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October 30, 2018

Subject: Re-submission of manuscript - **"**Demonstration of equal-intensity beam generation by dielectric metasurfaces" – by Gwanho Yoon, Dasol Lee and Junsuk Rho

Dear Editorial Board of *Journal of Visualized Experiments*,

We would like to express our gratitude for sending us the decision letter concerning our recent submission to *Journal of Visualized Experiments*. To address all the referees' and editorial comments point-by-point, we have carefully revised the manuscript.

The main concern is insufficient descriptions on the protocol. Therefore, we have added details on the method, clarified unclear steps, and address grammar issues. We have also added additional steps on sample observation process using a scanning electron microscope for perfect reproduction of our work by readers. A figure re-print permission link is included in the rebuttal letter that contains both reviewers' and editorial comments and our responses.

The Submission includes (1) a revised manuscript (clean), (2) a revised manuscript (tracked), (3) a rebuttal letter, and (4) figure files (300 dpi tif).

Thank you for considering our manuscript for publication and look forward to hearing from you soon.

Sincerely,
Junsuk Rho

TITLE:

Demonstration of Equal-intensity Beam Generation by Dielectric Metasurfaces

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KEYWORDS:

beam splitter, Fourier hologram, hydrogenated amorphous silicon, electron beam lithography, inductively coupled plasma–reactive ion etching, plasma-enhanced chemical vapor deposition, broadband, polarization independence

SUMMARY:

A protocol for the fabrication and optical characterization of dielectric metasurfaces is presented. This method can be applied to the fabrication of not only beam splitters, but also of general dielectric metasurfaces, such as lenses, holograms, and optical cloaks.

ABSTRACT:

The fabrication and characterization protocol for a metasurface beam splitter, enabling equal-intensity beam generation, is demonstrated. Hydrogenated amorphous silicon (a-Si:H) is deposited on the fused silica substrate, using plasma-enhanced chemical vapor deposition (PECVD). Typical amorphous silicon deposited by evaporation causes severe optical loss, impinging the operation at visible frequencies. Hydrogen atoms inside the amorphous silicon thin film can reduce the structural defects, improving optical loss. Nanostructures of a few hundreds of nanometers are required for the operation of metasurfaces in the visible frequencies. Conventional photolithography or direct laser writing is not feasible when fabricating such small structures, due to the diffraction limit. Hence, electron beam lithography (EBL) is utilized to define a chromium (Cr) mask on the thin film. During this process, the exposed resist is developed at a cold temperature to slow down the chemical reaction and make the pattern edges sharper. Finally, a-Si:H is etched along the mask, using inductively coupled plasma–reactive ion etching (ICP-RIE). The demonstrated method is not feasible for large-scale fabrication due to the low throughput of EBL, but it can be improved upon by combining it with nanoimprint lithography.

The fabricated device is characterized by a customized optical setup consisting of a laser, polarizer, lens, power meter, and charge-coupled device (CCD). By changing the laser wavelength and polarization, the diffraction properties are measured. The measured diffracted beam powers are always equal, regardless of the incident polarization, as well as wavelength.

INTRODUCTION:

Metasurfaces consisting of two-dimensional subwavelength antenna arrays have demonstrated many promising optical functionalities, such as achromatic lenses[1,2], holograms[3–6], and optical cloaks[7]. Conventional bulky optical components can be replaced with ultrathin metasurfaces while maintaining the original functionalities. For example, a beam splitter is an optical device used to separate an incident beam into two beams. Typical beam splitters are made by combining two triangular prisms. Since their interface characteristics determine the beam splitting properties, it is hard to reduce the physical size without functional degradation. On the other hand, ultrathin beam splitters can be realized with metasurfaces encoded with a one-dimensional linear phase gradient[8,9]. The thickness of metasurfaces is less than their working wavelengths, and separation properties can be controlled by the phase distribution.

We designed a metasurface beam splitter which can generate equal-intensity beams regardless of the incident polarization states[10]. This characteristic comes from a Fourier hologram. Due to the image of two white spots on a black background, generated hologram from the metasurface is the same as the encoded image. The Fourier hologram does not have a specific focal length, so the encoded image can be observed in the whole space behind the metasurface[11]. If the same two-spot image is generated behind the metasurface, it also works as a beam splitter. The Fourier hologram by the metasurface creates an inverted image, which is called a twin image, with respect to the orthogonal polarization states. The twin image is typically regarded as noise. However, the two-spot image encoded in this metasurface is origin-symmetric, resulting in a perfect overlap of the original and twin images. Since any polarization states can be represented by a linear combination of right-handed (RCP) and left-handed (LCP) circular polarizations, the device described here shows the polarization-independent functionality.

