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JOURNÉES DES SPECTROSCOPIES D'ELECTRONS

Characterization of a NiCr sub-nanometric layer / ZnO thin layer interface and its evolution with annealing

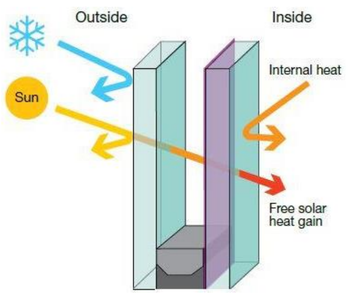
[1]Justine Voronkoff, [1]Hervé Montigaud, [2]Ludovic Largeau

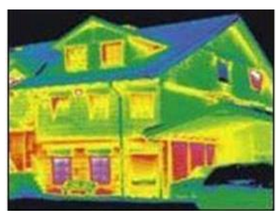
[1]SVI (Surface du Verre et Interfaces), CNRS/Saint-Gobain, Paris

[2]C2N (Centre de Nanosciences et de Nanotechnologies), CNRS, Université Paris Sud

Industrial context

Isolated Glazings

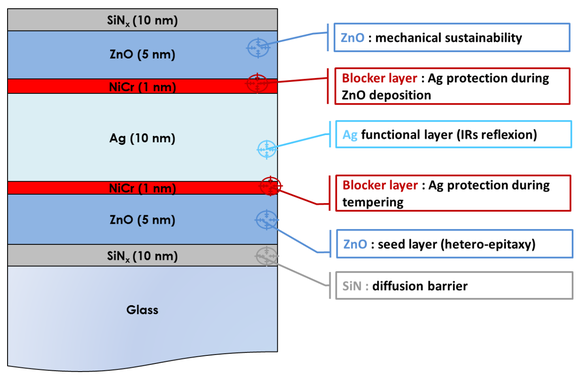




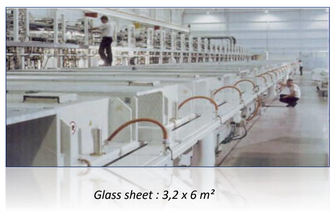
Low-Emissivity

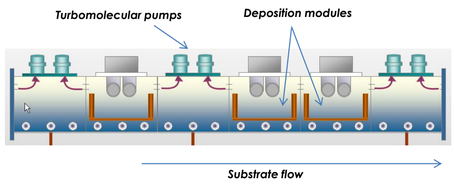
Silver-based stacks

Blockers at the interfaces Ag/dielectrics

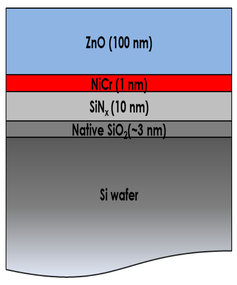


PVD magnetron sputtering





Simplified stack

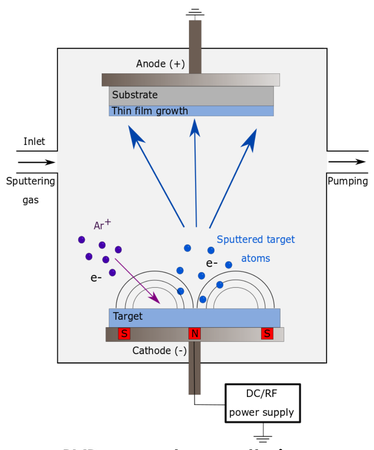


NiCr blocker-layer :

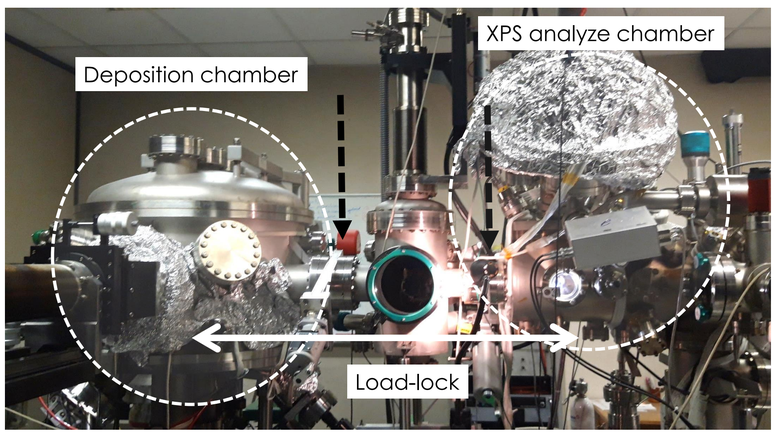
thickness = 1 nm
material : NiCr

1 nm interface "layer" characterization ?
Impact of the ZnO sputtered layer above ?
Evolution with annealing ?

