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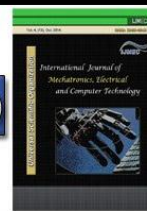
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## Optimization of Mid Infra-red Plasmonic Resonance for Sensor Applications

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### Abstract

Plasmonic resonances are strong candidates for applications in the fields of biomedical and environmental sensing. Previous related work has not investigated fully the optimization of resonant behaviour based on the various metallic metamaterial structure designs now available. The performance of structures that exhibit two distinct plasmonic resonances can be improved by varying the design parameters e.g. the period of metamaterial arrays and asymmetry in the basic elements. In addition, selection of an appropriate substrate for the metamaterial array should be considered for the production of plasmonic resonances in regions of the electromagnetic spectrum that are of particular interest. In the present work, we report work on optimization of the resonances produced by gold asymmetric split-ring resonators (A-SRRs) fabricated on fused silica and silicon-on-insulator (SOI) substrates. The specific technological aspects of using a SOI substrate are also highlighted.

**Keywords:** Plasmonic resonances, Metamaterial sensors, Mid infra-red region, Nano-fabrication, Computer simulation.

### 1. Introduction

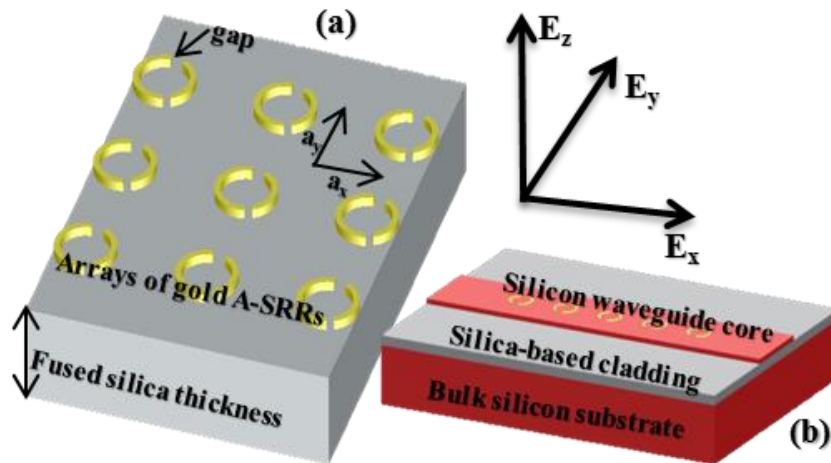
Metallic metamaterials are now widely used in sensor applications for biomedical and environmental monitoring. The metamaterial structures are formed from thin metallic resonators with dimensions much smaller than the operating wavelength. The geometric dimensions of the resonators can be tuned to provide plasmonic resonances at specific wavelengths that are matched to the molecular resonance of target molecules. Metamaterial sensors based on asymmetric Au resonators are promising devices that have been used to detect varieties of biochemical compounds, e.g. polymethyl methacrylate (PMMA), deoxyribonucleic acid (DNA), 17 $\beta$ -estradiol [1, 2, 3]. Plasmonic resonances produced by one type of metamaterial, an array of Au asymmetric split H-shape (ASH) structures, can enhance mid infra-red spectral features associated with a particular analyte [4]. It is essential that resonance peaks from metamaterial are distinct for efficient sensor applications in the mid infra-red as demonstrated with Au and Si patterns on fused silica and SOI substrates, respectively [1, 5]. In the present work, we have concentrated on the production of optimized plasmonic resonance peaks from an Au A-SRR based metamaterial.

## 2. Engineering design and Experiment

Arrays of Au A-SRRs were designed and fabricated on fused silica and SOI substrates, as shown in Figs. 1, 2 and 3.

