Programming emissivity on fully integrated VO$_2$ windows

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Abstract

The programmability of emissivity states in a monolithically integrated micro window based on vanadium dioxide (VO$_2$) thin film is demonstrated. The 400 $\mu$m window features a VO$_2$ thin film with integrated electrodes for actuation and sensing. The phase transition was induced by resistive heating, while the electrical resistivity and optical transmittance (for near IR wavelength of 1550 nm) of the VO$_2$ thin film were monitored simultaneously. Abrupt drops in electrical resistance and optical transmittance confirmed the quality of the VO$_2$ thin film. Electronic pulses were used to program emissivity states in the VO$_2$ window. The emissivity programmed state was shown for a specific DC current over imposed with the programming pulse; but any emissivity state that belongs to the minor hysteretic curves can be obtained by choosing different electronic inputs. The fully monolithically integrated device presented here can be used for IR cloaking applications, where different emissivity values can be programmed with electronic pulses. Copyright © 2018 VBRI Press.

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Introduction

In the last few years, adaptive camouflage has seen an increase interest for its wide array of applications. Many animals have natural camouflage capabilities for survival and hunting. Species such as cephalopods and reptiles, use a form of visual coloration to adapt to any surroundings [1–3]. Numerous past studies have tried to discern and incorporate such adaptive camouflage for commercial and military grade applications [4]. Similarly, infrared cloaking is of equal importance due to the ability to cloak and shape shift in thermal imaging which allows to incorporate any camouflage system on arbitrary surfaces [5–7]. In order to create a thermal cloaking device, either the temperature or the emissivity of the material must be modulated [8,9]. Emissivity modulation is more advantageous over thermal controlling, since it would not involve a real temperature change of the object and it will eliminate the need to compensate for heat dissipation limitations when controlling the temperature. However, emissivity modulation would require materials with tunable optical properties, especially for the thermal infra-red (IR) region.

Vanadium dioxide (VO$_2$), has shown to be a suitable candidate for such applications. It undergoes an insulator to metal phase transition above 68 °C [10], which can be done either by simple conductive, resistive heating, or photo-thermal heating [11–13]. Along with the phase transition, VO$_2$’s optical and electrical properties simultaneously change as well [14-15]. The material’s optical constants (n, k) suffer a significant change across the phase transition, abruptly causing a drop in transmittance. This change is more evident for near-IR (NIR) region up to 2500 nm [16,17], thus allowing VO$_2$ to be incorporated in smart window designs [18–21]. This was cleverly used by Xiao et al., when they reported the use of VO$_2$ for thermally adaptive camouflage. [8]. VO$_2$ also behaves as a highly disordered material for emissivity modulation, due to its change in emittance along the phase transition [22,23]. Furthermore, unlike most materials, VO$_2$’s emissivity decreases as the temperature increases through the phase transition region. This means that the material “looks colder” as the temperature is raised. Since the transition region of VO$_2$ extends only about 10 °C, inducing the change in emissivity of a VO$_2$ thin film coating requires much less energy than changing the emissivity of the coated element by a large increase in temperature. This makes VO$_2$ a great candidate for adaptive camouflage windows.

Here we propose a micro variable optical window based on VO$_2$ thin film. The window can be actuated via resistive heating which allows to program states in both transmittance and emissivity via electronic modulation. Via this change in transmittance and emissivity we can developed a smart VO$_2$ window that could act as a cloaking device. The intrinsic hysteresis of VO$_2$ allows for multiple optical states for a single temperature within the transition region. In this work, we exploit this to demonstrate electronically programmable emissivity states in a monolithically integrated VO$_2$ window.
Experimental

Device Fabrication

The fabrication of the VO$_2$-based window consisted of a 4-mask lithography process, with a minimum feature size of 10 μm. The fabrication process is shown in Fig. 1. A double sided polished SiO$_2$ wafer (2-inch diameter, 500 μm thick, SOF50D05C2, MTI) was used as the substrate. The metal layers (titanium/platinum (Ti/Pt) with thicknesses of 400 Å/1500Å, respectively) used for the heater and electrodes were deposited by evaporation and patterned using lift-off technique. The Ti layer was used only for adhesion purposes. The width of the heater loop was 10 μm and the electrodes was 15 μm. The gap between the heater loop and the electrode was 10 μm as well.

An insulating layer of SiO$_2$ of approximately 400 nm thick was deposited by plasma enhanced chemical deposition (PECVD). This was done in 3 steps of ~130 nm each to avoid possible voids through the SiO$_2$. After deposition, the SiO$_2$ layer was etched by reactive ion etching (RIE) to open vias to the Pt electrodes to the VO$_2$ thin film that was going to be deposited next. A layer of VO$_2$, approximately 170 nm thick was deposited by pulsed laser deposition (PLD). A KrF laser operated at 10 Hz with a laser fluence of ~2 J/cm$^2$ was used, with a deposition time of 25 minutes. The substrate was maintained at 595 °C in an oxygen environment at 15 mTorr pressure. After deposition, an annealing process under the same pressure and temperature conditions was performed for 30 minutes. To avoid any undesired material residual accumulation that would have affected the results for transmission experiments, the backside of the wafer was covered during the deposition. This was followed by the patterning of the VO$_2$ windows through photolithography process and RIE using [24]. Finally, another SiO$_2$ etching step was performed to open contact pads to the heater and electrodes. The 2 inch wafer was then diced into individual dies, each measuring 4 mm$^2$.

