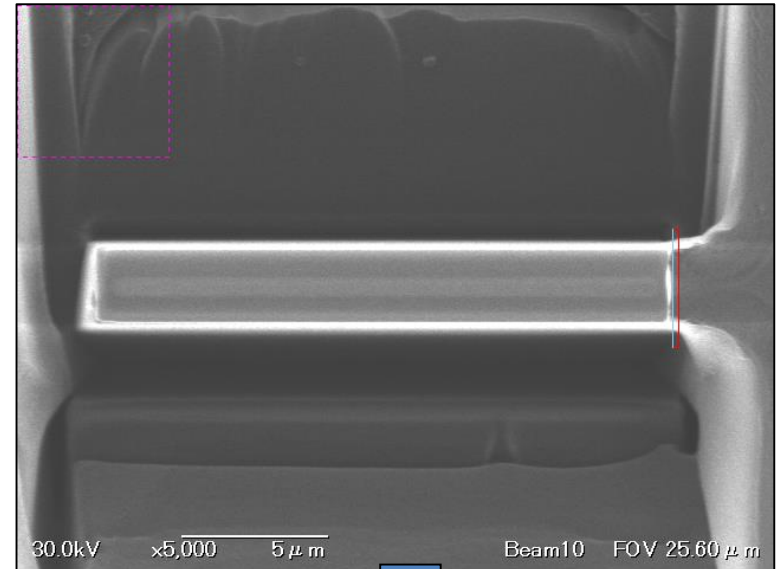
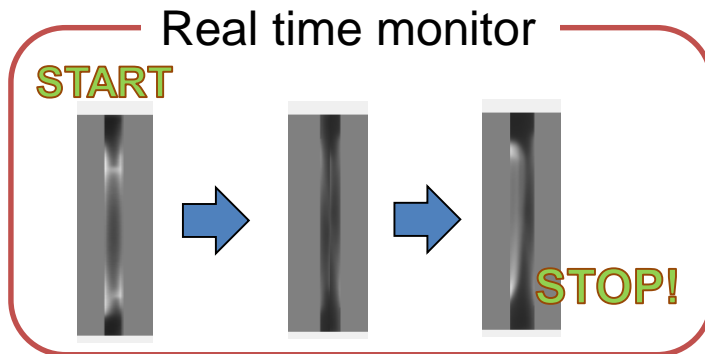
Sample will be attached on lower side, which is why redeposition is needed only for that side.



Sample block separation

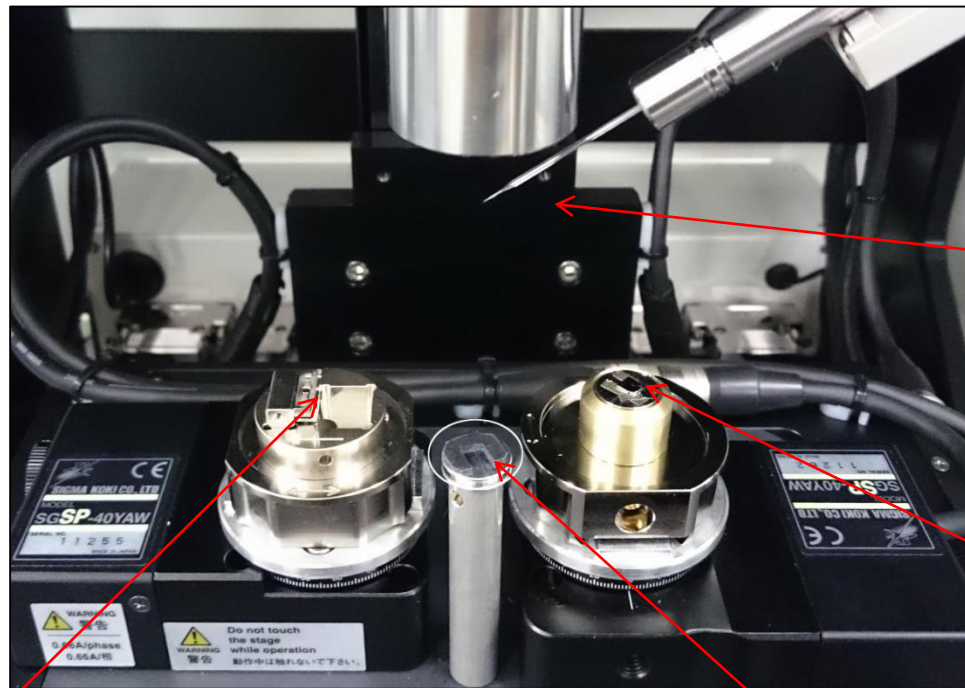
- Tilt=53°
- Mode: MILL
- 30kV, 100(8)-300pA (9)
- 0.2 μm (x) , 3-4 μm (y)
- Dose \sim 50

Stop it, when the piece is separated.



Pickup and Affixing to a FIB Grid (1/4)

