Double metal layer lift-off process for the robust fabrication of plasmonic nano-antenna arrays on dielectric substrates using e-beam lithography

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Abstract: Integrated plasmonic sensors often require the nanofabrication of metallic structures on top of dielectric substrates by nanolithographic methods such as electron beam lithography (EBL). One of the preferred metals for the realization of such nanostructures is gold given both its corrosion resistance and favorable refractive index in the visible and NIR regions. Due to its inert nature, gold offers very poor adhesion to dielectric layers, therefore often requiring the deposition of a thin metallic layer as adhesion promoter. The presence of this layer has a negative influence on the plasmonic behavior of the resulting nano-antennas. Thus, the thickness of the adhesion layer should be kept as thin as possible. Moreover, the use of EBL on non-conductive substrates leads to charge accumulation in the isolating materials (i.e., charging effect), which degrades the resolution of the lithography. A possible solution to this problem is the use of an anti-charging layer under the electron sensitive resist, which should be thick enough to offer high conductivity.

In this work, we present a nanofabrication process that decouples the contradicting requirements for the metal layers, permitting to independently optimize both the thickness and type of the metal used as anti-charging layer and as adhesion layer underneath the nanostructures. Additionally, the proposed method permits eliminating any metal residue during the lift-off process, leading to a perfectly clean device outside the nanostructured region, which is instrumental when the nanostructures are to be integrated with other photonic functions on the chip.

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1. Introduction

Since the first report of surface plasmons by Ritchie in 1957 [1], research in the field of plasmonics has paved the way to multiple applications, some of them having already led to commercial products, such as surface plasmon resonance (SPR) sensors [2], which are routinely utilized in analytical laboratories to monitor the kinetics of surface binding effects [3] and surface-enhanced Raman spectroscopy (SERS) [4,5], which is now a mature technique for which commercial substrates are available. Plasmonic antennas, which receive such name in direct analogy with their RF counterparts, are nanostructures capable of concentrating light in specific regions of the space known as hot-spots. The intense near-field in such regions can be used to boost the weak intensity of signals generated by the analytes of interest, such as Raman scattering in SERS. Plasmonic nano-antennas can be integrated onto optical waveguides, leading to the realization of integrated plasmonic sensors, which combine the signal enhancement of the plasmonic structures with the miniaturization, scaling and interfacing provided by the integrated waveguides [6–12]. Given the subwavelength dimensions of plasmonic nano-antennas and considering the inverse relationship between the Raman scattering cross-section and wavelength used, 785 nm is one the most used wavelengths to perform this technique since it offers a compromise between performance and technological requirements. Integrated optical waveguides operating at this wavelength are often
fabricated using dielectric materials, including silicon dioxide (SiO$_2$), silicon nitride (Si$_3$N$_4$) [13], aluminum oxide (Al$_2$O$_3$) [14,15] and titanium oxide (TiO$_2$) [16].

Gold (Au), silver (Ag) and copper (Cu) are the most commonly used metals in plasmonics due to their dielectric permittivity in the visible and the near-infrared wavelength ranges [17]. In cases where the nanoparticles are exposed to hostile environments, such as environmental sensors, gold is preferred given its inert behavior. However, noble metals show poor adhesion and mechanical properties when deposited on dielectric substrates such as metal oxides [18–20]. In order to improve adhesion, a thin layer of other material that acts as an adhesion promoter is typically utilized. Titanium (Ti) and chromium (Cr) are two of the most used materials for this task. Due to their highly reactive nature, such materials are able to oxidize and establish strong bonds between the substrate and the upper metallic layers. A very complete study on the mechanical, electrical and chemical properties of these metals when combined with gold layers is provided in [21]. Cr forms a Cr-Au inter-compound while Ti acts as a separate layer that provides the improved adhesion. Moreover, given its high electric conductivity, Cr is most useful when improved conductivity is required for the application, whereas Ti is a better candidate for plasmonic applications due to its lower influence on the optical properties of the Au material (i.e., lower diffusion of Ti atoms into the Au layer). The presence of Cr or Ti as adhesion layer has negative effects on the properties of the plasmonic nanoparticles, including broadening, intensity decrease and frequency shift of the plasmonic resonances [22–24]. The degrading effects augment as the thickness of the adhesion layer increases. Therefore, the thickness of the adhesion layer should be minimized for a good plasmonic performance.

