X-ray Based Metrology for Nanostructure Characterization and Its Applications to Semiconductor Industry

吳文立 (Wen-li Wu)

Fellow Emeritus
National Institute of Standards and Technology

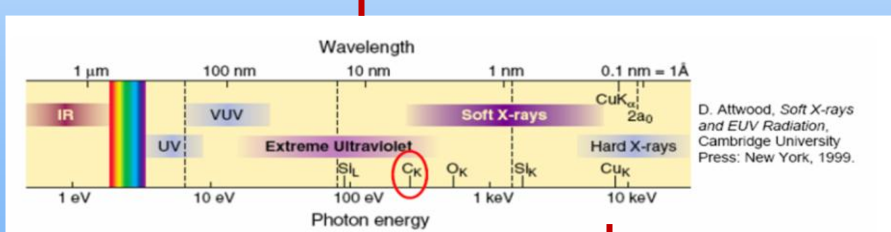
Advisor, CMS
Industrial Technology Research Institute

1

CMS-MML 30th Anniversary 5/3/2017

X-ray wavelength

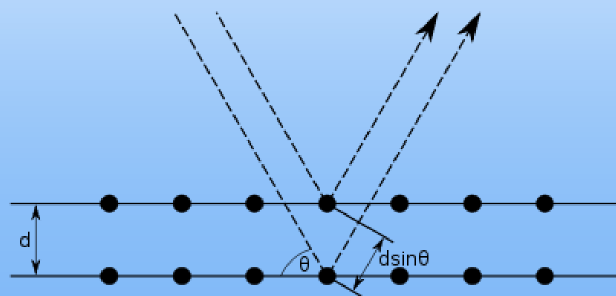
For a feature size of 5nm and below



To penetrate silicon wafer for TRANSMMISION measurements

2

Bragg's law: $2d \sin \theta = n\lambda$, $\sin \theta \leq 1$ i.e. $d \geq n\lambda/2$



3

ABSTRACT

X-ray diffraction has been used over one hundred years to exploit the beauty of symmetry in crystalline materials including minerals, inorganic substances, proteins and many others. **X-ray remains as the workhorse for crystallography** where the aim is to determine the position of the constituting atoms and molecules inside a crystal with a precision less than one Angstroms, a length scale comparable to the probing X-ray used. Nowadays with the progress in nanotechnology the feature size in many man made structures approaches rapidly to a few nanometers; an obvious example is IC chip where its feature size already reaches below 10 nanometers. For these feature sizes X-ray diffraction becomes a method of choice for quantitative measurements due to its short wavelength and its penetration power. Please note that there is an important difference between the traditional X-ray method used in crystallography and the X-ray applications for nanostructure; the aim of the latter is often **to determine the form factor of the constituent elements instead of their location or spatial arrangements**. In this talk **X-ray measurements in different modes including 1. transmission, 2. glazing incident and 3. reflection modes** will be discussed in terms of their merits and shortcomings. In addition, results from X-ray at different wavelengths will be provided to demonstrate how to enhance the scattering contrast between materials with different chemical structures.

OUTLINE

1. Transmission small angle X-ray scattering (tSAXS)
2. Grazing incident small angle X-ray scattering (GISAXS)
3. X-ray reflectivity (XRR)
4. CMS efforts to address/circumvent difficulty caused by the insufficient brilliance of current laboratory based X-ray sources

OUTLINE

1. **Transmission small angle X-ray scattering (tSAXS)**
2. Grazing incident small angle X-ray scattering (GISAXS)
3. X-ray reflectivity (XRR)
4. CMS efforts to address/circumvent difficulty caused by the insufficient brilliance in current laboratory based X-ray sources

Acknowledgements:

tSAXS team

Chengqing Wang (NIST now at KLA-Tencor)
Ronald Jones (NIST)
Tengju Hu (NIST now at DuPont in Shanghai)
Dan Sunday (NIST)
R. Joseph Kline (NIST)

