Zone Plate Development at the APS: Status and Plans

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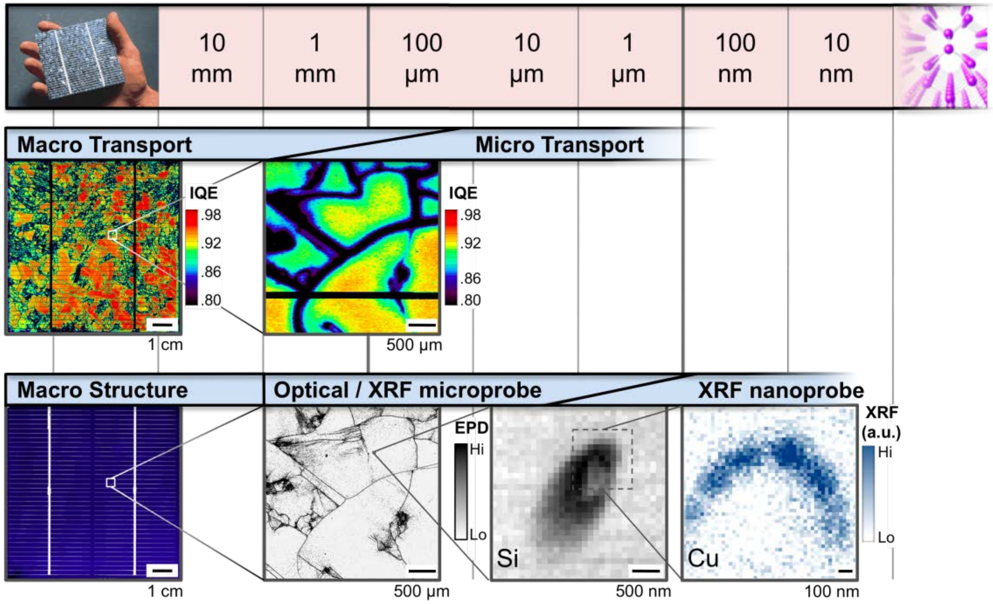
Outline

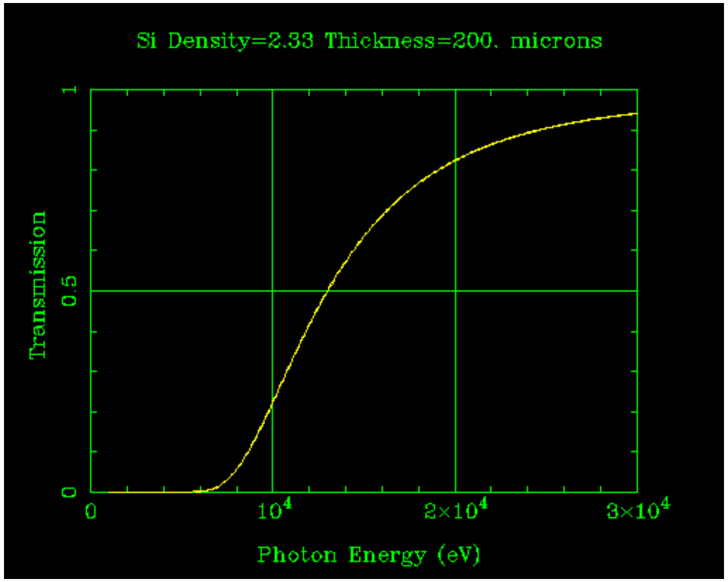
- Increasing Effective Energy and Resolution
- Some Zone Plate Background
- Current Zone Plate Development at Argonne
- Future Plans for Zone Plate Development at Argonne
- Conclusion

Higher X-ray Energy with Finer Resolution

- X-ray fluorescence microprobe and nanoprobe enable multi-length scale studies, whose capabilities only increase with greater range of probe energies.
- In-situ studies provide a better understanding of the system in real time, such as preferred defect reactions of metals during crystal growth and processing .
- Increasing the X-ray energy will allow thicker samples to be imaged, possibly for thick in-situ setups.



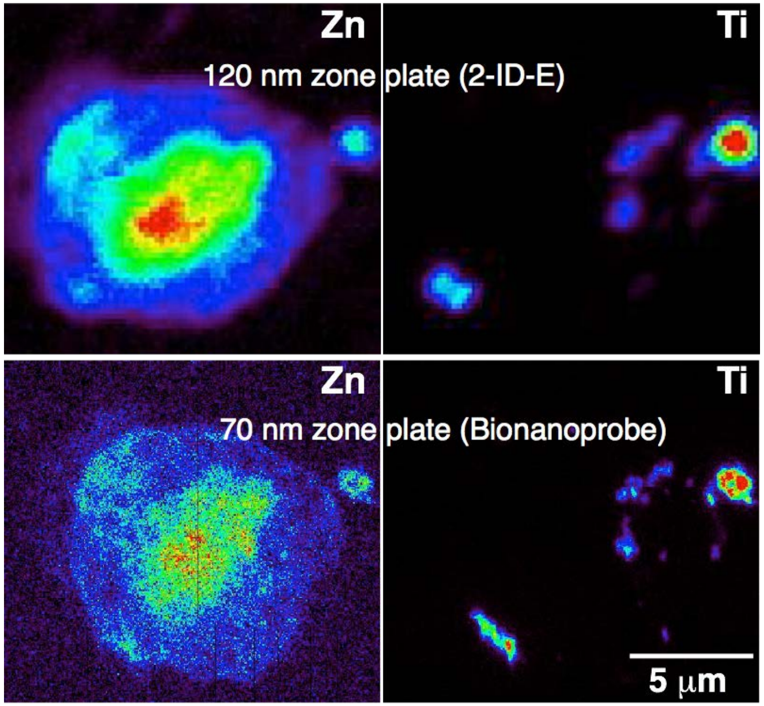
M. I. Bertoni *et al.*, Energy Environ. Sci., 2011, 4, 4252-4257.



http://henke.lbl.gov/optical_constants/filter2.html

Higher X-ray Energy with Finer Resolution

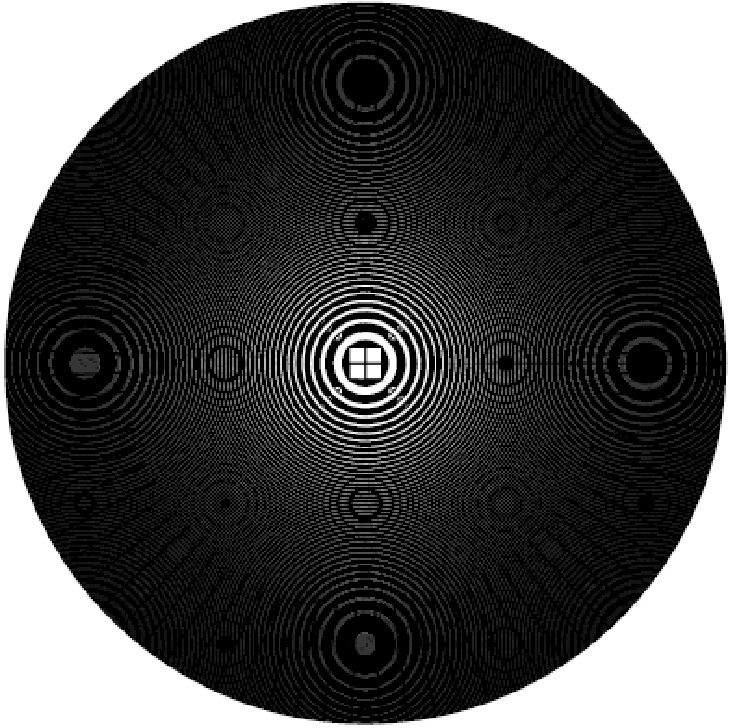
- Higher resolution lets us see more detail in materials

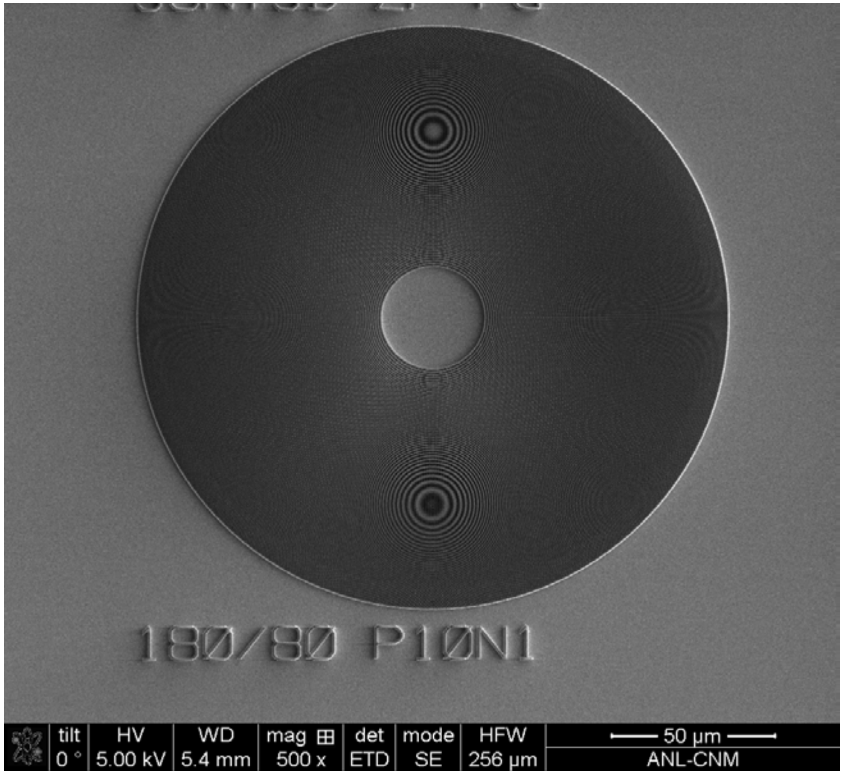


Paraffin-embedded HeLa transfected with TiO2-DNA conjugates (Paunesku, Woloschak *et al.*). Fluorescence maps obtained using two different zone plates. Improved spatial resolution lets one see if these anti-cancer agents are targeting mitochondria.

