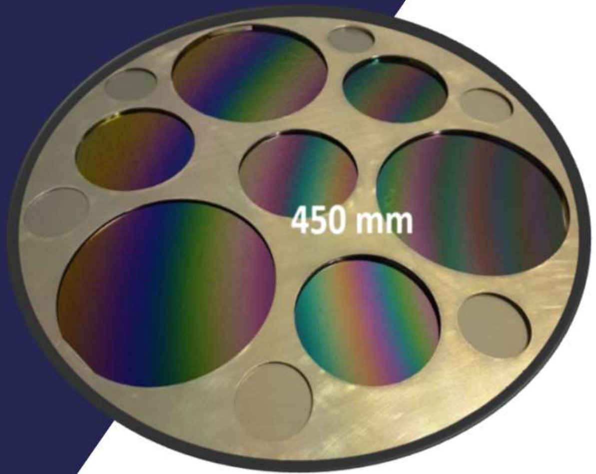
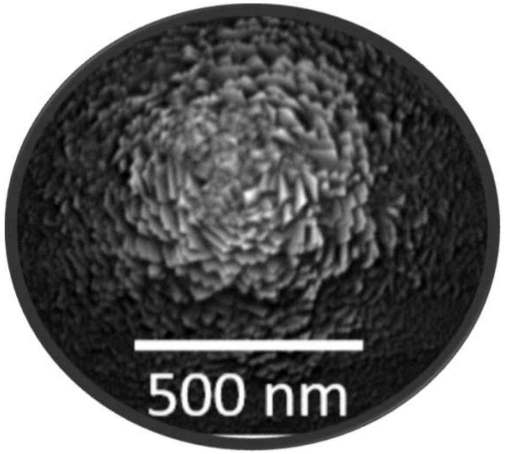
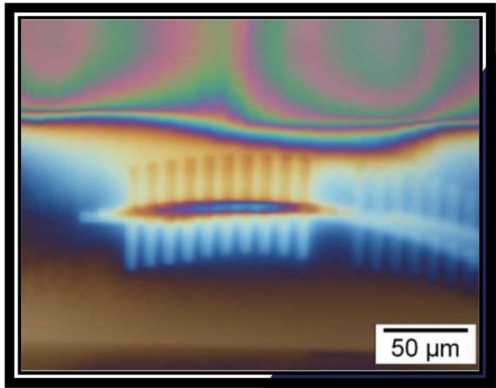
CHEMICAL BEAM EPITAXY LASER-ASSISTED
Sybilla equipment

Giacomo Benvenuti, PhD.

E. Wagner (PhD),
S.C. Sandu (PhD),
W. Maudez (PhD),
S. Bagdzevicius (PhD),
R. Rani (PhD)







19 November 2021   EPIDOX Conference

SUMMARY

WHY WE NEED A NEW TOOL?

CBE-CBVD & SYBILLA EQUIPMENT

COMBINATORIAL GROWTH

ADDITIVE GROWTH

SOME FUNCTIONAL MATERIALS

CONCLUSION & OUTLOOK

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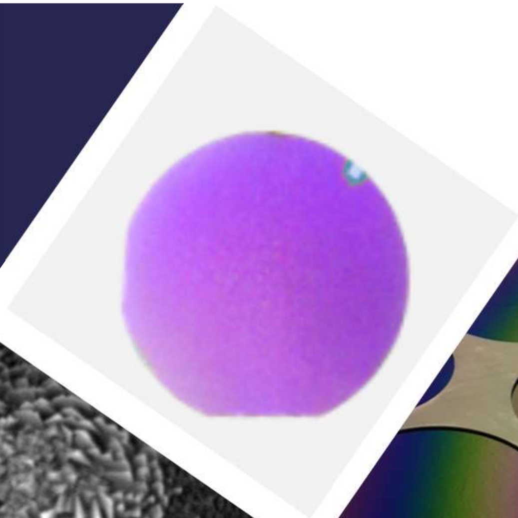
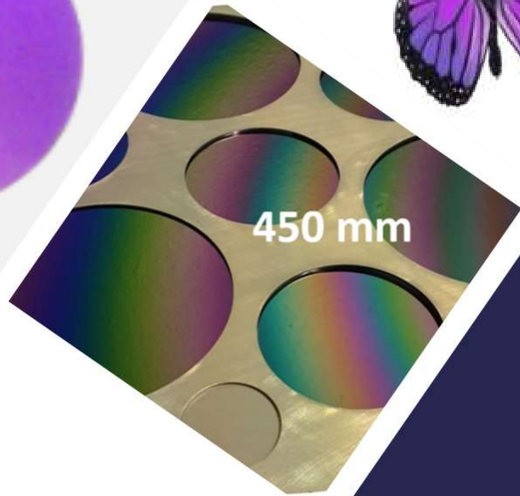
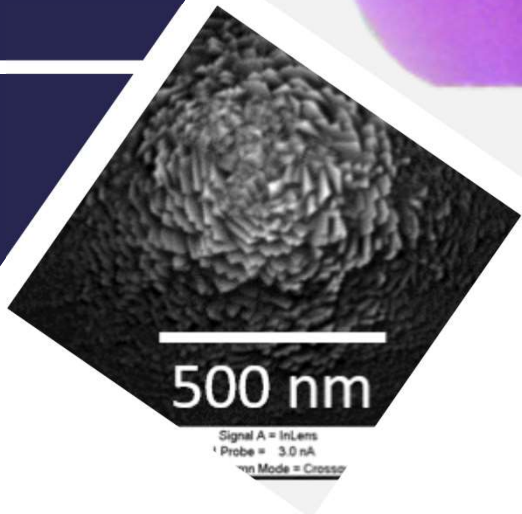
GLOBAL CONTEXT & 3D-OXIDES







3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

OXIDE THIN FILMS

3D-OXIDES MULTI-FUNCTIONAL THIN FILMS

OXIDE THIN FILMS OFFER UNIQUE OPPORTUNITIES

- Replace scarce or toxic elements
- Multi-functional materials for new devices (More than Moore)

HOWEVER, THEY ARE FAR MORE COMPLEX !

- Huge amount of combinations
- Higher temperatures for epitaxy and crystals quality
- Strong material properties variations for non perfect crystals
- Complex Figures of Merit for multi-functional materials

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Chemical Beam Epitaxy Laser-Assisted (CBE-LA)

Sybilla Equipment

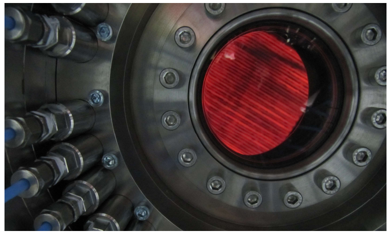



3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

CBE Strengths & Weaknesses

3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

Chemical Precursors in Molecular Vacuum



CBE vs. PVD techniques

+

- Chemical selectivity (2 different regimes)
- Lower process temperatures
- Less sensitive to gas contamination (reduced getter effects)
- Small gas sources for better control

−

- Chemistry
- Complex multi-parameter process

CBE vs. CVD techniques

+

MOLECULAR Vacuum

- No gas phase reaction
- No boundary layers with slow diffusive processes
- Line of sight & easy impinging rates modeling
- UHV characterization techniques
- Beam-assisted deposition & use of very reactive species

−

- Reduced number of available precursors

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Available Chemical Precursors

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	Ar
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	*	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
Fr	Ra	*	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Uub	Uut	Uuq	Uup	Uuh	Uus	Uuo

*	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
*	Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

Mastered in 3D-oxides

Under investigation in house

Reported in literature

Radioactive, toxic or unstable

Semiconductors in literature

Oxygen plasma

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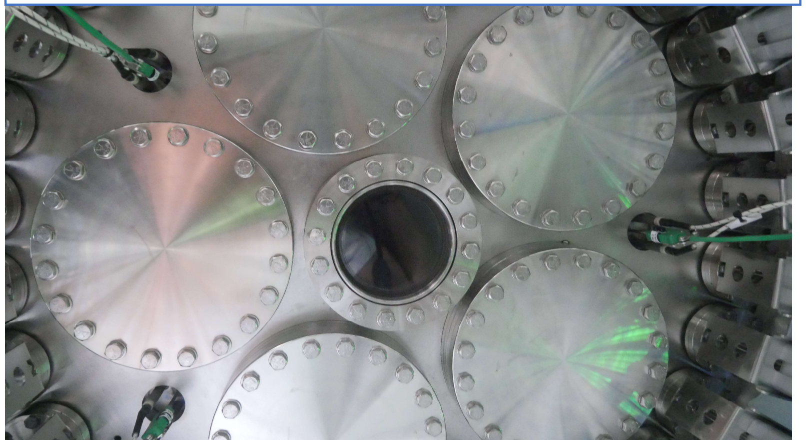
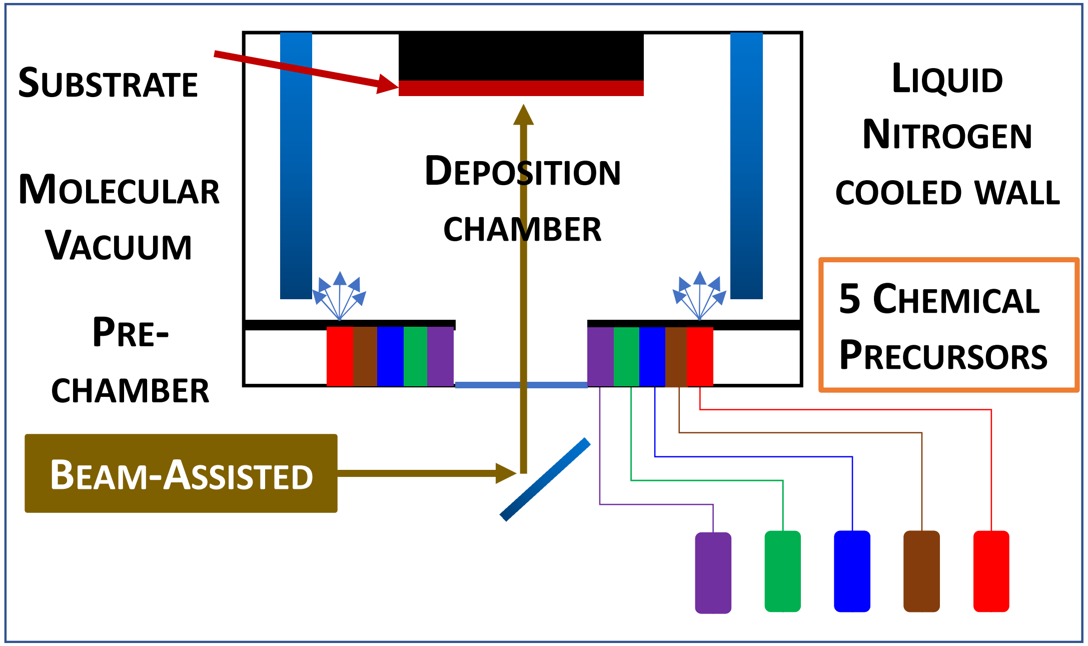
Sybilla for 450 mm substrates set-up

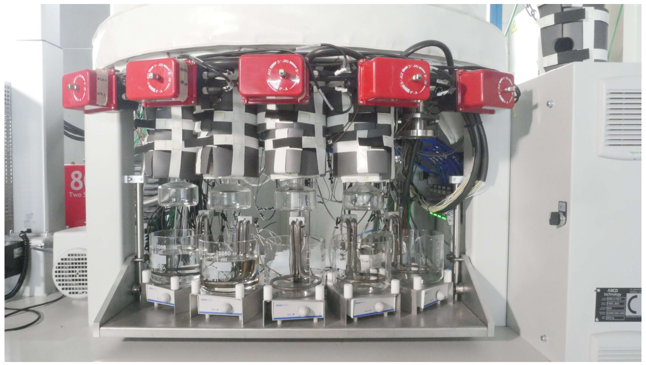
3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

Single wafer Sybilla-450 mm
Merging CVD and MBE assets



Windows for assisted deposition or characterization techniques





5 independent precursor sources

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SIDE AND TOP VIEWS OF SYBILLA EQUIPEMENT

