CONFORMAL OPTICAL COATINGS BY ATOMIC LAYER DEPOSITION

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OSA Thin Films Technical Group

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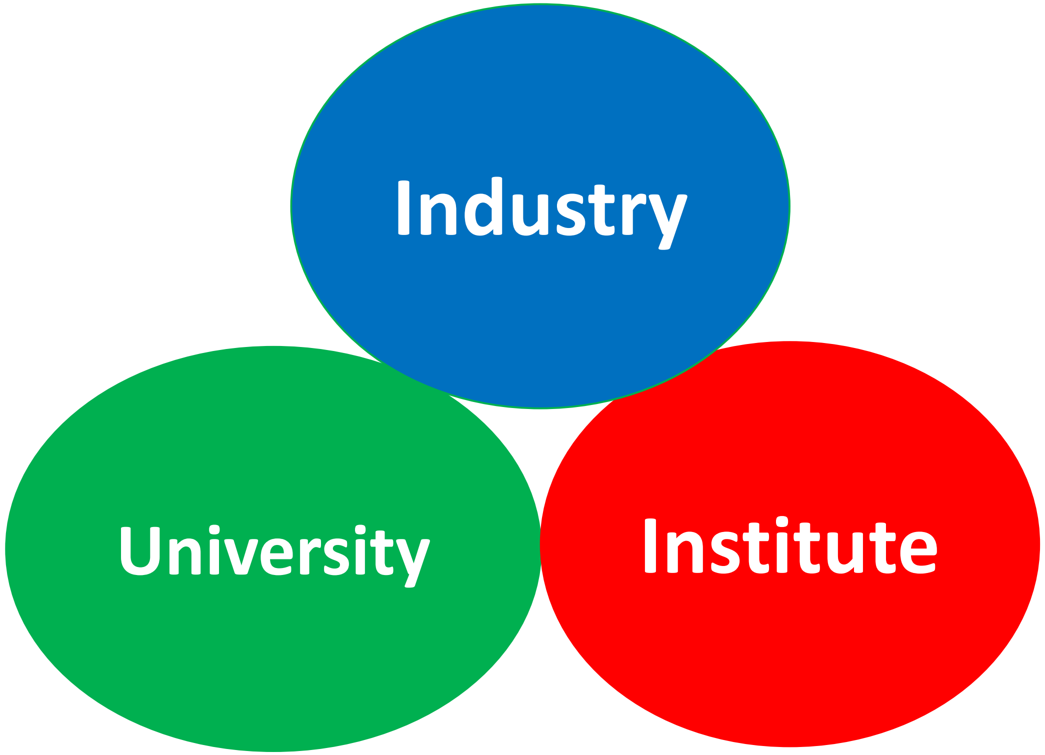
About the Thin Film Technical Group

Our mission:



Our recent webinars:

- Environmental stability of PVD coatings
- Surface coatings inhibit infection
- Metasurfaces: new generation building blocks for optics
- Nanoscale multilayers for EUV and X-ray applications

Connect with Thin Film Technical Group

- OSA Website: www.osa.org/ThinFilmsTG
- LinkedIn: www.linkedin.com/groups/4783616
- In Person at OIC 2022 (OSA - Optical Interference Coatings Conference)

https://www.osa.org/en-us/meetings/topical_meetings/optical_interference_coatings/

Attendees of OIC 2019, New Mexico, USA

Scheduled 19-24 June 2022, Whistler, Canada

OSA Thin Films Technical Group

Invited Speakers

Mr. Sami Sneck

- Business Executive at Beneq since 2005
- MSc degree in Chemical Engineering in 2001 from Helsinki Univ. of Technol.
- Introduced ALD to jewelry, photovoltaics, optical coatings, and semiconductors

Dr. John Rönn

- ALD for optics and photonics at Beneq
- Ph.D. from Aalto University School of Electrical Engineering in 2019
- Published ALD articles in Nature Communications and ACS Photonics

Conformal Optical Coatings by Atomic Layer Deposition

M.Sc. Sami Sneck & D.Sc. John Rönn

July 2nd, 2021

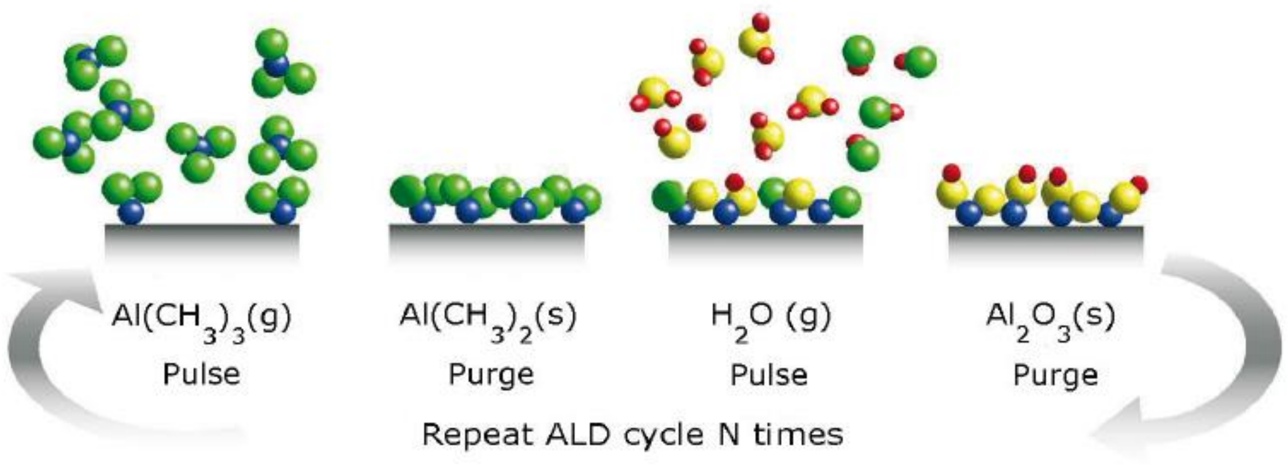
BENEQ

Contents

Principles of Atomic Layer Deposition (ALD)

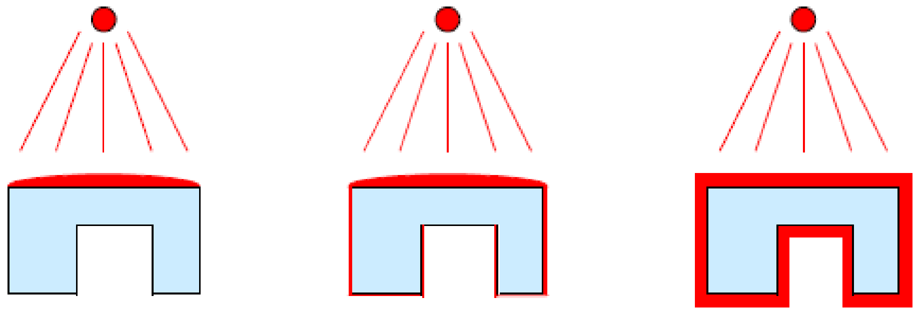
Atomic Layer Deposition

- A thin film deposition technique based on **saturated surface reactions** of the precursor and the surface species
- Precursors released on the surface alternatively
- Process typically in vacuum (~1 mbar) at 70-400°C



Atomic Layer Deposition

- Surface controlled layer-by-layer growth leads to
  - ✓ Excellent uniformity
  - ✓ Exceptional conformality
  - ✓ Accurate control of film thickness
  - ✓ Atomic scale engineering of the film properties



Source controlled gas phase process (PVD)

Semi-surface controlled gas phase process (CVD)

Surface controlled gas phase process (ALD)

BATCH PRODUCTION OF OPTICAL COATINGS

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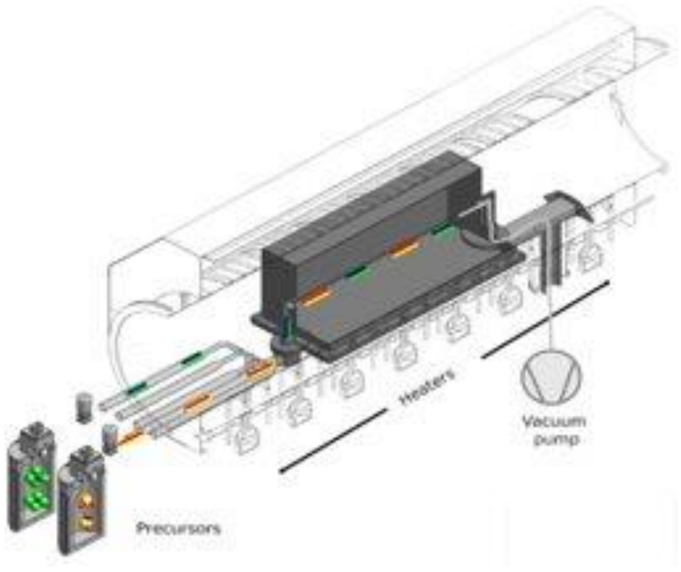
Batch production of optical coatings

- Although typical ALD processes suffer from low deposition rates, ALD itself is a highly scalable technology
  - ✓ Throughput can be increased by increasing the batch size
- Beneq's batch ALD-equipment offer extremely large batches to be coated in a single run

