

## Electromagnetic wave sensors

# Optical Limiting Sensor Based on Multilayer Optimization of Ag/VO<sub>2</sub> Phase Change Material

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**Abstract**—An optical limiting sensor working in the infrared (IR) was developed to address the need for eye and sensor protection against laser threats. Metallic and dielectric photonic resonators (thin-film multilayers) incorporating phase-change-materials, such as vanadium dioxide (VO<sub>2</sub>) were simulated and experimentally realized, with optimization of the deposition procedure by RF magnetron sputtering at low temperature. For the first time, the silver is placed between the substrate and the VO<sub>2</sub>, thus improving the device limiting performances. By maximizing the difference of transmittance between the “on” and the “off” states at the standard light wavelength for telecom applications (1550 nm), we calculated optimum thickness for VO<sub>2</sub> and silver films. The deposited thin films were characterized by SEM and Raman spectroscopy, and VO<sub>2</sub> transition temperature was investigated by measuring resistance changes. As a proof of concept of the device working principle, we calculated transmittance drop of 70% when the sensor is exposed to a laser pulse excitation (20 ps, 500 MW/cm<sup>2</sup>). Our results pave the way for multilayer with optical limiting properties.

**Index Terms**—Electromagnetic wave sensors, light intensity sensor, magnetron sputtering, optical limiting, phase-change material, vanadium dioxide (VO<sub>2</sub>).

## I. INTRODUCTION

Infrared (IR) light at high power can cause irreversible injury to the human eye. On the contrary, low-power IR radiation is not harmful to the eyesight. High-power lasers are widespread nowadays in several fields, such as spectroscopy, medical applications, production (laser cutting), and defence. It is important to take precautions because IR light is invisible, so the eyes will not take the protective measures, such as blinking or closing when a high-intensity beam of IR radiation shines into them. It is thus of utmost importance to develop a threshold sensor for high optical power that can sense it and switch to nontransmitting state preventing eyes from damaging. An additional application is the protection of detectors in power meters.

In the present work, we designed a dynamic device [1] whose transmittance depends on the intensity of the incident radiation, based on the use of a phase-change-material (PCM), namely, vanadium dioxide (VO<sub>2</sub>). The latter can be grown by different techniques [2], among those sputtering is one of the most industrially applicable [3], [4], [5], [6]. We investigated the condition to obtain the VO<sub>2</sub> phase by room temperature deposition and subsequent annealing.

VO<sub>2</sub> is a material endowed with interesting properties. It undergoes a reversible phase transition from monoclinic to tetragonal phase at a temperature around the insulator to metal transition temperature T<sub>IMT</sub> [5]. When the temperature is low, it is in the insulating state,

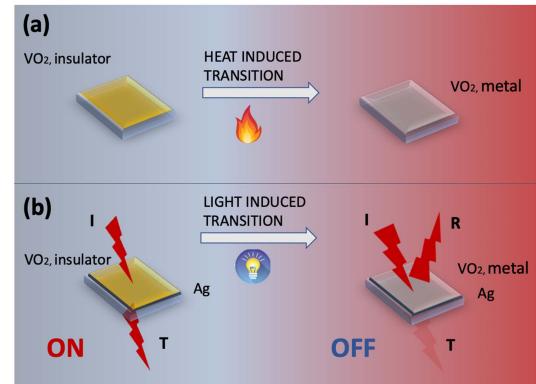


Fig. 1. Concept of optical limiting sensor. (a) VO<sub>2</sub> can be converted from insulator to metal by heating above T<sub>IMT</sub>. (b) By using an excitation with pulsed laser in the IR, at proper value of peak intensity and pulse duration, the IR absorption by the device raises the temperature in the VO<sub>2</sub> above T<sub>IMT</sub>, and the transmittance drops, so the device is in the OFF state. The sensor is reversible, coming back in the ON state when the stimulus is removed.

