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XPS – as a versatile tool in the research of the Department of Inorganic and Physical chemistry, UGent

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Annual XPS -users meeting 16-10-2009

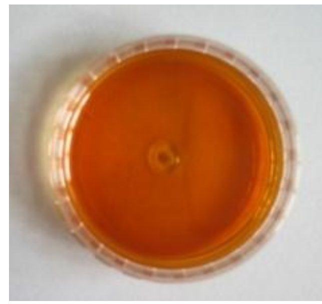
Content

- Thin film superconducting materials by sol gel chemistry
- Fibrous transistors, in cooperation with dep. of Textiles, Ugent
- Biogenic nanoparticles, in cooperation with labMET
- Supported metal oxide catalyst for liquid oxidation reactions

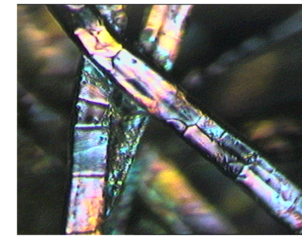
Principle

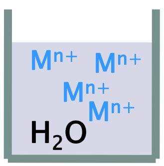
Deposition on substrates by dipcoating, printing

Gelation, T≤60°C

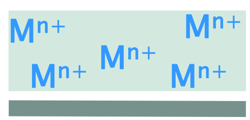


Heat treatment under controlled atmosphere





Sol

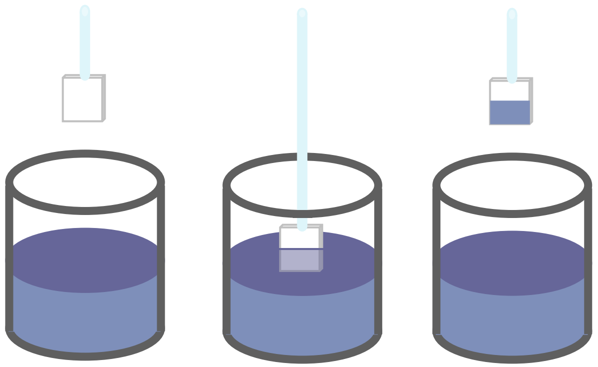


Gel

oxide thin film (5-100 nm)

Deposition

- dip-/spin-coating from aqueous solutions
- ink-jet printing from water-based inks



liquid precursor-layer

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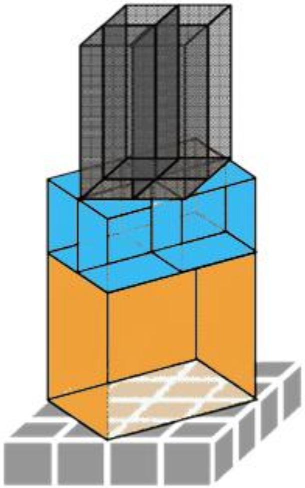
Analysis of solutions, gels and thin films:

- optical analysis : UV/Vis/IR/Raman spectroscopy
- rheology : viscosity, surface tension, contact angle, particle size, ...
- thermal analysis : TGA/DTA/DSC/TMA
- microscopy : AFM, optical, SEM/EDX, (HR)TEM with EELS, STEM, EDX
- structural analysis : XRD, pole figures, BET
- electromagnetic : resistivity measurements
- complexometry : potentiometric titrations
- XPS

Materials

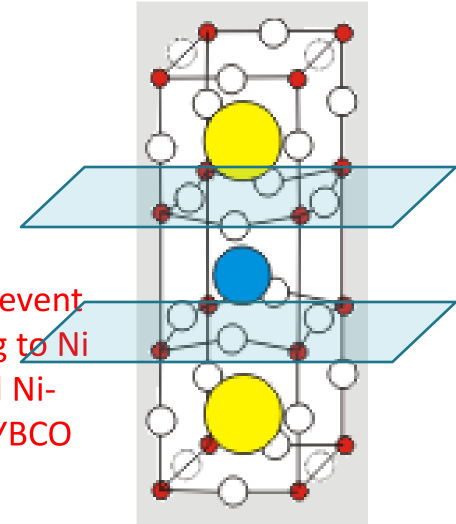
- ceramic high T_c superconductor architectures on NiW tapes :
  YBCO, CeO_2, $La_2Zr_2O_7$
- TiO_2 for self-cleaning surfaces : on ceramic tiles and steel
- YSZ for thermal barrier coatings and solid oxide fuel cells
- mesoporous organosilica layers for low-k dielectrics (through ...)
- Diesel soot catalyst

