

**PSI** Center for  
Photon Science

# **Advancements in Fabricating High-Precision VLS Blazed Gratings on Non-Flat Silicon Substrates**

**Dr. Kevin Hofhuis :: E-Beam Scientist**

**Nanotechnology group at Paul Scherrer Institute (CH)**

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## Goal

Fabricating sawtooth profiles on curved Silicon substrates for x-ray diffraction gratings

## Grayscale lithography

- Dose gradient shapes → a new design element for height varying resist exposures
- Through-mask oxidation → transfer technique from resist to Si substrate

## Variable line spacing (VLS) gratings

- Curved substrates → important for focusing x-ray optics
- Mainfield scaling → high precision placement changing the grid ever so slightly

# Blazed x-ray diffraction gratings

Blaze angle is optimized for a specific wavelength and diffraction order

- Highly efficient → as much light into the desired order

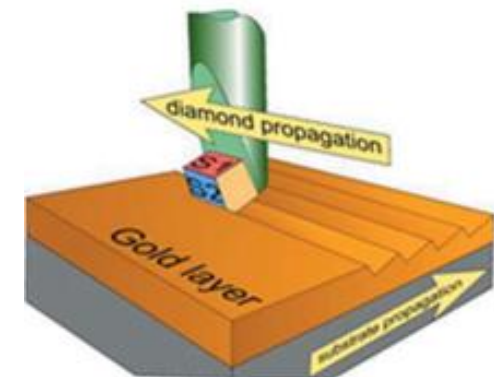
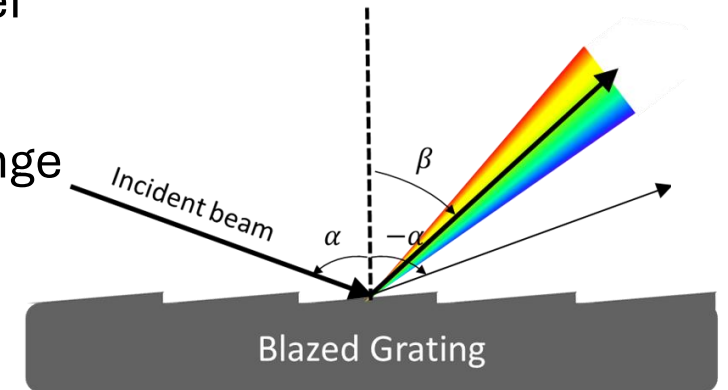
Application at Synchrotrons and XFELs in the soft and tender x-ray range

- Monochromators
- Analyzers for inelastic scattering
- Self-seeding and pulse compression

Issue: Long lead times and shortage of supply

- Commonly used manufacturing method is mechanical ruling
- Higher line densities → longer manufacturing times
- Highly specialized equipment (one in Europe)

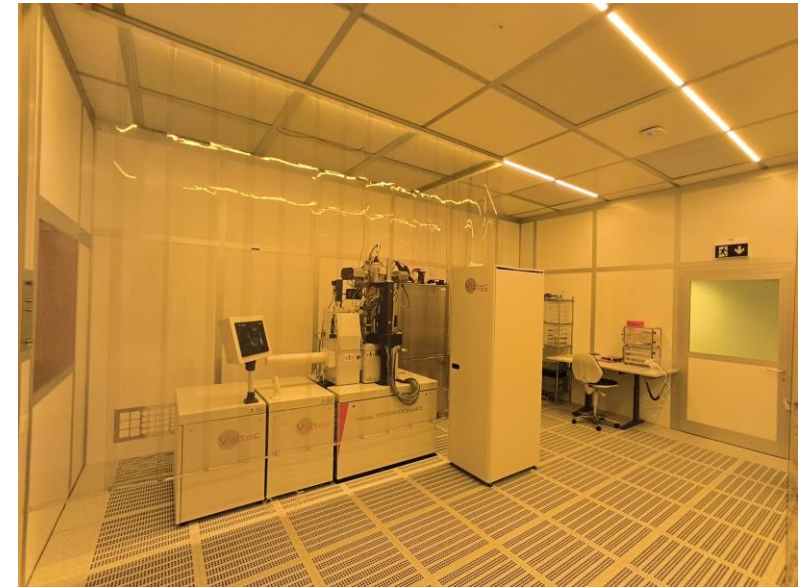
LEAPS Innov proposed solution is electron beam lithography



# Our e-beam machine and location

EBPG 5000+ with UPG (Universal Pattern Generator)

- Beyond trapezoid fracturing
- Nanometer placement ( $< 1 \text{ nm}$ )
- Sufficient throughput at exposure rates of  $1 \text{ cm}^2/\text{hr}$ 
  - Even for higher line densities
- Substrates up to  $140 \times 30 \times 10 \text{ mm}^3$



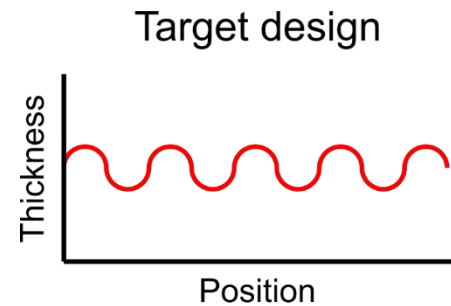
E-beam at PICO  
The new cleanroom in PiA