whereas when the temperature exceeds the transition temperature, it is metallic with high reflectance in the IR. The transition can be induced in several ways, by temperature increase and by optical absorption [7], as schematized in Fig. 1. In particular, the former scenario is depicted in Fig. 1(a), in which a device made by a VO<sub>2</sub> thin film transmits all the incident light when the temperature is lower than the T<sub>IMT</sub>; instead, when the temperature is higher than T<sub>IMT</sub>, the VO<sub>2</sub> converts

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to metal and the light is reflected. Fig. 1(b) shows the optical limiting concept: the absorption of a very intense light yields a temperature increase, exceeding  $T_{IMT}$ , and induces the switch of the device to a low transmission state. In the latter case, a silver (Ag) layer is added to increase the IR radiation absorption inside the  $\text{VO}_2$ , thus obtaining light-induced thermal switching of the  $\text{VO}_2$  layer.

COMSOL Multiphysics simulations in conjunction with a transfer matrix approach allow to perform the geometrical optimization and the theoretical characterization of the device. The threshold intensity at which the phase transition in  $\text{VO}_2$  occurs strongly depends on the design: At variance with [7], the Ag is placed between the substrate and the  $\text{VO}_2$ , thus maximizing the device limiting performances. We optimized the deposition condition to obtain a multilayer of Ag and  $\text{VO}_2$  films by using magnetron sputtering, also considering the influence of different substrates (fused silica and sapphire).

## II. METHODS

### A. Device Realization

Thin films multilayers were deposited by a modular sputtering system equipped with RF and dc generators (Advanced Energy).  $\text{VO}_2$  thin film was grown starting from a pure vanadium target, with RF magnetron sputtering in oxidizing conditions. The oxygen/Ar ratio during deposition was 50%, and the substrates were kept at room temperature. Glass and sapphire substrate were tested and the influence on  $\text{VO}_2$  phase analyzed. To realize the optical sensor, a thin Ag film was deposited by dc magnetron sputtering between the substrate and the  $\text{VO}_2$  to control the incident intensity threshold power at which the switching occurs. Annealing procedure was optimized to achieve the correct crystalline monoclinic phase: air or vacuum from a primary pump were investigated as annealing atmospheres.

PCM phase and morphology were investigated by Raman spectroscopy (RS) and field emission scanning electron microscope (SEM) LEO 1525. For RS, a modular micro-Raman confocal system from Horiba with laser excitation at 532 nm, 100 $\times$  objective and 1800 l/mm grating were used.

### B. Simulation and Test

Optimization of the geometrical parameters was carried out employing a transfer matrix approach, where the whole  $\text{VO}_2$  layer was assumed to be in its metallic or dielectric phase, corresponding to “OFF” and “ON” states, respectively. The theoretical characterization was achieved with transient opto-thermal simulations performed with COMSOL Multiphysics software [8], [9], [10], [11].

For testing the sensor performances, a home-built microscopy setup integrated with a tunable pulsed laser from an optical parametric oscillator was used. The transmission through the samples is recorded both on a power meter and on an InGaAs camera.

## III. RESULTS AND DISCUSSIONS

### A. Simulations for Geometrical Optimization

Before the experimental deposition of the multilayer, the device simulation was carried out to optimize the geometrical parameters (thickness of Ag and  $\text{VO}_2$  layer) in order to have maximum change in transmittance  $\Delta\text{Tr} = \text{Tr}_{\text{dielectric}} - \text{Tr}_{\text{metal}}$ , where  $\text{Tr}_{\text{dielectric}}$  ( $\text{Tr}_{\text{metal}}$ ) is the transmittance in the dielectric (metallic) state. All calculations were made at fixed wavelength at 1550 nm (value of interest for telecom applications), using  $T_{IMT}$  taken from literature [12]. A transfer matrix approach was implemented assuming that the whole  $\text{VO}_2$  layer was

