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Terahertz transmission properties of vanadium dioxide films deposited on gold grating structure with different periods

Min Gao1,2, Xu Wang1, Shengxian Luo1, Qingjian Lu1, Sheng-Nian Luo2*, Chang Lu1, Sihong Chen1, Fei Long3 and Yuan Lin1,4

1 State Key Laboratory of Electronic Thin Films and Integrated Devices, University of Electronic Science and Technology of China, Chengdu 610054, People’s Republic of China
2 The Peac Institute of Multiscale Sciences, Chengdu 610207, People’s Republic of China
3 Guangxi Key Laboratory of Optical and Electronic Materials and Devices, China & School of Materials Science and Engineering, Guilin University of Technology, Guilin 541004, People’s Republic of China
4 Authors to whom any correspondence should be addressed.

E-mail: mingao@uestc.edu.cn and linyuan@uestc.edu.cn

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Abstract

Vanadium dioxide (VO$_2$) is a typical thermal induced phase transition material, exhibiting a transition from metallic phase at high temperature to insulating phase at low temperature, which is also accompanied by a conductivity change of over several orders of magnitude. The transition property makes VO$_2$ prominent to achieve an effective degree of control of terahertz (THz) wave. In this paper, composite films consisting of metal grating with different periods and VO$_2$ film were prepared by polymer assisted deposition method. Although the conductivity change of VO$_2$ films deposited on gold grating structure across phase transition was declined to about two orders of magnitude, the amplitude modulation depth of THz of the composite films can still reach a high value. Furthermore, it was found that the THz modulation depth was related with the grating period. According to theoretical simulation, the fluctuation height of VO$_2$ films, caused by metal grating structure during growth, can be used to regulate THz wave. These results demonstrate an economic and unsophisticated method to fabricate VO$_2$ films with thickness fluctuation structure and then tune the THz waves.

1. Introduction

Terahertz (THz) wave refers to electromagnetic wave with a frequency range from 0.1 to 10 THz and the wavelength range is approximately 0.03 to 3 mm. It is a new source of radiation with many unique advantages, such as high spatial and temporal coherence, high signal to noise ratio and low photon energy. Therefore, it has received considerable attention in many fields like biochemical identification, safety monitoring, wireless broadband communication, spectral characterizations of materials, etc [1–3]. Owing to the different requirements for the band and intensity of THz in various fields, it is necessary to effectively manipulate THz wave. During the last several decades, plenty of approaches based on various materials have been studied to fabricate THz modulator [4–6]. One of the modulation methods is utilizing electromagnetic metamaterials (MMs) [7–10]. MMs are artificial materials consisting of periodically arranged structural units with special electromagnetic properties not found in natural materials. By adjusting the structure, critical dimensions and the type of material, the response of MMs to THz wave can be manipulated. The sub-wavelength grating structure, a relatively simple structure among various meta-structures, can modulate the polarization transmittance of THz wave [11]. And the properties of grating structure can be controlled by period, fill factor or material of grating. Meanwhile, many changeable properties of MMs based THz modulator have been realized by introducing active materials such as vanadium dioxide (VO$_2$) [12–15], photonic crystals [16–18], polymer
materials [19, 20] and graphene [21, 22]. In recent years, VO₂ has received increasing interest. It is one of the representative oxides with the property of thermal-induced phase transition, which can transfer from insulating state (monoclinic structure) to metallic state (tetragonal rutile structure) around 340 K [23, 24]. In the process of transition, it also causes dramatic change of electrical and optical properties in addition to structural changes [25–27]. The change in resistance can reach more than 4 orders of magnitude. This phase change property can be triggered on a sub-picosecond timescale by different external conditions, including temperature, stress, electromagnetic field, light, etc [28–30]. Due to its unique property, it is potential for the design of logic storage devices [31, 32], switching devices [33, 34], modulators [35, 36] and many other devices. Utilizing this material, the transmission of electromagnetic waves can be continuously and spontaneously tuned by means of temperature control over a wide spectral range, and thus VO₂ gives rise to great possibilities in the field of electromagnetic wave modulation and switching devices. Therefore, VO₂ films with sub-wavelength grating structure have great potential in modulating the THz waves. On the other hand, the transmittance change trend of the VO₂ can change with the thickness of films because of dramatic change of complex refractive index during phase transition [36]. Overall, fabricating thickness periodic distribution structure by VO₂ would be another way to further improve the properties of VO₂-based THz devices. VO₂ films can be patterned by standard photolithography and wet etching technique, but these methods are complex. In this paper, VO₂ films were deposited on sub-wavelength metallic grating structures by the polymer assisted deposition (PAD) method [37–40]. The atomic force microscopy images revealed that the thickness of VO₂ films formed periodic height fluctuations and the fluctuation height increased with the increase of grating period. By investigating THz transmission properties of these composite films at different temperatures, we found that the THz modulation depth was related with the grating period. Combining with the electrical results and theoretical simulation, it is suggested that the periodic fluctuation height of VO₂ films dominated the regulation of THz transmission.

