

Effect of Strain on Properties of Metal Doped VO₂ Based Thermal Sensors on Muscovite Substrate[†]

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Abstract: In this work, VO₂ based thermal sensing thin film synthesized on flexible muscovite substrates by direct oxidation of deposited vanadium metal, were investigated for the impact of doping and strain on their electrical properties. We investigated both undoped and Ti doped VO₂ on muscovite substrate and compared with those on Quartz substrate. Both doped and undoped VO₂ were found to undergo phase transition due to effect of heat as well as mechanical strain on muscovite substrate. On the other hand, the Ti doped VO₂, on both quartz and muscovite substrate showed significant reduction in the transition temperature compared to the undoped VO₂ thin films on these two substrates. When subjected to mechanical strain, the VO₂ thin film on muscovite substrates resulted in a decrease or an increase in resistance depending on whether the applied strain was tensile or compressive, respectively. The resistance change was also steeper around the transition temperature compared to room temperature, exhibiting high gauge factor. This metal doped VO₂ on flexible muscovite substrate has the significantly low transition temperature which causes the VO₂ film to undergo phase transition at a near-room temperature and enables it to be used as a temperature sensor with enhanced sensitivity.

Keywords: VO₂ thin film; transition temperature; flexible substrate; mechanical strain; tensile strain; compressive strain; gauge factor



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1. Introduction

VO₂ has many important applications because of its uniquely reversible phase transitioning properties [1–4]. There have been numerous studies reported on synthesizing high quality VO₂ on different substrates and their electrical and optical properties have been investigated in detail. While traditionally VO₂ is grown on sapphire, Si or quartz substrates, its synthesis on muscovite substrate has attracted special attention due to the optical transparency and flexibility of muscovite, which not only offers the possibility of modulating the optical properties of the transmitted light through phase transition but can enable the phase transition by application of mechanical strain. However, the typical transition temperature of 67 °C is not low enough to induce phase change by application of strain, in unheated substrates, which can enable a host of applications requiring high sensitivity associated with the phase change region. Therefore, reduction in the transition temperature of the VO₂ is very much desired, which will make the VO₂ thin film will be more sensitive to physical parameters such as temperature, strain or electric field even at room temperature without any need for heating, enabling it to be used as a thermal sensor with great sensitivity. This can be achieved by doping the vanadium with metallic dopants such as W, Ti and Cr, which can reduce the transition temperature of VO₂ thin films [5,6]. In our previous reports [7,8] we have demonstrated the synthesis and properties of VO₂ synthesized on various substrates, including piezoelectric and flexible ones. We

also displayed the shifting of transition temperature due to mechanical strain on VO₂ on muscovite [7]. In the current work, we present the phase transitioning properties of VO₂ doped with Ti and compared the characteristics of doped and undoped VO₂. Furthermore, we displayed the change of resistance of VO₂ film due to mechanical strain.

For measuring the surface resistance, the VO₂ thin film sample is connected to a data acquisition unit through contact points formed by conductive paste. The sample is placed at the very edge of the ceramic heater with a thermocouple on top of it to record the real-time temperature, operated from the data acquisition unit (Figure 1). The temperature is varied through by the ceramic heater, which is recorded by the data acquisition unit, alongside the real-time variation of resistance. In order to apply the mechanical strain, a wedge was placed on top or bottom of the sample, to apply tensile or compressive strain, respectively. The amount of strain was controlled by the rotation of a knob of the micropositioner attached to the wedge. In order to observe the effect of strain on surface resistance, the temperature of the sample was maintained at room temperature, and for each amount of tensile and compressive strain, the resistance was recorded. A gradual reduction of resistance was observed due to increase of compressive strain, whereas the opposite behavior is observed for tensile strain. The same measurement procedure was repeated at transition temperature 50 °C and similar results were observed. Gauge factor has been calculated for all the three combinations of applied strain on the VO₂/Ti/muscovite, i.e., tensile strain at room temperature, compressive strain at room temperature and compressive strain at transition region of VO₂. As expected, maximum gauge factor was obtained at the transition region of VO₂ due to compressive strain.

