## Tuned permeability in terahertz split-ring resonators for devices and sensors

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A process is demonstrated for tuning the magnetic resonance frequency of a fixed split-ring resonator array, by way of adding material near the split-ring elements. Applying drops of a silicon-nanospheres/ethanol solution to the surface of the sample decreases the magnetic resonance frequency of the split-ring array in incremental steps of 0.03 THz. This fine tuning is done post fabrication and is demonstrated to be reversible. The exhibited sensitivity of the split-ring resonance frequency to the presence of silicon nanospheres also suggests further application possibilities as a sensor device. © 2007 American Institute of Physics. [DOI: 10.1063/1.2768300]

Device design is quickly becoming a large part of metamaterial research. In the short half decade since its conception, understanding of the physics behind tailored electromagnetic responses in metamaterials has progressed far enough to where application demonstrations are surfacing. Prime examples include diffraction beating lenses,<sup>1,2</sup> fre-quency selective cloaks,<sup>3-5</sup> advanced optics,<sup>6,7</sup> and improved radomes.<sup>8</sup> Many of these devices have been demonstrated at the microwave frequencies, aided by the comparable ease of fabricating millimeter scale structures. Operation at higher frequencies-infrared and above-requires smaller structures which are usually patterned photolithographically.9,10 The photolithographic step is often a hurdle to creating infrared devices, as it requires designing and purchasing an expensive lithography mask, which then permanently fixes the metamaterial structures. To make matters more difficult, design methods rely on finite element or similar numerical solvers; so even the best efforts in design can incorrectly predict the resonant response frequency of a structure by 5%.

Given all these considerations, it is clear that there is significant utility in developing the ability to alter the response of any metamaterial structure without the need to design a new lithography mask. Active tuning based on modification of the electromagnetic properties of materials is one option being pursued,<sup>11</sup> with clear advantages for switchable devices.<sup>12</sup> Despite advantages, the architecture of initial active-tunable devices has been quite complicated, and the tunable frequency range limited. In this letter, we demonstrate a simple procedure for passive tuning of a split-ring resonator (SRR) response. Although the tuning process is certainly not limited to SRR's, we consider them exclusively here for simplicity and familiarity.<sup>13</sup> We use the same SRR array as in the work of Driscoll et al.<sup>13</sup> wherein gold SRR's are patterned on a 1 mm thick silicon substrate coated with a thin 6  $\mu$ m layer of benzocyclobutane (BCB). The BCB is used as a low-loss adhesive aid in performing the gold lithography. A photograph of this SRR array is shown in Fig. 1(a). This metamaterial is designed to have a magnetic resonance at 1.20 THz, which experimentally manifests as a sharp dip in sample transmission, shown in Fig. 2 as the black curve. Transmission is probed in a Fourier transform infrared spectrometer using s-polarized 45° incident light (see inset in Fig. 3). The details of this style of measurement and a more indepth discussion of how this dip is known to be the magnetic mode of the SRR can be found in Refs. 13 and 14. One improvement on the method in Ref. 13 is explored in this work: sample transmission spectra exhibit large Fabry-Pérot fringes, which arise from multiple reflections within the thick 1 mm silicon substrate. These fringes make precise determination of the resonance frequency difficult. The fringes have a known periodicity which is set by the thickness of the substrate, so in our analysis, we pass the transmission spectra through a digital notch filter which removes the fringes. Because the notch filter is sharply targeted at the substrate fringes, no other features of the spectra are significantly affected.

Passive tuning in this letter is accomplished by adding dielectric material to alter the capacitance of the SRR. A simplified model of the SRR can be thought of as an inductive-capacitive circuit element.<sup>15,16</sup> The self-inductance of each loop battles the capacitance of the split gap to determine the resonance frequency (and all other attributes of the resonance) following

$$\omega_0 \propto \frac{1}{\sqrt{LC}} = \frac{1}{\sqrt{L}\sqrt{\varepsilon_0} \int^v \varepsilon(v)E(v)dv}}.$$
 (1)

The electric field falls off quickly away from the metamaterial layer so the volume integral need only be considered in the vicinity of the SRR. Cross sections of the electric field distribution for our SRR are displayed in Fig. 1(g), as solved by finite-integration time-domain solver. The high intensity of the electric field E(v) in the gap region makes the resonance frequency sensitive to small changes in the dielectric  $\varepsilon$ . To take advantage of this, we prepare a 0.2% solution of silicon nanospheres (50 nm diameter, prepared by weight), suspended in ethanol using an ultrasonicator. The solution is then applied to the surface of our sample in small drops of 30  $\mu$ l (average drop volume as measured by weight). The