Here, we present a protocol for the fabrication and optical characterization of dielectric metasurfaces enabling equal-intensity beam generation. The phase distribution of this device is retrieved from the Gerchberg–Saxton (GS) algorithm, which is generally used for phase-only holograms[12]. a-Si:H of 300 nm thick is deposited on the fused silica substrate, using PECVD. A Cr mask is defined on the a-Si:H film, using EBL. The mask pattern corresponds to the phase distribution derived from the GS algorithm. ICP-RIE is exploited to etch the a-Si:H film along the Cr mask. The rest of the Cr mask is removed by Cr etchant finalizing the sample fabrication. The optical functionality of the fabricated metasurface is characterized using a customized optical setup. When a laser beam is incident to the metasurface, the transmitted beam is separated into three parts, namely two diffracted beams and one zeroth-order beam. The diffracted beams deviate from an extension of the incident beam path while the zeroth-order beam follows it. To verify the functionality of this device, we measured the beam power, beam profile, and diffracted angle using a power meter, CCD, and protractor, respectively.

All the fabrication processes and materials used are optimized for the target functionality. For visible working frequencies, the individual antenna sizes should be a few hundreds of nanometers, and the material itself should have a low optical loss at visible wavelengths. Only a few kinds of fabrication methods are applicable when defining such small structures. Typical photolithography, as well as direct laser writing, are incapable of the fabrication due to the diffraction limit. Focused ion beam milling can be used, but there are critical issues of gallium contamination, pattern design dependence, and the slow process speed. Practically, EBL is the only way to facilitate the fabrication of metasurfaces working at visible frequencies[13].

Dielectrics are usually preferred due to the unavoidable ohmic loss of metals. The optical loss of a-Si:H is low enough for our purpose. Although the optical loss of a-Si:H is not as low as low-loss dielectrics such as titanium dioxide[1,4] and crystalline silicon[14], the fabrication of a-Si:H is much simpler. Typical evaporation and sputtering processes are not capable of the deposition of an a-Si:H film. PECVD is usually required. During the PECVD process, some hydrogen atoms from SiH_4 and H_2 gases are trapped among the silicon atoms, resulting in an a-Si:H film. There are two ways to define a-Si:H patterns. One is the deposition of a-Si:H on a patterned photoresist, followed by the lift-off process, and the other is by defining an etching mask on the a-Si:H film, followed by the etching process. The former is well-suited to evaporation processes, but it is not easy to deposit a-Si:H film using evaporation. Hence, the latter is the optimal way to make a-Si:H patterns. Cr is used as the etching mask material because of its high etching selectivity with silicon.

PROTOCOL:

1. Fabrication of the dielectric metasurface

1.1. Precleaning of a fused silica substrate

1.1.1. Prepare a double-side polished, fused silica substrate (length: 2 cm; width: 2 cm; thickness: 500 µm).

1.1.2. Immerse the fused silica substrate in 50 mL of acetone and conduct the sonication process for 5 min at 40 kHz.

1.1.3. Immerse the substrate in 50 mL of 2-propanol (IPA) and conduct the sonication process for 5 min at 40 kHz.

1.1.4. Rinse the substrate with the IPA and blow nitrogen (N_2) gas to dry the substrate before the evaporation of the IPA.

1.2. Deposition of a-Si:H by PECVD

1.2.1. Locate the prepared substrates on a zig inside the load lock chamber of the PECVD system.

1.2.2. On PECVD software, set the chamber temperature to 300 °C and set the radio frequency power to 800 W.

1.2.3. Set the SiH_4 gas flow rate to 10 sccm and the H_2 gas flow rate to 75 sccm.

1.2.4. Set the process pressure to 25 mTorr. Click the **Start** button to start the deposition process, which takes ~300 s.

1.3. Formation of the Cr etching mask

1.3.1. Load the sample obtained from step 1.2.4 on the sample holder of the spin coater. Release poly(methyl methacrylate) (PMMA) A2 on the sample using a filter-mounted 5 mL syringe and start the coating process with a rotation speed of 2,000 rpm for 1 min.

NOTE: Released PMMA should cover the whole substrate; otherwise, the spin-coated film will not be uniform.