Park InnovAare

# Grayscale lithography - concept

Grayscale = thickness/height variations



New developments

1. Dose gradient primitive shape
2. Efficient exposure
3. Through-mask oxidation using HSQ resist

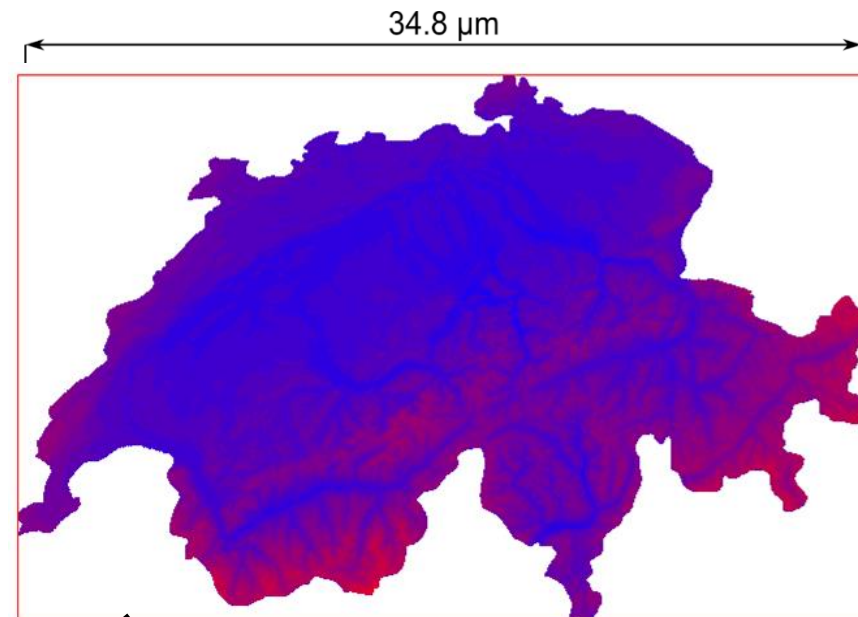
Let's go through an example

# Grayscale lithography - example

Target: Grayscale 16bit map

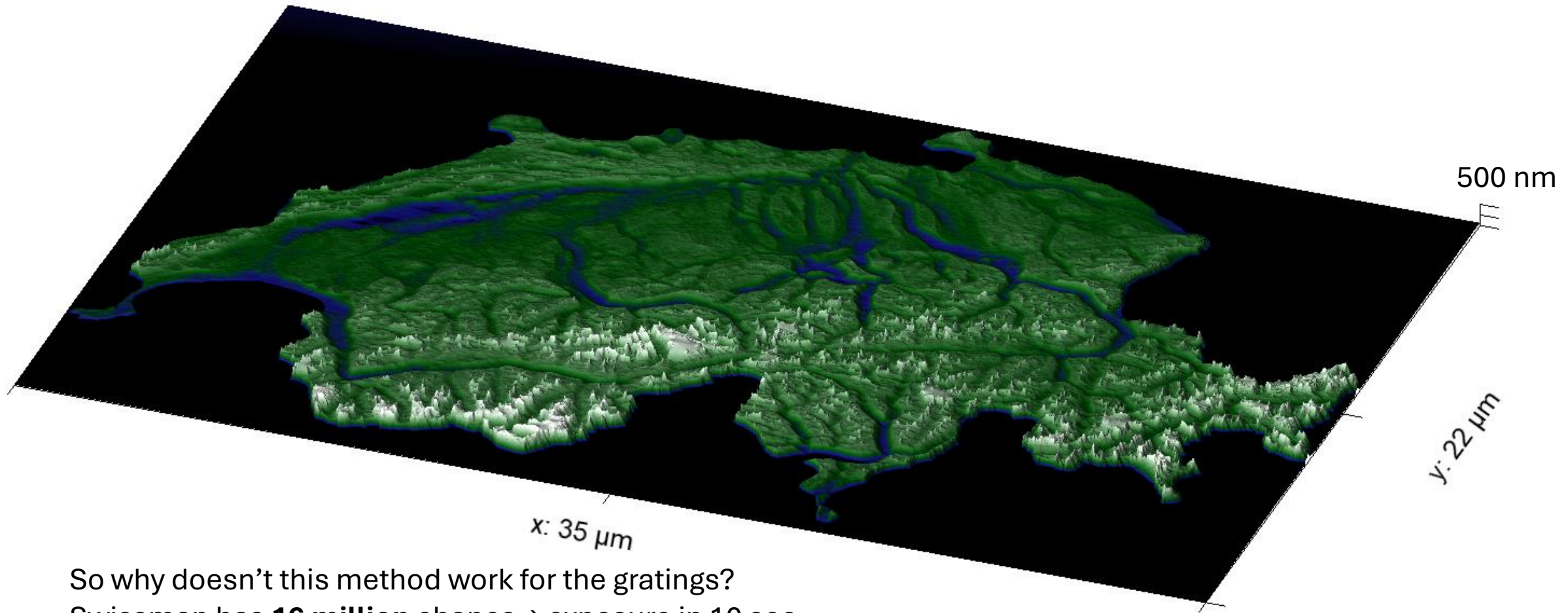


Data prep: 46 MB for 35 doses



BEAMER w/ 3D Surface PEC

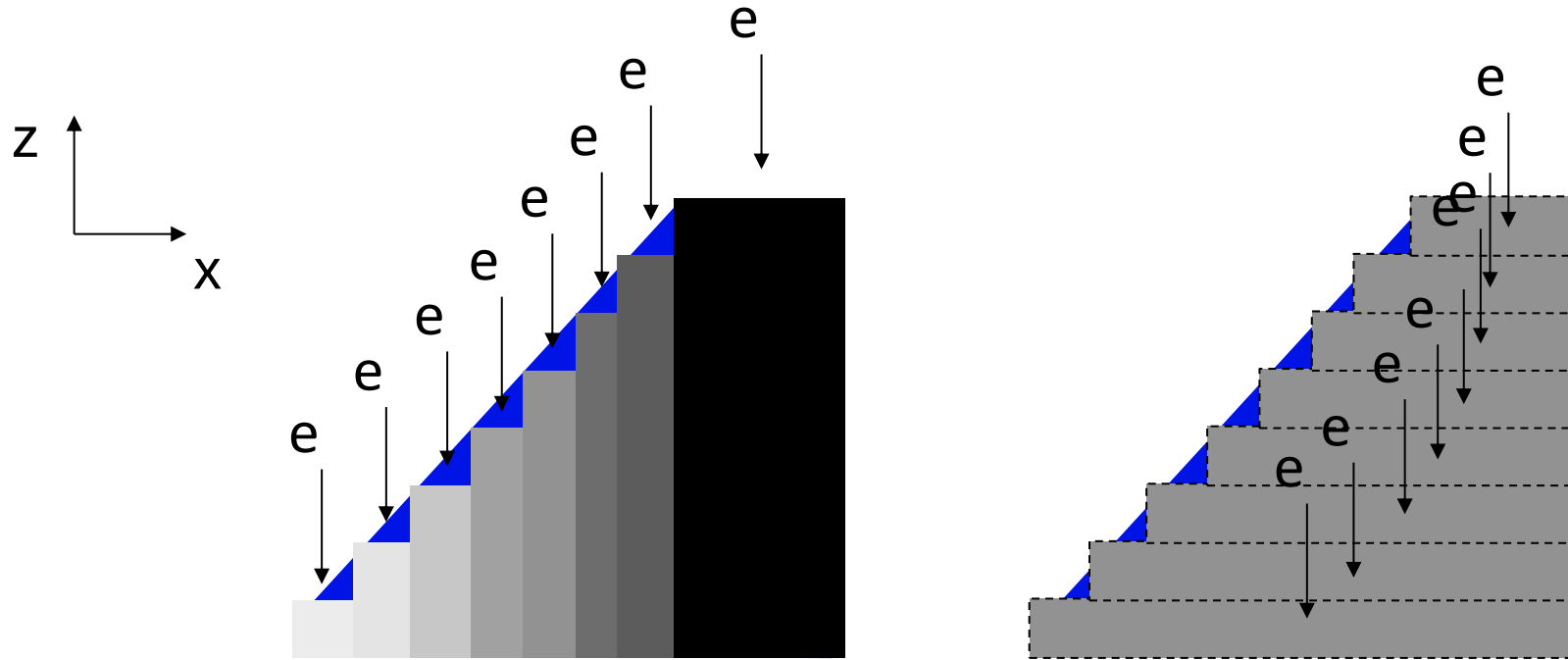
# Grayscale lithography - results



So why doesn't this method work for the gratings?  
Swissmap has **16 million** shapes → exposure in 10 sec  
Grating is **12 million** times larger