The main difficulty in the fabrication of plasmonic nano-antennas in the near-IR (e.g., 785 nm) is their nanometric dimensions, with elements of typical sizes below 100 nm. Several nanolithography techniques have been used to fabricate plasmonic nano-antennas, including focused ion beam (FIB) [25], nanoimprint lithography (NIL) [26] and electron-beam lithography (EBL) [27]. FIB creates nano-antennas with lower structural quality compared to EBL and presents multiple problems, such as metal redeposition, ion contamination and thickness variation [28]. Furthermore, NIL requires the use of other nanofabrication techniques to generate the mold necessary to replicate the structures. Thus, EBL is frequently used for the fabrication of plasmonic nano-antennas. PMMA (poly-methyl-methacrylate) is an electron sensitive positive resist that is often used in EBL processes given its high resolution, wide range of possible thicknesses, excellent adhesion to most substrates, and removal using standard cleanroom solvents such as acetone, photoresist thinners and positive resist removers [29]. DMSO (TechniStrip Micro D350) is a solvent that allows removal of PMMA [30] at high temperatures (80°C) given its high boiling point.

Accumulation of charge is one of the main issues that limits the ultimate achievable resolution when performing EBL on non-conductive substrates. Charging can be eliminated by using an anti-charging layer. Different schemes for anti-charging in EBL have been investigated [31], including the use of a metallic layer on top of the resist, the use of a water-soluble conductive polymer on top of the resist, and the use of a metallic layer underneath the resist. A metallic layer on top of the resist reduces the achievable resolution when targeting critical dimensions in the tens of nanometers. Although the use of a polymer conductive layer has been reported to deliver the required resolution [31], any defect in the resist will lead to metal deposition during the lift-off process, which limits the performance of the optical circuits around the metallic nano-antennas. A metallic anti-charging layer underneath the PMMA is, therefore, the selected option in this work. This layer should be sufficiently thick to provide enough conductance to eliminate the accumulated charge. However, a thick anti-charging metallic layer is in contradiction with the requirement for an as-thin-as-possible adhesion layer to optimize the plasmonic performance.

Recently, Hahn et al. proposed the fabrication of gold plasmonic structures on insulating substrates using a PMMA/Al bilayer [32]. In their approach, a thick (30 nm) aluminum layer is
used to provide anti-charging. The patterns are created on a thin (45 nm) PMMA layer, which provides extra resolution due to the reduced neighboring effect during EBL. Wet etching is then used to create an undercut in the Al layer to ensure a good lift-off process. The ultimate achievable resolution is determined by the aluminum layer thickness, which makes the fabrication of multi-element plasmonic antennas with small separation between elements challenging. Additionally, inhomogeneities in the wet etching process induce extra roughness on the resulting patterns, which limits the achievable resolution.

In this work, a new nanofabrication process is proposed whereby two metals, Ti and Cr, are used, thereby decoupling the contradictory requirements for anti-charging and adhesion promotion. In the proposed process flow, the best thickness can be chosen for each application. The process is based on a metal lift-off step followed by a selective metal etching step. In the method proposed here, an undercut of the anti-charging layer is not necessary as in the work of Hahn et al. [32] since this is achieved via a negative PMMA profile, permitting the fabrication of nano-antennas with smaller features. In this paper, details of the new process are given, together with the characterization results of arrays of bowtie nano-antennas designed for surface-enhanced Raman spectroscopy and fabricated using this method.