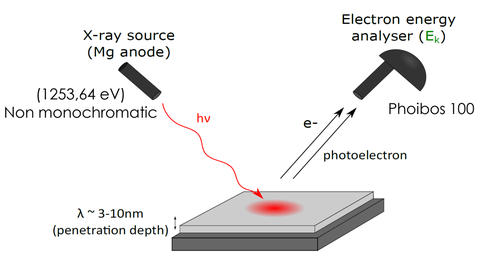
In situ XPS



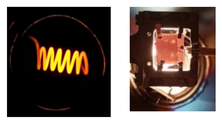
PVD magnetron sputtering principle



Deposition chamber linked to analyzes chamber

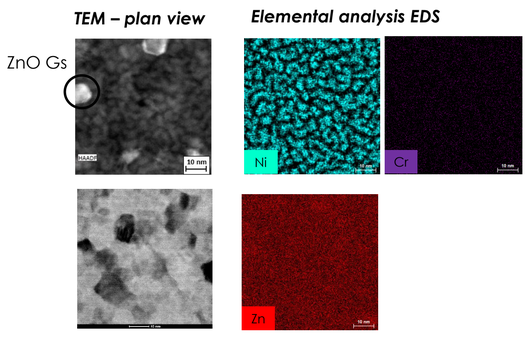


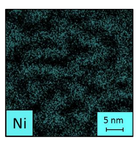
XPS and heater in situ



NiCr / ZnO interface

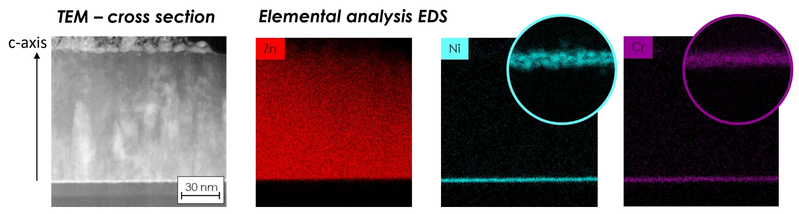
TEM – plan view *Elemental analysis EDS*





**On SiN under-layer without ZnO above

TEM – cross section *Elemental analysis EDS*



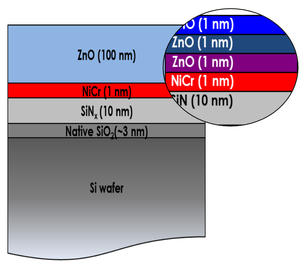
Microstructure

Ni discontinuous layer
**No modification with ZnO
Cr homogeneous layer

ZnO polycrystalline layer
Columnar growth (wurzite)

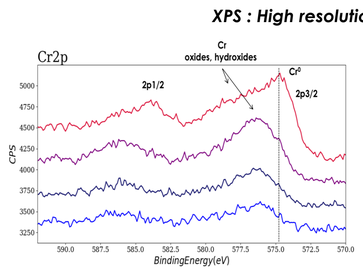
No link between ZnO grains localization & Ni distribution

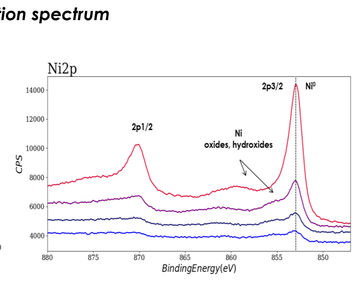
Oxydation state



Successive layers deposition

XPS : High resolution spectrum





	% Cr metallic	% Cr oxides + hydroxides
NiCr (1 nm)	36	64
ZnO (1 nm)	5	95
ZnO (2 nm)	0	100
ZnO (3 nm)	0	100