- The APS Upgrade has a Key Performance Parameter of 20 nm resolution at 25 keV.
- The APS Upgrade *In Situ* Nanoprobe beamline requires 20 nm resolution focusing optics; additional broad benefit to APS.

Zone Plate Background

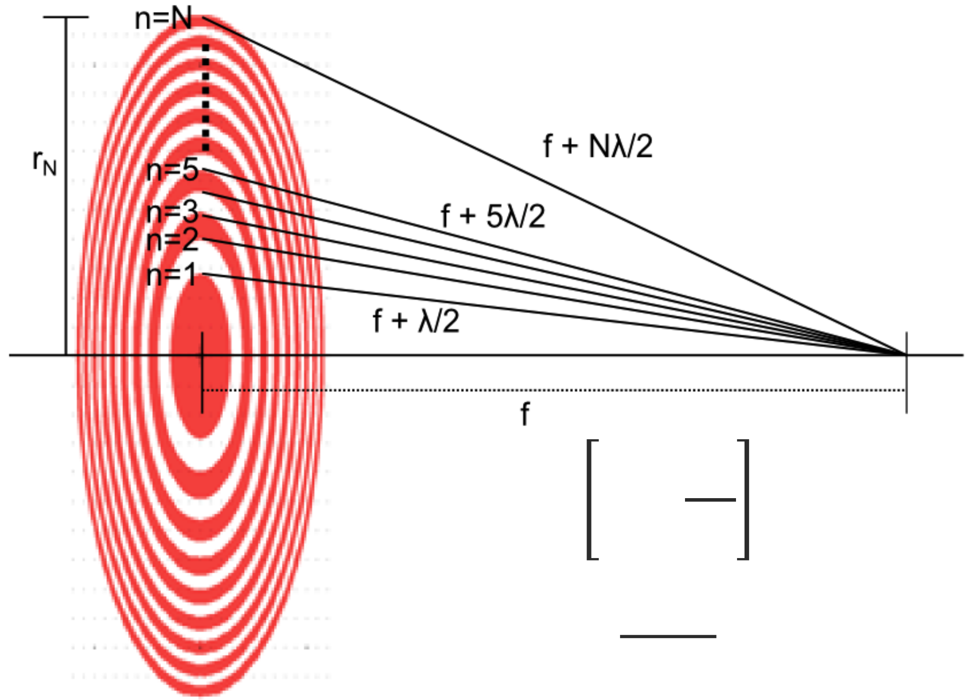




Zone plate pattern generated with custom software detailed in Lu, M. et al. J. Vac. Sci. Technol. B **24** 6 (2006).

Zone Plate: basics

- Diffractive optics with grating spacing radially varied
- Largest diffraction angle is given by outermost (finest) zone width dr_N as $\theta=\lambda/(2dr_N)$
- Thus the Rayleigh resolution is $0.61\ \lambda/(\theta)=1.22dr_{N.}$
  - Transfer function extends to a half period of half a zone width, or $dr_N/2$.
- Zones must be positioned to ~1/3 width over diameter, accurate zone placement and important for stacking.
- Zones must be thick enough along the beam direction to cause sufficient phase shift.

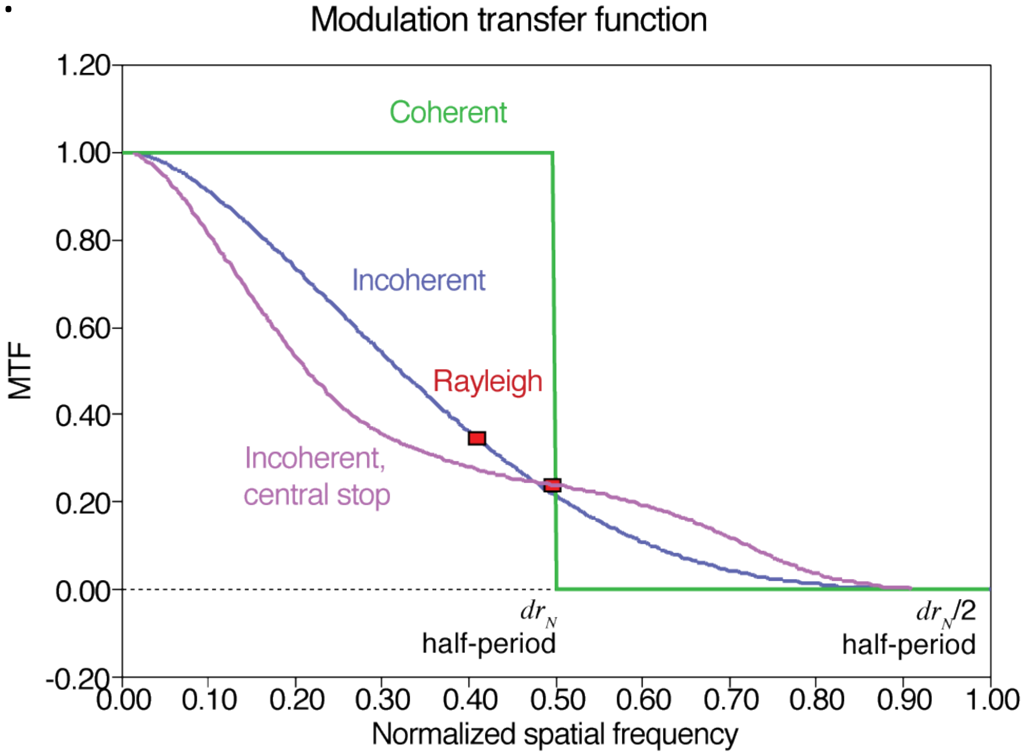


$$d\ dr_N = f\ \lambda$$

Where: diameter d, location of n[th] zone r_n, outermost zone width dr_N, focal length f, wavelength λ

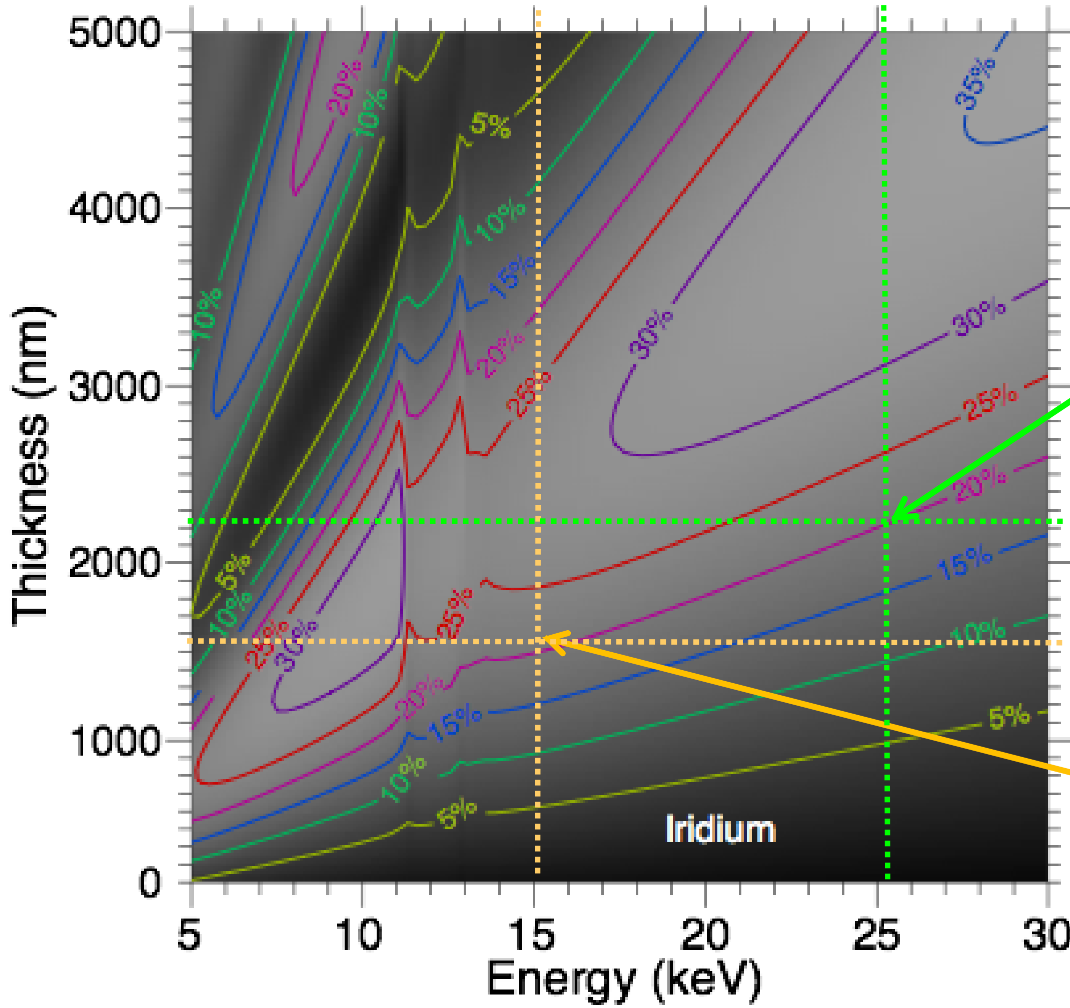
Zone Plate: resolution

- The Rayleigh resolution is equal to $1.22dr_N$.
- Incoherent transfer function reaches zero at a half-period equal to $0.5dr_N$, so a 20 nm zone plate can give optical response down to 10 nm half-period.