### A. Engineering Design

Previous work has reported the design of A-SRR arrays with two distinct plasmonic resonance peaks and demonstrated the suitability of this design as a sensor for the detection of molecular resonances [1, 6, 7]. Production of distinct plasmonic resonance peaks is an important feature to achieve high sensitivity. Gold is one of the preferred metals for the thin film deposition on the designed patterns because of its high reflectivity features compared with other metals or dielectric materials. The work reported here investigates optimisation of the design of A-SRRs fabricated on a fused silica and also looks at the fabrication of the same design on a SOI substrate. The SOI substrate allows the A-SRR arrays to be integrated with optical waveguides providing the opportunity for a range of novel integrated photonic sensors to be investigated. Arrays of Au A-SRRs were designed and fabricated on a fused silica substrate using 100 nm thick Au and with an equal period ( $a$ ) in both  $a_x$ - and  $a_y$ -axes. The arrays are orthogonal to the propagation direction of the light along the  $z$ -axis as shown in Fig. 1a. Electric ( $E$ ) field in the three dimensions are for light incident normally on to the A-SRR arrays designed on fused silica while we proposed light coupling to Si waveguide for the design on SOI substrate. The periodic spacing of the A-SRRs was varied between 2.0  $\mu\text{m}$  to 2.8  $\mu\text{m}$  while the gaps between the arcs were varied from 55 nm to 175 nm. The outer diameter of the A-SRR is 1.0  $\mu\text{m}$  and the width of arcs is 100 nm. Similar A-SRR arrays were also designed on a SOI substrate, as shown in Fig. 1b, with the silicon waveguide core separated from the bulk silicon substrate by a thin silica-based cladding.

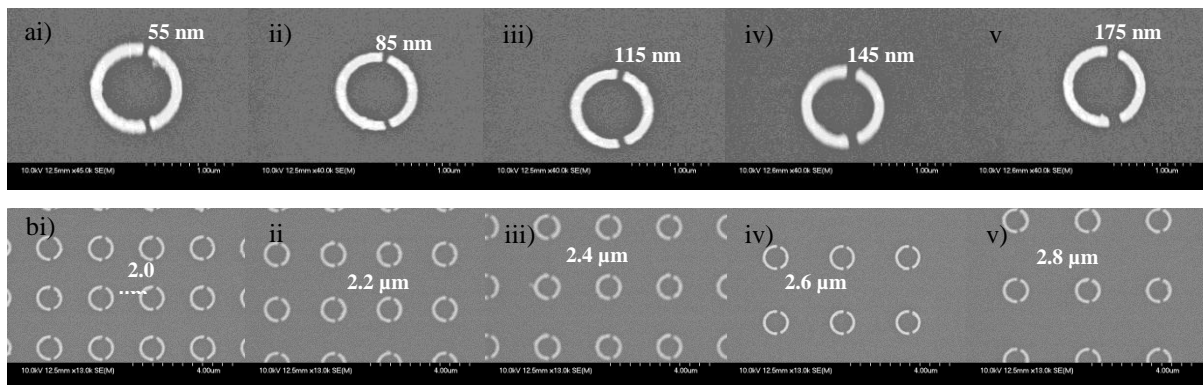


**Figure 1:** Schematic diagram of gold thin film A-SRRs on (a) fused silica (b) Silicon on Insulator (SOI) substrates

### B. Experimental

Arrays of A-SRR were fabricated using electron beam lithography on a 960  $\mu\text{m}$  thick fused silica substrate, and a SOI wafer which has 500 nm thick layers of Si core with lower silica-based cladding of 3  $\mu\text{m}$  and the bulk Si substrate. The optical properties of fused silica allow a broad range of transmission and SOI has the prospect of extending silicon devices into the integrated electronic and photonic circuit devices. Although, within the mid infra-red region the substrates are limited by strong  $\text{H}_2\text{O}$  absorption between 2.5  $\mu\text{m}$  to 2.8  $\mu\text{m}$  and  $\text{CO}_2$  at approximately 4.2  $\mu\text{m}$  as observed in the

reflectance measurements of Figs. 4 and 5. A background spectrum can compensate the atmospheric features that may change with several measurements. The reflectance of light from the fabricated Au A-SRR arrays as a function of wavelength is used to characterize the plasmonic resonance properties. A single layer of the Au A-SRR arrays was fabricated on a fused silica substrate following the same procedure in reference [1]. Arrays of A-SRR with gap of 55 nm, 85 nm, 115 nm, 145 nm, 175 nm and periodic spacing of 2.0  $\mu\text{m}$ , 2.2  $\mu\text{m}$ , 2.4  $\mu\text{m}$ , 2.6  $\mu\text{m}$ , 2.8  $\mu\text{m}$  were written using electron beam lithography tool in the James Watt Nanofabrication Centre (JWNC). The pattern was developed in isopropyl alcohol and methyl isobutyl ketone solutions, and 100 nm thickness of Au metal was evaporated on 10 nm Ti layer for adhesion, as shown in Fig. 2.