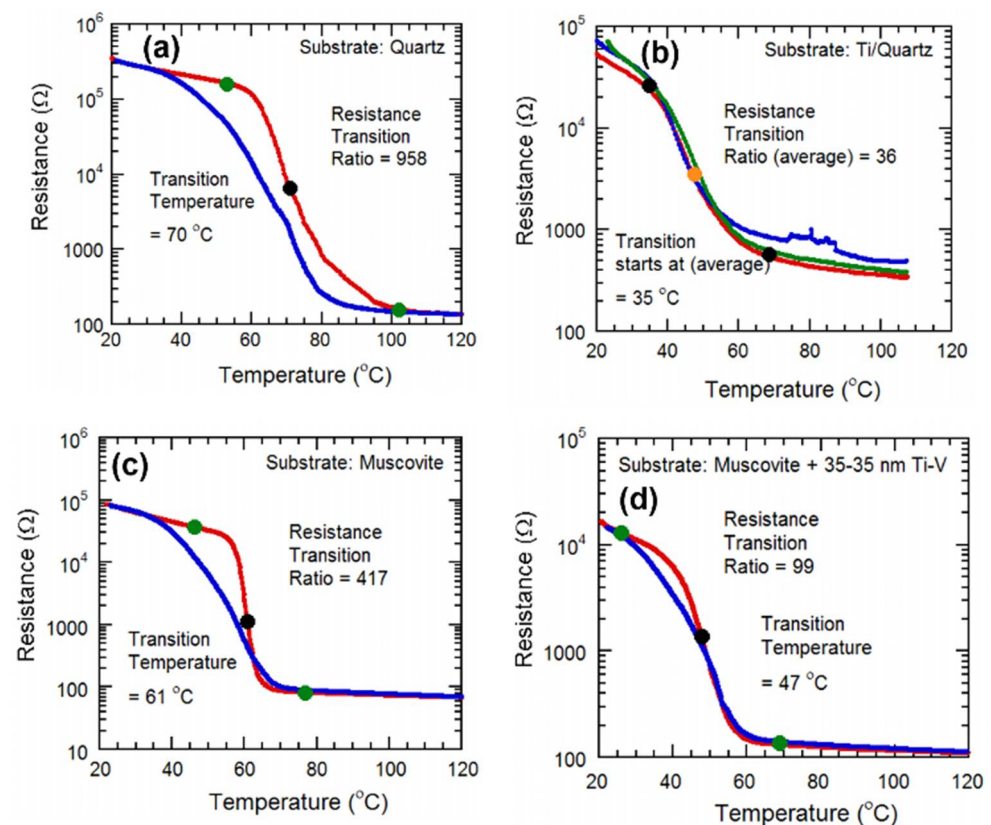


Figure 1. The phase transitioning plots of VO₂ thin films synthesized on (a) AT-cut quartz, (b) Ti (35 nm, doping layer) coated AT-cut quartz, (c) muscovite, and (d) Ti (35 nm, doping layer) coated muscovite.

2. Experimental Details

The synthesis of the VO₂ film-based sensors were achieved by using a homemade low-pressure furnace through the controlled oxidation of vanadium thin films of desired

thicknesses deposited on AT-cut quartz and muscovite. Prior to growth, the substrates were coated with vanadium thin films, using high purity (99.7%, from Kurt J. Lesker) vanadium pellets with a deposition rate of 1.5 Å/s in an electron beam evaporation system (70 nm thickness for undoped vanadium). The doped VO_2 film was synthesized by first depositing a 35 nm layer of Ti, which was followed by a deposition of 35 nm V. The chamber pressure is expected to reach 4.53 Pa under pump down, N_2 gas (purity 99.999% from Airgas) flow was started at a constant rate of 400 sccm until the chamber pressure stabilized at $\sim 2666 \text{ Pa}$. The furnace temperature was then increased to 475°C , and O_2 flow was initiated at the rate of 100 sccm. The oxidation durations were optimized over several growth iterations to ensure high quality of the VO_2 films, carefully avoiding under-oxidation and over-oxidation. In addition, it was ensured that the VO_2 film has uniformity all over the substrate, through detailed characterization.

