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A smart material for infrared modulation applied to IR sensor

S. Paradis
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Defence R&D Canada – Valcartier
Technical Report
DRDC Valcartier TR 2008-160
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Abstract

Vanadium dioxide (VO_2) is a smart material that offers substantial potential to modulate incident infrared radiation. Its property changes upon phase transition make it an attractive solution as a non-mechanical optical/electrical shutter or functional component in electro-optic systems. The purpose of this document is to report our latest results on the fabrication and characterization of the smart material VO_2 deposited by magnetron radio-frequency sputtering technique. The specific properties under study are the temperature of phase transition T_t, electrical resistivity, and optical transmittance and reflectance in the longwave infrared (LWIR) range. Tungsten (W) dopant was used to tailor the optical properties of VO_2. Films with tungsten concentrations of 1.8, 3.3 and 4.1at.% were studied. The results indicate a shift of T_t from 65°C for undoped film to room temperature (~19°C) for the 1.8at.% W film. The resistivity drop after phase transition for the 1.8at.% W films is 1.5 orders of magnitude compared to 3 orders of magnitude for the undoped films. With concentrations of >3.3at.% W, no phase transition was seen in the temperature range under study. For undoped films, the optical transmittance and reflectance contrast (namely the percentage of transmittance below T_t minus the percentage of the transmittance above T_t) at an IR wavelength of 8 μm are ~80% and ~ 40%, respectively. These values are relative to uncoated silicon wafer. For the 1.8at.% W films, the transmittance and reflectance contrast is ~60% and ~20%. Films with more than 3.3at.% W showed no phase transition, only a linear and small decrease of transmittance and reflectance in the temperature range under study. Electrical training on the undoped and 1.8at.% W films shows that good ohmic contact is achieved after 2-3 training runs with no degradation of the film's optical and electrical properties. The additional results of this study supplement our previously published results, and confirm that tailoring the temperature of transition of VO_2 to room temperature using 1.8at.% tungsten dopant can be done with reasonable compromises on optical properties.

Résumé

Le dioxyde de vanadium (VO_2) est un matériau intelligent qui offre un potentiel considérable pour la modulation de la radiation infrarouge. Ses changements de propriétés qui s'effectuent lors de la transition de phase en font un matériau intéressant pour des applications d'obturateur optique/électrique sans pièce mobile ou comme composante fonctionnelle dans les systèmes électro-optiques. Le but de ce document est de rapporter nos derniers résultats concernant la fabrication et la caractérisation du matériau intelligent VO_2 fabriqué par pulvérisation magnétron radio-fréquence. Les propriétés spécifiquement étudiées dans cette étude sont la température de transition T_t, la résistivité électrique, la transmittance et la réflectance optique pour le spectre infrarouge lointain (LWIR). Le tungstène (W) a été utilisé comme dopant pour ajuster les propriétés optiques du VO_2. Des films à des concentrations de W de 1,8; 3,3 et 4,1 at.% ont été étudiés. Les résultats indiquent un déplacement de la température de transition de 65°C pour un film non dopé à ~19°C pour le film dopé à 1,8at.%W. La baisse de résistivité après la transition de phase pour les films de 1,8at.%W est d'1.5 ordre de grandeur comparé à trois ordres de grandeur pour les films non dopés. À plus de 3,3 at.%W, il n'y a pas de transition de phase observée dans la gamme de températures à l'étude. Le contraste optique en transmittance et réflectance (c'est-à-dire le pourcentage de transmittance sous T_t moins le pourcentage de transmittance au-dessus de T_t) pour la longueur d'onde IR de 8 μm est respectivement de ~80% et de ~40% pour les films non dopés. Ces valeurs sont relatives à celles d'une tranche de silicium sans revêtement. Pour les films à 1,8at.%W le contraste en transmittance est de ~60% et celui en réflectance est de 20%. Les films à plus de 3,3at.%W ne montrent aucun contraste optique mais une petite diminution linéaire de la transmittance et de la réflectance dans la gamme de températures à l'étude. L'entraînement électrique des films non dopés et ceux dopés à 1,8at.%W montre que l'obtention de bons contacts ohmiques se fait en 2-3 passes d'entraînement. Aussi, aucune dégradation des propriétés optiques et électriques n'est observée en cours d'entraînement. Les résultats additionnels de cette étude complètent ceux que nous avons publiés antérieurement et confirment que, pour ajuster la température de transition du VO_2 près de l'ambiante, l'utilisation d'un dopage de 1,8at.%W peut se faire avec des compromis raisonnables sur les propriétés optiques.

Executive summary

A variety of electro-optic (EO) systems such as night vision cameras and infrared spectrometers are in use or being developed in the Canadian Forces to provide images of objects and signatures of gases present in various theatres of operation. For many of these systems, infrared (IR) light is the wavelength of interest to be used as the passive incident signal. A passive signal is a signal that comes essentially from the object or scene under observation, with no external radiation used to illuminate it. Modulation of the incoming IR light is desired for some of these EO systems. By modulation we mean the capability to partially or completely block or admit, either gradually or abruptly, the transmission of radiation at a specific bandwidth or wavelength. Examples where modulation is potentially needed include 1) protection of the sensitive infrared detectors of surveillance systems from strong laser radiation, 2) dispersive spectrometer where the radiation enters sequentially in an array of small windows (multiplexing) to improve the signal-to-noise ratio, 3) thermal regulation for spacecraft or buildings where IR radiation needs to be blocked or admitted to optimize the occupant comfort or spacecraft performance.

Some of the current and emerging technologies propose novel solutions for optical modulation such as micro-shutters and liquid crystal-based devices. But these technologies have limitations. Piezo-electric based micro-shutters are mechanical components that are subject to mechanical failure. They also have limited switching speed and require high voltage for actuation. Liquid crystal technology is mature but it suffers the constraints of low switching speed in the sub-millisecond range and relatively high IR absorption, which is disadvantageous in IR systems. One way to get around these limitations is to use a smart material for optical modulation. The term “smart material” is used here to describe a material that has the capability to change, in a reversible way, its optical and electrical properties upon proper triggering. There are a large number of so-called smart materials. At the Micro-Systems Laboratory, we focused on a smart material called vanadium dioxide (VO_2), which can be deposited as a thin film. Our interest for this smart material resides in the fact that, when heated to 68°C, it undergoes a transition from semiconductor to metallic phase. The phase transition transforms VO_2 from transparent to opaque for an incident infrared radiation. This smart behaviour obviates the need for mechanical and moving components to serve as an optical shutter. Tailoring the temperature of transition of VO_2 closer to room temperature while achieving good transmittance and reflectance contrast properties is a specific objective in this study.

This report supplements our previously published results on fabrication, characterization and tailoring of VO_2 films. Data presented here and in our previous publications support the finding that it is feasible to tailor the transition temperature of VO_2 while achieving good optical modulation, making it a viable solution for the modulation of infrared radiation in EO systems. The next step will be to examine the feasibility of a new design for a tunable uncooled infrared sensor. In the proposed design, VO_2 film acts as a non-movable smart mirror embedded in a resonant cavity of a microbolometer. The smart mirror can be tuned to opaque or transparent and permits the selection of the detected signal in the infrared band. This multispectral imaging capability is desirable to provide a spectral selectivity according to battlefield conditions and situations (e.g., 3 to 5 μm in hot/wet conditions and 8 to 12 μm in

cold/haze), and would have a higher probability of detection and recognition of camouflaged or lightly concealed targets. This project is currently under study and preliminary results should be published in the near future.

Sommaire

Au sein des Forces canadiennes, une variété de systèmes électro-optiques (EO) tels les caméras de vision nocturne et les spectromètres infrarouges sont utilisés ou en voie de développement pour pouvoir imager des objets et obtenir la signature de gaz. Chez plusieurs de ces systèmes, le signal infrarouge (IR) est la longueur d'onde d'intérêt qui sera utilisée comme signal passif incident. Le signal passif est un signal qui vient essentiellement de l'objet ou de la scène regardé, aucune illumination externe n'est utilisée. La modulation du signal entrant infrarouge est souhaitable pour certains de ces systèmes EO. Par modulation nous entendons le fait de pouvoir bloquer ou permettre, de façon graduelle ou subite, partiellement ou totalement la transmission d'une longueur d'onde spécifique. Les exemples suivants sont des situations où la modulation est potentiellement nécessaire 1) protection des détecteurs infrarouges à l'intérieur de systèmes de surveillance contre de puissantes radiations laser, 2) spectromètre dispersif où la radiation entre séquentiellement dans un réseau de petites fenêtres (multiplexage) pour améliorer le rapport signal sur bruit, 3) contrôle de température pour des engins spatiaux ou des édifices où la radiation infrarouge doit être bloquée ou admise à l'intérieur pour optimiser le confort des occupants ou la performance des machines.