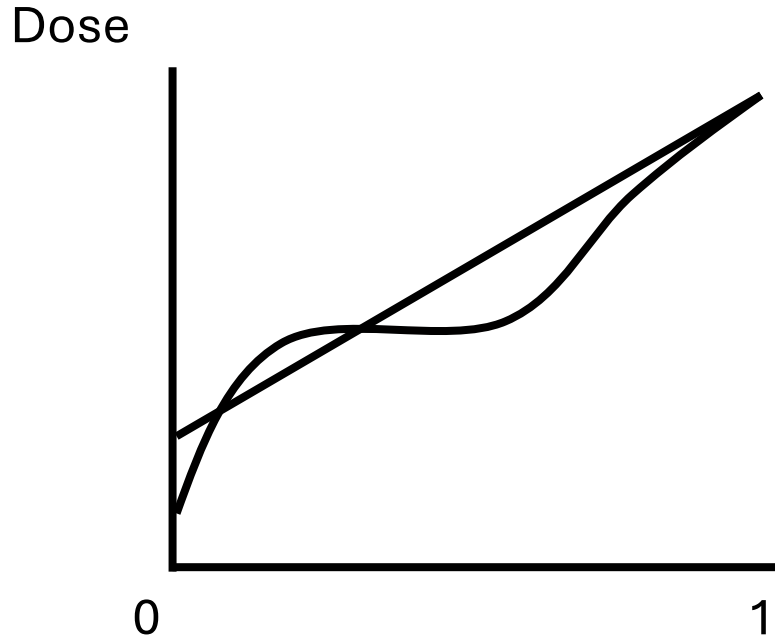
# Grayscale data preparation - fracturing

Current methods of fracturing

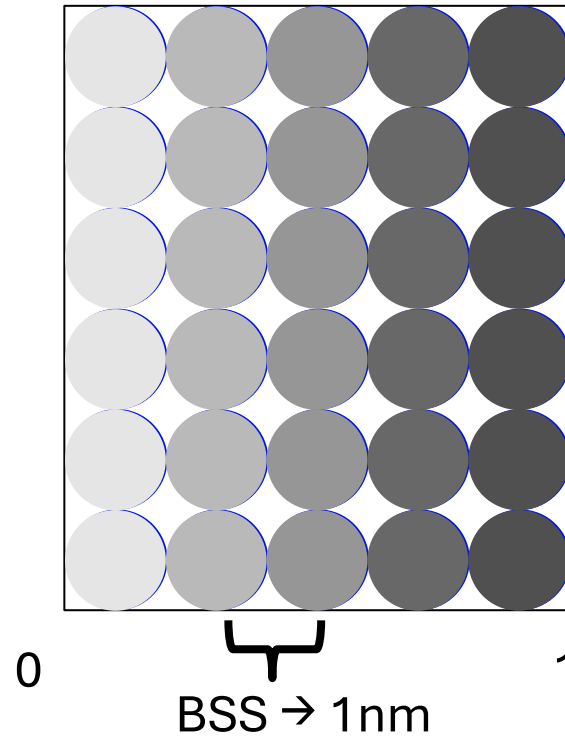


Each change in dose needs another new shape  
Amount of shapes explodes!

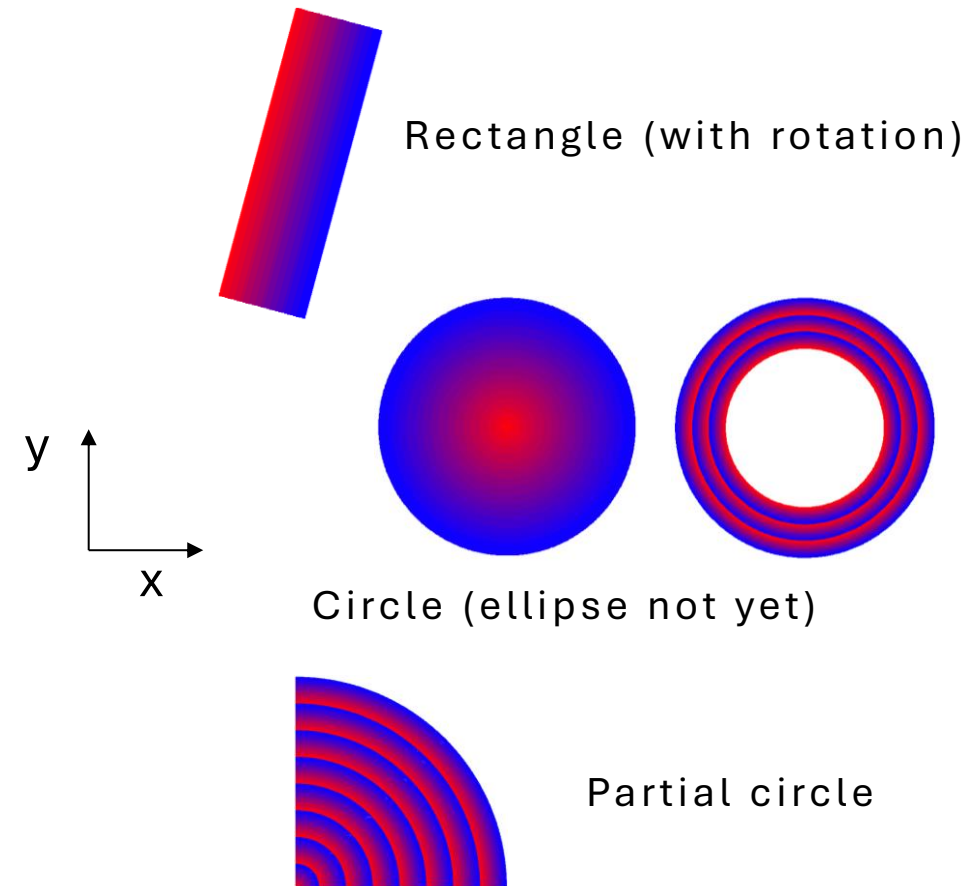
# Dose mapping




Dose gradient cube



Basic shapes



Result: Efficient grayscale lithography data preparation

Comparing old vs new method 

Design VLS of 120 x 20 mm<sup>2</sup>

## Old method

146 GB gtx (ascii)

11 GB gpf (binary)

**38 billion shapes**

## New method

30 MB gtx (ascii)

9 MB gpf (binary)

**950 million shapes**

Result: Storing the dose information inside the shape reduces the file size drastically, and is easier to program/debug