3. Results and Discussion

The experimental setup in Figure 1 was utilized to measure the electrical resistance variation. A two-point probe measurement setup was implemented to observe the effect of substrate heating on the electrical resistance of VO_2 following previously reported [9,10]. A ceramic heater is used to vary the temperature of the VO_2 film, assisted by a thermocouple for recording the temperature, and tungsten probes connected to a data acquisition unit for measuring the surface resistance. The results are plotted in Figure 1.

As seen from Figure 1a,b, the Ti-doped VO_2 shows a significant reduction in transition temperature from 70°C to 35°C . However, this is also associated with a reduction in the transition resistance ratio from 958 to 36. On the other hand, the transition temperature for the VO_2 synthesized on muscovite changed from 61°C to 47°C , while the transition resistance ratio reduced from 417 to 99.

The effect of strain on VO_2 based sensing films deposited on the muscovite substrate was studied using a home-made experimental set up as shown in Figure 2.

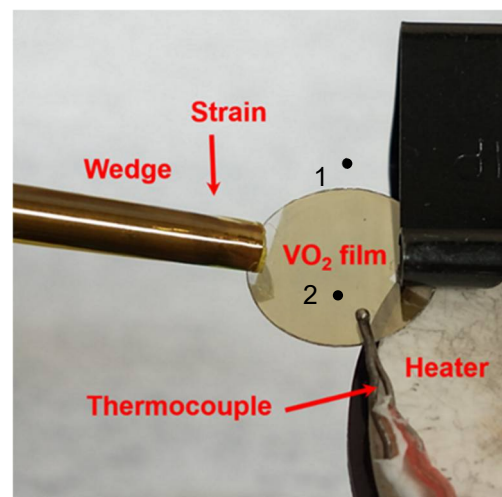


Figure 2. The experimental setup for application of mechanical strain applied on VO_2 thin film synthesized on muscovite substrate. Points 1 and 2 indicate the electrical contact points for resistance measurement.

A thermocouple is also shown touching the VO_2 substrate to measure the film temperature. We studied both tensile and compressive strain by bending the edge of the film using a micropositioner extended arm. The arm is pressed downwards on the top of the sample to create tensile strain, and it can be pressed upwards from the bottom of the sample to produce compressive strain. The tensile strain causes the surface resistance to increase while compressive strain results in a decrease in surface resistance in agreement with previous reports [8].

In Figure 3, the variation of surface resistance is plotted with respect to variation of deflection due to applied strain. The change of resistance is recorded with change of tensile strain at room temperature, compressive strain at room temperature and compressive strain around transition temperature at phase transitioning region. For tensile strain the change in resistance per °C comes out to be 3.55%. The slope of resistance change is significantly more at the phase transition region compared to room temperature. At room temperature due to compressive strain, rate of change of resistance per °C is 5.95% and for around transition temperature, it is 9.09%.