Certaines technologies courantes ou en cours de développement proposent des solutions novatrices pour faire de la modulation optique; les micro-obturateurs et les composantes à base de cristaux liquides en sont deux exemples. Cependant, il y a des limites à ces technologies. Les micro-obturateurs actionnés par des piézos-électriques sont des composantes mécaniques qui sont sujettes aux bris mécaniques. Ils ont aussi une vitesse de commutation limitée et requièrent un grand voltage pour l'activation. Pour sa part, la technologie du cristal liquide est mature, mais elle souffre de contraintes liées à de basses vitesses de commutation dans le domaine du sous milliseconde. De plus, l'absorption élevée de l'IR par les cristaux liquides est désavantageuse pour leur utilisation dans les systèmes IR. Une façon de contourner ces contraintes est d'utiliser un matériau intelligent. L'expression matériau intelligent est utilisée ici pour décrire un matériau qui a la possibilité de changer, de façon réversible, ses propriétés optiques et électriques lorsqu'on le déclenche de façon appropriée. Il y a plusieurs matériaux dits intelligents. Au laboratoire micro-systèmes, nous avons concentré notre attention sur un matériau intelligent appelé dioxyde de vanadium (VO_2) qui peut se déposer en couches minces. Notre intérêt pour ce matériau repose sur le fait que lorsqu'il est amené à 68°C, il subit une transformation de phase de semiconducteur à métallique. Cette transformation de phase le fait passer de transparent à opaque pour une radiation infrarouge incidente. Par conséquent, ce comportement intelligent permet d'éviter l'utilisation de composantes mécaniques mobiles à des fins d'obturateurs optiques. Un objectif spécifique de cette étude est d'ajuster la température de transition plus près de l'ambiant tout en maintenant de bons contrastes optiques en transmittance et réflectance.

Ce rapport complète nos résultats publiés antérieurement sur la fabrication, la caractérisation et l'ajustement des films de VO_2. Les résultats présentés ici et dans nos publications antérieures soutiennent le fait que l'ajustement de la température de transition du VO_2 peut être faite tout en maintenant une bonne modulation optique et constitue donc une solution intéressante pour la modulation IR dans les systèmes EO. La prochaine étape sera d'évaluer la faisabilité d'un nouveau design pour un détecteur IR non refroidi à bande spectrale

sélective. Dans ce design, le film de VO_2 agit comme un miroir intelligent fixe à l'intérieur de la cavité de résonnance d'un microbolomètre. Le miroir intelligent peut être ajusté pour être opaque ou transparent et permettre de sélectionner la bande infrarouge détectée. Cette capacité de faire de l'imagerie multispectrale est souhaitable pour permettre un choix spectral qui varie selon les conditions et la situation en terrain d'opération (par ex. le 3 à 5 μm pour les climats chauds/ humides et le 8 à 12 μm pour les climats froids/brumeux). Cela permettrait une plus grande probabilité de détection et de reconnaissance des objets camouflés ou cachés. Ce projet est actuellement en cours d'étude et les résultats préliminaires devraient être publiés sous peu.

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1. Introduction

A variety of electro-optic (EO) systems are in use or being developed in the Canadian Forces to provide images of objects and signatures of the gases present in various operational theatres. For many of these systems, infrared (IR) light is the wavelength of interest to be used as the passive incident signal. Modulation of the incoming IR signal is desired for some of these EO systems. By modulation we mean either a gradual change in the transmission (analog) or a sharp and abrupt change (on-off action), also called digital modulation. Mechanical shutters (like a window that opens or closes), can be used to modulate the signal. However, a mechanical shutter may present mechanical or robustness issues, as well as manufacturing problems if the shutter components are at the micro scale. One way to get around these limitations is to use a smart material. The term “smart material” is used here to describe a material that has the capability to change, in a reversible way, its EO properties upon proper triggering. The alteration of the material properties must be large enough to suit the needs of the intended application. The smart material can be produced in the form of a thin film that will eliminate the need for the mechanical and moving parts that comprise an optical shutter (or optical switch). This in turn should yield a more robust micro scale shutter that is easier to manufacture.

There are a large number of so-called smart materials, each offering different EO capabilities for different wavelength bands. The mechanisms used to trigger the change in material properties are also numerous (heat, electrical field, photoinduced, etc).

At the Micro-Systems Laboratory, we focused on a smart material called vanadium dioxide (VO_2), which can be deposited as a thin film. This material is known to have “smart” properties since 1959 [1]. Research has been done ever since to characterize and tailor its properties for specific applications. More details on the nature of VO_2 will be given in the following section. Our interest in this smart material lies in the fact that when it is heated to 68°C it undergoes a phase transition from semiconductive to metallic phase. This phase transition converts VO_2 from transparent to opaque for incident infrared radiation. This property could be exploited in military EO systems where modulation of incoming IR signals is desired. Potential applications include protecting sensitive infrared detectors from strong laser radiation [2], a digital light window to multiplex the incoming infrared signal in a miniature infrared dispersive spectrometer [3], and a smart tunable mirror in an uncooled dual band microbolometer [4]. Vanadium dioxide is also being studied for applications in other technologies, for example, thermal regulation for spacecraft [5] and smart window coatings to reflect solar energy and reduce cooling costs [6].

Work on VO_2 thin film was supported under Work Breakdown Element 15ea05 (previously15ea03) entitled “Miniature IR Spectrometer” from April 04 to March 07. DRDC Valcartier provided additional support on this work carried out under 42gc40 from April 07 to March 08. This report completes the electro-optic characterization and tailoring of VO_2 films.

Chapter 2 briefly describes VO_2 properties and the results of our previous work, and Chapter 3 and 4 present respectively the experimental description of the work and the electrical and optical properties observed for undoped thin films and 1.8, 3.3 and 4.1at.% W-doped thin films.

2. Background

2.1 Vanadium dioxide (VO_2)

Vanadium is a transition metal that forms more than a dozen oxides. These oxides exhibit a large variety of behaviours ranging from metallic (VO) to insulating (V_2O_5), as well as semiconductive (VO_2). This last oxide is of interest mostly because its structural atomic phase can be reversibly changed from semiconductive to metallic at 68°C, which is relatively near room temperature. Other oxides of vanadium exhibit phase transition as well, but at less convenient temperatures (V_2O_3 at -134°C, V_2O_5 at 250°C). The phase transition of VO_2 can be triggered in various ways, but the most common is thermal activation (obtained via thermal heating or via the Joule effect by application of an electrical field). Pure electronic transition triggered only by an electrical field (where heat effect plays no role in the transition) is difficult to obtain, although not impossible [7]. This method is very attractive because pure electronic transition provides higher switching speed. However, it is very likely that most of the time the two mechanisms (thermal and electronic) are present when an electrical field is used to trigger the phase transition.

At room temperature VO_2 is 80% transparent to infrared light, particularly LWIR. When it reaches the temperature of transition (T_t), VO_2 switches from semiconductive to metallic phase and thereby becomes opaque (<10% transmission) to infrared light. Figure 1 illustrates the effect of the phase transition on the transmittance of VO_2 as recorded with our IR camera. The transition is reversible, meaning that when the mechanism inducing the process is turned off, the VO_2 returns to its initial phase. This reversible change of optical and electrical properties is to be exploited in applications where an optical switch or shutter is required.

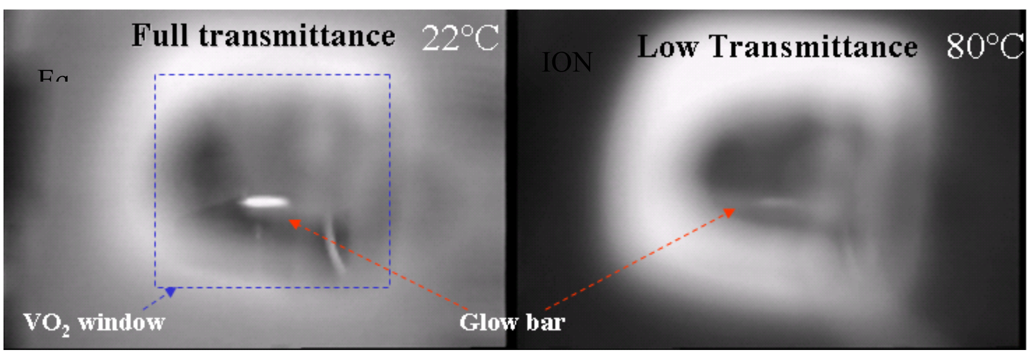

Figure 1: Thermal images of a 1 cm² window of VO_2 thin film that is heated above its temperature of transition via thermal heating. a) At RT, the VO_2 film is transparent for the IR source (cylindrical glow bar). b) When the transition is achieved at high temperature, opacity due to high reflectance of IR radiation is clearly seen.

Examples of potential applications of VO_2 for the military include protection of sensitive infrared detectors from strong laser radiation [2], optical smart slits in an IR micro spectrometer for detection of gas [3], and a smart tunable mirror in an uncooled dual band microbolometer [4]. In order to use VO_2 as a smart material for EO applications, it is often desirable to tailor the phase transition temperature while maintaining good EO properties. Tailoring can be done by adding impurity dopant to the VO_2 atomic structure.

