Self-assembly Nanostructured Gold for High Aspect Ratio Silicon Microstructures By Metal Assisted Chemical Etching

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Supplementary information

Experimental

Sample Preparation

P-type Si <100> 4 inch wafers with resistivity 1-30 Ωcm were used as substrates. After Oxygen plasma cleaning, a positive photoresist MICROPOSITTM S1805 was spin-coated at 4000 rpm on the substrate and cured by a thermal treatment at 115°C for 2 min. The resist was exposed to ultraviolet radiation through a Cr mask with a grating pattern and then developed in MICROPOSIT MF-321 Developer. Two grating patterns were used, with a pitch of 4.8 μm and 2.4 μm, and line width 2 μm and 1 μm, respectively. The patterned substrate was finally washed in deionized water and then dried in N_2 gas flow. Oxygen plasma etching was then performed in order to remove any residuals of photoresist and realize a uniform Oxygen-terminated Si surface before the Au deposition. A thin Au film was deposited on the patterned wafer by physical evaporation with a deposition rate of 0.4 Å/s. Lift-off was performed in acetone after the Au deposition in order to remove the photoresist mask. Some wafers were subjected to annealing in the temperature range 180-250 °C for 30 min in air by using a standard hot-plate. MACE was performed by dipping the whole wafer in the etching solution, at room temperature, in dark conditions and then quenched with a H_2O rinse. The pattern was dried using nitrogen. The semiconductor fabrication grade chemicals, HF (10 wt%), H_2O_2 (30-31 wt%), IPA alcohol (99 wt%) were provided by Technic and used for the experiments. Samples of Figure 1.d - 1.i were etched for 10 min with the following solution: 35 ml of HF, 24 ml of H_2O_2, 41 ml of H_2O (deionized water). Samples of Figure 1.j-l and Figure 2 were etched for 2 min (Figure 1.j), 10 min (Figure 1.l), 15 min (Figure 2.a) and 40 min (Figure 2. c, f, g), respectively, with the following solution: 35 ml of HF, 57 ml of H_2O_2, 8 ml of H_2O (deionized water). Samples of Figure 3.a and Figure 3.b were etched for 3 h and 6 h, respectively, with the following solution: 68 ml of HF, 18 ml of H_2O_2, 14 ml of IPA. Etching rate of Figure 1.j was measured with 10 min etching, the etching rate was linear with the time as it was verified by sampling at 5, 10, 15, 20, 30 min within the experimental error reported in Figure 1.j.

Characterization details

SEM was performed with a Zeiss Supra VP55 – Gemini column, AFM was performed with Veeco-Innova microscope, RBS was performed with a 3.5 MV Singletron Accelerator HVEE by using a 2 MeV $^4He^+$ beam with a scattering angle of 165°, RBS spectra were fitted by using RUMP.[27] For SEM cross-view, samples were cleaved along the <100> Si axis, which the grating pattern is aligned, as showed in the insert of Figure 3.c. SEM conditions were the following: 5 kV, in-lens detector, 3° tilt (Figure 1 and 2); 20 kV, 30° tilt with tilt correction and dynamic focus (Figure 3). Data of Figure 3.c were acquired by analyzing several SEM images at 5 kV, in-lens detector, 3° tilt, and magnification of 5000 X.