Recent advance in phase transition of vanadium oxide based solar reflectors and the fabrication progress

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ABSTRACT

Vanadium dioxide (VO₂) as a phase-change material controls the transferred heat during phase transition process between metal and insulator states. At temperature above 68 °C, the rutile structure VO₂ keeps the heat out and increases the IR radiation reflectivity, while at the lower temperature the monoclinic structure VO₂ acts as the transparent material and increase the transmission radiation. In this paper, we first present the metal-insulator phase transition (MIT) of the VO₂ in high and low temperatures. Then we simulate the meta-surface VO₂ of metamaterial reflector by Ansys HFSS to show the emittance tunability (Δε) of the rutile and monoclinic phase of the VO₂. In next section, we will review the recent progress in the deposition of thermochromic VO₂ on glass and silicon substrate with modifying the pressure of sputtering gases and temperature of the substrate. Finally, we present the results of the in-situ sputtered VOₓ thin film on thick SiO₂ substrate in different combination of oxygen and argon environment by V₂O₅ target at temperature higher than 300°C and then, analyze it with x-ray diffraction (XRD) method. The thermochromic VO₂ based metamaterial structures open a new route to the passive energy-efficient optical solar reflector in the past few years.

Keywords: Nanofabrication, Phase Transition Metamaterial, V₂O₅ target, Optical Solar Reflector, Vanadium Oxide

1. INTRODUCTION

The reversible metal-insulator phase transition (MIT) of VO₂ has gained enormous attention in thermal control system of spacecrafts recently. However, among other metamaterials great effort is still investigated to understand the MIT process. In this section we review the recent advance in MIT mechanism of VO₂ and discuss the physical properties of the rutile and monoclinic structures. V-O system is one of the most interesting materials with different compounds including VO, VO₂, V₂O₃, V₃O₅, etc. Some of these components show metal-insulator transition (MIT) with a big change in thermal, optical, or electrical properties. These special properties encouraged scientists to use the V-O system in many optical applications and therefore, learn the phase transition process. Among all various types of V-O system, VO₂ is considered for the temperature phase change mechanism [1, 2, 3]. Today, great effort has been made to understand the MIT phase change mechanism on various VO₂ morphologies acquired from different types of fabrication methods, [4, 5, 6]. Despite the great progress has been made in recent years, the phase transition is still an arguable mechanism. The rapid transition between rutile metallic phase and monoclinic insulator phase is followed by changes in the crystallographic and electrical properties of VO₂, accomplished by switching and sensing applications [7].

The isolator behavior of VO₂ with 3d₁ orbital occupancy in room temperature and, forming other isolator phases in specific temperatures, all make it hard to interpret the phase change properties. One explanation for this change can be described by Peierls mechanism and electron correlation technique, which address the lattice distortion and the band gap shift. On the other hand, in the 3d t₂g-derived states of the vanadium, when d₁ bands break into occupied

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bonding and empty antibonding, the $\pi^*$ states get depopulated and their energy increase that leads to create the bandgap. Therefore, a bandgap is created between the $d$ and the $\pi^*$ bands, illustrated in Figure 1. However, bandgap disappear in rutile phase to allow reflection of radiations. Therefore, we explain the lattice structures and electron interaction to learn more about the transition process.

![Figure 1. Bandgap in monoclinic (left) and rutile (right) phase of V](image)

The VO$_2$ can grow in the form of different crystal structures, including monoclinic (M) and rutile (R) phases which show reversible phase change close to room temperature. During the phase change, the d-electrons of V atoms in the symmetrical structure join to the varying V-V bonds in low symmetry monoclinic structure. There is an assumption that this V-V localized structure derived from the high temperature delocalized phase and shows insulative properties. On the other hand, the conductivity property in the high temperature rutile phase is due to the fermi level between the $\pi^*$ band and the $d\parallel$ band. While in M phase, the $d\parallel$ band split to two parts, make a gap between $d\parallel$ band and the $\pi^*$ band where Fermi level falls into that and lead to VO$_2$ to be insulative. However, this theory is no clarified yet, since some research show that lattice distortion is not the only reason for MIT phase transition. One hypothesis is that there are other phases rather than M, at the low temperature. With the help of doping, electron injection or stress effect, different phases of VO$_2$ like M2 monoclinic, triclinic VO$_2$ (T) or other metastable monoclinic metal phases are discovered, which indicated that V-V bond interactions is not the necessary step to open an insulating gap [1, 8, 9, 10]. Studying the transition phase of thermochromic VO$_2$ makes a new path to understand the correlation effects of other thermal sensors [11, 12, 13].