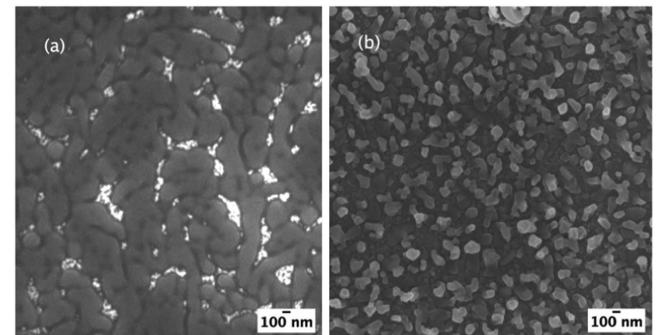


Fig. 2. SEM images of the (a)  $\text{VO}_2$  thin film compared with (b) Ag/ $\text{VO}_2$  thin films acquired at 50k $\times$  magnification. Grain size observed in  $\text{VO}_2$  thin film is much bigger than the one of the Ag/ $\text{VO}_2$  multilayer (100 nm).

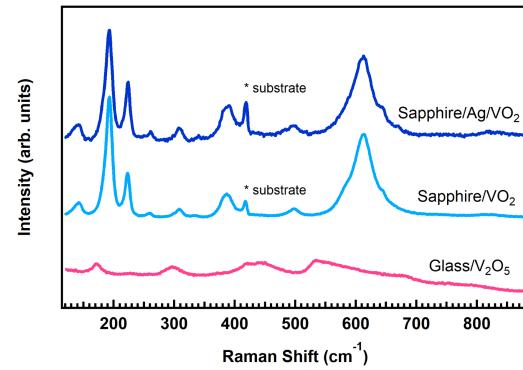


Fig. 3. Raman spectra acquired on vanadium oxide layers for different substrates. For sapphire substrate, both  $\text{VO}_2$  and Ag/ $\text{VO}_2$  layers (blue and light blue lines) present only vibrations from  $\text{VO}_2$ , (M1) phase, and sapphire substrate; (glass-magenta line) the phase obtained on glass substrate is poorly crystalline, with few peaks ascribable to  $\text{V}_2\text{O}_5$ .

assumed to be in its metallic or dielectric phase. For the optimal configuration, a transmittance drop of more than 70% is expected when the device is in the “OFF” state. The optimum thickness obtained for Ag/ $\text{VO}_2$  are 5/52 nm, respectively.

### B. Sensor Characterization

SEM microscopy revealed the morphology of the single and multilayer film (Fig. 2). Polycrystalline structure can be observed for  $\text{VO}_2$  thin film—Fig. 2(a)—many rounded particles with typical dimensions between 150 and 250 nm coalesce in a brain-shaped fashion. For multilayer film Ag/ $\text{VO}_2$ —Fig. 2(b)—the mean diameter of the observed particles is much smaller, being around 100 nm. Very porous structuring of the surface is observable at higher magnification. Despite the observed strong influence of the Ag layer on the  $\text{VO}_2$  film morphology, the correct phase was observed both for  $\text{VO}_2$  and for Ag/ $\text{VO}_2$  multilayer, as confirmed by RS.

RS was useful to characterize the quality of produced thin films. It is well known that the  $\text{V}_2\text{O}_5$  phase is unwanted, and the procedural window to obtain the pure  $\text{VO}_2$  phase is quite narrow. Reactive sputtering is needed when starting from a metal target, followed by annealing at 550 °C in vacuum. Air atmosphere during annealing produced the unwanted  $\text{V}_2\text{O}_5$  phase. Influence of the substrate for deposition is shown in Fig. 3, by comparing thin films deposited on sapphire and glass substrate. Lattice vibrations typical of the  $\text{VO}_2$  (M1) insulating phase [13], [14] can be observed for the thin film deposited on sapphire substrate (except for the peak of the sapphire substrate,