2. Experiments

In our experiments, the substrates were (10–10) Al₂O₃ with thickness of 500 μm and area of 1 cm². The VO₂ thin film was deposited by the PAD method. The first step is to prepare the precursor solution. 3 g of polyethyleneimine (PEI), 3 g of ethylenediaminetetraacetic acid (EDTA) and 1.2 g of ammonium metavanadate were dissolved in a certain amount of deionized water, followed by being stirred with a magnetic stirrer until they were absolutely dissolved to form a uniform solution with clear yellow-green color. Then the NH₄⁺ ions in the yellow-green solution were removed by an ultrafiltration cup to obtain about 20 ml of yellow-green viscous precursor solution. Meanwhile, the gold grating structures were prepared by lithography and magnetron sputtering on (10–10) Al₂O₃ substrates with a thickness of 100 nm. In order to ensure the adhesion of Au to the substrate, a thin layer of nickel was deposited before depositing the gold film. Patterns were prepared using the contact exposure method. The extra part was eliminated by the lift-off process. The lift-off step after the sputtering process was used to remove the excess metal parts by ethanol. The period of the grating structure was changed from 6 μm to 10 μm. To prepare VO₂ films, the substrates coated with the precursor solution were placed in an annealing furnace to crystallize the films. The detailed growth process has been reported in our previous work. [39] The pure VO₂ films deposited on Al₂O₃ substrates have a thickness of about 120 nm. The schematic diagram of the composite film is shown in figure 1(a).

The morphologies of the samples were characterized by optical microscopy and atomic force microscopy (AFM) and the microstructures of the films were analyzed by x-ray diffraction (XRD) and Raman spectroscopy. Electrical properties of VO₂ films were characterized at temperature between 25 °C and 100 °C using a four-probe test. A transmissive THz–TDS (THz time-domain spectroscopy) system was employed to measure the THz transmission in the THz frequency range of 0–3 THz. The pump source is a mode-locked titanium: sapphire femtosecond laser with power of 800 mW and 100 fs pulsewidth at a repetition rate of 80 MHz. The laser is used to generate nearly single-cycle THz pulses via GaAs photoconductive antenna. Frequency-domain transmission data were obtained from the Fourier transform of the time-domain THz transmission of the VO₂ films. A bare Al₂O₃ substrate was used as a reference sample to eliminate the influence of substrate.

3. Results and discussion

Figure S1 is available online at stacks.iop.org/MRX/7/056404/mmedia in the supplementary material shows the optical images of grating with the same duty cycle and different periods. From the optical images, it can be seen that the gold line width and internal width of the prepared grating structures are uniform. And the width of the gold line and the internal part is the same. Then VO₂ films were deposited on the prepared grating structures. The surface topography of the as-prepared sample was characterized by AFM, shown in figures 1(b)–(e). Figure 1(b) is a pure VO₂ film without grating structure, as a reference sample. The roughness of the pure VO₂
film is only 8.1 nm. Figures 1(c)–(e) are the morphologies and line profiles for the as-prepared VO₂-grating composite films with different grating periods, which show that the surface of the as-prepared VO₂ films on grating structure is not flat and the height difference between the top surface of metal grating and the internal
part increases with the period of the grating structure. The height difference presents a nearly linear relationship with the period of metal grating, shown in figure S2(a) in the supplementary material. The height difference changes are related with the different wettability of sapphire and gold surfaces, shown in figures S2(b)–(c) in the supplementary material.

Figure S3 in the supplementary material displays the XRD results of the pure VO2 film and the VO2 films on three kinds of metal gratings. The peak at 68° corresponds to (30–30) of the sapphire substrate. The peaks at 60.76° and 64.78° correspond to the (−313) and (−402) planes of the VO2 film (JCPDS Card No. 72-0514). From the XRD spectra, the pure VO2 film in this experiment is crystallized with a highly preferred orientation. As for the composite films, due to poor lattice matching between VO2 and Au, the film deposited on grating shows deteriorated crystallinity with more peaks of other orientations. The peaks at 27.7°, 33.1°, 44.3°, and 78° are corresponding to VO2(011), VO2(−102), Au(200) and Au(311) (JCPDS Card No. 72-0514 and No. 04-0784), respectively. XRD results indicate that the presence of the grating has an effect on the growth orientation of the VO2 film and the period of Arespectu grating has no obvious effect on the growth orientation.