Setting the specimen into the pickup system



Glass probe

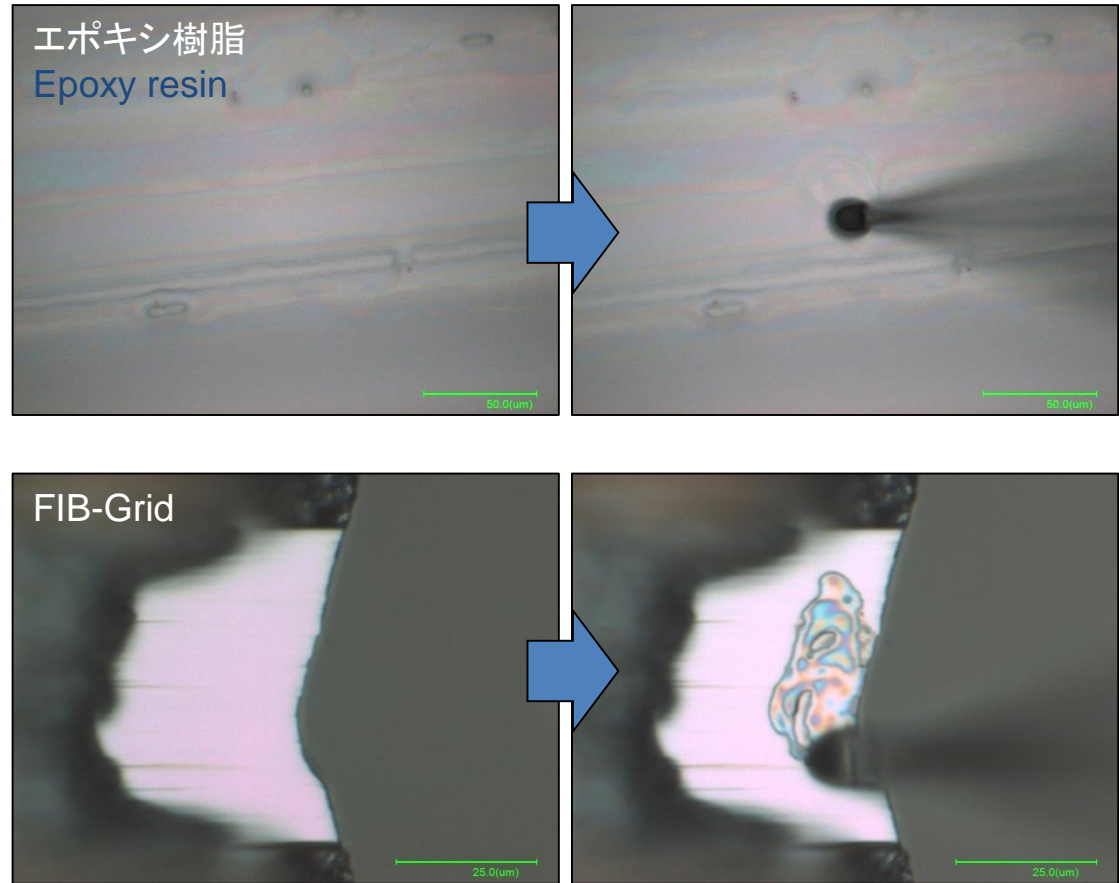
Sample

FIB-Grid

Epoxy resin

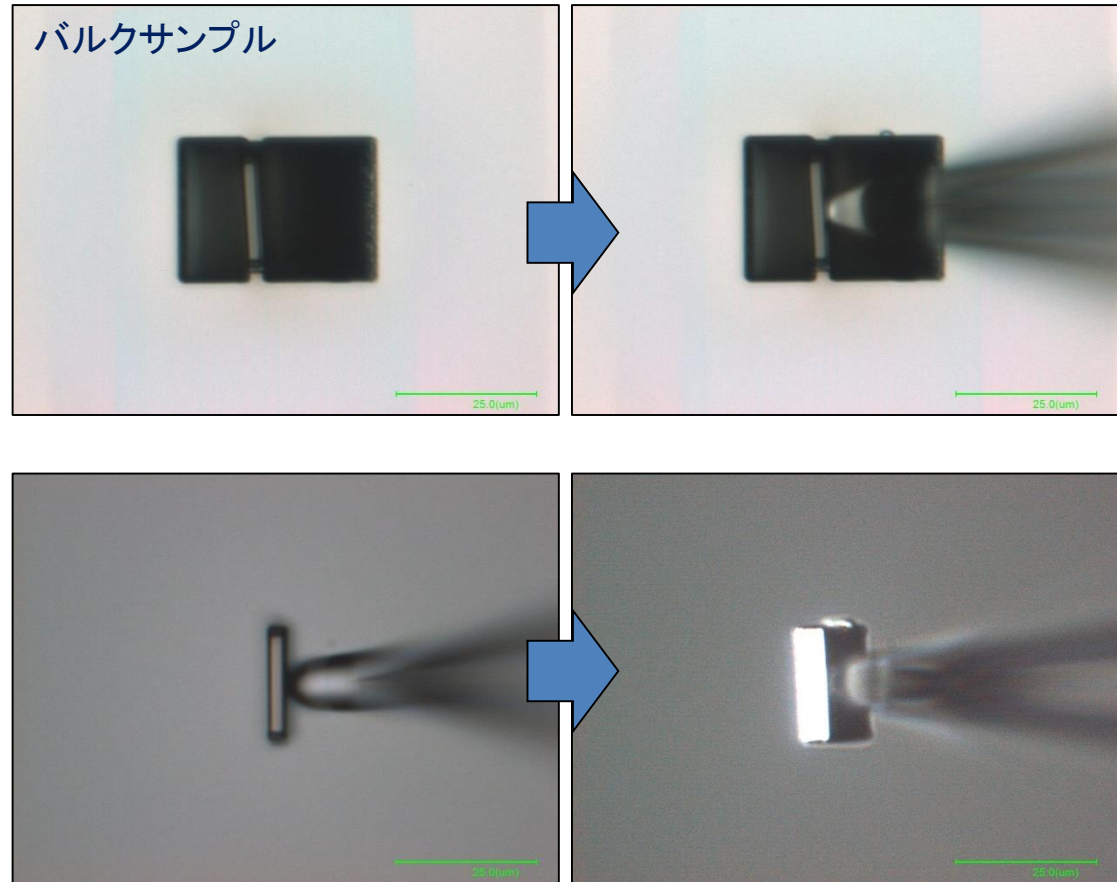
Pickup and Affixing to a FIB Grid(2/4)

- Applying epoxy resin to the FIB grid
 - Apply epoxy resin to the glass probe
 - Apply epoxy resin to the FIB grid



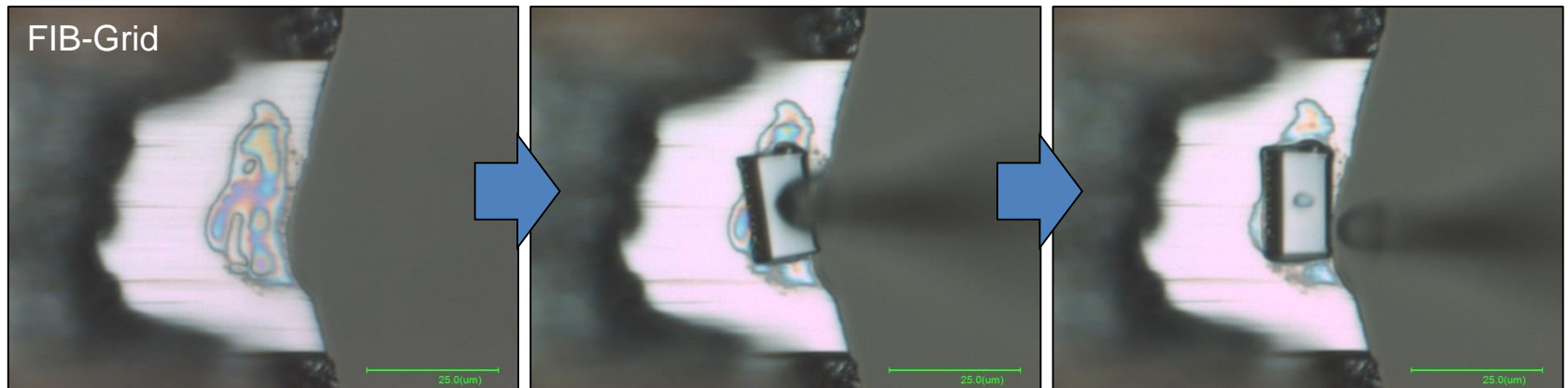
Pickup and Affixing to a FIB Grid(3/4)

