High density metamaterials for visible light

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**ABSTRACT**: Investigate a large exposure dose and characterize sub-100-nm sized split-ring resonator metamaterial structures in visible range which are potentially useful for cloaking and ultrasensitive biochemical sensors, and to explore the limit of resonator size with gold first and then extend the techniques to other metals for better characteristics. Bio-chemical sensors will be developed based on different split ring resonator geometries. In addition, metamaterials on flexible substrates for cloaking application for visible light will also be explored in this proposed effort.

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Abstract
In this project, we aimed to design, fabricate and characterize sub-100-nm sized split-ring resonator metamaterial structures in visible range which are potentially useful for cloaking and ultrasensitive biochemical sensors. We explore the limit of resonator size and also develop the application of SRR metamaterials for Bio-chemical sensing. Using our sonicated cold development electron beam lithography process that has recently been shown to overcome the tradeoff between resolution and throughput, we designed, fabricated, and characterized ultrasmall split-ring resonator metamaterial structures covering from ultraviolet to near-infrared frequency range. The smallest achievable dimensions were explored, enabling various investigations ranging from the interaction of resonance modes with the interband transition of a metal, to kinetic inductance limitation for the achievable magnetic resonance in split ring resonators. Rotationally symmetric resonator geometries were studied in detail and characterized in terms of their capability as a robust and efficient sensing platform.

1. Introduction
Metamaterial is formed out of an array of deep subwavelength metallic nanostructures that are designed to produce novel optical properties. The most widely studied aspect in metamaterials is the ability for light confinement in ultrasmall dimensions, which serves as the platform for light-matter interaction at the nanoscale [1]–[4], designing optical nanoantenna [5]–[7], and for biochemical sensing [8]–[16]. However, as the dimensional requirements are proportional to its operational wavelength, the metamaterial fabrication becomes increasingly challenging towards...
visible spectrum operation. This is attributable to the inherent tradeoff between resolution and throughput in any lithography process, where smaller dimensions can be patterned at the expense of large exposure dose, making large-scale fabrication of ultrasmall resonators not practical. Many methods have been proposed to overcome this challenge, including the use of self-assembly process in nanosphere lithography (NSL) [17], [18], and the combination of laser-induced-transfer (LIT) and focused-ion-beam (FIB) [19]. However, these methods lack the lithographic flexibility inherent in electron-beam-lithography (EBL) process and often involve complex fabrication steps. Therefore, the standard EBL method is still preferable over these non-standard methods.

We have proposed an EBL process that addresses the inherent tradeoff between resolution and throughput, where a mild sonication is introduced at specific timing for a specific duration during a cold development [20]–[22]. Based on this process, gold (Au) and silver (Ag) split-ring resonators (SRR) have been demonstrated, with the resonator size as small as ~60 nm and observed magnetic frequencies in the mid-visible spectrum range [13], [23]. Recently, we further improve this nanofabrication capability for realizing even smaller resonators, and show that such ultrasmall dimensions makes possible the experimental investigation of self-limiting mechanism for the achievable magnetic resonance that was theoretically studied over a decade ago [24]–[26]. We demonstrate aluminium split-ring resonators working on ultraviolet frequency spectrum, and discuss its unique optical properties and fabrication challenges [27]. Finally, we investigate variety of rotationally symmetric structures aimed for biosensing applications.

2. Experiment, Results, and Discussion

(1) Observation of the kinetic inductance limitation for the fundamental magnetic resonance in ultrasmall gold v-shape split ring resonators [Adv. Opt. Mater. 4, 1047 (2016)]

The kinetic inductance limitation has been proposed as the fundamental limitation for the attainable magnetic resonance frequency in SRRs, but its experimental verification remains challenging owing to the stringent dimensional requirements in the resonator nanofabrication. In this work, we report the first experimental observation for such saturation effects with saturated frequency at 520 THz using gold v-shape split ring resonators. The interplay between electric dipole and magnetic dipole as a function of SRR opening angle is theoretically studied and experimentally observed, showing the dramatic increase of intensity buildup and resonance contrast as the dominant mode changes from electric to magnetic.
Figure 1. The v-SRRs in square lattice configuration. The fabricated SRRs are of 30 nm metal thickness and 25-nm metal width. The opening angles were varied from $\theta = 180^\circ$ (horizontal nanorod) to $\theta = 0^\circ$ (vertical nanorod). The scale bars correspond to 100 nm.