Qinghuang Lin (IBM)
Kwang-woo Choi (Intel *retired*)
George Thompson (Intel)
James S. Clarke (Intel)
Steven J. Weigand (APS in Argonne Nat'l Lab)
Denis T. Keane (APS in Argonne Nat'l Lab)
Gila Stein (Univ. Houston)
Joy Y. Cheng (IBM, *TSMC*)

Benjamin Bunday (Sematech)

NIST / Intel CRADA 1893
NIST Office of Microelectronics Program

Introduction

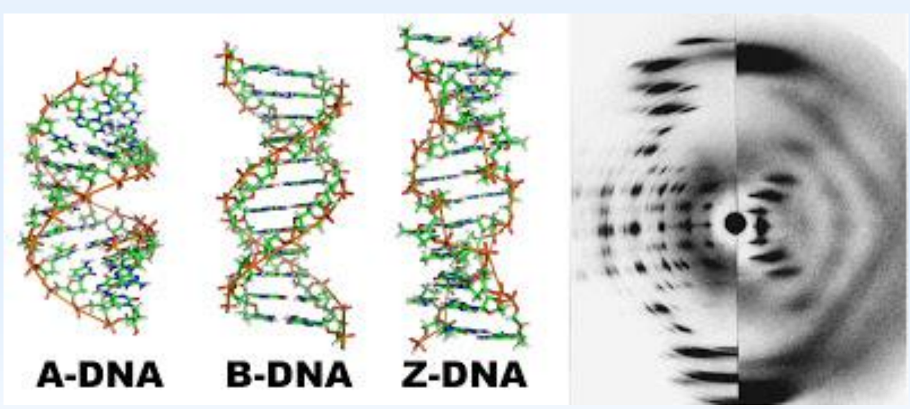
Transmission small angle X-ray scattering (tSAXS) is a technique to characterize nanostructures quantitatively and was pioneered in 2000 by Wu. Its main **advantages** over optical techniques and SEM are:
1. Short wavelength ~ 1 Å or less, hence, higher intrinsic resolution compared to any optical methods
2. High penetration power at this short wavelength, hence, applicable to buried structures, deep holes and structures with high aspect ratio.
3. Weak reaction with most materials, hence, non-invasive and the experimental data can be analyzed with a simple Fourier inversion.

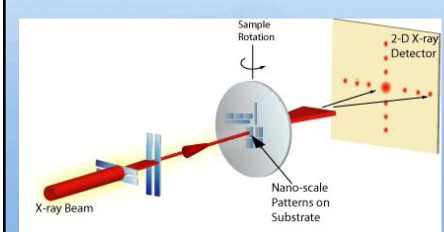
The main **drawback** is its long measurement time due to the lack of a laboratory based high brilliance X-ray source. Synchrotron X-ray source is not an option for daily measurements by industries. To address this drawback efforts have been launched with Wei-En Fu's team at CMS.

X-rays were discovered by Wilhelm Conrad Röntgen in 1895, just as the studies of crystal symmetry were being concluded. Von Laue's pioneer work using X-rays on a copper sulfate crystal and record its diffraction pattern on a photographic plate (Nobel Price in Physics 1914) marks the beginning of using X-ray diffraction to explore the beauty of symmetry of crystal lattices, this includes the work by Rosalind Franklin on the double-helix structure of DNA in 1950's.

There is a basic difference between the X-ray diffraction and tSAXS; the focus of former is the lattice structure whereas the form factor of the constituting object is the focus of tSAXS. For semiconductor industries the constituting object can be a via, a photoresist line or a FinFET. An important goal of tSAXS is to determine the structure details of these objects.

I(**q**) ~ L(**q**) F(**q**) where I(q) is the X-ray scattering intensity at a scattering vector **q**. The lattice factor L(**q**) is the main focus of crystallography or traditional X-ray diffraction whereas F(**q**) is the target of tSAXS.