Impurity doping of donors or acceptors can influence the semiconductive-metallic transition properties of VO_2 films. Studies [8-13] showed that doping VO2 films with impurities such as nobium, tantalum, molybdenum or tungsten (n-type doping) will lower Tt, while doping with aluminum, chromium, iron or titanium (p-type doping) will raise Tt. In addition to tailoring the temperature of transition, dopant elements will also affect contrast switching. By contrast we mean the variation of the IR electrical or optical signal with respect to temperature. Compromises must therefore be made between the desired Tt and the acceptable optical contrast for the specific application.

2.2 Previous work

In our previous work [14-16], we reported results on the fabrication and EO characterization of undoped VO_2 thin films. Thin films were deposited on SiO_2/Si substrates by reactive RF magnetron sputtering using a pure vanadium target under various ratios of argon and oxygen gases. The oxygen content in the mixed atmosphere has a significant influence on the semiconductor-metal transition characteristics. Phase transition triggered by thermal activation and by an electrical field was studied. Transmittance in the region of 8 to 12 μm was reported to be 90% at room temperature, the remaining 10% of the radiation being either absorbed or reflected. Above 65°C, less than 10% of incident radiation is transmitted. A corresponding variation of three orders of magnitude in electrical resistivity was observed over the transition. In the near infrared region of 1 to 2 μm, transmittance was also studied and was found to be about 60% at room temperature (RT) and very low (a few percent) above 65°C. Ellipsometry measurements were done to obtain the optical indexes n (refractive index) and k (absorption coefficient). These constants are useful parameters to consider when building multilayer stacks of thin films. The measurement was done for the wavelength range 1-2 μm (8-12 μm measurements are not yet possible in the lab), and showed that the optical constants change significantly when the transition occurs. The refractive index decreases from about 3.4 at RT to 1.6 at 80°C, and the absorption coefficient increases from less than 0.5 at RT to 3.4 at 80°C.

The phase transition of vanadium dioxide triggered by a pulsed voltage was investigated. The use of a short pulse was intended to minimize the Joule heating effect due to leakage current, which is a current that flows through an insulating layer due to residual imperfections in the film such as impurities, pinholes, or grain boundaries. Abrupt semiconductor-metal transition induced by 100 nsec pulsed voltage (AC) was observed in the VO_2 thin film. However, even with this brief pulse, pure electronic effect was difficult to isolate with certainty from thermal effect. More details can be found in [15].

In our previous work, we began tailoring the temperature of transition with tungsten (W), because it was reported to be the most effective dopant to decrease the T_t: only one atomic percent of W is needed to decrease the transition temperature by 23°C. Our goal was to bring the transition temperature closer to room temperature. A 1.5 atomic percent (at.%) W target was used to fabricate the film. Our results showed that the transition temperature was lowered substantially, dropping from near 68°C to room temperature (typically 22-24°C). This temperature decrease suggested a slightly higher dopant concentration than 1.5at.% of W.

The composition of the doped film was obtained by X-ray spectroscopy (XPS), and revealed a surface concentration of about 1.8at.% W. The need to cross-check the composition with another analytical technique should be examined in the future. We also suspected that the temperature of transition may be even lower than 22-24°C. Since we had no cooling set-up at that time, we could not start our measurement below RT.

The optical transmittance contrast observed for the doped films in the 8 to 12 μm IR band remained over 70%, while the electrical resistivity dropped approximately by one and a half orders of magnitude. The sharpness of the drop was less than for undoped VO_2. Both undoped and W-doped films had an optical and electrical hysteresis of about 10°C. Note that a hysteresis loop denotes an unstable area where heating and cooling curves diverge and where, in the case of VO_2, we find second-order hysteresis loops (small loops inside the large main loop).

Co-doping with tungsten and titanium (Ti) was also investigated in our inital work for its effect on the first-order hysteresis loop. A target with 1.5at.% W and 12at.% Ti was used. The effect of this co-doping was to suppress the hysteresis (both for resistivity and transmittance). On the other hand, the optical and electrical contrast became linear and gradual with no sharp phase transition. This behaviour can be useful in applications where a gradual change of optical or electrical properties is required, such as for pressure or temperature sensors. However, since our immediate interest is a material that offers abrupt optical change, the Ti-W co-doping was not investigated further in the present work.

The additional results presented in this report complete earlier investigation on the doping of VO_2 films. Higher concentrations of dopant are studied and the composition of films is cross-checked with another analytical method. Measurements of resistivity and transmittance below RT is reported for undoped and 1.8at% W films. Reflectance measurements and electrical training (defined in section 3.1.4) are also presented.

3. Experimental procedure

All films fabricated in this study were ~0.1 to 0.2 μm in thickness, which is sufficient to provide significant optical modulation. The fabrication techniques used influence the microstructure and the composition of the film. Consequently they have a substantial effect on the film's optical and electrical properties.

3.1 Thin film fabrication & characterization

3.1.1 Thin film structure and deposition

The device configuration silicon-silicon oxide-VO_2 (metal oxide semiconductor, MOS) and the radio frequency (RF) sputtering deposition technique used here have been described elsewhere [14,16]. The 1 cm^2 wafer was used as a transparent physical support for the VO_2 films. Four targets were used with a starting material composition of vanadium and 0, 1.5, 2 and 2.5at.% tungsten. The initial purity of the vanadium used was 99.7%. All targets were mounted on a planar magnetron gun. The substrate was heated to 500°C during deposition.

A reactive plasma was created in the vacuum chamber by introducing high purity Ar gas and O_2 gas. The gases were introduced through separate mass flow controllers.

Sputtered matter cross-contamination was observed when the undoped target was used after several high W content targets. This is discussed briefly in the results section.

The dopant concentration deposited in the VO_2 film structure was estimated by Rutherford backscattering spectrometry (RBS). This technique is based on collisions between a projectile ion beam and the target atomic nuclei (here the VO_2 thin film) and does not require standards. In addition, RBS quantitative analysis is highly sensitive to atoms with a large atomic number, which is the case for tungsten. Therefore it was decided to use the RBS technique to complement the previously used XPS technique [16]. The RBS measurements were done at the University of Montreal. Professor Sjoerd Roorda used a 2 MeV helium ion beam at normal incidence and collected the energy for a particle backscattering at 170°. Four samples were sent for RBS analysis: films fabricated with one undoped target and three other targets doped with 1.5, 2 and 2.5at.% W.

3.1.2 Thickness & resistivity measurements

Film thickness was determined by surface profilometry (Dektak 3030), and was typically ~0.1-0.2 μm. Electrical resistivity was measured using the standard four-point probe technique (Lucas Lab, model 302). Changes in resistivity were recorded every second during both heating and cooling cycles. The starting temperature was either RT or 0°C (achieved by Peltier cooling, also called thermo-electric cooling). The samples were heated to 100°C.

3.1.3 Optical transmittance and reflectance

The transmittance of the thin film substrates was measured at an IR wavelength of 8μm using an ARC SpectraPro-300i spectrometer and a sample heater coupled to a UP150 temperature controller with ramp-up capability. A classical mercury cadmium telluride (MCT, 8-12 μm wavelength range) detector was used, and a glow bar provided the infrared illumination for optical measurements in the LWIR. An improvised cooling system was used to lower the starting temperature for transmittance to -11°C. Thin films were also characterized for reflectance at an IR wavelength of 8 m from RT up to 80°C. The reference for reflectance measurements was chromium/gold thin film deposited on a silicon wafer and the incident angle was less than 60°.

3.1.4 Electrical training

Phase transition can be triggered by direct heating of the film, but an electrical field can also be used to heat the sample (by the Joule effect) or to trigger electronic transition. Low-resistance, stable contacts at the electrode level are important for the performance and reliability of the device when an electrical field is used. Therefore, ohmic contact (region on a semiconductor device that has been prepared so that the current-voltage (I-V) curve of the device is linear and symmetric) is a desirable feature and is achieved through electrical training. . Electrical training consists in biasing the sample to a voltage magnitude sufficient to effect transition, then repeating this process until the $Si/SiO_2/VO_2$ stack resistance, monitored through the rise of voltage across a resistance in series, stabilizes to a low resistance value. There is always a certain level of contact resistance, which causes some power dissipation, resulting in Joule heating. The latter is the dominant heating source for our samples when subjected to an electrical field. Another benefit of electrical training is that, although RF sputtering should produce polycrystalline VO_2 films, there could also be a thin layer of V_2O_5 (an insulator) or other oxide-rich substance at the surface of the film, which would prevent good ohmic contact with the electrode. Electrical training facilitates the transformation of V_2O_5 to VO_2 [17].

Aluminum electrodes were laid out in a specific pattern on the VO_2 thin film and on the underside of the Si substrate (Figure 2).

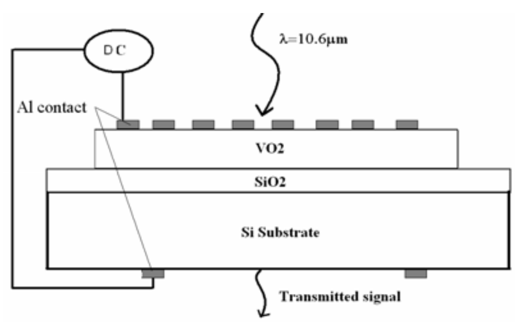

Figure 2: Schematic of MOS structure with the electrodes.