Beneq P400A



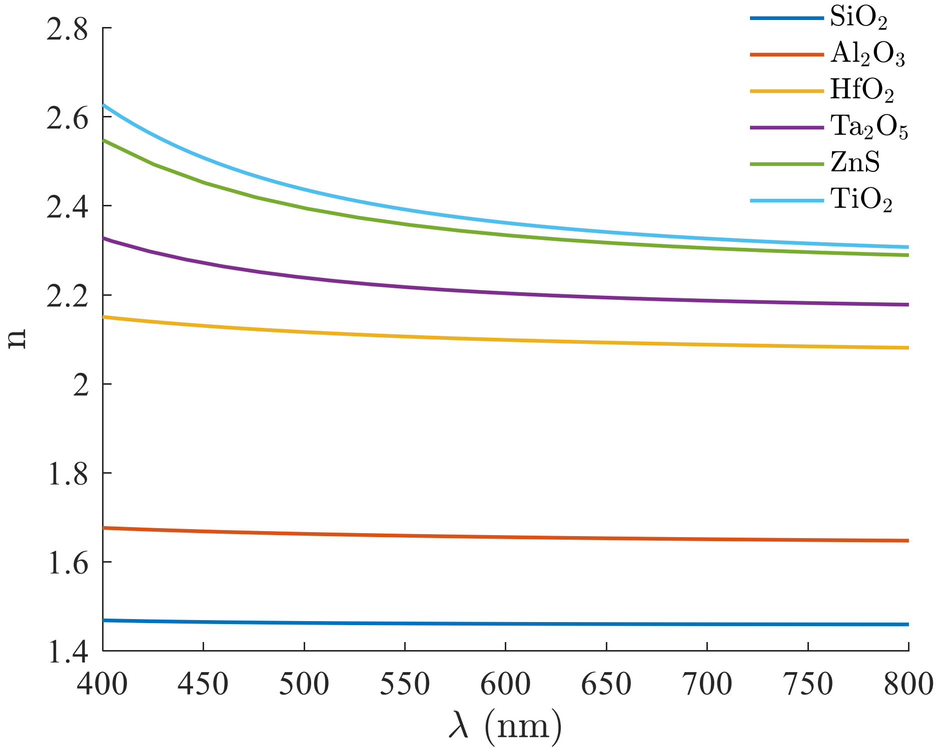
Typical dep rate 1-2 µm/day

8 m^2 of high accuracy films with P400A

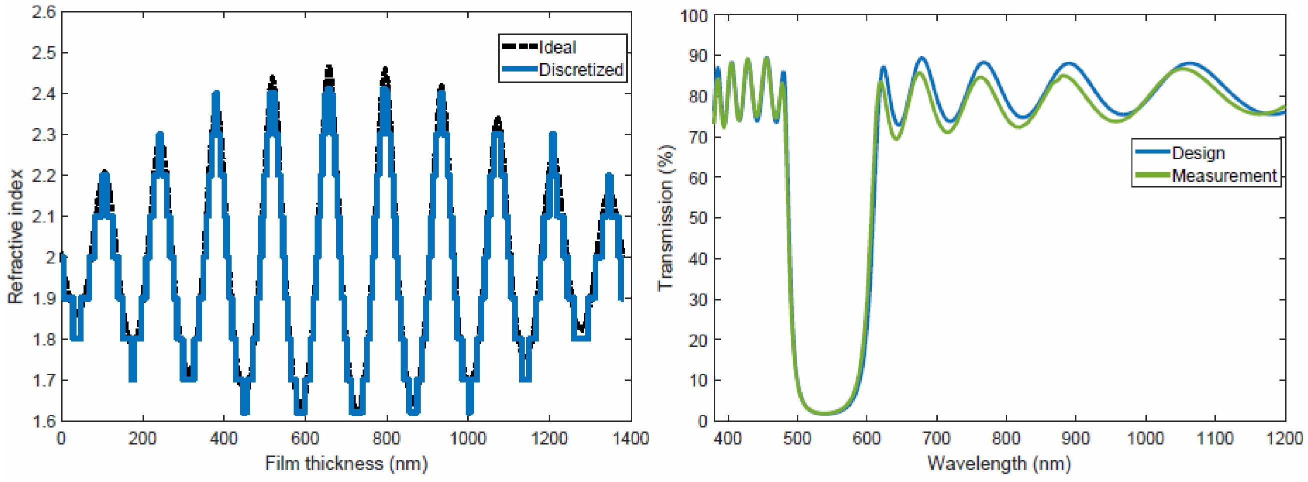
Batch production of optical coatings

Batch production of optical coatings

- Optical coatings realized using low-loss materials



- ✓ Edge-pass filters
- ✓ Band-pass filters
- ✓ Anti-reflective coatings
- ✓ Highly-reflective coatings



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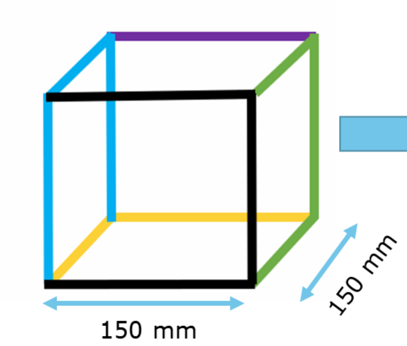
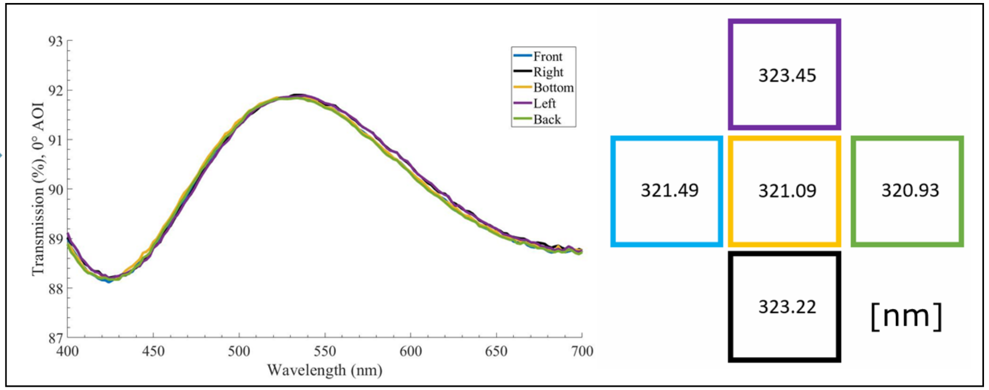
Batch production of optical coatings – 3D-structures

- ALD enables film deposition on the most complicated structures and surfaces
- Large 3D shapes, such as optical domes are examples of structures that are very difficult to coat conformally
- Example: Topless cube



Above: Schematic of a 150 mm –sided top-open cube. Glass substrate attached to each side.



Left: Measured transmission of glasses after ALD-Al_2O_3 deposition
Right: Fitted thickness values (nm) for corresponding sides. Variation < 0.4 %

K. Niiranen *et al.* "Atomic Layer Deposition of conformal Optical Interference Coatingss," in *Optical Interference Coatings Conference (OIC) 2019*, OSA Technical Digest (Optical Society of America, 2019), paper TE.5.

Batch production of optical coatings – 3D-structures

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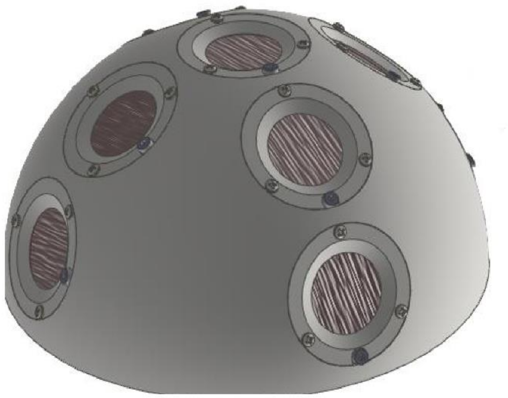
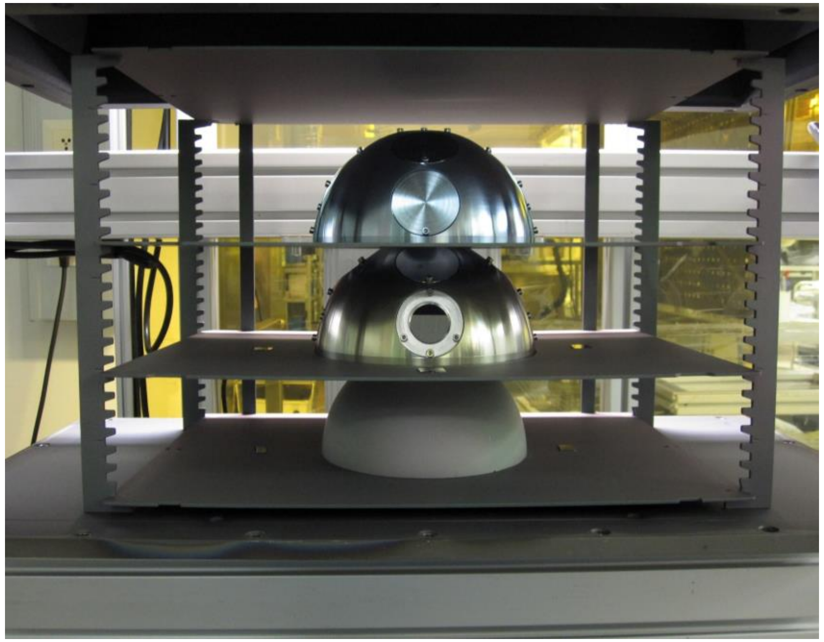
- Example: Optical domes



150 (142) mm



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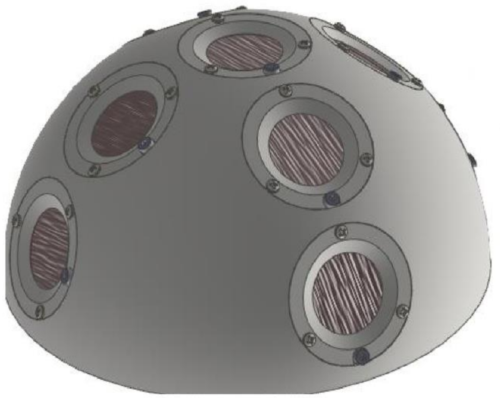
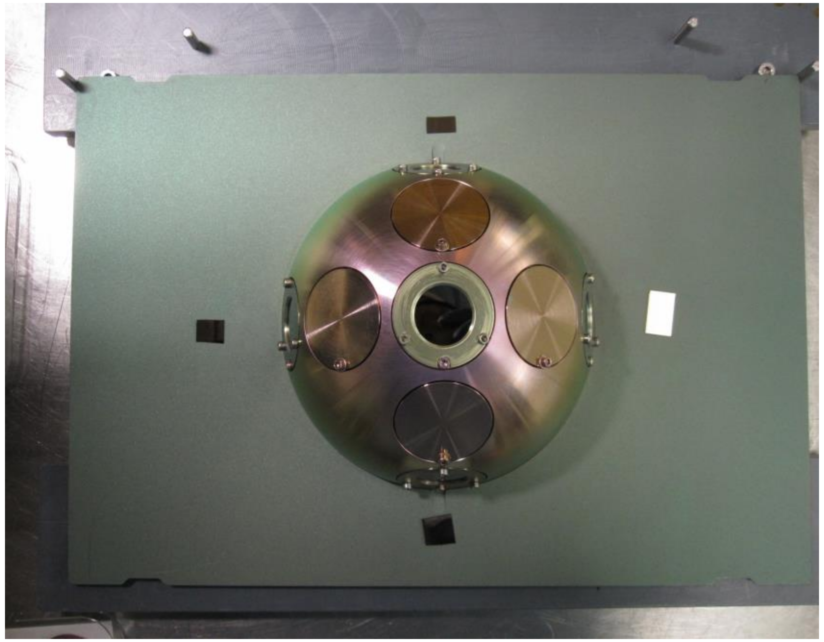