Transmission SAXS
- Silicon transparent for E > 13 keV
- Non-destructive / No sample prep
- Beam spot size (40x40) μm

Measure "3-D" and Buried patterns
- Contact holes
- Multilevel interconnects

High Precision for sub-45 nm
- Sub-nm precision in pitch and linewidth
- Pattern **Cross Section**

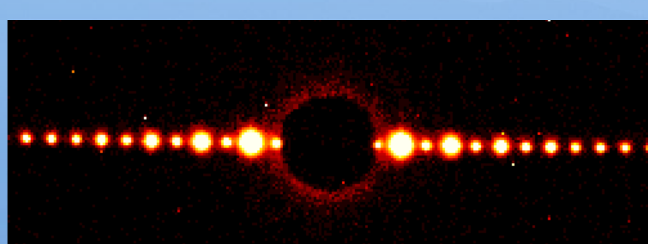
Current State of The Art of tSAXS

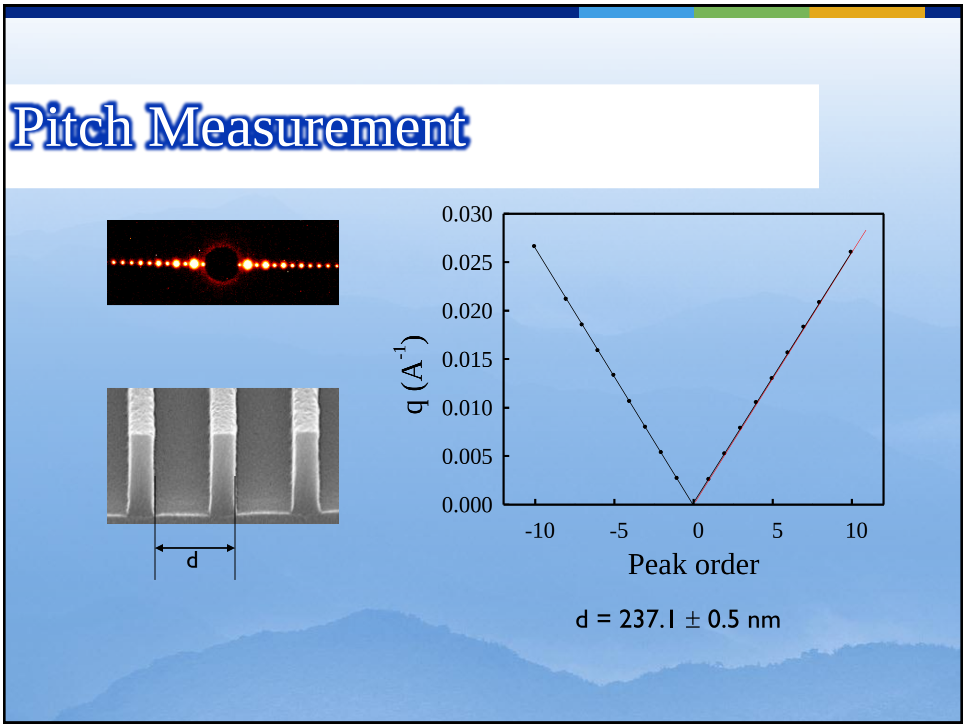
- Since 2005 tSAXS (called CD-SAXS then) has been listed in **International Technology Roadmap for Semiconductors [1]** as a potential solution to many future metrology needs.
- Excillum, Lyncean, Sigray and others have been working on new X-ray sources.
- KLA-Tencor and Applied Materials have also started their efforts on X-ray metrology.
- The current consensus is that the brilliance of Today's laboratory based X-ray source is still inadequate for in-line applications; at CMS there are two ongoing efforts to address this brilliance issue.