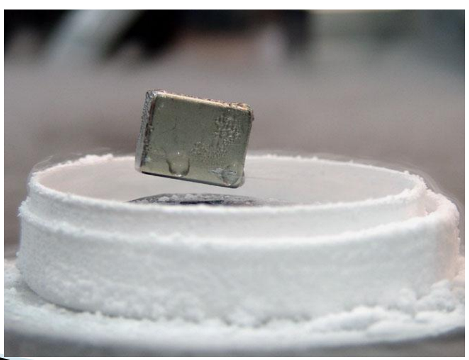
High T_c superconductor architecture :



- YBCO by dipcoating or printing from aqueous solutions
- CeO_2 (50-100 nm) by dipcoating from aqueous solutions
- LZO (250 nm) by dipcoating from aqueous solutions
- Textured, flexible NiW tape

Buffer layers prevent O from diffusing to Ni substrate and Ni-diffusion into YBCO layer



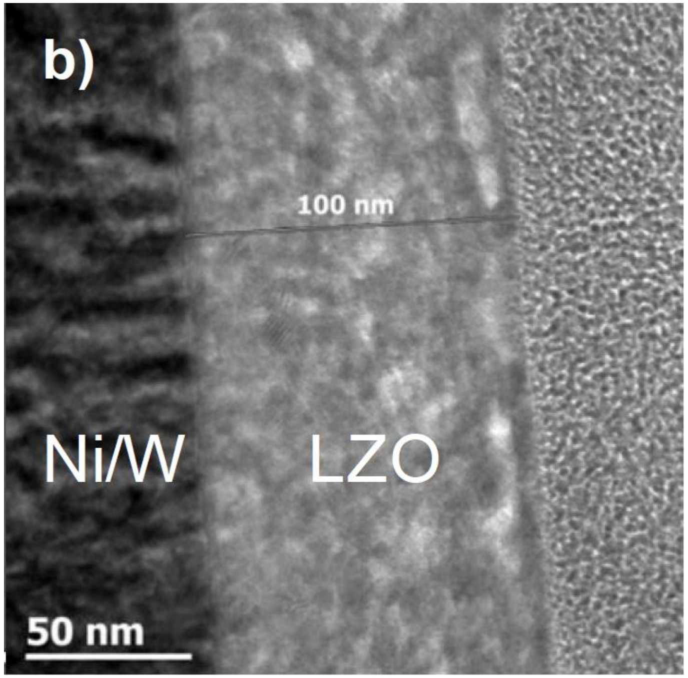


Applications :

-resistance ~0 at -180 °C
- second generation HTS wires
- fault current limiter
- magnets
- coils for renewable energy
- induction heaters ...

High T_c superconductor HTS architecture: XPS, TEM, FIB

(HR)-TEM analysis of interface, surface, crystalinity/texture

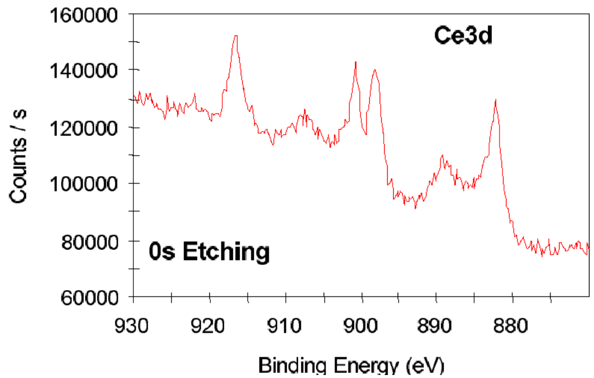


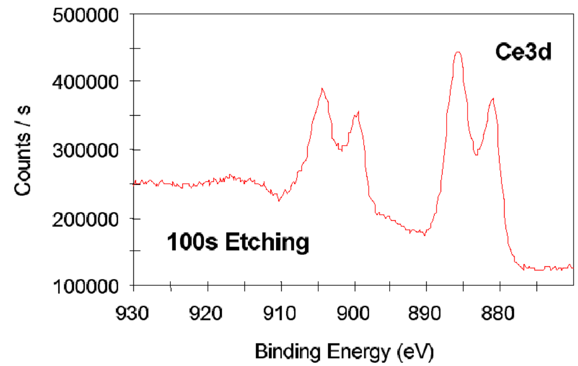
Cs-corrected TEM equiped with EELS, STEM, EDX ...

