Development and characterisation of nano-porous vanadium dioxide coatings for energy efficient windows

Lavinia Calvi

Interreg Vlaanderen-Nederland
Europese Unie
Europees Fonds voor Regionale Ontwikkeling
SiA Nationaal Regieorgaan Praktijkgericht Onderzoek
Brightlands Materials Center
IMO-IMOMEC
UHASSELT
imec

Contents

Problem Statement

- Increase in world energy needs → increase in efficiency & decrease in energy losses
- Buildings: 30-40% of world's total energy consumption
- Use of glass in construction has increased over the years

  + Light weight

  + Allows natural light in the building

  - Heat is lost in winter and gained in summer

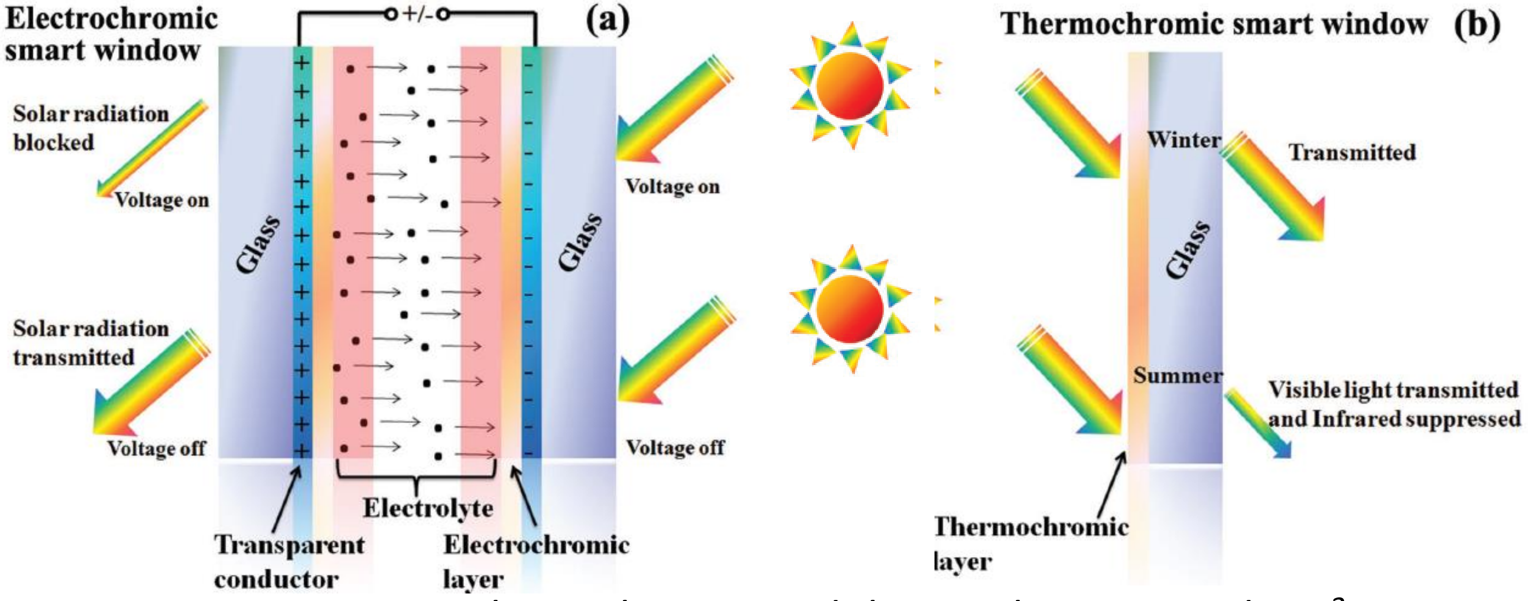
Hasselt City Hall[1,2]

1. "Stadhuis Hasselt." *TripAdvisor*, www.tripadvisor.com/Attraction_Review-g188650-d9879719-Reviews-Stadhuis_Hasselt-Hasselt_Limburg_Province.html.
2. "'T Scheep Hasselt." *UAU Collectiv*, 25 Sept. 2018, www.uaucollectiv.com/portfolio/nsh/.

Electrochromic & Thermochromic Windows

- Chromogenic materials exhibit changes in optical properties due to external stimulus
- Currently both types of glass become tinted
- Less visible light enters
- Active vs passive
- Voltage vs temperature

Suntuitive® Glass windows installed at an educational facility in Keller, Texas, USA. Photo courtesy of Pleotint, LLC.