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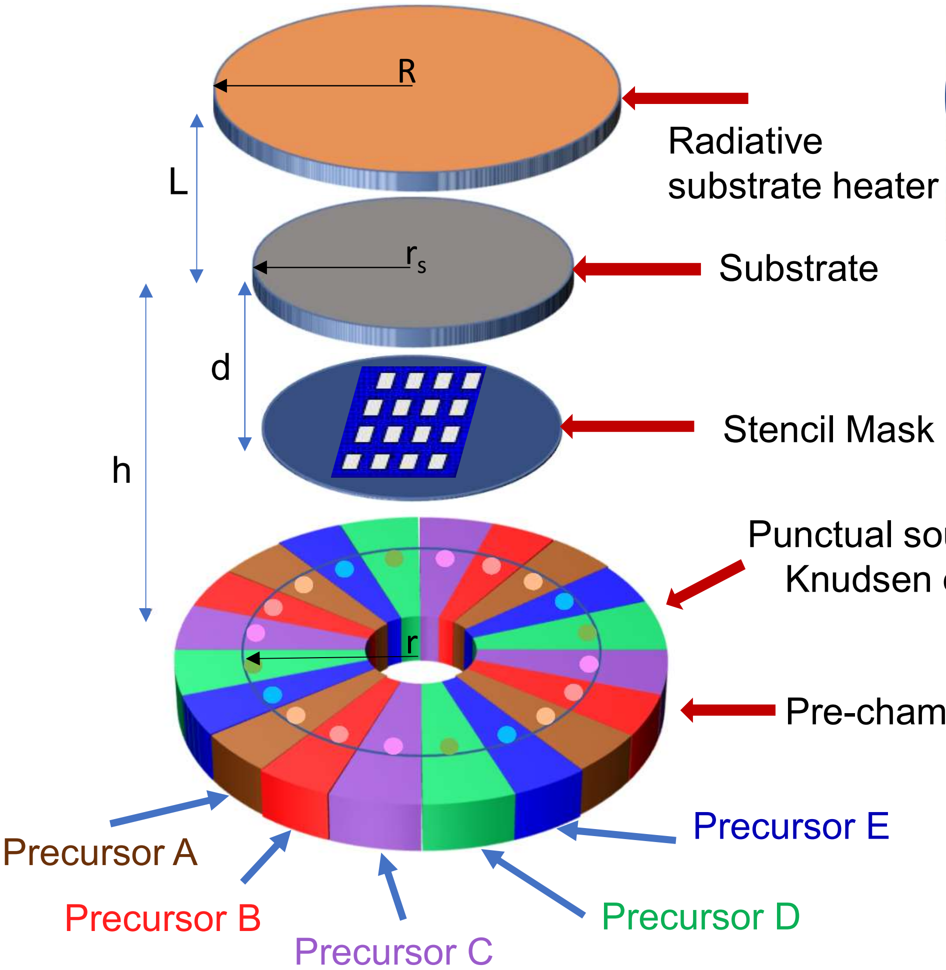
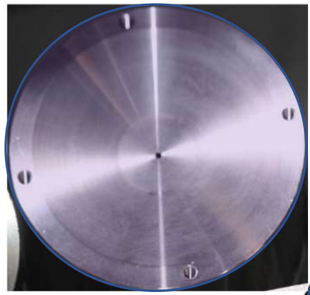
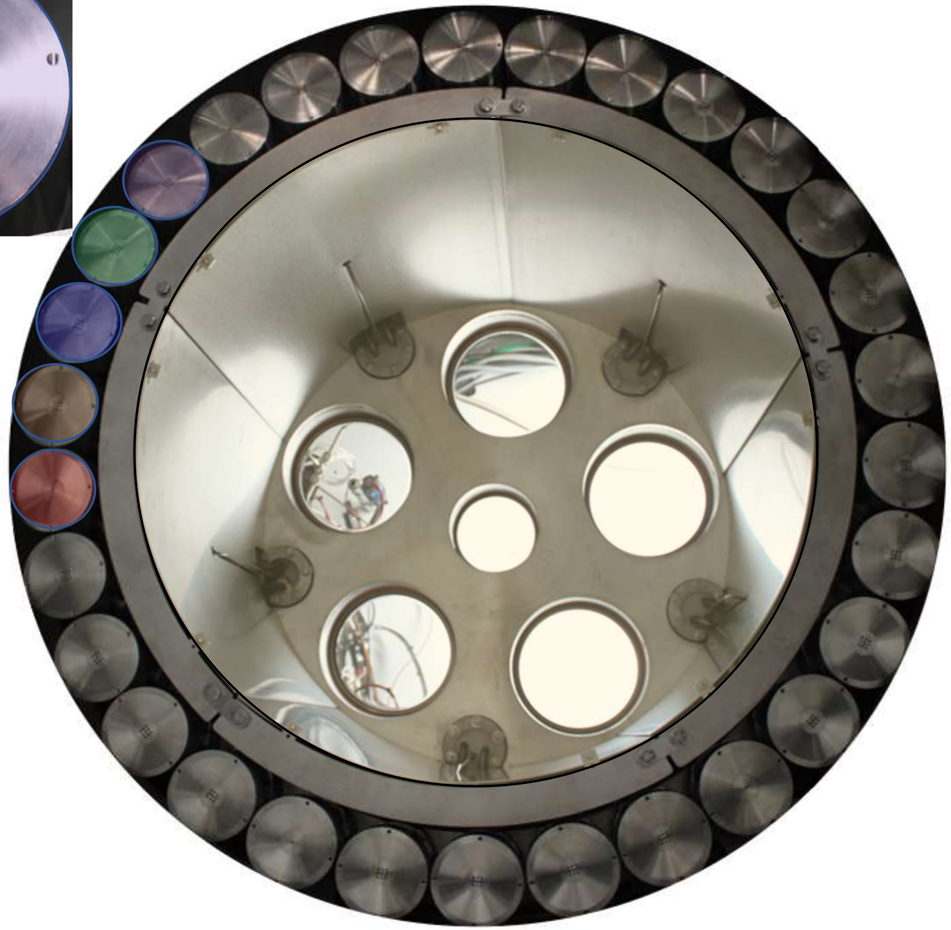
MULTI-FUNCTIONAL THIN FILMS







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SYBILLA (CBE-LA) PERFORMANCES

3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

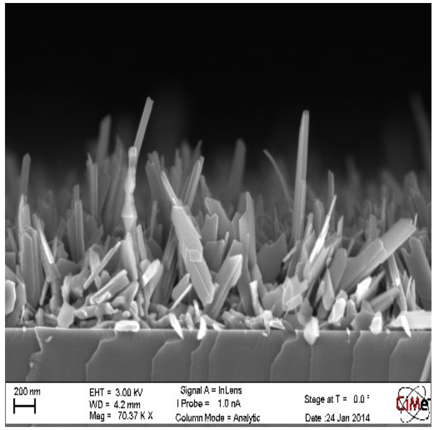
AGILE AND DISRUPTIVE R&D, RELIABLE AND COST EFFICIENT PRODUCTION

Growth rates:	5 nm h^{-1} up to 20 μm h^{-1}
Layer Quality:	Epitaxial to highly porous thin films
Substrate size:	Scalable to any size
Number of elements:	Actually 1-5, but scalable to 6 or even more

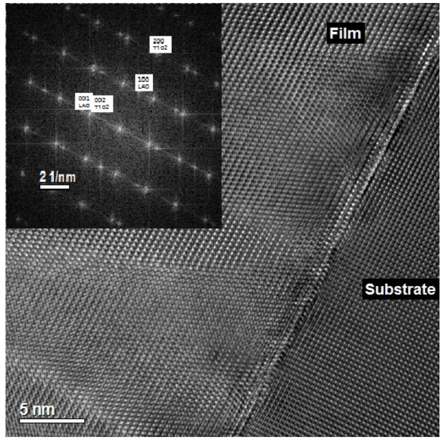
R&D results uptake:	Very fast as the same equipment is used
Process modification:	Very fast: process is not geometry dependent

Precursors use:	From 10% up to as high as 65%
Equipment life-time:	Extensive (different materials/applications)
Costs of ownership:	Possibly lower than few € / cm^2

Combinatorial production:	Monolithic integration of ≠ functionalities



V_2O_5 nano-wires



Epitaxial $LiNbO_3$

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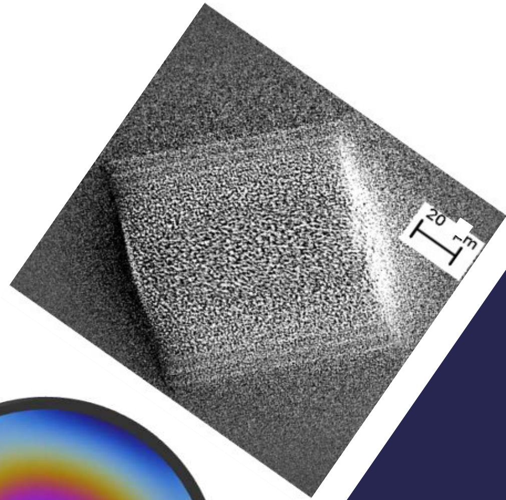
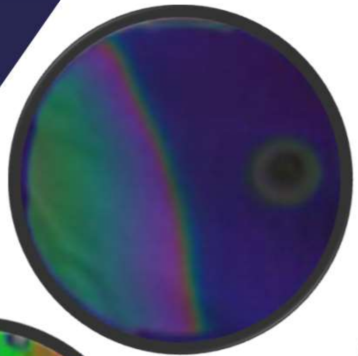
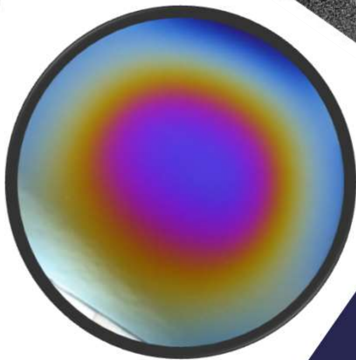
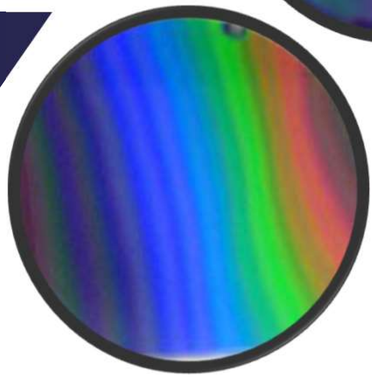
Combinatorial



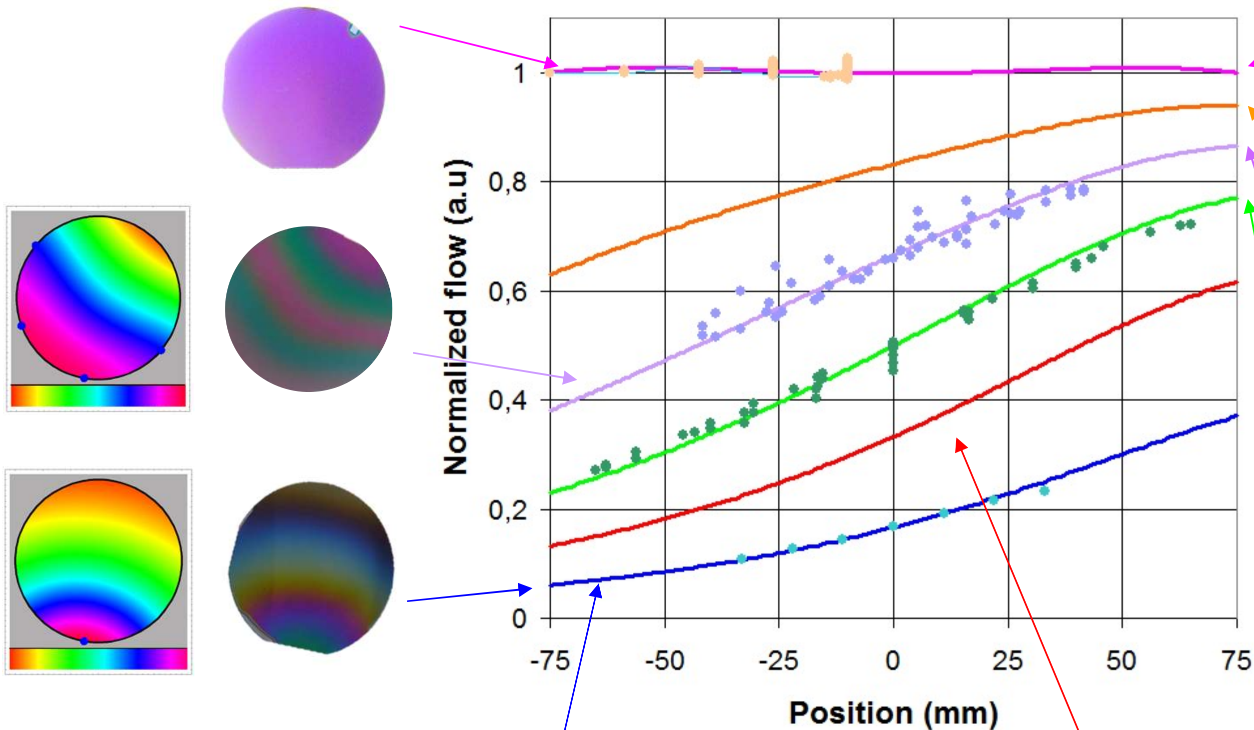




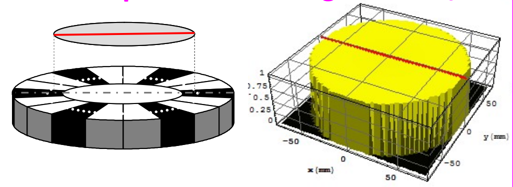


Precursor Flow Gradients

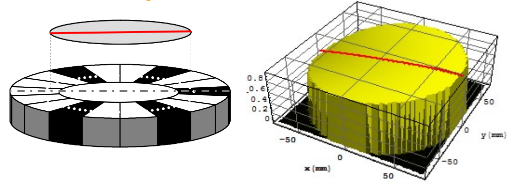
Wagner et al. (2016) ACS Combinatorial Science, 18(3) 154



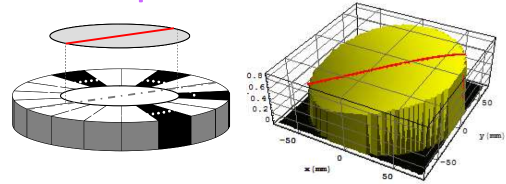
6 active quarters- Homogeneous +/-2%



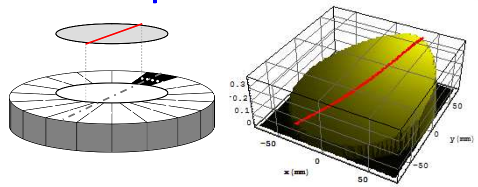
5 active quarters- Flow ratio: 1.5



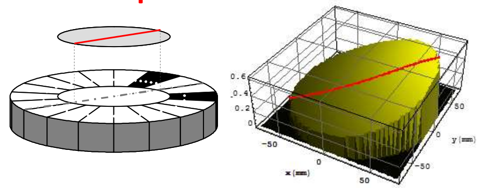
4 active quarters- Flow ratio: 2.2



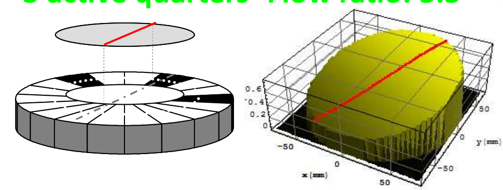
1 active quarter- Flow ratio: 6



2 active quarters- Flow ratio: 4.6



3 active quarters- Flow ratio: 3.3



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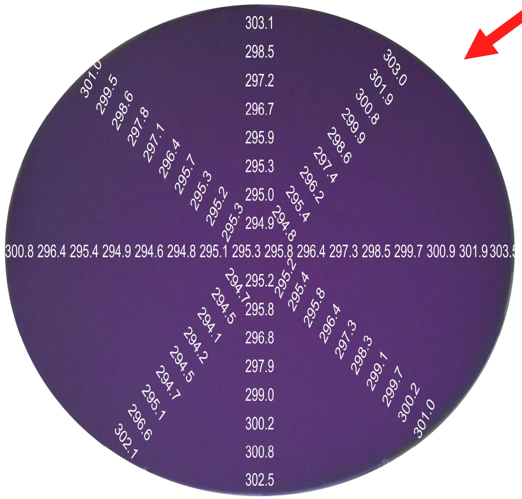
VERY HIGH THICKNESS UNIFORMITY 18’’ SUBSTRATES