Transfer functions and zone plate resolution:
Jacobsen *et al.*, *Ultramicroscopy* **47**, 55 (1992)

Zone Plate: efficiency of a single Iridium zone plate

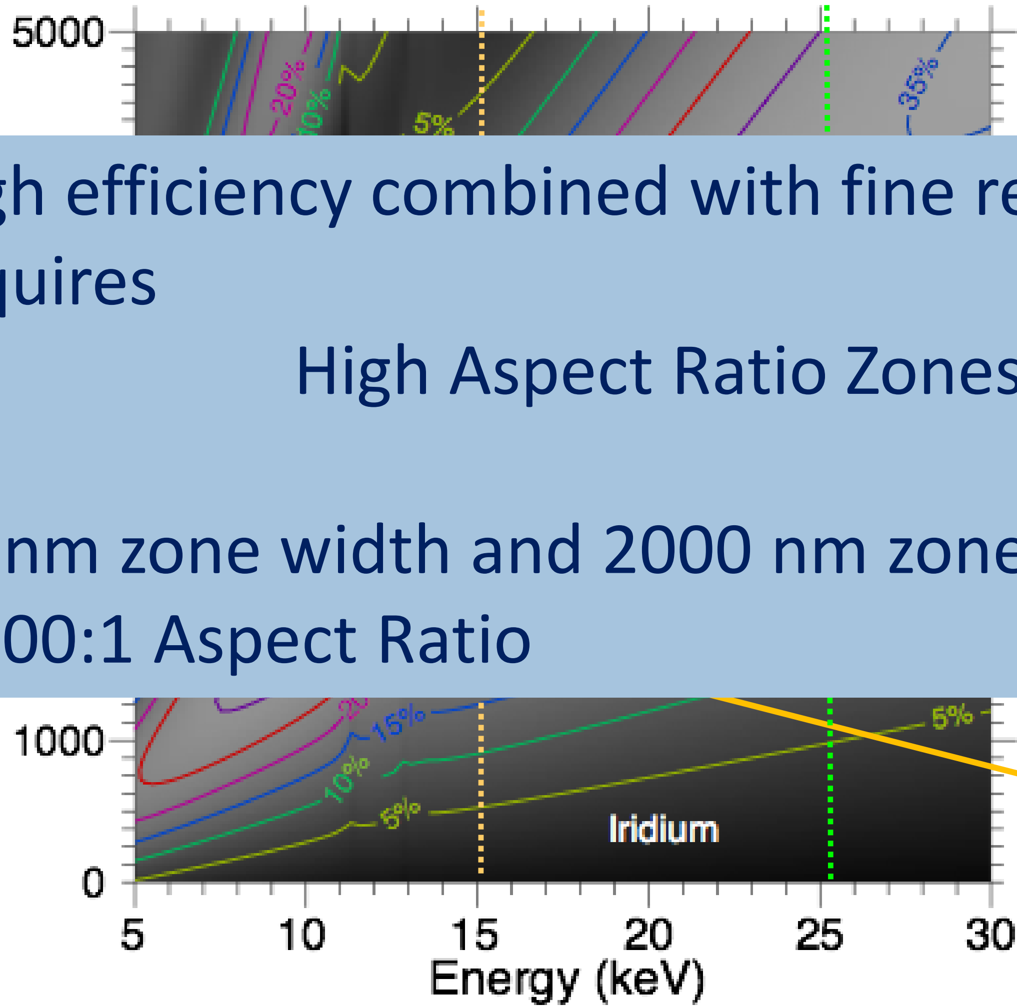


2.2 um Ir yields ~20% efficiency at 25 keV

1.5 um Ir yields ~20% efficiency at 15 keV

Calculation for scalar diffraction efficiency for binary zones [C. Jacobsen, following J. Kirz, *JOSA* **64**, 301, (1974)]

Zone Plate: efficiency of a single Iridium zone plate



High efficiency combined with fine resolution requires

High Aspect Ratio Zones

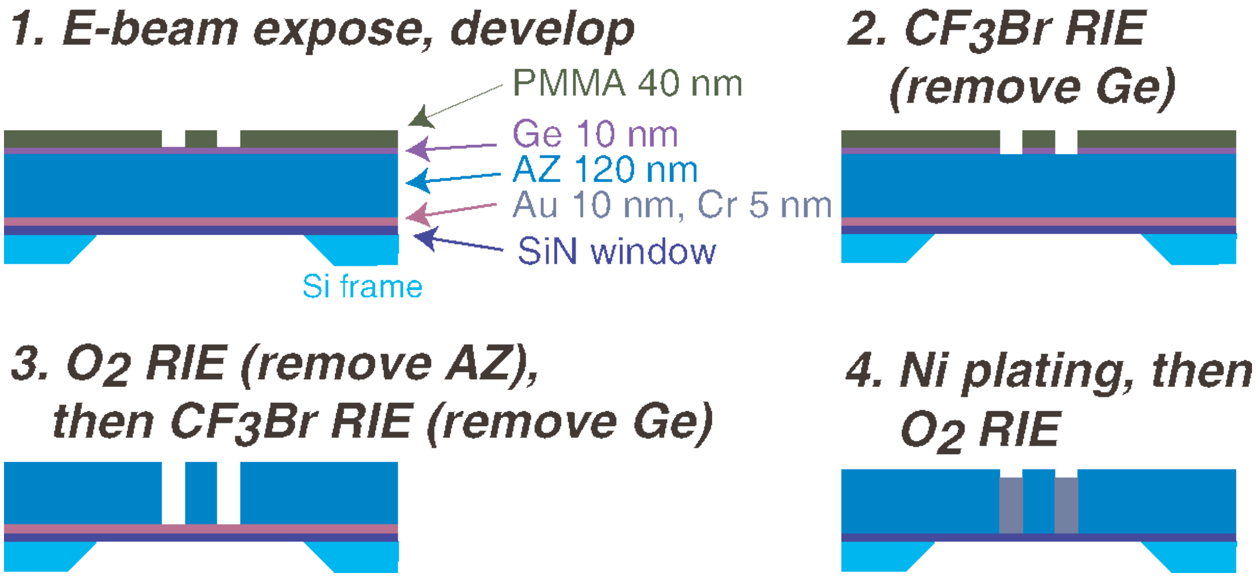
20 nm zone width and 2000 nm zone thickness = 100:1 Aspect Ratio

yields
ciency at

1.5 um Ir yields ~20% efficiency at 15 keV

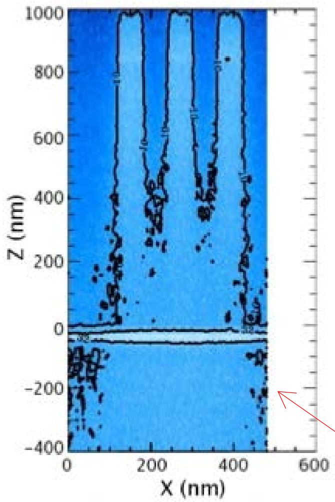
Calculation for scalar diffraction efficiency for binary zones [C. Jacobsen, following J. Kirz, *JOSA* **64**, 301, (1974)]

Zone Plate: example fabrication process

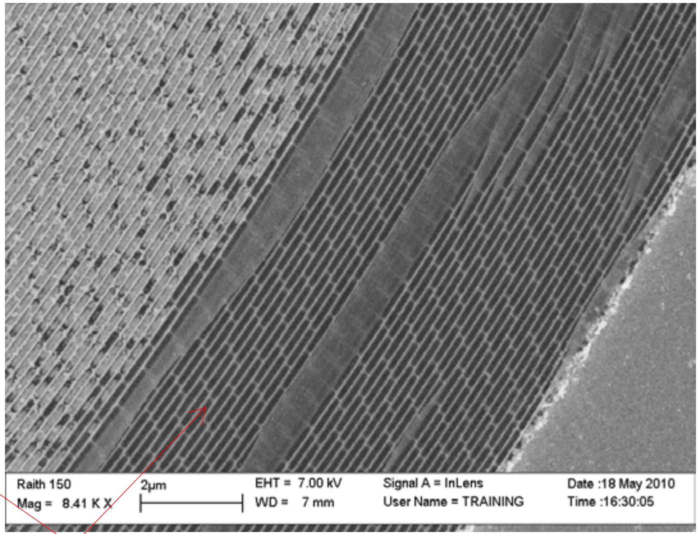


Process taken from Tennant, D., et al. *AIP Conference Proceedings*. Vol. 507. 2000.