Sputter XPS analysis depth profiling

- Ni - diffusion through buffer layers for ≠ processing
- determination of layer thickness
- compositional analysis
- degree of oxidation

Study of oxidation state of Cerium and Ni-diffusion, a single layer pHd Greet Penneman (2007) Escalab 250 (VG Scientific)





Ce^{+4} ion : (Xe) = $5p^6\ 4f^0$

Ce^{+3} ion: (Xe) $4f^{1}$ = = $5p^6\ 4f^1$

Ce–O is not 100 % ionic

ionic: Ce: $5p^6\ 4f^0$ O: $1s^2\ 2s^2\ 2p^6$

less ionic: Ce: $5p^6\ 4f^1$

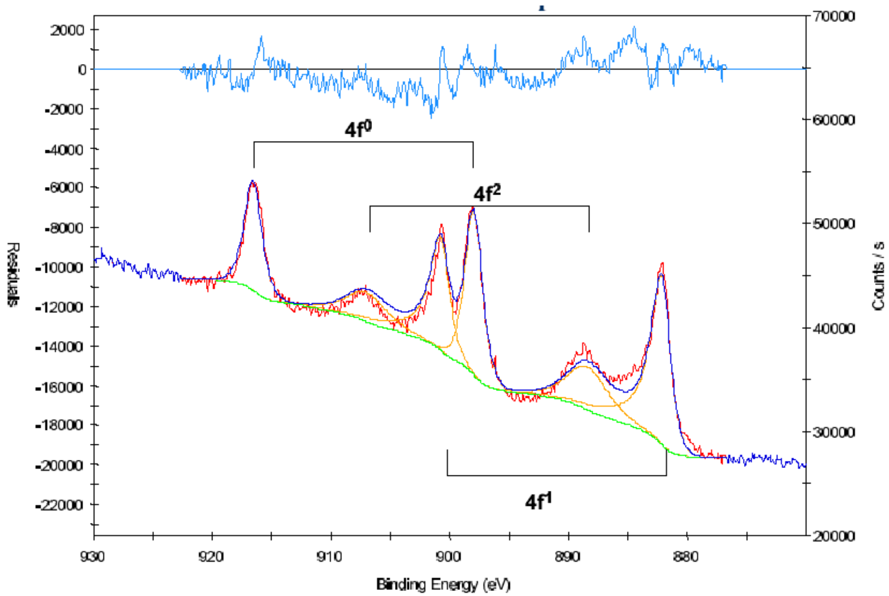
Ce: $5p^6\ 4f^2$

Ionic: Ce: $5p^6\ 4f^1$ O: $1s^2\ 2s^2\ 2p^6$

less ionic: Ce: $5p^6\ 4f^2$

Burrough's notation:		v_0	v	v′	v″	v‴
Ce $3d_{5/2}$	Origin	Ce^{3+}	Ce^{4+}	Ce^{3+}	Ce^{4+}	Ce^{4+}
	Shift (eV)	−36.1	−34.1	−30	−27.85	−18.3
	FWHM	4.11	5.77	3.76	2.69	3.96
		u_0	u	u′	u″	u‴
Ce $3d_{3/2}$	Origin	Ce^{3+}	Ce^{4+}	Ce^{3+}	Ce^{4+}	Ce^{4+}
	Shift (eV)	−17.8	−15.65	−13.65	−9.25	0
	FWHM	3.91	5.86	4.00	4.64	1.39

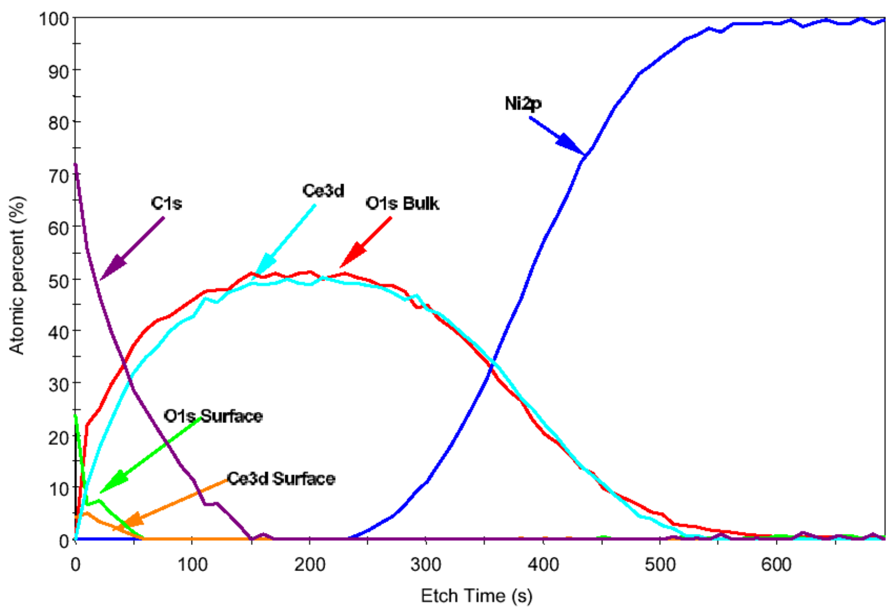
Study of oxidation state of Cerium and Ni-diffusion, a single layer pHd Greet Penneman (2007) Escalab 250 (VG Scientific)



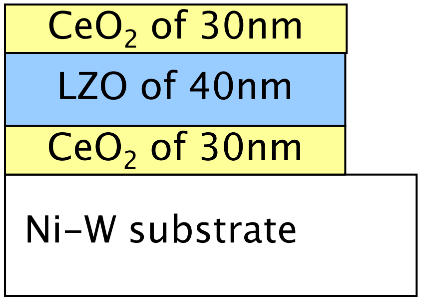
XPS narrow scan of the Ce 3d region showing the typical spectrum for Ce^{4+}.

