



## Bi-layered energy efficient coatings as transparent heat mirrors based on vanadium oxide thin films



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### ABSTRACT

Energy-efficient coatings based on two-layer-structured transparent heat mirrors were fabricated by depositing a vanadium oxide thin film on a silver thin film by thermal evaporation. Transparent heat mirrors are energy efficient coatings designed to save energy in hot climates by allowing only the visible part of solar radiation to pass through them and reflecting the infrared heat. These transparent heat mirrors can be used as energy-saving windows in energy-efficient buildings and green house agriculture. First, the optical, structural, and chemical properties of individual vanadium oxide thin films were investigated related to their application in transparent heat mirrors. Then, in order to realize their potential as energy efficient coating, optical characterization was performed on a two-layer structure. The performance of these coatings was calculated through integrated spectral transmittance and reflectance in the range of visible and infrared radiations. In addition, chemical depth profiling was performed to probe the diffusion of elements along the deposited layers. The fabricated coatings were found to show the expected behavior as transparent heat mirrors of high visible transparency and high infrared reflection.

### 1. Introduction

Multivalency, thermal and chemical stability, and excellent thermoelectric properties are characteristics that make vanadium oxide a favorable material for window glazing, optoelectronic electrochemical, and microelectronic devices [1–13].  $V_2O_5$  thin films have found diverse applications in technological fields, such as thermal and phase dependent electrical switches, optical switches, lithium batteries, electronic information displays, artificial muscles, smart windows, memory devices, and infrared detectors [1–8]. Vanadium oxide thin films have been fabricated using various deposition techniques, such as thermal oxidation of vanadium [1,5], thermal evaporation [2,10,11], spray pyrolysis [3], sputtering [4,12], electron beam evaporation [5–7,9], chemical vapor deposition [8], sol-gel deposition [13,14], and pulsed laser deposition [15]. For renewable energy applications,  $V_2O_5$  has the preferred properties of high refractive index and high visible transparency. The range of band gap values reported in literature for  $V_2O_5$  thin films is 2.04–3.25 eV [1,5–7], whereas the range of refractive index values reported in literature for pure  $V_2O_5$  thin films is 1.9–2.09 [2,6].

The solar spectrum extended from 400 nm to 3000 nm. The visible portion of the solar spectrum is associated to the light from 400 to

700 nm. The part of the solar spectrum below the visible is associated to UV radiation. The other part of solar spectrum from 700 to 3000 nm is associated to infrared (IR) radiation/heat. The home windows designed with ordinary and conventional glass pass almost all types of radiation. For that reason, energy efficient buildings are designed in this way to pass the visible light to the interior but simultaneously reflect the infrared heat back to the exterior. To acquire this design, the windows are glazed with different kinds of the thin films. Such thin film based coatings/windows are called spectrally-selective windows. The multi-layer structure has been established on basis of metal and dielectric thin films to attain the preferred conditions of visible transparency and reflection of the heat, such a type of coating is known as a transparent heat mirror (THM) [16–19].

A number of methods have been employed to reduce the heat absorption by a building and reduce the cooling load. One of them is green vegetation on the roof in order to reduce heat flux. However the problem with such type of structure is that we need to arrange for an extra expense of fuel to light up the building. In addition to green roof, the reflective roof was also used for the same purpose to reflect heat, but the problem with such type of roof is the loss of reflectivity due to dust and weather conditions [20,21]. Therefore, a need of a protected

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coating which is unaffected by weather conditions and has a sufficient passage of visible-light inside the building was inevitable. Vanadium oxide is a potential candidate for tunable window glazing due to its excellent electrochromic and thermo-chromic properties [22,23]. Electrochromic coatings are materials which change their optical properties by applying an electric field in one direction and attain their original state by applying the electric field in the reverse direction. They become either partially transparent or more reflective during this transition. The other advantages are: they require small power (power required for switching only), long term memory, specular during all states and ease of fabrication [22]. The large refractive index of  $V_2O_5$  make it suitable for use in antireflective coatings. Moreover, its refractive index can be tuned by layering with other materials [24]. Smaller refractive indices of the dielectric allow wider transmission range at the cost of a more gradual transition and smaller IR reflectance. [25]. Therefore a high refractive index dielectric is very suitable for antireflective coatings as the low values of refractive index of dielectric layer do not anti-reflect the metal layer properly. These controllable properties can be combined with energy efficiency properties of  $V_2O_5$  to make it a unique material for tunable windows.

In this work, a two-layer metal/dielectric structure has been used for the fabrication of transparent heat mirrors. Silver (Ag) was used as the metal and vanadium oxide ( $V_2O_5$ ) was used as the dielectric to enhance the visible transmission and stability of the THM. Optimum values of thickness were used in order to achieve required optical properties. In order to achieve a high IR reflection, the metal film should be as thick as possible. To increase visible transmittance, the thickness of the metal layer should be as small as possible. However, below a critical thickness, the IR reflectance will be degraded [17]. The transmittance band width becomes narrow as the thickness of the metal (silver) increases. However, the infrared reflectance increases with thickness [26]. The selection criterion for a dielectric with refractive index ( $n_D$ ) for any metal with extinction coefficient ( $k_M$ ) used in a THM within desired spectral range is given as: In order to achieve optimum performance, the  $n_D$  should closely match the  $k_M$  in a given spectral range [26]. Comprehensive analysis showed that dielectric thickness must be equal to  $\lambda/8n_D$ . But in most cases,  $n_D < k_M$ , therefore the thickness of the dielectric must be greater than  $\lambda/8n_D$ , and the metal should be thinner than given value [26,27]. Leftheriotis et al. [28] showed that the thickness of the dielectric layer can be changed in order to optimize transmittance without any undue changes in the IR reflectivity. They observed that deviations from the optimum thickness of the dielectric caused by errors in film deposition method are not expected to have a significant effect on the film performance. However the best results were obtained when the two dielectric layers have the same thickness. Fig. 1 shows the THM based on two-layer structure. The inspiration behind this work was to provide a new material that is suitable for energy efficient coatings, and economically affordable. This type of coating will not only save the electricity but also reduce the pollution in our environment. The novelty of this work is demonstrated in two ways. First,  $V_2O_5$  thin films have not been studied yet for applications in THMs. Second, instead of three layers, a two-layer structure was investigated for THM applications. This would provide not only ease of fabrication but also cost-effective windows for future buildings.