1.3.2. Transfer the sample from the sample holder to a hot plate, and bake the sample with a hot plate at 180 °C for 5 min. Then, cool the sample at room temperature for 1 min.

1.3.3. Load the sample on the sample holder of the spin coater. Release E-spacer on the sample, using a 1 mL pipette, and start the coating process with a rotation speed of 2,000 rpm for 1 min.

NOTE: Released E-spacer should cover the whole substrate; otherwise, the spin-coated film will not be uniform.

1.3.4. Load and fix the sample on the zig for EBL. Put the zig into the EBL chamber and, then, load it into the main chamber.

1.3.5. On the EBL console, push the **Isolation** button and, then, the **FC** button. Set the magnification to its maximum value using the magnification knob.

1.3.6. Turn the **Zero check** button on. Turn the **Beam current** knob to set the beam current value to 50 pA. Turn the **Zero check** button off.

1.3.7. Push the **Reference** button to move the stage to the reference position. Turn the **Blank** button off.

1.3.8. Set the magnification value to 100,000 using the magnification knob. Adjust the focus and stigmatism knobs to obtain the clearest image in the EBL display. Turn the **Blank** button on.

1.3.9. On the computer connected to the EBL console, run the Linux terminal. Move the current location to the folder that has the .gds file, using the **cd** command.

1.3.10. Enter **gds2cel** to convert the .gds file to a .cel file and wait until it finishes. Enter **job** to run the main software.

1.3.11. Click the **Chip size modification** menu. Select **600 µm x 600 µm** and **240,000 dots**. Click **Save** and then **Exit**.

1.3.12. Click the **Pattern data creation** menu. Enter **ps** in the command window to load the pattern .cel file generated from step 1.3.10. Enter **i** in the command window and click the pattern to magnify the pattern image.

1.3.13. Enter **sd** in the command window and **3** to set the dose time to 3 µs. Enter **sp** in the command window and **1,1** to set the exposing pitch to a normal condition. Enter **pc** in the command window and a filename to create a .ccc file. Click the center of the pattern.

1.3.14. Enter **cp** in the command window and click the pattern to apply the exposing conditions from step 1.3.13. Enter **sv** in the command window and a filename to create a .con file. Enter **q** in the command window to exit the **Pattern data creation** menu.

1.3.15. Click the **Exposure** menu. Enter **i** and the .con filename from step 1.3.14. Enter **e** and click the **Exposure** button to start the exposing process.

NOTE: The exposing time depends on the pattern area and density. General metasurface patterns of a 300 µm x 300 µm area take ~3 h.

1.3.16. When the exposing process finishes, turn the **Isolation** button off. Push the **EX** button to move the stage.

1.3.17. Unload the sample from the chamber after finishing the exposure. Immerse the sample in 50 mL of deionized (DI) water for 1 min to remove the E-spacer.

1.3.18. Prepare 10 mL of methyl isobutyl ketone (MIBK):IPA = 1:3 solution in a beaker surrounded by ice. Immerse the sample into the MIBK:IPA = 1:3 solution for 12 min. Then, rinse the sample with the IPA and blow N_2 gas to dry the sample.

1.3.19. Load and fix the sample on the holder of the electron beam evaporator. Mount the holder inside the chamber of the evaporator.

1.3.20. Load a graphite-crucible-containing piece-type Cr inside the evaporation chamber.

1.3.21. On the software of the electron beam evaporator, click the **Chamber pumping** button to create a vacuum on the inside of the chamber, and lower the pressure to 3 x 10^{-6} mTorr.

1.3.22. Select **Chromium** in the material section and click the **Material** button to apply it. Click the **E-beam shutter** button to open the source shutter. Click the **High voltage** and the **Source** button, in that order.

1.3.23. Click the upward arrow button to increase the electron beam power slowly, and repeat this until the deposition rate reaches 0.15 nm/s.

NOTE: One click per 5 s is slow enough.

1.3.24. Click the **Zero** button to reset the thickness gauge. Click the **Main shutter** button to open the main shutter. When the thickness gauge reaches 30 nm, click the **Main shutter** button to close the main shutter.

NOTE: Deposition time can be easily calculated from the deposition rate. A 30 nm-thick deposition takes ~200 s, in the condition used here.

1.3.25. Click the **E-beam shutter** button to close the source shutter. Click the downward arrow button to decrease the electron beam power slowly, and repeat this until the power reaches 0.

