Focused electron beam induced etching and *in-situ* monitoring: Fabrication of sub-beam sized nanoholes

Hiroyuki Miyazoe^{1,2}, Ivo Utke¹, Johann Michler¹, Kazuo Terashima²

¹EMPA - Materials Science & Technology, Laboratory for Mechanics of Materials and Nanostructures, Feuerwerkerstrasse 39, CH-3602, Thun, Switzerland

²The University of Tokyo, Graduate school of Frontier Sciences, 5-1-1- Kashiwanoha, Kashiwa-shi, Chiba, 277-8561, Japan

1. Introduction

Resolution of focused electron beam-induced etching process is not yet assessed scientifically compared to the deposition¹⁾. In this work, we investigated the etching into 10-nm-thin amorphous carbon membranes as a model substance. In-situ time resolved stage current monitoring, Monte-Carlo simulation, and beam size measurements were performed to rationalize the experimental results.

Figure 1: Principle of focused electron beam induced processing.

2. Experiment

Figure 2: Scheme of the experimental setup. (Not at scale)

1. Etching experiment:
H₂O and XeF₂ gas flow
2. Theoretical approach:
Monte-Carlo (MC) simulation (MOCASIM)²⁾
3. Verification of experimental results:
Knife edge measurement³⁾ under H₂O gas flow

Table 1: Experimental details.

Gas flow rate (molecule/s·cm ²)	2.0 × 10 ²⁰ for H ₂ O 1.3 × 10 ¹⁹ for XeF ₂
PE energy (keV)	5 and 25
Probe current (pA)	88-9822
Exposure time (s)	1-300

3. Morphology

SEM observation

Figure 3: SEM image of an array of 6 × 6 nanoholes etched with H₂O using 25 keV and 985 pA electron beam. The numbers indicate the exposure time in seconds. Inset: 18 nm diameter hole.

Figure 4: Radius of holes as a function of exposure (etching) time with a 5 keV and 1 nA electron beam using H₂O or XeF₂. Inset: SEM images of nanoholes with corresponding exposure time in second.

The smallest diameter: 18 nm
Etch rate: 2.3 times faster in H₂O

4. Stage current

Figure 5: Time resolved stage current measurement for six successive etch exposures with H₂O precursor (primary electron energy = 5 keV, probe current = 924 pA).

5. Analytic calculation

Current balance at sample surface
Time resolved stage current $I(t)$:

$$I(t) = I_p - I_{SE} \quad \dots (1)$$

I_p : Current of primary electrons, I_{SE} : Current of secondary electrons

Figure 6: Scheme of the current balance during etching experiment.

Assuming Gaussian distributed primary electron beam, $I_{SE}(r)$:

$$I_{SE}(r) = 2\pi\sigma^2\delta j_{peak} \exp\left(-\frac{r^2}{2\sigma^2}\right) \quad \dots (2)$$

σ : Standard deviation, δ : Secondary electron yield
 j_{peak} : Peak current density, r : Radius of nanohole

Stage current change during nanohole etching process, $I(r)$:

$$I(r) = I_{SE}(r) - I_{SE}(r=0) \quad \dots (3)$$

Analytical fitting using eq. (2) and (3):

Figure 7: Stage current change $I(r)$ as a function of radius of nanoholes (primary electron energy = 5 keV, and probe current = 924 pA). Analytic and MC models allow to determine the FWHM of the incident beam.

Theoretical approach ↔ Experiment
Good agreement

Determination of beam size and yield of secondary electrons (I_p : 5 keV, 924 pA):

Beam size (FWHM) 125 nm
SE-yield (δ) 0.18

$$(FWHM = 2(2\ln 2)^{1/2} \sigma)$$

6. Verification of Results

Beam current vs Beam size⁴⁾

(i) 10 keV < Primary electron energy < 30 keV:

$$I_p \propto FWHM^{8/3} \quad \dots (4)$$

(ii) Primary electron energy < 10 keV:

Broadening of beam size due to the spherical and chromatic aberrations

Figure 8: Beam size (FWHM) as function of probe current obtained from knife edge measurements (KE) and by analytical fitting of the current vs. nanohole diameter dependence for H₂O, and for XeF₂, at primary electron energies of (a) 25 keV, and (b) 5 keV. Minimum hole diameters (D_{min}) and saturated hole diameters (D_{sat}) are indicated.

$FWHM_{KE} \sim FWHM_{H_2O} \sim FWHM_{XeF_2}$
Etching evolution are reliable for beam size measurement.
Better control for the nanohole size.
 $D_{min,H_2O} \sim 0.2 - 0.4 \times FWHM$ in H₂O
Smaller nanoholes than beam size.
 $D_{min,XeF_2} \sim 0.4 - 0.8 \times FWHM$ in XeF₂
Precursor deletion in the center for XeF₂⁹⁾.
 $D_{sat} \sim 2 \times FWHM$
Corresponds to 6.25% of j_{peak} .

Minimum diameter < Beam size

7. Conclusion

Focused electron beam induced etching of a 10-nm-thick a-C membrane under H₂O and XeF₂ gas flux was examined in this work. The beam size of the primary electron beam was determined by *in-situ* stage current monitoring. Nanoholes with diameters having only 20-39% of the beam size were fabricated using an *in-situ* time resolved stage current control. Considering modern technology having nanometer electron beam size, it can be anticipated that nanometer sized membrane etching is attainable.

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Contact:

E-mail: hiroyuki.miyazoe@empa.ch

Tel: +41 33 228 40 39

Fax: +41 33 228 44 90

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