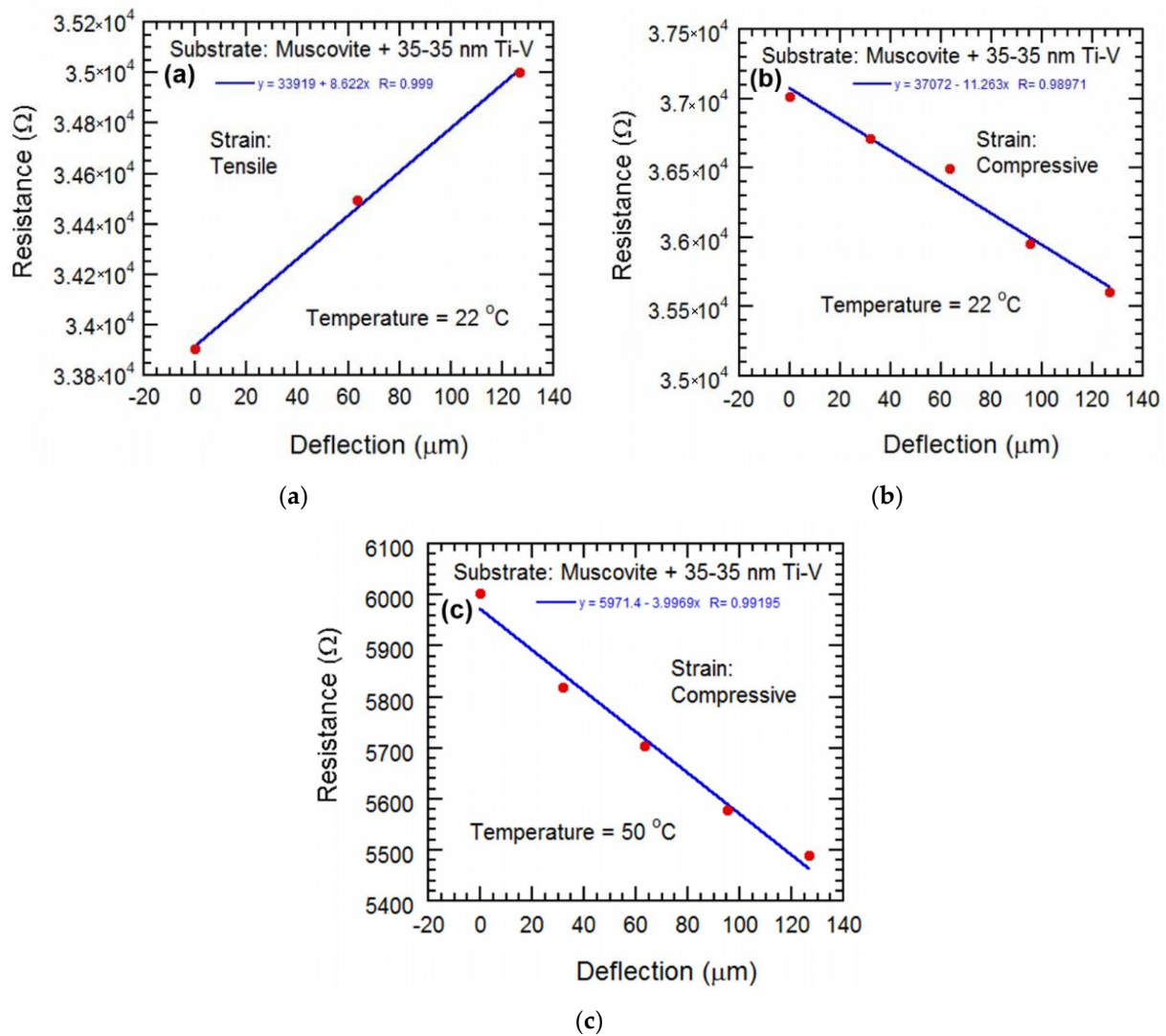


Figure 3. (a) The effect of tensile strain on the resistance between two contacts on Ti-doped VO_2 at 22 °C (room temperature). The effect of compressive strain on the resistance of Ti-doped VO_2 on muscovite (b) at 22 °C (room temperature) and (c) at 50 °C (transition region).

The $\text{VO}_2/\text{Ti}/\text{muscovite}$ thin film sample shown in Figure 2 can be approximated as a square cantilever with thickness $t = 150 \mu\text{m}$, and beam length $L = 11 \text{ mm}$ ($1.1 \times 10^4 \mu\text{m}$), one end attached and the other end as freely suspended. From Figure 3a–c, we see that the maximum deflection of the free end of the cantilever $Y_{\text{max}} = 127 \mu\text{m}$.