150 (142) mm



Batch production of optical coatings – 3D-structures

- ALD enables film deposition on the most complicated structures and surfaces
- Large 3D shapes, such as optical domes are examples of structures that are very difficult to coat conformally
- Example: Optical domes
  - ✓ Al_2O_3 at 120 °C

150 (142) mm

- Lower Dome

- Top [nm]

	55.1	
55.0	55.1	55.1
	55.1	

- Bottom [nm]

	55.4	
55.5	55.3	55.1
	55.2	

Batch production of optical coatings – 3D-structures

- ALD enables film deposition on the most complicated structures and surfaces
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- Example: Optical domes
  - ✓ Al_2O_3 at 120 °C

150 (142) mm

- Upper Dome

- Top [nm]

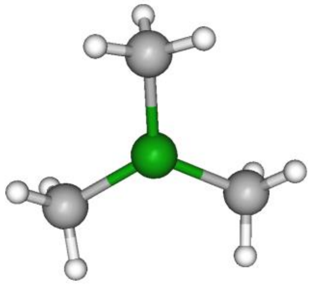
	55.3	
55.1	55.3	55.3
	55.1	

- Bottom [nm]

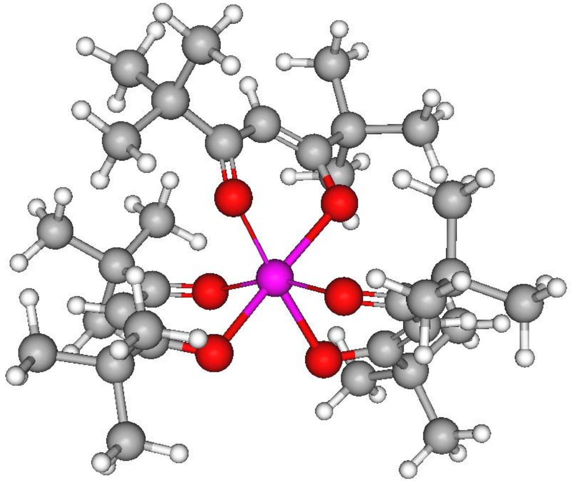
	55.3	
55.5	55.2	55.1
	55.2	

Batch production of optical coatings – 3D-structures

- ALD enables atomic scale engineering of the film properties
  - One of the key aspects in developing efficient active devices (lasers, amplifiers) e.g., for silicon photonics
- Example: Atomic-layer-deposited Er:Al_2O_3 as a silicon-integrated optical amplifier
- Erbium-doped Er:Al_2O_3 grown by sequentially depositing Er_2O_3 and Al_2O_3
  - For Al_2O_3, TMA + Water process
  - For Er_2O_3, $Er(thd)_3$ + Ozone process



TMA



$Er(thd)_3$

Batch production of optical coatings – Er-doped Al_2O_3

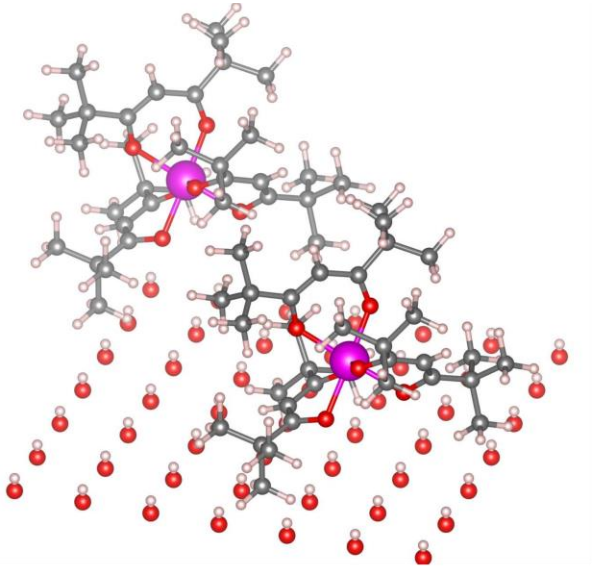
- Erbium oxide cycle

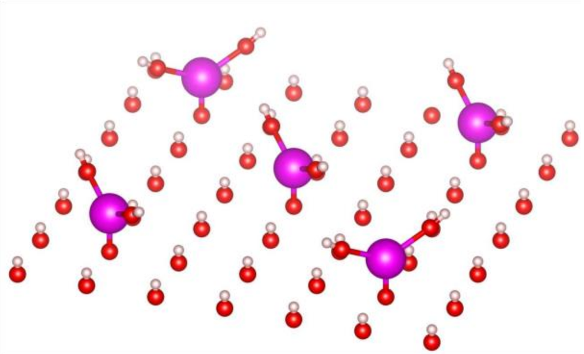
Step 1: Pulse $Er(thd)_3$ and purge

- The large size of the $Er(thd)_3$ molecule prevents close-packing of Er-ions

Step 2: Pulse ozone and purge

- The ozone burns or removes the organic ligands → Sub-monolayer of Er_2O_3

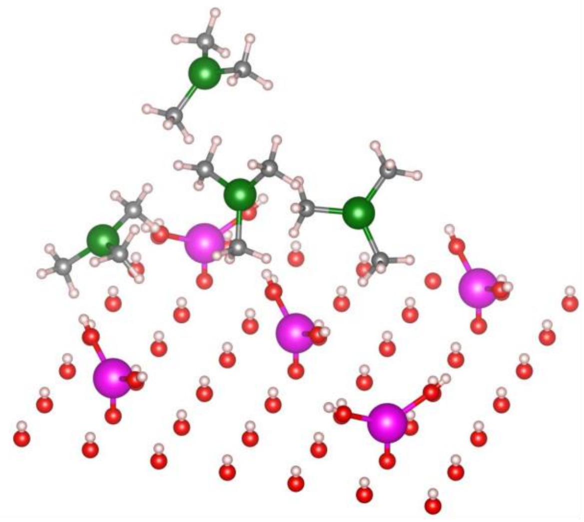




Batch production of optical coatings – Er-doped Al_2O_3

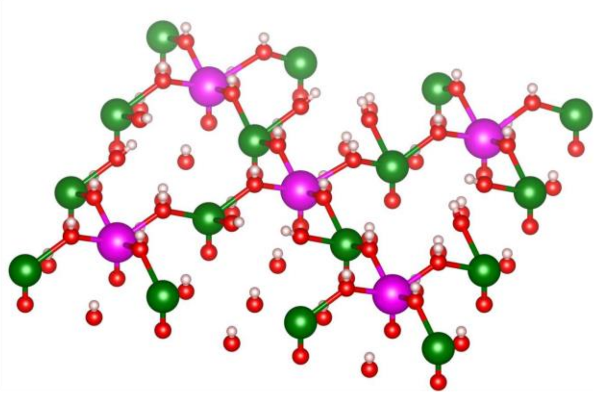
- Aluminum oxide cycle

Step 3: Pulse TMA and purge



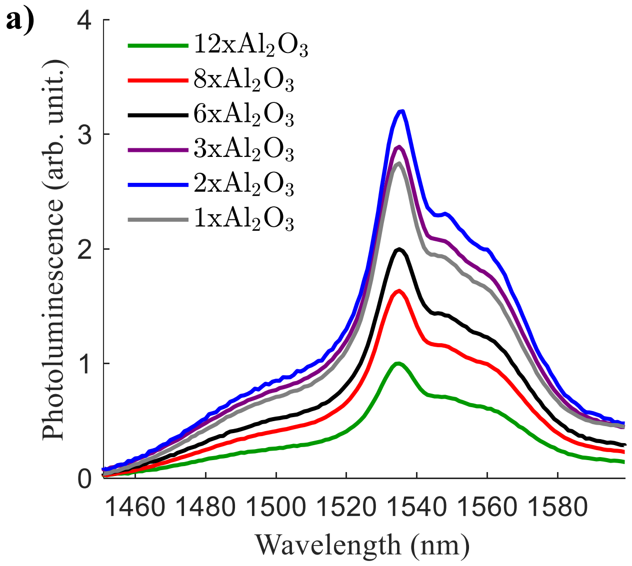
Step 4: Pulse water and purge

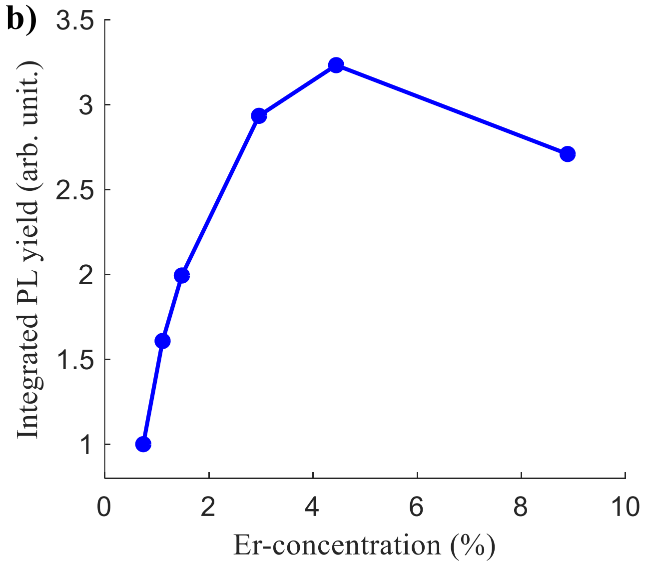
- Sub-monolayer of Er: Al_2O_3 with controlled Er-distribution is deposited