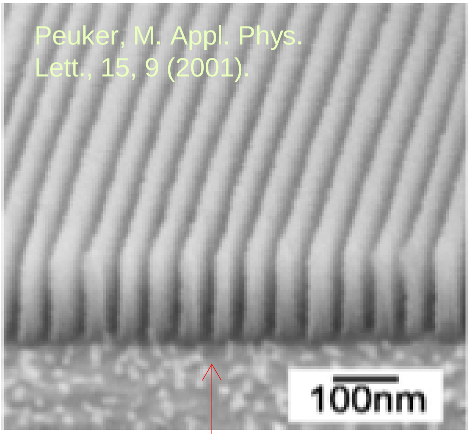
Zone Plate: aspect ratio challenges



M. Lu, et al, Proc. SPIE **7039**, 70390V (2008).

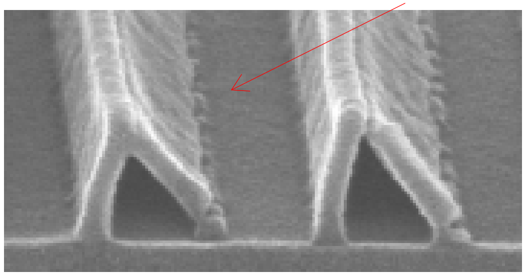


Lithography Limit

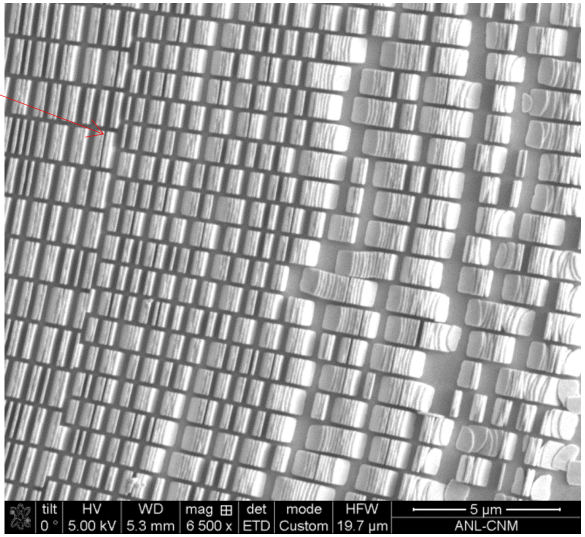


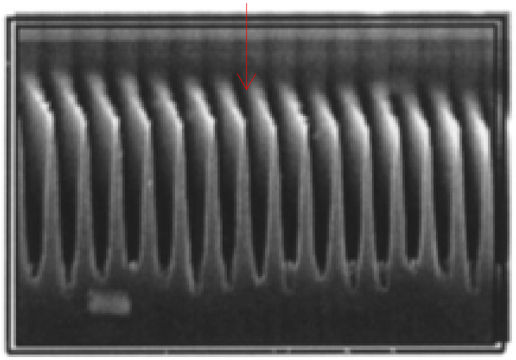
Pattern Transfer Limit

Pattern Collapse



http://ieuvi.org/TWG/Resist/2009/022609/IEUVI_TOK_Iwashita.pdf



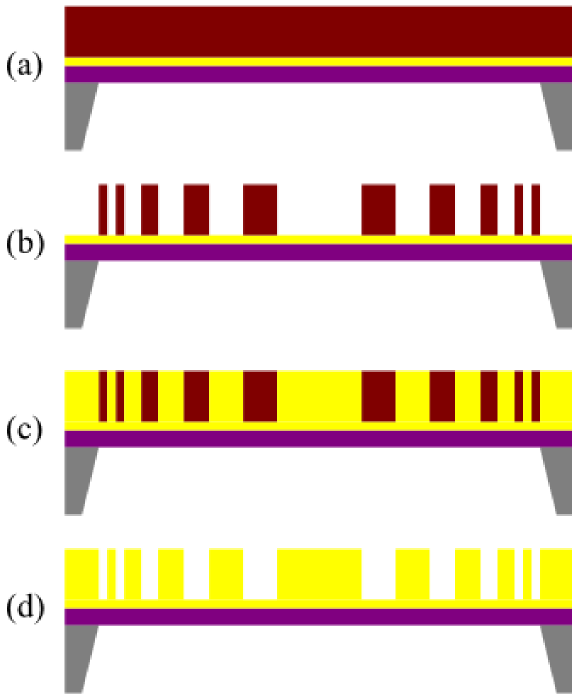


Stein, A. et al., J. Vac. Sci. Technol. B 21(1), (2003).

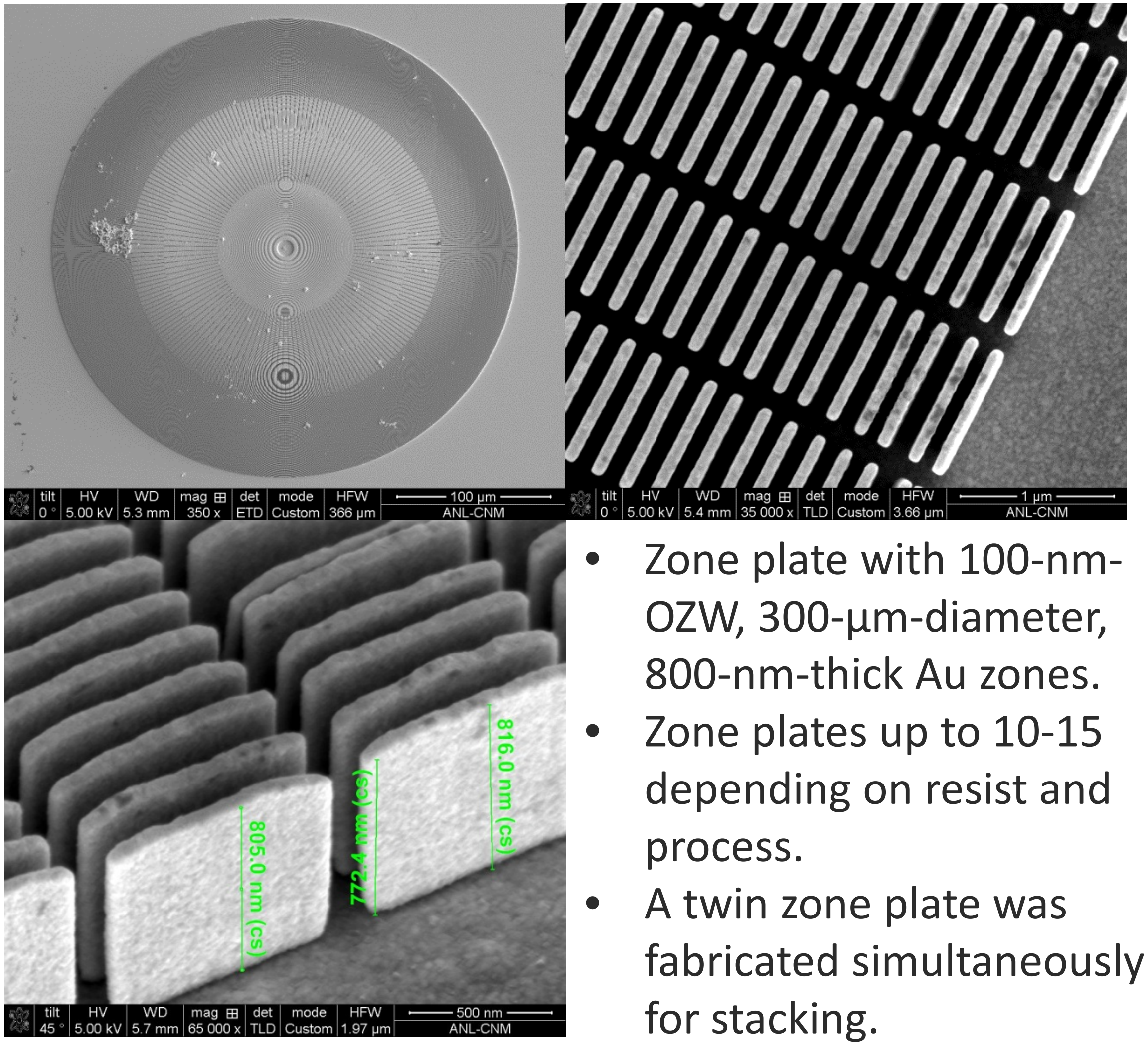
Current Zone Plate Research at Argonne:

What can be done and results from Center for Nanoscale Materials and Advanced Photon Source

Single Layer Zone Plate Fabrication: plating into a resist



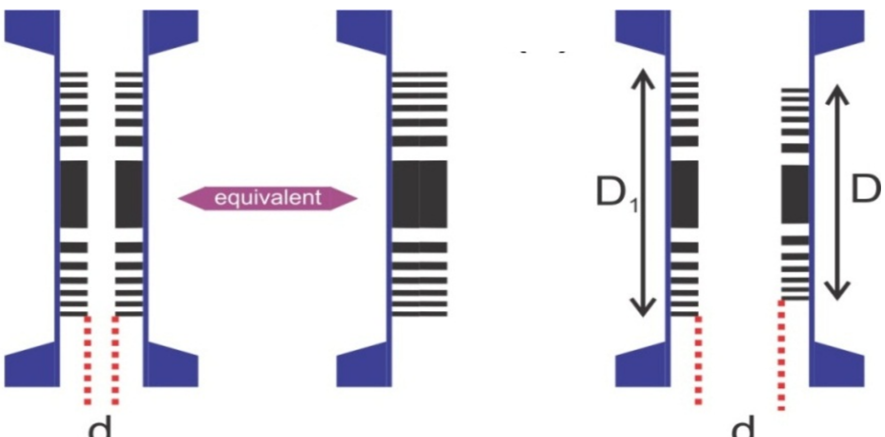
a) Sample preparation
b) E-beam lithography
c) Au electroplating
d) Resist removal



- Zone plate with 100-nm-OZW, 300-µm-diameter, 800-nm-thick Au zones.
- Zone plates up to 10-15 depending on resist and process.
- A twin zone plate was fabricated simultaneously for stacking.

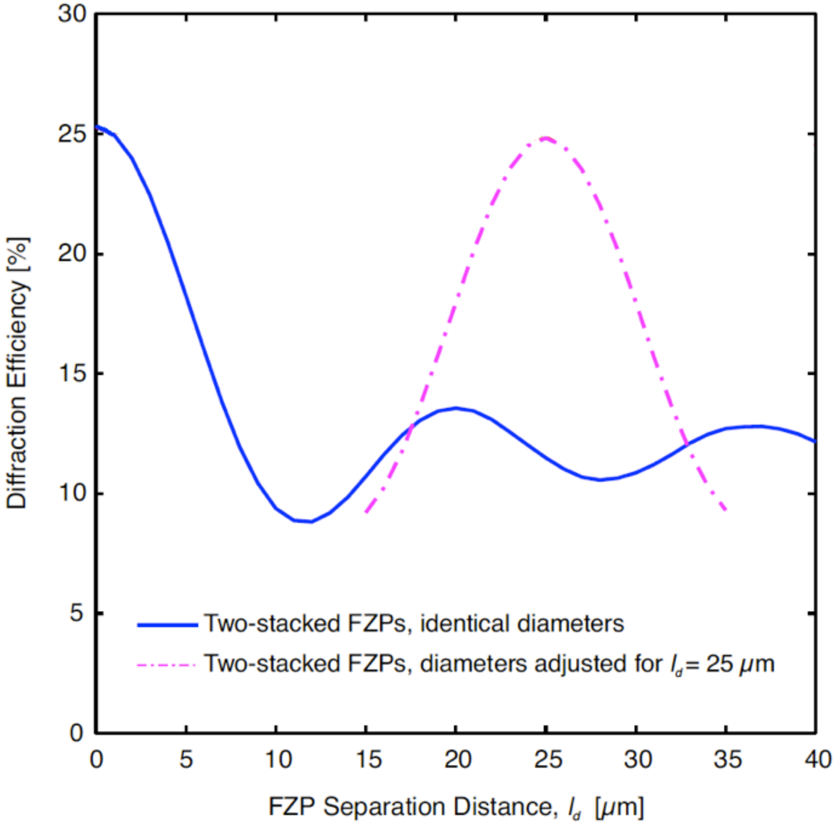
Zone plate stacking: background

- Stacking of two zone plates has been demonstrated [Shastri *et al.*, *Opt. Comm.* **197**, 9 (2001); Xradia].
- 200 µm Si wafer thickness makes it difficult (though possible) to stack more than 2 in close proximity.
- Greater separation: adjust diameter! (J. Vila Comamala *et al.,* J. Synchrotron Radiat. **20**, 3 (2013)).



