SUPPLEMENTARY MATERIAL

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# Variations of Milling Parameters and its Effect on the Volumetric Sputtering Yield of Silicon with the Use of a Plasma Focused Ion Beam

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## SUPPLEMENTARY RISK ASSESSMENT S1

**Supplementary Table S1.** Chemical risk assessment.

| Chemical | Risk | Hazard | Mitigation |
|---|---|---|---|
| 2-propanol (isopropanol / IPA) | Medium | Highly flammable liquid and vapour. Causes serious eye irritation. May cause drowsiness or dizziness. | Keep away from heat, sparks, open flames and hot surfaces. Use eye, face and skin protection. Handle with care. |
| 2-propanone (acetone) | Medium | May cause nausea, vomiting and inflammation of the mouth. Skin may be dry, red and inflamed. Eye contact with acetone vapours can lead to irritation or severe eye damage. May cause fires. | Avoid inhaling vapour. Avoid contact with eyes, skin and clothing. Keep away from flames and gas nozzles. Avoid spillage and do not drink. Wear hand and eye protection. |
| Nitrogen gas ($N_2$) | Low | May displace oxygen and may cause asphyxiation. | Work in a ventilated area with face protection. |

## SUPPLEMENTARY METHODS S1

### ImageJ analysis

### Calibration

1. Select the line tool.
2. Hold Shift while drawing a line across the horizontal width of the image.
3. Select Analyse → Set Scale → 20.7 $\mu$m → Global → OK.

### Set-up

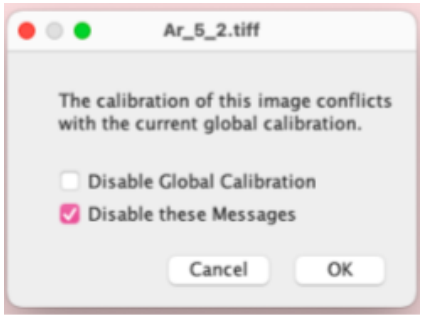
4. Select Analyse → Select Measurements. Deselect Area and Min & Max Grey Values, select Bounding Rectangle, and click OK.

### Analysis

5. Select the rectangle tool.
6. Draw a rectangle over the milled line.
7. Press Control + M to save the data.
8. Repeat for each TIFF image.

### Global calibration

If a calibration message appears when opening a new image after the first analysis, deselect "Disable Global Calibration" and select "Disable these Messages" (Supplementary Figure S1). This retains the global calibration after previous images are closed, so the calibration steps do not need to be repeated.




**Supplementary Figure S1.** ImageJ notification regarding global calibration.

## SUPPLEMENTARY CALCULATIONS S1

### Corrected milled depth

For the 5 kV, 3.1 nA condition, the measured depth is $d = 0.391\,\mu$m. The Helios stage is tilted at 52°.

$$d_m = \frac{d}{\sin(52^\circ)}, \tag{S1}$$

where $d_m$ is the corrected milled depth and $d$ is the measured depth.

$$\begin{aligned} d_m &= \frac{0.391\,\mu\text{m}}{\sin(52^\circ)} \\ &\approx 0.4962\,\mu\text{m}. \end{aligned}$$

## SUPPLEMENTARY CODE S1

### Code used to automate argon PFIB milling

```
import os
import time
import numpy as np
from datetime import datetime
from autoscript_sdb_microscope_client import SdbMicroscopeClient
from autoscript_sdb_microscope_client.enumerations import *
from autoscript_sdb_microscope_client.structures import *
from autoscript_sdb_microscope_client import SdbMicroscopeClient
from autoscript_sdb_microscope_client.enumerations import *
from autoscript_sdb_microscope_client.structures import *
import autoscript_toolkit.drift_correction as dc_toolkit
from datetime import datetime

# Get the current date and time
current_time = datetime.now()
print("Start time:", current_time)

microscope = SdbMicroscopeClient()
microscope.connect("192.168.0.1")

microscope.imaging.set_active_view(2)
microscope.imaging.set_active_device(ImagingDevice.ION_BEAM)
microscope.beams.ion_beam.horizontal_field_width.value = 40e-6

microscope.patterning.set_default_beam_type(BeamType.ION)
microscope.patterning.clear_patterns()

rect = microscope.patterning.create_rectangle(0, 0, 10e-6, 10e-6, 1e-6)
rect.dwell_time = 1e-6
rect.overlap_x = 0.5
rect.overlap_y = 0.5

microscope.beams.ion_beam.source.plasma_gas.value = PlasmaGasType.ARGON

voltages = [5000, 8000, 10000, 12000, 30000]

currents_5kV = [0.2e-9, 3.1e-9, 4.8e-9, 9.3e-9, 20e-9]
currents_8kV = [0.2e-9, 3.9e-9, 6.1e-9, 9.5e-9, 20e-9]
currents_10kV = [0.21e-9, 1.4e-9, 5.9e-9, 12e-9, 35e-9]
currents_12kV = [0.21e-9, 1.2e-9, 5.8e-9, 14e-9, 50e-9]
currents_30kV = [0.2e-9, 0.74e-9, 2e-9, 7.6e-9, 28e-9]

for i, volt1 in enumerate(voltages):
    if i == 0:
        currents = currents_5kV
    elif i == 1:
        currents = currents_8kV
    elif i == 2:
        currents = currents_10kV
    elif i == 3:
        currents = currents_12kV
    elif i == 4:
        currents = currents_30kV

    microscope.beams.ion_beam.high_voltage.value = volt1
    print(f"For {volt1} kV:")

    for current1 in currents:
        microscope.beams.ion_beam.beam_current.value = current1
        rect.time = 30
        print(f"For {current1} A:")
        microscope.patterning.run()
```

```
        time.sleep(5)
        microscope.specimen.stage.relative_move(StagePosition(x=+30e-6))

    microscope.specimen.stage.relative_move(StagePosition(y=-30e-6))
    microscope.specimen.stage.relative_move(StagePosition(x=-150e-6))

# Get the current date and time
current_time = datetime.now()
print("End time:", current_time)

print('milling finished')
```