The v-SRRs were fabricated by the sonicated cold development process with achievable pitch of ~40 nm. [20], [22] After lithographic patterning, the samples were evaporated with 2-nm thick titanium adhesion layer and 30-nm thick gold on ITO-coated glass, followed by lift-off in n-methyl pyrrolidone (NMP) for 10 minutes. Figure 1 shows the examples of the fabricated v-SRRs, which are characterized by the arm length ($s$) and the opening angle ($\theta$). The left panel shows the v-SRRs with 100-nm arm length and 60$^\circ$ opening angle; while the right panels show the v-SRRs with opening angles varied from $\theta = 180^\circ$ to $\theta = 30^\circ$, for a fixed arm length of 100 nm. Figure 2 shows FDTD calculations of the intensity buildup in the v-SRR as a function of the opening angle, with its corresponding electromagnetic fields distribution. The typical side and top view of the $E_x$-field distribution at the fundamental magnetic resonances is shown in the insets of Figure 2a, while the intensity buildup is numerically measured at the point “B” in the simulations. The intensity buildup is rather flat at large angles, but begins to increase sharply below $\theta \sim 50^\circ$. This remains true as the arm length is varied from $s = 100$ nm to $s = 60$ nm. The electromagnetic field distributions of v-SRR are presented in Figure 2b, showing the evolution from electric-dipole to magnetic-dipole as the opening angle is decreased from $\theta = 180^\circ$ to $\theta = 30^\circ$. At $\theta = 180^\circ$, we have a nanorod situation where the $E_x$-fields are confined at both tips, with $E_y$-fields and $H_z$-fields symmetric with respect to the horizontal axis.
In this case, the resonator behaves as a plasmonic particle, with pure electric dipole resonances corresponding to plasmonic oscillation in the direction of incidence polarization. As the angle is decreased, the accumulated electric charges (from the two tips) begin to interact capacitively, skewing the $E_y$-field and $H_z$-field distributions towards the inner side of the $v$-SRR. Thus, both electric-dipole and magnetic-dipole are generated in this situation, where the former is due to the conduction current from plasmonic oscillation in the longitudinal direction, and the latter is due to displacement current from the capacitive interaction between the $v$-SR R tips. As the gap separation decreases, the ohmic resistance remains unchanged due to a fixed metal length, while the capacitive impedance becomes smaller. This leads to a higher conduction current and stronger $E_x$-fields in the SRR gap. Thus, the electric field enhancement in the SRR gaps translates to the magnetic dipole strength, suggesting that the excitation of magnetic-dipole is more preferable at small opening angles. This is indeed depicted by the mode evolution in Figure 2b, where the transition from plasmonic particle (corresponding to electric dipole) to split-ring resonator (corresponding to magnetic dipole) occurs at $\theta \sim 50^\circ$. The lateral sizes of the $v$-SRR in the X and Y direction for a given opening angle ($\theta$) and arm length ($s$) are given by $s_x(\theta) = 2s \sin(\theta/2)$ and $s_y(\theta) = s \cos(\theta/2)$, respectively. Thus, the condition where the resonator sizes are the same in both direction, i.e., $s_x(\theta) = s_y(\theta)$, is satisfied at $\theta_T = 2 \tan^{-1}(1/2) \approx 53^\circ$, which is interestingly close to the transition angle observed in Figure 2a. Thus, from plasmonic particle perspective, the resonance for X-polarization is longer (shorter) than that for Y-polarization at $\theta > \theta_T$ ($\theta < \theta_T$). As the resonance wavelength of $m_0$ is always longer than that of $e_0 [x]$, it then follows that the $m_0$ resonance behaves more as an electric dipole at $\theta > \theta_T$ and more as a magnetic dipole at $\theta < \theta_T$. This could be the likely cause for the transition from electric-dipole to magnetic-dipole at $\theta \sim 50^\circ$ in Figure 2a.