Batch production of optical coatings – Er-doped Al_2O_3

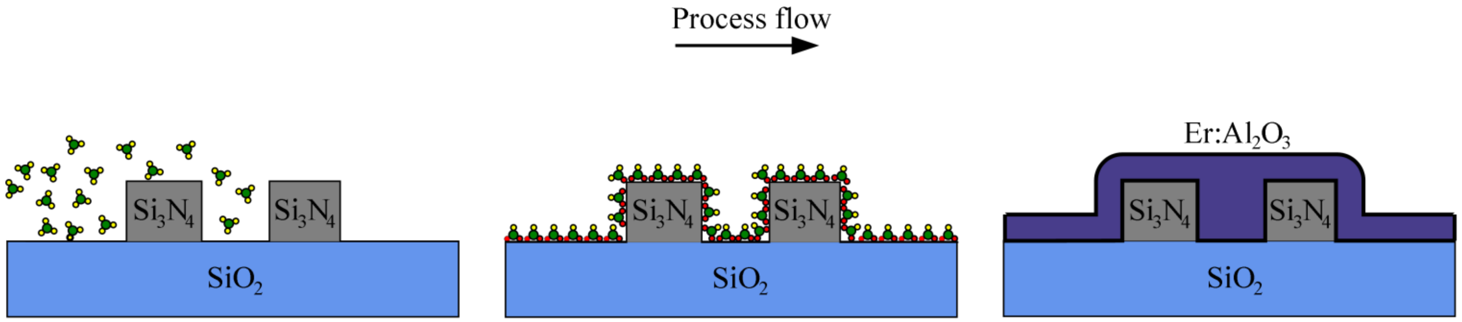
- PL response of Er-doped Al_2O_3 films with varying number of Al_2O_3 cycles in each Al_2O_3-Er_2O_3 supercycle
- The Er-concentration varies from ~0.75 to 9 %
- Peak PL at ~4.5% of Er-ions (=2xAl_2O_3), ~9% (=1xAl_2O_3) doping causes severe quenching

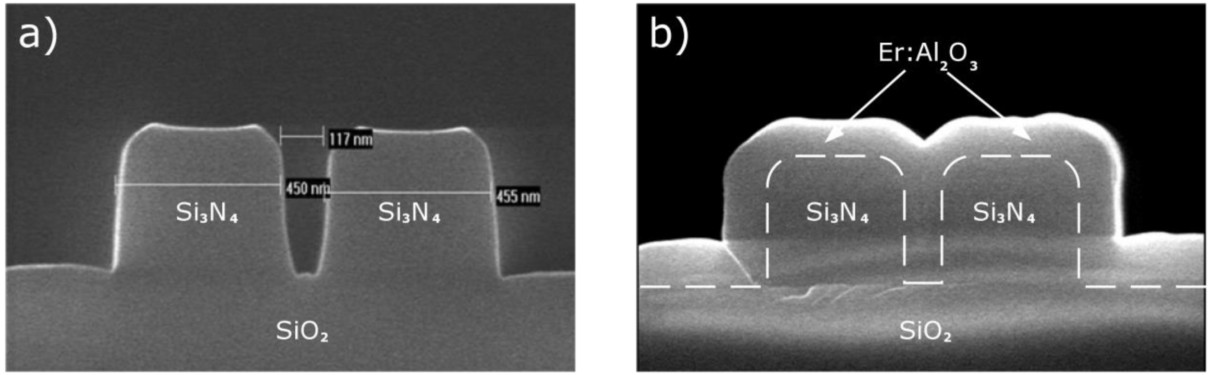




Batch production of optical coatings – Er-doped Al_2O_3

- The $Er:Al_2O_3$ can be readily deposited e.g., on a silicon integrated platform to form the amplifier

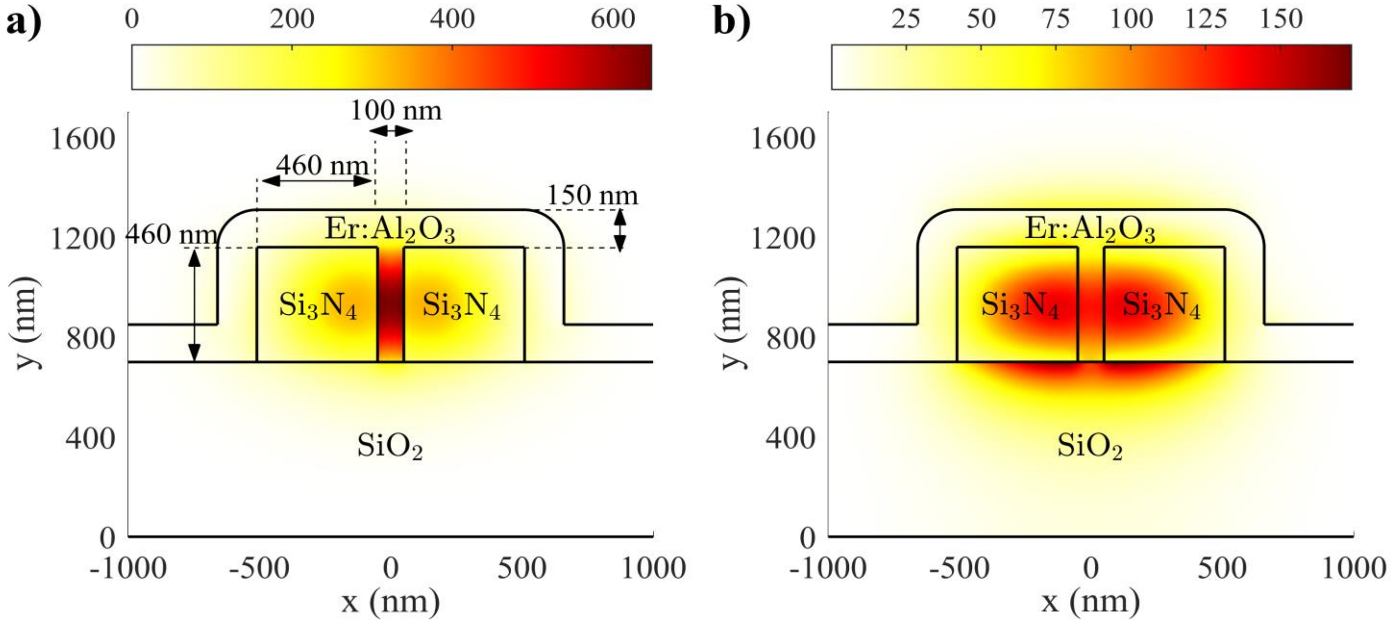




Batch production of optical coatings – Er-doped Al_2O_3

- Mode distribution in the device

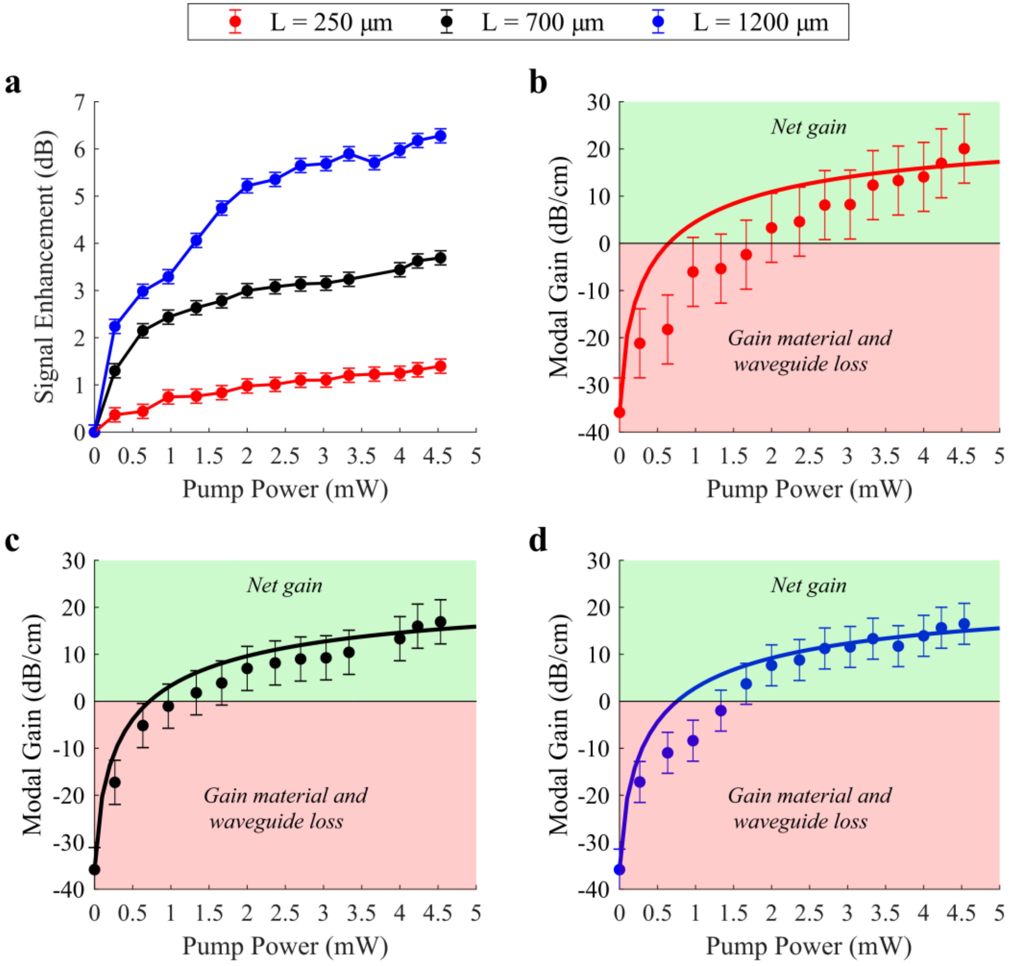
$|\vec{E}_x|^2$ of TE-mode $|\vec{E}_y|^2$ of TM-mode



33% power confinement 19% power confinement

Batch production of optical coatings – Er-doped Al_2O_3

- Pump at 1480 nm, Stimulate at 1533 nm



nature COMMUNICATIONS

ARTICLE

https://doi.org/10.1038/s41467-019-08369-w OPEN

Ultra-high on-chip optical gain in erbium-based hybrid slot waveguides

John Rönn[1], Weiwei Zhang[2,3], Anton Autere[1], Xavier Leroux[2], Lasse Pakarinen[1], Carlos Alonso-Ramos[2], Antti Säynätjoki[1,4], Harri Lipsanen[1], Laurent Vivien[2], Eric Cassan[2] & Zhipei Sun[1,5]

Rönn, John, et al. "Ultra-high on-chip optical gain in erbium-based hybrid slot waveguides." *Nature communications* 10.1 (2019): 1-9.