The pattern of finger electrodes consists of a series of 0.5 mm-wide aluminum lines separated by 0.4 mm-wide spaces. The VO_2 samples were connected to a voltage source operating in DC mode to modulate the VO_2 semiconductor-metal transition. A 220 ohm series resistance was used to monitor changes in resistance (thereby the quality of ohmic contact) in the sandwiched MOS structure $Si/SiO_2/VO_2$. A continuous CO_2 laser beam (λ= 10.6 µm) modulated by a chopper operating at a frequency of 20 Hz was used. An MCT detector coupled to a lock-in amplifier was used to observe optical switching and modulation characteristics at RT.

4. Results and discussion

4.1 Thin film composition and electrical properties

The composition of the films was investigated by RBS. RBS spectra of undoped VO_2 films reported no W content, as expected (Figure 3a). Any W present would have been observed at 950MeV (Figure 3a). The corresponding resistivity plot for this sample shows that the temperature of transition (T_t) – defined here as the point on the heating branch with the greatest change in slope – is in the vicinity of 65°C, as shown in Figure 3c. This value is similar to those found in the literature, where T_t is reported to range from 60° to 68°C for polycrystalline films. The hysteresis is about 10°C and a sharp electrical contrast of three orders of magnitude is observed.

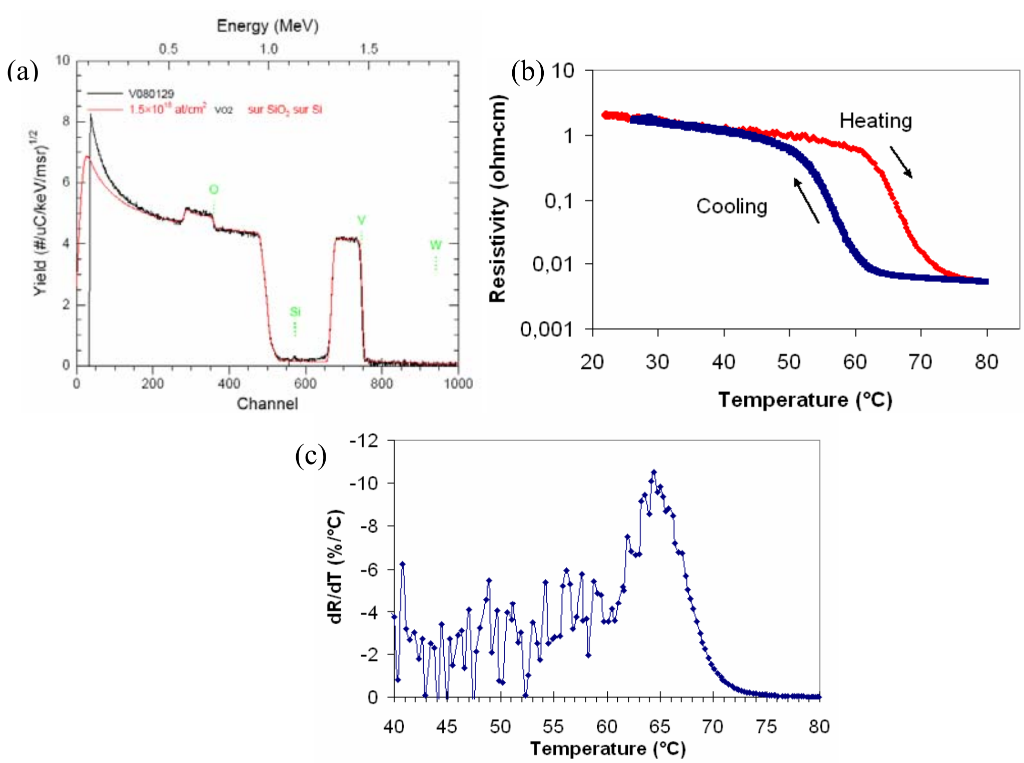

Figure 3: a) RBS spectra of the undoped VO_2 film b) the corresponding electrical resistivity plot shows a drop of 3 orders of magnitude c) the derivative plot shows a T_t around 65°C

The deposition of the $V_{1-x}W_xO_2$ films using the vanadium tungsten (V-W) target with x = 1.5at.% W produced a film with a higher dopant concentration than the alloy target. The dopant concentration found by RBS is 1.8at.% (Figure 4a), corroborating our previous results found by XPS. This time, the variations in resistivity as a function of temperature were recorded with the starting temperature below RT to ensure that the doped film was not tested in the hysteresis area. Based on the relationship described in the literature, where it has been reported that 1at.% of W dopant decreases the VO_2 transition temperature by 23°C, for 1.8at.% W one should expect a T_t around 23°C. On the derivative plot (Figure 4c) we observe peaks at 13°C, 18°C and 28°C. We must specify that our thermocouple was not directly in contact with the coated substrate but was sitting in a groove machined into the surface of the sample holder. Heat dissipation or non-uniformity in the local heating rate may play a more significant role when the starting temperature is below RT. Presence of more than one peak could also simply be caused by local non-uniformity in the stochiometry of the film.

Regarding resistivity following the phase transition, it was noted that the magnitude of the resistivity drop is only half that observed with the undoped film. This illustrates that tailoring the T_t near room temperature is done at the compromise of electrical contrast (and to a lesser extent optical contrast, as we shall see) but electrical and optical contrast are still acceptable for potential applications as a functional part.

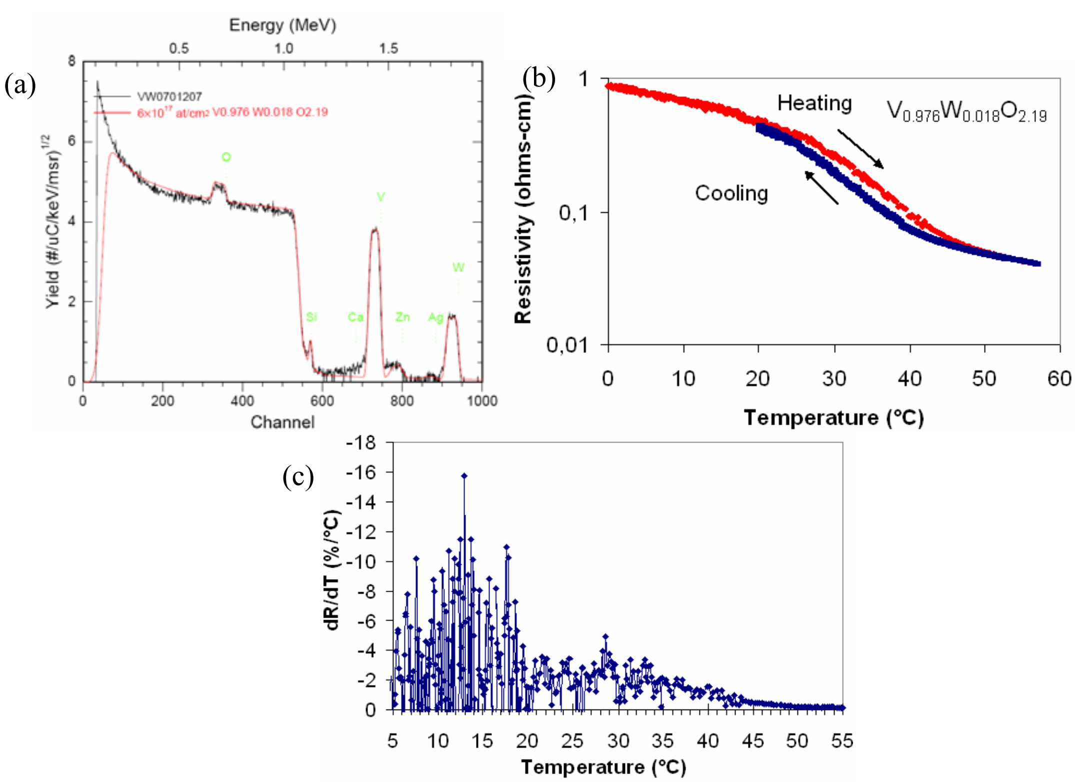

Figure 4: a) RBS spectra for the film deposited with 1.5at.% W target shows a dopant concentration of 1.8at.% W. Ca and Ag peaks are also present and denote trace impurities b) the corresponding electrical resistivity plot shows the amplitude of the drop is half that observed with undoped film c) the derivative used to determine T_t shows peaks at 13°C, 18°C and 28°C.

Films made with the 2at.% W alloy target showed a composition with a surface gradient of 3.3-3.9at.% W, and those made with the 2.5at.% W alloy target had a composition of 4.19at.% W (Figure 5 a & b).