Electrochromic and thermochromic windows[3]

3. Li, M.; Magdassi, S.; Gao, Y.; Long, Y. *Small J.* **2017**, *13*

Smart Windows Concept

- Aim: decrease solar heat gain
- True definition: passive & switchable

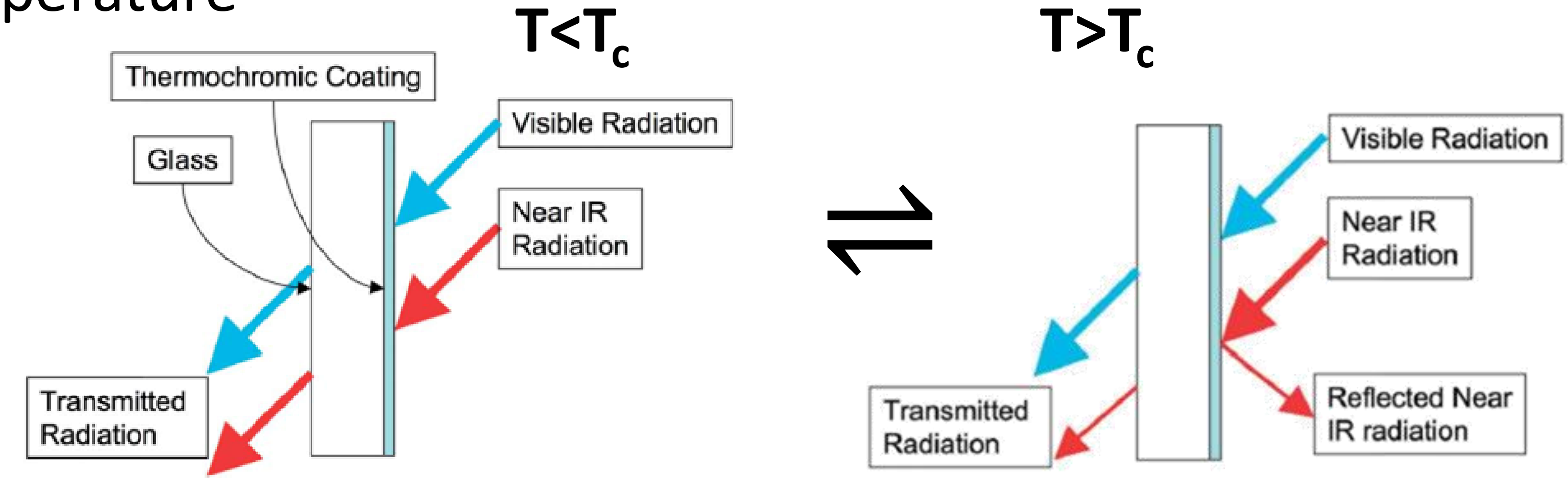
Mechanism for property change

Variable Transmission	Passive	Active
Switchable	Thermochromic	Electrochromic
Static	Low-e & tinted glass	Privacy & Solar glass

Based on: Sandburg, Kyle. “Are Smart Windows the Future? – Strategy Dynamics – Medium.” *Medium.com*, Medium, 9 Aug. 2018, medium.com/strategy-dynamics/are-smart-windows-the-future-b7bc1567105f.

Vanadium Dioxide Coating Principles

- Thermochromic – change optical properties with temperature change
- Critical / switching temperature (T_c) - where ‘switch’ occurs
- Vanadium dioxide (VO_2) - infrared properties change with temperature



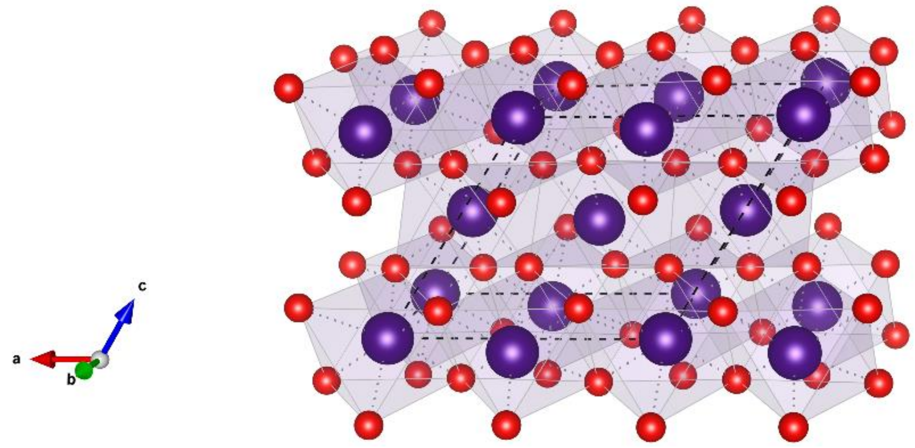
Schematic of thermochromic behaviour[4]