Near field stacking

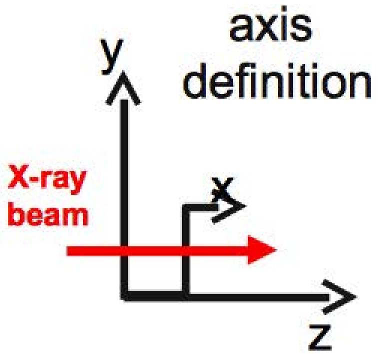
Intermediate field stacking

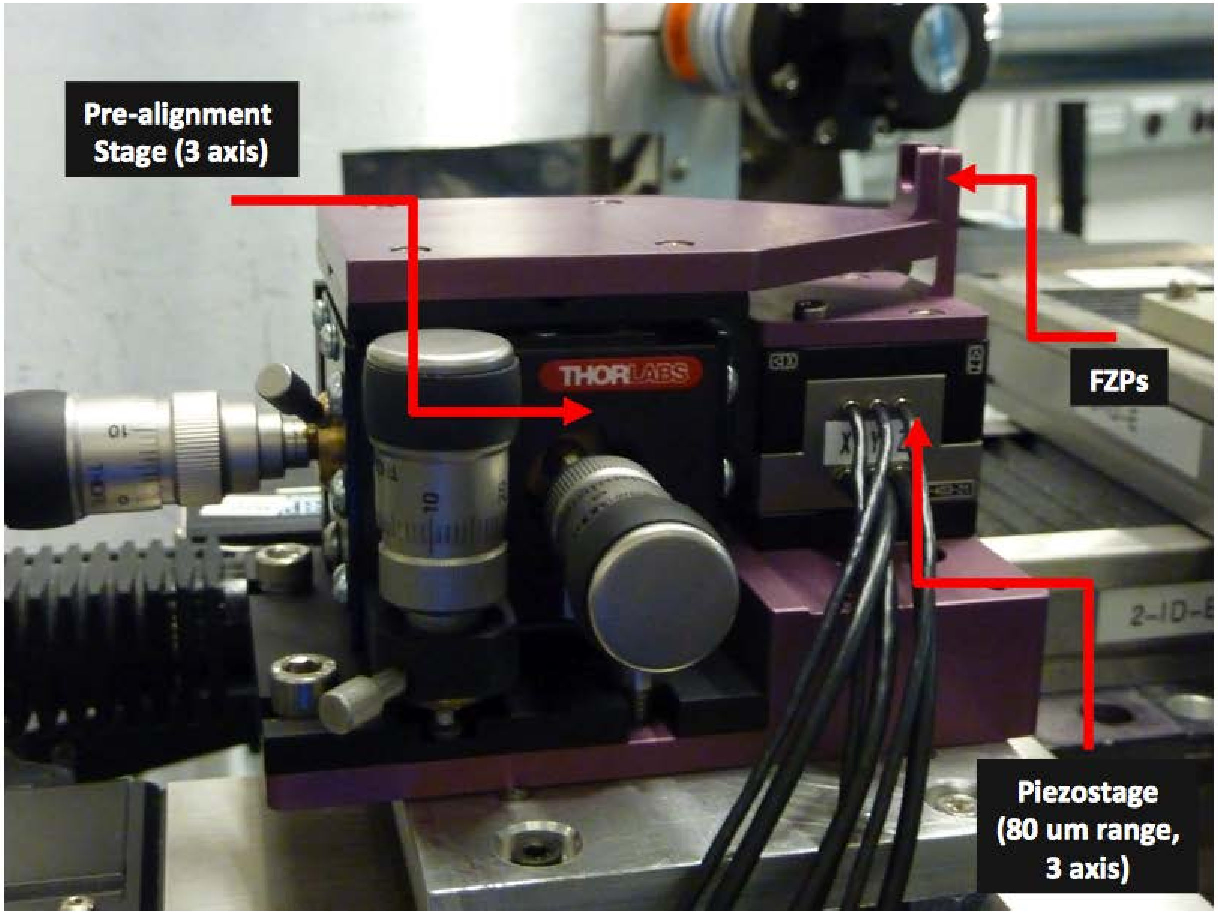


Simulation: two zone plates each with dr_N=20 nm, t=500 nm Au, diameter=45 µm at 6.2 keV. Adjusted zone plate diameter=44.8 µm. J. Vila Comamala *et al.,* J. Synchrotron Radiat. **20**, 3 (2013).

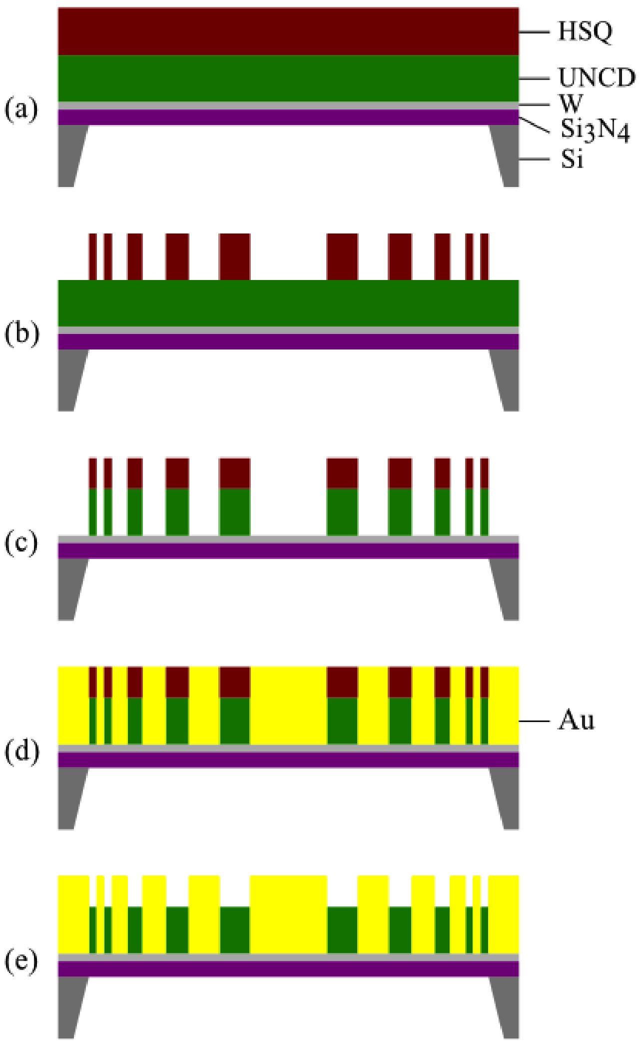
Zone plate stacking: example at APS

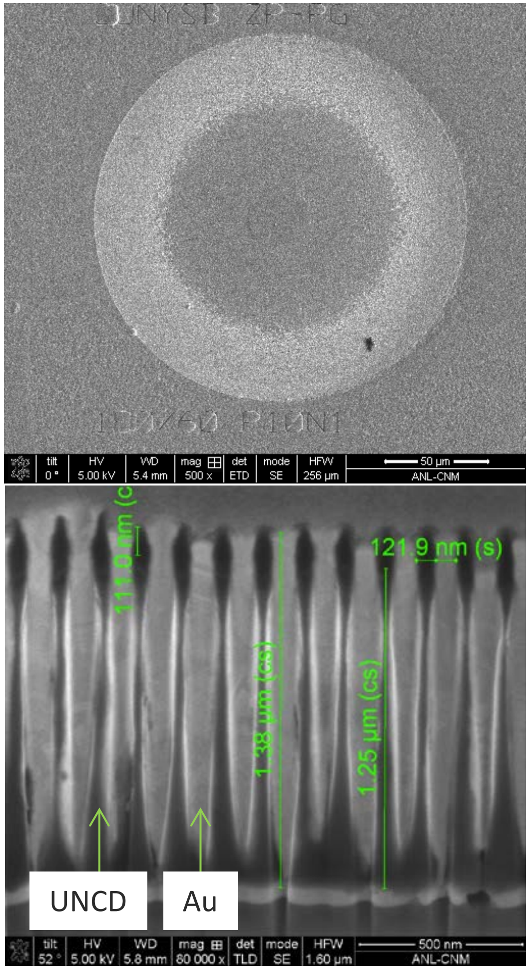
Two Au zone plates fabricated with 400µm diameter, drn: 100nm, thickness per zone plate approx 1.2-1.3µm, dof approx 160µm at 10keV. Experiment done at 2-ID-E by Joan Vila-Comamala and Charlotte Gleber. Preliminary results in preparation for publication.



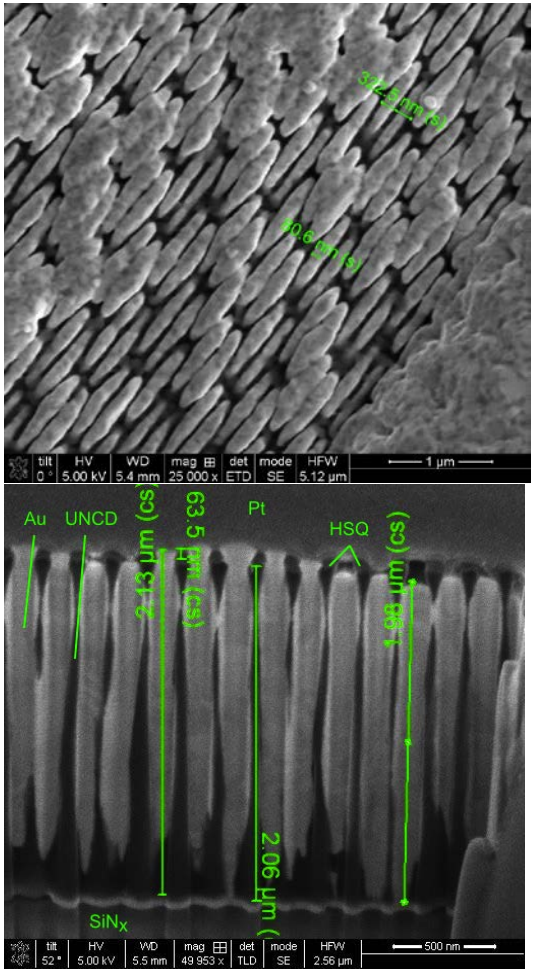


Zone Plates with Ultrananocrystalline Diamond (UNCD)