2. Fabrication of plasmonic nano-antenna arrays on non-conductive substrates using EBL

The proposed fabrication process uses two different metals, one as anti-charging layer and the other one as adhesion promoter. The nanofabrication process flow can be seen in Fig. 1. Firstly, the substrates are cleaned using nitric acid (HNO$_3$) for 10 minutes, rinsed in DI water using a quick dump rinse (QDR) system and spin-dried. Then, the metal chosen as the anti-charging layer (metal A) is deposited using an e-beam evaporator (modified Balzers BAK 600). After the evaporation process, the samples are rinsed with IPA and a 160–170 nm PMMA layer is deposited using a spin coater. The e-beam lithography process is carried out using a Raith 150 system. Plasmonic bowtie antennas were chosen as test patterns since they are well known in the literature [33–35]. Figure 2(left) shows the typical structure of a bowtie antenna. Figure 2(right) shows the EBL mask used for the exposure of an individual antenna. After exposure, the sample is developed by direct immersion in a beaker in a mix of methyl isobutyl ketone and isopropanol (MIBK:IPA, 1:3) for 30 s followed by a 30 s rinse in a beaker with IPA. After development, the samples are stored in a beaker with deionized (DI) water to avoid wetting problems during the following etching step due to the small dimensions of the created apertures in the resist layer. Wet etching is used to eliminate the conductive layer in the open areas. Before the evaporation of the nano-antenna metals, ozone cleaning (UV ozone oven, t = 300 s, etching rate = 0.72 nm/min) is done in order to eliminate any organic residue and rests of resist in the open areas of the patterns (i.e., descum). Evaporation of the material chosen as the adhesion layer (metal B) and the gold material for the antennas is done again using e-beam evaporation. After evaporation, a lift-off process is carried out in a DMSO ultrasonic bath (VWR USC-THD) at 80 °C for 30 minutes. A frequency of 45 KHz and maximum power are used. After lift-off, the sample is first immersed in a beaker with acetone, then it is immersed in a beaker with IPA and finally it is rinsed with DI water using a QDR. At this point of the process, it is possible to inspect the sample in order to look for defects in the antenna arrays using SEM. Selective wet etching is then carried out to eliminate the conductive layer without altering the adhesion layer. As a final step, the sample is cleaned in a nitric acid (HNO$_3$) bath for 10 minutes in order to eliminate any organic residue, rinsed in DI water and spin dried.

Two experiments were carried out in order to test the previously introduced fabrication method. Cr and Ti are the two materials that have been tested both working as anti-charging and adhesion layers.
First, the fabrication of Cr/Au bowtie nano-antennas on fused silica substrates was demonstrated. The fused silica substrates had a surface of $1 \times 1$ cm$^2$. A 6 nm Ti layer was evaporated as anti-charging layer (base pressure = 1e-8 mBar, process pressure = 2.2e-7 mBar, acceleration voltage = 10 kV, e-gun intensity = 96 mA and deposition rate = 0.05 nm/s). Spin coating of PMMA 950 K A4 was made at 6000 rpm during 30 seconds. Resist pre-exposure bake was made at 180°C for 90 s using a hotplate. Characterization with ellipsometry showed a layer thickness of 167 nm. The mask for the EBL process included a dose test with bowtie nano-antenna arrays with varying length (200–320 nm) and fixed gap (20 nm), angle ($\alpha = 60^\circ$) and reduction constant ($K_c = 15$ nm). A dose variation in the range of 112–420 $\mu$C/cm$^2$ was applied in order to adjust the EBL process. A voltage of 10 kV and an aperture of 20 $\mu$m were used to create a negative slope resist cross-section (Fig. 3), beneficial during the subsequent lift-off process. The arrays included 100 $\times$ 100 antennas separated by a distance of 1 $\mu$m, having an approximated area of 100 $\times$ 100 $\mu$m. After development, selective Ti wet etching was done with 0.1% HF for 90 s in order to fully etch the Ti layer. An etching rate of ~5–10 nm/min was determined by etching a test sample with a Ti layer of equal thickness as the process sample before each individual etching step. No significant damage to the fused silica substrate was observed given the low etching rate of SiO$_2$ in 0.1% HF [36]. A Cr layer was used as adhesion promoter in this case. E-beam evaporation was used to deposit 1.8 nm of Cr and 30 nm of Au (base pressure = 3.5e-7 mBar, process pressure for Cr = 2e-7 mBar, acceleration voltage for Cr = 8 kV, e-gun intensity for Cr = 24 mA, deposition rate for Cr = 0.08 nm/s, process pressure for Au = 9.2e-7 mBar, acceleration voltage for Au = 10 kV, e-gun intensity for Au = 236 mA and deposition rate for Au = 0.08 nm/s). After lift-off, the Ti
layer was eliminated using selective wet etching. At the end of the fabrication process, high-resolution SEM (Zeiss MERLIN HR-SEM) was used to explore the resulting nanostructures. Some examples of nano-antennas created by this method and their dimensions can be seen in Fig. 4.