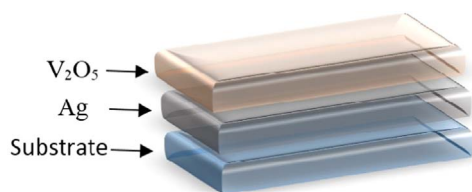


Fig. 1. Schematic diagram of a transparent heat mirror using  $V_2O_5$  thin film (dielectric) over an Ag film (reflector).

## 2. Experimental details

$V_2O_5$  and Ag thin films were prepared by thermal evaporation in a Leybold model L560 box coater that was pumped to  $3 \times 10^{-5}$  mbar base pressure. Fused silica and tantalum substrates were used to deposit the films in order to study their optical and chemical properties. Substrate rotation was ensured for smooth fabrication of the films during deposition at constant rate, where the source-to-substrate distance was maintained at 40 cm. The substrates were maintained at ambient temperature. Source material ( $V_2O_5$  powder) of high purity (99.995%) was evaporated from a tungsten boat. The rate of evaporation of 0.1 nm/s and final thickness (between 35 and 160 nm) were controlled and monitored using a quartz crystal thickness monitor and rate controller. For the THM ( $V_2O_5/Ag$ ), the  $V_2O_5$  (40 nm) was coated over the silver film by the same procedure described above. A high quality silver powder (Alfa Aesar, 99.999%) was deposited over the substrate with a thickness of 40 nm. A molybdenum boat was used for thermal evaporation of silver with a constant evaporation rate of 0.2 nm/s. The bilayer coatings for THM were fabricated without breaking the vacuum.

To study the physicochemical properties and performance of the individual films and THMs, a number of characterization techniques were employed. X-ray photoelectron spectroscopy (XPS) was used to study the chemical composition of the individual films and THM. Thermo Scientific Escalab 250Xi spectrometer with a monochromatic X-ray source Al  $K\alpha$  (1486.6 eV) was used for this purpose. The resolution of the instrument was 0.5 eV. Ambient temperature and a pressure of  $5 \times 10^{-10}$  mbar was maintained during XPS analysis. The adventitious C 1s peak is used as reference of binding energy at 284.5 eV. THM based on  $V_2O_5/Ag$  layers was investigated through elemental depth profiling, where XPS measurements were taken sequentially after each 20-s ion etching. The ion gun with 2-keV Ar<sup>+</sup> ion beam focused on a 1-mm<sup>2</sup> surface area of the coating was used for etching. The chamber pressure was maintained at  $3 \times 10^{-8}$  mbar during the experiment. The ion current was kept less than 1  $\mu$ A at the sample surface during the depth profiling. The complete etching of film took twelve cycles of profiling. In order to overcome and reduce the charging effect due to non-conducting samples, the films were deposited over the tantalum substrates. Atomic force microscopy (AFM) (Veeco Innova diSPM) was used in contact mode in order to study the surface morphology of the individual films of vanadium oxide and silver. A silicon tip of 10 nm radius was used to probe the surface of the samples. The tip was oscillating at 300 kHz (resonant frequency) during the experiment with a scan rate of 2 Hz over a scanning surface area of  $2 \times 2 \mu\text{m}^2$ . X-ray diffractometer (Rigaku Ultima IV) was used for x-ray diffraction (XRD) in the  $\theta$ - $2\theta$  configuration using Cu  $K\alpha$  radiation ( $\lambda_{Cu} = 1.54 \text{ \AA}$ ), in order to investigate the structural properties of the films. The  $2\theta$  step and the step acquisition time were  $0.02^\circ$  and 1.00 s, respectively. Jasco V-570 spectrophotometer was used to study the optical properties of the individual  $V_2O_5$  films and their THMs. The normal-incidence transmittance ( $T$ ) of individual films was measured in the range of 200–800 nm, whereas normal-incidence reflectance ( $R$ ) and transmittance spectra of  $V_2O_5/Ag$  based THM were measured in the range of 200–2000 nm.

## 3. Result and discussion

### 3.1. $V_2O_5$ film

Fig. 2(a) shows the XRD pattern of a vanadium oxide ( $V_2O_5$ ) thin film. The pattern depicts a broad peak, characteristic of an amorphous structure, that was due to the fused silica substrate. No reflection due to  $V_2O_5$  was observed, indicating that the as-deposited vanadium oxide thin films were amorphous, this is because the kinetics energy of evaporated species is small and lower than the energy necessary for initiation of crystalline growth. Amorphous films are usually characterized by smoother surfaces. For this particular application, it is desirable

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