Figure 2. Mode evolution from electric dipole-like to magnetic dipole in $v$-SRR. (a) Intensity buildup as a function of opening angle. (b) The evolution of electromagnetic field distributions ($|E_x|$, $|E_y|$, $|H_z|$) of $v$-SRR along with increasing opening angles.
In order to demonstrate the magnetic resonance frequency saturation resulting from kinetic inductance limitation, we show in Figure 3 the mapping of $m_0$ mode as a function of inverse resonator size. Here, the resonance positions of the $v$-SRRs are combined with those of the $u$-SRRs from our previous work. [13] There are 5 geometric variations for $v$-SRR with $s = 100$ nm, and 4 variations for $v$-SRRs with $s = 50$-70 nm. The variations for $s = 100$ nm are (1) $\theta = 90^\circ$, (2) $\theta = 60^\circ$, (3) $\theta = 30^\circ$, $d = 12$ nm, (4) $\theta = 30^\circ$, $d = 8$ nm, (5) $\theta = 30^\circ$, $d = 0$ nm, while the variations for $s = 50$-70 nm are (1) $\theta = 90^\circ$, (2) $\theta = 60^\circ$, (3) $\theta = 30^\circ$, $d = 14$ nm, (4) $\theta = 30^\circ$, $d = 12$ nm.

The resonator size ($s_{\text{eff}}$) of $v$-SRRs is approximated as $s_{\text{eff}} \approx (A_{\text{SRR}})^{1/2}$, where $A_{\text{SRR}} = 1/2 \times (\text{base}) \times (\text{height})$ is the area of the $v$-SRR with base and height obtained from SEM inspections. The resonator size for the $u$-SRR structures is defined as the length of the bottom arm. The saturation effect can be seen clearly at a very small resonator size, where the $m_0$ mode saturates at ~520 THz (578 nm). The same saturation effect is observed for the $e_0$ mode (not shown here), where the $e_0$ mode saturates at ~530 THz (565 nm). Note that the observed two saturation frequencies are still lower than the interband transition of gold at 556 THz (539 nm). This is as expected since the plasmonic oscillation is significantly suppressed by the interband transition. The role of interband transition in the magnetic resonance is also investigated numerically, where finite difference time domain calculations are performed based on Drude metals and on gold permittivity according to Johnson and Christy [28].

(2) Preferential excitation of the hybrid magnetic-electric mode as a limiting mechanism for achievable fundamental magnetic resonance in planar aluminium nanostructures [Adv. Mater. 28, 889 (2015)]

Owing to its bulk plasma oscillation in the deep ultraviolet and higher free electron density, aluminum nanostructures has been shown to exhibit better plasmonic performance as compared
to its gold and silver counterparts in the UV-Vis frequency range [29], [30]. Different kinds of aluminum nanostructures have been explored [31]–[34], and their electric dipole modes have been experimentally studied and mapped [31]. Meanwhile, the magnetic dipole resonances have also been demonstrated at up to 532nm in planar split ring resonator (SRR) structure [35], which also proved useful for enhancing Raman signal in the graphene [15]. Interestingly, the realization of magnetic resonances in the deep visible spectrum remains challenging despite the fact that the fundamental kinetic inductance limitation has not yet occurred in such frequency range. This is mainly attributed to the aluminum interband transition (at 800 nm) that is known to impart red shifts of the resonance modes. Thus, the existing size dependence of split ring resonators [36], coupled with the need for compensating the red shift effects from the interband transition, has made the dimensional requirements of Al-based SRR more challenging than those based on other plasmonic metals. Other practical challenges include native surface oxidation of aluminum nanostructures, metal roughness, and most importantly cross-contamination during aluminum physical deposition step [34] that has been shown to suppress plasmonic oscillations. In this work, we attempt to address these challenges by fabricating aluminum SRR structures in the \textit{u}-shape and the \textit{v}-shape geometries, whose magnetic, electric, and magnetic-electric resonances all fall within the UV-Vis-NIR frequency spectrum. We discuss the mode excitations based on longitudinal and transverse plasmons, where the former corresponds to the well-known magnetic and electric modes in SRR, and the latter points to the unexplored hybrid magnetic-electric mode. In particular, we demonstrate that the preferential excitation of magnetic-electric mode over the fundamental magnetic mode becomes a limitation for the achievable magnetic resonances in aluminum SRR even before the fundamental kinetic inductance limitation sets in. Finally, we also discuss the interaction of these modes with aluminum interband transition, which results in the resonance splitting around 800 nm.