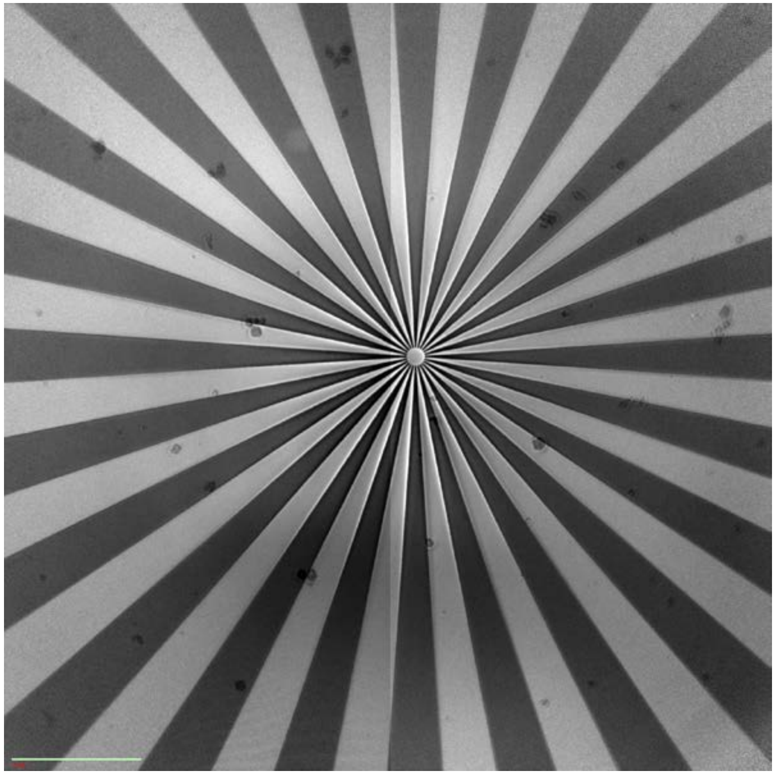
60-nm-OZW, 23 aspect ratio zone plate



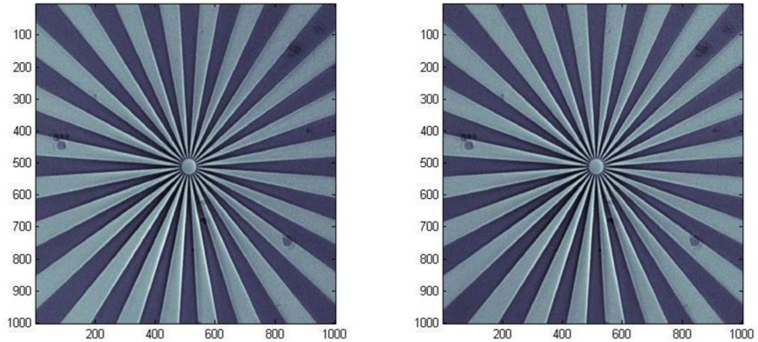
80-nm-OZW, 25 aspect ratio zone plate (submitted)

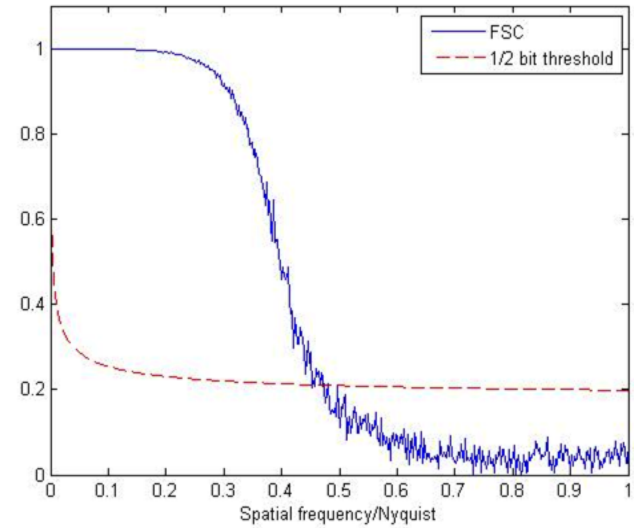
UNCD Zone Plate Characterization at Sec 32 TXM

Zone plate with 60-nm-OZW and 1400-nm-thick tested at TXM



100 nm Siemens star imaged
15% focusing efficiency at 8 keV
(In preparation for publication)

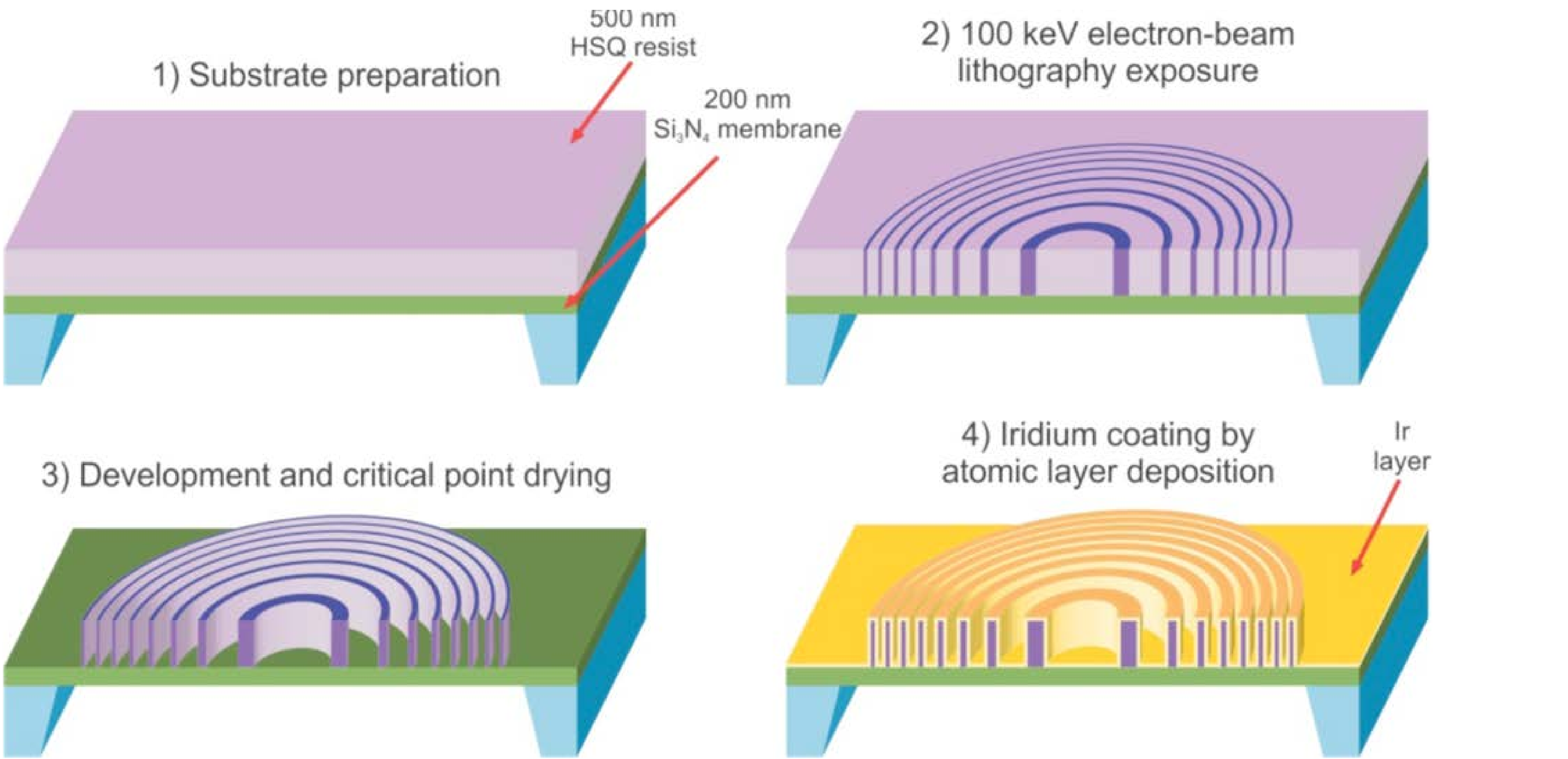




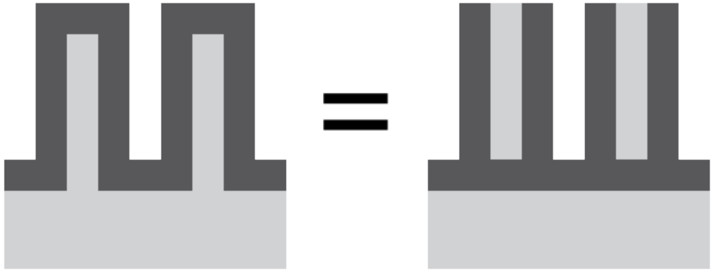
1/2 –bit threshold = 71.3 nm

FRC method: see *M. van Heel* and *M. Schatz, J. Struct. Biol.* **151**, 250-262 (2005)

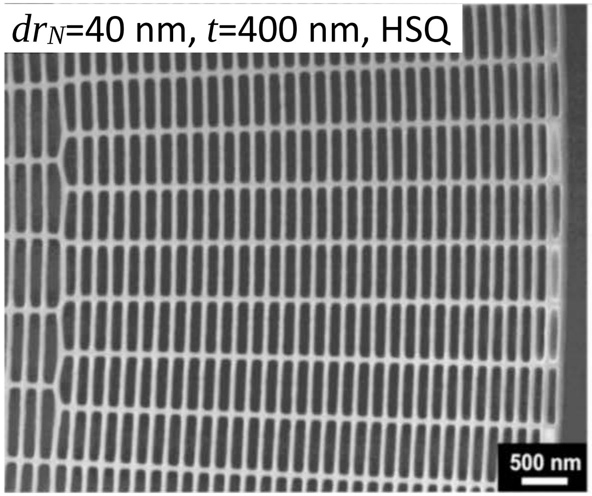
Zone Doubling Zone Plates



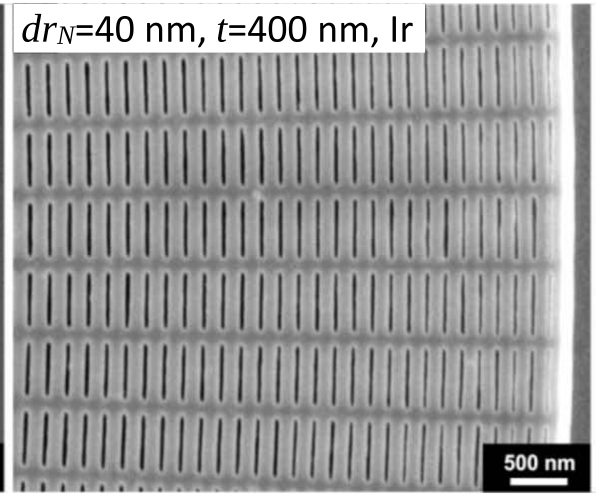
Jefimovs *et al.*, *Phys. Rev. Lett.* **99**, 264801 (2007).



