

Fabrication of Nanohole Arrays with Nano-Imprint Lithography

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Abstract and Introduction:

Sub-wavelength metallic nanostructures can excite surface plasmons, which are electromagnetic waves at the metal interface. When light is incident on a periodic array of sub-wavelength holes in a noble metal film, more light passes through the array than predicted by aperture theory. This is called extraordinary optical transmission (EOT) and believed to be a result of surface plasmon resonance (SPR). SPR biosensors have been used in measuring the binding kinetics of biomolecules [1].

A method for fabricating nanostructures, specifically nanohole arrays, is necessary for this biosensing application. Current fabrication methods for nanohole arrays include focused ion beam (FIB) milling and e-beam lithography (EBL). These techniques are not practical for large-scale production. Nanosphere lithography can do this [1]; however, the process requires numerous steps and uniformity is limited. Another technique, nano-imprint lithography, offers the advantages of low cost, high throughput, and uniformity.

This research focused on nano-imprint for the fabrication of nanohole arrays in metal films, and three methods for mold fabrication: (a) polystyrene nanospheres, (b) double imprinting, and (c) metal masking.

Mold Fabrication:

Reactive ion etches used:

NIL: 10 sccm O₂ at 50W RF power and 20 mtorr

OXIDE: 50 sccm Ar, 25 sccm CF₄ and 50 sccm CHF₃ at 150W RF power and 75 mtorr

O2SHRINK: 10 sccm Ar and 35 sccm O₂ at 60W RF power and 100 mtorr

SISLW: 40 sccm CF₄ and 4 sccm O₂ at 25W RF power and 100 mtorr

Nanosphere Lithography. A solution of nanospheres was prepared with 5:5:1 ratio of ethanol, de-ionized water and polystyrene nanospheres. A droplet of nanosphere solution was applied to a silicon wafer, resulting in nanosphere self-assembly. The patterned nanospheres were then shrunk with 1 min of O2SHRINK and etched with SISLW for 150 sec, resulting in silicon pillar nanostructures (Figure 1). The

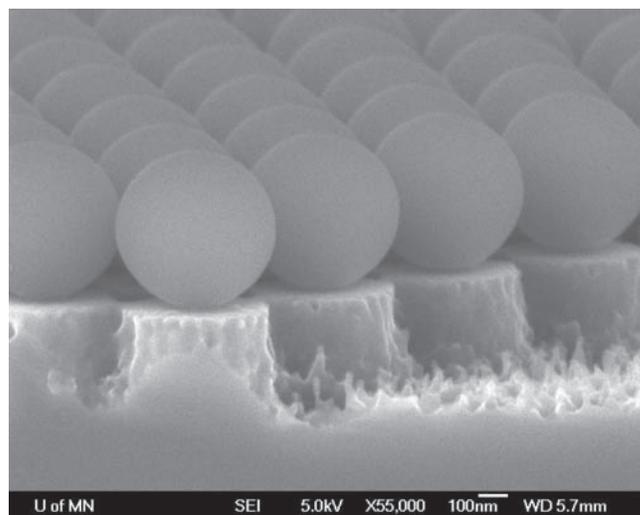


Figure 1: Nanopillars etched with nanosphere mask.

nanospheres were removed with a 30 min piranha clean (1:1 H₂SO₄:H₂O₂). The mold was placed in a desiccator with 4 drops of (heptadecafluoro-1,1,2,2-tetrahydrodecyl) trichlorosilane for 1 hour, forming a self-assembled monolayer (SAM) release layer.

Double Imprinting. Polymethylmethacrylate (PMMA) resist was spin coated on a thermally oxidized (100 nm) silicon wafer (EBL#1) at 3000 RPM for 30 sec and baked at 180°C for 90 sec. A dot array was exposed using electron beam lithography at 20 kV and with a 10 μm aperture. The PMMA was developed for 15 sec in a bath of 1:3 methyl isobutyl ketone (MIBK) and isopropyl alcohol, leaving holes in the resist. EBL#1 was etched with two RIE recipes—4 min of OXIDE and 3 min with SISLW—then cleaned for 30 min in piranha. A trichlorosilane release layer was applied to EBL#1. Another 100 nm thermally oxidized wafer (NIL#1) was coated in NXR-1025 thermal resist by spin coating at 1000 RPM and baking at 155°C for 1 min. This gave a 300 nm resist layer. The mold EBL#1 was imprinted to the new wafer at 175°C and 300 psi for 1 min. Three RIE recipes were applied to NIL#1—3 min NIL, 4 min of OXIDE, and 4 min of SISLW—leaving pillar features in SiO₂/Si. The SAM trichlorosilane release layer was applied to NIL#1.

Metal Masking. EBL was employed to pattern PMMA on a silicon wafer with 100 nm of thermally grown oxide, EBL#2. 30Å of Au/Pt was slowly deposited using sputtering over the PMMA and bare wafer. The resist was then stripped using acetone and sonication for 10 min, leaving dots of Au/Pt. Two RIE recipes were used on EBL#2; first, 3 min of OXIDE followed by 3 min of SISLW. EBL#2 was coated with the trichlorosilane release layer.