# Grayscale EBL and through-mask oxidation

## Spin coating

- HSQ as resist, Thickness  $\sim 300$  nm

## Grayscale exposure

- Exposed area is converted to  $\text{SiO}_x$

## Development

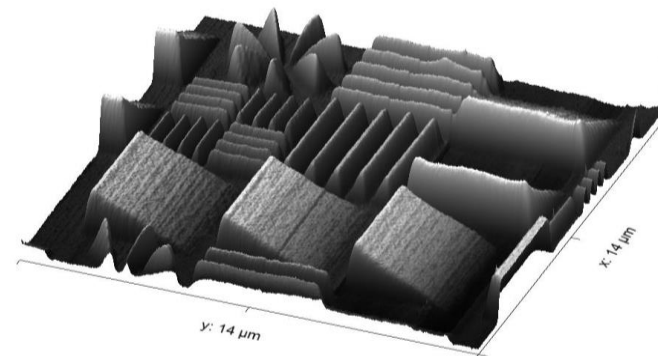
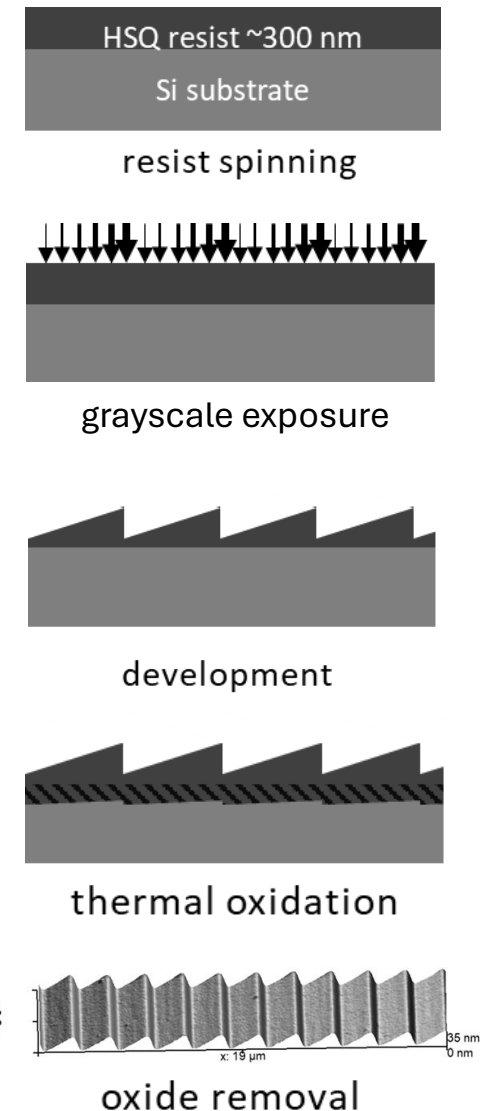
- NaOH based developer

## Thermal oxidation

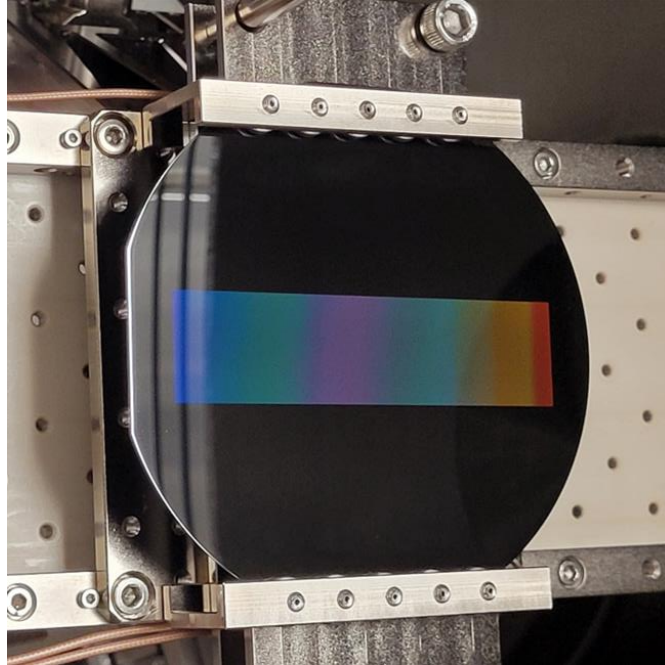
- Dry oxidation, Different  $\text{SiO}_x$  thickness  $\rightarrow$  different oxidation speed

## Oxide removal

- Wet chemical oxide removal with HF  $\rightarrow$  reduces the roughness



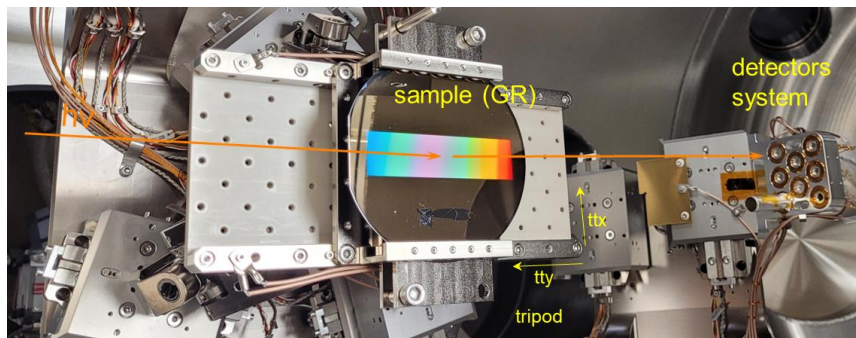
# Grayscale EBL and through-mask oxidation



Result: Smooth surface due to height feature reduction (10x)