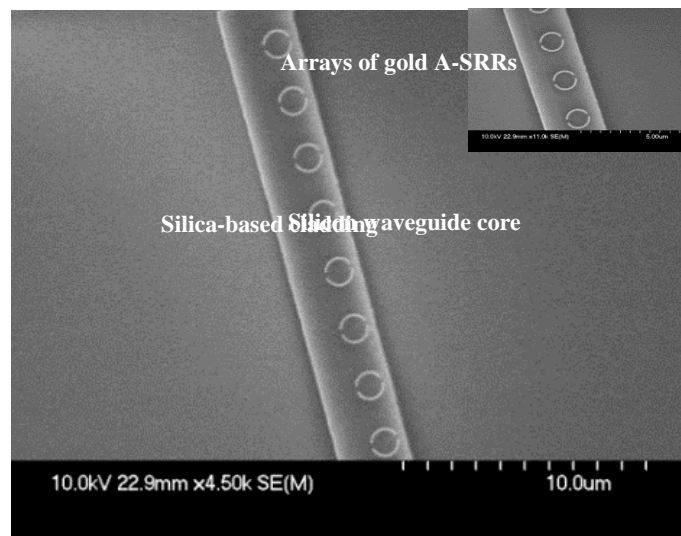


**Figure 2:** Scanning electron micrograph image of A-SRRs on fused silica substrate, a(i-v) Variation of gaps between the asymmetric rings b(i-v) periodic variation of arrays

Lift-off process was used to produce the Au A-SRR arrays pattern that have equal diameter of 1.0  $\mu\text{m}$ , arc width of 100 nm, and equal periodic spacing in x- and y-axes, as shown in Fig. 2. Through a rigorous experimental process of choosing suitable aspect ratio, the present work fabricated 55 nm gaps between two complementary geometries as shown in Fig. 2ai. Nanofabrication of gap smaller than the 55 nm could be difficult and not ideal for production of plasmonic resonance in the mid infrared region of the present work interest. Again, the choice of fabricating A-SRR arrays with periodic spacing from 2.0  $\mu\text{m}$  to 2.8  $\mu\text{m}$  as shown in Fig. 2b was considered suitable for optimizing the plasmonic resonance that will occur between wavelengths of 2  $\mu\text{m}$  to 6  $\mu\text{m}$ . Fabrication of A-SRR arrays with periodic spacing greater than 2.8  $\mu\text{m}$  for the present design will have plasmonic resonance of very low reflection magnitude as observed in the reflectance spectra of Fig. 4.

The electron beam lithography patterning of A-SRR arrays on the silicon waveguide of SOI was slightly more complicated during fabrication. This was because of the alignment of the A-SRR arrays at the centre of the Si waveguide width, as shown in Fig. 3. There were two main steps of electron beam lithography patterning required. We have used top-down approach, as it is most common in fabrication of nanostructures [8]. In the top-down technique, a mask protects the pattern and plasma, mechanical or chemical etching can be used to etch away the exposed material. In the present work, Polymethyl methacrylate resist was used firstly to write the topmost pattern, A-SRRs. After development and metallization of the A-SRRs, the sample was processed again for the second lithography. Hydrogen silsesquioxane (HSQ) resist was used for patterning the Si waveguide before dry etching was carried out. Surface Technology Systems (STS) was used for the plasma etching and high-density low pressure (HDLP) method of SOI etching was applied. The HDLP used was generated by excitation of inductively coupled plasma (ICP) on the STS equipment. Reactive ion etch employed utilizes the mix process, which exhibit no scalloping on etched sidewalls. The etching parameters were

set to a temperature of 23<sup>0</sup>C, chamber pressure of 10 mT, flow rate of 30 sccm and RF power level 300 W for 3.2 minutes, which was sufficient to etch through the 500 nm silicon core layer. The scanning electron micrograph (SEM) image in the Fig. 3 shows properly aligned A-SRRs on the Si waveguide core.



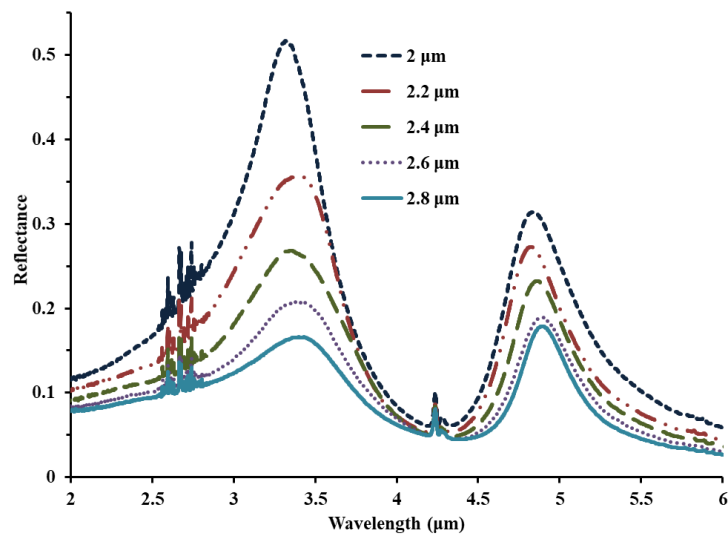
**Figure 3:** Scanning electron micrograph image of A-SRRs arrays on SOI substrates