- Picking up sample block
 - Pick up the bulk sample
 - Rotate (180) the glass probe so that the cross section faces down.



Pickup and Affixing to a FIB Grid(4/4)

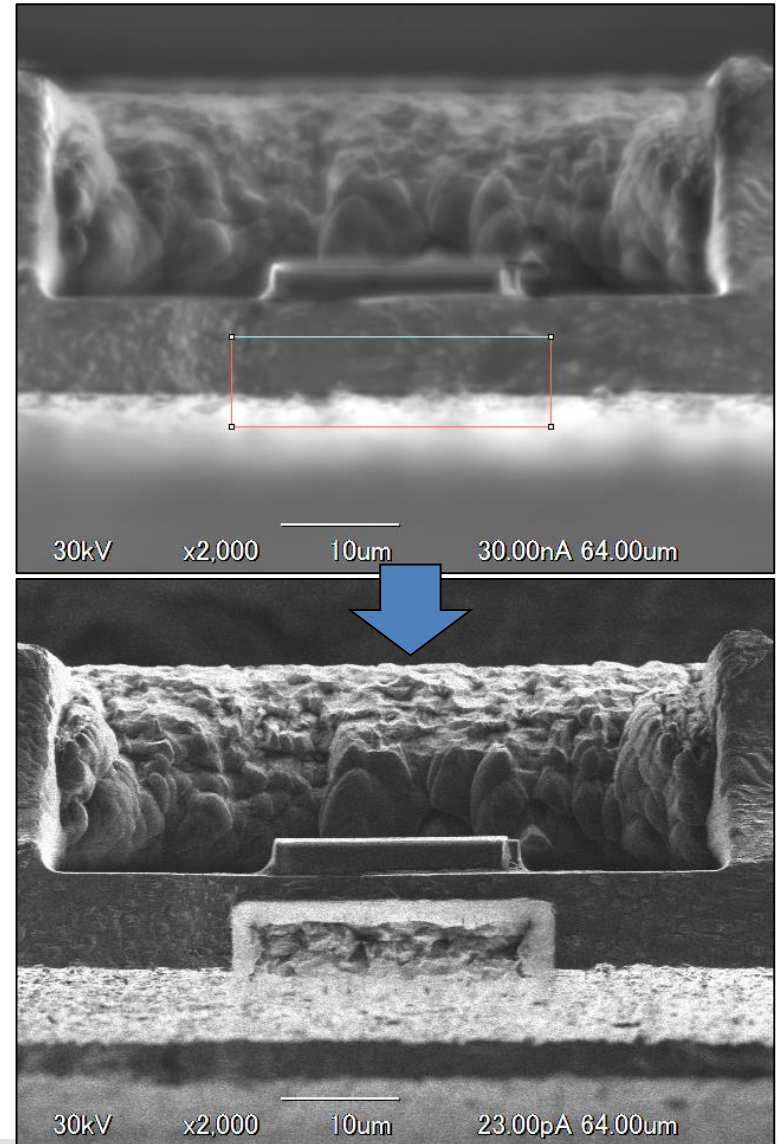
- Affixing the sample block
 - Put the sample on the TEM GRID
 - Adjust the position of the sample (nudge with glass needle).



FIB grid milling (1/2)

- Tilt=53+1.5°
- Mode : RAPID
- 30kV, 10nA (4)-50nA (5)
- $\sim 27\mu\text{m}(\text{x})$, $\sim 5\mu\text{m}(\text{y})$
- Dose ~ 30