Fitting: contstraints: doublet separation, peak position, FWHM, peak shape

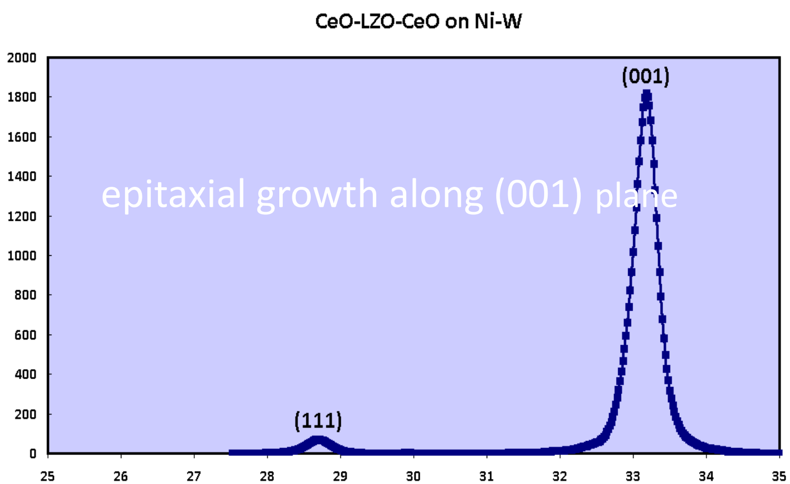
Top: Ce (IV)
Core: Ce (III)
Due to sputtering?



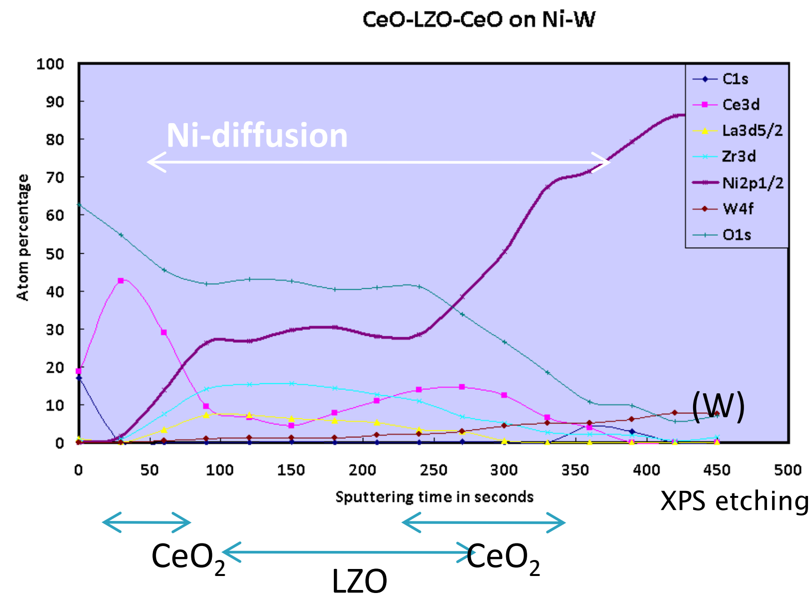
Continuation : Multiple layers, Vyshnavi Narayanan



The objective is to study the role of buffer layers in transferring the texture of Ni-W substrate to YBCO (XRD) and its effective role in prevention of penetration of Ni into YBCO



XRD spectrum



"Multiple buffer layer architecture in HTSC- Deposition and characterization"
V. Narayanan, V. Cloet, N. Van de Velde, P. Lommens, K. De Buysser, E. Bruneel, I. Van Driessche and S. Hoste, Eucas 2009, Berlin

Biogenic metals: XPS analysis of cerium from organic origin

B. De Gusseme, Prof. Verstraete (LabMET)

Goal:

In a biological process cerium-ions are removed from a solution into a solid state. The question was to identify the oxidation number of the cerium in the solid state.

