Characterization of Phase Transition Behavior in VO2 Thin Films

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Introduction.

Vanadium dioxide (VO_2) interests researchers with its ability to transform from an insulator to a metal at a readily achievable temperature of around 67 $^{\circ}$ C. This shift, known as the metal-insulator transition (MIT), triggers a 10,000-fold increase in electrical conductivity.

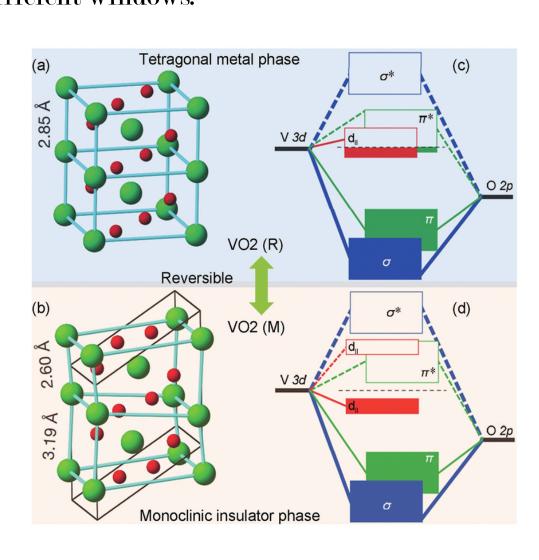
Our research focuses on the controlled production and detailed characterization of high-quality VO₂ thin films. We use a sol-gel approach with standard precursor solutions to create very uniform thin films by spin coating.

Annealing in a custom-built vacuum furnace allows for the tuning of properties of the VO_2 films. To quantitatively examine the MIT and its related characteristics, we use the Van der Pauw four-point-probe approach for electrical resistivity measurements.

By doing this research, we hope to develop a standard procedure for fabricating VO₂ thin films at IWU.

Background

Vanadium dioxide (VO₂) undergoes a well-studied phase transition between solid states triggered by factors like heat, light, or electric fields. This transition leads to significant changes in VO₂'s electrical and optical properties. Across this transition, VO₂'s electrical properties can change by factors of 1,000 to 100,000, while its optical properties vary based on the wavelength of light (increasing in contrast in infrared wavelengths). These properties make VO₂ appealing for applications such as optical switches, holographic storage, hydrogen catalysis, and energy-efficient windows.



Materials or Methods

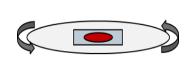
Sol-Gel Process

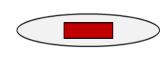
The sol-gel process is a versatile wet chemical technique for producing solid materials from small molecular precursors. It involves the formation of a colloidal solution (sol) and the subsequent gelation of the sol to form a solid gel network. In this work, V_2O_5 (99.9%), and H_2O_2 (30 wt.%) were used without further purification.

Spin Coating

Spin coating is a widely used technique for depositing uniform thin films onto substrates. The sol-gel precursor solution will be dispensed onto clean substrates, which will then be rotated at a speed of 300 rpm to spread the precursor out, then accelerated to a speed of 3000 rpm. The high speed will spread the solution radially outward, resulting in a thin, uniform film deposition. Multiple coating cycles may be employed to achieve the desired film thickness.









Annealing

After spin coating, the as-deposited films underwent a thermal annealing process to promote the formation of the desired VO_2 phase. The films were annealed in a controlled low-pressure oxygen atmosphere at a temperature of around 450° C for 30 minutes. This step is crucial for promoting crystallization of the VO_2 phase (by reducing the V_2O_5 phase).

Four-Point Probe for Resistivity Measurements

The electrical resistivity of the VO₂ thin films were measured using the Van der Pauw four-point probe technique. This method involves bringing four probes into contact with the corners of the film's surface. A current is passed through two adjacent corners, and the voltage drop is measured across the other two corners. By accounting for the film thickness and the probe geometry, the resistivity could be calculated, but without thickness, only the "sheet" resistance can be calculated. By performing this measurement at low temperatures (room temperature) and high temperatures (greater than 70°C), this technique minimizes the effect of contact resistance and provides accurate resistivity measurements, allowing for the characterization of the metal-insulator transition in VO₂.

Results

Film Appearance

The vanadium oxide films had a generally good appearance, but some spotting was visible on the surface. Future studies will evaluate the electrical properties of these thicker films.





Spins	Sintering Temperature	Annealing time	Annealing Temperature
4	150° C	30 minutes	450° C
8	150° C	30 minutes	450° C
8	250° C	30 minutes	450° C

The films sintered at different temperatures showed distinct visual characteristics. Specifically, the film sintered at 250° C had a grayish-green color and a slightly rougher surface, while the film sintered at 150° C appeared more golden-yellow with a smoother texture. The higher sintering temperature not only changed the color but also affected the surface texture of the vanadium oxide coating.

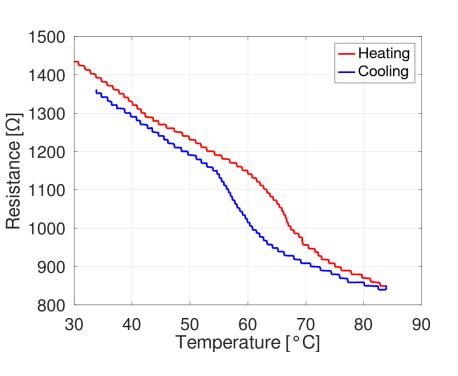
Sheet Resistance

We measured the sheet resistance using a Keithley Model 2400 to both supply a constant current of 100 uA and measure voltage across the sample. We measured the voltage at room temperature and on a hot plate set to 110°C. These voltages were used to calculate the sheet resistances of the various samples at these temperatures, which can be seen in the table below.

	Sample sintered at 150 C	Sample sintered at 250 C
Room Temp (20°C)	8550Ω	4932Ω
Hot Plate (100°C)	4674Ω	1777Ω
D/ D	4.00	0.775

Conclusions

The thickness of the vanadium oxide films prepared by the sol-gel method was found to be a crucial factor in obtaining films suitable for electrical characterization. When the films were spin-coated only 4 times, the thickness was not sufficient for reliable electrical measurements. However, increasing the number of spin-coating cycles to 8 resulted in films with adequate thickness, enabling further electrical characterization to be performed.



Acknowledgements

Wyatt Broers developed a system we used to measure our resistance as a function of temperature.

Literature cited

[11] N. Wang, S. Magdassi, D. Mandler, and Y. Long, Simple Sol—Gel Process and One-Step Annealing of Vanadium Dioxide Thin Films: Synthesis and Thermochromic Properties, Thin Solid Films 534, 594 (2013).

Future Work

Further investigations are needed to explore surface treatments or modifications of the glass substrates prior to film deposition. Such treatments may improve the film quality and uniformity, potentially addressing the issue of spotting observed in the current films. Additionally, surface treatments could enhance the adhesion and interfacial properties between the vanadium oxide films and the glass substrates, which is essential for various applications.