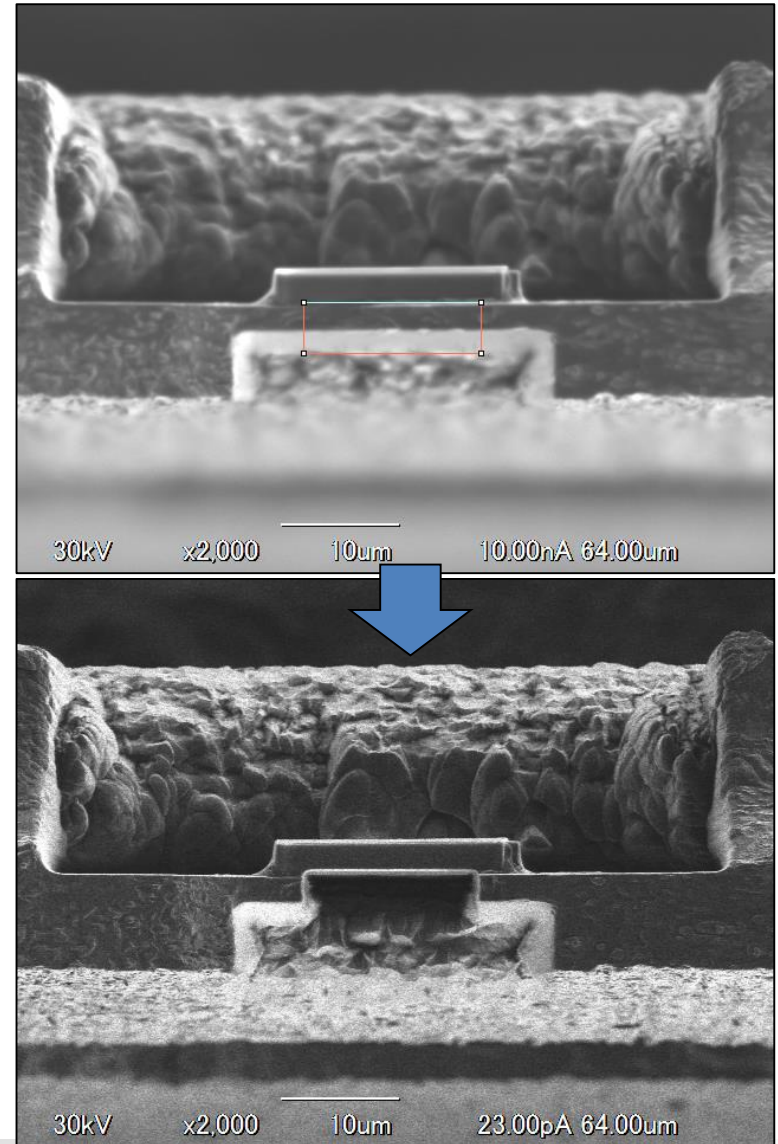
Beam 2-4



FIB grid milling (2/2)

- Tilt=53+1.5°
- Mode : MILL
- 30kV, 1nA (6)-10nA (4)
- $\sim 15\mu\text{m}(\text{x})$, $\sim 3\mu\text{m}(\text{y})$
- Dose ~ 30

Beam 4-6



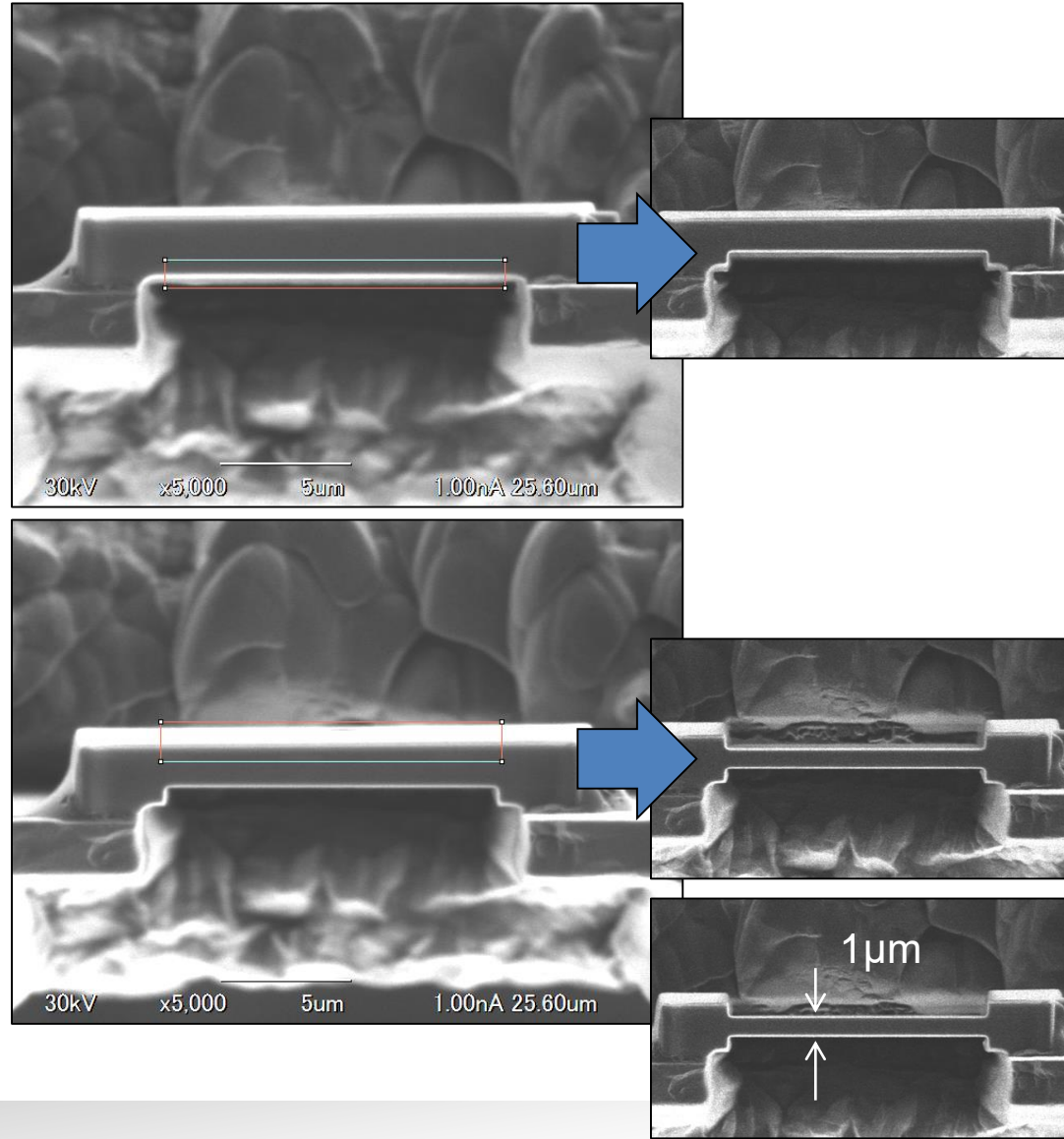
Thinning sample(1/4)

Reducing thickness to about 1.0um

- Tilt= $53 \pm 1.0^\circ$
- Mode: MILL
- 30kV, 1nA (6)
- Lower side (54.0°)
 - $13\mu\text{m}(x)$, $\sim 2\mu\text{m}(y)$
 - Dose ~ 8
- Upper side (52.0°)
 - $13\mu\text{m}(x)$, $\sim 2\mu\text{m}(y)$
 - Dose ~ 8
- Tilt= 53°

Measure the thickness at Tilt= 53° degree.