4. M. E. A. Warwick and R. Binions, *J. Mater. Chem. A*, vol. 2, pp. 3275–3292, 2014.

VO_2 Structure

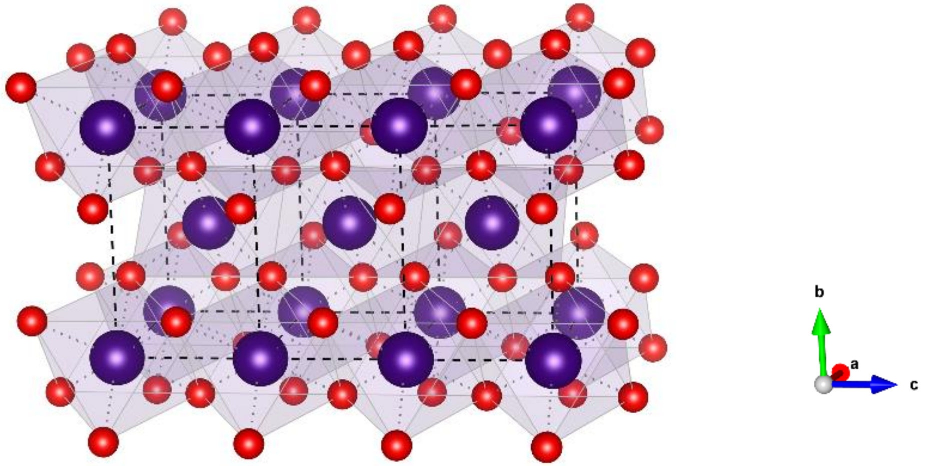
- Low temperature ($T<T_c$)
- Monoclinic lattice
- Semi-conducting phase
- Tilt in the c-axis

- High temperature ($T>T_c$)
- Rutile structure
- Metallic phase
- Infrared reflecting



$\rightleftharpoons$

Reversible 1st order metal-insulator transitions (MIT)

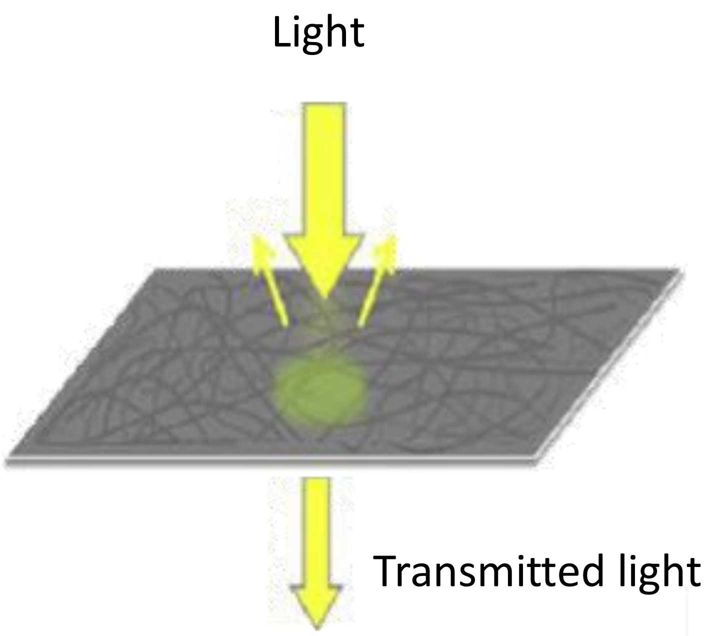


VO_2 Structure

Challenges of VO_2

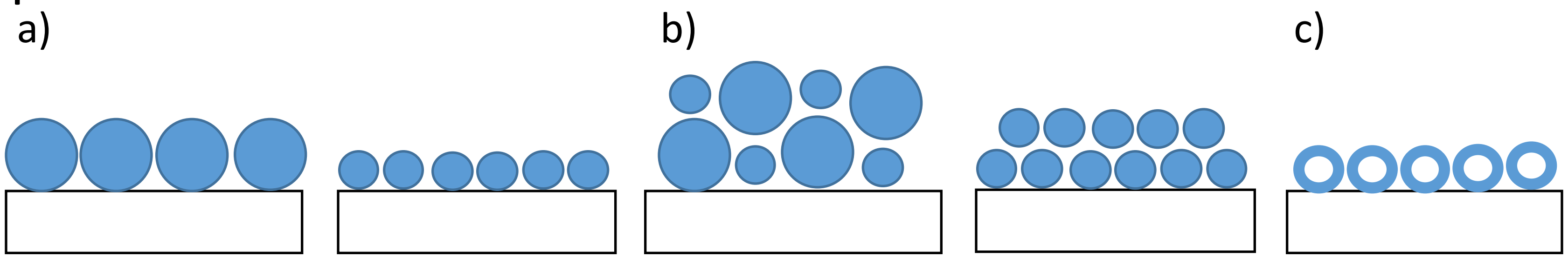
1. Relatively high switching temperature – about 68°C
   - Doping the material can be used to lower this temperature

2. Low luminous transmittance → increase porosity to lower refractive index and increase transmission in visible
   a) Decreasing particle size → avoid scattering
   b) Stacking of particles
   c) Encapsulation of air



Glass coatings light transmission[5]