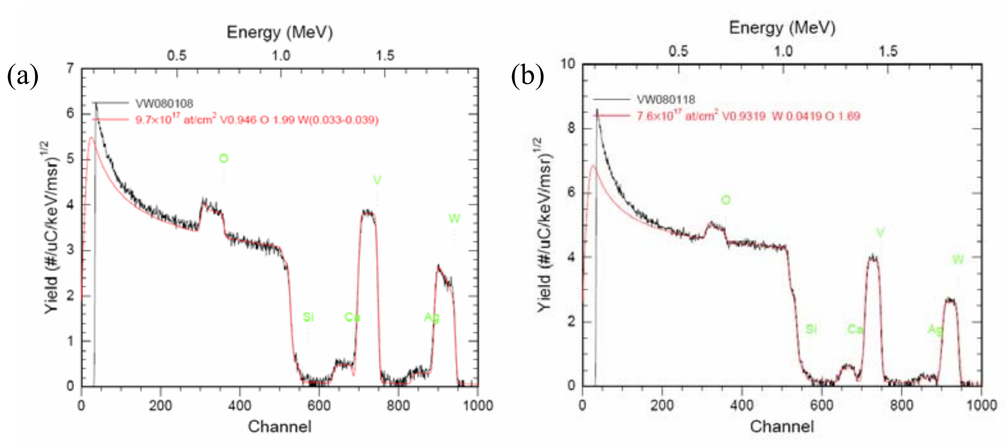

Figure 5: a) RBS spectra for film deposited with 2 at.% W target. A surface gradient of 3.3 to 3.9at.% W is present b) RBS for film with 2.5at.% W target shows 4.19 at.% W concentration. In both cases Ca and Ag peaks are present and denote trace impurities.

These values are significantly different from those expected. The dopant concentration deviation from that of the alloy targets has been reported to be not unusual when using alloy targets. Although the starting material is precisely known in these targets, the sputtering mechanisms that occur during deposition can lead to such variations. The temperature of transition for these films is not what we had initially expected, namely around 0°C for a W content of 2.5at.%. The real W concentration of these films being closer to 4% than to 2%, we would have needed to start below -30°C in order to observe a phase transition, if any. The lowest start temperature we were able to achieve for our experiment was -15°C, and indeed, no phase transition was observed in that temperature range, only a very gradual and linear change in resistivity (Figure 6). These films with a very gradual and small linear change in resistivity in the vicinity of room temperature are less suitable for optical or electrical shutter applications.

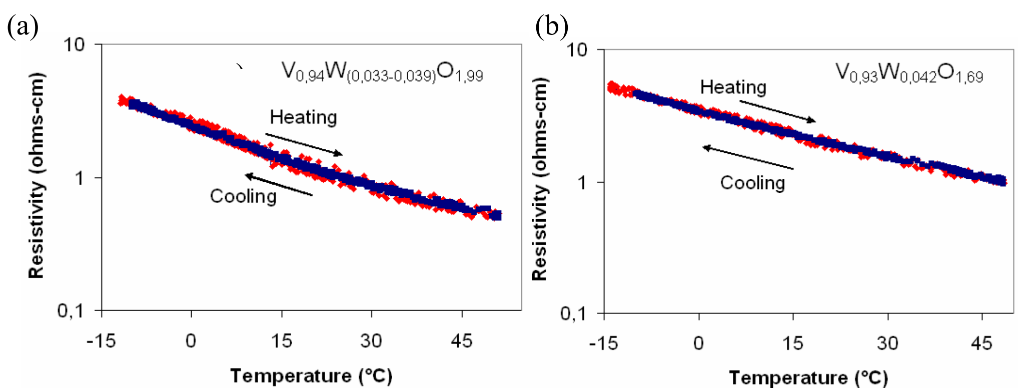

Figure 6) Electrical resistivity plots: a) $V_{0.946}W_{(0.033\text{-}0.039)}O_{1.99}$ and b) $V_{0.93}W_{0.042}O_{1.69}$ showing a very gradual and linear change of resistivity.

Cross-contamination was observed when a sputtering experiment with undoped VO_2 was done just after several sputtering experiments with the 2at.% W alloy target. The first three runs with the undoped target produced films with a temperature of transition very different from the expected ~65°C for undoped films (see Figure 7 a, c, d). These results suggest that the stochiometry of the films included tungsten contamination. After three deposition runs with the undoped target, the film stochiometry showed the expected temperature of transition. No RBS measurements were taken on these involuntarily doped films, but the T_t deduced from the derivative plots (~43°C and ~47°C) led us to suspect that the dopant concentration due to cross-contamination was ~1at.% W (runs 1 and 2) or 0.8at.% W run 3. The drop in the resistivity of these doped films was almost the same as with the undoped film, whereas with 1.8at.%, the magnitude of the drop was much greater. It shows how a slight change in the concentration of dopant can greatly influence the film's electrical properties.

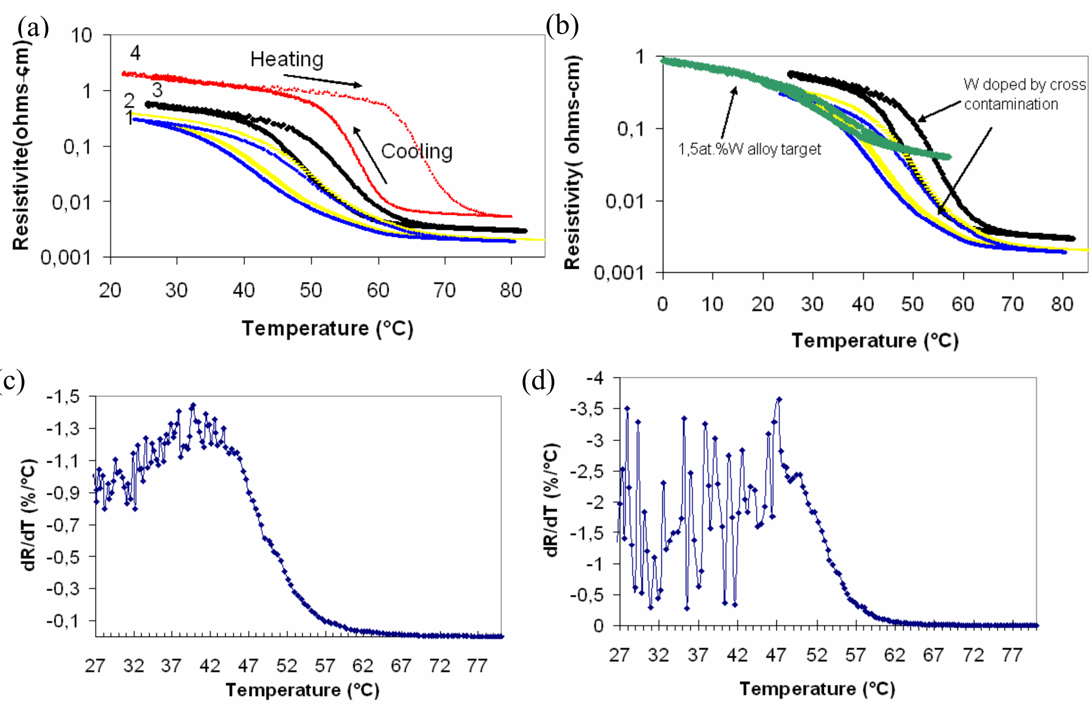

Figure 7a) Electrical resistivity plot shows cross-contamination occurred when deposition of undoped film followed deposition runs with the 2at.% W alloy target. Numbers refer to order of deposition runs b) the drop in resistivity of these involuntarily doped films is similar to that of the undoped film) T_t for films 1 & 2 is ~42°C and that of film 3 is a bit higher, 47°C, as shown with the derivatives

At that point we wanted to show that W cross-contamination can occur between different runs with different target alloy compositions. The specimen holder as well as the entire vacuum chamber receive some deposition material, which is liable to be re-sputtered on the next run. This is an inherent drawback of the sputtering technique which one must bear in mind. One could argue that cross-contamination can also occur between targets with concentrations of 1.5%, 2% or 2.5% dopant. However, our observations show that when the 1.5% target was used, the T_t of the films deposited was reproducible in successive runs. Further investigation of this cross-contamination phenomenon is needed to fully understand it.

4.1.1 Optical properties

The use of a smart material for optical shutter applications is based on its capacity to switch reversibly its optical properties. Transmittance of infrared light at 8 μm was recorded from a sub-zero temperature, i.e., ~-10°C for doped films. The capability to start measurements below RT for the doped films was lacking in our previous work due to the absence of a proper cooling system. This time, a custom-made cooling system was built with perforated Peltier plates coupled to a water cooling system. The temperature regulating setup provided a temperature range of -10°C to 100°C.