The \textit{u}-SRR and \textit{v}-SRR are schematically shown in Fig. 4a, with some of their smallest fabricated structures displayed in Fig. 4b. The experimentally measured transmissions of \textit{u}-SRRs under \textit{x} and \textit{y} incidence polarizations are presented in Fig. 5a and 5b, respectively. We show the FDTD calculations of the \textit{u}-SRR structures (indicated by the dashed lines), where we assume the presence of ~4-nm thick aluminum oxide layer.

\textbf{Figure 4.} Aluminum split ring resonators (SRR) on ITO glass substrate. (a) Schematic representations of \textit{u}-SRRs and \textit{v}-SRRs with lattice constant \textit{a}. (b) SEM images of the fabricated \textit{u}-SRRs [1st column, (1)-(3)] and \textit{v}-SRRs [2nd and 3rd column, (4)-(9)] with different resonator sizes (\textit{s}) and opening angles (\textit{θ}). The scale bar corresponds to 100 nm. The resonator sizes for the \textit{u}-SRRs are 100 nm (1), 80 nm (2), and 60 nm (3); while for the \textit{v}-SRRs are 100 nm (4, 7), 80 nm (5, 8), and 70 nm (6, 9). For all SRR structures, the lattice constant is designed to be two times the resonator size (\textit{a} = 2\textit{s}), and the feature width is 20–25 nm.
(Al₂O₃) on the SRR sidewalls. The fundamental and higher order magnetic modes are denoted by circles and triangles, respectively. As resonator size is decreased, it can be seen that the magnetic mode moves from ~1663 nm (s = 150 nm) to ~1271 nm (s = 70 nm), before interacting with the interband transition at s = 60 nm, giving split resonances at ~766 nm (m₀₁ mode) and ~1042 nm (m₀₂ mode). Meanwhile, at increasing resonator size, the higher order magnetic mode moves from ~452 nm (s = 60 nm) to ~530 nm (s = 70 nm) before interacting with the interband transition at s = 80 nm, resulting in split resonances at ~657 nm (m₁₁ mode) and ~961 nm (m₁₂ mode). The m₁₁ mode then moves asymptotically around 800 nm at larger sizes, while m₁₂ mode moves from ~961 nm (s = 80 nm) to ~1119 nm (s = 200 nm). The same observation is found under the y-polarization incidence (Fig. 5b), where the electric mode moves from ~558 nm (s = 60 nm) towards the interband transition, and results in resonance splitting at ~713 nm (e₀₁ mode) and ~943 nm (e₀₂ mode). The e₀₁ mode then follows the same asymptotic path around 800 nm, while the e₀₂ mode moves from ~943 nm (s = 80 nm) to ~1324 nm (s = 200 nm). As expected from the red shift from the aluminum interband transition, the achievable magnetic resonance frequencies remain lower than the gold and silver counterparts [13], [23], in spite of the higher free electron density in aluminum.