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FIG. 1. (Color online) Photographs of the gradual addition of silicon nanospheres by solution. Panel (f) shows the sample after removing most of the silicon by ultrasonics. Panel (g) shows cross sections of the electric field intensity (solved by finite integration time domain). The scale is logorithmic: green is the incident field intensity and red is 100 times incident.

sample is heated to 60 °C, so shortly upon contact with the sample the ethanol evaporates leaving behind only the silicon nanospheres. This procedure produces a fairly uniform layer (see Ref. 17). Figure 1(b) shows a photograph of the SRR array after this first application. The introduction of the silicon material onto the SRR effectively increases the capacitance and thus decreases the resonant frequency, and we observe that the transmission dip decreases by  $\sim 0.05$  THz (Fig. 2, blue curve). Repeated applications of 30  $\mu$ l drops of this solution deposit additional silicon spheres [pictured in Figs. 2(c)-2(e), which continue to decrease the resonance in steps. Saturation of this effect begins by picture/line f, and further applications reduce the resonance frequency in diminishing increments. A weaker solution of nanospheres could presumably give even finer step sizes, and an exact resonance frequency can be created via a guess and check methodology. Another interesting effect of the silicon addition is an observed sharpening and deepening of the magnetic resonance dip. This is qualitatively sensible, as the Qfactor of a parallel RLC circuit should increase with increasing capacitance. This sharpening is also an indicator that addition of the silicon nanospheres does not appreciably add



FIG. 2. (Color online) Fine tuning: addition of silicon nanospheres increases the average dielectric of the SRR capacitance and shifts the magnetic resonant frequency downwards. Thick gray (line f) shows near restoration of the original response by removal of the nanospheres in an ultrasonicator.

to the damping of the SRR resonator (the nanospheres are nearly intrinsic silicon and so have a very small imaginary permittivity at tetrahertz).

The accumulation of the silicon spheres is also reversible. We briefly (30 s) submerge the sample in an ultrasonicated ethanol bath, and most of the silicon is removed [Figure 2(f)]. The resonance frequency also returns nearly to its original position. Not entirely all of the spheres come off. We see that the spheres clustered near the edges of the SRR's remain. This reversibility is an important attribute; if the desired resonant frequency is overshot, we can reset and begin the application process anew.

The observed sensitivity of the SRR metamaterial resonance frequency to the introduction of microliters of our weak solution of silicon nanoparticles also brings to mind the



FIG. 3. (Color online) Coarse tuning: three values of the BCB spacer thickness showing thinner spacers decrease the SRR magnetic resonance position

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possibility of using such devices as sensors. The 30  $\mu$ l drop contains an amount of silicon so small (less than 1 ng), that we cannot detect it via standard transmission-amplitude experiment.<sup>18</sup> The silicon layer which we add to the SRR's is many times thinner than the skin depth of tetrahertz radiation. Using our SRR array as a probe offers a significant advantage as we transform detection of the silicon nanospheres from a transmission-amplitude level measurement to a resonance frequency position measurement, which is often much more accurate.<sup>19</sup> The resonance frequency of the SRR is sensitive to very small quantities of material as the concentrated fields within the split gap most strongly interrogate a volume of only  $(\sim 3 \ \mu m)^3$ . The field concentration increases with increasing Q factors, and for SRR's the Q is generally around 10. A cleverly designed metamaterial sensor with high Q might thus be able to detect quantities of material even several orders of magnitude less than that demonstrated in this letter, and we are in the process of designing and investigating such devices.

Fine tuning by addition of silicon nanospheres gives control over the resonance frequency within a change of  $\sim 10\%$ , as we see in Fig. 2. We can effectively extend this tuning range by also varying the thickness of the BCB spacer used. Since BCB has a lower dielectric than silicon, a thinner BCB layer lets more of the SRR fields penetrate the silicon substrate, and lowers the resonance frequency. Figure 3 demonstrates this, showing that using a thinner BCB layer creates a lower resonance frequency, acting as a method for coarse tuning. Imprecision in the BCB spin-coating process limits this coarse tuning to an accuracy of 5%-10%, which meshes nicely with our fine tuning. By combination of the coarse and fine tuning methods, we can feasibly create a metamaterial with a precise magnetic resonance frequency anywhere over the octave from 0.70 to 1.20 THz (all without needing to change the feature sizes). Combined with the possibility of sensors which probe subwavelength volumes, the sensitivity of metamaterials to passive material inclusions holds potential for interesting designs and applications.

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