NOTE: One click per 5 s is slow enough.

1.3.26. Click the **Source** and, then, the **High voltage** button. Wait for 15 min to cool the chamber. Click the **Vent** button to vent the chamber and unload the sample from the holder.

1.3.27. Immerse the sample in 50 mL of acetone for 3 min. Conduct the sonication process for 1 min at 40 kHz. Rinse the sample with IPA and blow N_2 gas to dry the sample.

1.4. Etching process of a-Si:H

1.4.1. Spread thermal glue to the back of the sample. Attach the sample on the zig and load the zig on the etching system.

1.4.2. On the software, set the chlorine (Cl_2) gas flow rate to 80 sccm and the hydrogen bromide (HBr) gas flow rate to 120 sccm. Set the source power to 500 W and the bias to 100 V. Click the **Start** button to start the etching process for 100 s.

1.4.3. Unload the sample and remove the thermal glue with a dustproof wiper.

1.4.4. Immerse the sample in 20 mL of Cr etchant for 2 min and in 50 mL of DI water for 1 min. Rinse the sample with DI water and blow N_2 gas to dry the sample.

1.5. Obtaining the scanning electron microscope image of the fabricated metasurface

1.5.1. Load the sample on the sample holder of the spin coater, release the E-spacer on the sample using a 1 mL pipette, and start the coating process with a rotation speed of 2,000 rpm for 1 min.

1.5.2. Fix the sample on the sample holder of the scanning electron microscope (SEM), using carbon tape. Put the holder in the load lock chamber of the SEM and create a vacuum in the load lock chamber.

1.5.3. Transfer the holder from the load lock chamber to the main chamber. Turn the electron beam on with a 15 kV acceleration voltage.

1.5.4. Move the stage to a 1 cm working distance. Find the metasurface by moving the stage horizontally. Adjust the stigmatism and focal length until the image becomes clear.

1.5.5. Capture the images.

1.5.6. Turn off the electron beam. Move the stage to the extraction position. Transfer the holder from the main chamber to the load lock chamber.

1.5.7. Vent the load lock chamber and unload the sample.

1.5.8. Immerse the sample into 50 mL of DI water for 1 min to remove the E-spacer. Blow N_2 gas to dry the sample.

2. Optical characterization of the dielectric metasurface

NOTE: Direct radiation of a laser can damage eyes. Avoid direct eye exposure and wear the appropriate laser safety glasses.

2.1. Mount a 635 nm-wavelength laser on the optical table (**Figure 1a**). Turn the laser on and wait for 10 min to stabilize the beam power.

2.2. Adjust the horizontal and vertical alignment of the laser using an alignment screen both near and far from the laser.

2.3. Place a neutral density filter in front of the laser. Mount the first convex lens behind the neutral density filter. Place an iris at the back focal plane of the convex lens to remove noise.

2.4. Mount the second convex lens with twice the focal length from the first convex lens. Place a linear polarizer behind the second convex lens. Place a right-handed circular polarizer behind the linear polarizer.

2.5. Mount the third convex lens behind the circular polarizer. Mount the fabricated metasurface on the holder. Locate the metasurface at the back focal plane of the convex lens.

NOTE: The laser beam should be incident from the substrate to the patterned area.

2.6. Place a thick white paper screen, which has a 1 cm-diameter hole in the center, behind the metasurface. Mount a protractor on the optical table aligning the origin with the metasurface.

2.7. Measure the power of the three diffracted beams, which are three bright spots on the screen, using a power meter.

NOTE: If the laser beam power is not maintained at a constant, calculate the average beam power over a period of time.

2.8. Replace the right-handed circular polarizer with a left-handed circular polarizer. Measure the three diffracted beam powers, using the power meter.

2.9. Remove the left-handed circular polarizer. Measure three diffracted beam powers, using the power meter.

2.10. Decrease the laser beam power, using the neutral density filter to allow a CCD measurement. Place the right-handed circular polarizer. Capture the three diffracted beam profiles using the CCD.

NOTE: Weaker laser beam power is preferred, to prevent CCD damage. A beam power of 300 μW has been used in this work.

2.11. Replace the right-handed circular polarizer with the left-handed circular polarizer. Capture the three diffracted beam profiles, using the CCD.