**Figure 5.** The resonance modes of aluminum u- SRR and their interactions with aluminum interband transition at λₕ = 800 nm. Near the interband transition, the fundamental magnetic mode (m₀) splits into m₀₁ and m₀₂ while the electric mode (e₀) splits into e₀₁ and e₀₂. The experimental (solid) and calculated (dashed) transmission spectra of u-SRRs on ITO glass for (a) x-polarization, s = 60 – 100 nm, w = 25 nm, and (b) y-polarization, s = 120 – 200 nm, w = 40 nm. The fundamental magnetic modes are indicated by filled (m₀₁) and hollow (m₀₂) circles, while the higher order magnetic modes are indicated by filled (m₁₁) and hollow (m₁₂) triangles. The electric modes are indicated by filled (e₀₁) and hollow (e₀₂) squares.
As the SRR size from our previous works is constrained to $s = 60$ nm [13], [23], a different SRR geometry is clearly needed to further push the magnetic frequency into the short visible wavelength spectrum. This is accomplished by a $v$-shape SRR (Fig. 4b), which we believe can effectively reduce the SRR area (or SRR inductance) within the same dimensional constraints. In addition, the magnetic resonance can be more flexibly tuned in the $v$-SRR by virtue of its opening angle ($\theta$) and size ($s$), as compared to that in the $u$-SRR which is tunable only by its resonator size ($s$). In another perspective, the tuning of resonance wavelength via the opening angle can also be understood as changing the SRR capacitance between SRR arms, which in turn leads to the tuning of SRR effective refractive index. [37] The experimental measurements are presented in Fig. 6, where the $v$-SRRs were fabricated by the same patterning process except for the 2-nm thick coating layer that was changed to titanium instead of nickel. This is because we found that titanium prevents native oxidation better than nickel. The experimental transmission spectra are shown in Fig. 6a and Fig. 6b for $x$ and $y$ polarizations, with their FDTD calculations denoted by dashed lines based on the same considerations as those in the $u$-SRR cases. The existence of $m_{01}$ (squares) and $m_{02}$ (circles) modes is again observed for $\theta = 90^\circ$ and $\theta = 60^\circ$ under the $x$ polarization, where these modes are going into and out of interband transition at decreasing resonator sizes. For $\theta = 90^\circ$, the $m_{01}$ moves from $\sim 781$ nm ($s = 100$ nm) to $\sim 654$ nm ($s = 70$ nm), while the $m_{02}$ moves from $\sim 1275$ nm ($s = 100$ nm) to $\sim 1035$ nm ($s = 80$ nm). Likewise, for $\theta = 60^\circ$, the $m_{01}$ moves from $\sim 764$ nm ($s = 100$ nm) to $\sim 533$ nm ($s = 70$ nm), while the $m_{02}$ moves from $\sim 1124$ nm ($s = 100$ nm) to $\sim 880$ nm ($s = 80$ nm). Meanwhile, the $m_{e0}$ mode (triangles) is observed around $\sim 400$ nm, which are more pronounced at decreasing resonator size for both opening angles. This lack of size dependence of $m_{e0}$ positions supports the hypothesis that this is indeed the hybrid magnetic-electric mode (which is also verified numerically from our FDTD simulations). For $\theta = 90^\circ$, the $m_{e0}$ positions are $\sim 445$ nm (for $s = 80$ nm) and $\sim 410$ nm (for $s = 70$ nm); while for $\theta = 60^\circ$, the $m_{e0}$ positions are at $\sim 437$ nm (for $s = 80$ nm) and $\sim 386$ nm (for $s = 70$ nm), which already enters the ultraviolet regime.
Figure 6. Mode excitation through longitudinal and transverse plasmons: (a) The $x$-polarized experimental (solid) and calculated (dashed) transmissions of $v$-SRRs of $\theta = 90^\circ$ and $\theta = 60^\circ$ for different sizes, where the split magnetic modes $m_{01}$ (circles), $m_{02}$ (squares), and the magnetic-electric mode $me_0$ (triangles) are all indicated; (b) The $y$-polarized experimental (solid) and calculated (dashed) transmissions of $v$-SRRs of $\theta = 90^\circ$ and $\theta = 60^\circ$ for different sizes, where the electric modes ($e_0$) are denoted by triangle markers.