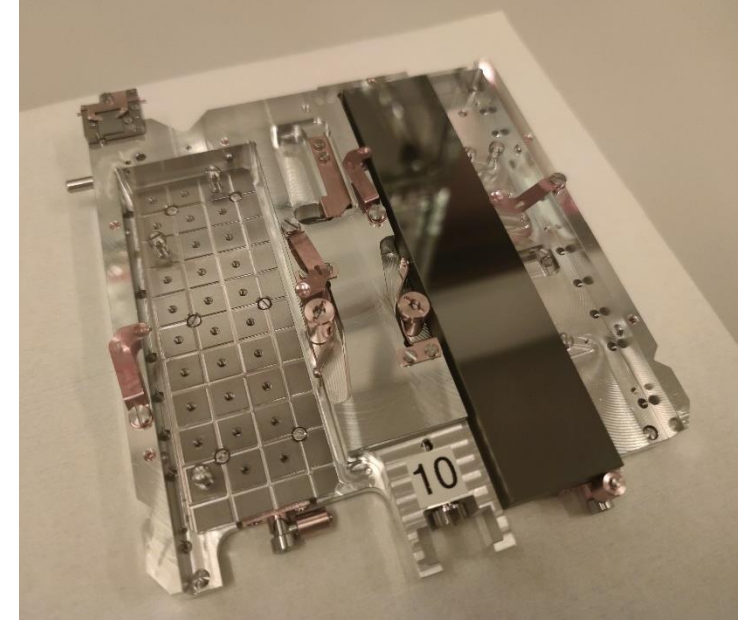
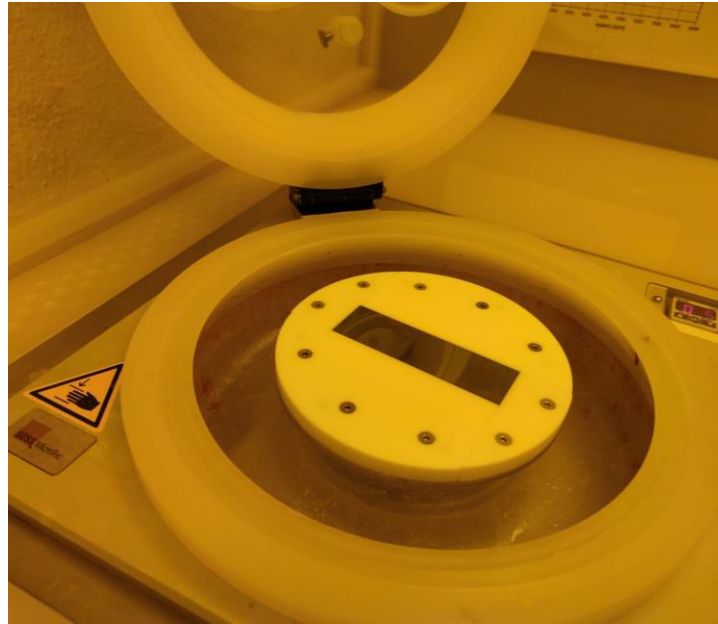
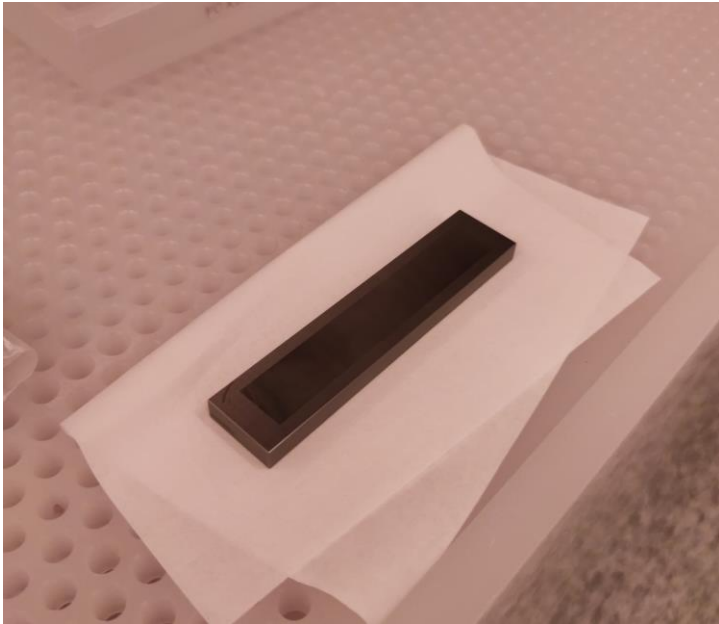


development



oxide removal

# Thick curved substrates



Radius of curvature is 28 m or 12 m

- Maximum height difference: 100  $\mu\text{m}$  to 200  $\mu\text{m}$  (tool limit)

In the near future: Z-stage in 2025 with up to 10 mm range

Combined with a new feature for tilt corrections  $\rightarrow$  even curvier substrates

# High-precision placement

Example: Write lines with a pitch of 100.4 nm

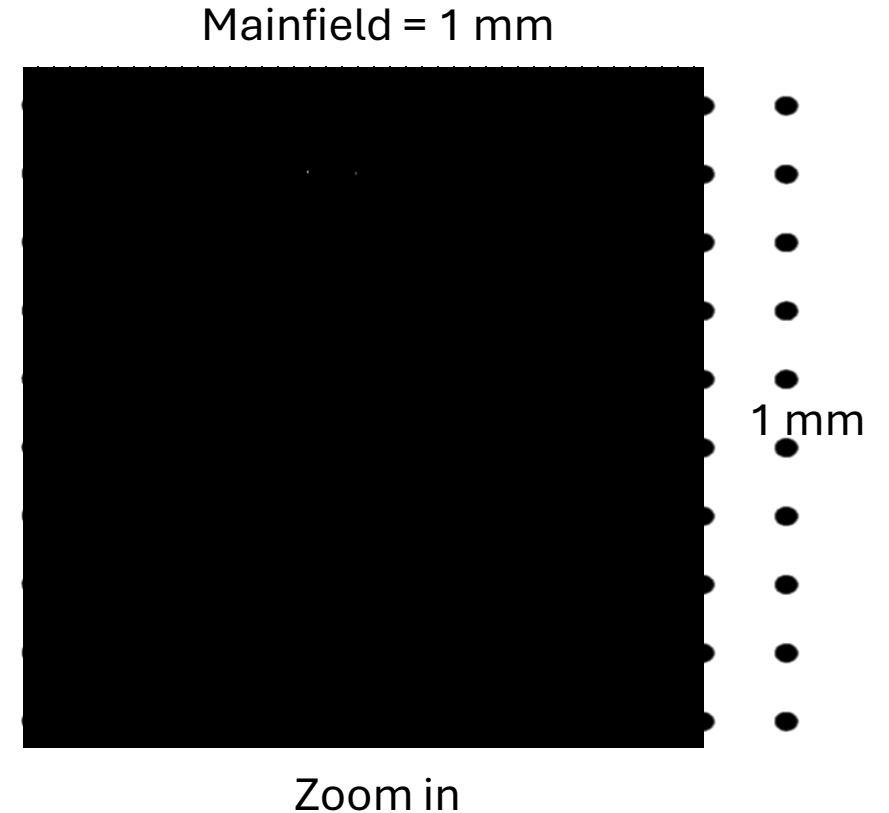
- Grid = 1 nm (20 bits  $\rightarrow$  min 0.07 nm)

Problem: Grid snapping

Solution: Mainfield scaling

- Stretching or compressing the mainfield
- Maximum scaling  $\pm 1\%$

How about VLS gratings where pitch is continuously changing?