2.12. Remove the left-handed circular polarizer. Capture the three diffracted beam profiles, using the CCD.

2.13. Replace the 635 nm-wavelength laser with a 532 nm-wavelength laser.

2.14. Repeat steps 2.2 to 2.12.

REPRESENTATIVE RESULTS:

The measurement results show the polarization-independent functionality of the device presented here (**Figure 1**). Measured beam powers of diffraction orders of $m = \pm 1$ are equal regardless of the incident polarization state (i.e., RCP, LCP, and linear polarization). Since any arbitrary polarization states can be decomposed by the linear combination of RCP and LCP, the device's functionality can be maintained, regardless of polarization states. The diffraction angles are 24° and 28.5° for the wavelengths of 532 nm and 635 nm, respectively, and the angles can be controlled by changing the encoded hologram.

Diffraction efficiency is defined by the ratio of the diffracted beam powers (m = ± 1) to the incident beam power. The device presented here consists of same-sized nanorods with different orientations based on the geometric-phase-resulting broadband operation (**Figure 2**). Theoretically, the efficiency should be higher than 20% for both wavelengths. However, the measured diffraction efficiencies are 18.3% at λ = 532 nm and 9.1% at λ = 635 nm. The discrepancy mainly comes from the beam size being larger than the metasurface itself. The measured profiles of the zeroth-order beam clearly show that the incident beam size is larger than the metasurface (i.e., the excess portion of the incident beam directly goes to the power meter without interacting with the metasurface, reducing the diffraction efficiency) (**Figure 3**).

FIGURE LEGENDS:

Figure 1: Diffracted beam power measurement. (**a**) The optical setup for the laser illumination. The next two panels show the measured diffracted beam power (**b**) at λ = 532 nm and (**c**) at 635 nm. Since the laser beam power was not consistent, the measured beam powers are calculated by time-averaging recorded values. The error bars in the figure represent the maximum and minimum values during the recording time. This figure has been modified from Yoon et al.[10].

Figure 2: SEM images of the fabricated metasurface. (**a**) Top view of the metasurface. (inset) Geometry of the unit cell: length (L) = 150 nm, width (W) = 80 nm, height (H) = 300 nm, and pitch (P) = 240 nm. (**b**) Perspective view of the metasurface. This figure has been modified from Yoon et al.[10].

Figure 3: Captured beam profiles by CCD taken 14 cm behind the metasurface. The beam diameters can be estimated at ~3 mm at λ = 532 nm and ~5 mm at λ = 635 nm. The corresponding beam diverging angles are approximately 2.5° and 4.1°, respectively. The captured beam profiles have laser speckles, but they can be removed by diffusers[1] or Dammann grating[3,15]. This figure has been modified from Yoon et al.[10].

DISCUSSION:

Some fabrication steps should be conducted carefully, to generate a metasurface that is the same as the original design. In the resist development process, a low-temperature solution is usually preferred. The standard condition is room temperature, but the reaction speed can be slowed down by decreasing the solution temperature to 0 °C. Although the corresponding reaction time becomes longer, a finer pattern can be obtained than with standard conditions. The reaction time control is also easy owing to the low reaction speed. Another critical step for a fine pattern is drying IPA after the resist development. The N_2 gas moves and evaporates the rest of the IPA on the sample. Some amount of IPA does not move, creating randomly distributed islands. If the IPA islands are formed and then evaporated, the sample will be damaged. Therefore, to minimize IPA island formation, strong blowing is better than weak blowing, unless the substrate is broken by the strong air flow. An appropriate power of the sonication is helpful to clearly peel off the thin film. After the lift-off step, it is possible to check whether the thin film clearly peels off or not by using a conventional optical microscope. Fortunately, if any Cr thin film remains on the patterned area, it is possible to remove the residue by an additional sonication process. This is a

considerable advantage of the Cr mask, because masks made of other materials, such as gold, are extremely difficult to remove once the residue dries.

EBL is an effective method to fabricate nanoscale structures, but this method suffers from a low throughput, impinging large-scale manufacturing. One way to improve productivity is by making master molds, using EBL, and printing the pattern using the master mold. This method is called nanoimprint lithography. Although the fabrication of the mold using EBL takes a long time, the result is that patterns can be transferred in a short time, using the mold that can be used repeatedly. Moreover, it is also possible to transfer the pattern onto a flexible substrate by modifying the printing processes.