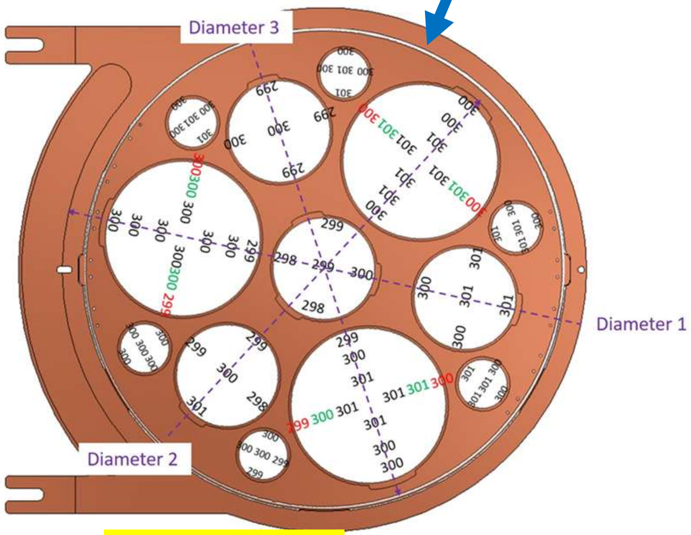
3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

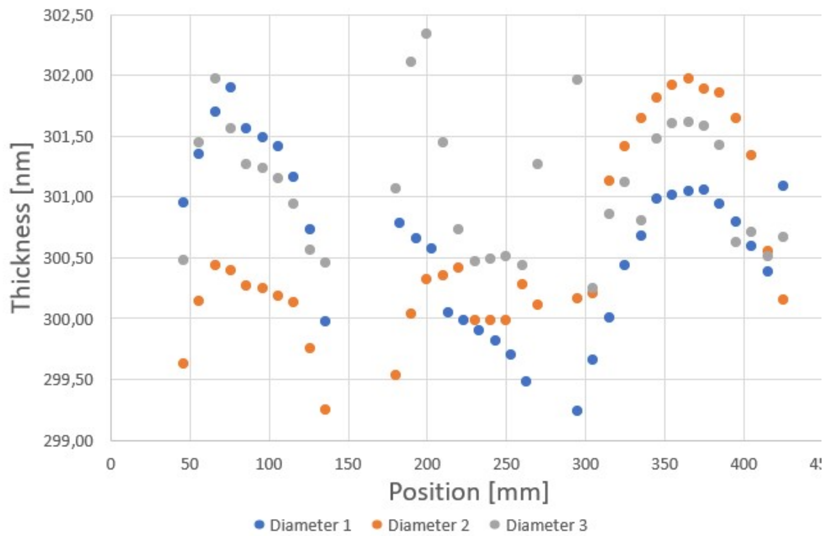
+/- 1,5% uniformity

+/- 0,5% uniformity

	All points	Without extrema
min	299.23	299.66
max	302.34	302.34
average	300.77	300.85
sdtv	0.71127994	0.6668543
Homogeneity	1.03%	0.89%







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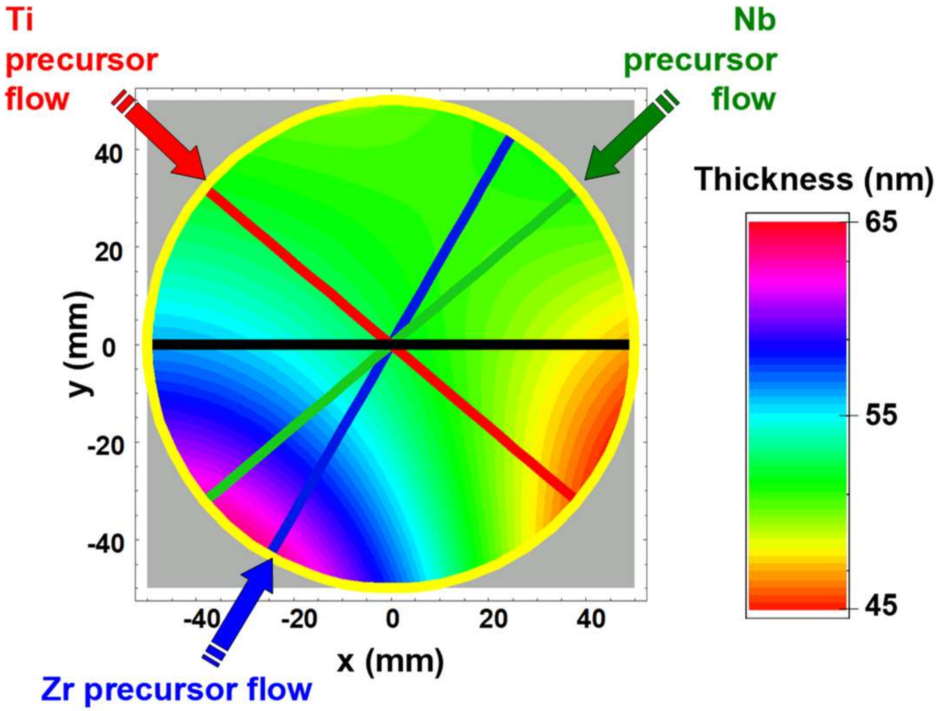
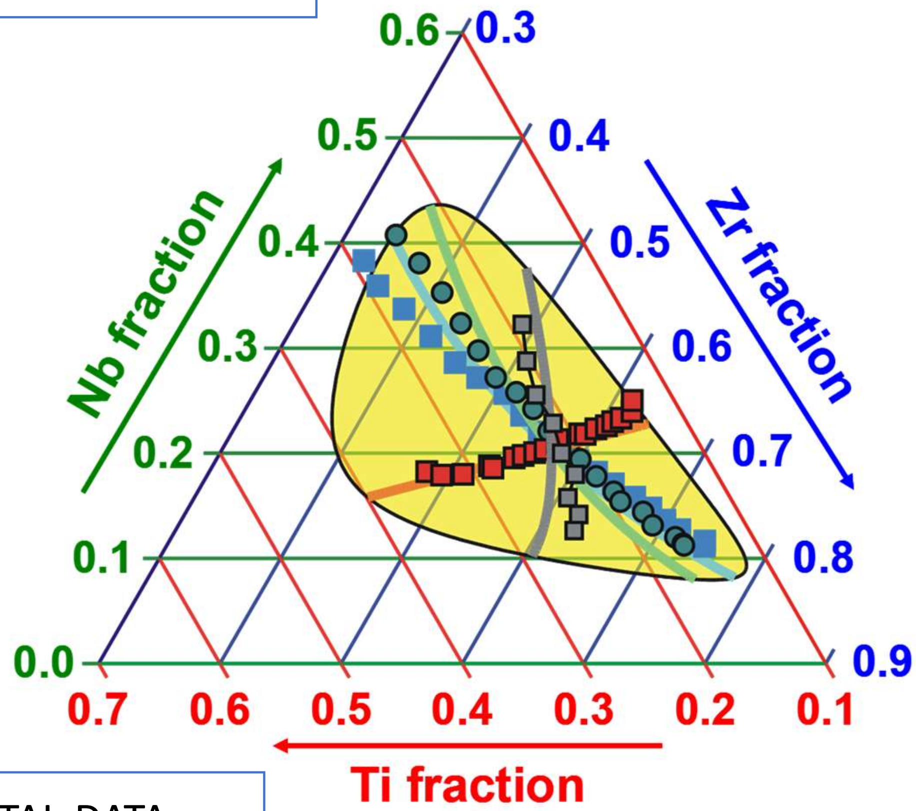
Material Properties vs Composition

Wagner et al. (2016) ACS Combinatorial Science, 18(3) 154

Combinatorial with 3 elements





Simulation vs Experimental data

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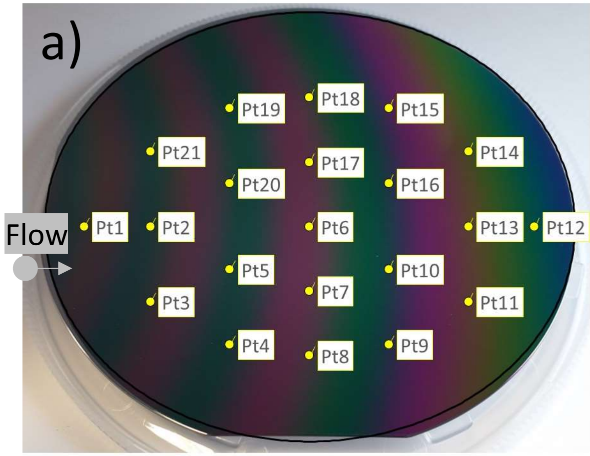
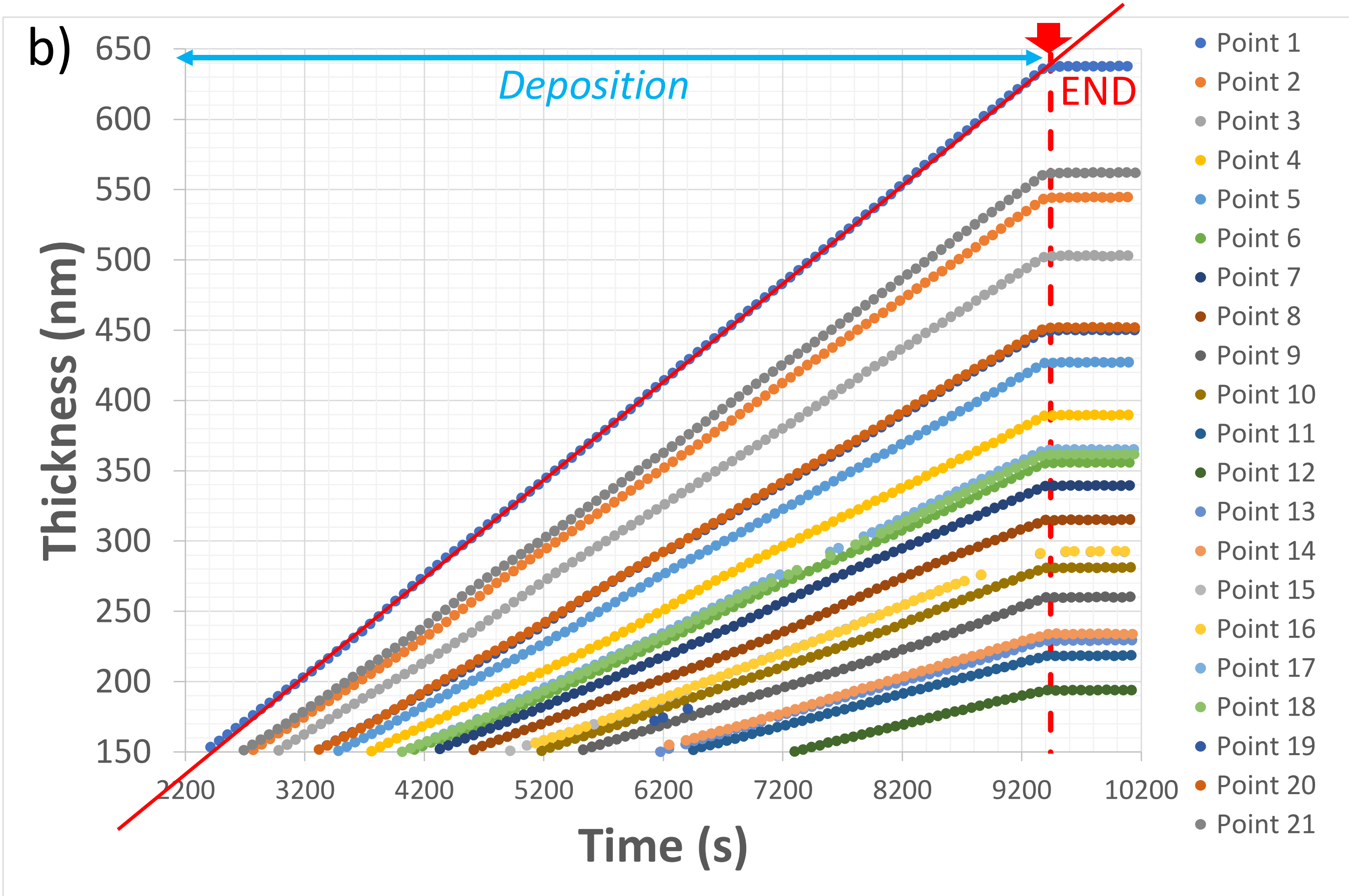
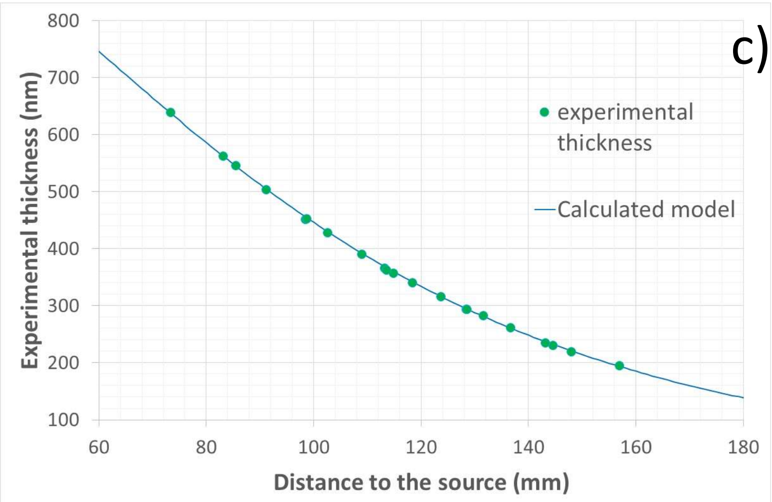
COMBINATORIAL IN-SITU SPECTRAL REFLECTOMETRY