![Figure 2. Vanadium dioxide semiconductor (left) to metal (right) phase transition at 68 °C shows change in infrared reflectivity](image)

As a thermochromic material, VO$_2$ goes through semiconductor to metal phase transition at the specific temperature of 68 °C which makes the change in infrared reflectivity, as shown in Figure 2. The transmission radiation changes with temperature and thickness of the VO$_2$ material, which is demonstrated in Figure 3. The broadest extensive hysteresis transition belongs to the thickest VO$_2$ with 50nm. By increasing the temperature above 68 °C at wavelength
of 2 µm, the material keeps the heat out and IR radiation reflection increased since little radiation is transmitted into. To manipulate the transition temperature of the VO₂ film different method were introduces including the adding various dopants, using multiple layers, or size effectiveness of VO₂ lattice [14, 15, 16, 17].

Figure 3. Transmission hysteresis loops for different thickness of VO₂ thin film at wavelength 2µm [14]

2. NUMERICAL SIMULATION OF FILTER

In [18], the optical solar reflector (OSR) composed of plasmonic VO₂ meta-surface is introduced to control the temperature of the satellite devices. The grating thermo-chromic VO₂ in the OSR filter improve the absorption (α) and emittance tunability Δε with the help of plasmonic and thermo-chromic properties of VO₂. This leads to higher absorption of the structure in higher temperature and lower absorption in temperature below the critical level. The smart meta-surface reflector shows higher tunability Δε at lower absorbance in comparison with the planar thin film design [19]. The OSR reflects the radiation of the sun and dissipates the thermal spectrum of onboard instruments to reduce the solar absorption and keep down the thermal fluctuation of the satellite. In contrast with active systems, the smart passive systems keep the heat at low temperature (low emittance) and dissipates extra heat at high temperature (high emittance) to keep the temperature at optimal ranges. This makes the passive systems cost and energy effective with lower weight. In passive thermal control structures, thermo-chromic VO₂ works as a reflector and shows semiconductor to metal phase change at specific temperature 68°C with high tunability of emittance. The critical temperature can be manipulated by doping or defect engineering methods to be near room temperature. The performance of the passive smart system can be explained by the dynamic emittance tunability of Δε = ε_hot - ε_cold. VO₂ based metamaterials exhibits phase transmission on plasmonic effects that shows higher absorption in metallic state and lower emittance in monoclinic phase, applicable over a wide range from visible and NIR to MWIR.

The insulator SiO₂ in [18] is sandwiched between the meta-surface VO₂ and aluminum reflector, shown in Figure 4, makes the destructive interference effect to improve the blackbody spectrum absorption and high tunability Δε. The Al₂O₃ layer is considered as adhesion material.

Figure 4. VO₂ based OSR Schematic Simulated in HFSS
The proposed structure is composed of the array of metasurface VO\(_2\) in the shape of the square grating and simulated by Ansys HFSS, demonstrated in Figure 4. The absorption infrared spectra of the monoclinic grating VO\(_2\) with grating squares size 2.8\(\mu\)m and different gaps between grating square changes from 3.3-4.35\(\mu\)m at temperature 30°C is low at 5-7\(\mu\)m and 12-18\(\mu\)m which is illustrated in Figure 5.

![Absorption infrared spectra of the monoclinic grating VO\(_2\)](image)

The emittance of a reflector is described in Eq. 1 as the average emittance weighted by blackbody spectrum at temperature (T).

\[
\varepsilon = \frac{\int (1-R(\lambda))B(\lambda, T)d\lambda}{B(\lambda, T)d\lambda}
\]  \[1\] [18]

Using Eq 1, the emittance of the meta-surface VO\(_2\) with low-emissivity dielectric spacer in high and low temperature is calculated by the measuring the reflection spectra from FTIR method, as shown in Figure 6 (a). Therefore, it’s not hard to find the emittance tunability \(\Delta \varepsilon\) of the meta-surface VO\(_2\) at the peak of the blackbody spectrum, which is plotted in Figure 6 (b). The latter shows that by increasing the gap size between features (or decreasing the feature size of the grating VO\(_2\)), maximum emittance tunability \(\Delta \varepsilon\) improved about %30 in comparison with the planar film of VO\(_2\).
Figure 6. Measured emittance (left) and emittance tunability (right) of planar film (dash line) and meta-surface VO$_2$ for high and low temperatures [18]