The transmittance results reported here are relative transmittance, that is, T/T_0 where T_0 is the transmittance of the uncoated Si/SiO_2 substrate. Figure 8 shows the relationship between transmittance and temperature for the uncoated substrate. It is linear regardless of the temperature; therefore the change in transmittance observed in coated films is solely due to the coating itself. The film is initially highly transparent at room temperature, and at high temperatures it becomes almost fully opaque for the infrared incident radiation. The resulting optical contrast (defined as the percentage transmittance below T_t minus the percentage transmittance above T_t) averages nearly 90% at 8 μm. Our previous study showed that this is also true for the entire LWIR band (8 to 12 μm range). The temperature of transition for transmittance is found to be 65°C (the same as for resistivity) and the hysteresis for

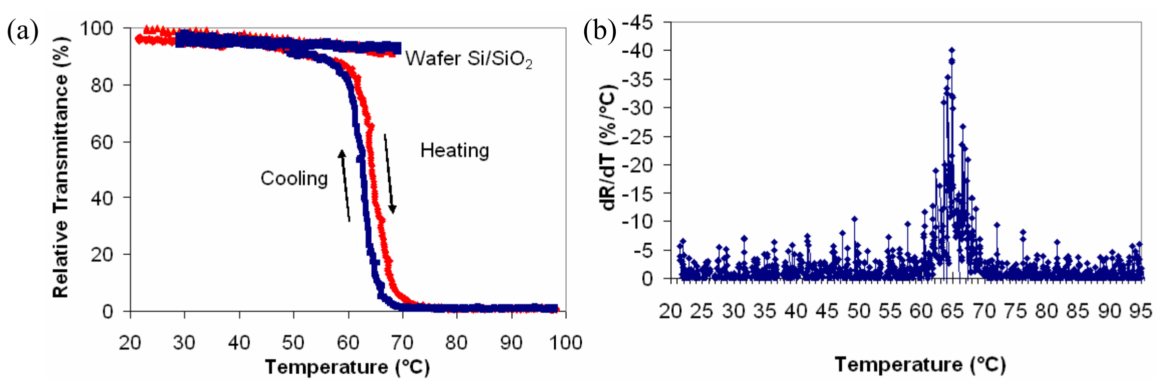

Figure 8: a) Temperature dependence of relative transmittance for undoped VO_2 at λ = 8 μm. The silicon wafer with SiO_2 is the reference substrate for these measurements b) the derivative shows a temperature of transition around 65°C.

For the tungsten-doped films, the three starting temperatures used were -11°C, RT and 10°C. Figure 9 depicts the relationship between transmittance and temperature with the 1.8at.% W-doped VO2 film. The temperature of transition seen is 15°C for the curve started at -11°C, 19°C for the curve started at 10°C, and 22°C for the curve started at RT. The curve started at

RT is considered the least trustworthy as it starts directly in the hysteresis area. The farther the measurements are from the hysteresis, the better it is because this first-order hysteresis zone is known to have second-order hysteresis loops. Therefore the starting point for the measurements should be in the linear portion of the curve. The two plots with a starting temperature outside the hysteresis zone show a value of T_t with a difference of 4°C.

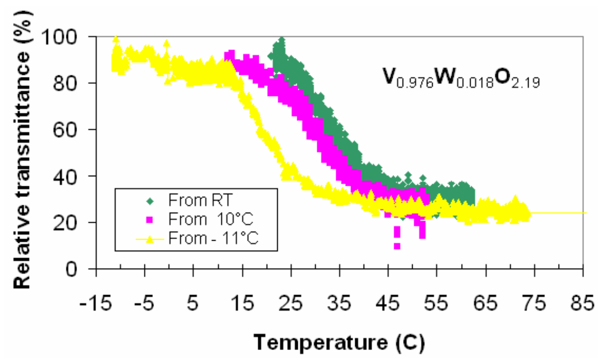

Figure 9: Optical transmittance of 1.8at.% W-doped VO_2 film, Recorded at three different starting temperatures: RT, 10°C and -11°C.

Again, our thermocouple is not directly in contact with the coated substrate but is sitting in a groove machined into surface of the sample holder. Heat dissipation or non-uniformity in the local heating rate may explain the gap of 4°C when the sample holder is cooled to well below 0°C. Overall, there is substantial agreement between the electrical T_t and the optical T_t. The optical contrast of transmittance decreases slightly in comparison to the undoped film: 60% as against ~80%. While the optimal transmittance contrast is achieved within few degrees of the T_t with undoped film, with W-doped films the transmittance starts to decrease at T_t but reaches its lowest transmittance value 20°C later. This is an important factor to consider for doped film in optical switch applications, because one must bear in mind that complete elimination of transmittance will not be achieved at room temperature even though the T_t is at RT.

When over 3.3at.% W dopant is added to the film, we see in Figure 6 that the resistivity slope is linear with no phase transition in the temperature range under study. The same observation is made for the transmittance of 8 µm IR. Figure 10 shows a linear relationship between transmittance and changes in temperature for doped films with over 3.3at.% W. Reflectance measurements for undoped and 1.8at.% W doped films were performed at 8 µm. Films with doping levels >3at.% W were not tested since they undergo no phase transition in the temperature range under study. The reference substrate for the reflectance measurements is a silicon wafer coated with chromium and gold.

(a) (b)

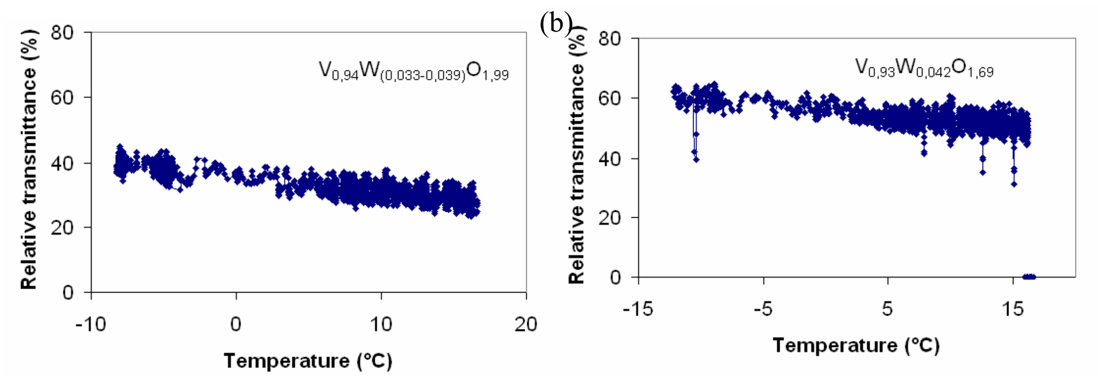

Figure 10: Optical transmittance of doped films: a) 3.3-3.9at.% W and b) 4.2at.% W.

Figure 11 shows the reference substrate reflectance decreasing slightly in a linear way as it is heated to a maximum temperature of 80°C. One challenge encountered in our reflectance measurements is warping. Warping is a distortion of the surface that occurs during a change in temperature due to the differences in thickness and CTE (coefficient of thermal expansion) of the films, substrates and sample holders used. For example, we may expect that the thin film will warp when the film and sample holder are cooled then heated and to the phase transition temperature of the VO_2. Initially, all reflectance measurements were to be started at -10°C.

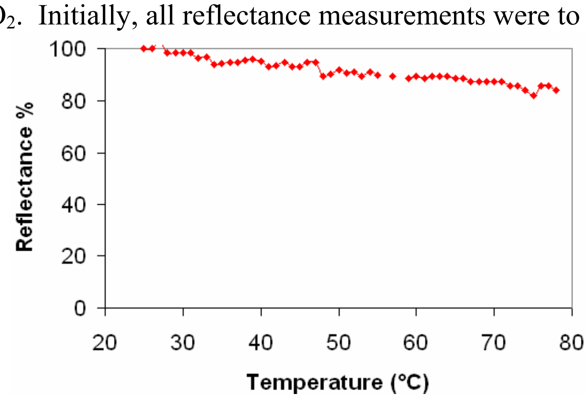

Figure 11: Optical reflectance at 8 μm for reference sample consisting of Cr/Au thin films deposited on Si wafer.

However, persistent warpage problems arose when we cooled the sample holder and then reached the phase transition temperature. We would simply lose the radiation signal, indicating clearly that warpage of the sample, sample holder or both had caused a minute change in the angle of the reflected signal, which affected the signal received by the detector. Warpage is not necessarily linear or isotropic. Sample warpage was observed previously in the 4-point measurements of resistivity. In that case though, pressure is applied to make contact during measurements, so warpage problems could be minimized by choosing probes with larger radius tips to prevent them from piercing the film should it become convex/concave due to warpage. In transmittance measurements, warpage had little impact because the signal transmitted is the signal detected. Therefore, even if warpage is present it does not divert the signal from its optical path to the detector.

Hence, for reflectance measurements we were compelled to do all measurements from RT, as we noticed that warpage of the sample holder/sample setup was minimized at that starting temperature. The sample was always initially placed at the best possible position to get the highest reflected signal at the detector at RT and this position was maintained for the rest of the experiment.

Figure 12a) shows the reflectance of the undoped film; the sample has low reflectance at low temperature (RT) and high reflectance once its phase transition is achieved at high temperature (80°C). The reflectance contrast is about 40% when VO_2 is going through the phase transition and becomes metallic. This value for reflectance contrast is only half of that observed for transmittance contrast. Initial reflectance below T_t is probably due to background reflectance from the underlying Si substrate. When in the metallic phase at a temperature above T_t, we saw that absorption coefficient k increases by a factor of 7 (from less than 0.5 at RT to 3.4 at 80°C) [15]. Such high absorption limits the reflectance contrast that is achievable. A hysteresis loop of 10°C was present.