Beam 6



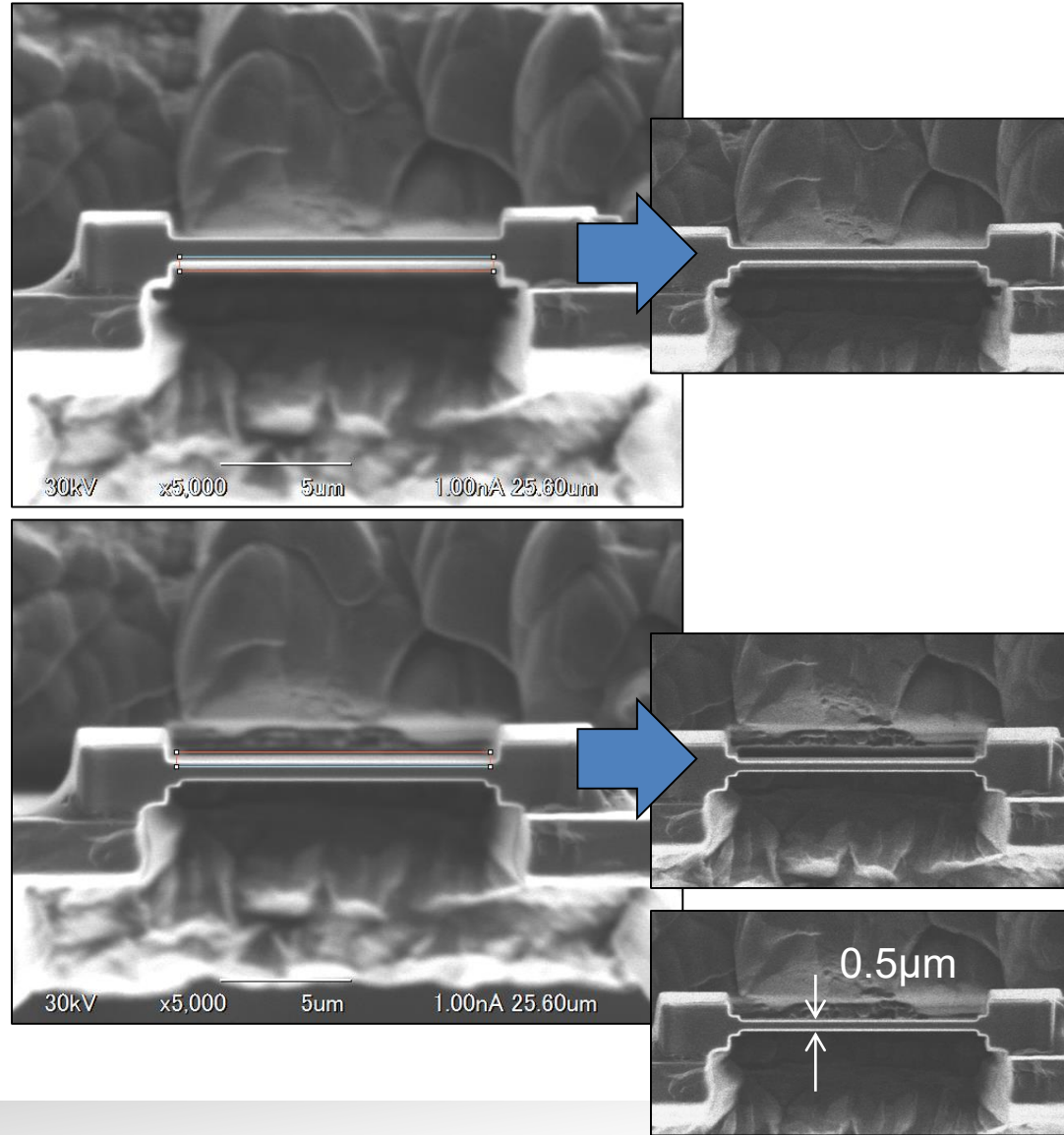
Thinning sample (2/4)

Reducing Thickness to about 0.5 μ m.

- Tilt= $53 \pm 1.0^\circ$
- Mode: MILL
- 30kV, 500pA (7)-1nA (6)
- Lower side (54.0°)
 - 12 μ m(x), $\sim 1\mu$ m(y)
 - Dose ~ 5
- Upper side (52.0°)
 - 12 μ m(x), $\sim 1\mu$ m(y)
 - Dose ~ 5
- Tilt= 53°

Beam 6-7

Measure the thickness at Tilt= 53° degree.



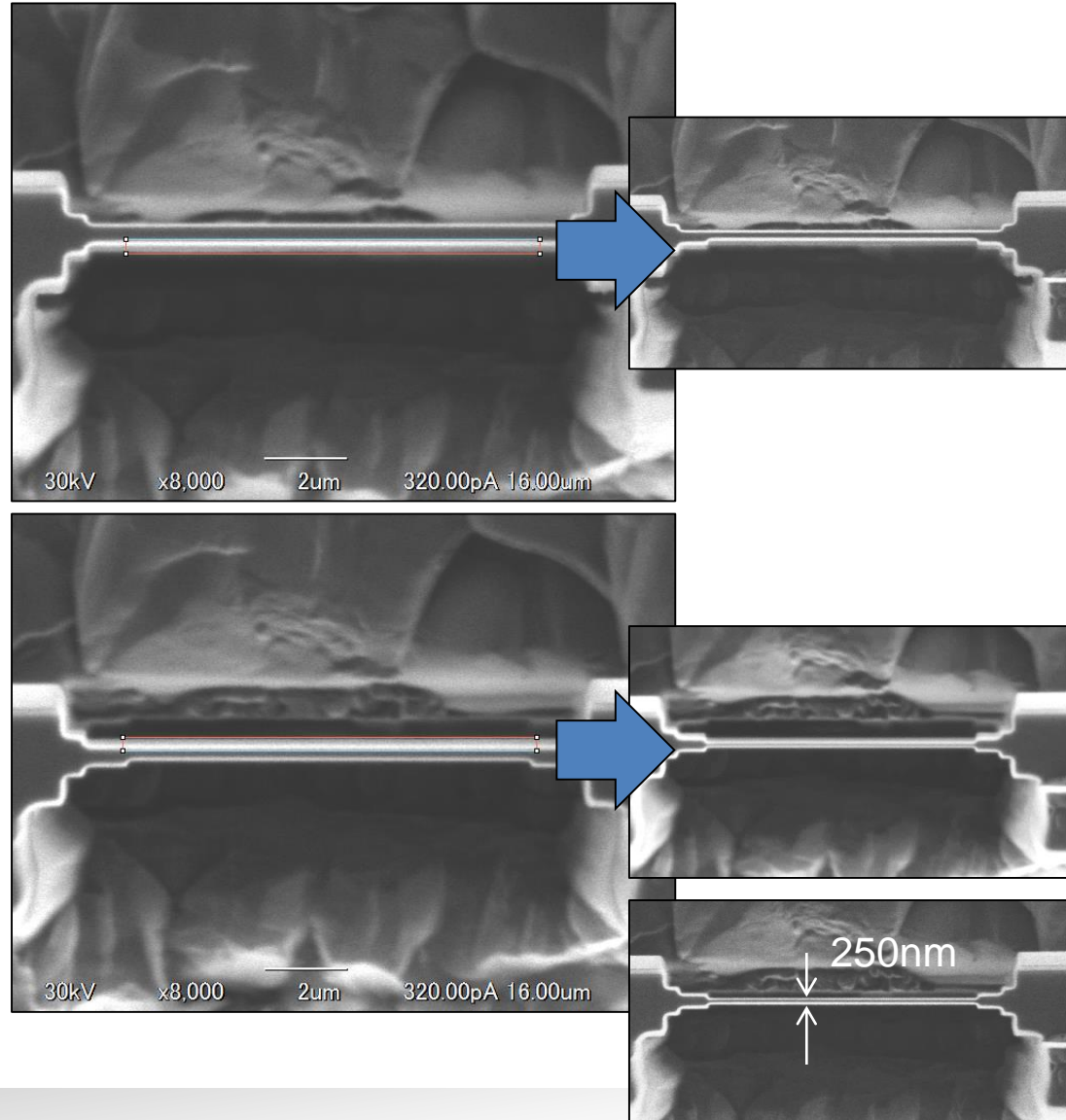
Thinning sample(3/4)

