Scalable method for the fabrication and testing of glass filled, three-dimensionally sculpted extraordinary transmission apertures

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1 Detailed Fabrication

The fabrication was done on single-side polished (100) silicon wafers. PMMA A4 950mw was spun onto the wafers at 4000 RPM for 1 minute, to a height of about 125nm. The e-beam resist was baked at 180 $^\circ$C for 5 minutes. The patterns were written with a Leica EBPG 5000+ 100kV electron beam writer with a dose of 1350 $\mu$C/cm$^2$ and developed for 45 seconds in a 1:3 solution of MIBK:IPA. Aluminum Oxide was sputtered into the regions where the PMMA was removed. The sputtering was done with a DC magnetron sputtering gun at 10 mTorr with a 5:1 Ar : O$_2$ gas mixture at 400 W. Approximately 30nm was deposited. The pattern was lifted off with sonication in dichloromethane for 1 minute.

Etching was performed in an Oxford Plasmalab 100 ICP-RIE 380. Etching was conducted with simultaneous etching (with SF$_6$) and passivation (with C$_4$F$_8$). Tuning of the relative flow rates of the etching and passivation gasses allowed for sidewall sculpting as the etch progressed into the silicon. This step is shown schematically in figure S1(a) and an SEM of an array of circular pillars is shown to the right. The samples were then oxidized at 1000$^\circ$C for 8 hours in a dry oxygen ambient (shown in figure S1(b)) to ensure that no silicon was left within the structures. Note that in this step an equally thick field of silicon dioxide is grown on the backside of the sample. A 2 nm titanium wetting layer was sputtered on the structures followed by 250nm of gold. Both were performed with DC magnetron sputtering in an Ar atmosphere. The conformal metal coating in this step is shown schematically in figure S1(c) and is also seen in the middle SEM.

The samples were then reflowed using a rapid thermal annealer (RTA). The sample temperature was ramped from room temperature to 650 $^\circ$C in 90s in a 5%/95% H$_2$ : N$_2$ atmosphere. The temperature was held for 7 minutes and lowered to room temperature in 2 minutes. The rapid heating and cooling caused the gold on the side-walls to wick and bead atop the structures. This is shown schematically in figure S1(d) and an SEM of the resulting beading is shown at the bottom.
A thin layer of PMMA (A2 950 mw) was spun on to protect the surface and the glass structures were snapped off using a clean-room compatible q-tip for mechanical cleavage. The samples were then sonicated in dichloromethane to remove the PMMA protection and any fragments of the structures left on the surface. These steps are shown schematically in figure S2(a-c). The resulting glass structures protrude from the gold surface at the height of the PMMA protection (shown in the top SEM image). The protruding tips are flattened with a $CF_4 : CHF_3 : Ar$ (45:15:60 sccm at 20mTorr, 80W fwd) RIE etch. The use of fluorocarbons preferentially etched the glass while leaving the gold chemically untouched.

A thick layer of PMMA (A8 950mw) is spun on to protect the front side and photoresist is sput on the backside of the sample (AZ 5214). A Karl-Suss MA6/BA6 backside mask-aligner is used to align a window in the photoresist to the apertures on the front-side. The photoresist is developed and buffered hydrofluoric acid is used to etch a window on the backside of the sample. The photoresist is stripped in IPA. The silicon dioxide window is used to mask an isotropic $XeF_2$ and etch terminates on the silicon dioxide surface on the front-side of the sample. Finally the protective PMMA layer is removed with an oxygen plasma. These steps are shown schematically in figure S3(a-d). Images are also shown of the front and backside of a sample with a dime to show the scale.

2 Polarization rotation animation

An animation demonstrating the effect of rotating the polarization of the input light on the color transmitted through an array of apertures is available on the web. The figure shows, centered, the Caltech logo in which the flame ‘turns-on’ as the polarization filter on the input light is aligned along the short axis of the rectangular apertures that constitute the flame. For comparison the logo to the left of the frame has only circular apertures (with the same spacing, area and distribution) and thus the colors do not change during the polarization rotation.
Figure S1: Sample fabrication flow. (a) Silicon nanostructures are etched using an ICP-RIE using electron beam lithography and an alumina hard mask. (b) The structures are oxidized to make them transparent. (c) Ti/Au 2/250nm is conformally sputtered on to the structures. (d) The gold is reflowed off of the side-walls to the top of the structures to make mechanical cleavage easier. SEMs show (top) an array of silicon pillars (middle) The conformal gold coating (bottom) gold nano-beads atop the pillars as a result of the reflow step.
Figure S2: Sample fabrication flow. (a) A thin layer of PMMA is spun onto the sample to protect the surface during mechanical cleavage. (b) A clean room compatible q-tip is used to snap off the glass pillars. (c) The PMMA protection and any re-deposited fragments are removed via sonication in dichloromethane. The apertures at the end of this step protrude slightly from the gold surface. (d) An RIE etch is used to flatten the glass apertures. SEMs show (top) an array of slightly protruding glass apertures after the mechanical cleavage step and (bottom) an aperture that is flush with the gold surface after the RIE etch.
Figure S3: Sample fabrication flow. (a) PMMA is used to protect the front-side and photoresist is used to pattern a window aligned to the apertures on the backside. (b) Bufferend hydrofluoric acid is used to transfer the window pattern into the oxide on the backside and the photoresist is removed. (c) $XeF_2$ is used to etch a window through the wafer and create an optical path. (d) The PMMA protection is removed from the front-side. Images show (top) the final gold surface of a sample and (bottom) the backside of the sample, highlighting the window from the back to the front. A dime is included for scale.