The length to thickness ratio of the approximated cantilever is 73:1, which is more than 10:1. Therefore, the average strain ε due to deflection of the free end of the cantilever can be calculated from the formula stated below [11]:

$$\varepsilon = 1.5 \left(\frac{t}{L^2} \right) Y \quad (1)$$

Here, t is the cantilever beam thickness, L is the cantilever beam length, and Y is the cantilever deflection at the free end.

Plugging in the values of the parameters of the right-hand side of the Equation (1), we can calculate the strain as $\varepsilon = 2.36 \times 10^{-4}$. From Figure 3a, the fractional resistance change (over the entire deflection range) can be calculated as $\Delta R/R = 0.032$ for tensile strain at 22 °C. From the maximum deflection $Y_{\max} = 127 \mu\text{m}$, the gauge factor GF is calculated from the formula [12]:

$$GF = \frac{\Delta R/R}{\varepsilon} \quad (2)$$

Plugging in the values of the right-hand side of the Equation (2), the calculated gauge factor = 136. Following the same procedure, the relative resistance change for 127 μm deflection in Figure 3b comes out to be the $\Delta R/R = 0.038$, which yields a GF value for the compressive strain at 22 °C as 161. Similarly, from the plot in Figure 3c, we find $\Delta R/R = 0.086$, and for the compressive strain of $\varepsilon = 2.36 \times 10^{-4}$, the GF turns out to be 364 at 50 °C.

Comparing the GF values in all three cases, we find that the gauge factor is maximum when the relative variation of resistance is maximum during compressive strain, which happens at the phase transition region (50 °C).

Comparing the GF values with those of common semiconductors, we find that these values are significantly higher than the best values reported for Si piezoresistors, which have the maximum gauge factor 95 [12]. They are also comparable to the GF values reported for III-nitride based piezoresistors, although they are lower than that which is possible with the III-Nitride piezotransistor technology. For III-V based piezoresistive microcantilevers, the gauge factor reported previously for tensile strain was 75 [13]. The GF of 136 determined for tensile strain in this work is higher than the GF calculated for tensile strain on III-V based piezoresistive microcantilevers [13].

For III-V nitride-based pressure sensors with integrated transistors [14], the maximum gauge factor reported be 260 for operation in the sub-threshold region, which is still less than the maximum gauge factor 364 found for our flexible VO₂/Ti/muscovite thin films at phase transition temperature. However, piezotransistors formed by GaN microcantilever with embedded AlGaIn/GaN HFET provides gauge factor ~8700 for gate bias voltage −3 V, caused by a deflection of only 1 μm [15], and 3200 due to gate bias voltage of −3.1 V [16], which are still much superior compared to the values reported here for our macroscopic devices. Nonetheless, the presented VO₂ based sensing devices required no specialized and expensive layer structures such as the III-Nitrides or complicated fabrication methodology. Thus, they can be promising for applications requiring near room temperature strain sensing requiring high sensitivity.

4. Conclusions

In conclusion, we have synthesized and characterized undoped and Ti-doped VO₂ films on quartz and muscovite substrates. It was observed that the transition temperature for Ti-doped VO₂ on both substrates reduced significantly compared to the undoped ones, although it was associated with a reduction in the transition resistance ratio. The flexible muscovite substrate was utilized in order to study the effect to both compressive and tensile strains, which resulted in a reduction in resistance for the former, and an increase for later. The resistance change due to strain were found to be steeper around the transition temperature compared to room temperature, as expected. The gauge factor of the cantilever modeled VO₂/Ti/muscovite due to the maximum deflection turned out to be 364 at

transition temperature under compressive strain, which is much improved compared to conventional Si and III-Nitride based piezoresistive microcantilever sensors.