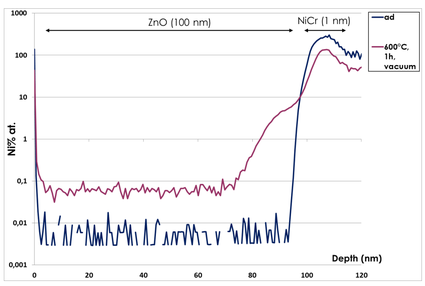
Chromium fully passivated

	% Ni metallic	% Ni oxides + hydroxides
NiCr (1 nm)	45	55
ZnO (1 nm)	32	68
ZnO (2 nm)	34	66
ZnO (3 nm)	46	54

Nickel partially oxidized ~ 50%

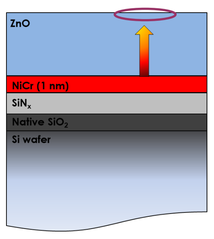
Vacuum annealing impact

Ni migration



Tof-SIMS depth profiles

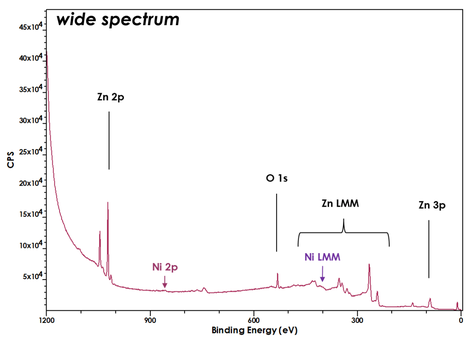
Ni diffusion within ZnO



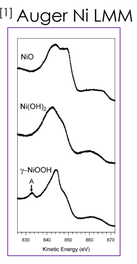
600°C, 1h, vacuum

Ni chemical forms

XPS in situ heating

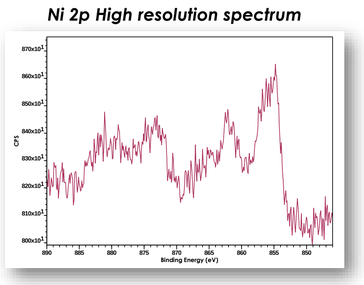


[1] Auger Ni LMM

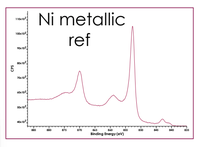


Ni LMM within Zn LMM

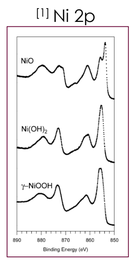
Ni 2p High resolution spectrum



Ni^{2+}/Ni^{3+} mixture at the surface

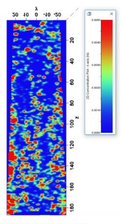


[1] Ni 2p



Nanoscale evolution

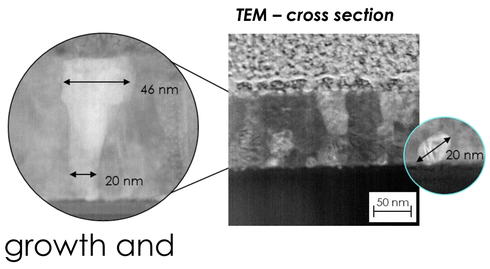
Atom Probe Tomography
[Credits : Jacques Perrin Toinin, RWTH]



2D Ni concentration

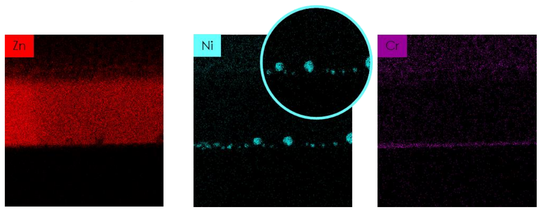
ZnO grain boundaries filled by Ni

TEM – cross section



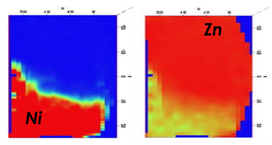
ZnO grains growth and crystallization improvement

Elemental analysis EDS



Ni-rich clusters formation at the NiCr / ZnO interface @600°C

Atom Probe Tomography
[Credits : Jacques Perrin Toinin, RWTH]



element	O	Zn	Ni
Concentration* (at%)	27	19	54

*underestimation of O–content : evaporation rate of oxygen slower than for Ni and Zn with those conditions

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References

[1] M.C. Biesinger et al., Applied Surface Science 257 (2011) 2717–2730
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