# Continuous high-precision placement

$$\text{Density (lines/mm)} = a_0 + a_1x + a_2x^2 + a_3x^3$$

## Settings

- Mainfield = 80  $\mu\text{m}$
- Grid = 0.5 nm

Scaling each mainfield goes as follows

Parameter	Specified
Grating Period ( $a_0$ )	800
1st Coefficient ( $a_1$ )	2.81 $\text{mm}^{-1}$
2nd Coefficient ( $a_2$ )	2.2e-3 $\text{mm}^{-2}$
3rd Coefficient ( $a_3$ )	6.4e-6 $\text{mm}^{-3}$

	Pitch (nm)	Nom. Pitch (nm)	Scaling factor
Field 1:	1346.804249	1347	-14532
Step 40 $\mu\text{m}$			
Field 2:	1346.738446	1346.5	17708
Step 40 $\mu\text{m}$			
Field 3:	1346.672648	1346.5	12821
etc...			
#3202:	1152.851544	1153	-12875

## Advantages

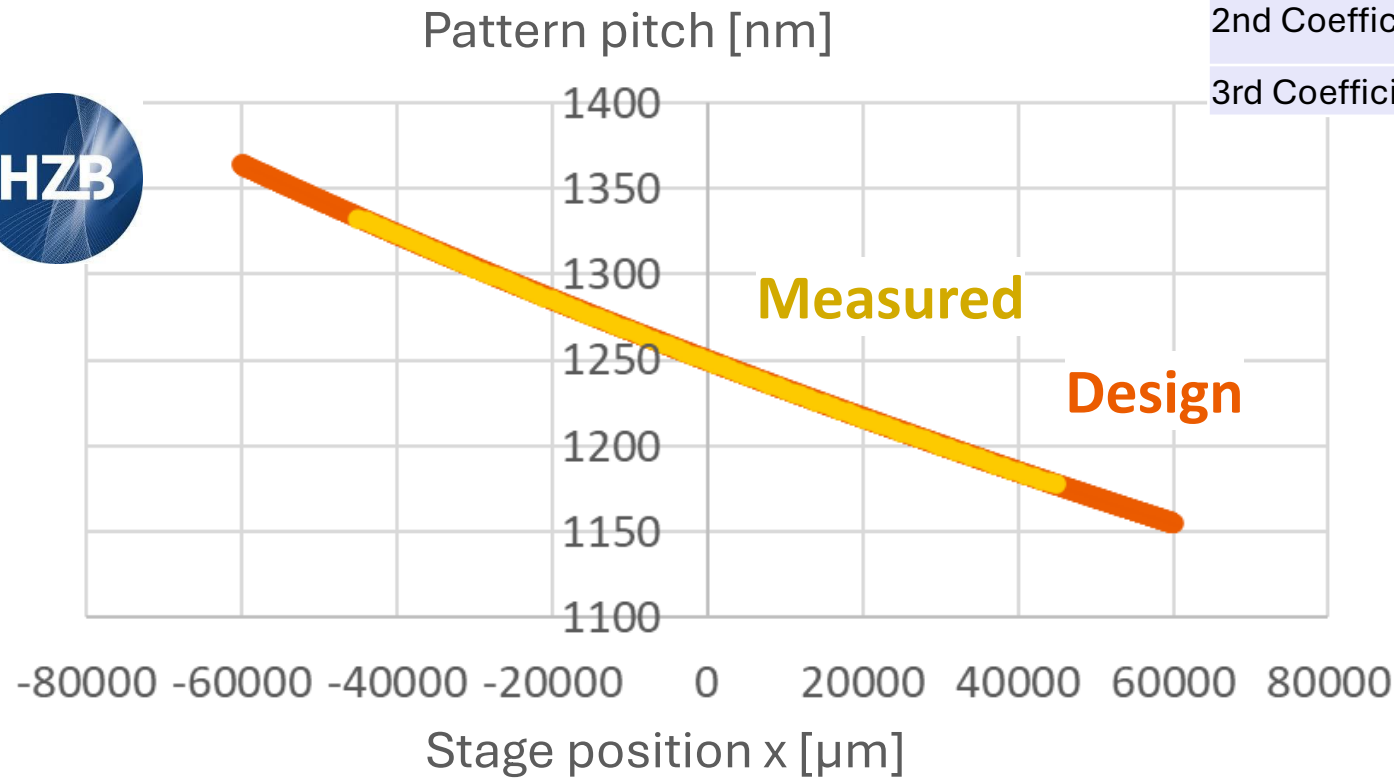
- Multipass averages out the errors at the edges of the mainfields
- Each mainfield writes 60 lines reducing stage movements (time saving)
- Accuracy better than addressing grid

# VLS gratings – early results

Variable line spacing using cont. scaling of the grid

$$\text{Density (lines/mm)} = a_0 + a_1x + a_2x^2 + a_3x^3$$

Parameter
Grating Period ( $a_0$ )
1st Coefficient ( $a_1$ )
2nd Coefficient ( $a_2$ )
3rd Coefficient ( $a_3$ )