### 3. Results and Discussion

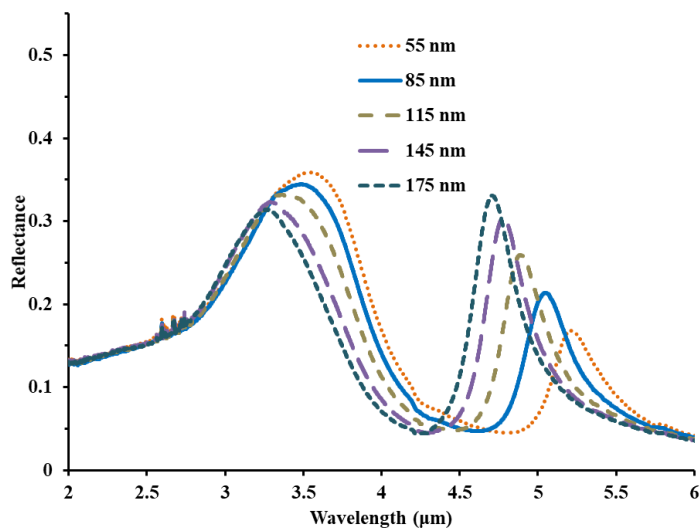
In some related publications, the importance of producing optimized plasmonic resonance peaks was observed [1, 6, 9, 13]. In the present work, we demonstrate the effect of the fused silica substrate on the plasmonic resonance peaks from the Au A-SRR arrays. Gold A-SRR can couple incident light to free electrons (plasmons) or phonons (phonon polaritons) but at a high cost of energy dissipation that leads to a low efficiency of the metamaterial device [10]. Metamaterial structures formed by only dielectric such as SOI, which have a higher refractive index than fused silica, have shown interesting results due to low non-radiative and Ohmic losses [10, 11]. We have experimentally shown in Fig. 3 a novel idea of combining both metal and a high refractive index dielectric. Our introductory work of A-SRR arrays on SOI substrate is a potential solution that will both activate use of surface-enhanced infra-red absorption (SEIRA) spectroscopy when illuminating wavelengths in the mid infra-red region and reduce losses resulting from the light incident normally on the A-SRR arrays design on fused silica substrate. Meanwhile, the periodic arrangement of metallic metamaterial arrays has been revisited, with interesting new results of a high quality factor of the plasmonic resonances in the EM spectrum [9, 12].

In the previous article, the A-SRR arrays periodic spacing have values that are less than the diameter of the designed arcs and the gaps between the split arcs are  $> 100$  nm [1]. Different periodic spacings and gaps were optimized to produce results, as shown in Fig. 4 and 5. A closed-packed periodic arrangement in an array of metallic metamaterial structures can produce the desirable combination of high peak reflectivity and strong resonance enhancement [12, 13]. Because of the significant coupling between individual arc elements in an array, the density of the A-SRR arrays determines the obtainable peak reflectivity, while controlling resonance bandwidth and the effective resonant spectral selectivity, as shown in Fig. 4. Measurements results from Fourier transform infrared (FTIR) spectrometer coupled to a microscope shows two distinct plasmonic peaks at shorter and longer wavelengths correspond to the small and large arcs of the designed A-SRR respectively, as shown in

Figs (4-5). The optimized double peaks were achieved by applying suitable period of A-SRR arrays from 2  $\mu\text{m}$  to 2.8  $\mu\text{m}$  in the interested mid infra-red region of 2  $\mu\text{m}$  to 6  $\mu\text{m}$ , as shown in Fig. 4.