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References

1. Baqir, M.A.; Choudhury, P.K. On the VO₂ metasurface-based temperature sensor. *J. Opt. Soc. Am.* **2019**, *36*, F123–F130. [CrossRef]
2. Cui, Y.; Ke, Y.; Liu, C.; Chen, Z.; Wang, N.; Zhang, L.; Zhou, Y.; Wang, S.; Gao, Y.; Long, Y. Thermochromic VO₂ for Energy-Efficient Smart Windows. *Joule Cellpress Rev.* **2018**, *2*, 1707–1746. [CrossRef]
3. Masina, B.N.; Akande, A.A.; Mwakikunga, B. Effect of laser wavelength in PLD in the orientation and thermochromic properties of VO₂ (M1) on a glass substrate. *MRS Adv.* **2020**, *5*, 1121–1132. [CrossRef]
4. Lappalainen, J.; Heinilehto, S.; Saukko, S.; Lantto, V.; Jantunen, H. Microstructure dependent switching properties of VO₂ thin films. *Sens. Actuators* **2018**, *142*, 250–255. [CrossRef]
5. Zhang, J.; He, H.; Xie, Y.; Pan, B. Giant reduction of the phase transition temperature for beryllium doped VO₂. *Phys. Chem. Chem. Phys.* **2013**, *15*, 4687–4690. [CrossRef] [PubMed]
6. Matsuguchi, M.; Uno, T. Molecular imprinting strategy for solvent molecules and its application for QCM-based VOC vapor sensing. *Sens. Actuators B* **2006**, *113*, 94–99. [CrossRef]
7. Azad, S.; Gajula, D.; Koley, G. Infrared Transmission Characteristics of Phase Transitioning VO₂ on Various Substrates. *Micromachines* **2022**, *13*, 812. [CrossRef] [PubMed]
8. Azad, S.; Gajula, D.; Koley, G. Synthesis and characterization of VO₂ thin films on piezoelectric substrates. *Thin Solid Films* **2021**, *742*, 139055. [CrossRef]
9. Chen, H.-W.; Li, C.-I.; Ma, C.-H.; Chu, Y.-H.; Liu, H.-L. Strain engineering of optical properties in transparent VO₂/muscovite heterostructures. *Phys. Chem. Chem. Phys.* **2021**, *23*, 8908–8915. [CrossRef] [PubMed]
10. Kikuzuki, T.; Lippmaa, M. Characterizing a strain-driven phase transition in VO₂. *Appl. Phys. Lett.* **2016**, *96*, 132207. [CrossRef]
11. Lee, C. Improving Snapfit Design. 2007. Available online: <https://knowledge.ulprospector.com/1248/pe-snapfit-3/> (accessed on 19 February 2007).
12. Yu, X.M.; Thaysen, J.; Hansen, O.; Boisen, A. Optimization of sensitivity and noise in piezoresistive cantilevers. *J. Appl. Phys.* **2002**, *92*, 6296–6301. [CrossRef]
13. Qazi, M.; DeRoller, N.; Talukdar, A.; Koley, G. III-V Nitride based piezoresistive microcantilever for sensing applications. *Appl. Phys. Lett.* **2011**, *99*, 193508. [CrossRef]
14. Gajula, D.; Jahangir, I.; Koley, G. High Temperature AlGaIn/GaN Membrane Based Pressure Sensors. *Micromachines* **2018**, *9*, 207. [CrossRef] [PubMed]
15. Talukdar, A.; Khan, M.F.; Lee, D.; Kim, S.; Thundat, T.; Koley, G. Piezotransistive transduction of femtoscale displacement for photoacoustic spectroscopy. *Nat. Commun.* **2015**, *6*, 7885. [CrossRef] [PubMed]
16. Talukdar, A.; Koley, G. Impact of Biasing Conditions on Displacement Transduction by III-Nitride Microcantilevers. *IEEE Electron Device Lett.* **2014**, *35*, 1299–1301. [CrossRef]

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