In this work, we present a detailed process for the fabrication of dielectric metasurfaces. The method is not limited to the application of beam splitters; other metasurface applications, such as lenses, holograms, and optical cloaks, can be realized via this method. Compared to plasmonic metasurfaces, dielectric metasurfaces provide a much higher efficiency at visible wavelengths due to low optical losses of dielectrics. Hence, this protocol can pave the way to study and develop practical metasurfaces.

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DISCLOSURES:

The authors have nothing to disclose.

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Figure 1

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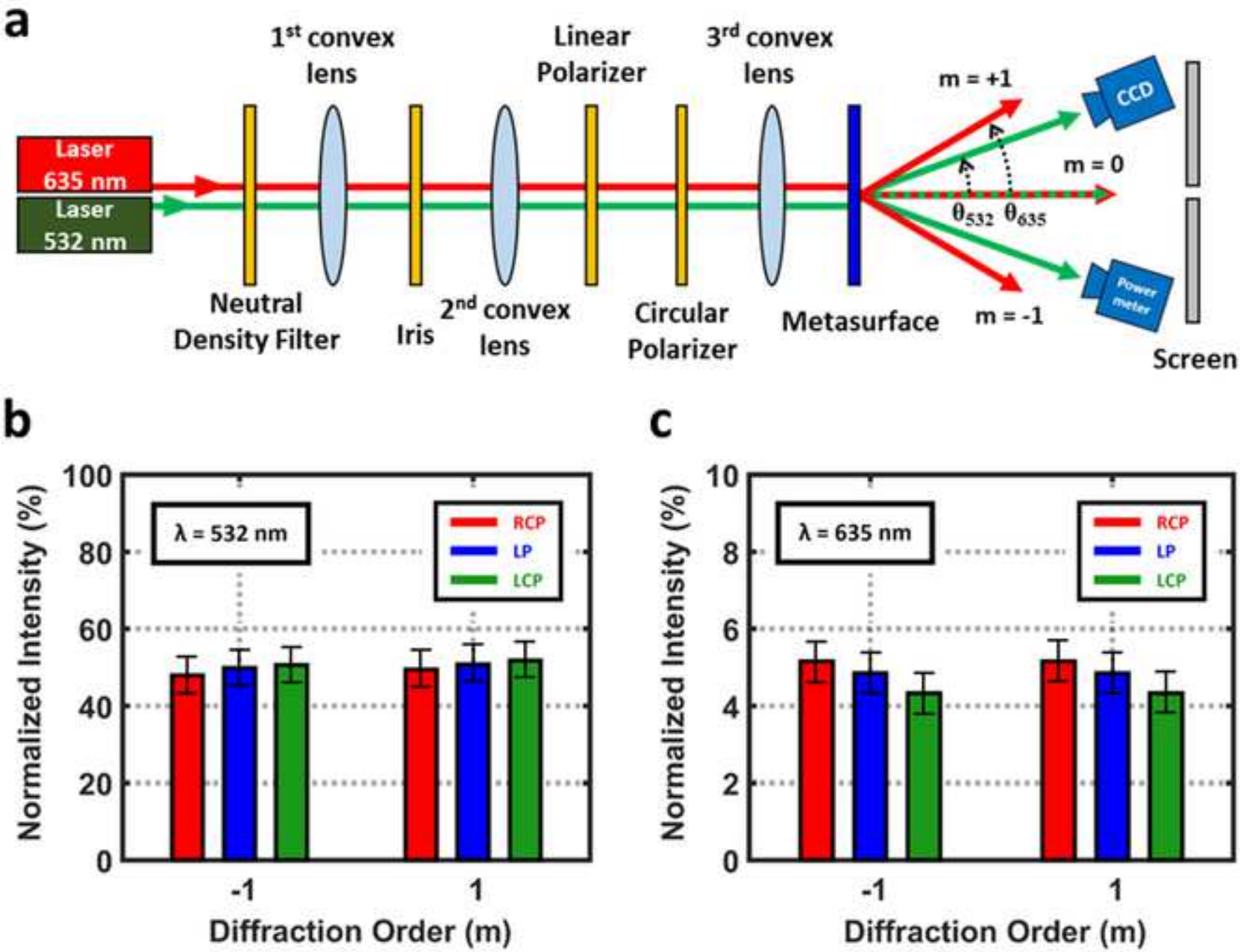