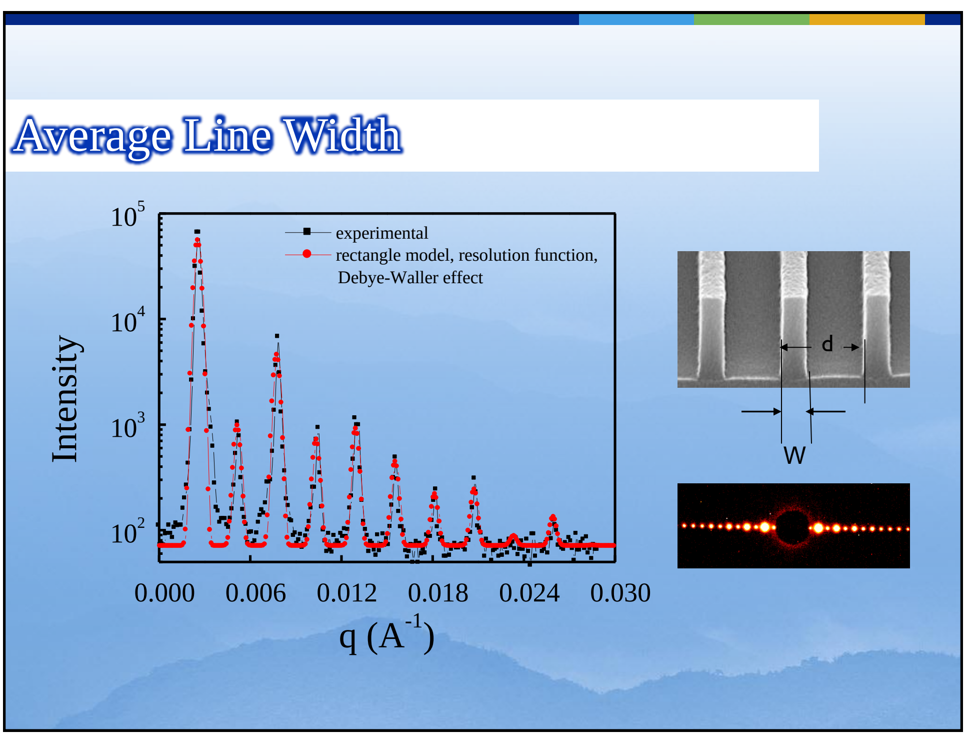
1. The **International Technology Roadmap for Semiconductors** is a set of documents produced by a group of semiconductor industry experts. These experts are representative of the sponsoring organizations which include the Semiconductor Industry Associations of the US, Europe, Japan, South Korea and Taiwan.
The documents represent best opinion on the directions of research into the following areas of technology, including time-lines up to about 15 years into the future.

Normal Incidence Scattering:

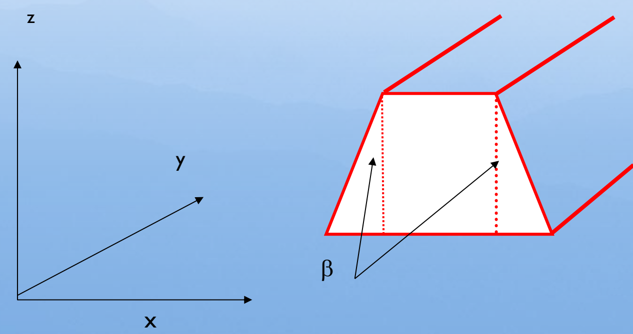
•Pitch & its modulation which occurs often in double or quadruple patterning
•Average line width