3D-OXIDES MULTI-FUNCTIONAL THIN FILMS







Mass Transport Limited Regime conditions

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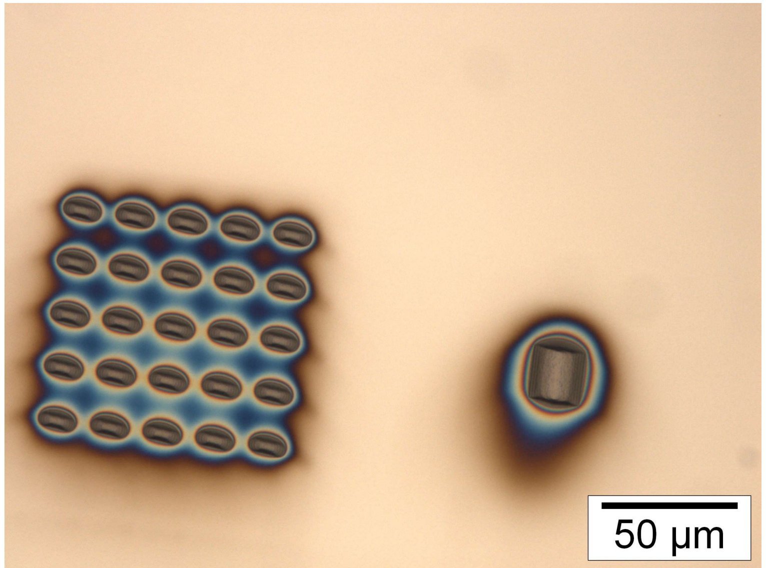
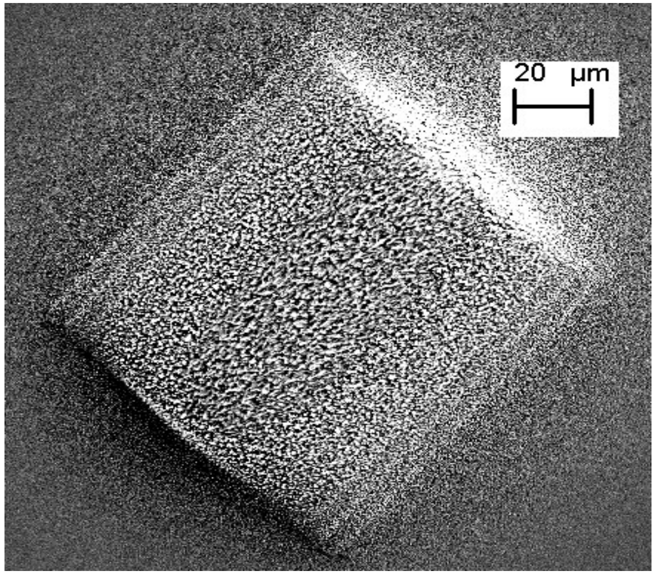
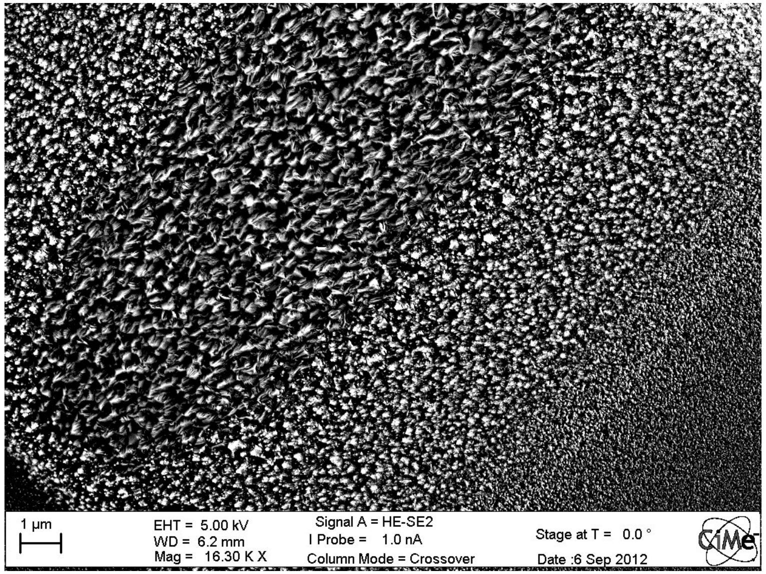
Material Properties vs Thickness

3D-Pyramid like structure







ROUGHNESS INCREASING WITH THICKNESS FOR COLUMNAR GROWN TiO_2 ANATASE

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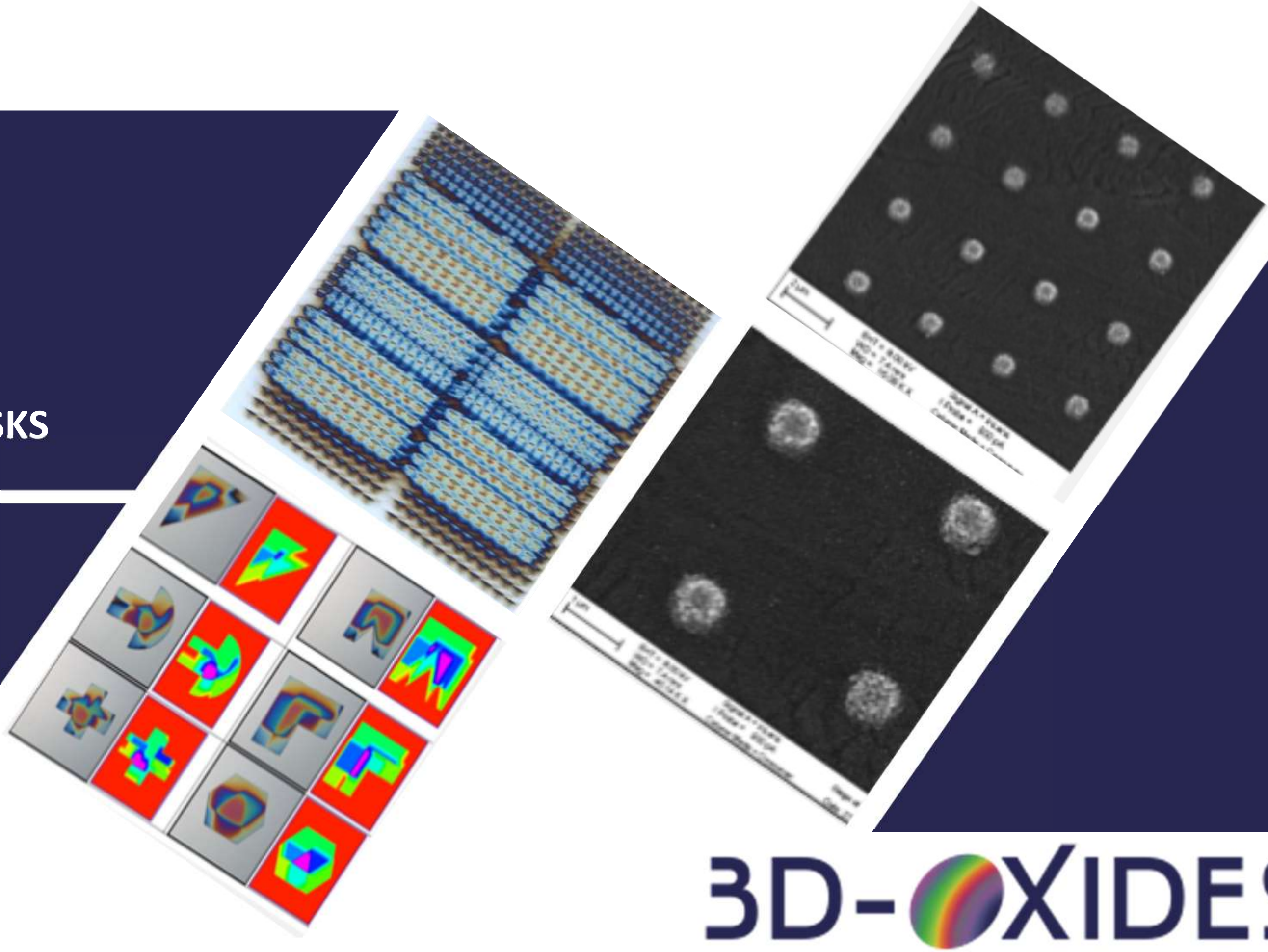
Additive Growth

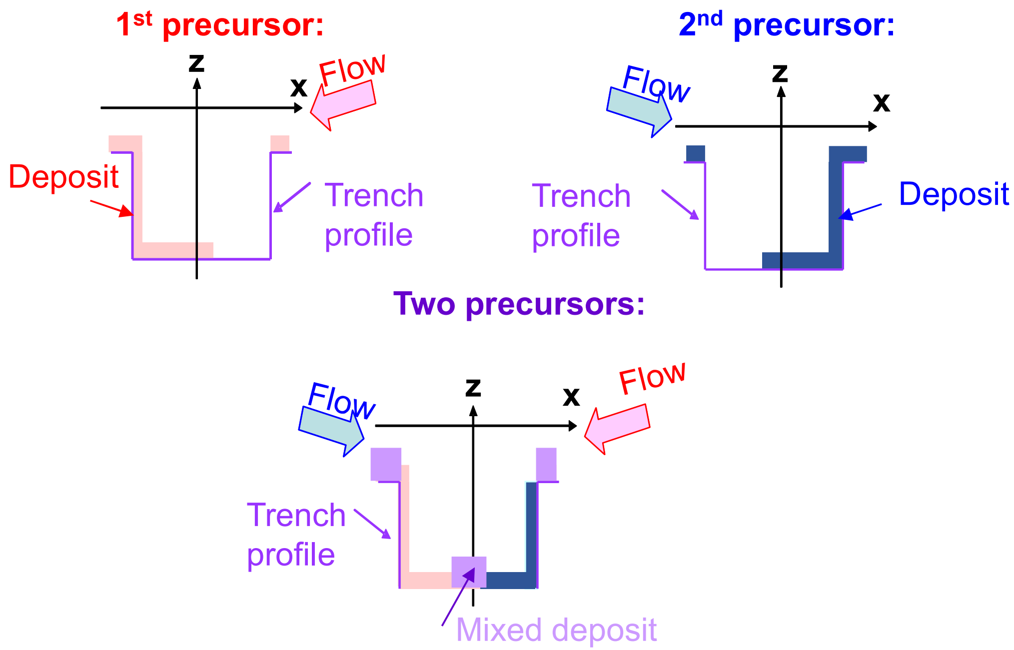
Patterned substates / Stencil Masks

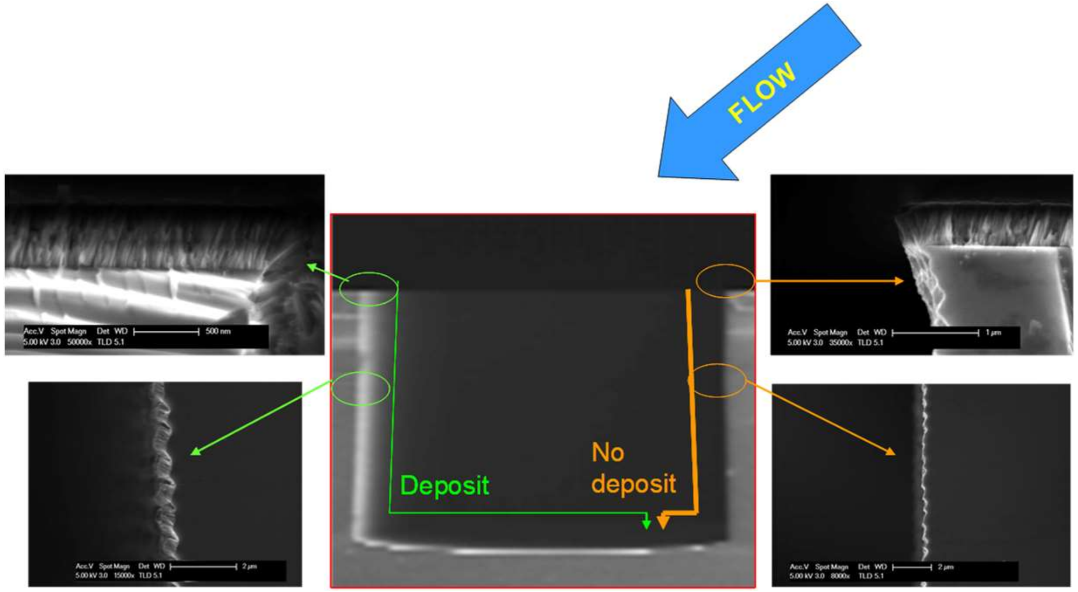


3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

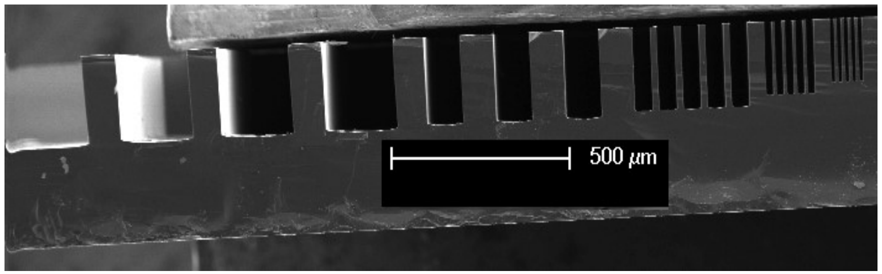
PATTERNED SUBSTRATES

3D-OXIDES MULTI-FUNCTIONAL THIN FILMS





Deposition on patterned substrates



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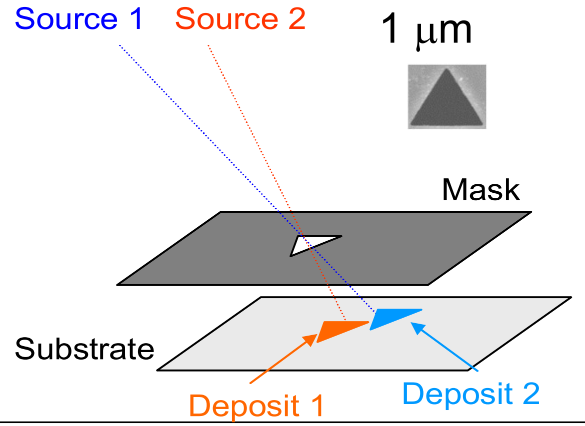
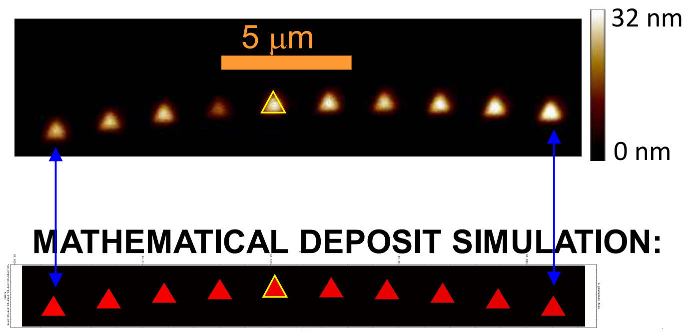
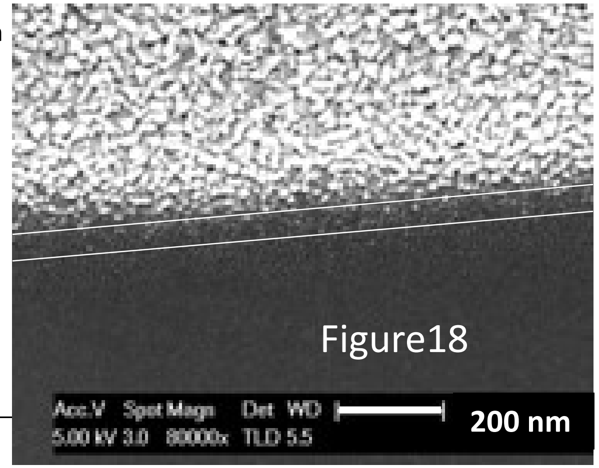
MORPHOLOGICAL PATTERNING

