SP1- 4p

On the synthesis of VO_x thin films by reactive magnetron sputtering

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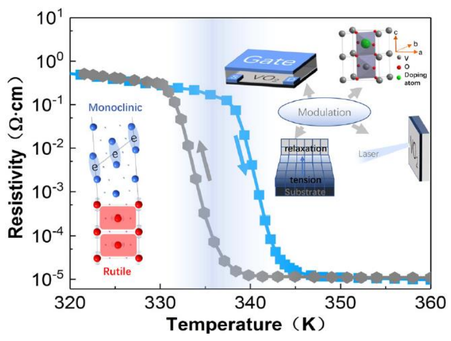
Introduction

Vanadium dioxide (VO_2) is an interesting material due to its semiconductor-to-metal transition at 68˚C [1]. At this temperature, the material undergoes a phase transition from monoclinic to tetragonal which yields to a change in the optical and electrical properties. Here we report our first attempt to study the effect of plasma conditions on the properties of 100 nm thick VO_x films deposited inside the so-called transition zone. In particular, we study the relationship between the deposition conditions and the film crystallinity and optical properties during reactive direct current magnetron sputtering (R-dcMS) of a V target in Ar/O_2 atmosphere by using voltage feedback control loop.



Z. Shao et al, Materials today, Vol 21, 2018, 875-896

Experimental

Table1 Experimental conditions

V target size	[cm^2]	20.3
Base pressure	[mTorr]	$< 2.0 \times 10^{-6}$
Working pressure	[mTorr]	10
Ar flow rate	[sccm]	60
O_2 flow rate	[sccm]	Variable
Power	[W]	Variable
Target current	[mA]	300
Substrate		Glass Conductive Si
Feedback system		Voltage feedback
Bias voltage	[V]	Floating, -100

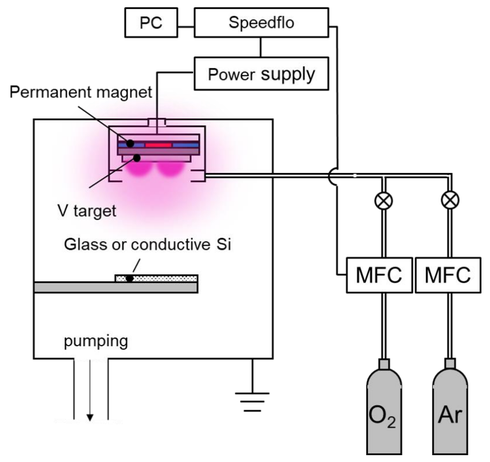


Fig.1 Schematic illustration of the R-DCMS deposition system.

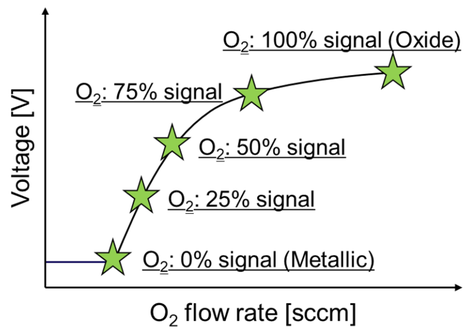


Fig.2 the voltage feedback control loop (Speedflo by Gencoa, UK) to stabilize the reactive process.

Table2 Post deposition annealing conditions[2][3]

Atmosphere		N_2
Temperature	[˚C]	400
Time	[min]	90

Characterization techniques

- Grazing incidence XRD
- Optical transmission and reflection measurements. Reflectivity is performed as a function of temperature

Phase constitution of the VO_x thin films

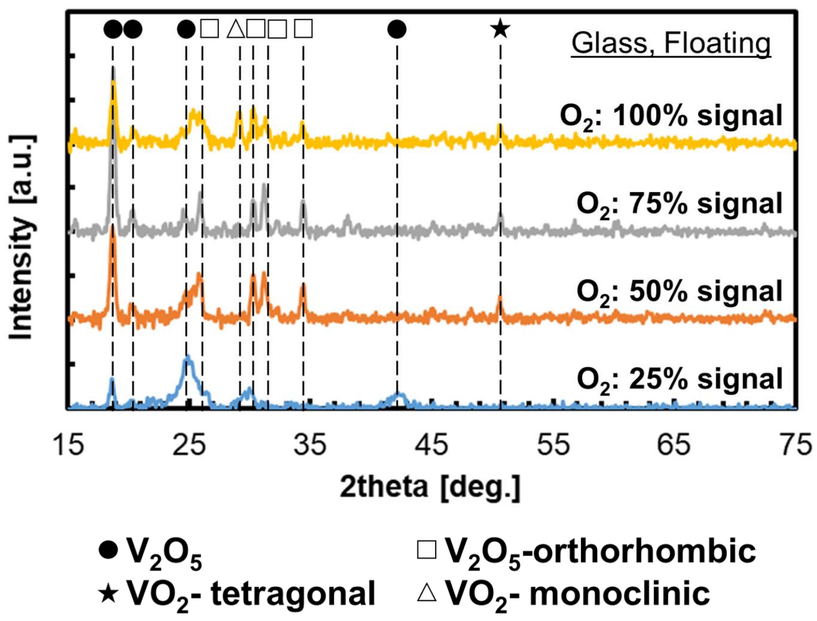


Fig.3 GIXRD scans of thin films grown at various O_2 signal setpoints ranging from 25 to 100%. As-deposited films on glass were XRD amorphous.
Sample deposited at 25% signal setpoint exhibits V_2O_5 crystal structure, while the samples of 50 and 75% signal setpoints shows mixed phase of phase of V_2O_5 and tetragonal VO_2. Film grown at 100% signal setpoint is characterized by a mixed phase of V_2O_5 and VO_2 (monoclinic and tetragonal).