Fig. (1). Fabrication process for the VO$_2$ based window. (a) SiO$_2$ substrate, (b) metallization of heater and electrodes. (c) SiO$_2$ insulating layer, (d) opening of the electrodes. (e) VO$_2$ deposition and window patterning, and (f) opening of contact pads for electrical connections.

Experimental setup

Fig. 2 (a) shows the electro-optical setup used to test the VO$_2$ window. The die containing the four micro VO$_2$ windows was mounted and wire-bonded into a circular package which has a hole in the middle to facilitate the transmittance measurements. The package was then mounted to a custom built printed circuit board (PCB) with a centered hole and electrical connections for both the heater and electrodes. Once wired and mounted, the PCB was placed on an X-Y-Z translational stage to align the IR laser beam with incidence normal to the window. A Thorlabs NIR laser diode ($\lambda$ = 1550 nm, ML925B45F) was operated below its stable power of 5 mW for measurement taking. To make sure that the beam spot was properly aligned with the window, a Thorlabs NIR laser diode ($\lambda$ = 980 nm, L980P010) was used. Both laser diodes were passed through a 50:50 NIR beam splitter (Thorlabs, BS015- 50:50, 1100 nm – 1600nm), then coupled into a single mode optical fiber and focused with a lens of 15 mm focal length. The lens was mounted on a micro positioner rail to control the diameter of the beam. A laser beam profiler (LBP, Newport, Model number LBP-4-PCI) was used to assist in the alignment of the focused laser beam and to obtain an approximate value of the beam diameter. After passing through the sample, the laser beam was focused into an optical sensor (S144C, Thorlabs) connected to a power meter (PM100D, Thorlabs), which communicates with a LabVIEW computer interface to facilitate data gathering.

For measurements, the electrical contacts on the PCB were connected to a National Instruments data acquisition (NI USB-6001) control for data acquisition. To actuate the window, a heater current $I_H$ was used while the voltage across the VO$_2$ ($V_{VO_2}$) was measured. Using a voltage divider and $V_{VO_2}$, the resistance of VO$_2$ was calculated (see insert in Fig. 2). For the voltage divider, a series resistor of $R_S$ = 6.67 kΩ and a supply voltage of $V_C$ = 10 V were used to measure the VO$_2$ resistance. Thus, the setup allows to simultaneously measure and drive the device.

Fig. (2). Optical set-ups used to measure the VO$_2$ window: (a) Electro-optical setup used for measurement taking. (b) IR camera setup.
The electro-thermal actuation, temperature distribution and emissivity were investigated by IR thermal imaging (OptoTherm, Infrasight MI320) as shown in Fig. 2 (b). Emissivity measurements were taken as a function of both temperature (i.e. conductive heating) and current (i.e. resistive, or Joule heating). For the temperature measurements, the die containing the window was attached to a Peltier heater. In order to obtain the value of the thermal emissivity, it is necessary to have a material with a well-defined emissivity (i.e. a benchmark). To this end, a piece of masking tape with a known value of emissivity ($E = 0.95$ [25]) was placed near the window of VO$_2$. Then the emissivity of the VO$_2$ was modified in the thermal camera until the temperature measured by the IR thermal imaging system in the VO$_2$ region was equal to the temperature measured in a selected region inside the masking tape.

**Results and discussion**

Characterization of both optical and electrical transitions were performed simultaneously for the 400 $\mu$m sized VO$_2$ window as shown in Fig. 3. In order to obtain the major hysteretic loops, current steps of increasing amplitude (0.1 V, or 0.63 mA) were applied to the heater electrodes until the phase transition was complete. This resulted in the major heating hysteretic loop. Then, current steps of decreasing amplitude were applied until reaching 0 mA, which resulted in the major cooling hysteretic loop. Each current step lasted 1 s, and the measurement was taken after waiting 900 ms from the beginning of the step. During this input to the heater, the VO$_2$’s resistance and transmittance were being monitored. A drop of approximately 3 orders in magnitude is visible for the resistance of VO$_2$ across its phase transition. This drop in resistance of VO$_2$ is to be expected and confirms the overall good quality of the sample. The average resistance drop for the sample was from $R_L = 631 \, k\Omega$ to $R_f = 676 \, \Omega$, having an average $\frac{R_L}{R_f}$ ratio of $\sim 9.35$. For the optical transmittance, the power of the IR laser before the sample (1.22 mW) was used to normalize the transmitted power through the sample. A transmittance drop from $T_L = 0.36$ to $T_f = 0.14$ was observed for the 400 $\mu$m window, giving a ratio of $\frac{T_L}{T_f} = 2.57$ for the infrared region ($\lambda = 1550 \, nm$). To demonstrate the programmability stages of VO$_2$, several minor loops were measured for both electrical and optical transitions. Fig. 4 shows the plots for resistance and transmittance. The input used for obtaining these minor loops is shown in the insert of Fig. 4.