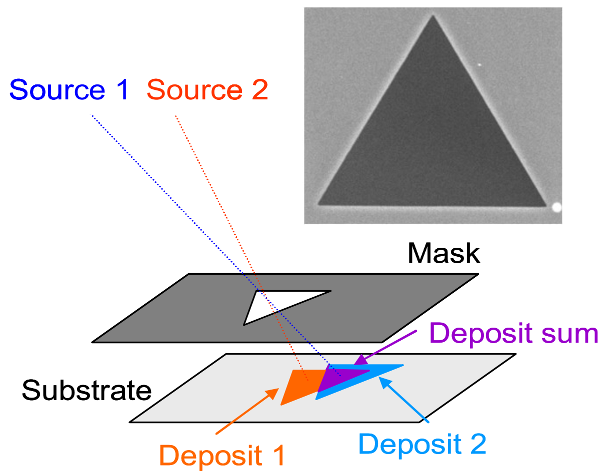
3D-OXIDES MULTI-FUNCTIONAL THIN FILMS

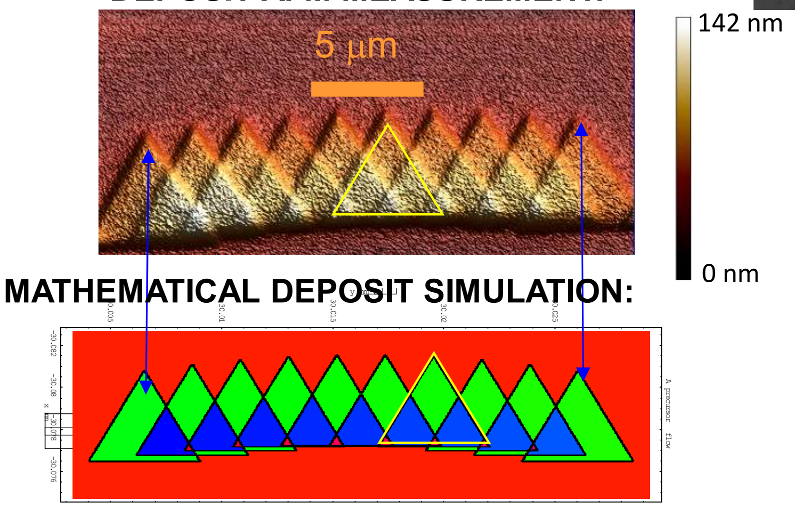
Wagner et al. (2015) Thin Solid Films 586 64

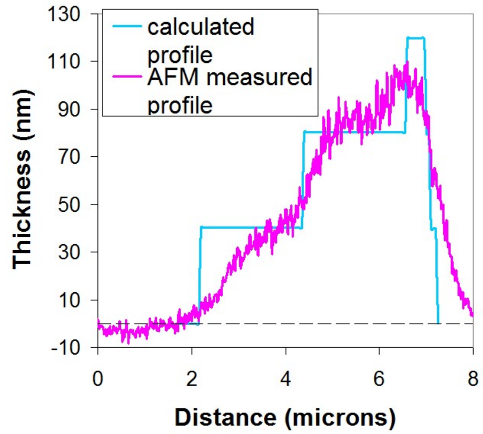












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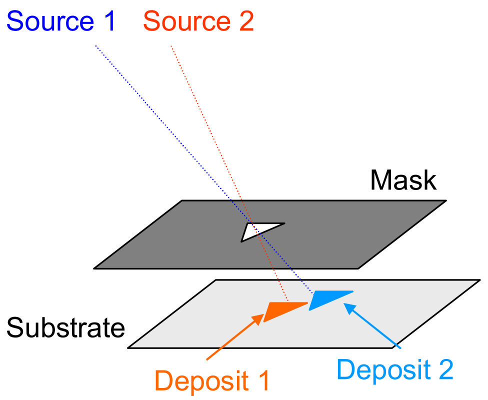
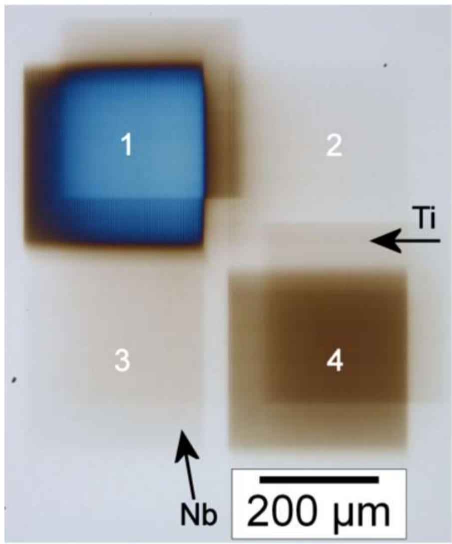
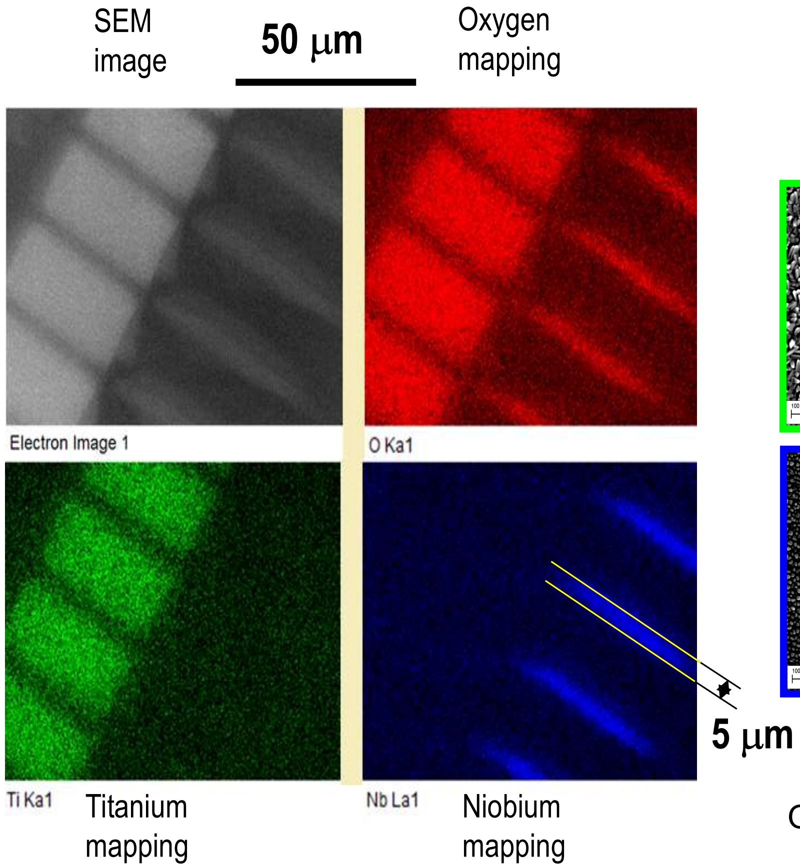
CHEMICAL PATTERNING (SEPARATED ELEMENTS)

3D-OXIDES MULTI-FUNCTIONAL THIN FILMS

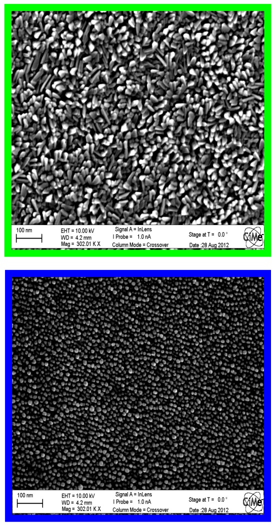




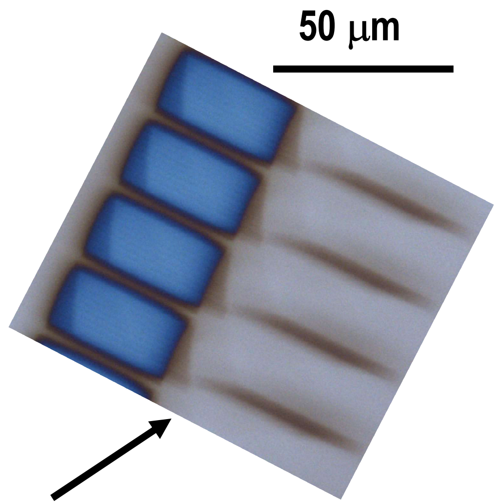
EDX mapping



SEM top-view



Optical microscopy



Optical microscope picture of the layer 80 nm for TiO_2, 40 nm for Nb_2O_5

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CHEMICAL PATTERNING (SUPERPOSED ELEMENTS)

3D-OXIDES
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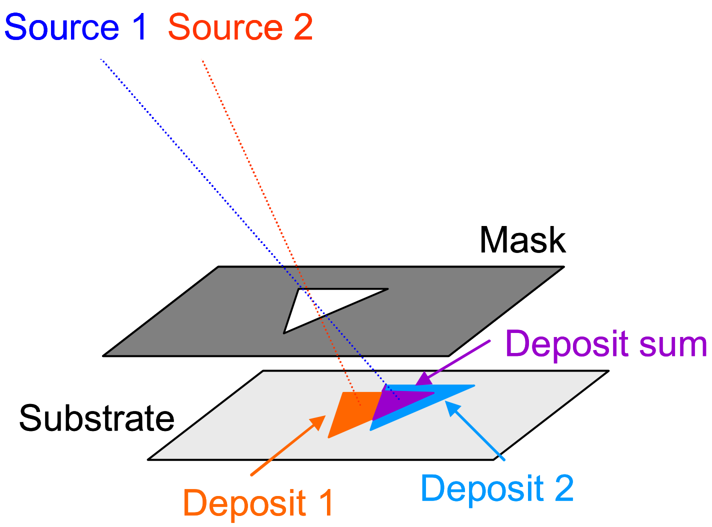