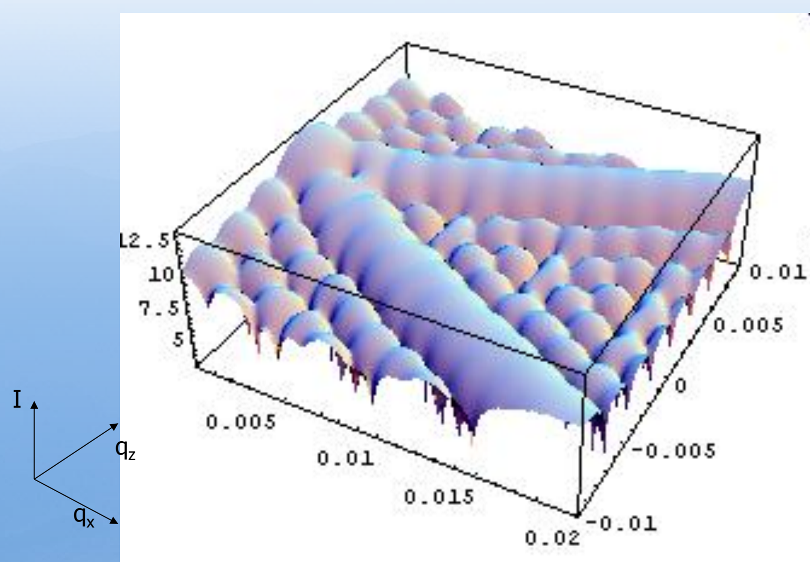


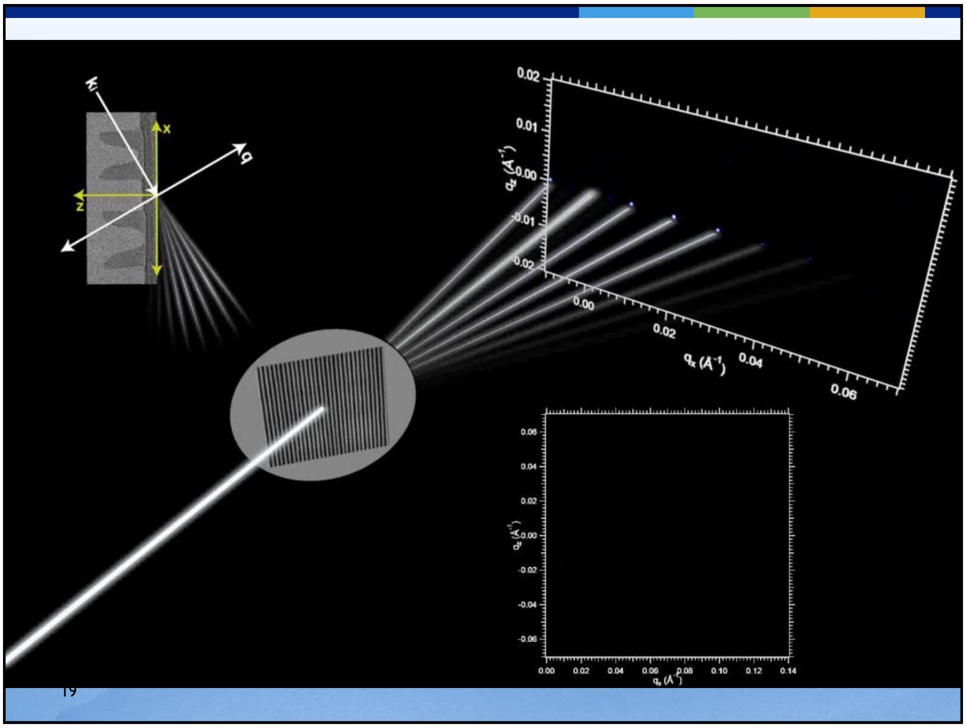

Pitch Measurement
d
0.030
0.025
0.020
0.015
0.010
0.005
0.000
q (A-1)
-10
-5
0
5
10
Peak order
d = 237.1 ± 0.5 nm