The metal–insulator transition (MIT) characteristics of the pure VO2 film and the VO2-grating composite films were measured by resistance-temperature curves. The four-probe method was used to test the resistance of films. The samples were mounted on a holder whose temperature was controlled by an external temperature controller Linkam HFS600E-PB2 and monitored by a platinum resistance temperature detector. Each temperature measurement point was kept for 2 min to ensure the data stability. The results are illustrated in figure 2. Pure VO2 film shows an abrupt electrical resistivity change of four orders of magnitude above and below the transition temperature \( T_{\text{MIT}} \left( T_{\text{MIT}} < 200 \text{°C} \right) \). And as for the composite films, they all show electrical resistivity change of two orders of magnitude. Compared to pure VO2 film, the resistances of the composite films in the insulating phase are lowered, and the resistances in metallic phase are higher. The decrease of magnitude of electrical resistivity change across the phase transition for the composite films is from the poor lattice matching between the film and the metal, which affects the crystalline quality of the VO2 film on the Au pattern. To illustrate the electrical properties of phase transition temperature and hysteresis width, the electrical resistivity curves were differentiated, as shown in figures S4(a)–(d) in the supplementary material. The maximum differential absolute value is corresponded to the phase transition temperatures for the heating and cooling curves, defined as \( T_{\text{heating}} \) and \( T_{\text{cooling}} \), respectively. The phase transition temperatures can be assumed as \( T_{\text{c}} = (T_{\text{heating}} + T_{\text{cooling}})/2 \). From figure S4, we can get that the transition temperature and hysteresis width for the pure VO2 film and the VO2-grating composite films have little change, which is 58 °C and 8 °C, respectively.

To verify the existence of VO2 in the gap and surface of metal grating, we performed micro-Raman spectroscopy on the internal and the top area of the metal grating (shown in the figure S5 of the supplementary material), respectively. Figure S5(a) is the Raman spectrum for VO2 film directly deposited on sapphire substrate without metal grating. Figures S5(b)–(d) are the Raman spectra, measured on the internal and the metal area of grating structure with different periods. All the Raman spectra in figure S5 are the intrinsic spectra of VO2 films. And we did not observe obvious change on the width or position of Raman peaks. The Raman results indicate that VO2 films were successfully deposited on the top and the internal area of metal grating structures.

THz properties of all the samples were performed using a THz–TDS system. An external heater was used to control the temperature of the film. The setup of home-made THZ-TDS system has been described previously [41]. Since THz wave is sensitive to water molecules, the tested area was treated by nitrogen gas before measurement to prevent the absorption of water molecules on the surface of the films. All the samples were placed along the same direction to avoid the effect of substrate orientation on THz response. The polarization direction of THz waves was set in perpendicular to the periodic direction of metal grating structures. The THz
response of the samples has little difference when the phase transition is finished, and we focus on the change of THz response between two different phase states. In the following part, we only exhibit the temperature-dependent data in the heating process. Figures 3(a)–(d) are the THz transmission waveforms for the pure VO2 film and the composite films obtained at different temperatures. As the temperature increases, the amplitude of THz waves decreases, which indicates that the THz transmission intensities of all the samples decrease with the increasing temperature. In the heating process, the VO2 films transited from the insulating phase to the metallic phase, and the crystal structure transited from monoclinic structure (referred as VO2(M)) to tetragonal rutile structure (referred as VO2(R)). In VO2(R), the unfilled π∗ and d|| bands partially overlap, and the Fermi level falls in the overlap region [42], resulting in the increasing of carries. More carries would enhance the reflection and absorption of THz waves, thus decrease the transmittance. Composite films also exhibit the same properties of the THz response. The test results of the samples with different grating periods at different temperatures are shown in figures 3(b) to (d). It is clear that the amplitude change ratio with temperature for the composite films is smaller than that of the pure VO2 film. To better analyze the experimental results, the THz response in time-domain was transformed to frequency response by Fourier transforming, which is shown in figure S6 in the supplementary material.

Then, the transmittances in frequency domain for the samples can be calculated by the following equation:

\[ T(\omega) = \frac{E_{\text{sample}}(\omega)}{E_{\text{substrate}}(\omega)} \]  

where \( E_{\text{sample}}(\omega) \) is frequency response of the VO2 film and \( E_{\text{substrate}}(\omega) \) is frequency response of the sapphire substrate. The transmittances in frequency domain for the pure VO2 film and the composite films are shown in figure 4.