The paste like substance was spread onto a Sn-substrate. It is shown that the presence of cerium in the sample could be confirmed. The oxidation state of the cerium is Ce(III)

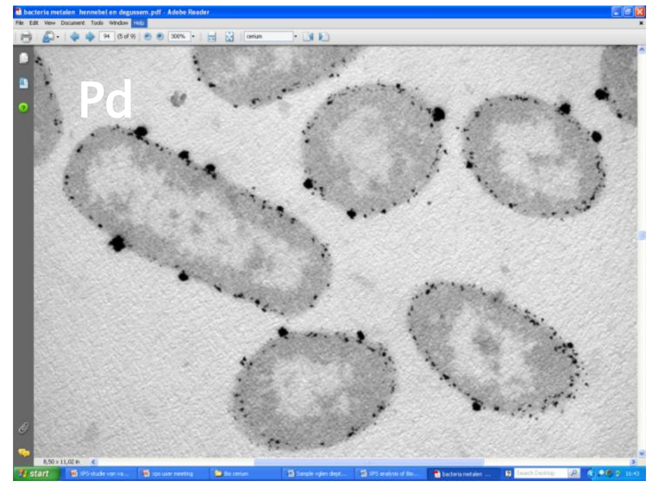
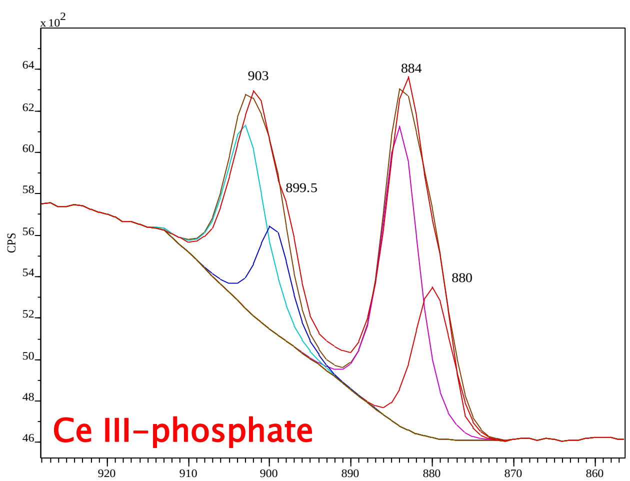


Illustration: TEM of zerovalent paladium particles precipitated at the cell surface of Shewanella oneidesis

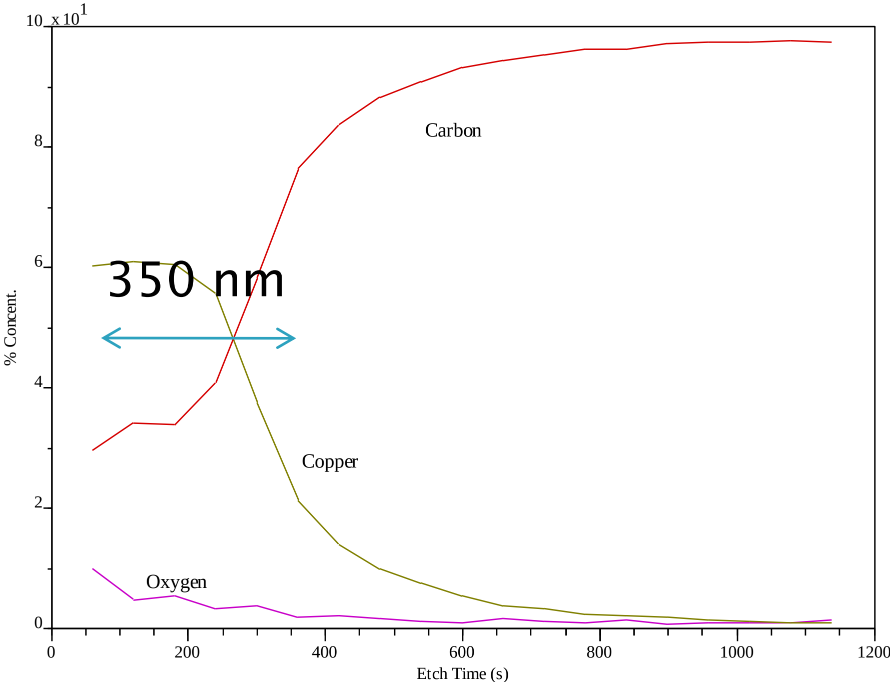
Oxidation state	BE (eV)
Ce(IV)	898.4 $3d^{5/2}$ 916.9 $3d^{3/2}$
Ce(IV)	888.9 $3d^{5/2}$ 907.5 $3d^{3/2}$
Ce(IV)	882.3 $3d^{5/2}$ 901.0 $3d^{3/2}$
Ce(III)	880 $3d^{5/2}$ 899 $3d^{3/2}$
Ce(III)	886 $3d^{5/2}$ 904 $3d^{3/2}$