Optical properties

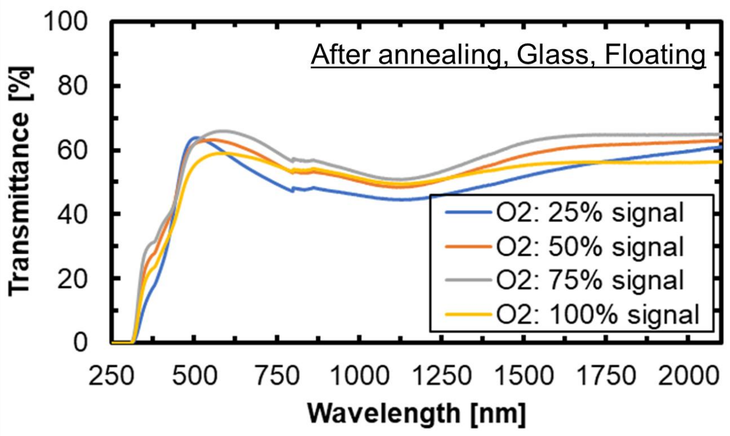
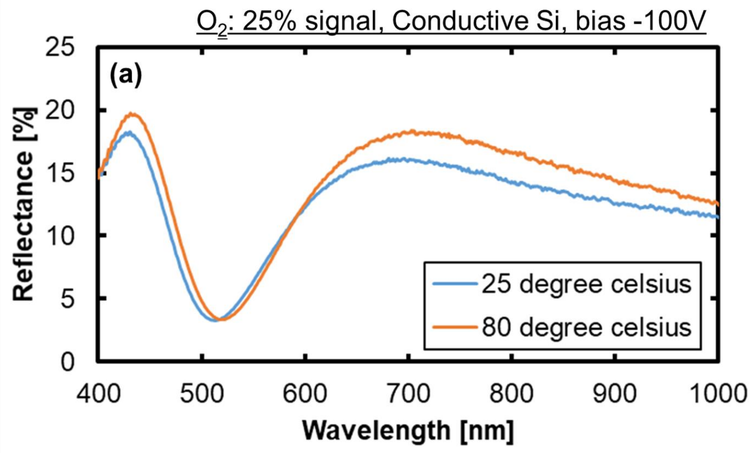


Fig.4 Transmittance spectra at room temperature with different O_2 signal setpoints ranging from 25 to 100% after annealing.



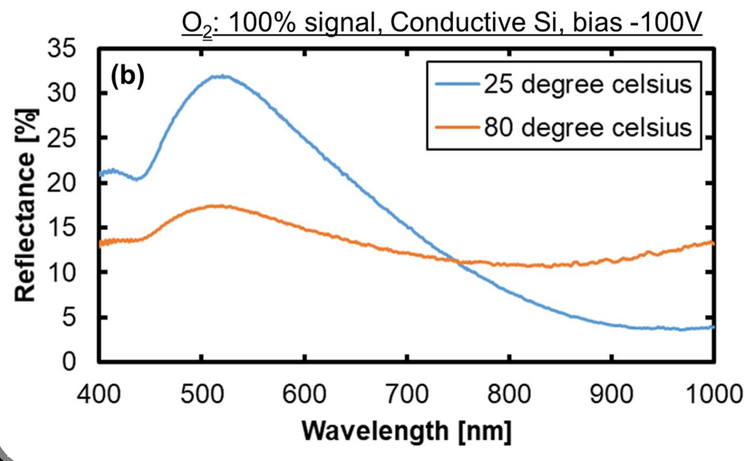


Fig.5 Reflectance spectra as a function of temperature for annealed films grown at (a) 25% and (b) 100% signal setpoints.

(a) The film grown at 25% signal setpoint doesn't show any change with temperature.

(b) The film grown at 100% signal setpoint show a significant variation of the reflectivity in the IR range at 25 ˚C and at 80 ˚C.
→This can be attributed to the presence of monoclinic VO_2 crystals.

References

[1] K. Liu et al, Mater. Today 21, 875 (2018)
[2] T. Rattana et al. Materials Today: Proceedings 5, 13870 (2018)
[3] M. Taha et al, Sci. Rep. 7, 1 (2017).

Summary

100 nm thick VO_x films were deposited on glass and conductive Si substrate at various positions inside the transition zone and substrate conditions (floating and -100V bias) during R-dcMS of a V target in mixed Ar/O_2 atmosphere. The crystal structure changes as a function of the deposition conditions. Every sample contains the V_2O_5 phase and only the one synthesized at 100% setpoint signal is characterized by a mixed phase of V_2O_5 and VO_2 with monoclinic and tetragonal. Reflectivity data revealed a significant increase of the reflectivity in the IR range as the temperature increases for the sample deposited at 100% setpoint value on a biased substrate and annealed at 400˚ C in nitrogen. Future work aims at investigating in more details the influence of deposition conditions to finally reach a phase pure VO_2 monoclinic films that we can deposit in Glancing Angle Deposition mode to fabricate VO_2-based nanocolumns.