Electronic supplementary information (ESI)

Flexible and In-Situ Fabrication of Nanochannels with High Aspect Ratios and Nanopillar Arrays in Fused Silica Substrates Utilizing Focused Ion Beam

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Experimental

Preparation of Gold-Deposited Fused Silica Substrate. Before depositing gold, fused silica substrates (Sendai Quartz, Sendai, Japan) were cleaned with the following procedure. The substrates were first treated with one-minute ultrasonic cleaning in acetone (99.7%, Wako Pure Chemical Industries, Ltd., Tokyo, Japan) for three times by using an ultrasonic cleaner (US-1, SND Co., Nagano, Japan). Then, the substrates were ultrasonically cleaned for 5 min in a commercial cleaning solution Semico-Clean 26 (Furuuchi Chemical Corp., Tokyo, Japan) which is widely used in semiconductor cleaning process. After that, the substrates were further treated with one-minute ultrasonic cleaning in ultra-pure water for three times, followed by one-minute ultrasonic cleaning in isopropyl alcohol (99.9%, Wako). Finally, the substrates were dried by nitrogen blow for deposition of gold. A 100-nm thick gold layer was deposited on the fused silica substrate using a vacuum deposition apparatus (EX-200, ULVAC, Kanagawa, Japan). The high purity (99.99%) gold grains (Tanaka Kikinzoku Kogyo, Tokyo, Japan) were used in the deposition process.

FIB milling. A FIB system (FB-2100, Hitachi, Tokyo, Japan) having a gallium ion (Ga^+) gun coupled with low spherical aberration ion optics was used. All FIB millings were performed using the milling mode of the FIB system with an accelerating voltage of 40 keV, which is routinely used in milling processes. The beam diameter was adjusted by changing the aperture size. The actual beam diameter was calculated from a beam profile which was determined by detecting secondary electron intensity during beam scanning on the sample surface. For milling of high-aspect-ratio nanochannels, the ion beam scan regions were defined by vector data of rectangular patterns whose widths were set to be the width of nanochannels. For milling of nanochannels with nanopillar arrays, the ion beam scan regions were defined by bitmap data of rectangular patterns and square patterns for nanochannels and pillars, respectively.

Characterization of FIB-milled nanochannels. The widths of nanochannels were determined by a scanning electron microscope (SEM; ELS-7500, Elionix, Tokyo, Japan), after removing the gold by using an etchant solution (4.0g KI + 1.0 g I_2 + 40 mL H_2O). The depths of nanochannels before gold removal were measured by a confocal laser scanning microscope with a high resolution of 120 nm (CLSM; LEXT OLS3000, Olympus, Tokyo, Japan), and thereby the actual depths of nanochannels were obtained by subtracting the thickness of the gold layers (100 nm) from the measured depths. The morphologies of nanochannels were characterized by using an atomic force microscopy (AFM; SPA-400, SII, Tokyo, Japan). The cross-sectional shapes of the nanochannels were characterized from scanning ion microscope (SIM) images of the FIB-milled cross-sections titled 60^{o} with respect to the ion beam. The nanochannels with nanopillar array were characterized from their SIM images. All SIM images were obtained using the observation mode of the FIB system (FB-2100) with an accelerating voltage of 40 keV and a beam current smaller than 0.01 nA to avoid damage on the sample surfaces resulting from the beam irradiation.