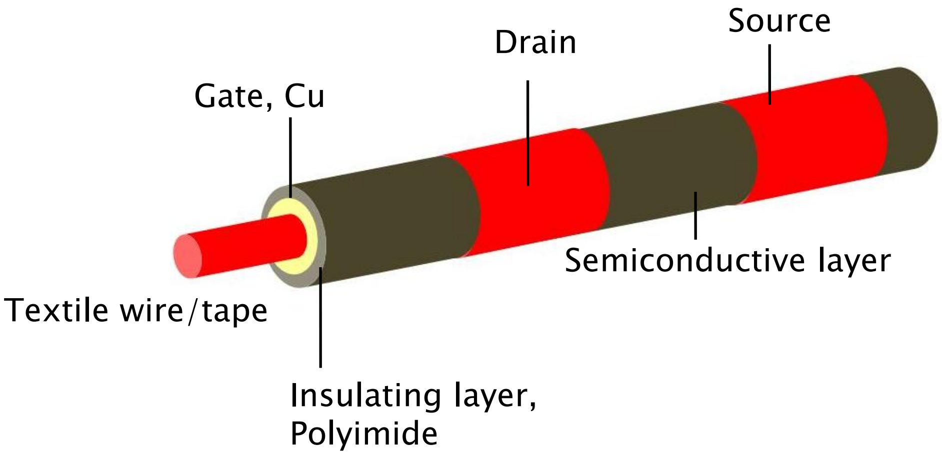
Table 1: Overview literature Ce3$d^{3/2}$ and 3$d^{5/2}$



Biogenic metals in advanced water treatment
TRENDS IN BIOTECHNOLOGY 27,2 , 90-98 FEB 2009 Tom Hennebel, Bart De Gusseme, Nico Boon, Willy Verstraete, UGENT

Textile based transistor
Dep. Textiles A. Swhwarz, prof. Van Langenhove





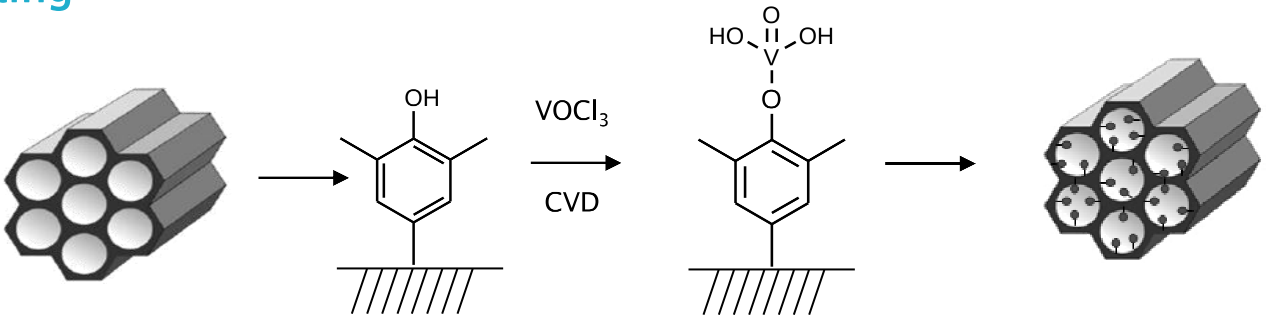
Determination of the thickness of the Cu-layer. Based on differences in etch-rate between Ta_2O_5 and Cu

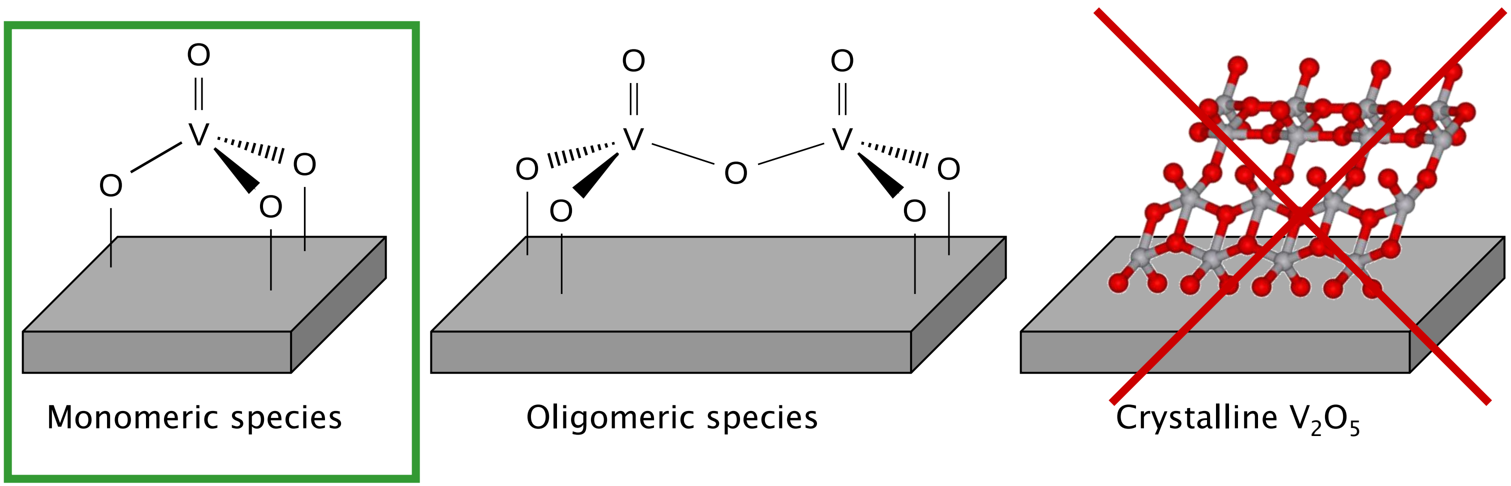
Steps towards a textile based transistor – Development of the gate and insulating layer, A. Schwarz, J. Cardoen, E. Bruneel, J. Hakuzimana, Philippe Westbroek, S.Hoste, Lieva Van Langenhove, submitted

