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Accurate Measurement of THz Dielectric Constant Using Metamaterials on a Quartz Substrate

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We present dielectric constant measurements of thin films using THz metamaterials fabricated on a quartz substrate. The resonance shifts of the metamaterials exhibit saturation behavior with increasing film thickness. The saturation frequency shift varies with the real part of the dielectric constant, from which the numerical expression for the particular metamaterial design was extracted. We first performed finite-difference time-domain simulations to find an explicit relationship between the saturated frequency shift and the dielectric constant of a thin film, which was confirmed by the experimental results from conventional techniques. In particular, the quartz substrate enables us to determine their values more accurately, because of its low substrate index. As a result, we extracted the dielectric constants of various films whose values have not been addressed previously without precise control of the film thickness.

Keywords : Terahertz spectroscopy, Metamaterials, Sensor
OCIS codes : (300.6495) Spectroscopy, terahertz; (160.3918) Metamaterials; (280.1415) Biological sensing and sensors

I. INTRODUCTION

Metamaterials have been received great attention in the last decade and become an active research field, due to their interesting properties such as cloaking, negative refraction, superlensing, phase modulation, perfect absorption, and sensing [1-6]. A metamaterial can be considered as an equivalent circuit when a strong surface current occurs in the structure by interacting with incident light. Its resonant properties can be tuned by controlling geometrical and material parameters, such as gap width, metal thickness, and substrate index [7-10]. In particular, the incident light can couple to the *LC* resonance of a metamaterial described by $f_0 = 1/(2\pi\sqrt{LC})$, where *C* is the capacitance of the gap structure and *L* is the inductance of the side-arm structure [10]. It is obvious from this relation that the *LC* resonant frequency is strongly dependent on the effective dielectric constant of the gap structure. Therefore, when a dielectric material is located in the gap area, the resonant-frequency shift Δf occurs due to the effective index change in the gap area [3, 11, 12].

On the other hand, terahertz time-domain spectroscopy (THz-TDS) has been considered as a competitive technique for detection and inspection of target materials, because it enables label-free, noncontact, nondestructive detection [13-16]. However, it is difficult to address the dielectric properties of these materials especially when they are transparent against THz waves, because a large quantity is needed to measure dielectric characteristics [3]. For instance, film thicknesses of more than ~200 μm were required to measure the dielectric constants of microbial substances such as penicillium and yeasts. Therefore, it is necessary to find an alternative way to address their dielectric properties in the THz frequency region.

Recently, we have shown that the dielectric constants of thin films and polar liquids can be measured directly using THz metamaterials, without the need to prepare thick films [17]. This was possible because the effective sensing volume of a THz metamaterial is highly localized near the surface [18]. The vertical extent of the sensing volume has been

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estimated as 3~4 μm, and the resonant-frequency shift saturates at a specific thickness (~10 μm) of target material deposited on the THz metamaterial [7, 18]. In particular, in our previous work we extracted the numerical expression relating the saturation value of the resonant-frequency shift Δf_{sat} and the dielectric constant of target material by using THz metamaterials fabricated on a Si substrate [17]. Metamaterial sensing has proven to be an effective method for finding the dielectric constants of thin films; however, it has not been applied to address various materials of practical importance in THz applications. In addition, the Si substrate has a relatively high index, which restricts accurate measurement of the dielectric properties of target materials [7].

In this paper, we measure the dielectric constants of various thin films using THz metamaterials fabricated on a quartz substrate with relatively low dielectric constant. We first performed finite-difference time-domain (FDTD) simulations to extract the numerical expression relating saturated frequency shift and dielectric constant of target materials for the particular THz metamaterials. Metamaterial sensing based on the low substrate index allows us to address very accurately the dielectric properties of thin films whose dielectric constants have not been reported before.

II. SAMPLE PREPARATION AND EXPERIMENTAL METHODS

The transmission amplitudes of the THz-metamaterial devices were obtained from a conventional THz-TDS system [17, 19, 20]. A linearly polarized THz pulse was generated from a photoconductive antenna by illumination with a femtosecond laser at $\lambda = 800$ nm. The THz pulse was focused on the THz metamaterial with ~1 mm^2 focusing area, under ambient conditions. The amplitude and phase of the transmitted THz electric field in time traces were obtained by changing the time delay between the THz pulse and the probe beam. We could obtain the THz spectrum by solving a fast Fourier transform for the transmitted THz electric field in the time traces.