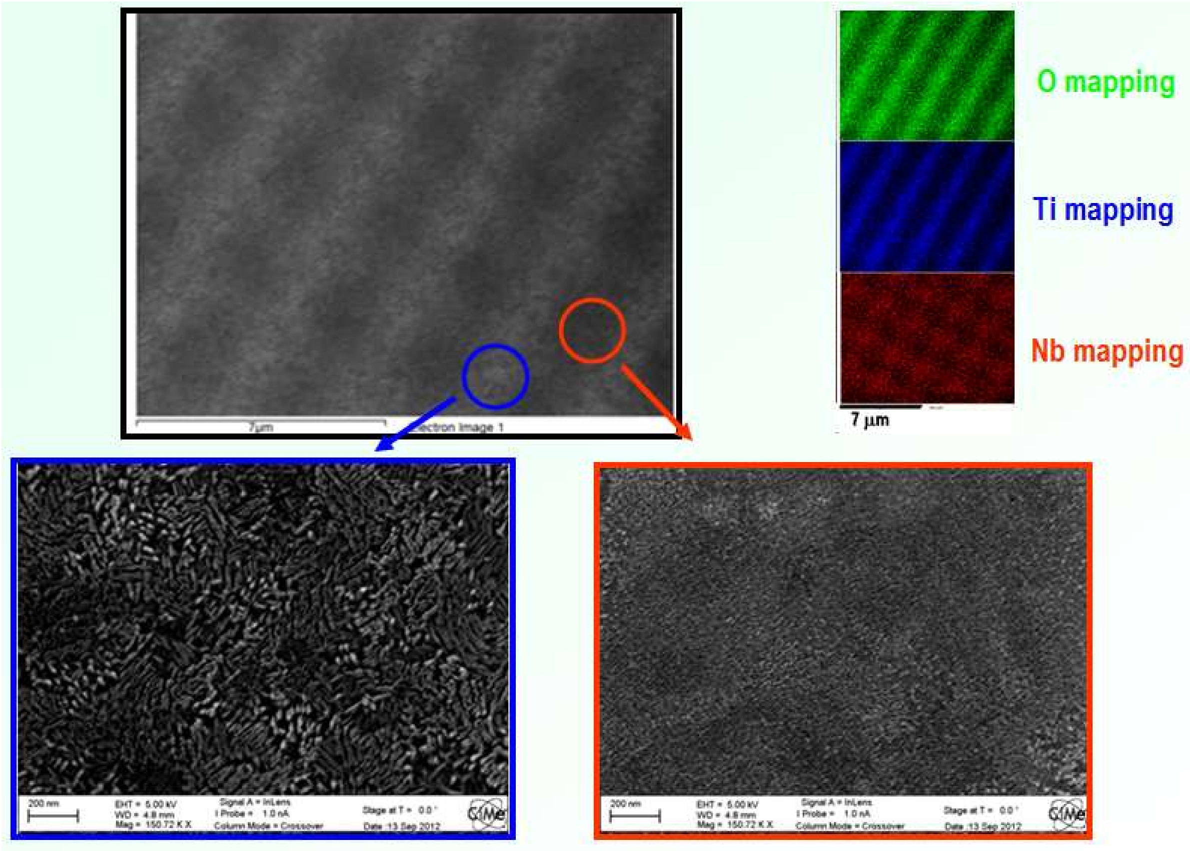

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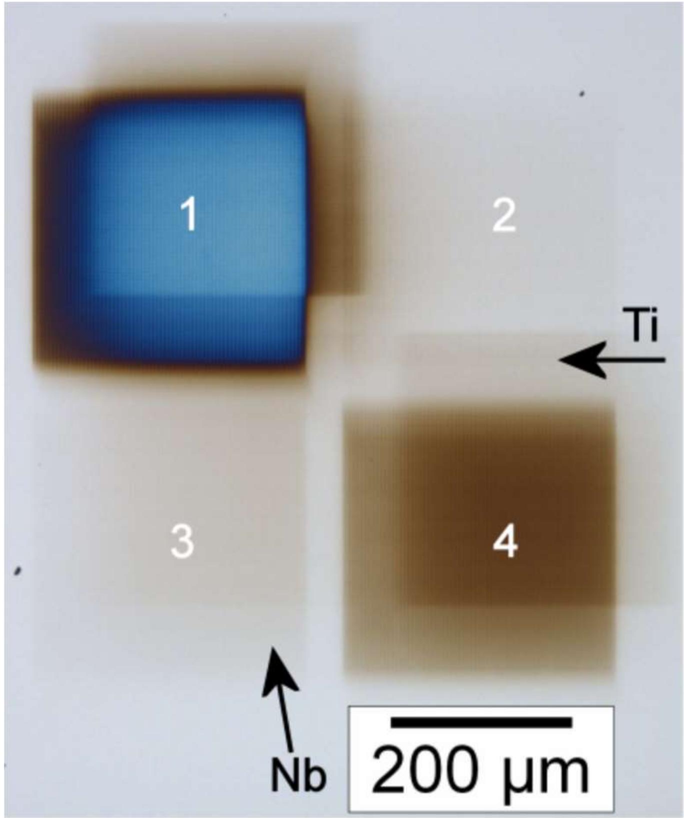
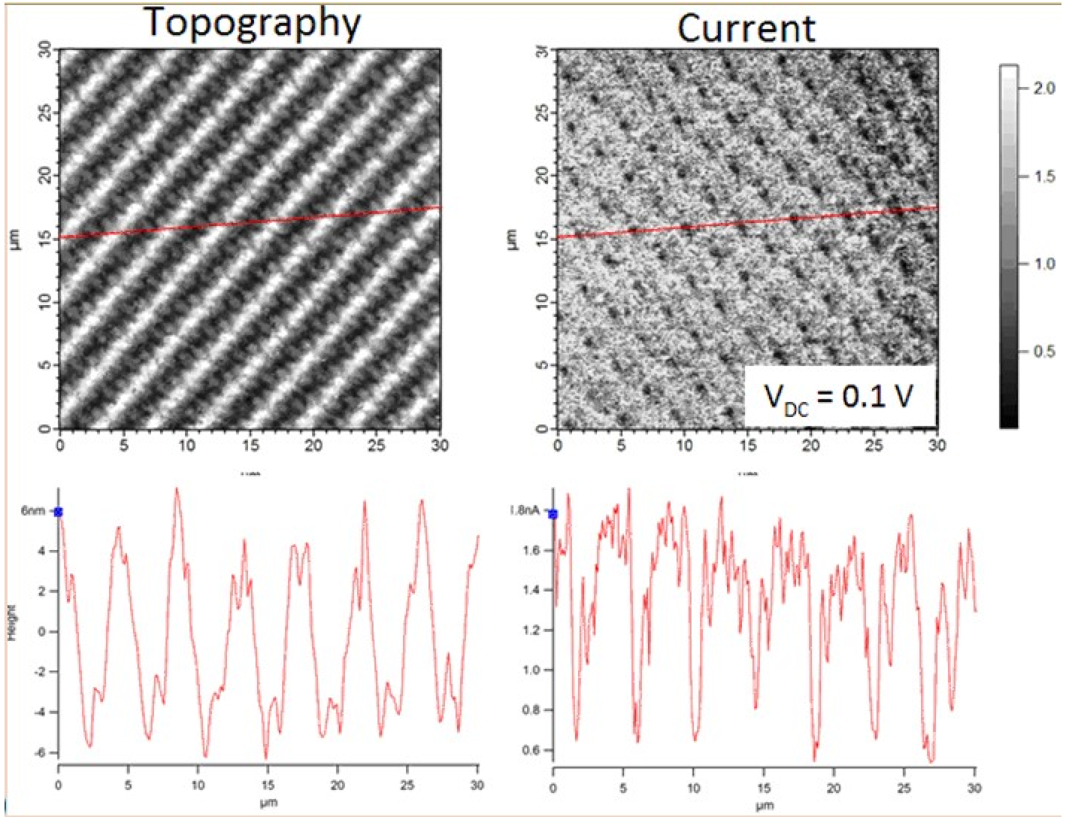
FUNCTIONAL PROPERTIES PATTERNING

3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

Variable electrical conductivity.
Most functional properties can be modulated and patterned.





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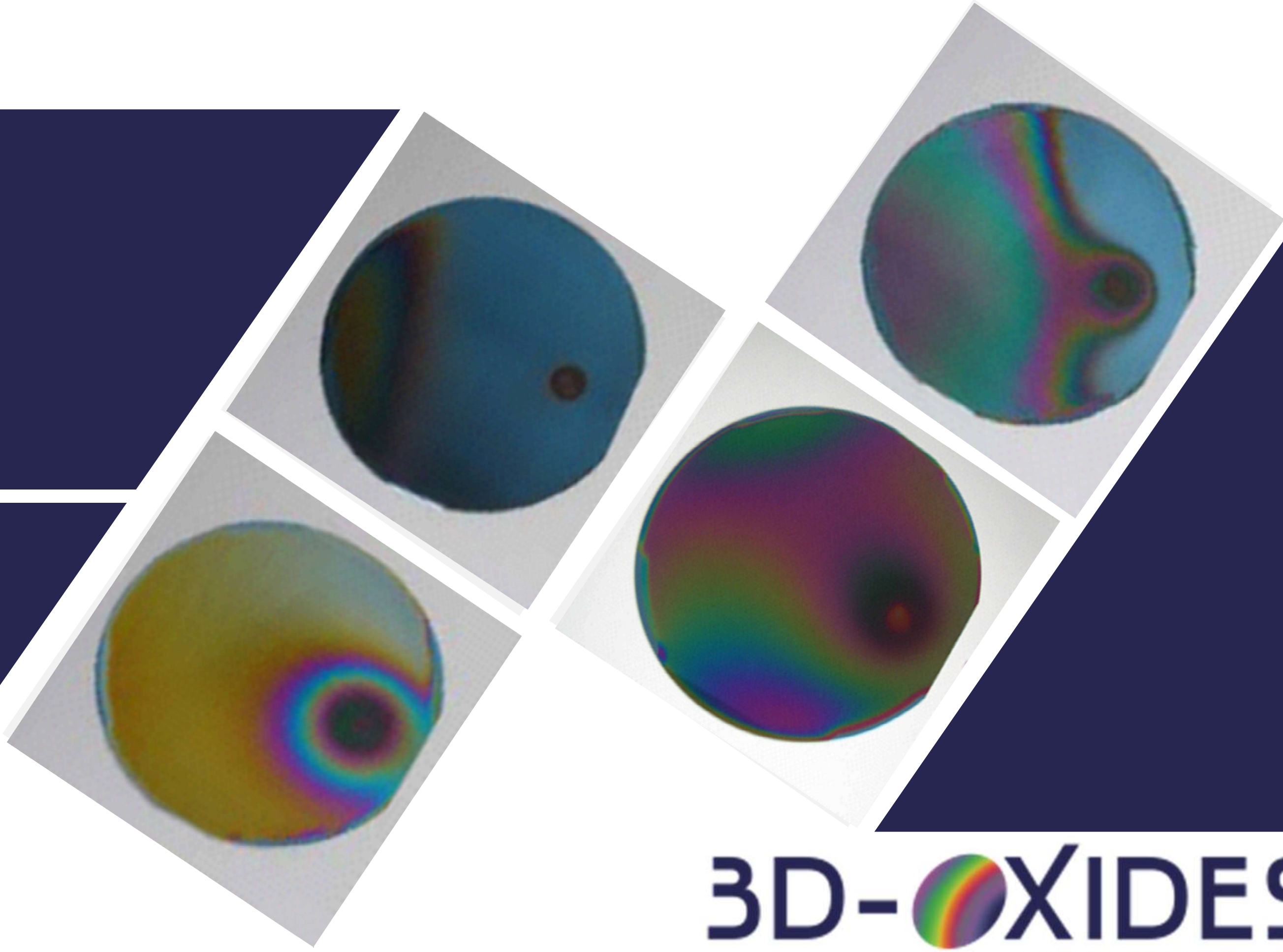
Additive Growth

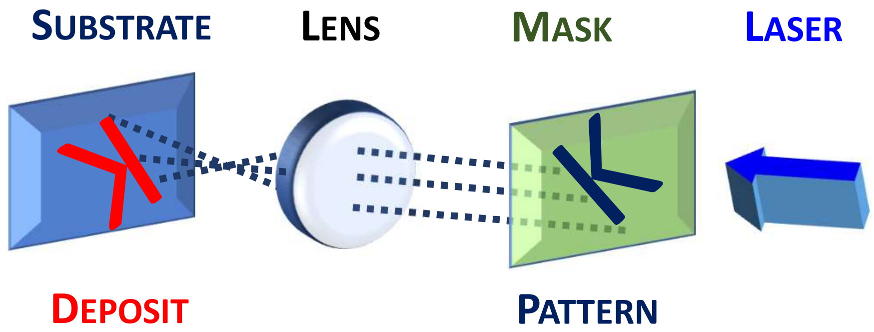
CBE-Laser-Assisted



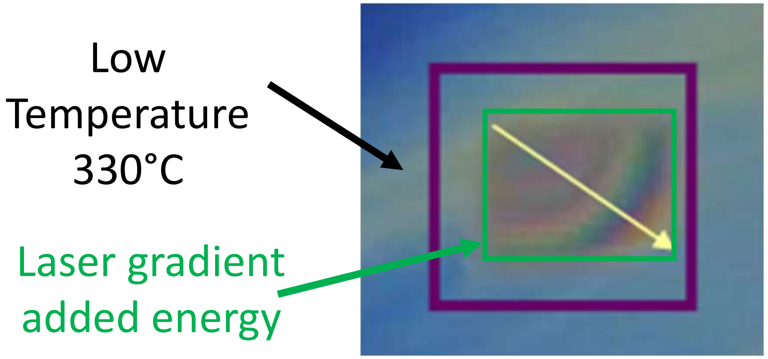
3D-OXIDES

MULTI-FUNCTIONAL THIN FILMS

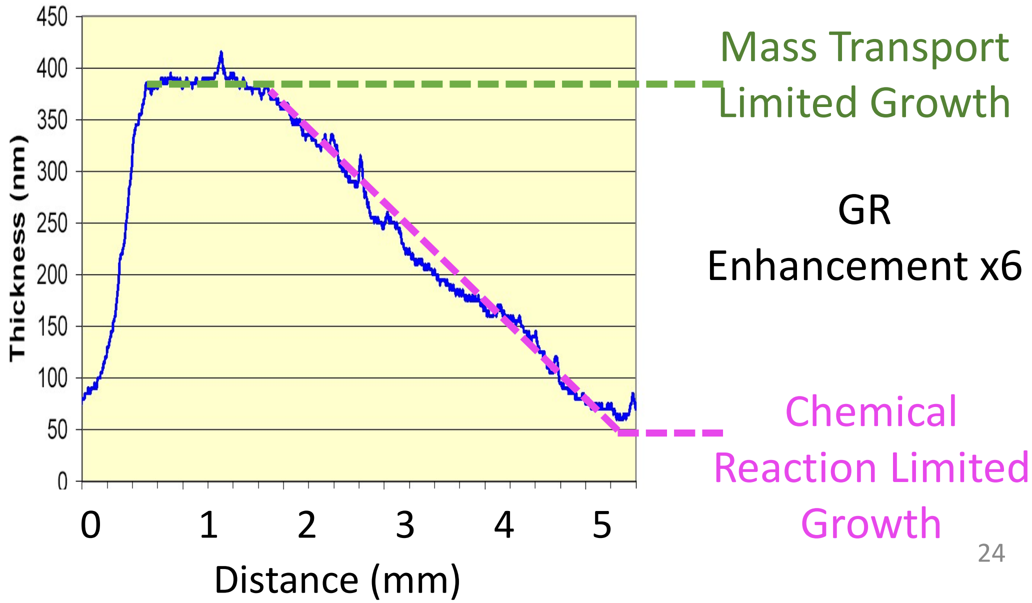
LASER PATTERNING (GROWTH RATES)



DEPOSITION / ABLATION / ETCHING
IS LIMITED TO THE IRRADIATED AREA



ENERGY Source	Thin Film Colour	TiO_2 layer (thickness nm)
Thermal deposition	Blue	70.0
Fluence 1	Yellow	112.7
Fluence 2	Blue	190.3
Fluence 3	Red	360.6
Fluence 4	Blue	402.1



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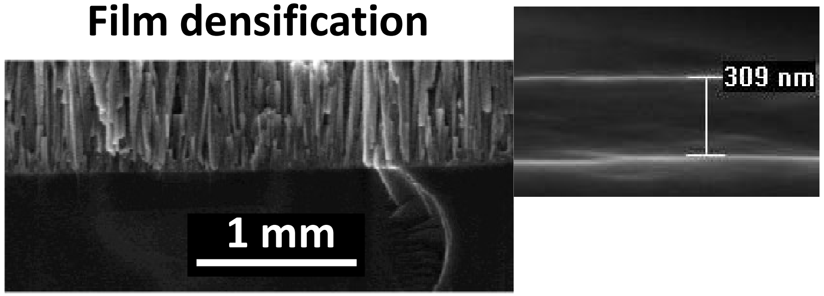
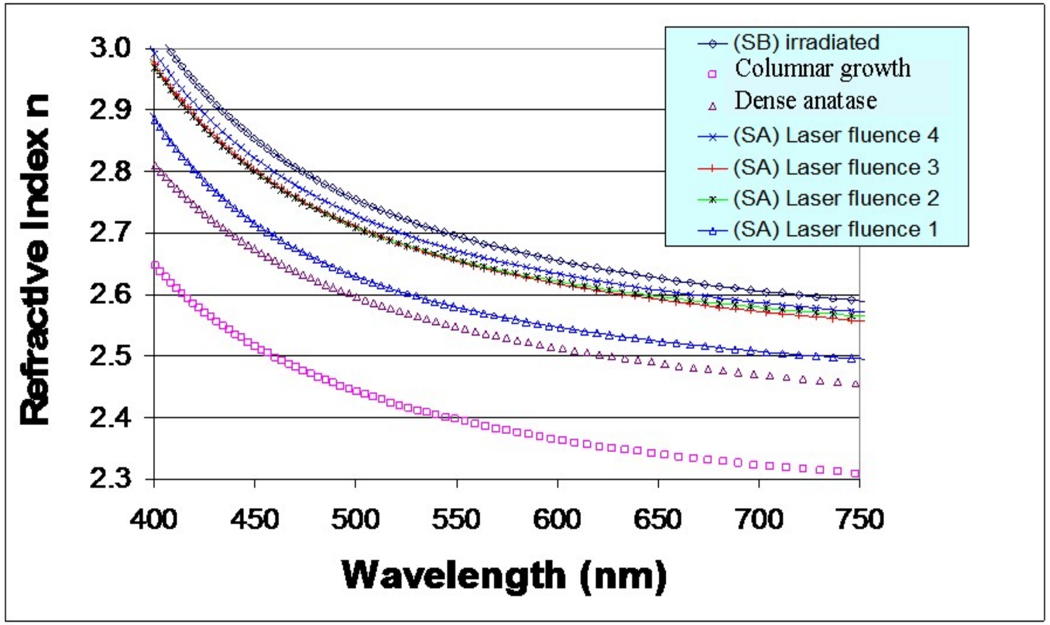
Selective modification of TiO_2 thin films properties