**Fig. 3.** SEM inspection of a bowtie nano-antenna on a test sample during the development of our fabrication process. A trench was created using focused ion beam (FIB) to explore the resist profile. The PMMA layer appears as the dark layer underneath the evaporated metal. Scale bar is 200 nm.

**Fig. 4.** HR-SEM images of bowtie nano-antennas fabricated in the first experiment and average dimensions (length and gap) in the image. All dimensions are given in nanometers. Scale bar is 200 nm.

The proposed fabrication method in the first experiment can be successfully applied on non-conductive substrates such as SiO$_2$ or Si$_3$N$_4$. However, when applied on other substrates, such as Al$_2$O$_3$ or TiO$_2$, both interesting materials for the fabrication of photonic devices, problems may arise due to the lack of selectivity of the titanium wet-etching process with respect to the substrate material. An etch rate of 0.75 nm/min was observed for TiO$_2$ in 0.1% HF. Despite the still high selectivity achieved by the etching process (>6:1), the resulting etch rate for TiO$_2$ is high enough to produce undesirable etching and modification of the cross section of any pre-existing photonic devices. The previous fabrication method can be modified to avoid the
accidental etching of the TiO$_2$ material by interchanging the metals used as the anti-charging and the adhesion layer.

In the second experiment, we tested the fabrication of Ti/Au bowtie nano-antenna arrays on a TiO$_2$ sample. The sample consisted of a 90 nm thick TiO$_2$ layer sputtered on top of an 8 $\mu$m thermal SiO$_2$ layer on a Si substrate. In the experiment, a 7 nm Cr layer was used as anti-charging layer (base pressure = 2.7e-7 mBar, process pressure = 1e-7 mBar, acceleration voltage = 8 kV, e-gun intensity = 28 mA and deposition rate = 0.06 nm/s). The spin-coating of PMMA was carried out using lower speed (4000 rpm) and longer time (45 s) in order to create a relatively thick and homogenous resist layer. Resist pre-exposure bake was made at 180$^\circ$C during 120 s using a hotplate. A new dose test was designed to create bowties with varying length in the range of 160–180 nm and fixed gap (20 nm), angle ($\alpha$=60$^\circ$) and reduction constant ($K_c$=0 nm). The nano-antennas were arranged in individual lines separated by 2 $\mu$m in both the horizontal and vertical directions. An arrow pattern was used to mark the beginning of each line. The new bowtie nano-antenna arrays were designed to operate in water (n = 1.33), which explains the reduced length considered in this case. A dose variation in the range of 460–750 $\mu$C/cm$^2$ was applied in order to adjust the EBL process. In this case, a voltage of 20 kV and an aperture of 20 $\mu$m were used to reduce the neighboring effect. The Cr material was etched after e-beam lithography and development using a mixture of perchloric acid and ceric ammonium nitrate (TechniEtch Cr01), which provides an etching rate of 60 nm/min. A maximum etching time of 15 s is utilized to open up the nano-antenna patterns prior to deposition of the Ti/Au layer. Before Au evaporation, a 2 nm Ti layer is deposited in this case as adhesion promoter (base pressure = 2.9e-7 mBar, process pressure for Ti = 6.4e-7 mBar, acceleration voltage for Ti = 10 kV, e-gun intensity for Ti = 112 mA, deposition rate for Ti = 0.07 nm/s, process pressure for Au = 6.4e-7 mBar, acceleration voltage for Au = 10 kV, e-gun intensity for Au = 256 mA and deposition rate for Au = 0.07 nm/s). The parameters for the rest of the steps of the fabrication process route remain unmodified. During this experiment, the sample was inspected by SEM (FEI NOVA600 DUALBEAM FIB/SEM) after the lift-off step. Thanks to the existence of the anti-charging layer, and despite the floating nature of the resulting nano-antennas, imaging using SEM with low-charging effect was possible. A general overview of the dose test and a detail of one of the fabricated bowtie antennas can be seen in Fig. 5.