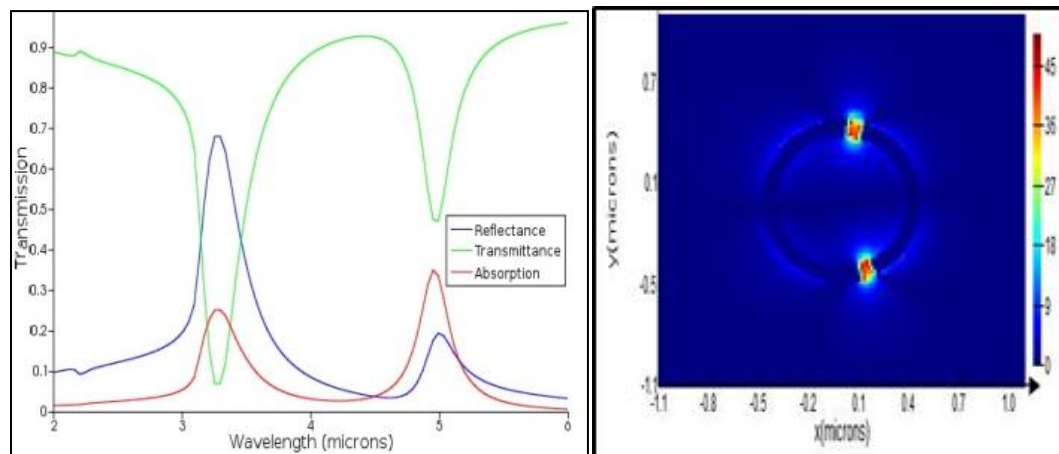
**Figure 4:** Reflectance spectra from FTIR measurements of A-SRRs arrays on a fused silica substrate for different periodic arrays



**Figure 5:** Reflectance spectra from FTIR measurements of A-SRRs arrays on a fused silica substrate for different gaps between the arcs

Fig. 5 shows resonance peaks at different wavelengths position due to the A-SRR gap variations: 55 nm ( $\theta^0 \sim 6.30$ ), 85 nm ( $\theta^0 \sim 9.70$ ), 115 nm ( $\theta^0 \sim 13.20$ ), 145 nm ( $\theta^0 \sim 16.60$ ) and 175 nm ( $\theta^0 \sim 20.10$ ) of reflectance spectra from FTIR measurements. The nanometre gap was calculated based on length of a sector ( $s$ ) equation;  $s = (\text{diameter} \times \pi \times \theta^0) / 360$ . The two gaps between the complementary asymmetric arcs were equally varied by simultaneously increasing the dimensions of both arcs, thus reducing the gap from 175 nm to 55 nm. As the gaps decreases the peaks of the reflectance spectra are redshifted, as shown in Fig. 5. The sequential redshift of the resonance peaks are attributed to high amount of gold as the dimension of the arcs were increase in order reduce the gaps. The tuning characteristics of metallic metamaterial were observed from the resultant plasmonic resonance. Ding et

al have published the idea of maintaining a constant nanometre gap, thereby demonstrating the effect of different asymmetric arcs on resonance peaks through computer simulations [14]. We also performed numerical simulations of the designed A-SRRs using Lumerical software. Lumerical is based on finite difference time domain technique of which the simulation region on the x- and y-axes for the A-SRR used periodicity condition. Fig. 6 shows the transmission spectra from simulating A-SRR design of 85 nm gap and periodic spacing of 2.2  $\mu\text{m}$ . There is observation of high field intensity at the 85 nm gap from the electric field distribution plots for the resonance peak wavelength position, as shown in Fig. 6. Nanometre gap together with sharp edges or tips of metallic metamaterial can give rise to hotspots which is important for sensors [14].



**Figure 6:** Transmission spectra and electric field magnitude plot of an A-SRR from computer simulation

#### 4. ACKNOWLEDGEMENTS

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#### CONCLUSION

We have demonstrated experimentally how variation of nanometre gap between the asymmetric split rings and A-SRR arrays periodic spacing can significantly support optimization of plasmonic resonances. Densely A-SRR arrays amplify the resonance magnitude and the smallest gap is redshifted mostly. The computer simulations for the 85 nm gap are in moderately good agreement with the reflectance peaks from the FTIR measurements. We have also briefly explained the novel idea of A-SRR arrays on SOI. The losses through the normal incident radiation can be minimize by coupling the light to Si waveguide core while the gold A-SRRs enhance the resonances. The sensor device can be extended to cover all EM spectrum region based on the smart geometrical properties of metamaterial. Applications in the areas of biomedical and environmental requiring spectra enhancement and analysis could benefit from the results of this work.

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