(a) (b)

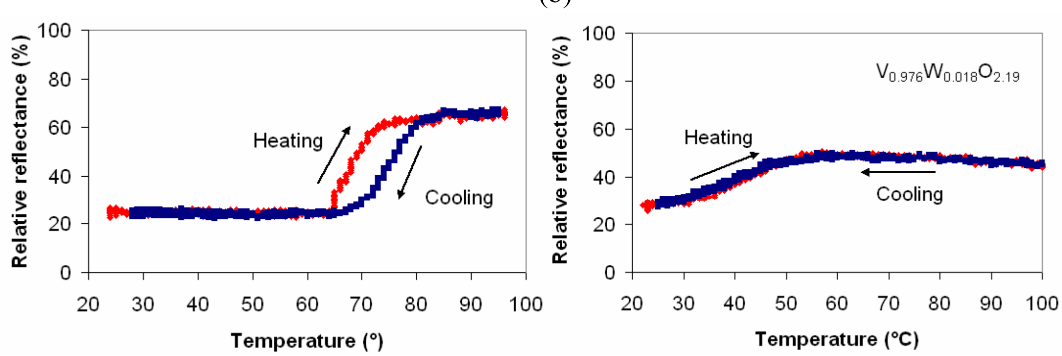

Figure 12: Optical reflectance results at 8 μm for a) undoped sample and b) 1.8at.% W doped film.

The doping effect is again significant for reflectance contrast; it becomes 20% for the 1.8at.% W doped film as against 40% for the undoped film (Figure 12b). The steepness of the drop decreases in comparison to undoped film. On the other hand, hysteresis vanishes completely, as was the case for transmittance.

The effect of long-term air storage on the reflectance of an undoped film is seen in Figure 13. After being in storage for 11 months, the aged film has a few percentage points less reflectance and its T_t rises 3 degrees. This is possibly caused by changes in the stochiometry or composition of VO_2 films.

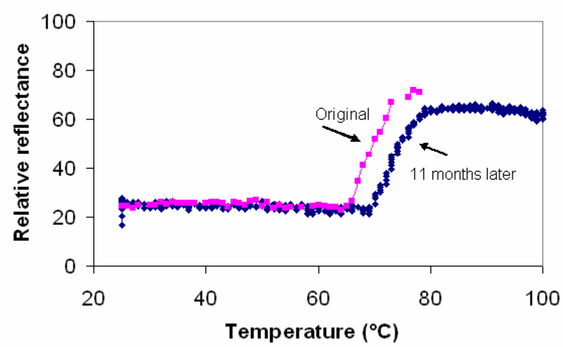

Figure 13: Optical reflectance slopes at 8 μm for undoped sample taken 11 months apart.

4.2 Electrical training

The $Si/SiO_2/VO_2$ stack resistance was monitored during the electrical training (as defined in section 3.1.4), and showed that at least three runs were necessary to achieve a stabilized low $Si/SiO_2/VO_2$ resistance indicating a good ohmic contact under the electrodes. In order to determine whether or not the quality of the film was degraded during the electrical training process, the electrical resistivity of the undoped film was monitored before and after each run. The optical switch (on/off) capability of the film resides in the optical contrast it can offer in transmittance. Therefore, the measurement of transmittance for an incident IR radiation at a wavelength of 10.6 µm was performed simultaneously during the electrical training. What triggers the transition in the VO_2 film of the $Si/SiO_2/VO_2$ stack is the heating of the film above the temperature of transition generated by the Joule effect. Joule heating is the process by which electric current leakage generates heat.

Figure 14 shows no degradation of optical and electrical contrast after the aluminum electrodes are positioned and throughout the successive electrical training runs. Table 1 reports that a minimum of three runs of electrical training were necessary to stabilize the MOS value, which dropped from 4.8 M ohms to less than 35 K ohms. The DC voltage applied across the MOS structure was 15 V for all runs except the first, where 30 V was needed to allow the film to fully reach the transition temperature as shown in Figure 14b. The higher voltage required for the first electrical training run indicates that resistance is highest at the contact level, which illustrates why we need to do electrical training: to achieve better ohmic contact. The transmittance contrast remains over 80% after each run and its temperature of transition was around 70°C (Figure 15).

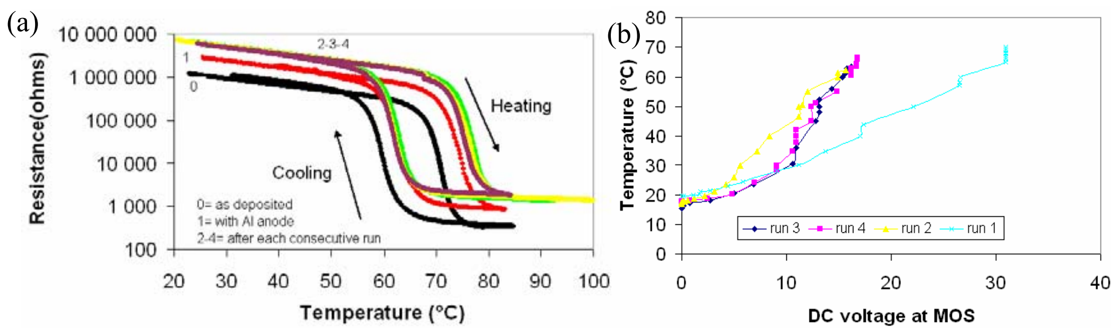

Figure 14: a) Changes in undoped VO_2 film resistance in the course of electrical training. The integrity of the film remains constant after each run. b) Electrical field induces Joule heating of the MOS structure. Nearly 30 V was needed for the first run when the MOS resistance was high. In the subsequent runs, better ohmic contact was in place at the electrode level and voltage required to reach 60°C decreased to 15 V.

Table 1: Electrical training of undoped VO_2 film required three runs to lower and stabilize the MOS resistance value; this indicates good ohmic contacts.

Run	*MOS resistance (ohms)at end of run*	*Transmittance contrast at 10.6 μm*	*Total applied DC voltage (V)*	*Voltage at MOS to see complete phase transition (V)*
0	4.8 M		---	---
1	2.6 M	80-90%	50	30
2	20 K	80-90%	40	15
3	20 K	80-90%	40	15
4	32 K	80-90%	40	15

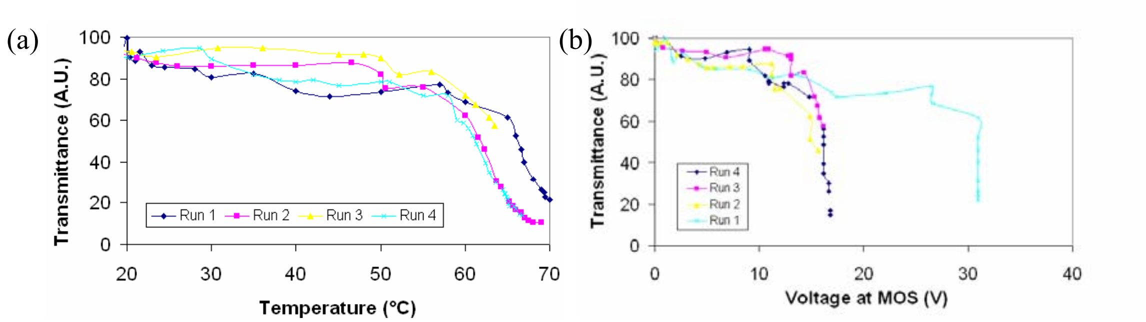

Figure 15: a) Transmittance contrast at 10.6 μm during each run of electrical training for undoped film b) relationship between transmittance and voltage at MOS leve

The electrical training of 1.8at.% W film was done. Addition of a metallic dopant enhances the conductivity of the film; thus the initial starting MOS resistance value was 4.8 MΩ vs 1.6

MΩ for undoped film. Two runs were needed to stabilize the MOS resistance from 1.6 MΩ to less than 25 kΩ as shown in Table 2. No cooling system could be used for the transmittance measurement. Therefore the starting temperature was RT and we acknowledge the fact that the experiment started in the hysteresis area. However, this is not critical here since the purpose of observing the transmittance change was not to determine the precise temperature of transition, but rather to confirm that there was still a phase transition after the successive electrical training runs. No degradation of the transmittance contrast was observed throughout the training process, as shown in Figure 16.

Table 2: Electrical training of VO_2 film with 1.8at.% W dopant required three runs to lower and stabilize the MOS resistance value; this indicates good ohmic contacts.

Run	*MOS resistance (ohms) at the end of the run*	*Transmittance contrast at 10.6 μm*	*Total applied DC voltage (V)*	*Voltage at MOS to see complete phase transition (V)*
0	1.6 M		----	---
1	18 k	80%	20	12
2	20 k	80%	20	12
3	21 k	80%	20	12

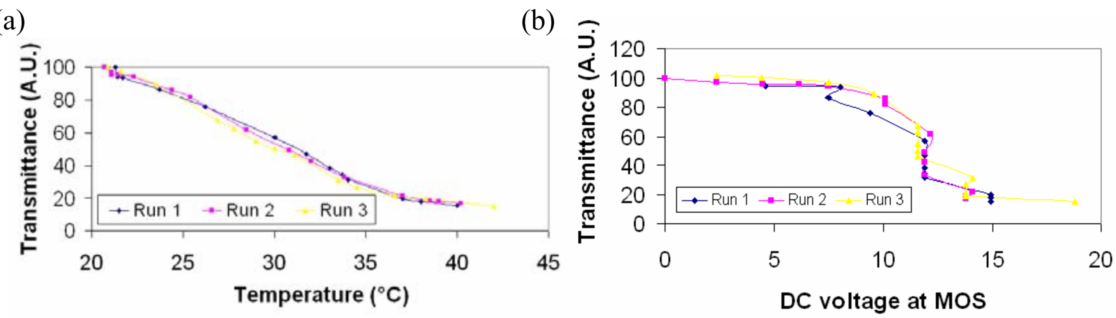

Figure 16: Transmittance contrast at 10.6 μm as observed after each run of electrical training for 1.8at.% W doped film b) relationship between transmittance and voltage at MOS level.