Max. residual in pattern pitch  
< 80 picometers

# Latest X-ray results - ESRF

Beamline ID 32 XES spectrometer

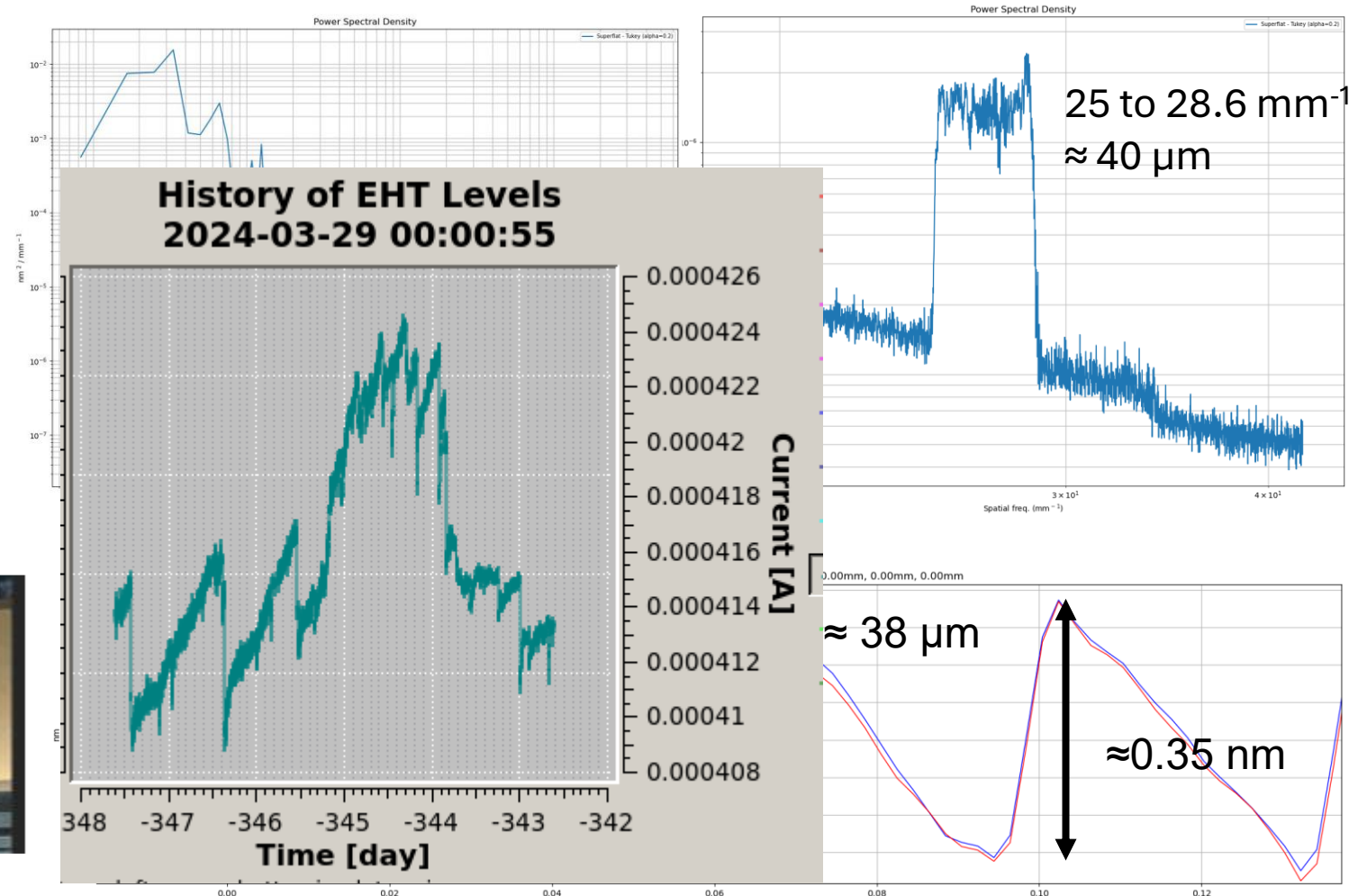
Mainfield settings show up,

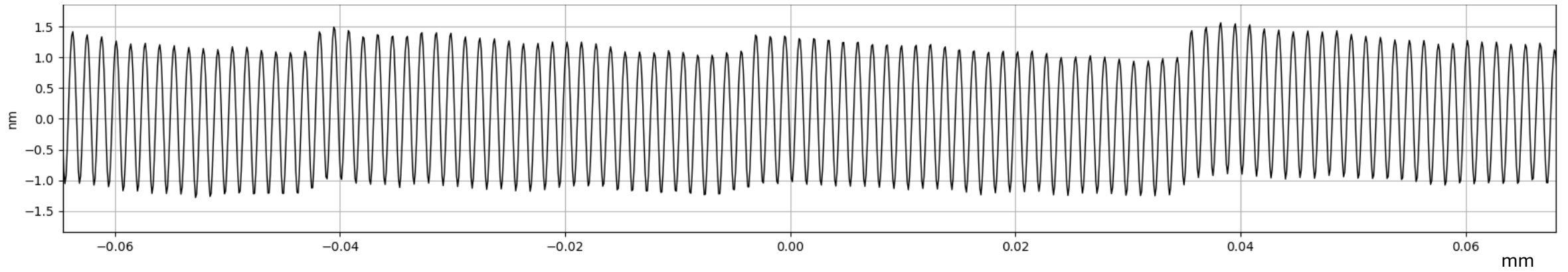
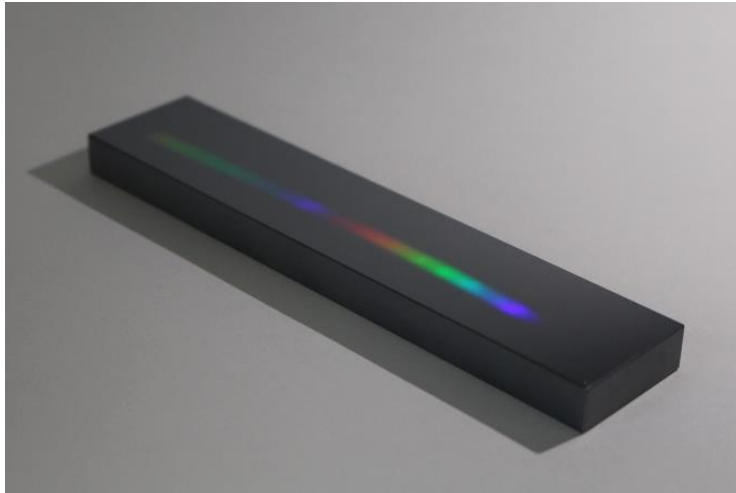
- Mainfield =  $80 \times 860 \mu\text{m}^2$   
MP = 2 so  $\approx 40 \mu\text{m}$

6 Steps visible by naked eye

Period  $\approx 18.6 \text{ mm}$

Amplitude  $\approx 0.2 / 0.3 \text{ nm}$





Effect of curvature?

# Conclusion

- VLS gratings in thick curved Silicon
  - Up to  $140 \times 30 \times 10 \text{ mm}^3$
- Picometer precision pitches
- Tests ongoing at the beamline(s)
- Soon commercially available at XRnanotech
- Through-mask oxidation process for smooth structures

## Future developments:

- New tool in 2025
- Expanding applications to metalenses and photonic devices



# Acknowledgements



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Andrey Sokolov  
Frank Siewert

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Christiaan Zonneville  
Bas Ketelaars  
and the entire team



**Thank you for listening!**



# Conclusion

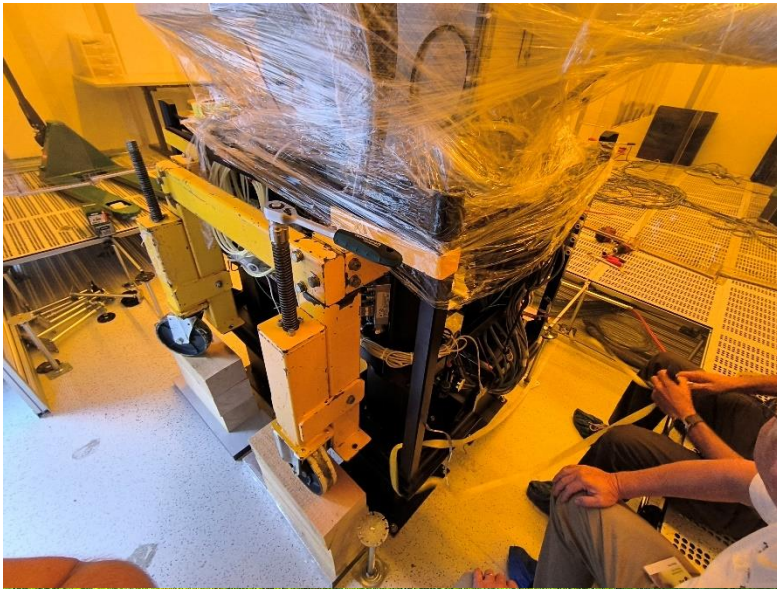
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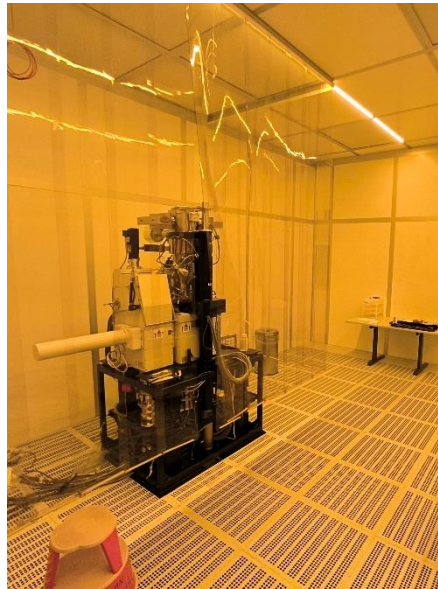
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# E-beam tools - move and acquisition