We fabricated the THz metamaterial on quartz substrates (1 mm thick) by employing a conventional photolithography technique, followed by metal evaporation of Cr/Au (2 nm/98 nm). The THz metamaterial consisted of a 40 × 40 array of electrical split-ring resonators with a side-arm length of 36 μm, line width of 4 μm, gap width of 3 μm, and periodicity of 50 μm [1]. A schematic of the dielectric constant measurement of thin films using THz metamaterial is shown in Fig. 1(a). We measured the THz transmission amplitude of the THz metamaterial before and after deposition of polymer layers, to extract the dielectric constant of thin films from the saturated frequency shift. Figure 1(b) shows a picture of a polymer layer (GXR-601, AZ Electronic Materials Inc.) deposited on the THz-metamaterial device. We prepared various polymer films such as polydimethylsiloxane (PDMS), poly (methyl methacrylate) (PMMA) (MicroChem Inc.), GXR-601 (AZ Electronic Materials Inc.), SU-8 2002 (MicroChem Inc.), Ma-P 1210 (MicroChem Inc.), Ma-N 2405 (MicroChem Inc.), and polyimide (PI). We poured the solution containing the polymers into a PDMS well to fabricate films with uniform thickness of ~40 μm, which is well above the saturation thickness (typically less than 10 μm) in metamaterial sensing. This allows us to measure the dielectric constants accurately, without precise control of the film thickness.

III. RESULTS AND DISCUSSION

We begin by finding the relationship between Δf_{sat} and the dielectric constant for our devices (*i.e.* metamaterial on quartz substrate) by using FDTD simulation. Here, we modeled the THz-metamaterial devices using a Lumerical FDTD simulation with a linearly polarized plane wave under periodic boundary conditions. To mimic our measurements, the same geometrical factors as in the experiments were used. We considered the metal film as a perfect electrical conductor. The smallest mesh size was 500 nm, and the FDTD simulation took 15 minutes. Figure 2(a) shows the THz transmission amplitude of the metamaterial, before and after the deposition of a dielectric layer with dielectric

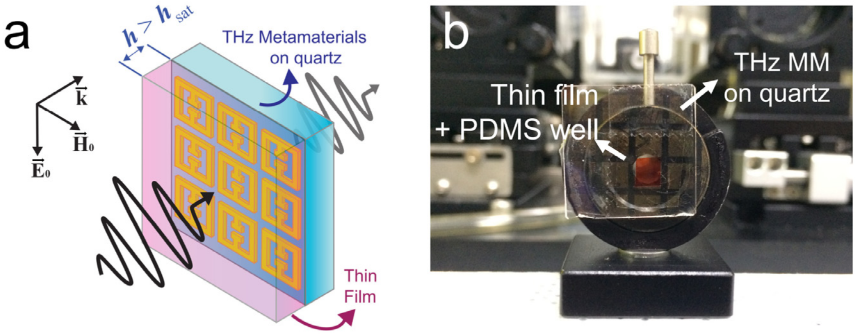
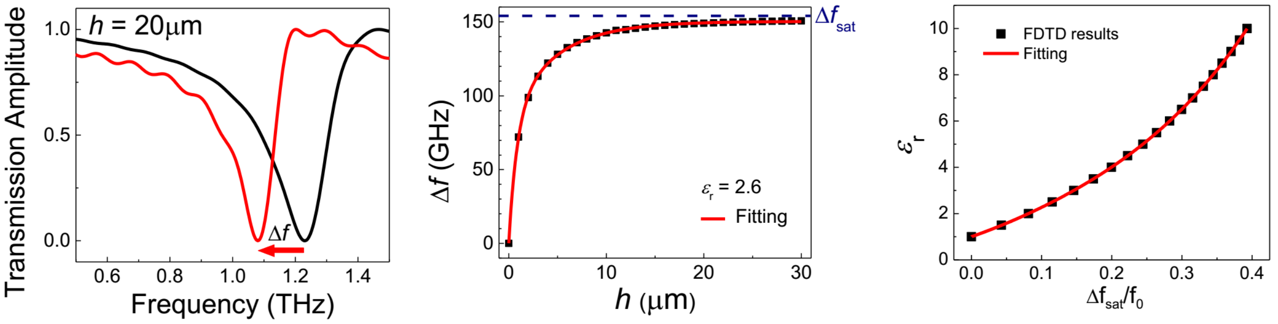


