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Nanoparticles doped film sensing based on terahertz metamaterials

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ABSTRACT

A nanoparticles concentration sensor based on doped film and terahertz (THz) metamaterial has been proposed. By coating the nanoparticles doped polyvinyl alcohol (PVA) film on the surface of THz metamaterial, the effects of nanoparticle concentration on the metamaterial resonances are investigated through experiments and numerical simulations. Results show that resonant frequency of the metamaterial linearly decreases with the increment of doping concentration. Furthermore, numerical simulations illustrate that the redshift of resonance results from the changes of refractive index of the doped film. The concentration sensitivity of this sensor is 3.12 GHz/0.1%, and the refractive index sensitivity reaches 53.33 GHz/RIU. This work provides a non-contact, nondestructive and sensitive method for the detection of nanoparticles concentration and brings out a new application on THz film metamaterial sensing.

1. Introduction

For its unique advantages, terahertz (THz) technology has promising application potentials in wireless communication [1], radar [2], imaging [3], security [4], especially in the spectrum detection and sensing [5,6]. Many important spectral information are located in the THz regime, for instance, the rotational and vibrational modes of molecules [7], band gap and inner band level of superconductors [8] and semiconductors [9]. For the lack of natural materials which can generate electro-magnetic response with THz wave, researchers have come up with artificially designed metamaterials [10–12]. By designing their sub-wavelength structures, metamaterials have been demonstrated to have lots of special characteristics [13–18], such as negative refraction, artificial resonance, extraordinary transmission and perfect absorption. Besides, the resonances of metamaterials are highly sensitive to the surrounding dielectric properties, making them ideal for THz sensing [19–21].

A series of researches have been made in THz sensing during the last few years [22–28]. Reinhard et al. [22] reported a metamaterial-based THz sensor for thickness measurements of subwavelength-thin materials and refractometry of liquids and liquid mixtures. Fan et al. proposed a real-time quantitative microfluidic amount sensor [23] and a THz refractive index sensor for liquids [24] based on photonic crystal column array filled with micro fluids. Wang et al. [25] mentioned a sensitive refractive index sensor based on THz metamaterial absorber which may be used for detecting flammable gases and poisonous materials. It can be seen that current THz sensing studies are mostly based on the sensitivity of the electromagnetic response characteristics of microstructures such as gratings, photonic crystals, metamaterials and metasurfaces to their surrounding dielectric materials. However, these sensing techniques are primarily applicable to gas or liquid materials which can be conveniently filled into the microstructures. Nevertheless, many powdery nanoparticles are difficult to adhere to the surfaces of these microstructure sensors, making them unreliable for precise control and quantitative detection. Moreover, the required sample amount is immensely large, which increases the test expenses since the nanoparticles are usually expensive. Therefore, a high-precision and non-contact sensing method for nanoparticles is in great demand to solve these problems.

In this work, a nanoparticles doped film sensing method based on THz metamaterial has been demonstrated through experiments and numerical simulations. Polyvinyl alcohol (PVA) films doped with different concentrations of magnetic nanoparticles were spun and coated onto the surface of THz metamaterial. Electromagnetic response spectra show

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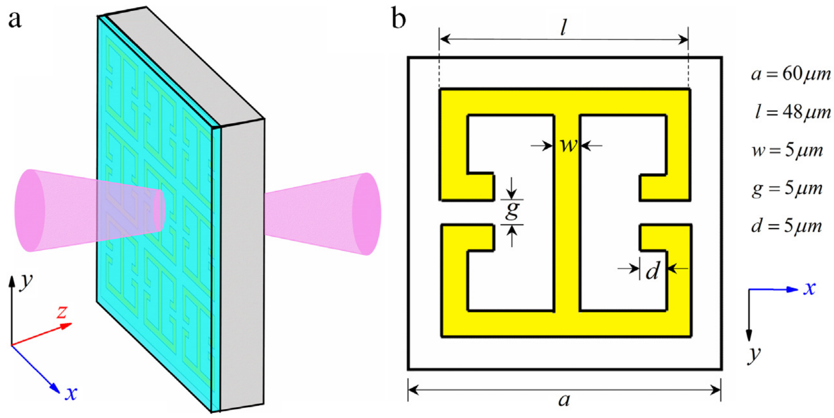

Fig. 1. (a) 3D Schematic of the film sensor. The gray part is the silicon substrate, the yellow part is the gold microstructure, and the blue part is the nanoparticles doped film. THz wave vector is parallel to the z-axis; (b) Metamaterial unit structure and its geometry.