5. S. Wang, M. Liu, L. Kong, Y. Long, X. Jiang, and A. Yu, "*Prog. Mater. Sci.*, vol. 81, pp. 1–54, 2016.



Particle thin films

VO_2 Powder Synthesis

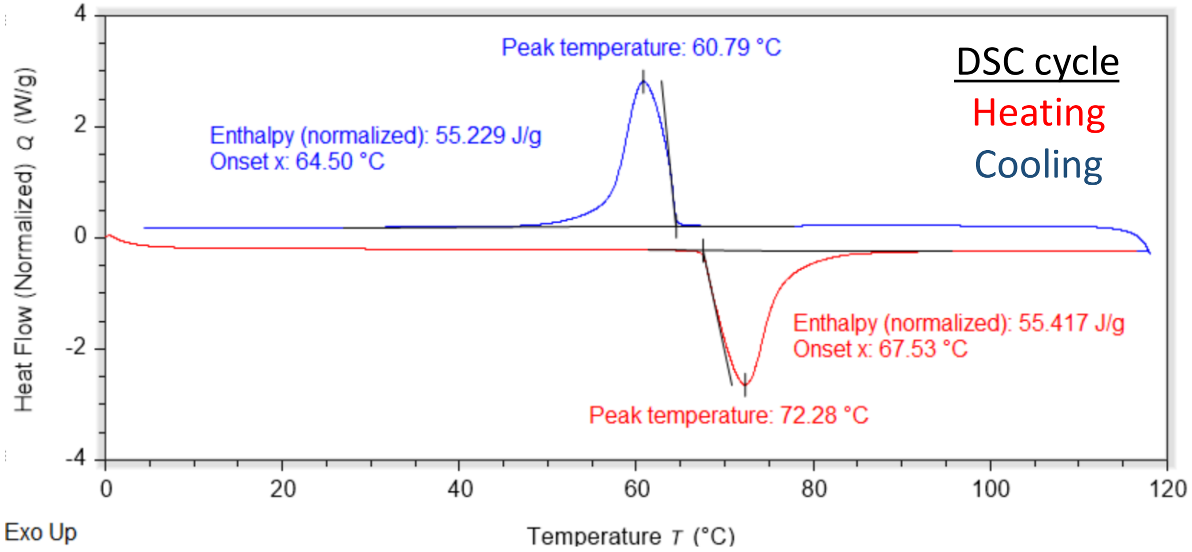
- Oxalic acid → V_2O_5 solution
- Drying
- Annealing in tube furnace
- Powder characterisation
- Milling
- Powder characterisation

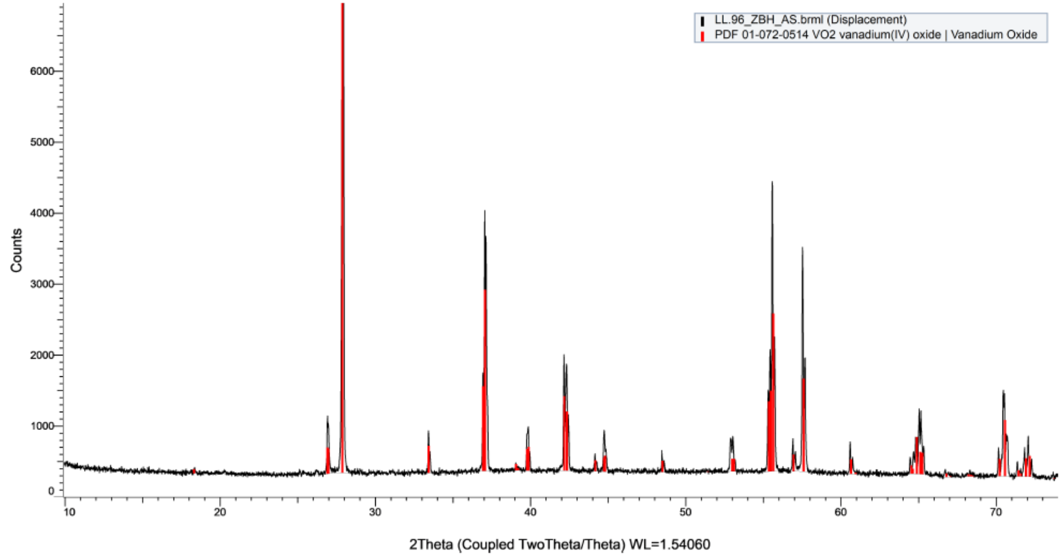
- V_2O_5 is dispersed in a solvent and then a reducing agent is added
- Freeze drying or rotary evaporator is used
- Annealing of the dried precursor is sensitive to oxygen
- High temperature curing process to form crystalline VO_2 & remove organics
- If too much oxygen is present in the tube furnace the material will oxidise

Interreg Vlaanderen-Nederland
Europees Fonds voor Regionale Ontwikkeling
SIA Nationaal Regieorgaan Praktijkgericht Onderzoek
IMO-IMOMEC UHASSELT imec
Brightlands Materials Center
ChemCYS