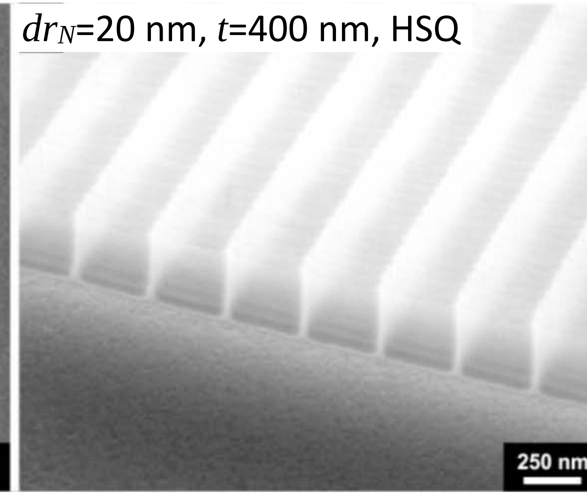
Zone doubling results at Argonne APS/CNM



dr_N=40 nm, t=400 nm, HSQ

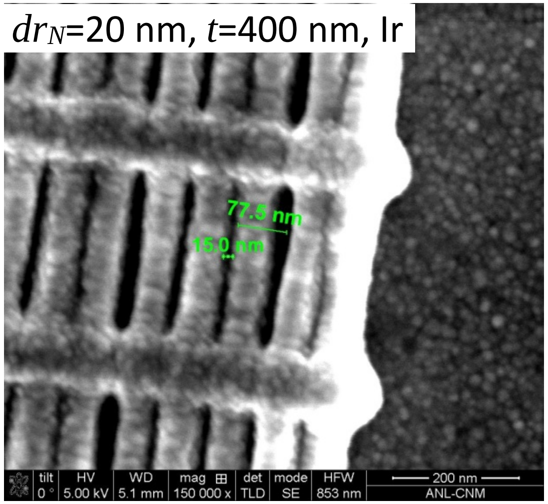


dr_N=40 nm, t=400 nm, Ir

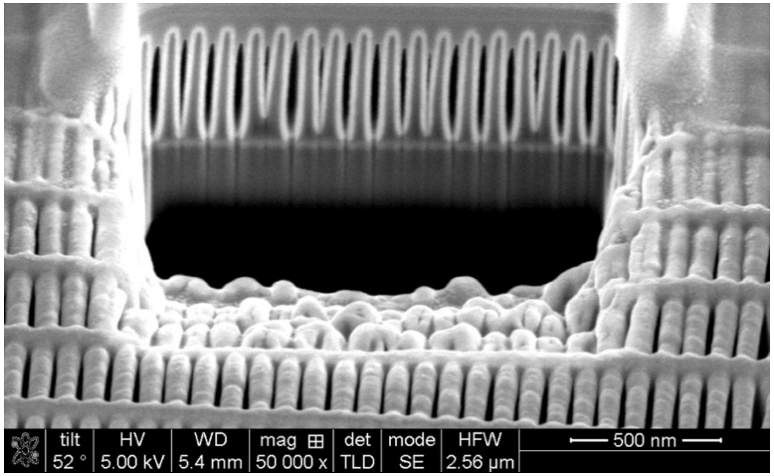


dr_N=20 nm, t=400 nm, HSQ

- Replication of processes pioneered at the Paul Scherrer Institute
- Lithography by Joan Vila-Comamala
- ALD by Energy Systems Division: J. Elam and D. Comstock



dr_N=20 nm, t=400 nm, Ir



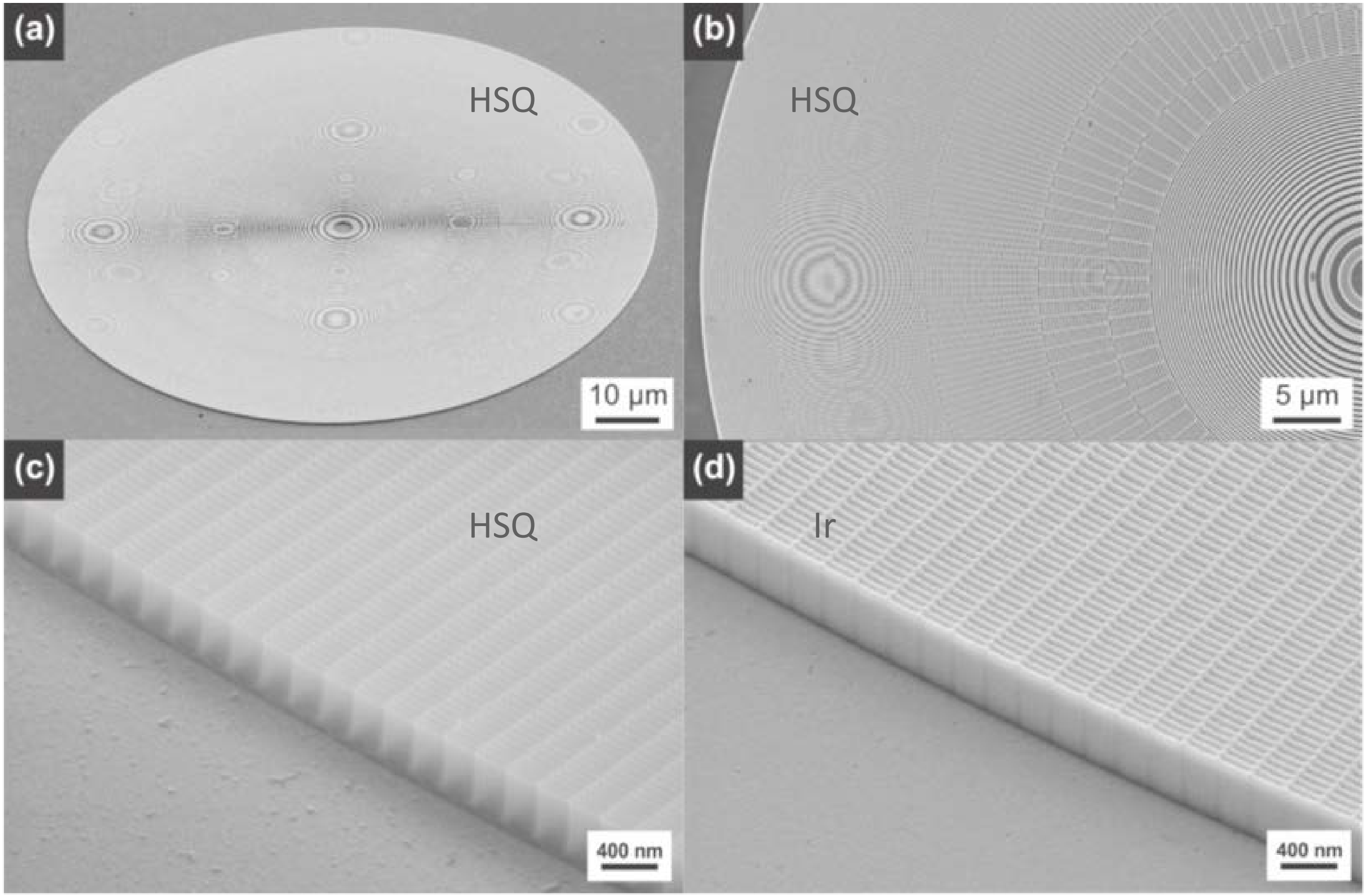
Focused Ion Beam (FIB) cutaway of 20 nm doubled Ir zones

Plans for Zone Plate Development:

In process or soon to begin

Increasing Aspect Ratio of Zone Doubling

Have room to improve zone doubling technique at Argonne, from 20 aspect ratio up to 27-30 possible.