08/17/2021

Ultra-fast film deposition with spatial ALD

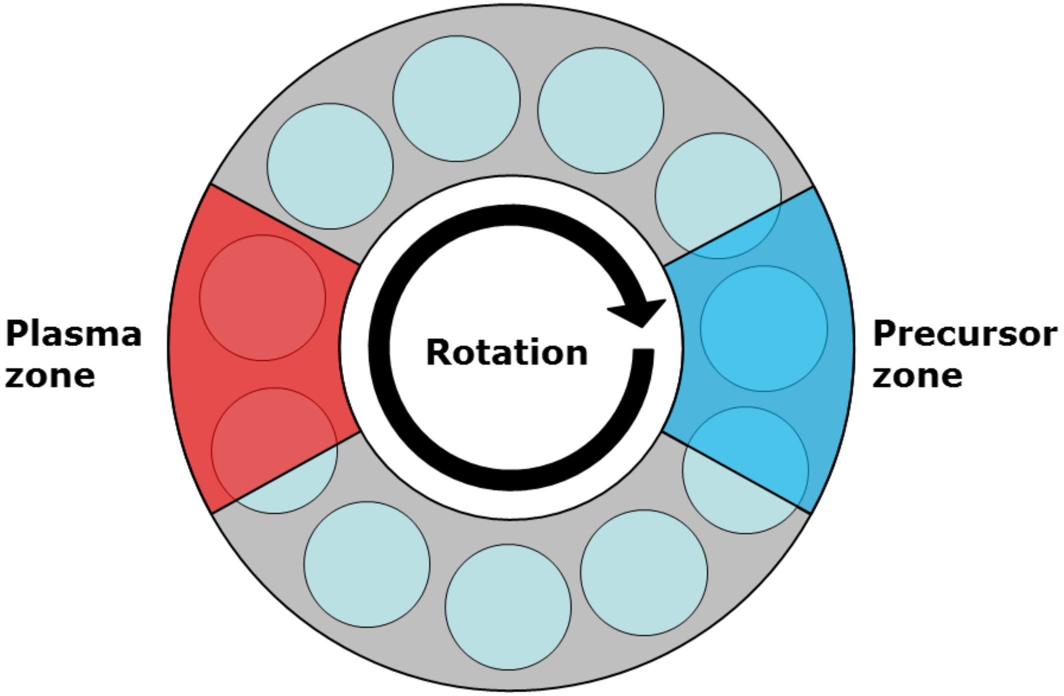
08/17/2021

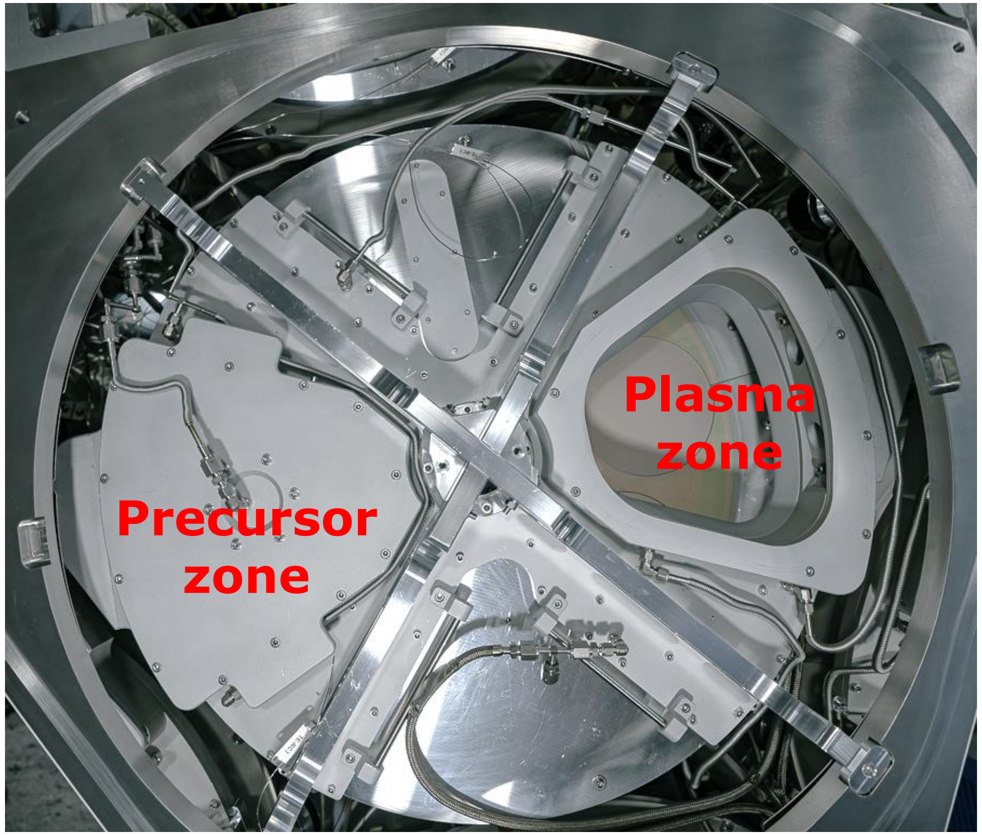
Beneq C2R

- Ultra-high deposition rates, up to several micrometers per hour
- Rotary spatial Plasma Enhanced ALD
- Single side coating
- Batch PEALD process for up to 7 pcs of 200 mm wafers
- For lenses and other 3D substrates with thickness up to 30 mm
- High film thickness uniformity, suitable for optical coating applications
- Can be equipped with a load lock or wafer automation.

Rotary Spatial PEALD

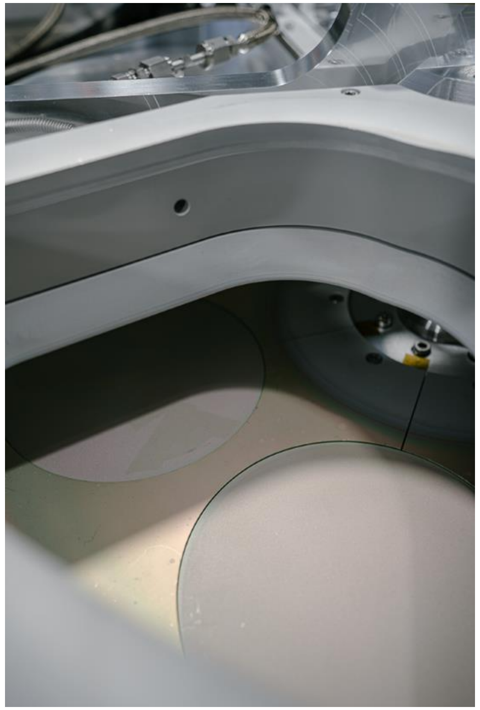
- ➢ Substrates are placed on a turn-table and rotated through precursor zone and plasma zone multiple times
- ➢ One revolution equals one ALD cycle
- ➢ High rpm's enable high deposition rates
  - At 200rpm, the cycle time is 0.3s





Beneq C2R

Item	Specification
Batch size	- 7 pcs of 200mm wafers - 13 pcs of 100mm wafers - 384 pcs of 25mm wafers
Process temperature	25 - 200°C
Process pressure	~2 mbar
Substrate orientation	Face up
Substrate loading	- Manual - Load lock (wafers only) - Transport Module (wafers only)



C2R Precursor Cabinet

- Number of Precursor sources: 4 pcs
  - Heatable, max temperature: 120°C
- Precursor canister volume: 3 liter
- Plasma gas lines: 3 pcs
- Instrumentation (MFCs, valves etc.)
- Ventilation connection and related safety components

C2R in action

➢ Check the video from beneq.com

Rotary Spatial PEALD for optical coatings

- Processes for low and high refractive index materials
  - SiO_2 from tetraethylsilanediamine (SAM-24)
  - TiO_2 from titanium tetraisopropoxide (TTIP)
  - Ta_2O_5 from Tris(ethylmethylamido)(tert-butylimido)tantalum (TBTEMT)
- Choose processes which result in dense, amorphous, optically homogenous coatings
- Use low substrate temperatures, for broad substrate compatibility
  - Also for minimization of CTE-mismatch induced film stress

Characterization Methods

- Stoichiometry was determined using RBS of 50nm films
  - SIMS was used for detection of residual carbon from precursors and aluminum from plasma electrode
- Optical constants were determined by spectral analysis
  - Films 250-1,000 nm thick on fused silica, analyzed using OptiLayer software
- Optical loss at 1064 nm wavelength determined using a laser ring-down technique
- Surface roughness measured using a Zygo 5500 Heterodyne profilometer
- Conformality determined by cross-section SEM imaging of coated trenches in silicon

In collaboration with

Analytical Results – RBS and SIMS

	Metal: Oxygen ratio	Ideal	Residual Carbon	Residual Aluminum
SiO_2	0.46	0.5	< 0.1%	0.3%
TiO_2	0.50	0.5	2.5%	0.3%
Ta_2O_5	0.37	0.4	2.0%	1.0%

- Near ideal stoichiometry for TiO_2, slightly oxygen rich for SiO_2 and Ta_2O_5
- Al concentration shows inverse relationship with rotation speed when using plasma electrode made of Aluminum (can be eliminated by using Titanium electrode)

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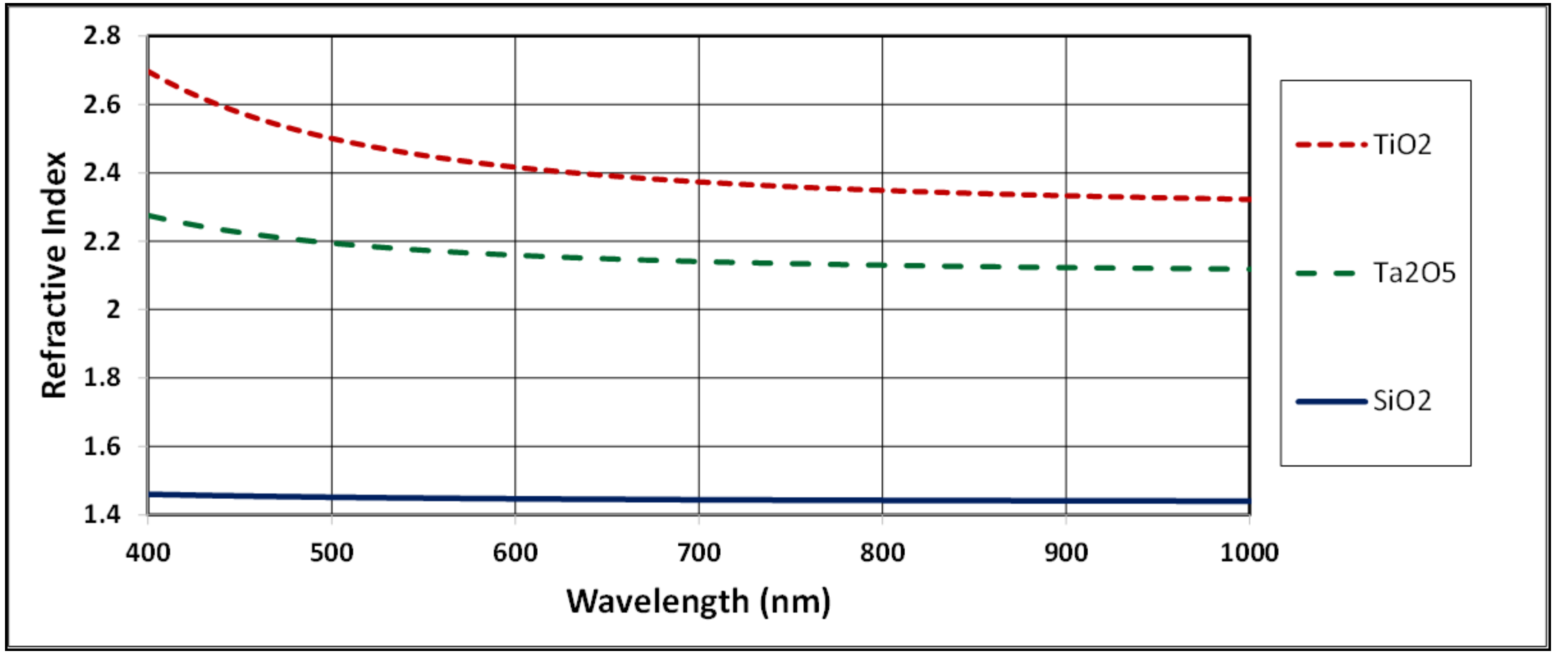
Optical Properties