3. REVIEW OF RECENT VO$_2$ DEPOSITION WORKS

In the recent years, different methods were advised to sputter vanadium films [20, 21, 22]. In order to make thermochromic VO$_2$ of metamaterial structures, we review some deposition approaches with the help of modifying the pressure of gases and temperature of the substrate [23, 24, 25]. One method is to sputter thin film with vanadium target and then oxidized it in high temperature annealing environment at temperature 300°C or above. The other popular way is reactive sputtering deposition which is performed with the partial oxygen pressure %0 to %20 of the total pressure (argon/oxygen atmosphere) at 300-600°C to create different mixtures of VO$_x$ (V$_2$O$_3$, V$_2$O$_5$ or VO$_2$) on the substrate. The films can be analyzed with XRD, atomic force microscopy and X-ray photon spectroscopy (XPS) methods, in addition to checking the color of different composition of VO$_x$ visually [26]. Furthermore, post annealing in 300°C or higher is recommended to form various types of VO$_x$ in other works. In [23], different forms of VO$_x$ were deposited on substrate with RF magnetron deposition by V$_2$O$_5$ target at temperature 300 to 527°C without annealing process. At the oxygen partial pressure less than $10^{-15}$ in room temperature in RF magnet deposition process, the VO$_2$ is the most stable phase of the other composition of VO$_x$ from the V$_2$O$_5$ target. The VO$_2$ thin film deposited on the glass substrate at 125W plasma power with the oxygen partial pressure changing from 0-20% of argon/oxygen for 100 minutes. Meanwhile, at the oxygen partial pressure higher than $10^{-29}$, V$_2$O$_5$ grows on the substrate as shown in Figure 7 [23, 27, 28].

Figure 7. Monoclinic (M) and tetragonal (T) VO$_2$ and V$_2$O$_5$ thin films growth from V$_2$O$_5$ target at partial oxygen pressures [23]

Reactive hydrogen is used for Rf sputtering of vanadium film with V$_2$O$_5$ target to reduce oxygen, whereas reactive oxygen is suggested for metallic vanadium or V$_2$O$_3$ target. The V$_2$O$_3$ target provides broader flow ratio during sputtering vanadium oxide, but it is more expensive than other two targets. Furthermore, metallic target gets oxidized during sputtering process, therefore, V$_2$O$_5$ is the most stable and best sputtering target to deposit rutile VO$_2$. However, due to safety requirement, reactive oxygen is recommended with V$_2$O$_5$ target at higher temperature. In [24], rutile VO$_2$ is deposited on fused silica glasses, Si or thick SiO$_2$ on Si substrate from a V$_2$O$_5$ target by tuning substrate temperature and the oxygen flow rate. The temperature of substrate changed from 300 to 500°C during the deposition with the total...
oxygen/argon pressure 12.5 mTorr at the power of 60 or 120W and the oxygen flow ratio \( R_{fo} \) is adjusted between 0.042 to 0.1 based on Eq 2, where \( f \) is the flow rate of the gas.

\[
R_{fo} = \frac{f_{O_2}}{(f_{Ar} + f_{O_2})} \tag{2} \quad [24]
\]

The temperature of the substrate plays an important role for adjusting the oxygen component of the thin film. Figure 8 shows at lower temperature 300°C, the \( V_2O_5 \) phase starts to grow on Si substrate. As temperature increases, \( V_7O_{13} \) and meta-stable phase \( VO_2 \) (A) phases show at \( R_{fo} = 0.1 \) due to less oxygen content of the films. Other phases with lower oxygen grow on the Si substrate when \( R_{fo} \) is decreased. At higher temperature 500°C, only rutile \( VO_2 \) grows on the Si substrate with \( R_{fo} \) between 0.06 and 0.042 in power 60W.

![Figure 8. XRD result of thin films on Si substrate at different temperature from 300°C to 420°C for \( R_{fo} = 0.3 \) [24]](image)

Figure 9 shows that the increasing the temperature of substrate not only form the phases with lower oxygen, but also increase the average grain size of the deposited thin film.

![Figure 9. AFM results for grain sizes of mixed rutile \( VO_2 \) and \( VO_2(A) \) phases at T = 450°C (left) vs single phase rutile \( VO_2 \) film at T = 500°C (right) [24]](image)

In [25], \( VO_2 \) is sputtered from \( V_2O_5 \) target with reactive hydrogen (2.5-10% of \( H_2+Ar \)) at pressure 0.2 Pa and power 100W. After 30 min pre-sputtering, the temperature of the fused silica glass substrate kept at 400°C to grow the \( VO_2 \) smoothly. In [29], the deposited precursor vanadium on the SiO\(_2\)/Si substrate with metal vanadium target is oxidized to \( VO_3 \) in chamber at temperature 370-415°C and oxygen pressure 0.2Torr. Furthermore, post annealing at 270°C (better result at 450°C) is an alternative method to oxidize the deposited vanadium to shape the \( VO_2 \). However, the best results were related to the in-situ deposition immediately after deposition of precursor vanadium. In [30], the single (001) orientation orthorhombic metal vanadium is in situ deposited on sapphire, silicon, and glass substrates by RF sputtering with vanadium target in partial oxygen pressure 0-25% at temperature less than 100°C. In [31], the deposited vanadium post-annealed at 530°C with an oxygen pressure between 14 -18 Pa to not oxidize changing to
In [32], the phase transition of VO\textsubscript{2} is improved by further post-annealing at 300\textdegree C in high-pressure oxygen 10–25 mTorr. The XRD result in Figure 10, shows thin film VO\textsubscript{2} sputtered by pulsed laser deposition (PLD) method on thick oxide (SiO\textsubscript{2}/Si) substrate [33].