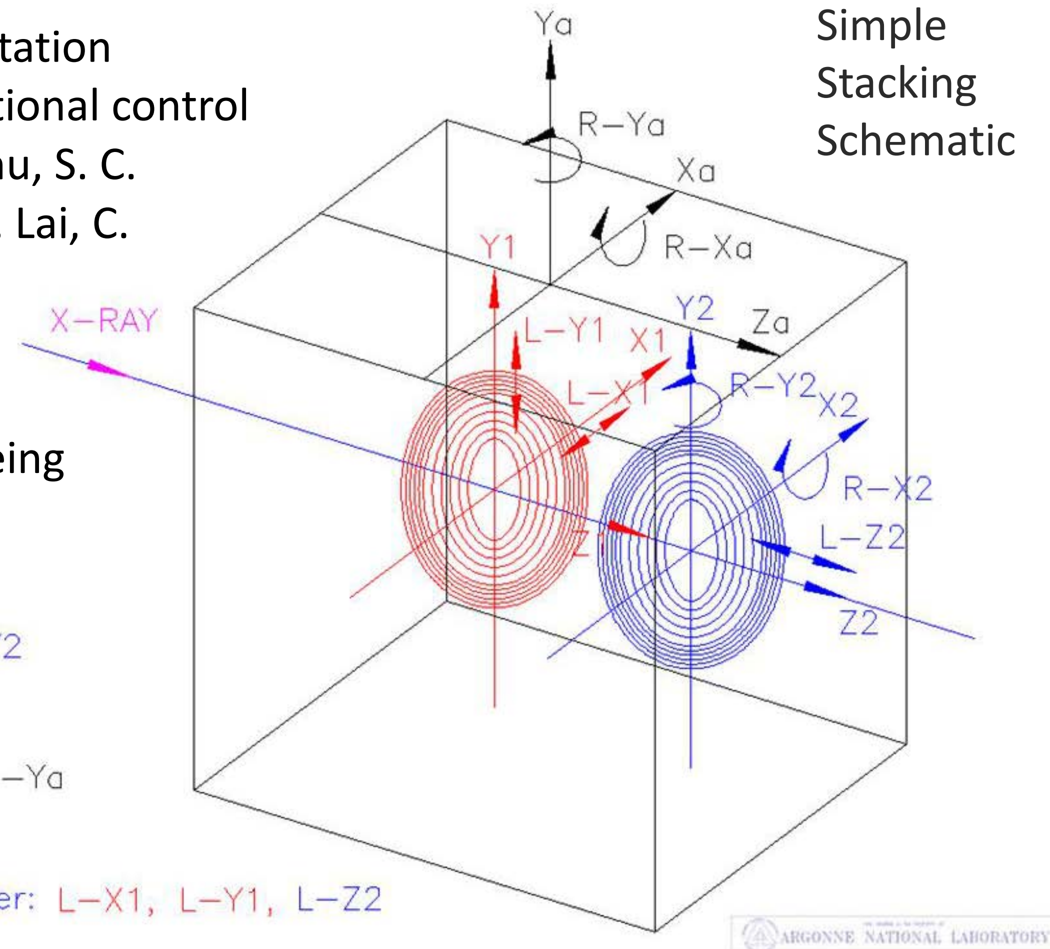
dr_N=20 nm, t=550 nm Ir on HSQ resist (C. David, J. Vila Comamala *et al.,* unpublished).

Process explained in Vila-Comamala, Joan, et al., *Nanotechnology* 21.28 (2010).

Next Generation Zone Plate Stacking System

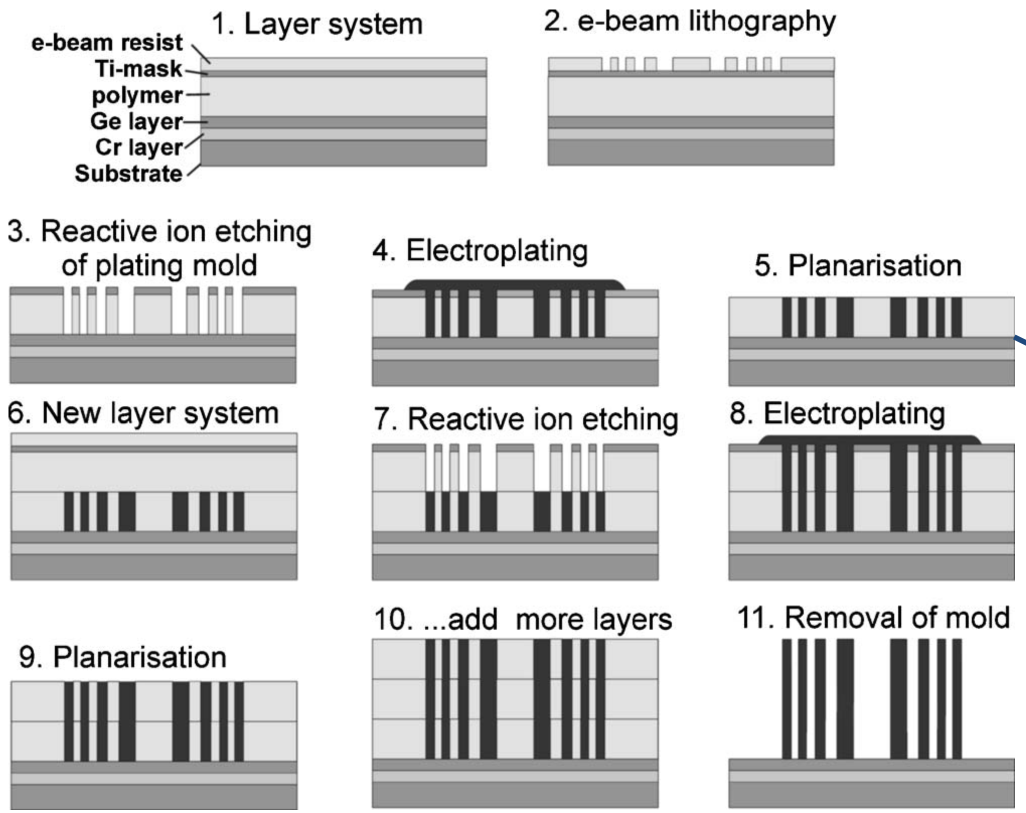
- Stacking system shown before had limited degrees of freedom
- Develop new stacking setup with rotation control as well as improving translational control
- Design and operation by J. Liu, D. Shu, S. C. Gleber, J. Vila-Comamala, S. Vogt, B. Lai, C. Roehrig, and M. Cummings
- For both contact and intermediate-field stacked zone plates
- Intermediate stacked zone plates being developed by K. Li

Simple Stacking Schematic



Multi-layer/Bragg zone plates

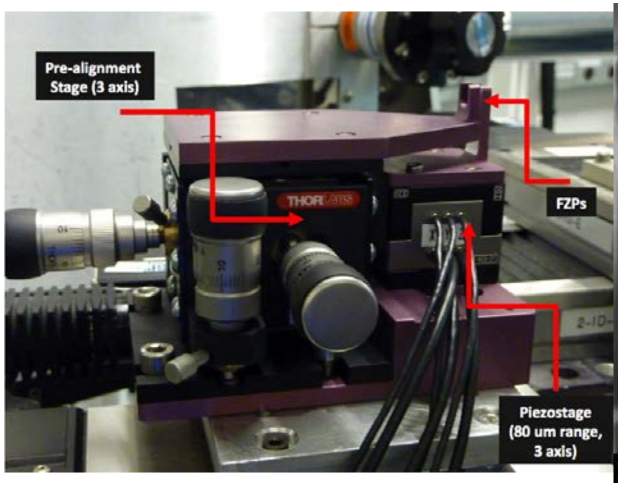
- Can do higher aspect ratio with sequential layers [Rehbein *et al.*, *J. Vac. Sci. Tech. B* **25**, 1789 (2007)]
- Planarization tests have been done, but much more work required.

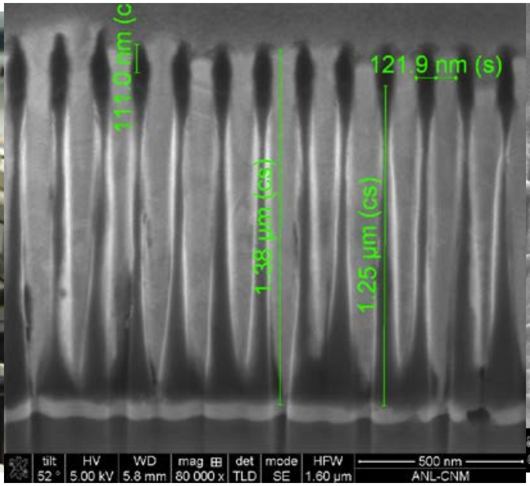


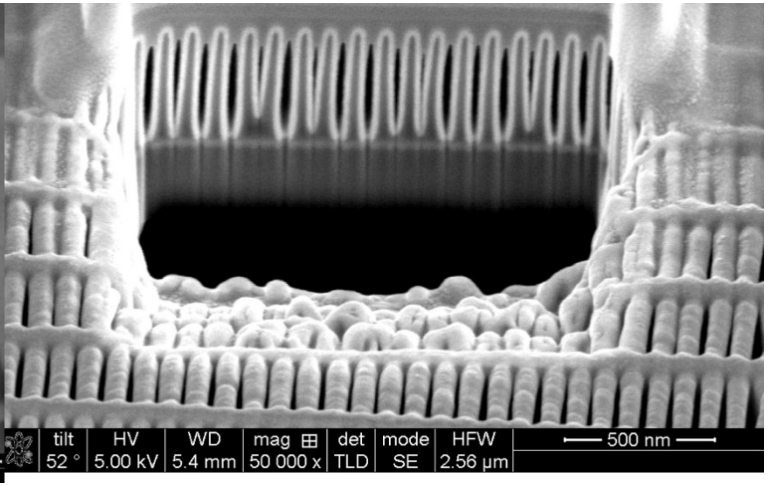
Logitech Orbis CMP (at CNM)

Conclusion

- Several techniques for ~20 aspect ratio zone plates developed at APS and CNM including
  - An example of a zone plate stacking system tested at APS
  - Zone plates fabricated with UNCD
  - Zone doubled zone plates with HSQ and Ir
- Plans introduced to further increase aspect ratio towards the goal of 20 nm resolution at 25 keV
  - Increasing aspect ratio of zone doubled method
  - Next generation zone plate stacking system
  - Multi-layer zone plates







Acknowledgements

Other Collaborators
Ralu Divan[1]
Anirudha V. Sumant[1]
Daniel Rosenmann[1]
Liliana Stan[1]
Suzanne Miller[1]
Francesco De Carlo[2]
Ming Lu[3]
Steve Wang[4]
Derrick Mancini[1,2,5]
Carlo Segre[5]

I would like to thank the organizers of the Optics Work shop for giving me the opportunity to speak today.

I would like to acknowledge Alexandra Joshi-Imre from Center for Nanoscale Materials. I would also like to acknowledge Advanced Diamond Technologies for the UNCD growth.

[1]Center for Nanoscale Materials at Argonne National Laboratory
[2]Advanced Photon Source at Argonne National Laboratory
[3]Center for Functional Nanomaterials at Brookhaven National Laboratory
[4]Illinois Tool Works
[5]Illinois Institute of Technology

Use of the Center for Nanoscale Materials and the Advanced Photon Soure was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357

Thank you for your attention

Questions?