The amplitude modulation depth (MD) of THz wave is an important index for VO2 based THz devices. It can be calculated using this equation [43, 44]:

\[ \text{MD} = \frac{(T_{25^\circ\text{C}}(\omega) - T_{100^\circ\text{C}}(\omega))}{T_{25^\circ\text{C}}(\omega)} \]  

where \( T_{25^\circ\text{C}} \) and \( T_{100^\circ\text{C}} \) are the transmittances at 25 °C and 100 °C, respectively. The calculated results for samples with different periods are shown in figure 5(a). The MD at 1.0 THz for the composite films with grating periods of 10 μm, 8 μm and 6 μm are around 60%, 50% and 40%, respectively, which shown in figure 5(b). It is

Figure 3. Time-domain THz response for all the samples in heating process. (a) VO2 film without grating. (b–d) VO2 films on metal grating with different periods: (b) period is 6 μm; (c) period is 8 μm; (d) period is 10 μm.
obvious that the MD increases with the increase of period. It is well known that the MIT properties of VO$_2$ are strongly related to the MD. The transmittance can be estimated as

\[ T(\omega) = \frac{1 + n_{\text{substrate}}}{1 + n_{\text{substrate}} + Z_0 \sigma(\omega) d_{\text{film}}} \]

where $Z_0$ is the impedance of free space, which is set to 377 $\Omega$, $n_{\text{substrate}}$ is the refractive index of the sapphire substrate, which is 3.3, $\sigma$ is the conductivity of the film, and $d$ is the thickness of the film. Based on this equation, the MD is related with the resistance change across the MIT. A high resistance drop during the transition can increase the MD. In figure 2, the pure VO$_2$ film shows an abrupt electrical resistivity change of four orders of magnitude.

![Figure 4](image-url)  
**Figure 4.** Frequency-domain THz transmittance for all the samples at different temperature. (a) VO$_2$ film without grating. (b)–(d) VO$_2$ films on metal grating with different periods: (b) period is 6 $\mu$m; (c) period is 8 $\mu$m; (d) period is 10 $\mu$m.

![Figure 5](image-url)  
**Figure 5.** (a) Modulation depth of all the samples with different grating periods as a function of frequency from 0.5 to 2.5 THz. (b) The modulation depth at 1.0 THz versus the grating period.
magnitude across MIT, while the composite films only show the electrical resistivity change of about two orders of magnitude. The decrease of magnitude for electrical resistivity change across transition for composite films led to the decrease of THz transmittance compared with the pure VO₂ film. On the other hand, no obvious correlation between the electrical resistivity change and the period of the gold grating for the composite films, as shown the figure 2. Even more, the composite film with 10 μm gold grating has the smallest electrical resistivity drop whereas it possesses the highest MD in the three types of composite films, which means there should be other factors to modulate THz transmittance. From the AFM images, the VO₂ films deposited on the gold grating structure are not flat, and the fluctuation height is related with grating structure period. It is suggested that the fluctuation height may be critical in modulating THz transmittance. Larger periodic fluctuation height of VO₂ film may enhance the MD.

To verify the effect of fluctuation height on the THz transmission properties of VO₂ films, CST STUDIO SUITE software was used to simulate. In order to simplify the simulation, the calculated model was constructed as a symmetrical periodic unit structure along X direction and Y direction, while the Z direction is set to open, which means a free space. The fluctuation height and period were set to different values. In this simulation, the average thickness of the VO₂ film with different fluctuation heights is the same, which is set to 120 nm, and the relative dielectric constant of the film at room temperature and high temperature is set to 10 and 4, respectively. The calculated transmittance for different films at high temperature has little difference, which is close to zero, but the calculated transmittance at room temperature depends on the fluctuation height, which is shown in figure 6(a). The transmittance at room temperature increases with the increase of fluctuation height, which is consistent with our experimental results. On the other hand, the calculated transmittance at room temperature keeps the same while changing the period if the fluctuation height is set at the same value, as shown in figure 6(b). The intensity of light decreases exponentially with the thickness of VO₂ film, so the transmittance is related with the thickness change when the average thickness is the same. Higher thickness change value can increase the transmittance of VO₂ film at insulating phase. However, thin VO₂ film may increase the transmittance of VO₂ film at metallic phase, which would decrease the modulation depth. In one word, the simulation results confirm that adjusting the fluctuation height of VO₂ film is another effective way to regulate THz wave, but the height change has a limited value change.

4. Conclusion

VO₂ films deposited on Al₂O₃ (10–10) substrate with different gold grating structure using PAD method showed periodic height fluctuations. And the height difference increases with the increase of gold grating period. The THz transmission properties of these composite films show that the THz modulation depth increases as the increase of grating period. Basing on the simulation results, the modulation ability of composite films is from the fluctuation height difference of VO₂ films. Our results indicate preparing VO₂ films with thickness fluctuation is another method to enhance the properties of VO₂ films, which could broaden the applications of VO₂ films on arbitrary substrates and THz regulators.
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ORCID iDs

Min Gao https://orcid.org/0000-0003-3899-2933
Sheng-Nian Luo https://orcid.org/0000-0002-7538-0541

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