Obtaining the minor loop plots allows for a more reliable way to program the desired values for transmittance and therefore emissivity. For this case, the same current-increase input steps used to obtain the major heating hysteretic loop was used, until reaching 15 mA.

This was used as the pre-heating current, from which the first electrical/optical state was measured. Programming of a second electrical/optical state was achieved by applying an electrical pulse (also following the same step input) up to 16.97 mA (see Fig. 3). Since this value of current is not enough to complete the phase transition of VO$_2$, once the pulse is over, the resistance...
and transmittance comes back to the pre-heating current, but following one of the minor hysteretic loops. The resulting pre-heated and programmed states in electrical resistance and optical transmittance are shown in Fig. 3, and the corresponding minor loop is identified in Fig. 4. It should be noted that there is a DC shift in transmittance between the minor loops and the programming pulse, which is most likely due to a small difference in the background light when measurements were taken. Although this shows only one programmed state, essentially any electrical resistance/transmittance value that belongs to the minor loops can be programmed by simply using a different pulse magnitude or pre-heating current.

In order to know the required minimum sampling rate for the 400 μm window, the device’s thermal time constant was measured. This was done by measuring the voltage in VO₂ ($V_{VO₂}$) resulting from a single current step input $I_H$. Fig. (5) shows the thermal time constant ($\tau_{therm} = 21.68$ ms) for the device for the case when the step was released (see Fig. 5). This measured time constant of ~ 20 ms is much faster than the 100 ms used for the current step input pulses, which indicates that the pulses will be enough to reach steady-state.

The change in VO₂’s optical properties across its phase transition is larger for wavelengths in the infra-red (IR) region [18]. The material’s ability to radiate thermal energy (i.e. emissivity) also changes abruptly during the material’s phase change, which allows for selective thermal emission. Fig. 6 shows the VO₂ emissivity as a function of both temperature and current. For both plots, VO₂ shows negative differential emissivity at the onset of the phase transition, which occurs around $T_{PT} \approx 68$ °C and $I_{PT} \approx 19.9$ mA. The VO₂ window shows a large thermal emissivity change from 0.76 below the transition point to 0.54 above the transition point. To measure emissivity as a function of current, the temperature of the VO₂ window was first measured as a function of current. Given that the emissivity of VO₂ changes with temperature, we needed a benchmark for calibration. Using the masking tape mentioned earlier for conductive experiments from Peltier heater would not work in this case, since it would require increasing the temperature of the tape by Joule heating through the same resistive heater used for the window. Therefore, we used the platinum heater as a temperature sensor, by monitoring its resistance as the current was applied. The heater’s temperature as a function of current was used to obtain the VO₂ window’s temperature. This allowed for mapping VO₂’s emissivity to obtain figure (6-b). On comparing the transition point for the plots on Fig. (4) to the plot in Fig. (6-b), there is a difference in current of $I_{PT} \approx 4.5$ mA. This is most likely due to the method for measuring the emissivity as a function of current as previously mentioned. It should be noted that the hysteresis curves have similar shapes, which suggests that the difference is most likely a “DC offset”, which would be corrected by an additional integrated device that can be used as a benchmark for calibration.

**Fig. 5.** Time constant measurement for the 400 μm window.

**Fig. 6.** Emissivity as a function of temperature (a) and current (b) for the 400 μm window.

**Fig. 7.** Thermal image for a 400 μm window before and after actuation using an electric pulse (1 mA), supplied in the form of short current steps –as depicted for the electrical and transmissivity experiments. Although both states correspond to the same temperature –since after the pulse the current returns to the pre-heated value–, the thermal image after the pulse clearly shows a lower irradiance, which is mapped to a lower temperature. This is due to the lower emissivity of the VO₂ after the programming pulse.

**Fig. 7.** Thermal image for the 400 μm window before programming pulse (a), and after programming pulse (b).
Conclusion

We have developed a VO$_2$ based window of 400 $\mu$m$^2$ that can be used as a smart window or thermal camouflage system. The electrical and optical transition in VO$_2$ where investigated, and hysteretic curves for minor loops where obtained. The minor loops inside the hysteresis of the VO$_2$ allows to program any state for transmissivity inside the window. Since the optical properties of VO$_2$ change greatly in the IR-region, then the emissivity will change during the transition. The emissivity for the VO$_2$ film as a function of both temperature and current was determined, and confirmed that the film shows a negative differential emissivity with the phase change. Emissivity states were programmed by electronic pulses to change the window’s thermal radiation, which can be used for real-time thermal cloaking. The rapid tune-ability of both transmissivity and emissivity in VO$_2$ suggest that the film could be incorporated in the use of adaptive thermal camouflage devices.

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Author’s contributions

Conceived the plan: J.F., Y.C., and N.S.; Performed the experiments: J. F. and Y. C.; Data analysis: J. F. and T. W., and D.T.; Wrote the paper: J.F., Y.C, T.W, D.T., and N.S. Authors have no competing financial interests.

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