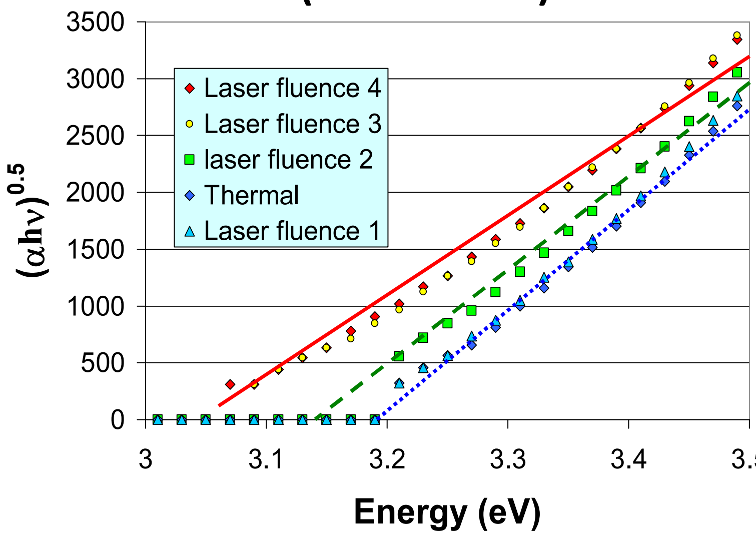
Film densification



Refractive index fine tuning



BandGap shifting (Tauc Model)



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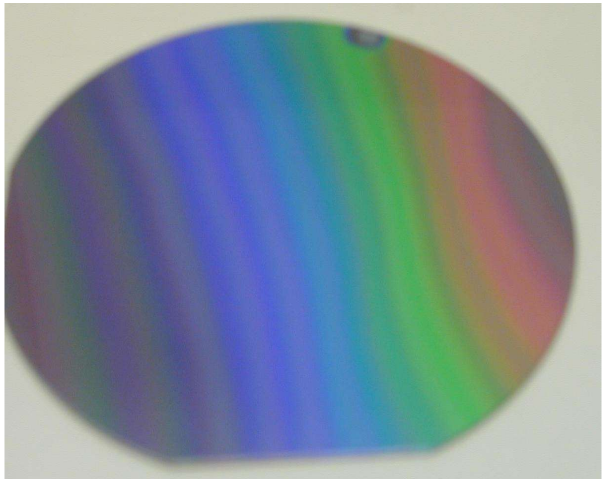
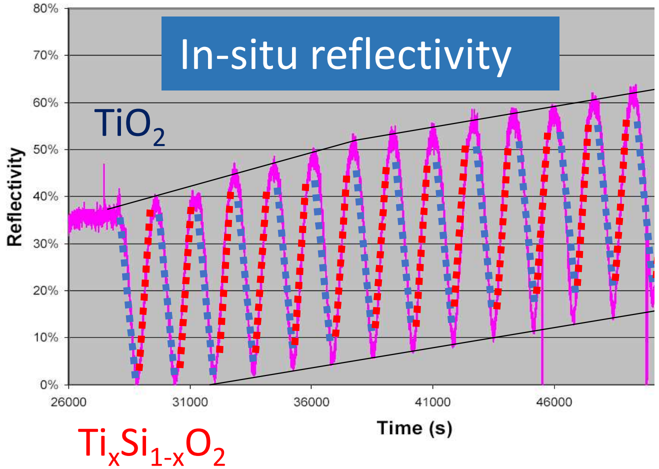
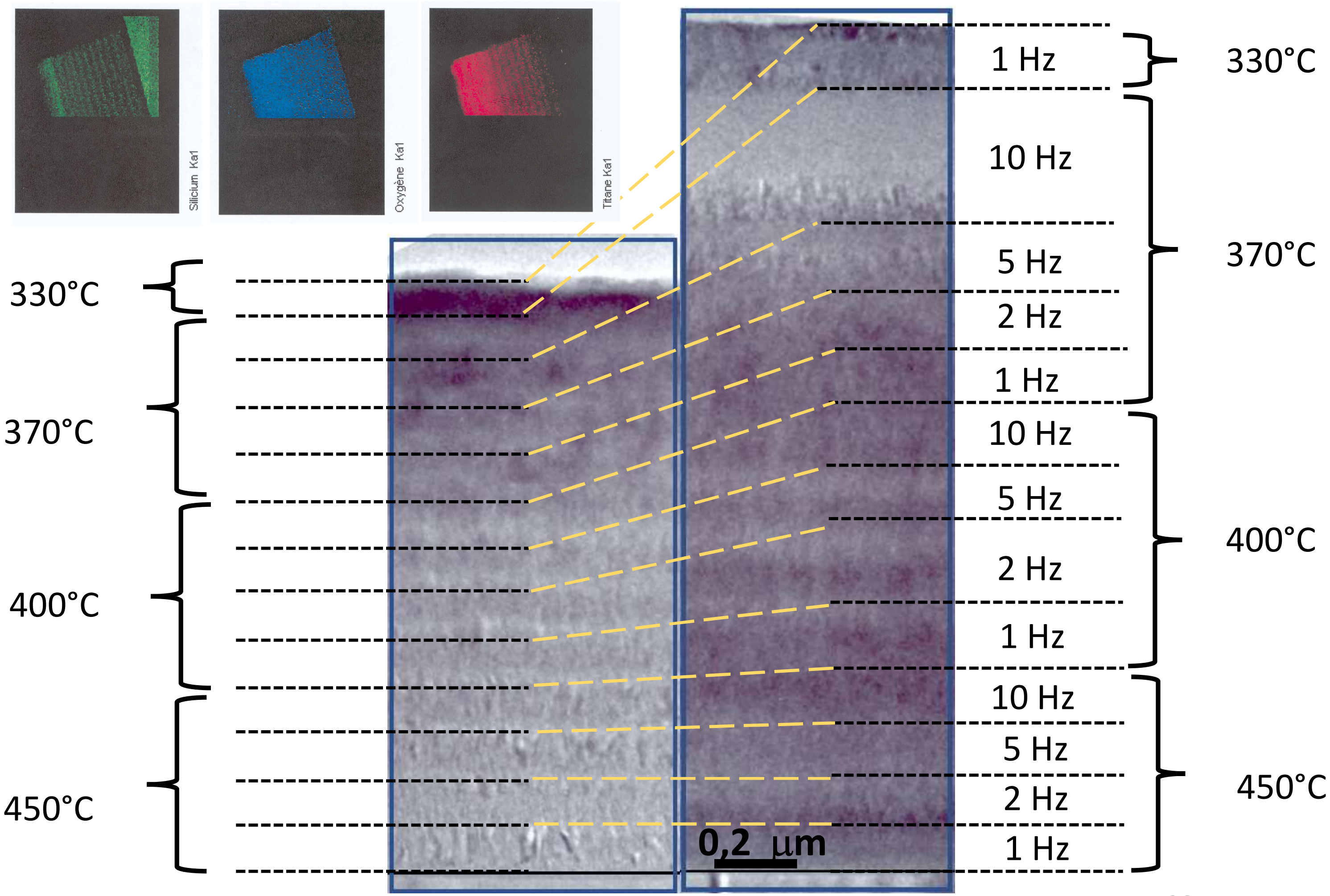
LASER 3D-PATTERNING (CHEMICAL COMPOSITION)

3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS







THERMAL GROWTH   THERMAL GROWTH + LASER

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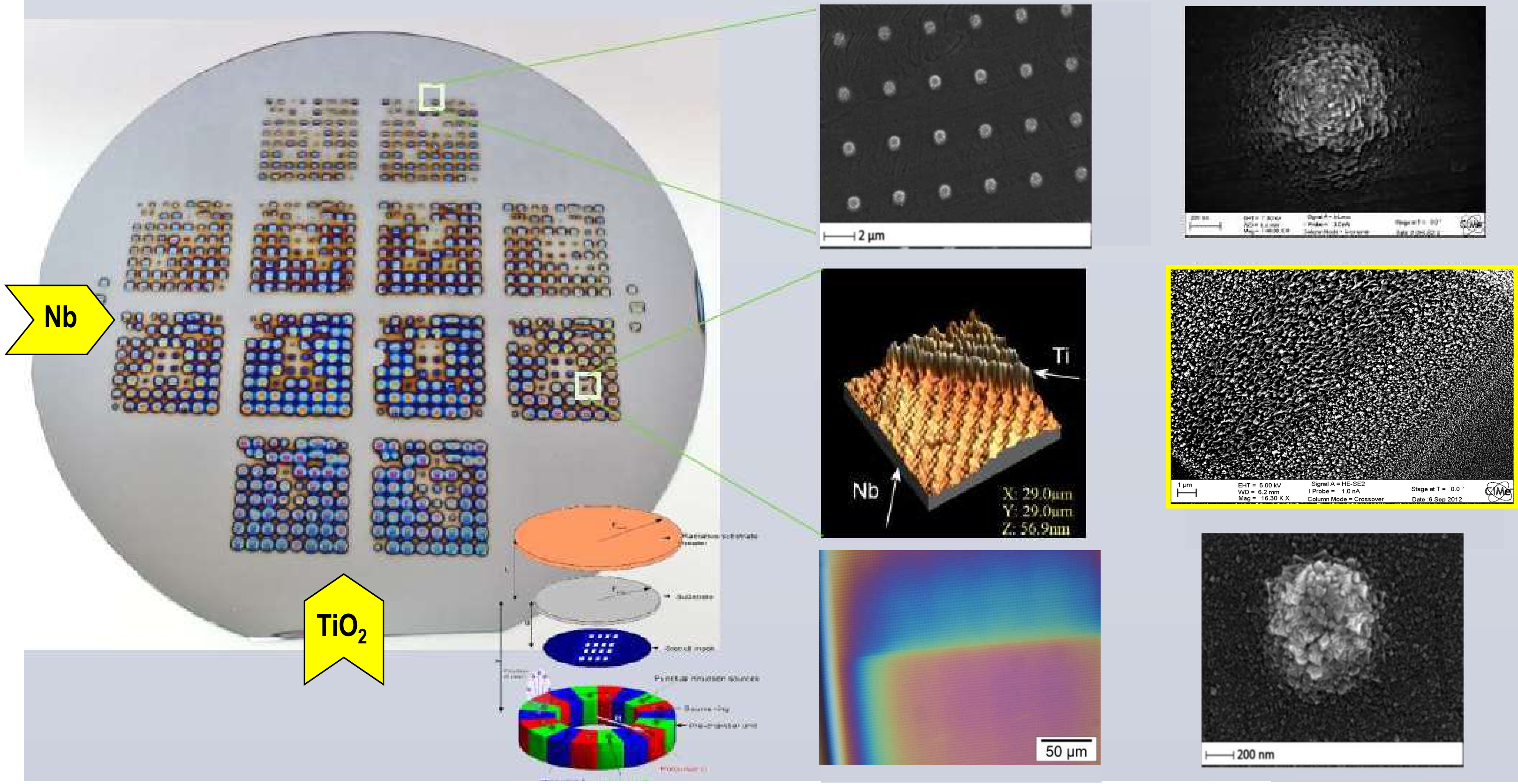
Combinatorial structures obtained via a stencil mask

Top-down patterning | Bottom-up patterning



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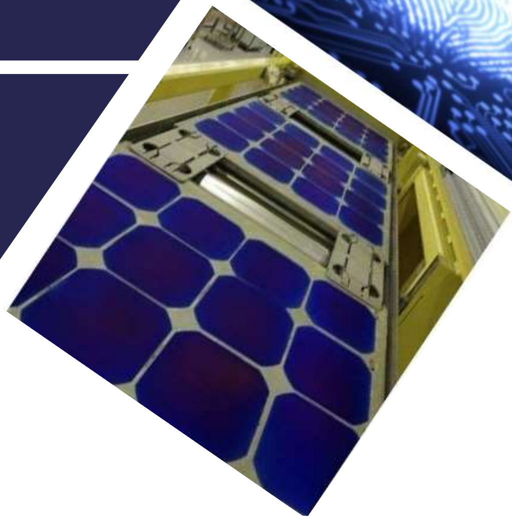
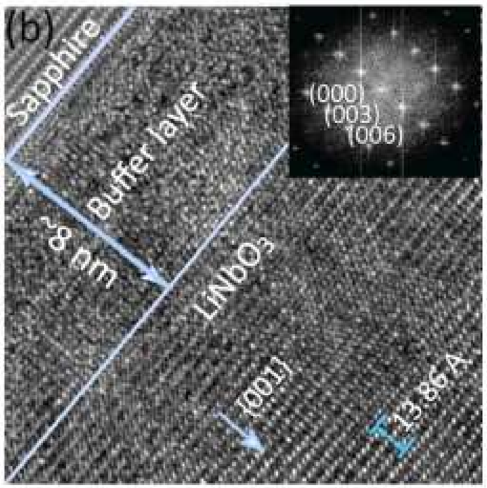
FUNCTIONAL MATERIALS



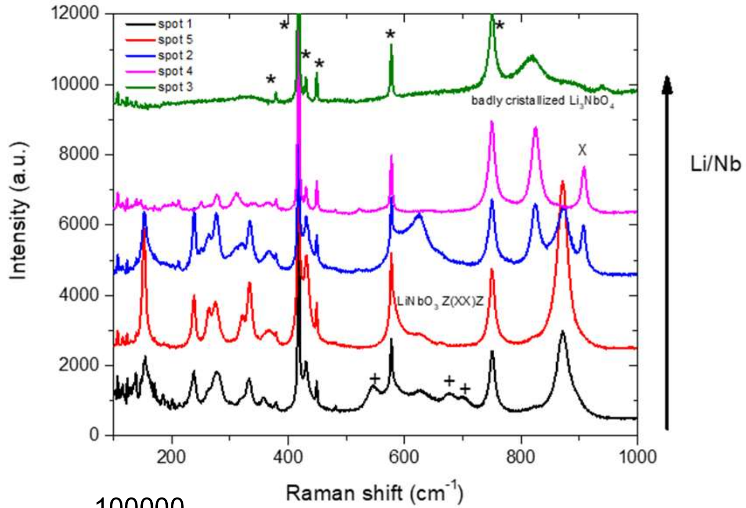
3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