Reducing thickness to about 250nm.

- Tilt= $53 \pm 1.0^\circ$
- Mode: MILL
- 30kV, 100pA (9) -300pA (8)
- Lower side (54.0°)
 - $10\mu\text{m}(\text{x})$, $\sim 0.5\mu\text{m}(\text{y})$
 - Dose ~ 5
- Upper side (52.0°)
 - $10\mu\text{m}(\text{x})$, $\sim 0.5\mu\text{m}(\text{y})$
 - Dose ~ 5
- Tilt= 53°

Measure the thickness at Tilt= 53° degree.

Beam 8-9



Thinning sample(4/4)

Reducing thickness to about 120nm

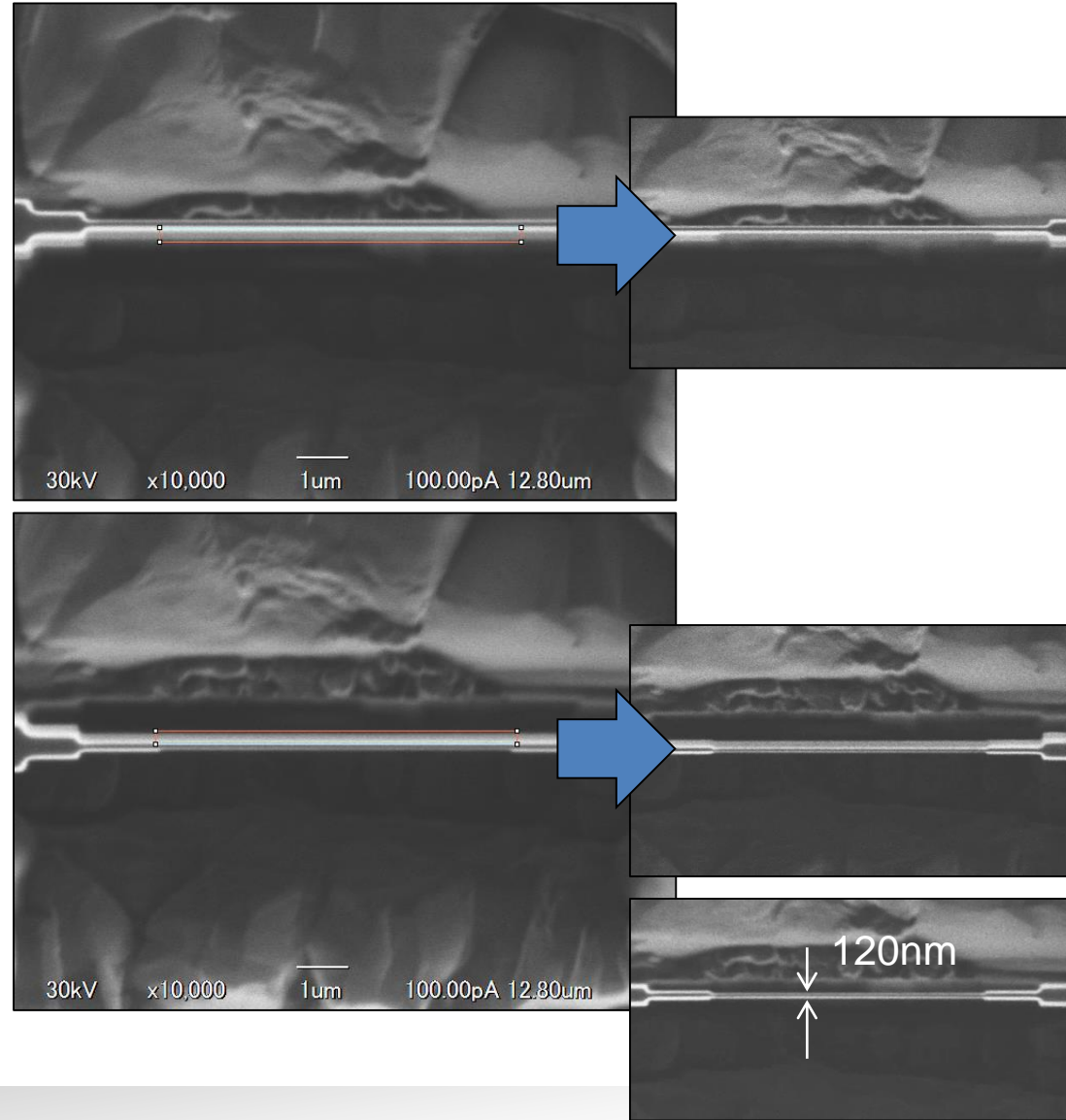
- Tilt= $53 \pm 0.8^\circ$
- Mode: MILL
- 30kV, 30pA (10)-100pA (9)
- Lower side (53.8°)
 - $\sim 7\mu\text{m}(x)$, $\sim 0.2\mu\text{m}(y)$
 - Dose $\sim 1 \times 3^*$
- Upper side (52.2°)
 - $\sim 7\mu\text{m}(x)$, $\sim 0.2\mu\text{m}(y)$
 - Dose $\sim 1 \times 3^*$
- Tilt= 53°

Beam 9-10

Measure the thickness
at Tilt= 53° degree.