	Refractive Index @ 550 nm	Refractive Index @ 1000 nm	Optical Loss @ 1064 nm	Normalized Loss @ 1064 nm for 1µ Thick Layer
1,000 nm SiO_2	1.45	1.44	~ 16 ppm	~ 16 ppm
240 nm TiO_2	2.45	2.32	~ 18 ppm	~ 75 ppm
250 nm Ta_2O_5	2.17	2.12	~ 40 ppm	~ 160 ppm

- Good optical materials with low optical loss

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Lotus applied technology

Beneq

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Surface Roughness

	Film Thickness	RMS Roughness	Peak-Valley Roughness
SiO_2	1,000 nm	0.8 Å	5.5 Å
TiO_2	240 nm	0.6 Å	4.0 Å
Ta_2O_5	250 nm	0.5 Å	2.9 Å

- Films Deposited on ¼″ thick "Super Polished" fused silica substrate (pre-characterized)

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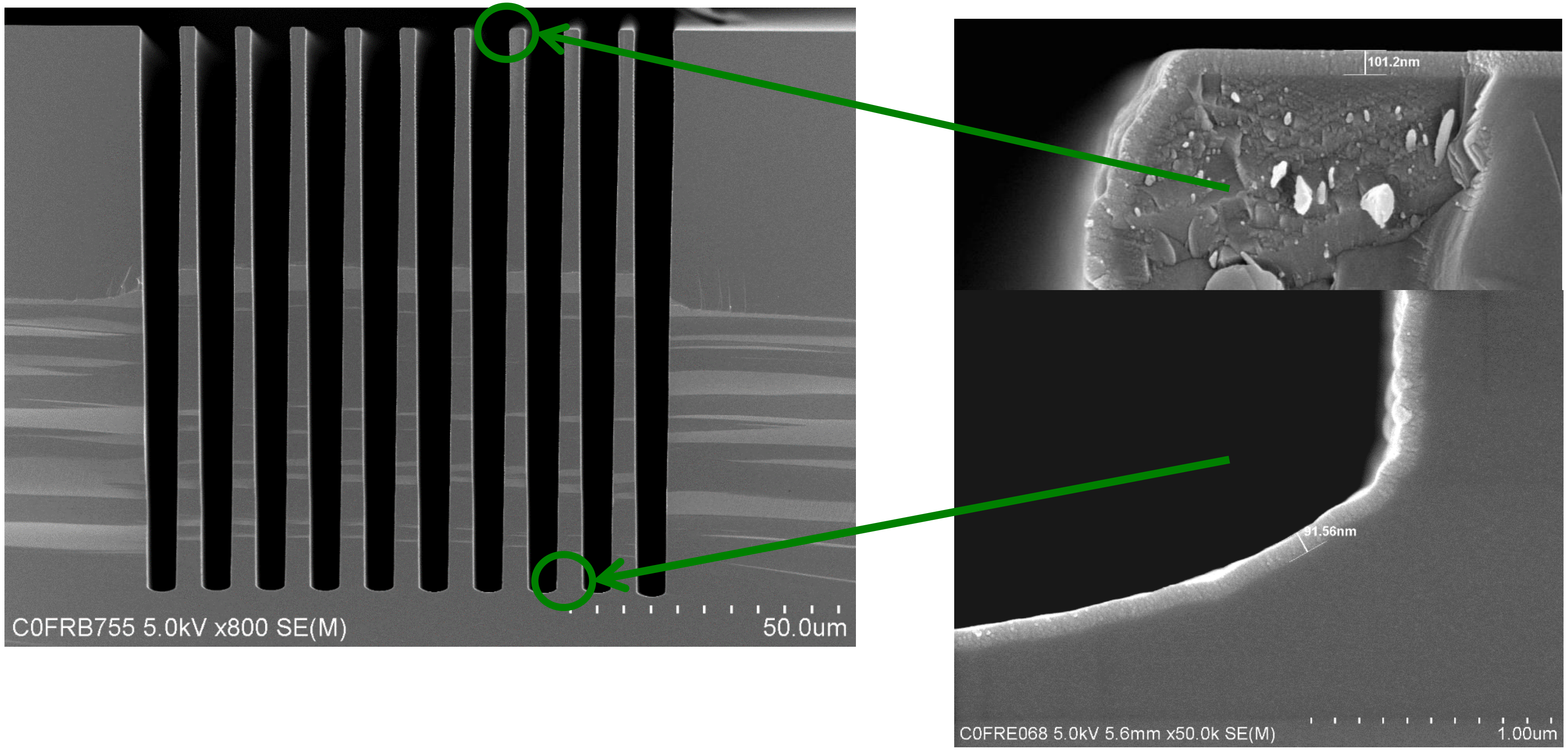
Lotus applied technology

Conformality Measurement

Ta2O5 Deposited at 150 RPM



- Silicon substrates prepared using “Bosch” Deep Reactive Ion Etch to mill trenches
  - Nominally 5-7μ wide by ~ 120μ deep

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lotus applied technology

beneq

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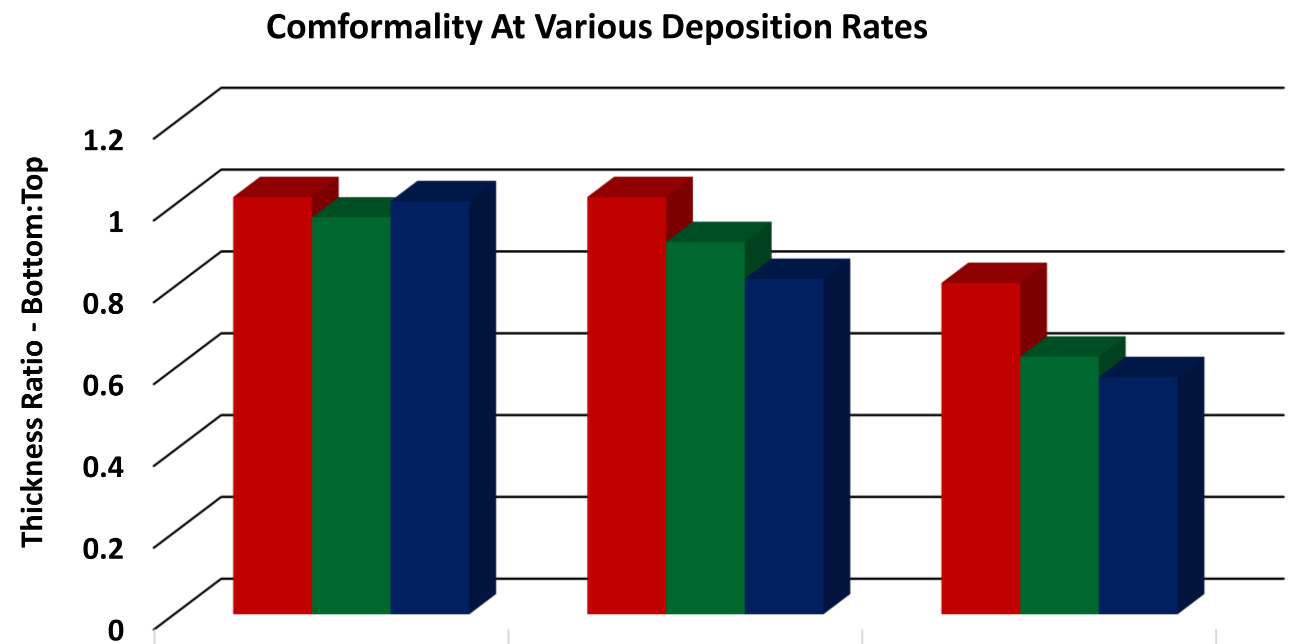
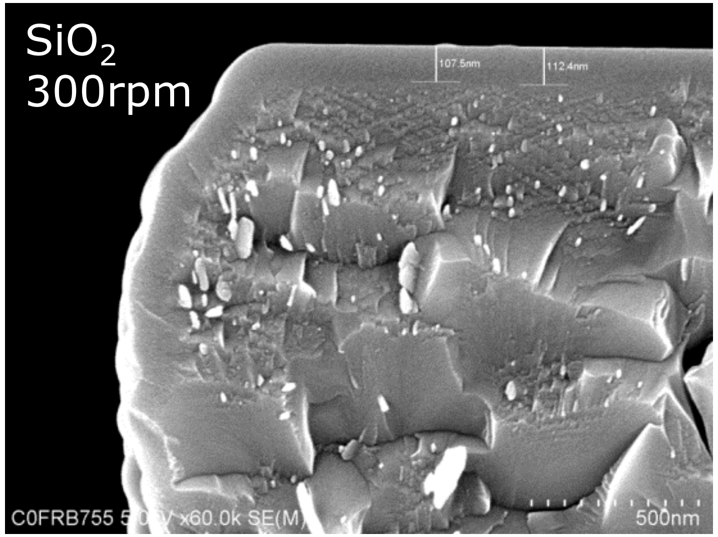
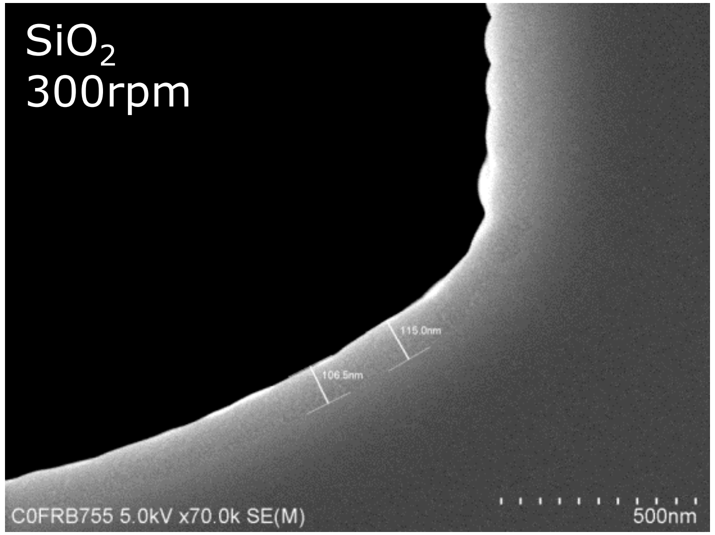
Conformality Results