**Supplementary Table S2.** Full dataset: milling conditions, measured and corrected depths, removed volumes, charges and volumetric sputtering yields.

| Voltage (kV) | Current (nA) | Measured depth ($\mu$m) | Corrected milled depth ($\mu$m) | Volume removed ($\mu$m$^3$) | Charge (nC) | Sputtering yield ($\mu$m$^3$/nC) |
|---|---|---|---|---|---|---|
| 5 | 0.2 | 0 | 0 | 0.00 | 6 | 0.0000 |
| 5 | 3.1 | 0.391 | 0.4962 | 49.62 | 93 | 0.5335 |
| 5 | 4.8 | 0.579 | 0.7348 | 73.48 | 144 | 0.5103 |
| 5 | 9.3 | 0.62 | 0.7868 | 78.68 | 279 | 0.2820 |
| 5 | 20 | 0.606 | 0.7690 | 76.90 | 600 | 0.1282 |
| 8 | 0.2 | 0 | 0 | 0.00 | 6 | 0.0000 |
| 8 | 3.9 | 0.35 | 0.4442 | 44.42 | 117 | 0.3796 |
| 8 | 6.1 | 0.458 | 0.5812 | 58.12 | 183 | 0.3176 |
| 8 | 9.5 | 0.377 | 0.4784 | 47.84 | 285 | 0.1679 |
| 8 | 20 | 0.647 | 0.8211 | 82.11 | 600 | 0.1368 |
| 10 | 0.21 | 0 | 0 | 0.00 | 6.3 | 0.0000 |
| 10 | 1.4 | 0.067 | 0.0850 | 8.50 | 42 | 0.2024 |
| 10 | 5.9 | 0.35 | 0.4442 | 44.42 | 177 | 0.2509 |
| 10 | 12 | 0.647 | 0.8211 | 82.11 | 360 | 0.2281 |
| 10 | 35 | 0.903 | 1.1459 | 114.59 | 1050 | 0.1091 |
| 12 | 0.21 | 0 | 0 | 0.00 | 6.3 | 0.0000 |
| 12 | 1.2 | 0.108 | 0.1371 | 13.71 | 36 | 0.3807 |
| 12 | 4.8 | 0.418 | 0.5304 | 53.04 | 144 | 0.3684 |
| 12 | 14 | 0.882 | 1.1193 | 111.93 | 420 | 0.2665 |
| 12 | 50 | 1.779 | 2.2576 | 225.76 | 1500 | 0.1505 |
| 30 | 0.2 | 0 | 0 | 0.00 | 6 | 0.0000 |
| 30 | 0.7 | 0 | 0 | 0.00 | 21 | 0.0000 |
| 30 | 2 | 0.121 | 0.1536 | 15.36 | 60 | 0.2559 |
| 30 | 7.6 | 0.364 | 0.4619 | 46.19 | 228 | 0.2026 |
| 30 | 28 | 1.577 | 2.0012 | 200.12 | 840 | 0.2382 |

## SUPPLEMENTARY CALCULATIONS S2

### Volumetric sputtering yield

The charge is calculated as

$$q = It, \tag{S2}$$

where $q$ is charge, $I$ is current and $t$ is milling time. Here, $t = 30\,\mathrm{s}$ and $I$ depends on the milling condition.

The volume removed is calculated as

$$V = L \times w \times d_m, \tag{S3}$$

where $V$ is the volume removed, $L$ and $w$ are the length and width of the milled area, and $d_m$ is the corrected milled depth. The milled area is $L \times w = 100\,\mu\mathrm{m}^2$ and the depth depends on the milling condition.

The volumetric sputtering yield is

$$Y_s = \frac{V_{\mathrm{removed}}}{q}, \tag{S4}$$

where $Y_s$ is volumetric sputtering yield and $V_{\mathrm{removed}}$ is the volume removed, denoted by $V$ in Equation (S3).

**Worked example:** $5\,\mathrm{kV}$, $3.1\,\mathrm{nA}$

$$\begin{aligned} q &= It \\ &= (3.1 \times 10^{-9}\,\mathrm{A}) \times 30\,\mathrm{s} \\ &= 9.3 \times 10^{-8}\,\mathrm{C} = 93\,\mathrm{nC}. \end{aligned}$$

$$\begin{aligned} V &= L \times w \times d_m \\ &= 100\,\mu\mathrm{m}^2 \times 0.391\,\mu\mathrm{m} \\ &= 39.1\,\mu\mathrm{m}^3. \end{aligned}$$

$$\begin{aligned} Y_s &= \frac{V_{\mathrm{removed}}}{q} \\ &= \frac{39.1\,\mu\mathrm{m}^3}{93\,\mathrm{nC}} \\ &\approx 0.42\,\mu\mathrm{m}^3/\mathrm{nC}. \end{aligned}$$