Ultra-stable and zero leaching supported metal oxide catalyst for liquid oxidation reactions

Study of Vanadiumoxide on mesoporous fenol resins
Ilke Muylaert, prof. Van Der Voort

Vanadium grafting





Muylaert I, *et al.*, Phys. Chem. Chem. Phys., (2009),

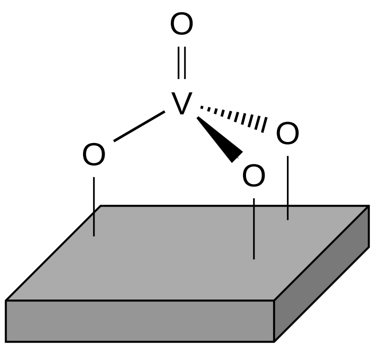
Characterization – XPS
Determination oxidation state of Vanadium

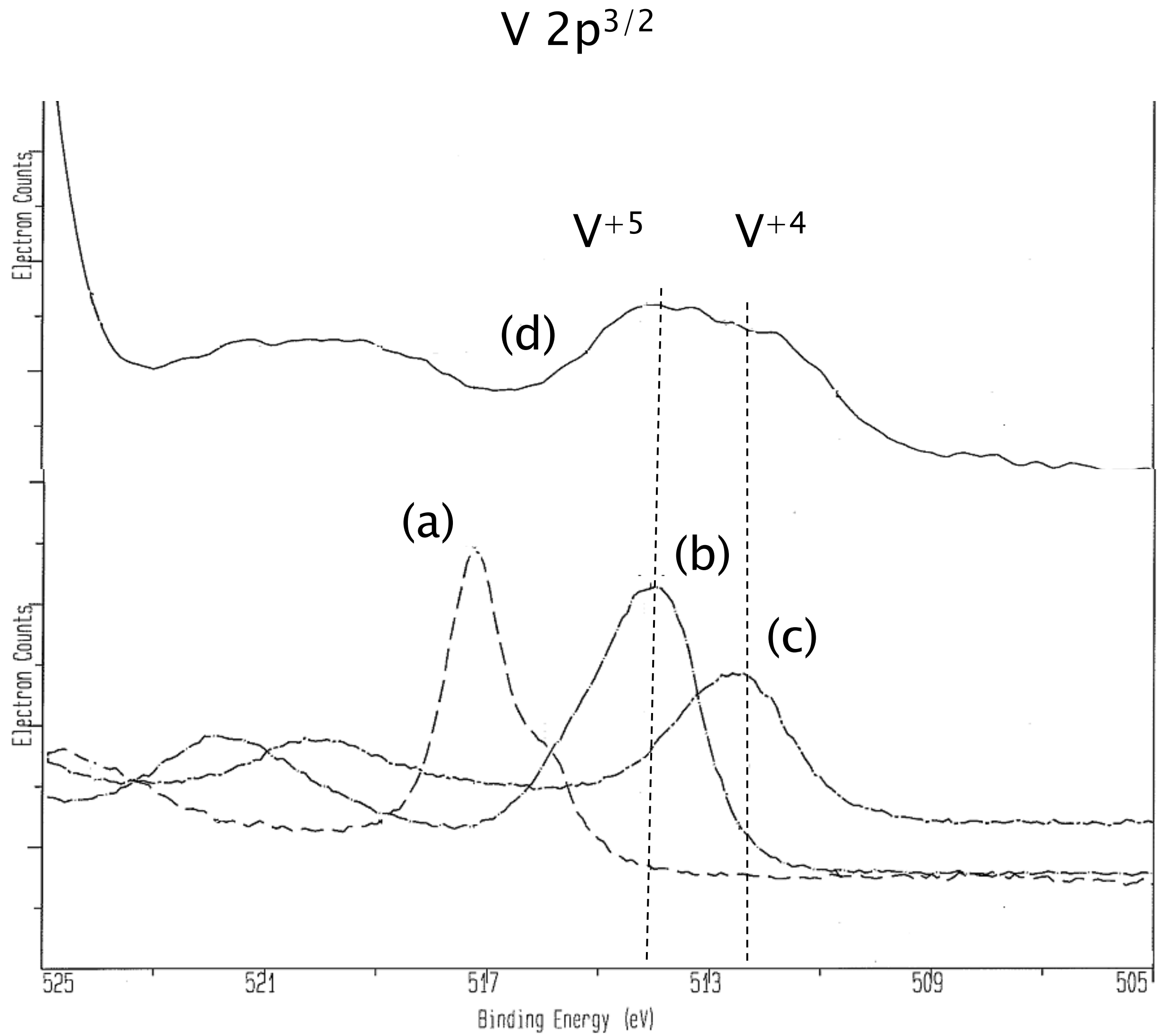