Figure 2

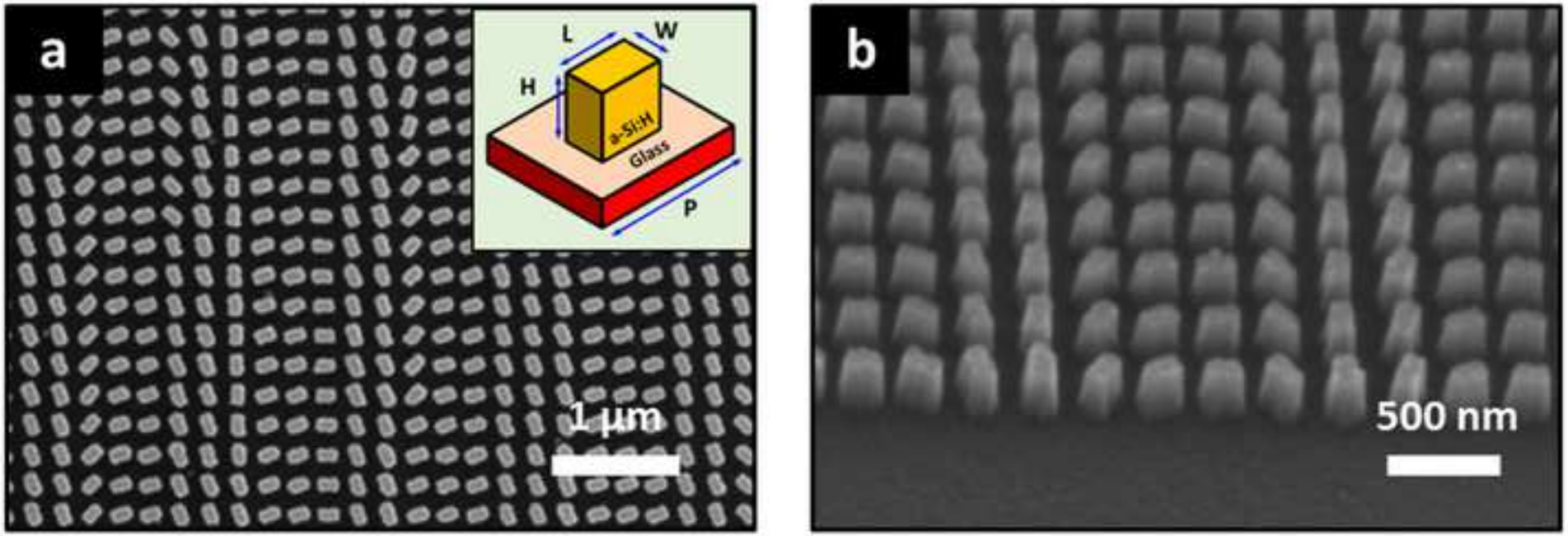
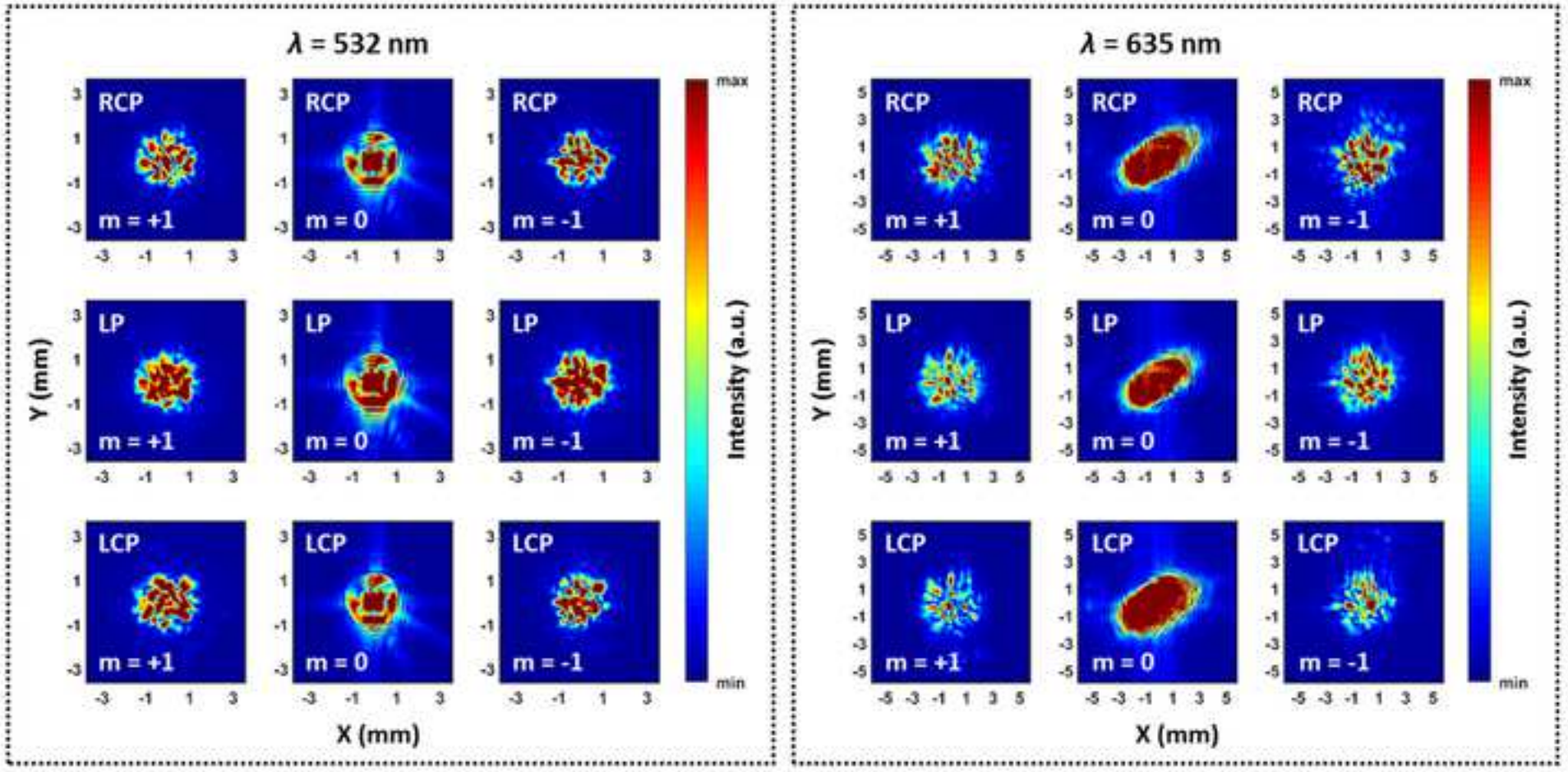