VO_2 Characterisation

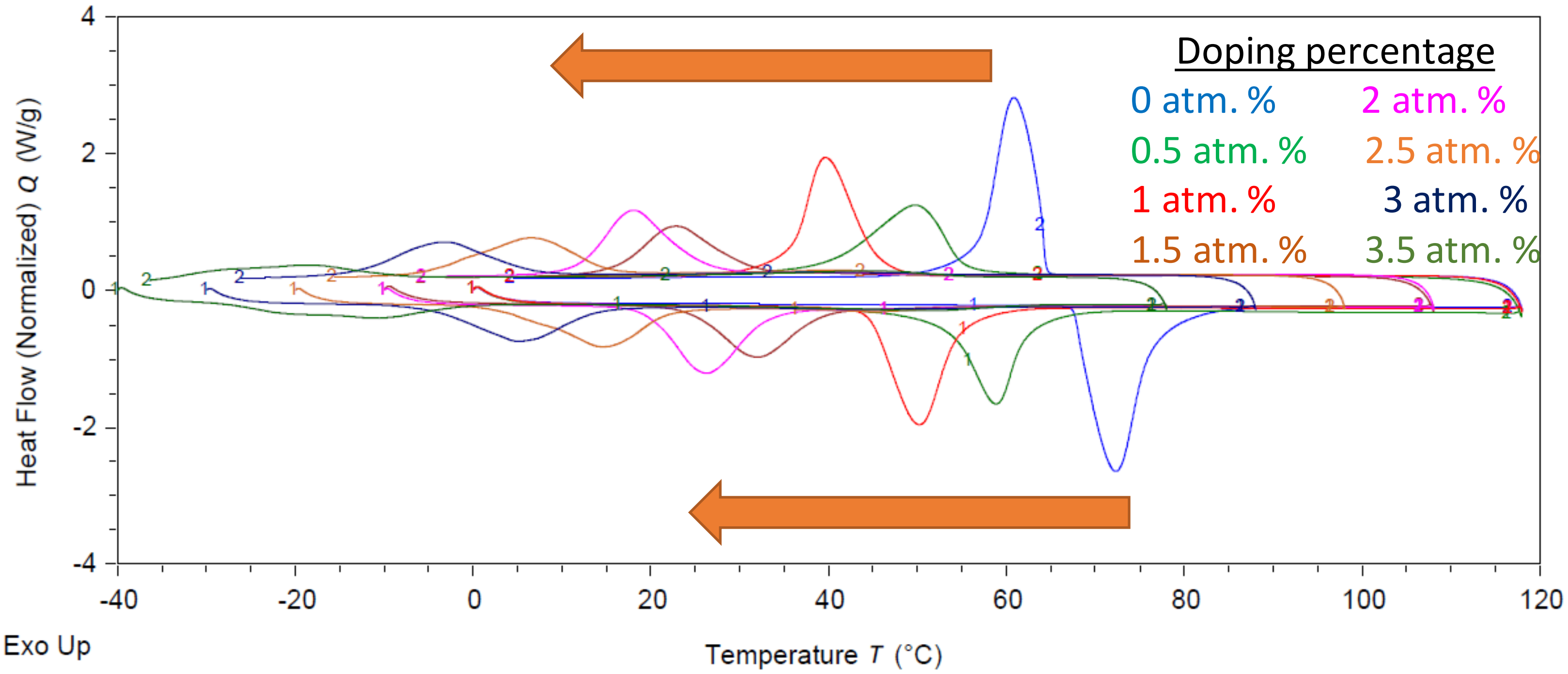
- DSC is used to measure various parameters of the VO_2 powders
- Integration of peak over time gives the enthalpy of the switch from monoclinic to tetragonal
  - 55 J/g is the theoretical values
  - Used to evaluate purity
- Hysteresis is the difference between the two peak temperatures
  - 72.28 °C – 60.79 °C = 11.49 °C
- XRD is also used to confirm the VO_2 structure but does not show amorphous phases



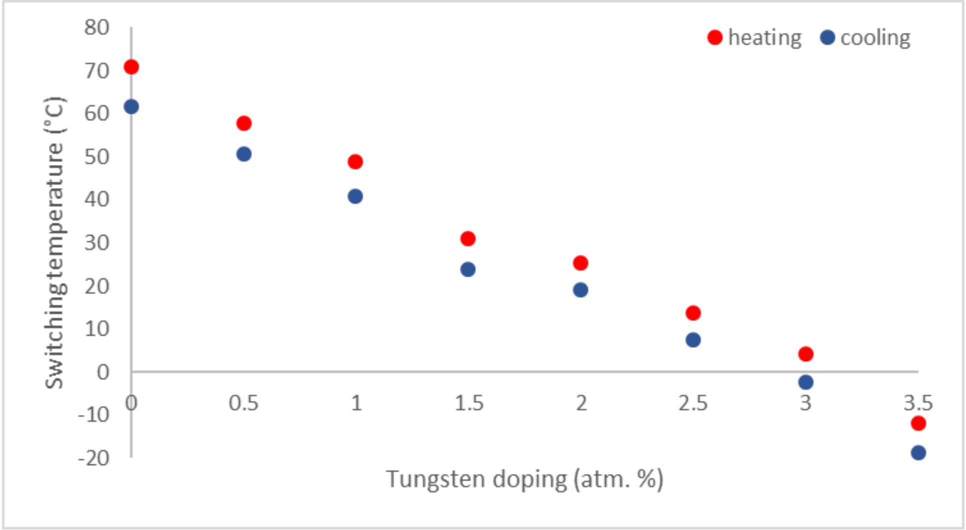


1. Decrease of switch temperature

- V_2O_5 solution ← Oxalic acid
- ← Tungsten solution
- ↓ Mixing
- ↓ Drying
- ↓ Annealing in tube furnace