![XRD result](image)

Figure 10. XRD results for VO\textsubscript{2} film deposited on SiO\textsubscript{2}/Si substrate [33]

### 4. EXPERIMENTAL RESULT

In our work, we sputtered 50nm VO\textsubscript{x} film on SiO\textsubscript{2}/Si substrate by V\textsubscript{2}O\textsubscript{5} target. The substrate is composed of 1.5\textmu m thermal SiO\textsubscript{2} deposited on silicon wafer by plasma-enhanced chemical vapor deposition (PECVD) method. During RF sputtering process, total gas pressure was kept at 12.5 mTorr for 2hours deposition process. We did several sputtering tests for different temperature of substrate varied from 300\textdegree C to 500\textdegree C in-situ at a power of 120W for different oxygen and argon flow ratio. The wafer height in the chamber held 20cm from target. The surface of the thin film is characterized by XRD at temperatures 300\textdegree C, 400\textdegree C and 500\textdegree C in Figure 11, shows that for flow ratio O\textsubscript{2}=2sccm and Ar(P)=32sccm (R\textsubscript{f0} 0.058), the deposited film on thick SiO\textsubscript{2} is V\textsubscript{2}O\textsubscript{3}. We repeated the test for R\textsubscript{f0} between 0.042 to 0.06, however the measured surface of the thin film by XRD technique showed V\textsubscript{2}O\textsubscript{3} growth on the thick oxide substrate. The result did not change significantly after post-annealing process for partial oxygen pressure 100Torr at 300\textdegree C, 400\textdegree C and 500\textdegree C. For the total volume of oxygen and nitrogen equal to 10 lit/min, %13 oxygen (1.3 lit/min) and %43 nitrogen (8.6 lit/min) were considered to do post-anneal the sputter film about 10 minutes. In [34], the ALD deposited amorphous vanadium oxide crystallized in furnace annealing process in higher rate of nitrogen ratio (more than %98) at temperature of 425\textdegree C and higher, as shown in Table 1 [35, 36, 37, 38]. The gray rows in Table 1 show the successful annealing process of create polycrystalline stoichiometric VO\textsubscript{2} phase.

| Anneal Temp | Annealing time and gas ratio under atmospheric pressure | Annealing time and gas ratio under low vacuum (10^{-2} Torr) |
|-------------|----------------------------------------------------------|----------------------------------|
| 420 \textdegree C | 30 min | 98.78\% N\textsubscript{2} + 1.22\% O\textsubscript{2} + 1\% O\textsubscript{2} + 1.5\% O\textsubscript{2} |
| 420 \textdegree C | 30 min | 99.85\% N\textsubscript{2} + 0.15\% O\textsubscript{2} + 0\% O\textsubscript{2} + 0\% O\textsubscript{2} |
| 420 \textdegree C | 5 min | 98.2\% N\textsubscript{2} + 1.8\% O\textsubscript{2} + 0.6\% O\textsubscript{2} + 0.4\% O\textsubscript{2} |
| 450 \textdegree C | 30 min | 5 min |
| 450 \textdegree C | 30 min | 5 min |
| 450 \textdegree C | 30 min | 5 min |
| 450 \textdegree C | 5 min | 10 min |
| 450 \textdegree C | 10 min | 60 min |

Table 1. The furnace annealing result to crystallize the ALD deposited amorphous vanadium oxide film [34]
5. CONCLUSION

In this article, the crystallographic and electrical properties of VO₂ were reviewed to show the metal-insulator phase transition of the film at temperature 68°C. The proposed VO₂ based metamaterial structure was simulated with HFSS to demonstrate the emittance tunability (Δε) of rutile and monoclinic states of VO₂. We summarized the recent progress in the deposition of thermochromic VO₂ on glass and silicon substrate by focusing on changing the partial pressure of the oxygen and argon. Furthermore, we sputtered VOₓ by V₂O₅ target with Rfo between 0.042 to 0.6 at temperature 300°C and higher and measured it with XRD method, which resulted in forming the V₂O₃ on the thick oxide on silicon wafer. For future work, it is recommended to sputter the thin film VOₓ on sapphire or silicon substrate with V₂O₃, since VO₂ grows smoothly on sapphire in comparison with the thick thermal SiO₂. Thin films thermochromic vanadium dioxide is currently attracting much attention in areas such as microelectronics and spacecraft control systems for their electronic behaviors in MIT.
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