FIG. 1. (a) A schematic diagram of dielectric-constant measurement of a thin film using terahertz metamaterial on a quartz substrate. (b) A picture of a polymer film (GXR 601) deposited on a THz-metamaterial device.



(a) (b) (c)

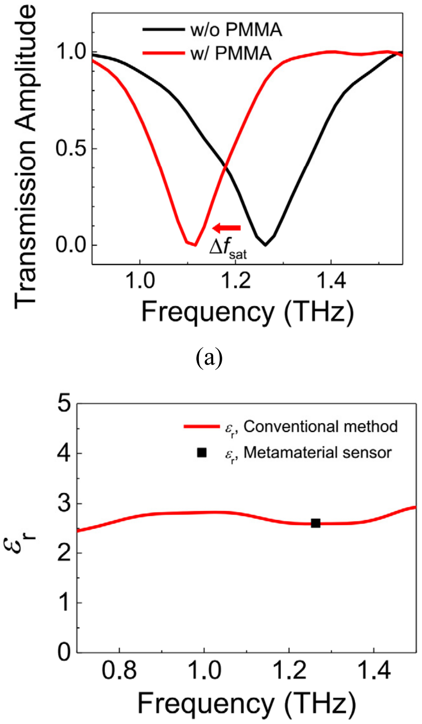
FIG. 2. (a) FDTD simulation results for the THz transmission spectra of metamaterial with (red line) and without (black line) the deposition of dielectric film ($\varepsilon_r = 2.6$) of thickness 20 μm. (b) Δf as a function of the thickness of the dielectric film ($\varepsilon_r = 2.6$). (c) $\Delta f_{sat}/\Delta f_0$ as a function of ε_r with nineteen different ε_r's from 1 to 10 (black boxes) and fitting results (red solid line).

constant ε_r of 2.6. We can clearly observe that the *LC* resonance shifts to lower frequency, because the dielectric film causes a change in the effective dielectric constant in the gap area of the THz metamaterial. Δf can be explained by the following relationship: $\Delta f/f_0 \approx \alpha(\varepsilon_r - \varepsilon_{air})/\varepsilon_{eff}$ [3, 17], where α is the sensitivity coefficient, ε_{air} is the dielectric constant of air, and ε_{eff} ($= n_{eff}^{\ 2}$) is the effective dielectric constant without the dielectric film coating.

On the other hand, Δf goes higher as we increase the film thickness, until it saturates at a specific thickness, as shown in Fig. 2(b). As mentioned, we have shown that the detection volume of THz metamaterial is highly confined to the surface [18], so we can extract the dielectric constant of a film from Δf_{sat} when the film thickness is thick enough (~30 μm). In Fig. 2(c) we plot $\Delta f_{sat}/\Delta f_0$ as a function of ε_r with nineteen different ε_r's from 1 to 10, obtaining a simple relationship between $\Delta f_{sat}/\Delta f_0$ and ε_r by fitting with a polynomial function: $\varepsilon_r = 1 + \eta_1(\Delta f_{sat}/f_0) + \eta_2(\Delta f_{sat}/f_0)^2 + \eta_3(\Delta f_{sat}/f_0)^3 + \eta_4(\Delta f_{sat}/f_0)^4$, which yields $\eta_1 = 10.6$, $\eta_2 = 21.5$, $\eta_3 = -18.4$, and $\eta_4 = 110$. From this relationship we can easily extract ε_r values from $\Delta f_{sat}/\Delta f_0$, for various thin layers. We also note that the nonlinear function has been introduced to fit the curve accurately over the broad frequency range, whereas a simple linear function was adopted in the previous case of the Si substrate [17].