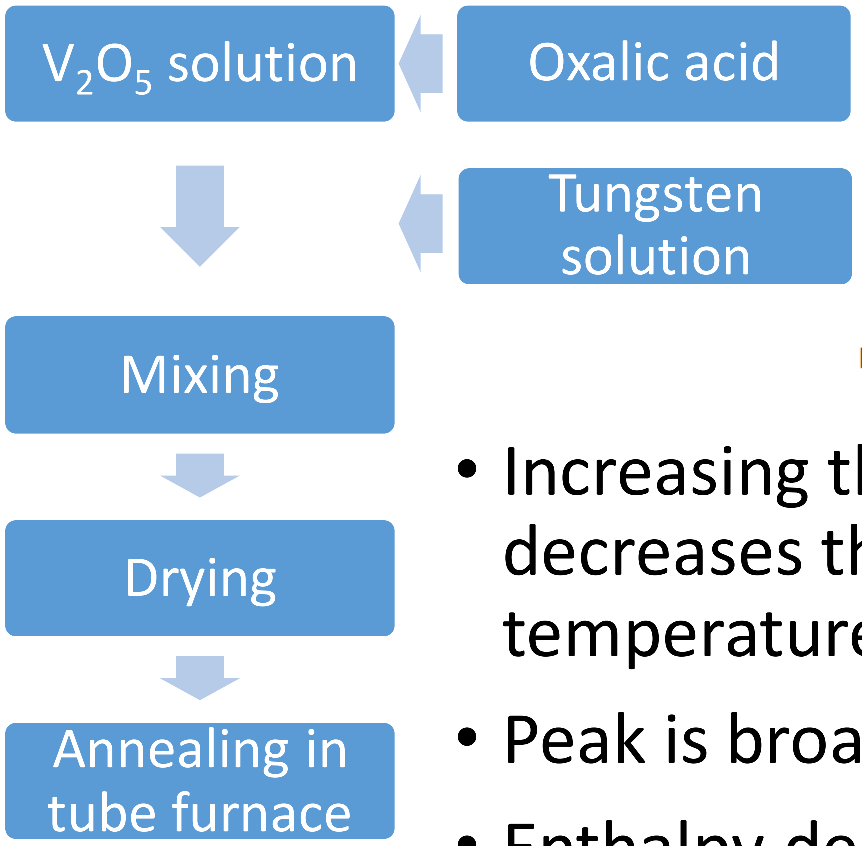


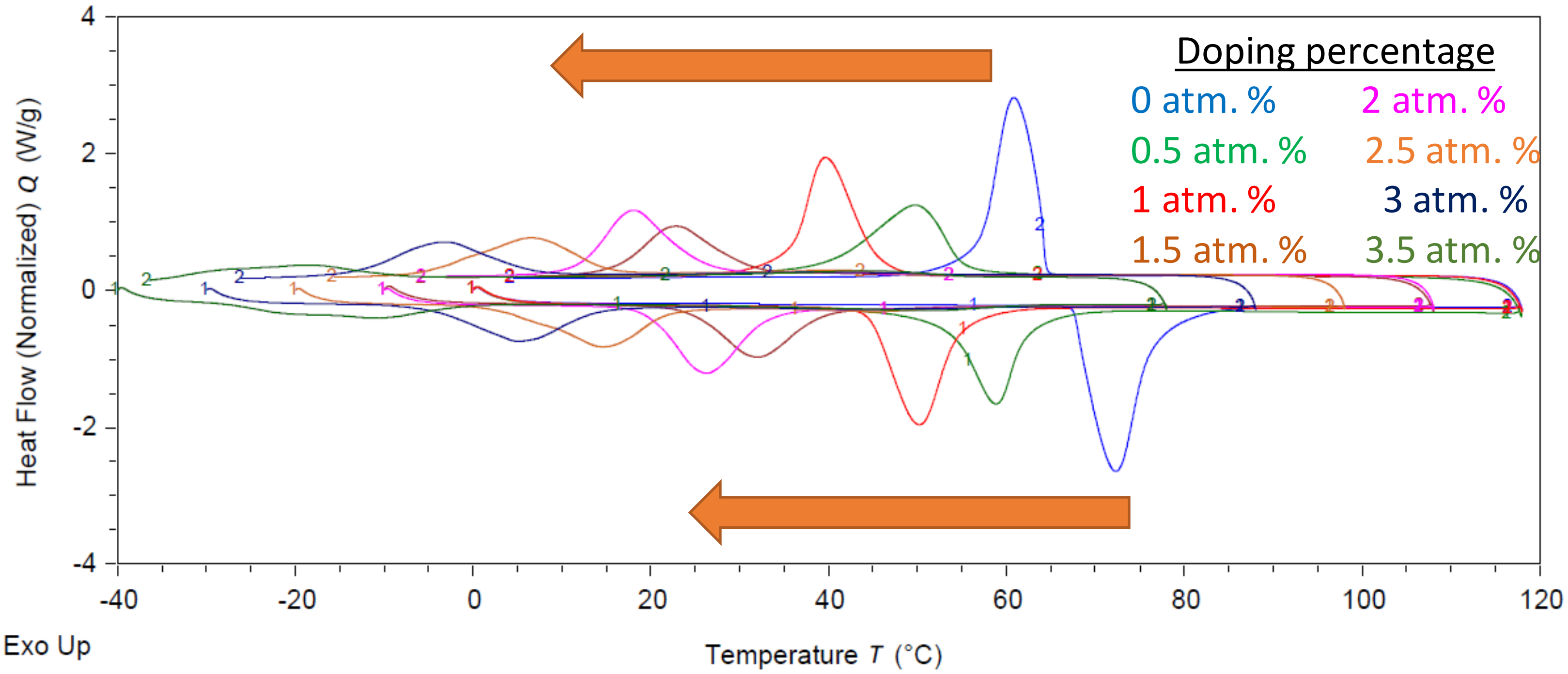
- Increasing the tungsten doping decreases the switching temperature
- Peak is broadened