Figure 3



Name of Material/ Equipment	Company	Catalog Number	Comments/Description
Plasma enhanced chemical vapor deposition	BMR Technology	HiDep-SC	
Electron beam lithography	Elionix	ELS-7800	
E-beam evaporation system	Korea Vacuum Tech	KVE-E4000	
Inductively-coupled plasma reactive ion etching	DMS	-	
Ultrasonic cleaner	Honda	W-113	
E-beam resist	MICROCHEM	495 PMMA A2	
Resist developer	MICROCHEM	MIBK:IPA=1:3	
Conducting polymer	Showa denko	E-spacer	
Chromium etchant	KMG	CR-7	
Acetone	J.T. Baker	925402	
2-propanol	J.T. Baker	909502	
Chromium evaporation source	Kurt J. Lesker	EVMCR35D	
Collimated laser diode module	Thorlabs	CPS-635	wavelength: 635 nm
ND:YAG laser	GAM laser	GAM-2000	wavelength: 532 nm
power meter	Thorlabs	S120VC	
CCD Camera	INFINITY	infinity2-2M	
ND filter	Thorlabs	NCD-50C-4-A	
Linear polarizer	Thorlabs	LPVISA100-MP2	
Lens	Thorlabs	LB1676	
Iris	Thorlabs	ID25	
Circular polarizer	Edmund optics	88-096	
sample holder	Thorlabs	XYFM1	
PECVD software	BMR Technology	HIDEP	

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Point-by-point response to editorial comments

We appreciate for the constructive editorial comments on our manuscript. Followings are our point-by-point responses, and they have been applied to the revised manuscript.

Comment #1

1. Please provide software information in the Table of Materials.

Response #1

We have included software information in the Table of Materials.

Comment #2

1. Please combine some of the shorter Protocol steps so that individual steps contain 2-3 actions and maximum of 4 sentences per step.

2. Please check that the highlighted steps are continuous and tell a complete story.

Response #2

We have combined the shorter protocol steps, and revised the highlighted steps to tell a complete story.

Comment #3

3. Please note that step numbering is not continuous here. Please check whether steps are missing here. Otherwise please update the numbering.

Response #3

We have corrected the numbering typo.