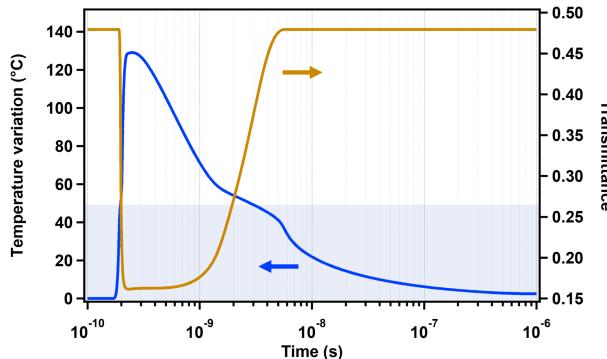


Fig. 4. Simulated temperature variation (blue) and transmittance (brown) temporal dynamics following the excitation from a single laser pulse (1550 nm, 20 ps FWHM, peak intensity 500 MW/cm<sup>2</sup>). The shaded area represents the region with temperature lower than  $T_{IMT}$ . The device undergoes a change of optical properties only when  $T > T_{IMT}$ .

visible at 417 cm<sup>-1</sup>). On the contrary, when a glass substrate was used, the correct phase was not achieved, and broad peaks of V<sub>2</sub>O<sub>5</sub> appear to be superposed to the amorphous band of the glass substrate. VO<sub>2</sub> (M1) phase is achieved as well for multilayer Ag/VO<sub>2</sub>.

To investigate the  $T_{IMT}$ , we placed the device on a Peltier cell (Linkam THMS600) for controlled temperature change. The Raman-active phonon modes of the VO<sub>2</sub> disappear across the semiconductor–metal phase transition because the unit cell of VO<sub>2</sub> changes to the rutile phase. The transition was tested also by measuring the resistivity of the Ag/VO<sub>2</sub>, confirming that  $T_{IMT} = 65$  °C during the heating cycle and  $T_{IMT} = 59$  °C during the cooling cycle. The cycle is fully reversible and was repeated several times with no observable changes. Moreover, stability tests performed over samples kept in ambient air at room temperature for up to four months showed no variation in the phase by RS.

The insulator to metal transition was confirmed by transmittance measurements performed at 1550 nm for VO<sub>2</sub> and Ag/VO<sub>2</sub> sample, with calculation of the hysteresis cycle.

### C. Simulations to Investigate the Thermal Dynamics

We studied the transient response of the sensor device when illuminated by a laser pulse of duration 20 ps and peak intensity 500 MW/cm<sup>2</sup> (details of the modeling are reported in [15] for Au/VO<sub>2</sub> multilayer). Fig. 4 reports the transient response of the sensor. As we can see, after the pulse arrival (occurring at 200 ps) the temperature in the VO<sub>2</sub> layer (blue curve) increases and overcomes the critical value; hence, the transmittance (brown curve) drops from 0.47 to 0.15. The response time of the layer, which is the time interval necessary to heat the sensor to the maximum temperature, is the same as the pulse duration [20 ps at full-width at half-maximum (FWHM)]. Afterward, the device cools down and after 3 ns the temperature becomes lower than the critical value, hence restoring the high transmittance state. The complete cooling occurs on longer time scales, as shown in the Fig. 4. We noticed that the  $T_{IMT}$ , and thus the OFF state, is reached for the high intensity source. In the case of a low-intensity light, the equilibrium temperature does not exceed the critical one, and thus, the sensor is not activated.

## IV. CONCLUSION

We designed and realized a planar multilayer sensor with vanadium dioxide to achieve a self-activating optical limiter with ON–OFF behavior. The mechanism is activated by the temperature increase induced by high-power laser in the IR (1550 nm).

After the geometrical optimization of the device to maximize the change of the transmitted power, we obtained a reduction up to 70% after transition in the OFF state. The device was realized by magnetron sputtering deposition of multilayer Ag/VO<sub>2</sub>. The characterization via RS and SEM allowed to obtain an optimized device that shows good agreement with the theoretical predictions. Our results open the opportunity for novel optical sensor with optical limiting properties.

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