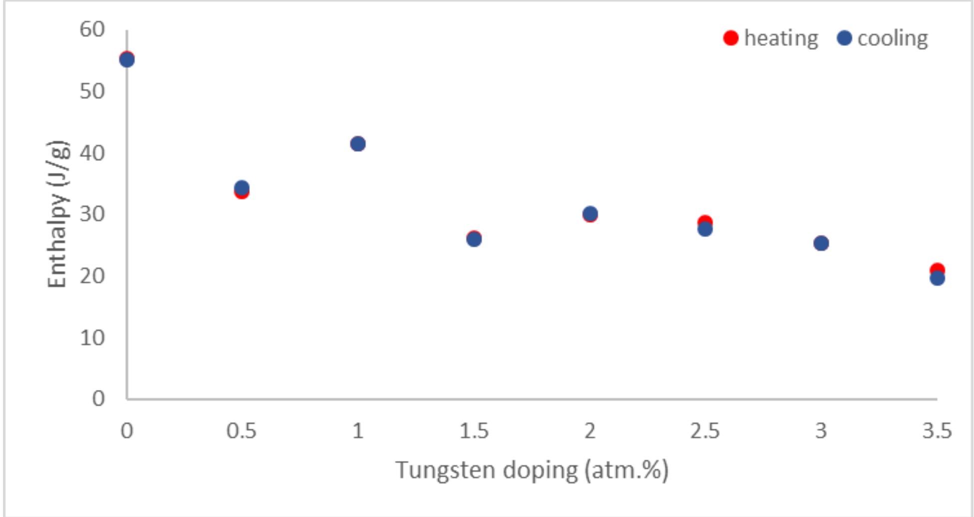
Interreg Vlaanderen-Nederland
Europees Fonds voor Regionale Ontwikkeling
SiA Nationaal Regieorgaan Praktijkgericht Onderzoek
IMO-IMOMEC UHASSELT imec
Brightlands Materials Center
ChemCYS

1. Decrease of switch temperature



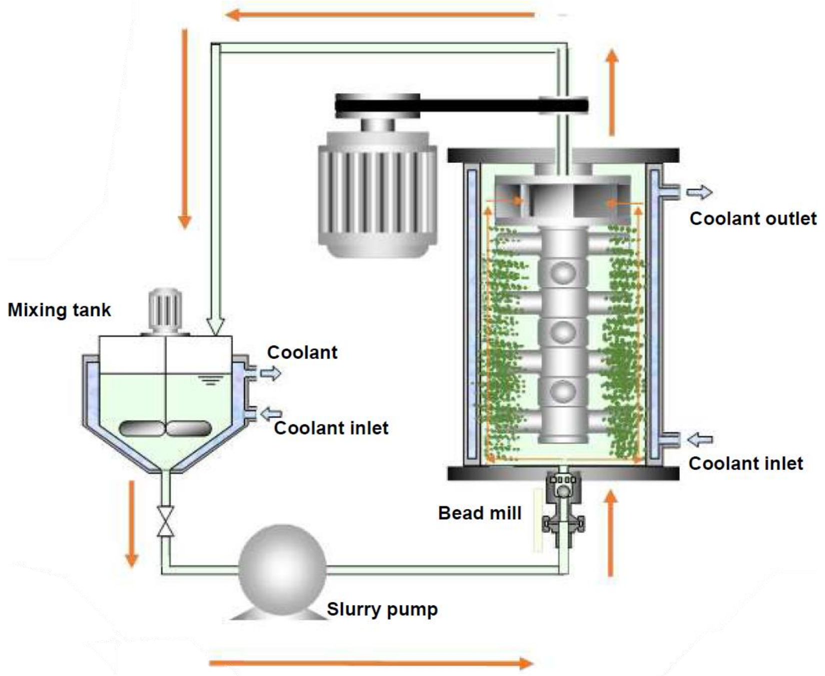


- Increasing the tungsten doping decreases the switching temperature
- Peak is broadened
- Enthalpy decreases - impurities



