Improved Thermal Radiative Properties of Tungsten Doped VO2 Thin Films Grown by High-Temperature Oxidation and Reduction Processes in Low-Oxygen Environment

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Thermochromic vanadium dioxide thin films have attracted much attention recently for constructing variable-emittance coatings upon its insulator-metal phase transition around 68ºC for dynamic thermal control applications. The phase transition temperature can be lowered to room temperature or even below with a tungsten doping method. However, most VO_2 thin films prepared in rich-oxygen environment such as commonly-adopted reactive magnetron sputtering and our previously-reported furnace oxidation methods suffer from the surface over-oxidation. This leads to higher infrared transmission for semitransparent metallic VO_2 thin film around 10 µm wavelength and higher infrared emissivity from variable-emittance coatings made with metallic VO_2 film, which diminishes the variation in transmittance or emittance upon the phase transition from expected. In order to minimize and ideally eliminate the surface over-oxidation, we propose to oxidize the vanadium films in low-oxygen environment with O_2 partial pressure around 1 Pa via constant nitrogen gas purging followed by a surface reduction process with O_2 partial pressure less than 0.05 Pa. Systematic studies on the nitrogen purging rate (0.5 ~ 4 liter per minute), oxidation time (0.5 ~ 3 hrs), and oxidation temperatures (300°C ~ 700°C) are conducted experimentally for vanadium thin films with different thicknesses (15 ~ 50 nm) sputtered onto intrinsic silicon wafers. Reduction at the same oxidation temperature with different time periods (10 ~ 60 mins) is also tested for achieving the optimal VO_2 quality with surface over-oxidation removed. Tungsten doped vanadium films with doping levels of 1%, 2% and 3% are also prepared from W/V alloy sputtering targets with the same thicknesses along with the undoped vanadium films. SEM and XRD characterizations will be done for examining the surface morphology and crystallinity of undoped VO_2 films prepared at different temperatures, while XPS measurement will be conducted to confirm the tungsten doping levels in doped VO_2 thin films. Temperature dependent infrared transmittance from room temperature to 100°C of all undoped and doped VO_2 samples with different thicknesses will be measured with an FTIR spectrometer equipped with a home-built sample heater. By coating the backside of the intrinsic silicon wafers with aluminum, the undoped and doped VO2 samples become opaque, and their temperature dependent infrared reflectance from 5 °C to 100 °C will be measured by the FTIR equipped with a specular reflection accessory along with a home-built temperature stage. To fully reveal the behaviors of doped VO2 thin films with the transition temperature close or lower than the room temperature, a cryostat is used along with the FTIR to measure their infrared transmittance from -50 °C to 100 °C. The dielectric functions of the undoped and doped VO_2 thin films will be fitted from these temperature-dependent transmission and reflection measurements. Finally, variable-emittance coatings made of a Fabry-Perot structure with undoped and tungsten doped VO_2 thin films will be fabricated from both rich- and low-oxygen furnace oxidation processes. The enlarged variation in infrared emissivity upon the phase transition from the tunable coatings prepared by the proposed low-oxygen processes will be experimentally demonstrated by the measured temperature dependent infrared reflectance along with validations from optical modeling.