Table 3 summarizes the electrical and optical properties of all our VO_2 films.

Table 3: Summary of semiconductor to metallic phase transition properties for 1 cm² vanadium dioxide films with a thickness of 0.1 to 0.2 μm.

	Undoped VO_2 tested from RT	1.8at.% W doped tested from as low as -15°C	>3.3at.% W tested from -15°C	1.5at.% W + 2at.% Ti tested from RT
Presence of phase transition	yes	yes	no	no
Magnitude of resistivity drop	3 orders	1½ order	linear	linear
Electrical hysteresis	~10°C	~10°C	none	suppressed
Temperature of transition	~65°C	~15-19°C	none observed in this T range	none
Transmittance contrast at 8 μm	~90%	~80%	linear	linear
Hysteresis for transmittance	<10%	<10%	----	suppressed
Delta T needed to fully reach minimum contrast	~10°C	~25°C	not applicable	not applicable
Reflectance contrast at 8 μm	~40%	~20%	-----	-----
Hysteresis for reflectance	~10°C	suppressed	-----	-----
Measured refractive index *n* (at 1-2 μm)	3.4 at RT, 1.6 (above T_t)	-----	-----	-----
Measured absorption coefficient *k* (at 1-2 μm)	0.5 at RT, 3.4 above T_t	-----	-----	-----
Runs of electrical training needed to stabilize MOS resistance	3	2	------	----
DC voltage needed to trigger phase transition	30 V, then 15 V	12 V	-----	-----
Transmittance contrast at 10.6 μm triggered by field effect	~90%	~80%	-----	-----

5. Conclusions

Vanadium dioxide is a smart material that is an excellent candidate for applications where modulation of infrared radiation is needed. Data presented here and in our previous publications support the finding that it is feasible to tailor the transition temperature and still achieve good transmittance and reflectance contrast properties.

Tungsten (W) dopant was used to tailor the optical properties of VO_2. Films with concentrations of 1.8, 3.3 and 4.1at.% W were studied. The results indicated a shift of T_t from 65°C with undoped film to room temperature (~19°C) with 1.8at.% W doped film. The resistivity drop following phase transition for the 1.8at.% W films is 1.5 orders of magnitude compared to 3 orders of magnitude for the undoped films. The sharpness of the electrical and optical transition is slightly degraded in comparison to undoped films, suggesting that compromises must be made between the optical requirements in a specific application and the desired temperature of transition. With concentrations of >3.3at.% W, no phase transition was seen in the temperature range under study for these films, that is, -15°C to 45°C. For an incident wavelength of 8 µm, undoped films have optical transmittance and reflectance contrasts of ~80% and ~40%, respectively. For the 1.8at.% W doped films, the transmittance and reflectance contrast are ~60% and ~20%, respectively.

This report completes our initial published results on fabrication, characterization and tailoring of VO_2 films. The next step will be to evaluate the feasibility of a tunable uncooled infrared detector where the reversibility of optical properties of VO_2 would permit the creation of an imager that has the capability to select a particular infrared band. This multispectral imaging capability is desirable to provide a spectral choice according to battlefield conditions and situations (e.g. 3 to 5 µm in hot/wet conditions and 8 to 12 µm in cold/haze), and would have a higher probability of detection and recognition of camouflaged or lightly concealed targets. This project is currently under study and results should be published in the near future.

6. References

1 Morin, J.(1959).Oxides which show a metal-to-insulator transition at the Neel temperature. *Physical Review Letters*, 3, 34-36.

2 Chen S., Hong M., X. Yi, T. Xiong, H. Wang C. Ke (2004).Smart VO2 thin film for protection of sensitive infrared detectors from strong laser radiation. *Sensors and Actuators A*, 115, 28-31.

3 Laou, P.(2004).TIF project: Miniature IR spectrometer.

4 Laou, P. and Paradis, S.(2007). Proposal: Tunable uncooled infrared detectors. *TM 2007-001.* DRDC Valcartier.

5 Kruzelecky, R. V.(July 2003). Passive Dynamically Variable Thin-Film Smart Radiator Device. *Society of Automotive Engineers,* Document Number: 2003-01-2472.

6 Parkin, I.P. and Manning, T.D. (2006). Intelligent thermochromic windows. *Journal of chemical education*, 83 (3) 393-400.

7 Stefanovich, G., Pergament, A., Stefanovich, D. (2000). Electrical switching and Mott transition in VO2. *J. Physics:condens. Matter*, 12, 8837-8845.

8 Xu, S.-Q., Ma, H-P., Dai, S.-X., Jiang, Z.-H. (2003). Switching properties and phase transition mechanism of Mo^{6+}-doped vanadium dioxide thin film. *Chin. Phys. Lett*., 20 (1),148-150.

9 Swann, J.T., De Smet, D.J. (1985). Ellipsometric investigation of vanadium dioxide films. *J.Appl.Phys*., 58(3),1335-1338.

10 Greenberg, C. B. (1983). Undoped and doped VO2 films grown from VO(OC3H7)3, *Thin solid films*, 110 73-82.

11 Lee, J.C., Jorgenson, G.V. and Lin, R.J. (1986). Thermochromic materials Research for Optical switching. *SPIE* 692, 2-7.

12 Béteille, F., Morineau, R., Livage, J. and Nagano, M. (1997). Switching properties of V1-xTixO2 thin films deposited from alkoxides. *Materials research bulletin* 32(8),1109-1117.

13 Soltani, M., Chaker, M., Haddad, E., Kruzelecky, R.V. and Margot, J. (2004). Effects of Ti-W codoping on the optical and electrical switching of vanadium dioxide thin films grown by a reactive pulsed laser deposition. *Applied Physics Letters*, 85 (11), 1958-1960.

14 Paradis,S. (2005). Étude du VO2 déposé par pulvérisation magnétron RF comme matériau fonctionnel pour la commutation IR. *TN 2005-375*, DRDC Valcartier.

15 Paradis, S., Laou, P., Mérel, P., Alain, D. (2006). Vanadium oxide films for optical modulation applications. In *Proceedings of SPIE: International Conference on Application of Photonic Technology: Photonic Devices*. Québec, Canada.

16 Paradis, S., Laou, P., Alain, D.(2007). Doped vanadium dioxide with enhanced infrared modulation *TM 2007-002.* DRDC Valcartier.

17 Pergament, A. L.,Stefanovitch G.B.,Chudnovski, F.A. (1994). Metal-semiconductor phase transition and switching effect in oxides of transition metals. *Phys. Solid State*, 36 (10), 1590-1597.

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Vanadium dioxide (VO2) is a smart material that offers substantial potential to modulate incident infrared radiation. Its property changes upon phase transition make it an attractive solution as a non-mechanical optical/electrical shutter or functional component in electro-optic systems. The purpose of this document is to report our latest results on the fabrication and characterization of the smart material VO2 deposited by magnetron radio-frequency sputtering technique. The specific properties under study are the temperature of phase transition Tt, electrical resistivity, and optical transmittance and reflectance in the longwave infrared (LWIR) range. Tungsten (W) dopant was used to tailor the optical properties of VO2. Films with tungsten concentrations of 1.8, 3.3 and 4.1at.% were studied. The results indicate a shift of Tt from 65°C for undoped film to room temperature (~19°C) for the 1.8at.% W film. The resistivity drop after phase transition for the 1.8at.% W films is 1.5 orders of magnitude compared to 3 orders of magnitude for the undoped films. With concentrations of >3.3at.% W, no phase transition was seen in the temperature range under study. For undoped films, the optical transmittance and reflectance contrast (namely the percentage of transmittance below Tt minus the percentage of the transmittance above Tt) at an IR wavelength of 8□m are ~80% and ~ 40%, respectively. These values are relative to uncoated silicon wafer. For the 1.8at.% W films, the transmittance and reflectance contrast is ~60% and ~20%. Films with more than 3.3at.% W showed no phase transition, only a linear and small decrease of transmittance and reflectance in the temperature range under study. Electrical training on the undoped and 1.8at.% W films shows that good ohmic contact is achieved after 2-3 training runs with no degradation of the film's optical and electrical properties. The additional results of this study supplement our previously published results, and confirm that tailoring the temperature of transition of VO2 to room temperature using 1.8at.% tungsten dopant can be done with reasonable compromises on optical properties.

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optical shutter, vanadium dioxide, IR modulation, doped thin films, smart material

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