※For Dose: change to get desired time (30 seconds)

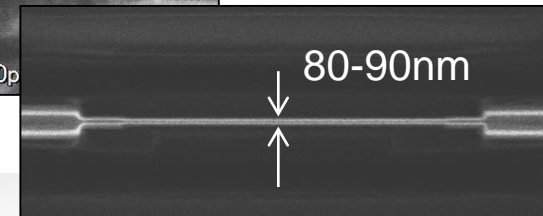
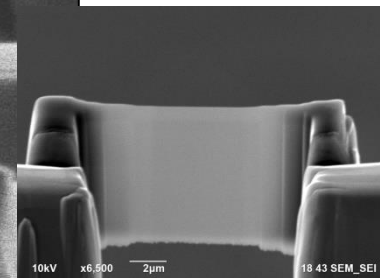
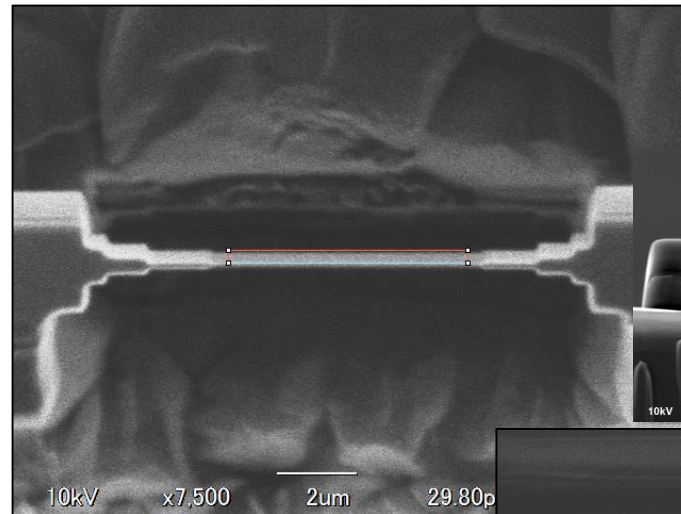
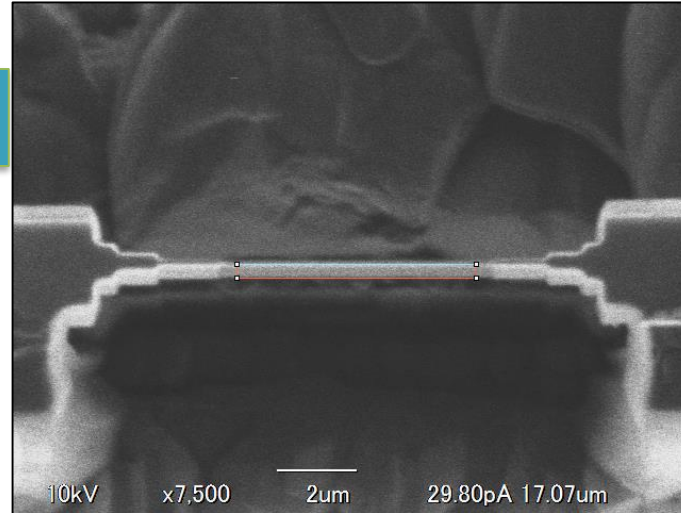
Repeat about 30 seconds milling. Longer time milling may cause sample distortion.



❌ *Processing with a low-acceleration Ga ion beam (1)*

Reducing thickness to 80~90nm

- Tilt= $53 \pm 1^\circ$ - $53 \pm 1.5^\circ$ Medium3(beam6), Fine2(beam8)
- Mode: MILL
- **10kV**, 30pA (8)-100pA (9)
- **Lower side** (54° - 54.5°)
 - $\sim 6\mu\text{m}(\text{x})$, $\sim 0.5\mu\text{m}(\text{y})$
 - 30sec
- **Upper side** (51.5° - 52.0°)
 - $\sim 6\mu\text{m}(\text{x})$, $\sim 0.5\mu\text{m}(\text{y})$
 - 30sec
- Repeat 30seconds milling on each side until the thickness becomes 80~90nm.
- Use SEM to monitor

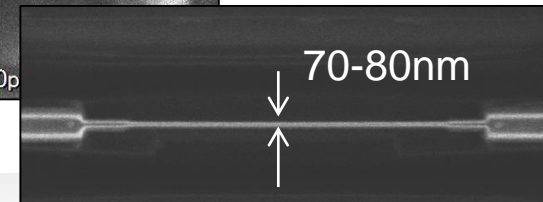
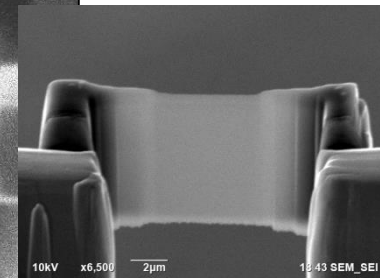
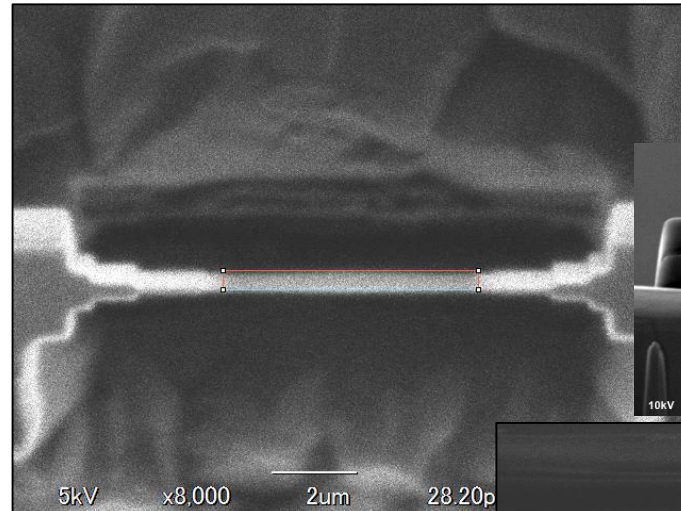
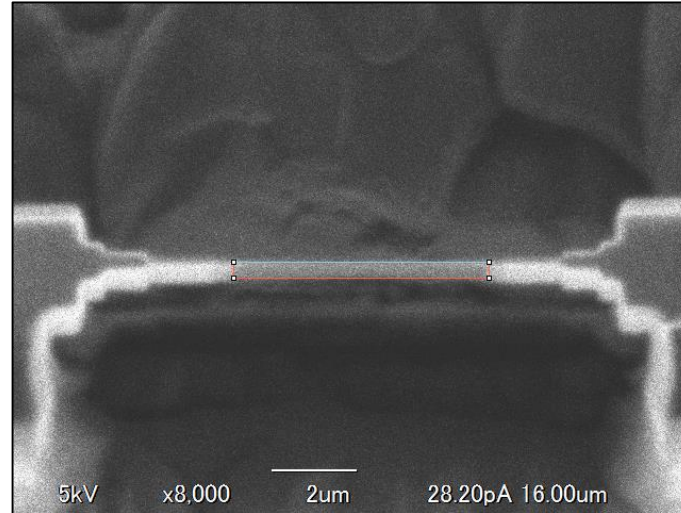