- Compare the coating thickness at the bottom and top of trench



	SiO2	Ta2O5	TiO2
60 RPM	1.02	1.02	0.81
150 RPM	0.97	0.91	0.63
300 RPM	1.01	0.82	0.58





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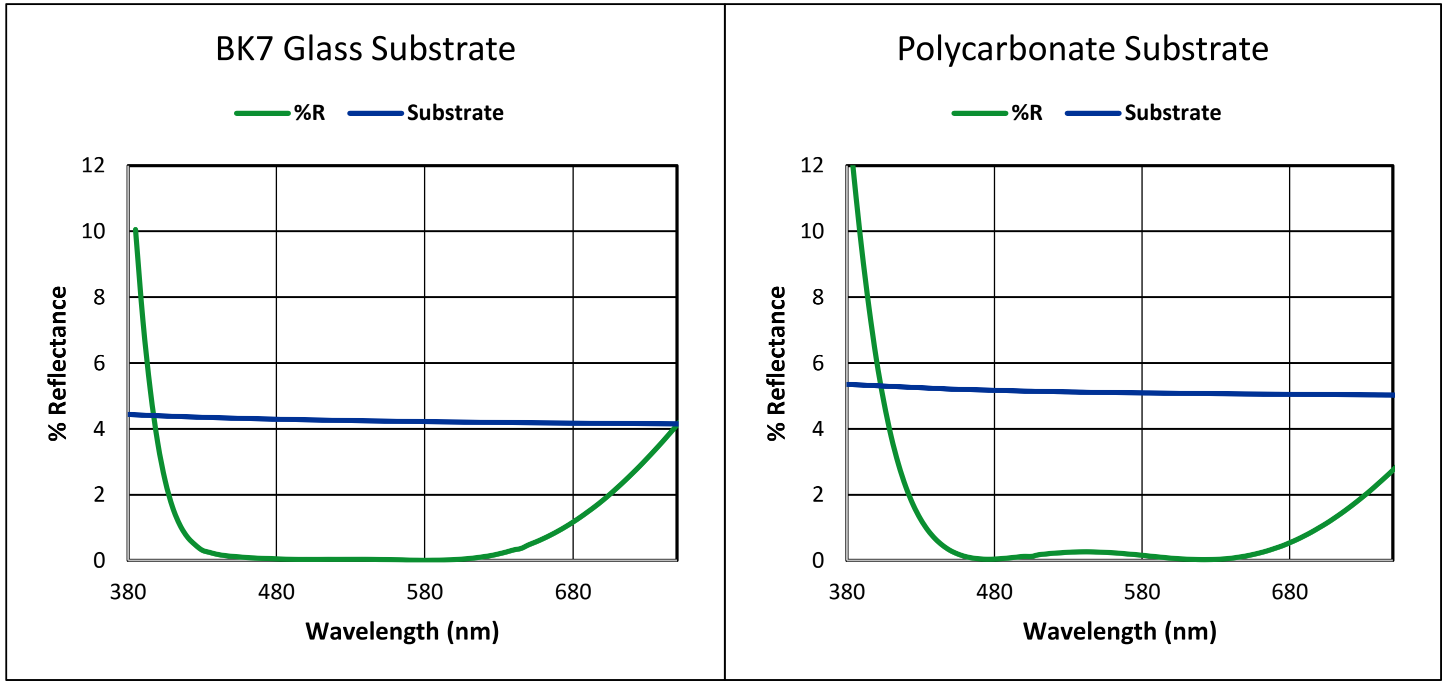
Deposition of an Anti-Reflection Coating

- SiO_2 (L) and TiO_2 (H)
  - Design: 0.22H + 0.37L + 2.0H + 0.95L
  - 85°C for polycarbonate substrate, 90°C for BK7 glass
  - Rotation speed of 200 RPM (200 ALD cycles per minute)
  - Total deposition time of ~ 18 minutes



- Weighted photopic reflectance of about 0.07% for AR on glass
- R < 0.1% for range of 460nm to 615nm

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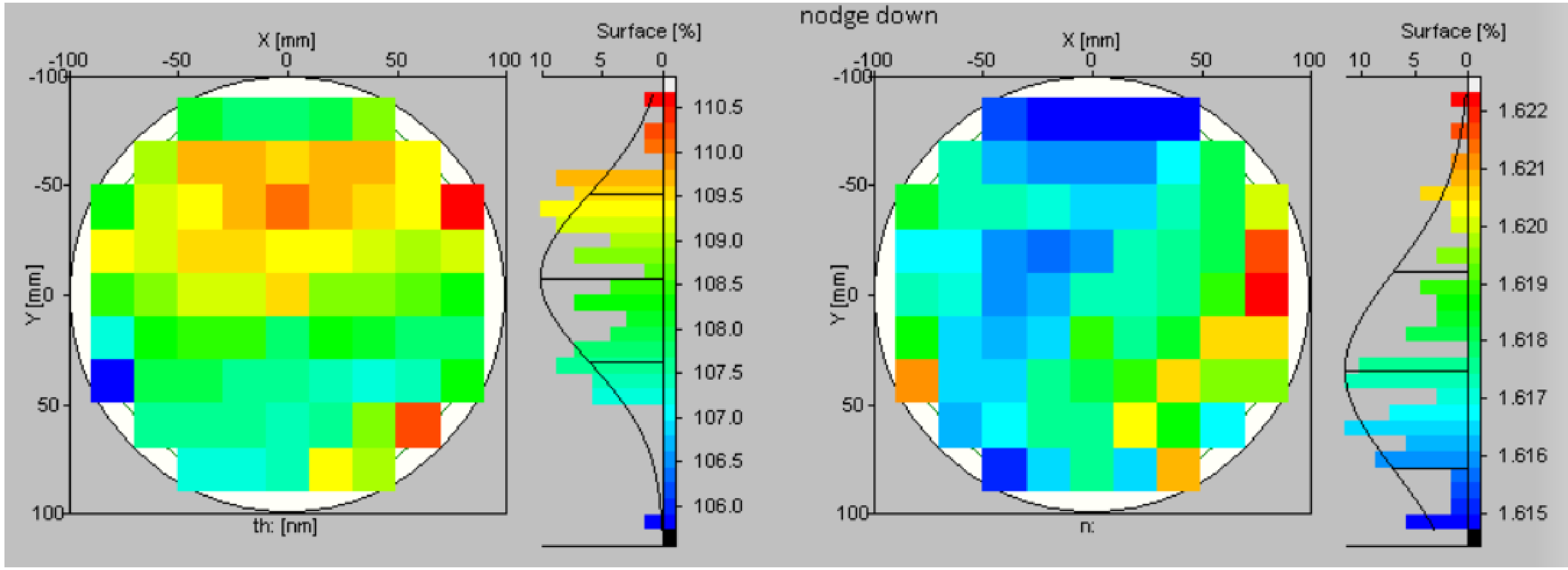
Beneq C2R

Preliminary process results from
C2R-101 testing at Beneq

Al_2O_3

- Precursors: TMA + O_2-Plasma

Temp. (°C)	RPM (1/min)	Cycles	Duration (min)	Thickness (nm)	GPC (nm/c)	Growth Rate (nm/h)	Non-Uni. (+/- %)	n
90	**50**	**765**	**15.3**	**108.7 ± 3.81**	**0.142**	**426.3**	**1.75**	**1.617 ± 0.011**
120	100	1000	10	134.3 ± 7.59	0.134	805.6	2.82	1.622 ± 0.006
120	200	1000	5	128.64 ± 6.36	0.129	1543.6	2.65	1.614 ± 0.007



Beneq

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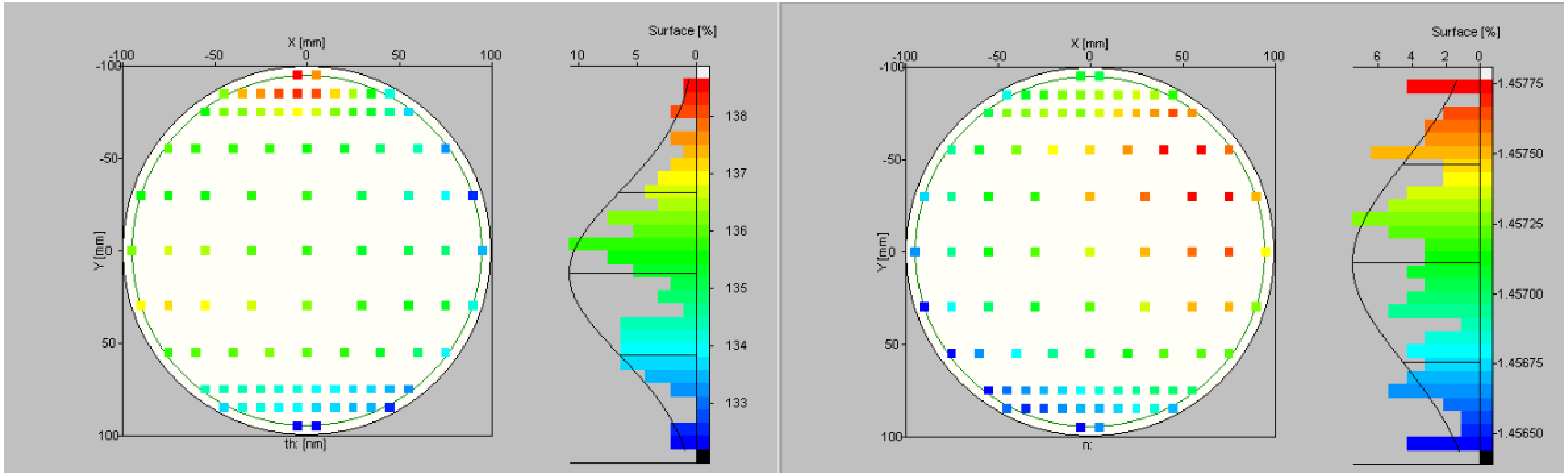
SiO$_2$