EMMO cleanroom



Park Innovaare

# Through-mask oxidation for metalenses (preliminary results)

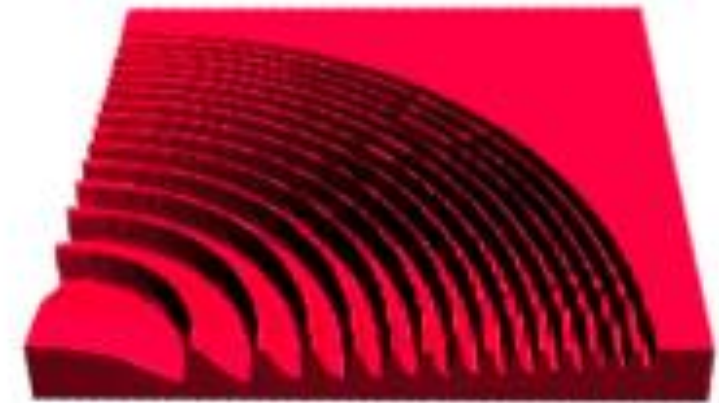
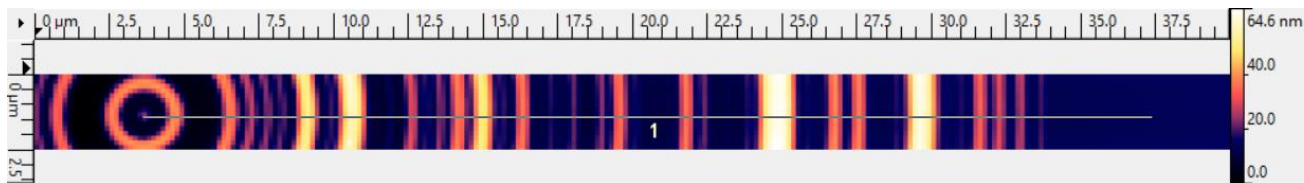
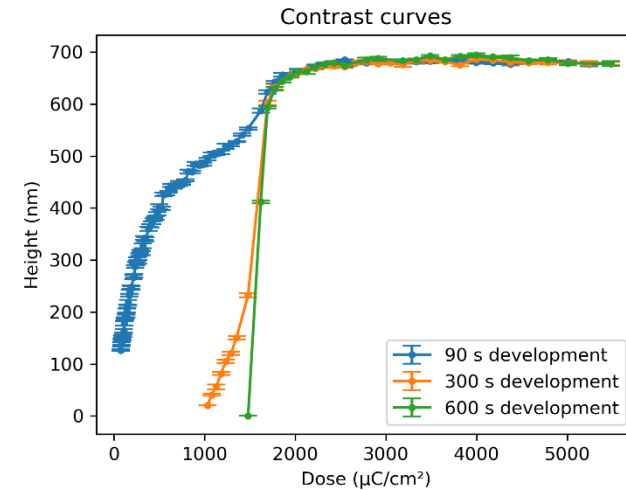
Benchmarking tests (ongoing)

- Smoothly varying structures
- Abrupt changing is possible

Silicon structuring

- Metalenses
- Photonic devices
- Pushing the limits for gratings
  - High line densities, Kinoform, sharper/larger angles

End goal: Stacking metasurfaces with high precision



# Going from one MF to the next (MP=2)



x='	'stageposX='	Polynomial line density	polynomial pitch	'scaleforchirp ='	'patternpitch='	'roundedpatter npitch='	'scaleX='	scaled pitch correction	final pitch (nm)	final line density	MF (um)
0	-60000	742.4984	1346.804249	1.077443399	1346.804249	1347	-14532	-0.19574604	1346.804254	742.4983973	40.406
1	-59959.596	742.534679	1346.738446	1.077390757	1346.738446	1346.5	17708	0.23843822	1346.738438	742.5346835	40.404
2	-59919.194	742.570959	1346.672648	1.077338118	1346.672648	1346.5	12821	0.172634765	1346.672635	742.5709665	40.402
3	-59878.794	742.607241	1346.606854	1.077285483	1346.606854	1346.5	7935	0.106844775	1346.606845	742.6072457	40.4
4	-59838.396	742.643522	1346.541065	1.077232852	1346.541065	1346.5	3049	0.041054785	1346.541055	742.6435284	40.398
5	-59798	742.679805	1346.475282	1.077180225	1346.475282	1346.5	-1835	-0.024708275	1346.475292	742.6797997	40.396
6	-59757.606	742.716089	1346.409503	1.077127602	1346.409503	1346.5	-6720	-0.0904848	1346.409515	742.7160821	40.394
7	-59717.214	742.752374	1346.343728	1.077074983	1346.343728	1346.5	-11605	-0.156261325	1346.343739	742.752368	40.392
8	-59676.824	742.788659	1346.277959	1.077022367	1346.277959	1346.5	-16490	-0.22203785	1346.277962	742.7886574	40.39
9	-59636.436	742.824946	1346.212194	1.076969755	1346.212194	1346	15764	0.21218344	1346.212183	742.8249516	40.388
10	-59596.05	742.861233	1346.146434	1.076917148	1346.146434	1346	10879	0.14643134	1346.146431	742.8612346	40.386
11	-59555.666	742.897521	1346.080679	1.076864543	1346.080679	1346	5994	0.08067924	1346.080679	742.8975212	40.384
12	-59515.284	742.93381	1346.014929	1.076811943	1346.014929	1346	1109	0.01492714	1346.014927	742.9338114	40.382
13	-59474.904	742.9701	1345.949184	1.076759347	1345.949184	1346	-3775	-0.0508115	1345.949189	742.9700976	40.38
14	-59434.526	743.006391	1345.883443	1.076706754	1345.883443	1346	-8659	-0.11655014	1345.88345	743.0063874	40.378
15	-59394.149	743.042684	1345.817706	1.076654164	1345.817706	1346	-13543	-0.18228878	1345.817711	743.0426808	40.377
16	-59353.774	743.078978	1345.751973	1.076601578	1345.751973	1346	-18426	-0.24801396	1345.751986	743.0789703	40.375
17	-59313.401	743.115272	1345.686245	1.076548996	1345.686245	1345.5	13842	0.18624411	1345.686244	743.1152725	40.373
18	-59273.03	743.151567	1345.620522	1.076496418	1345.620522	1345.5	8957	0.120516435	1345.620516	743.1515704	40.371
19	-59232.661	743.187864	1345.554804	1.076443843	1345.554804	1345.5	4073	0.054802215	1345.554802	743.1878645	40.369
20	-59192.294	743.224161	1345.48909	1.076391272	1345.48909	1345.5	-810	-0.01089855	1345.489101	743.2241546	40.367