![Fig. 5. SEM characterization of bowtie nano-antennas on top of a TiO$_2$ substrate. (left) general overview of dose test. Scale bar: 1 $\mu$m. (right) detail of one of the bowtie nano-antennas in the dose test. Dimensions of the antenna (gap and length) are indicated in the image. Scale bar: 150 nm.](image)

As mentioned above, the proposed method has the further advantage of protecting the substrate outside of the patterned nano-structures from potential defects appearing during the fabrication process that could lead to metal residues being left over it. During our fabrication experiments,
and given the short distance between the metal source and the samples in the e-beam evaporator utilized, which increased the temperature of the PMMA layer during the process, damage to the PMMA layer was observed after Cr/Au or Ti/Au evaporation. The damage consisted of bubbling and delamination of the PMMA layer [Fig. 6(b)] with the subsequent deposition of metal on undesired locations on the substrate [Fig. 6(c)]. This effect was never observed in the areas where the antennas and other patterns had been defined by e beam lithography. However, the presence of these defects is fatal if metal deposition occurs on photonic devices outside the nano-antennas region. In a conventional lift-off process, in which either a conductive polymer or metal layer is applied on top of the PMMA to prevent charging, residual fragments of gold will remain attached all over the substrate [Fig. 6(c)]. In the proposed process flow, wet-etching of the anti-charging metal layer lifts the metallic residue, producing perfectly clean samples after the completion of the process [Fig. 6(d)].

Fig. 6. (a) General overview of different bowtie nano-antenna arrays and additional test patterns in the same sample after the development and etching steps. (b) PMMA bubbling problem, (c) Defects after the lift-off process and (d) Defect-free sample after metal etching.

3. Optical characterization of plasmonic nano-antenna arrays

Arrays of bowtie nano-antennas were fabricated using the double-metal lift-off process described in the previous section (Fig. 1). The antennas were designed to have a broad resonance covering both the Raman excitation line at 780 nm and the Raman scattering signal (800 - 880 nm). Variations in the applied dose during the e-beam lithography step results in variations of the gap size and length of the bowtie antennas, tuning the resonance wavelength of the devices.

A dark-field microscopy setup was utilized to measure the scattering spectra of the different fabricated Cr/Au plasmonic antenna arrays. A schematic of the setup can be seen in Fig. 7. The setup uses a broadband light source (Fianium FemtoPower1060 Supercontinuum source SC450) and a computer-controlled monochromator (Bentham M300) to select the excitation wavelength.
A polarizing beamsplitter is used to set the polarization of the excitation beam parallel to the surface of the substrate. The sample is rotated so that the electric field oscillates along the main axis of the bowtie antennas (i.e., maximum scattering signal). The power of the excitation signal is monitored in real-time using a calibrated silicon diode (FDS1010) connected to a transimpedance amplifier and a data acquisition card (National Instruments USB-6009). The wavelength is monitored using a portable optical spectrometer (Ocean Optics USB2000+ UV-VIS-ES). Silver mirrors are used to direct the excitation signal to the different elements of the setup and to set the excitation angle, which is fixed to 23 degrees. A low-NA and extra-long working distance objective (Nikon M Plan 40 0.5 ELWD) is used to capture the generated scattering avoiding the collection of excitation signal [Fig. 7(b)]. A maximum excitation angle of 60 degrees is supported in this configuration. In order to minimize the effect of multiple-beam optical interference, the sample is mounted on a 10 mm thick BK-7 optical glass dish and refractive index matching fluid (liquid paraffin, $n = 1.48$) is deposited in between the sample and the dish. An EM-CCD camera (Andor DV885jcs-vp) is used to select the area under study and collect the scattering signal. A NIST traceable Teflon reflectance standard (LakePhotonics DRS-30-99-C) is used to calibrate the setup. LabVIEW is used to control the setup and extract the calibrated scattering spectra directly during the measurement process.