- Precursors: SAM24 + O_2-Plasma

Temp. (°C)	RPM (1/min)	Cycles	Duration (min)	Thickness (nm)	GPC (nm/c)	Growth Rate (nm/h)	Non-Uni. (+/-%)	n
120	**100**	**1824**	**18.24**	**135.3 ± 6.45**	**0.074**	**444.9**	**2.38**	**1.457 ± 0.001**
120	150	1824	12.16	111.3 ± 10.3	0.061	549.4	4.64	1.457 ± 0.001
120	200	1824	9.12	99.8 ± 11.4	0.055	656.8	5.70	1.457 ± 0.003



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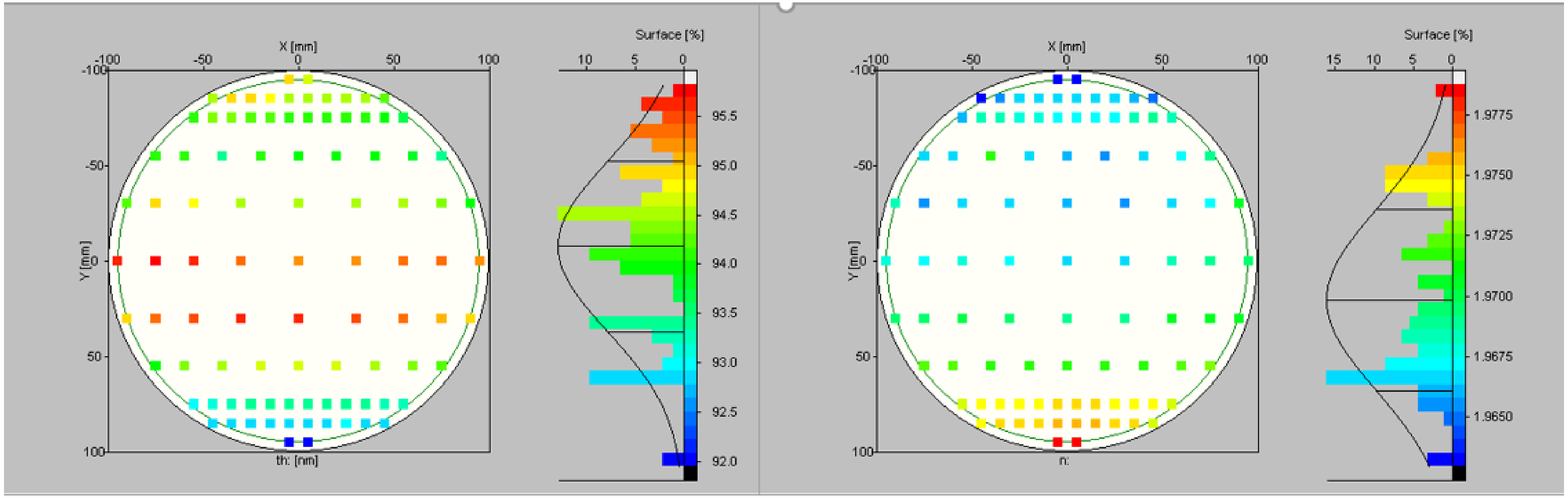
Ta_2O_5

- Precursors: TBTEMT + O_2-Plasma

Temp. (°C)	RPM (1/min)	Cycles	Duration (min)	Thickness (nm)	GPC (nm/c)	Growth Rate (nm/h)	Non-Uni. (+/-%)	n
120	**100**	**1100**	**11.0**	**94.2 ± 2.06**	**0.086**	**513.7**	**2.06**	**1.970 ± 0.016**
150	150	1100	7.33	82.2 ± 3.16	0.075	672.9	3.16	2.021 ± 0.132
150	200	1100	5.50	74.7 ± 5.60	0.068	814.7	5.60	1.996 ± 0.023



08/17/2021

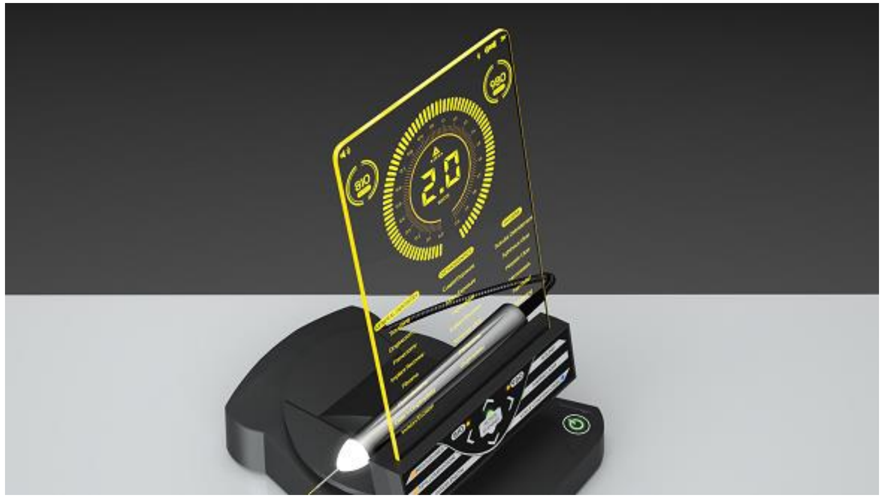
Beneq C2R - Summary

- Ultra-high deposition rates
- Single side coating
- Enables PEALD advantages in production scale
    - Plasma-enabled materials; low temp SiO_2, SiN…
    - Stress control
    - Lower process temperatures
- For lenses and other 3D substrates with thickness up to 30 mm
- Can be equipped with a load lock or wafer automation.

Beneq – Home of ALD

BENEQ – HOME OF ALD

- World's leading ALD-dedicated company
  - ✓ 40+ years of ALD expertise
  - ✓ 40+ dedicated ALD-system in operation 24/7
  - ✓ 170+ personnel



08/17/2021

BENEQ – HOME OF ALD

- Focus on
  - **ALD-equipment**
  - Coating services
  - Development services

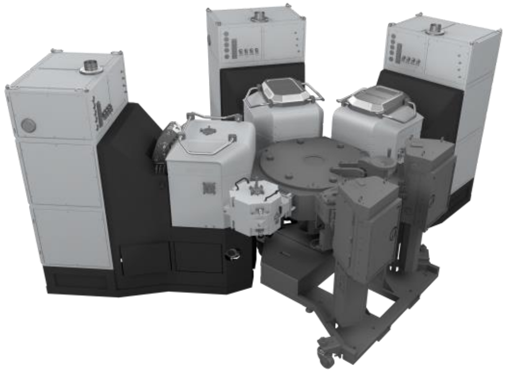
P400

P800

TFS 200

TFS 500

BENEQ C2R



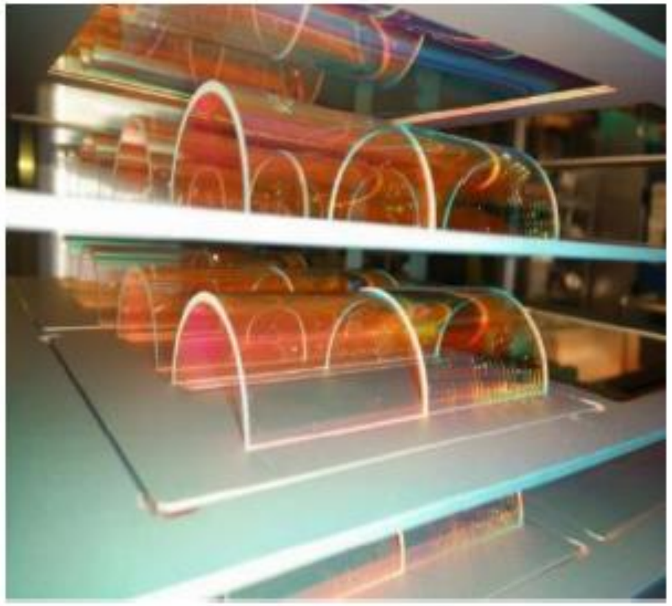
BENEQ Transform™

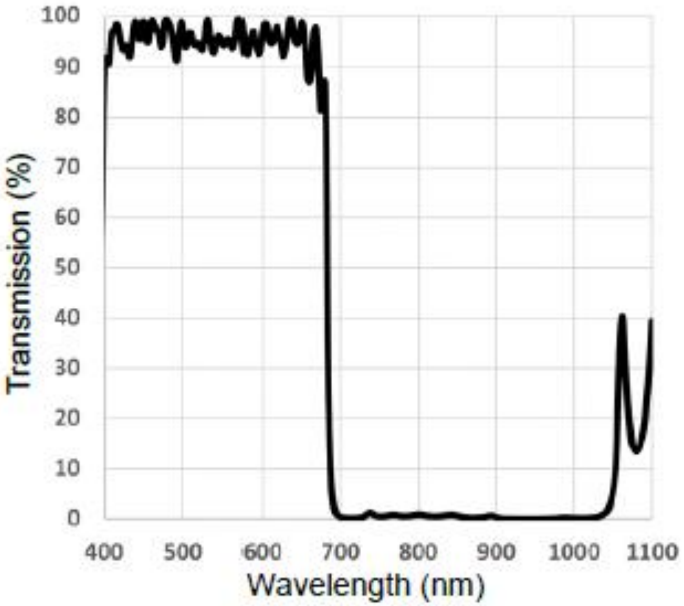
BENEQ – HOME OF ALD

- Focus on
    - ALD-equipment
    - **Coating services**
    - Development services

BENEQ – HOME OF ALD

- Focus on
  - ALD-equipment
  - Coating services
  - **Development services**





Beneq® is a leading supplier of production and research equipment for atomic layer deposition (ALD), a provider of thin film coating services, and the world’s premier manufacturer of thin film electroluminescent (TFEL and TASEL) displays.

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