To confirm the validity of our approach for thin polymer films, in Fig. 3 we compare the dielectric constant of PMMA film extracted using our method to that obtained by conventional THz transmission measurement [3]. Figure 3(a) shows the THz transmission amplitude of the THz metamaterial before and after deposition of PMMA layers. We coated the metamaterial with a PMMA layer (40 μm thick) to obtain $\Delta f_{sat}/\Delta f_0$. ε_r extracted from metamaterial sensing (black boxes) is shown together with ε_r obtained from conventional THz transmission measurement (red solid line) in Fig. 3(b) [17]. We dried the PMMA layer on a hotplate at 110°C for 20 min to prevent humidity from affecting the measured dielectric constant. From the metamaterial sensor we obtained an ε_r of 2.61 at 1.26 THz, which is in good agreement with that obtained by the conventional method. These results validate our approach, in which the dielectric constants of various thin films can be found effectively, without the need to prepare thick films [18].

Finally, we extracted ε_r for 7 different materials, including PMMA. These materials have potential use in future THz



(a)

(b)

FIG. 3. (a) Normalized THz transmission spectra of metamaterial with (red line) and without (black line) deposition of PMMA film of thickness 40 μm. (b) Dielectric constants obtained from metamaterial sensing (black box) and from conventional THz transmission measurement (red solid line) for the PMMA film.

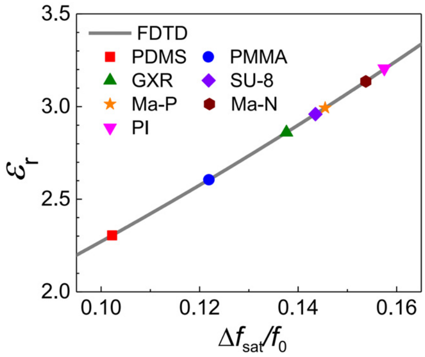


FIG. 4. Dielectric constants of various thin films extracted from THz metamaterial sensing.

applications, such as flexible substrates and composite host materials. Again, all polymer layers were dried on a hotplate at 110°C for 20 min. Although the study of the dielectric properties of these polymers is crucial, their dielectric constants have been largely unexplored. As summarized in Fig. 4, we obtained dielectric constants of 2.30, 2.61, 2.86, 2.96, 2.99, 3.14, and 3.25 for PDMS, PMMA, GXR-601, SU-8 2002, Ma-P 1210, Ma-N 2405, and PI respectively. The dielectric constants of PMMA, SU-8, and PI have been addressed in our previous work on the Si substrate, whereas those of the other materials have not been addressed previously in the THz frequency range. As a result, we found that the Ma-N and PI can be useful when a high substrate index is required, whereas PDMS is a good candidate when low substrate index is preferred. We also point out that the metamaterial fabricated on the quartz substrate is better in terms of sensitivity, because it has a low substrate index compared to the Si case. The sensitivity of THz metamaterial on the quartz substrate in terms of refractive index unit (RIU) is found to be 250 GHz/RIU, which is three times as high as that on a Si substrate (81 GHz/RIU) [17]. Therefore, by using a quartz substrate, we can achieve an accuracy three times as high as in the Si case.

IV. CONCLUSION

To conclude, we have shown that the dielectric constant of a thin film can be explicitly obtained from the saturated frequency shift of a THz metamaterial. We performed FDTD simulations to get a numerical expression relating the dielectric constant and the saturated values for devices fabricated on quartz. The dielectric constant extracted from our technique agreed well with that measured in conventional THz transmission experiments. As a result, we were able to address the dielectric constants of various thin films that had not been addressed before. We found that PI and Ma-N have relatively high indices, greater than 3, whereas PDMS has a low value of 2.3. In particular, the dielectric constant can be obtained without needing to prepare a large amount of target material, and also without precise control of the thickness. Importantly, the use of the quartz substrate with a lower index makes it possible to improve the accuracy by more than a factor of three, relative to the Si case. Dielectric measurement based on metamaterial sensing can be applied to other types of materials, including chemical and biological samples, and this information will play a crucial role in finding future applications in the THz frequency range.

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