(a) V_2O_5 Crystalline

(b) NH_4VO_3 +5

(c) $VO(acac)_2$ +4

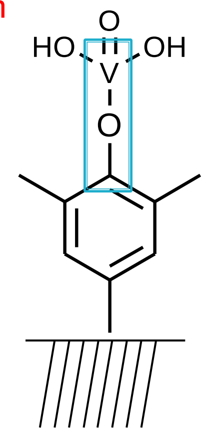
(d) P/F Resin +5 (+4)



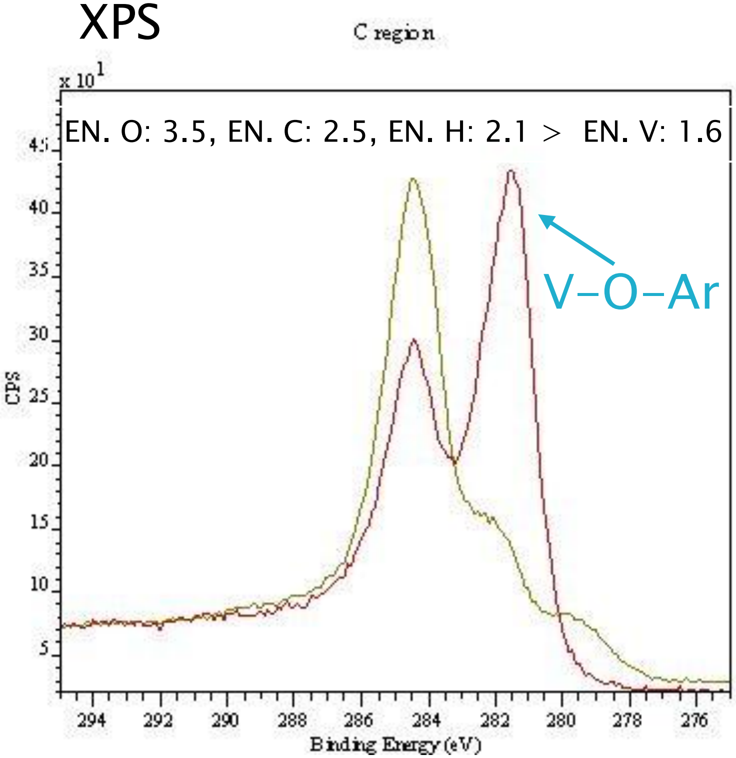


Structural information:
C– peak at low energy 281.5 eV: C–OV
Due to low electronegativity and soft center of vanadium atom

XPS and DRIFT spectra before and after grafting with Vanadium

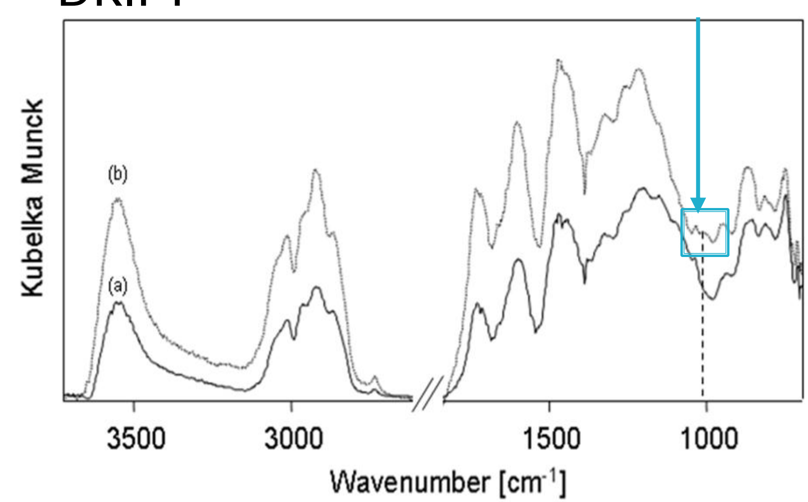


XPS



DRIFT

V–O–Ar



Thanks