![Fig. 7.](image)

(a) Dark-field microscopy setup and schematic showing the polarization of a bowtie nano-antenna array during the measurement process, (b) Comparison between the incident angle ($\alpha_{inc} = 23^\circ$) and the admission angle ($\beta_{ad} = 30^\circ$) showing the rejection of specular reflection of the excitation signal.

Finite-difference time-domain simulations (Lumerical FDTD Solutions) are carried out in order to obtain the expected reflectivity spectra of the antennas characterized by the previous method. A broadband (680–880 nm) plane-wave source is used for excitation. The simulated antennas have 1.8 nm thick Cr layer, 30 nm thick Au layer and a 50 nm curvature radius. The refractive index values for the Au and Cr materials are extracted from the literature [37]. The fused silica material is considered as a perfect dielectric with 1.45 refractive index. Symmetric and periodic boundary conditions are set in the X and Y boundaries (plane of incidence) to reduce computational time. Therefore, an infinite array of nano-antennas is simulated. Perfect match layers (PML) boundary conditions are set in the Z boundaries in order to absorb completely the light transmitted through the substrate or reflected towards the end of the simulation region. A distance of 4 µm between the nano-antennas and the PML boundary conditions and a number of 24 PML layers are chosen to avoid artifacts in the simulation results. A frequency domain power monitor located 3 µm above the structures calculates the reflectivity values.
The measured scattering spectra of the Cr/Au bowtie nano-antenna arrays, characterized by HR-SEM in the previous section (Fig. 4), and the comparison with the expected scattering spectra from FDTD simulations can be seen in Fig. 8. Given the arbitrary character of the scattering intensity scale, the results from the simulations have been re-scaled and re-aligned one-to-one according to the baseline and mean value of the corresponding measured spectra in order to make the comparison meaningful. The experimental results are in agreement with the predictions from the simulations and results found in the literature [38,39] (i.e., an increase of the antenna length results in a red-shift of the plasmon resonance and a larger gap further induces a blue-shift in the resonance together with a decrease of the magnitude of the resonance peak).

4. Conclusion

A new fabrication process flow to create gold plasmonic nano-antenna arrays on dielectric substrates using EBL has been introduced. The fabrication process is based on a double metal layer lift-off process that permits decoupling the contradicting requirements of metal thickness for the anti-charging layer (i.e., metal should be sufficiently thick) and for the adhesion layer (i.e., metal should be as thin as possible to preserve the good plasmonic performance of the gold nano-antennas). A further advantage of the method is that the anti-charging layer protects the surface of the sample outside the nano-structures area from undesired metal deposition or adhesion, which is of key importance in integrated plasmonic and photonic sensors. Arrays of bowtie antennas of different lengths and gaps were fabricated using this method. The measured scattering spectra show the expected trend from FDTD simulations.

The developed fabrication method has led to promising results in the creation of bowtie nano-antenna arrays. The rounded edges and relatively large gaps obtained in the fabricated antennas, which are due to the limitation of our lithography system, are the main factors responsible for the broad resonances observed in the measurements. The optimization of the thickness of the different metallic layers, possible thanks to the created double metal layer lift-off process, and the use of plasmonic metals (such as aluminum) as adhesion layer could also help to improve the final antenna response.

Funding

Stichting voor de Technische Wetenschappen (STW) (STW-13328).
Acknowledgements

The authors would like to thank the members of the user committee of the WaterPrint project for their insightful conversations during the project meetings.

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