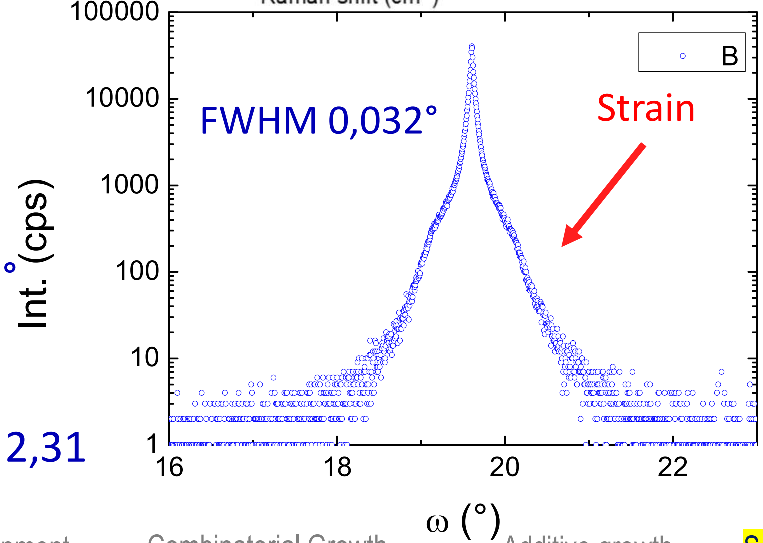
$LiNbO_3$

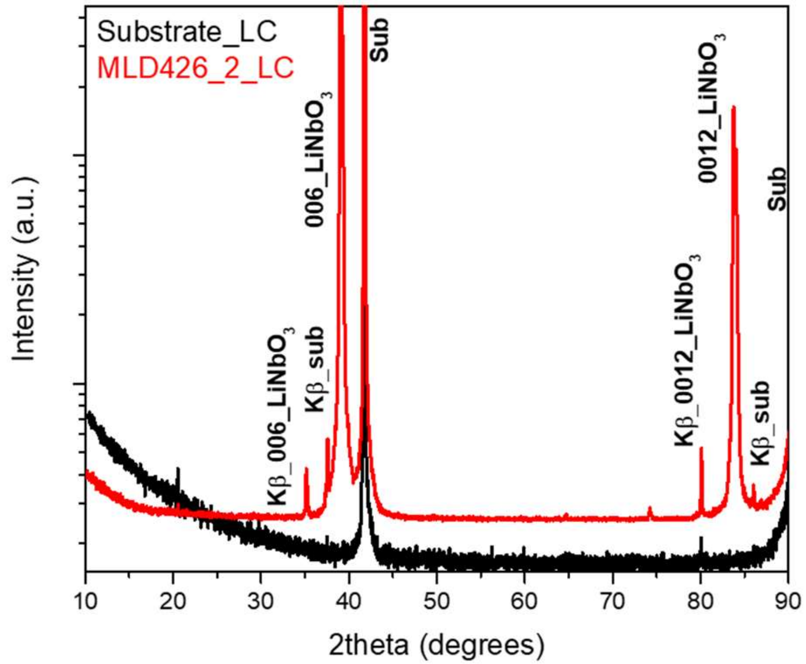
3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

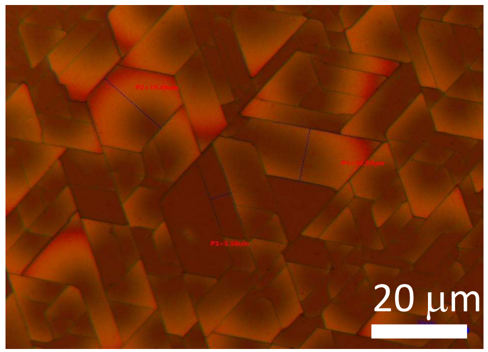


Combinatorial Chemical Beam Epitaxy of Lithium Niobate Thin Films on Sapphire
A. Dabirian, et Al.; Journal of The Electrochemical Society, 158 (2) D72-D76 (2011).
DOI:10.1149/1.3519843









Single orientation 006/0012
Rocking curves: FWHM 0,03°
Roughness RMS 1,39 nm
Raman: ok
Refractive index: 2,26 < n < 2,31

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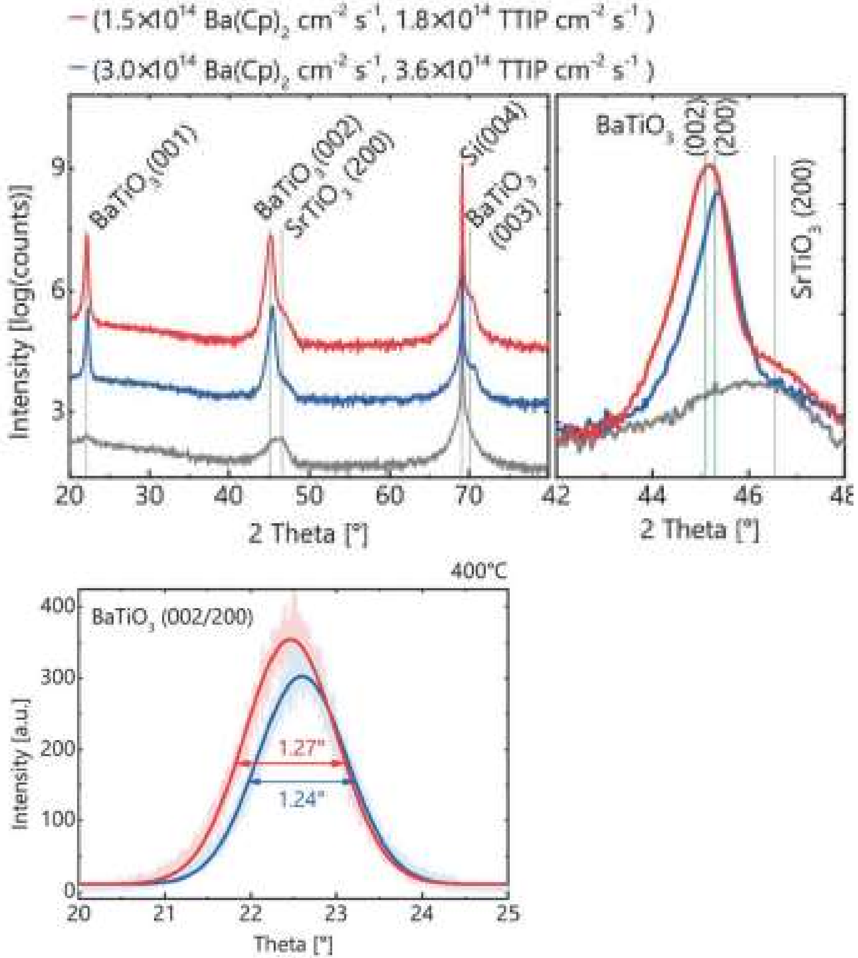
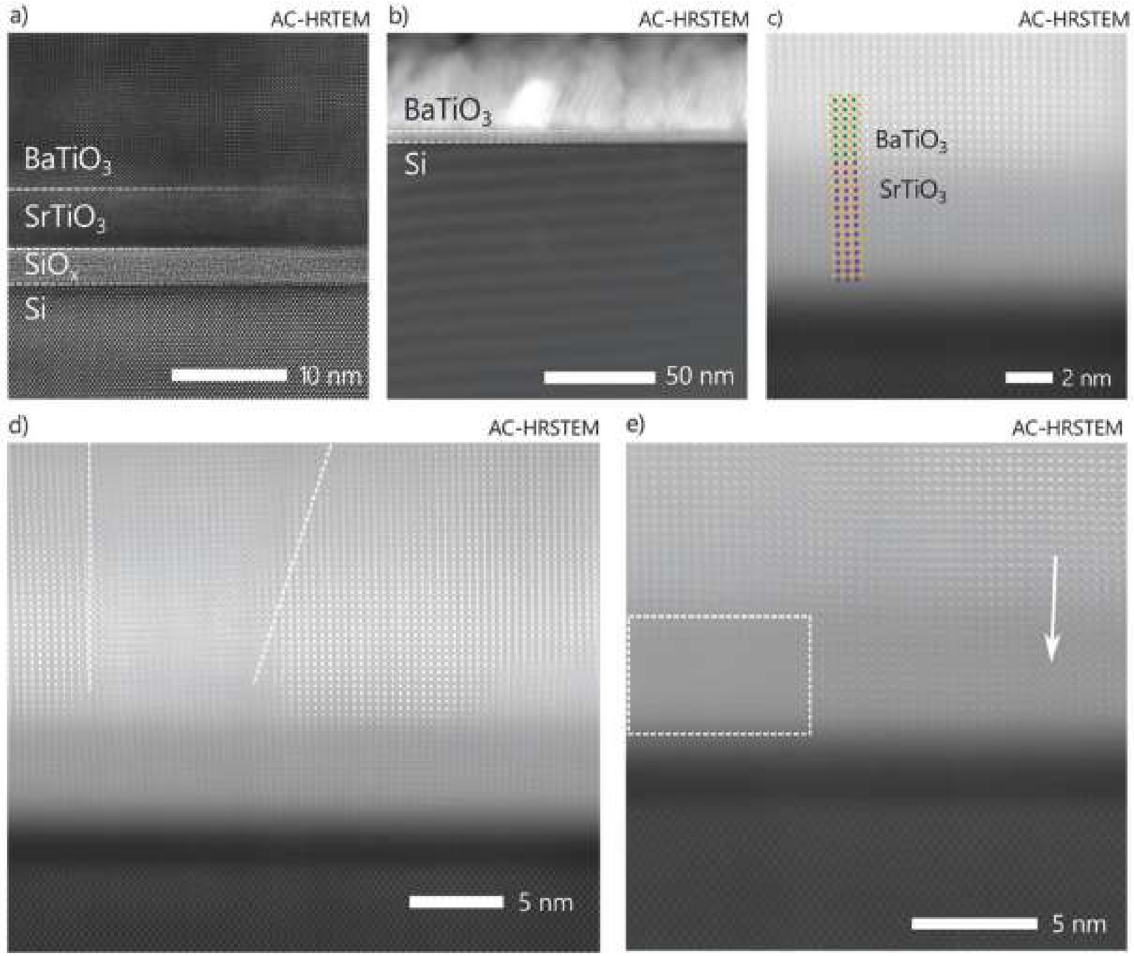
EPITAXIAL $BaTiO_3$ ON SI (EPFL AND IBM)

3D-OXIDES MULTI-FUNCTIONAL THIN FILMS





Reference: Low Temperature Epitaxial Barium Titanate Thin Film Growth in High Vacuum CVD; M. Reinke et Als; Adv. Mater. Interfaces 2017, 1700116 DOI: 10.1002/admi.201700116

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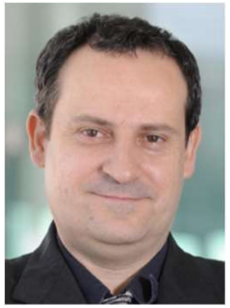
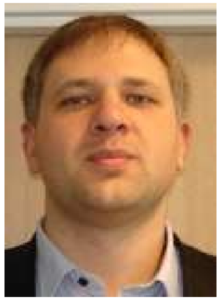
Conclusion & Outlook





3D-OXIDES
MULTI-FUNCTIONAL THIN FILMS

A NEW TOOL TO ADDRESS CURRENT NEEDS

Top level properties on many different materials:

$LiNbO_3$, $BaTiO_3$, $SrTiO_3$, TiO_2, $Hf_{(1-x-y)}Ti_xZr_yO_2$, Nb_2O_5, Ta_2O_5, Al_2O_3, ZnO, Vo_x, etc…

Upscalable to production

1. Very high control accuracy on process
2. Lower thermal budget (CMOS compatible)
3. Large substrates (mass production)
4. More elaborated architectures for better devices

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A Disruptive Tool for new oxide thin films devices

3D-OXIDES
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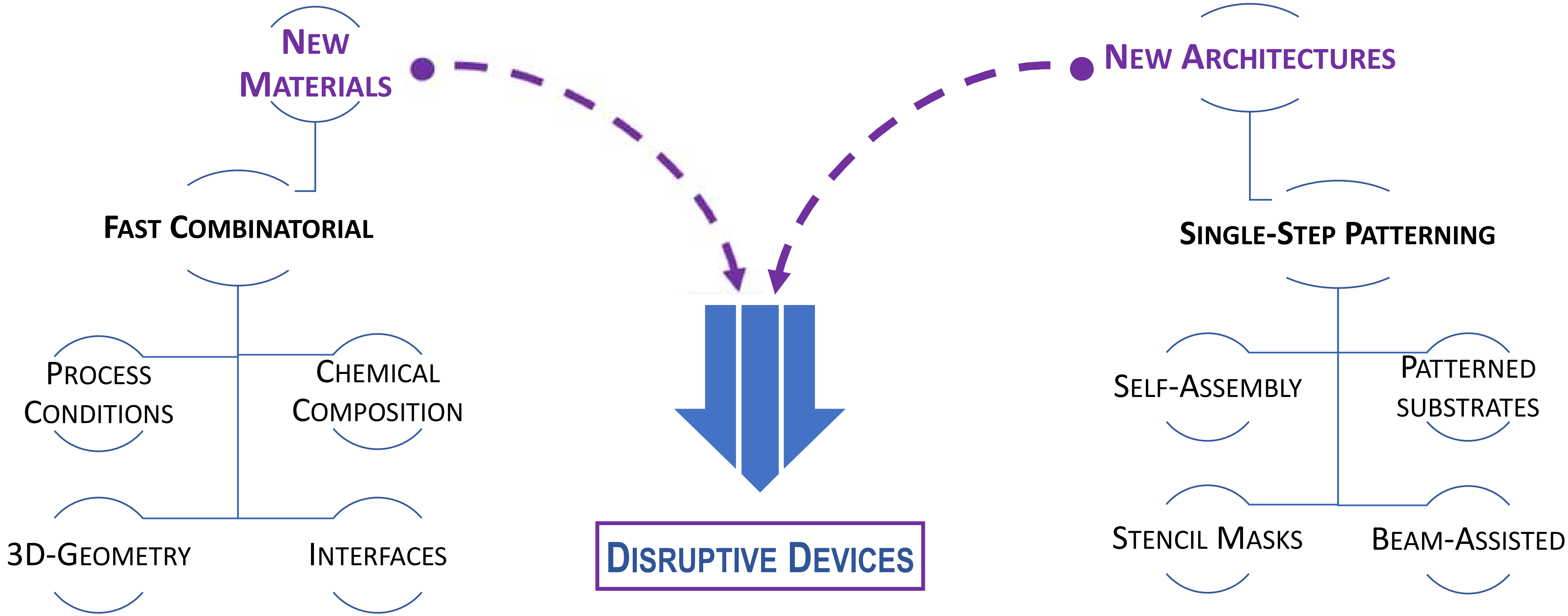

Fast technological approach addressing simultaneously several Markets

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MULTI-FUNCTIONAL THIN FILMS

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UNION EUROPÉENNE

THANK YOU FOR YOUR ATTENTION

L'EUROPE S'ENGAGE en région Auvergne-Rhône-Alpes

Phone : +33-(0)7 777 54 397
giacomo.benvenuti@3d-oxides.com