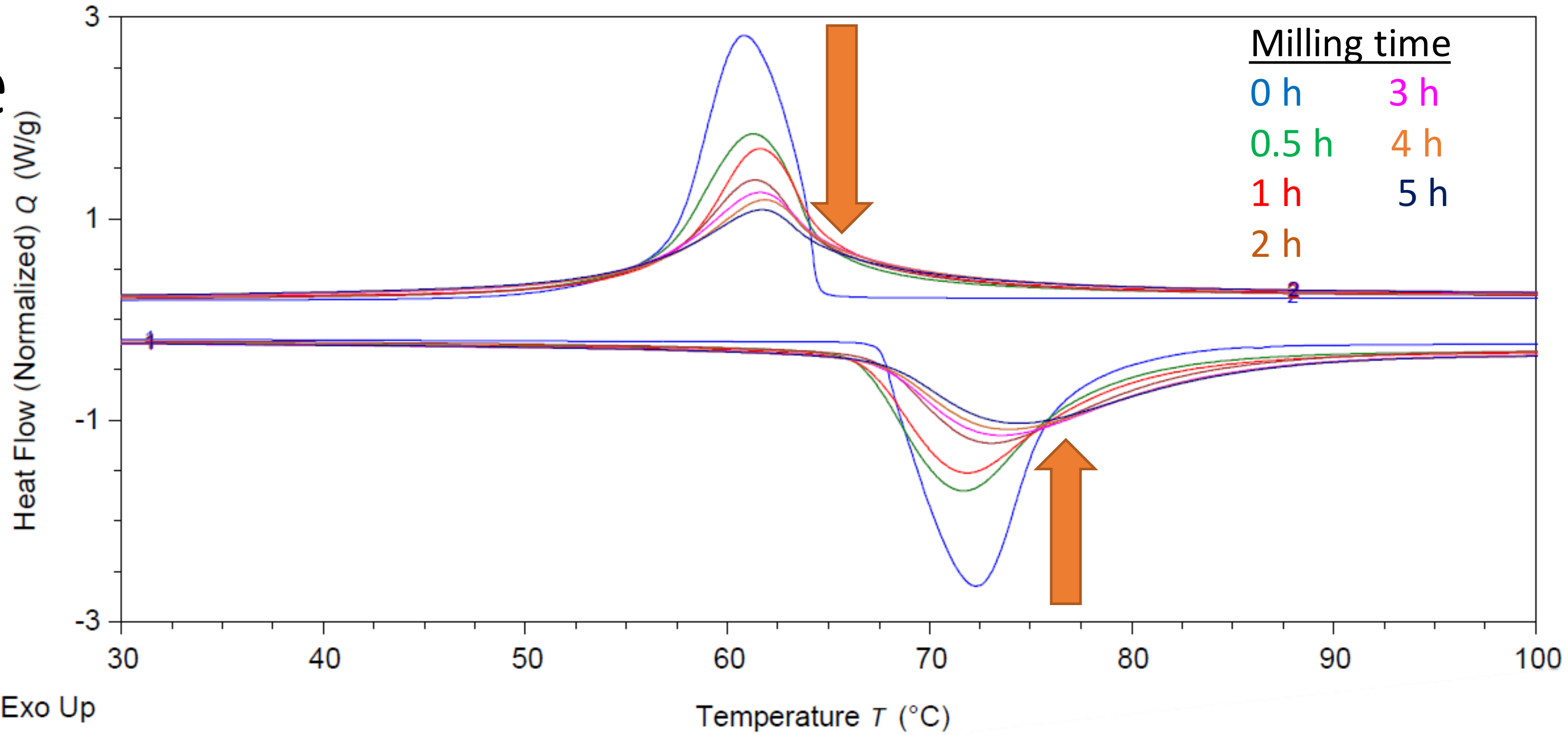
Interreg Vlaanderen-Nederland
SiA Nationaal Regieorgaan Praktijkgericht Onderzoek
IMO-IMOMEC UHASSELT imec
Brightlands Materials Center
ChemCYS

2. Decrease of particle size



Schematic diagram of bead mill[6]

6. Chemtech Company Hiroshima Metal & Machinery CO., L. Bead Mill - Grinding & Dispersing http://www.hiroshimamm-chemtech.com/en/knowledge/detail01/ (accessed Feb 18, 2020).



- Wet bead milling – decreases agglomeration of particles
  - Powders are dispersed in solvent
  - Solvent is degassed to decrease chance of oxidation
  - 5-10 µm to 800 nm (determined by SEM)
- Water cooling during milling
- Effects on structure need to be fully characterised

Conclusion & further works

- Successfully synthesised VO_2 thermochromic powder with **high** purity
- Introduced tungsten successfully as a dopant to lower and tune the switching temperature (-15 to 68 °C)
- Reduced the particle size using bead milling from 5-10 µm to 800 nm
- During the milling the switching enthalpy decreases – further research is require to determine the cause
- Looking into the kinetics of the switch from monoclinic to tetragonal using DSC to compare the activation energy for the different powders

Acknowledgements

- Research group Design and Synthesis of Inorganic Nanomaterials for Energy Applications

M.K. Van Bael, A. Hardy, K. Elen & P. Buskens

- Brightlands Materials Center

R. Habets, L. Leufkens, C. Yeung, D. Mann, T. Kusters & P. Buskens

- Project Funding