For completeness, we also present the results of $v$-SRRs under the $y$ polarization incidence (Fig. 6b), which show the traces of electric modes (denoted by triangle markers) at decreasing SRR size. For $\theta = 90^\circ$, the $e_0$ mode moves from ~630 nm ($s = 100$ nm) to ~454 nm ($s = 70$ nm), while for $\theta = 60^\circ$ the $e_0$ mode moves from ~619 nm ($s = 100$ nm) to ~446 nm ($s = 70$ nm). There are also subtle transmission dips around ~300 nm which likely originate from transverse plasmons along the $y$-direction. However, the resonance magnitude is independent of the resonator size, indicating that such a resonance dip comes from isolated transverse plasmons in the $y$-direction. This is in contrast to the $me_0$ mode which originates from the interaction between transverse plasmons in the $x$-direction. We further reduced the $v$-SRR size by decreasing the SRR opening angle to $\theta = 30^\circ$ (for $s = 70$ nm), with 14 nm lateral offset between the SRR arms to minimize e-beam proximity effects. From SEM inspection, the gap separation and the feature width are 28 nm and 21 nm, respectively, which we believe mark the smallest fabricated aluminum split ring resonator so far. Their measurements are presented in Fig. 7, where for clarity the inverse transmission spectra are plotted in log scale. For $s = 80$ nm, the resonance positions are ~604 nm (for $m_{01}$ mode), ~481 nm (for $e_{01}$ mode), and ~425 nm (for $me_0$ mode); while for $s = 70$ nm, ~469 nm (for $m_{01}$ mode), ~429 nm (for $e_{01}$ mode), and ~376 nm (for $me_0$ mode). It is interesting to note that $m_{01}$ mode is progressively overshadowed by $me_0$ mode at decreasing SRR size, and that the $m_{01}$ almost entirely diminishes in $s = 70$ nm. This clearly shows $me_0$ is more preferred to $m_0$ in the short visible wavelength, and we believe such a preferential excitation could serve as the other limitation for the achievable fundamental magnetic mode apart from the well-known kinetic inductance limitation [25], [26].
As a follow up of our previous work which showed that incorporating fourfold rotationally symmetric lattice could improve the optical response under unpolarized light illumination [13], various resonator designs with different rotational symmetry have been studied. We further explored different degrees of inherent rotational symmetry and studied their role in optical performances in the context of sensing application. From three-fold ($C_3$), four-fold ($C_4$), five-fold ($C_5$), six-fold ($C_6$), and eight-fold ($C_8$) rotational symmetry, we found that optical performance gets progressively better as the rotational symmetry is increased (see Fig. 8). We observe an enhancement in reflection (and scattering) by as much as ~1.6 times as the rotational symmetry is increased from $n = 3$ to $n = 8$ for a fixed arm length and period. In addition, we found that such resonant enhancement is caused by the interplay among longitudinal plasmons that are in conductive coupling with each other. The optical spectrum and resonance behavior of these structures have been theoretically studied and experimentally characterized, showing the figure-of-merit (FOM) of these structures in relation to sensing application.

**Figure 7.** Preferential excitation of magnetic-electric mode in the ultraviolet spectrum. The magnetic, electric, and magnetic-electric modes of v-SRR ($\theta = 30^\circ$ with 14 nm lateral offset between the arms) for $s = 80$ nm and $s = 70$ nm. The insets show the fabricated v-SRR, where the scale bars correspond to 100 nm. The gap separation and feature width are 28 nm and 21 nm, respectively.
In terms of inter-resonator coupling, we have studied the evolution of resonance modes as the inter-resonator spacing is decreased. Of particular interest is the transition from isolated split ring resonator arrays in four-fold rotationally symmetric lattice ($C_4$ u-SRR) into a gammadion-type resonator with inherent fourfold rotational symmetry ($U_f$). Our numerical simulations show that as the inter-resonator gap decreases the magnetic mode ($m_0$) diminishes and the electric mode ($e_0$) split into two modes that eventually become hybrid magnetic-electric modes ($me_0$ and $me_1$). The first mode ($me_0$) is the longitudinal plasmons with magnetic characteristics, whose field distribution is qualitatively similar to that in $m_0$ mode for isolated SRR structure. On the other hand, the second mode ($me_1$) corresponds to higher order longitudinal plasmon.

Experimentally (Figure 9), we observe that $U_f$ structure exhibit much stronger scattering characteristics compared with isolated SRR ($C_4$ u-SRRs) of the same resonator size. The papers detailing these results are still in preparation.

Figure 8. The role of rotational symmetry in the resonance strength. The optical response increase progressively as the internal rotational symmetry changes from three-fold ($n = 3$) to $n = 8$ eight-fold ($n = 8$). The arm length ($s$) and periodicity ($p$) are $s = 100$ nm and $p = 300$ nm, respectively. The scale bars in all the insets represent 100 nm.

Figure 9. The effect of internal rotational symmetry in improving the optical response under unpolarized light illumination. The back scattering response of the Uf-SRR (red) and $C_4$ U-SRRs (blue) for $s = 80$ nm and $s = 60$ nm. Inset: the fabricated $C_4$ U-SRR and Uf-SRR. The scale bars represent 100 nm.
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References


3. List of publications and significant collaborations that resulted from your AOARD supported project:

(a) Papers published in peer-reviewed journals (all papers acknowledged AOARD’s support)


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