Imprinting and Etching to Metal Film. A wafer with a 200 nm silver (or gold) film, deposited with electron beam evaporation, was coated with NXR-1025 thermal resist and placed on top of the nano-imprint mold. The two wafers were placed in the nano-imprinter and run for 1 min at 175°C and 300 psi. After imprinting, the mold was removed by carefully creating a gap with a razorblade and separating with a gust from a nitrogen jet. After imprinting, there was a residual layer of resist. The residual layer was removed with the RIE recipe: NIL. This recipe gave an etch rate of approximately 900Å/min for the NXR-1025 thermal resist. Etch times varied depending upon the features imprinted. When the residual layer of resist was removed, the silver (Ag) or gold (Au) layer below was etched using argon ion sputtering.

Results and Conclusions:

In this research, nano-imprint lithography was capable of transferring nanoscale features from one mold to a substrate. It was used to replicate molds, requiring only one master mold. Imprinting was performed with pieces as small as 2 × 2cm², and has the potential to efficiently fabricate nanohole array patterns to be etched into a metallic film. Both EBL and NSL were capable of nanohole array molds with varying periodicities. Nanosphere lithography (Fig.2) eliminated EBL patterning at the loss of precise feature design, and has possibilities for cheap large-scale patterning. There was no significant difference in image quality between the metal masking (Fig.3) and double imprinting (Fig.4) techniques.

The double imprinting method was preferred because it was cheaper and created a master mold in the process. The EBL patterning incorporated in the metal masking and double imprinting processes allowed for more pattern control, with options for alternative features. All three molds lost feature quality when the pattern was transferred to the metallic layer. No method was clearly superior.

Future Research:

Future research should focus on; 1) optimization of the Au/Ag etch process for improved feature quality, and 2) nanosphere self-assembly for increased patterning area.

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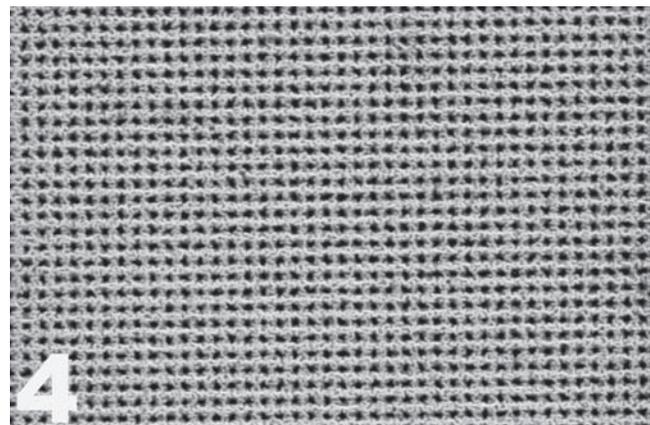
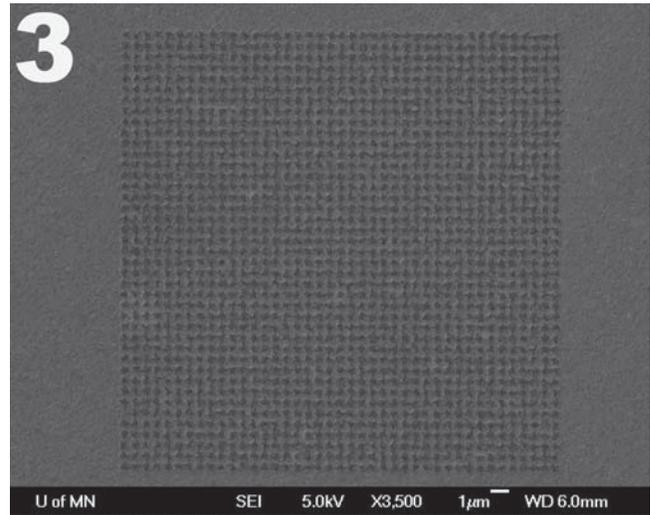
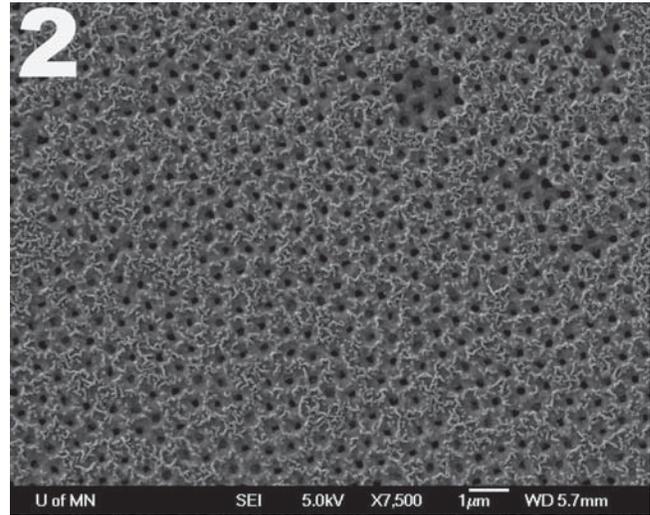


Figure 2: Nanoholes in silver patterned by nanosphere mold.

Figure 3: Nanoholes in silver patterned by metal masking mold.

Figure 4: Nanoholes in Ag patterned by double imprinting mold.

References:

- [1] Lindquist et al. Lab Chip, 9, 382-387 (2009).
- [2] T.W. Ebbesen et al. Nature, 391, 667-669 (1998).