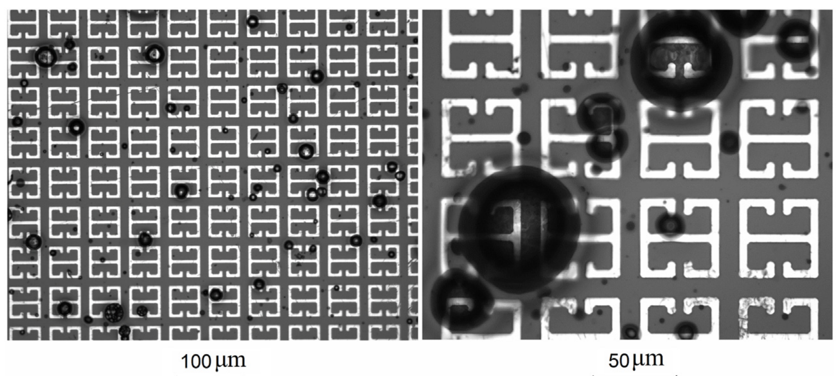

Fig. 2. Micrographs of the THz metamaterial with 0.16% nanoparticles doped PVA film.

that the resonance frequency linearly decreases with the increment of doping concentration of nanoparticles, illuminating the realization of concentration sensing for nanoparticles in the THz regime. The concentration sensing sensitivity reaches 3.12 GHz/0.1%, and the refractive index sensitivity reaches 53.33 GHz/RIU.

2. Experimental system and samples preparation

The schematic of the experiments is shown in Fig. 1(a). All the experiments were carried out in a terahertz time-domain spectroscopy (THz–TDS) system [21] at temperature of 23 °C and humidity of 10%. A 75fs, 800 nm Mai Tai titanium-doped sapphire femtosecond laser was applied in the system and the femtosecond beam was divided into two paths. One illuminated the low-temperature grown GaAs photoconductive antenna to generate THz wave, and the other illuminated ZnTe crystal to detect THz wave. Experimental metamaterial with 200 nm periodic gold microstructures were fabricated on a 500 μm high-resistivity silicon substrate, and a PVA film doped with magnetic nanoparticles was coated on the surface of metamaterial. The unit cell structure of the metamaterial is shown in Fig. 1(b), where a = 60 μm, l = 48 μm, $w = g = d$ = 5 μm.

First, we fabricated the metamaterial chips by lithography, and then, prepared PVA solution containing different concentrations of magnetic nanoparticles. Fe_3O_4 nanoparticles and PVA particles were added into deionized water and the mixed solution was heated at 95 °C in a water bath with magnetic stirring. The rotational speed of magnetic rotor was set to be 40 r/min for two hours. After mixed and dissolved uniformly, the nanoparticles doped PVA solution was obtained. We prepared five different PVA solutions with doping concentrations of 0% (pure PVA), 0.16%, 0.32%, 0.48%, and 0.80%. At last, the doped PVA solution was coated on the surface of the metamaterial to be a film by a spin coater. The maximum rotational speed was set to be 12 000 r/min to ensure uniformity of the film. Eventually, 5 groups of samples with different doping concentrations and a group of bare metamaterials without PVA film as the reference sample were obtained. In addition, there were six pieces for each group sample to ensure measurement accuracy by averaging. The film thicknesses of the five samples were all measured to be 5.5 μm by a profiler. The micrographs of 0.16% sample are shown in Fig. 2. We can see that the magnetic nanoparticles form magnetic clusters of micrometers size, and the nanoparticles accumulate in a large number at the boundary of the clusters. This phenomenon is caused by the mixture of magnetic nanoparticles with PVA and the formation of the solid film.

3. Results and discussion

Before investigating the sensing properties of doped films, the electromagnetic response of the reference sample was studied first. The experimental time-domain signals of air and reference sample with x-polarization (TE mode) and y-polarization (TM mode) are shown in Fig. 3(a). Compared with air signal, only partial electric field strength passes through the reference sample, and a phase delay occurs. By performing Fourier transform of the time-domain signals, the amplitude spectra of the reference samples $|E_r(\omega)|$ and air $|E_a(\omega)|$ are obtained, thus the transmission intensity of reference samples can be calculated by:

$$T = |E_r(\omega)| / |E_a(\omega)|. \tag{1}$$

Transmission spectra are shown in Fig. 3(b), which exhibits a −26 dB resonance at 0.825 THz when the incident THz wave is x-polarized, and a −11 dB resonance at 0.387 THz when it is y-polarized.