❌ *Processing with a low-acceleration Ga ion beam (2)*

Reducing thickness to 70~80nm

- Tilt= $53 \pm 1.5^\circ$ - $53 \pm 2.0^\circ$
- Mode: MILL
- 5kV, 10pA (9)-100pA (7)
- Lower side ($54.5 - 55.0^\circ$)
 - $\sim 6\mu\text{m}(\text{x})$, $\sim 0.6\mu\text{m}(\text{y})$
 - 30sec
- Upper side ($51.0 - 51.5^\circ$)
 - $\sim 6\mu\text{m}(\text{x})$, $\sim 0.6\mu\text{m}(\text{y})$
 - 30sec
- Repeat 30 seconds milling on each side until the thickness becomes 70~80nm.

Fine1 (beam7),
Fine3 (beam9)



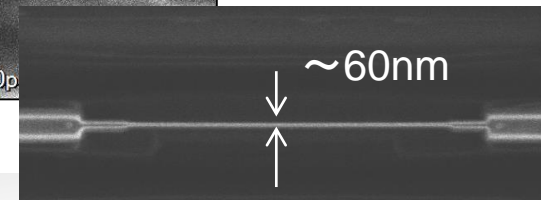
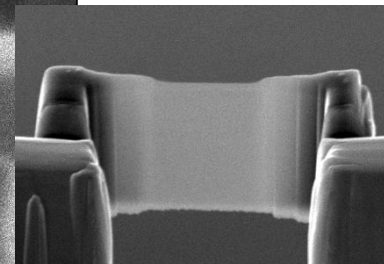
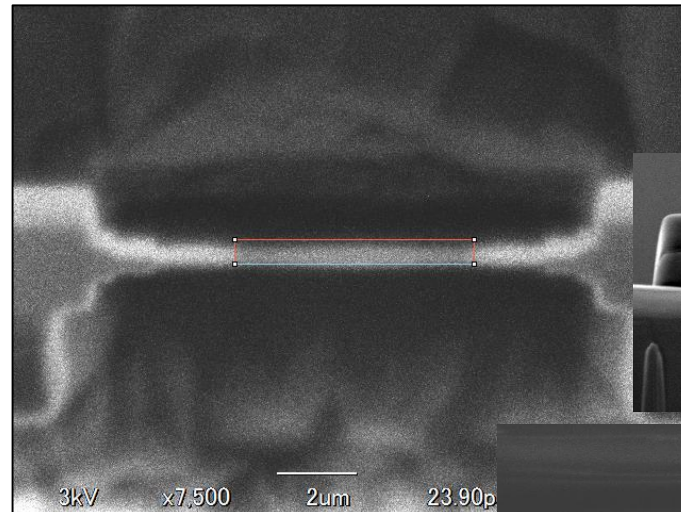
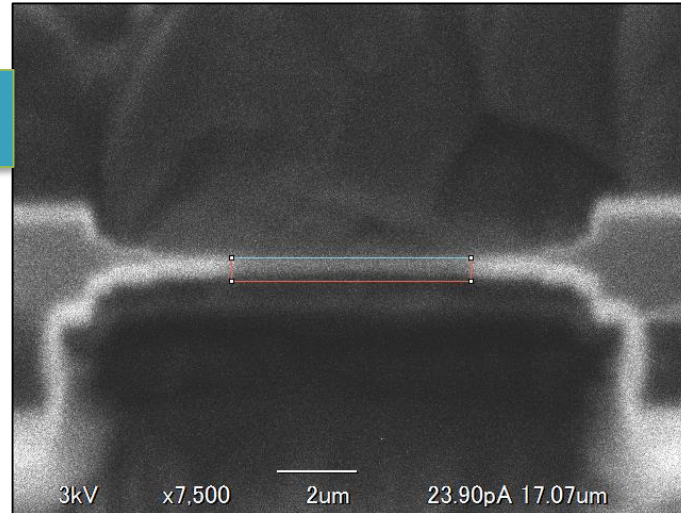
Low acceleration voltage processing should be done, if the customer wants less amorphous material.

※ *Processing with a low-acceleration Ga ion beam (3)*

Reducing the thickness to less than 60nm

- Tilt= $53 \pm 1.5^\circ$ - $53 \pm 3.0^\circ$
- Mode: MILL
- 3kV, 10pA (8)-100pA (5)
- Lower side ($54.5 - 56.0^\circ$)
 - $\sim 6\mu\text{m}(x)$, $\sim 0.5\mu\text{m}(y)$
 - 30sec
- Upper side ($50.0 - 51.5^\circ$)
 - $\sim 6\mu\text{m}(x)$, $\sim 0.5\mu\text{m}(y)$
 - 30sec
- Repeat 30seconds milling on each side until the thickness becomes $\sim 60\text{nm}$.

Medium2(beam5),
Fine2(beam8)



※

Low acceleration voltage processing should be done, if the customer wants to remove amorphous layers.

TEM Sample Thinning Procedure for JIB-4700F by using the Bulk Pickup Method