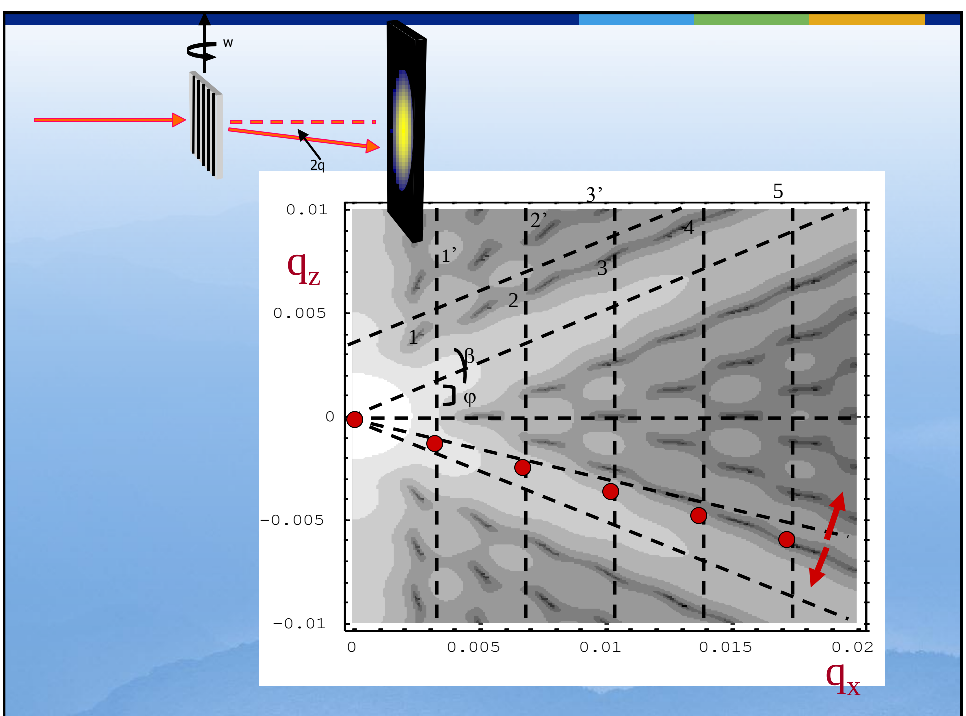

Average Line Width
10^5
10^4
10^3
10^2
Intensity
experimental
rectangle model, resolution function, Debye-Waller effect
0.000
0.006
0.012
0.018
0.024
0.030
q (A-1)
d
W

Sidewall Angle : *Start with a Trapezoid cross section*



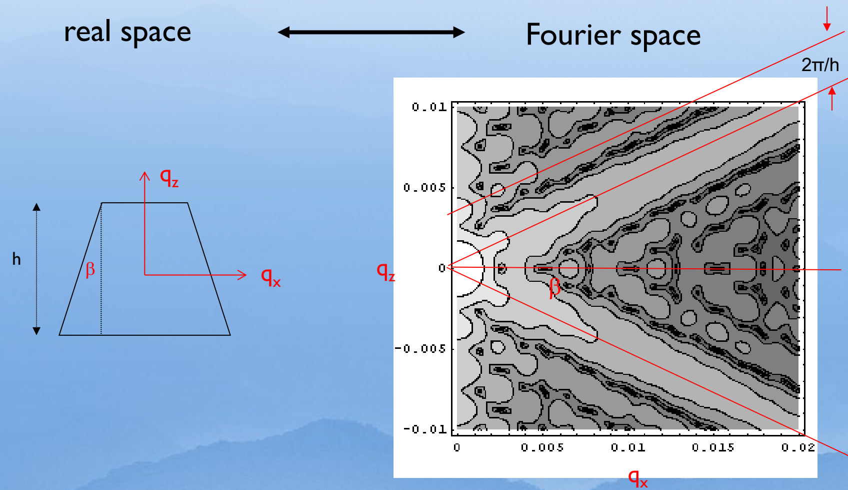


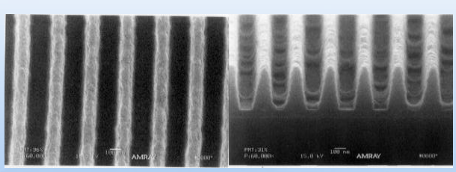

k
x
q
z
0.02
0.01
0.00
-0.01
-0.02
0.00
0.02
0.04
0.06
19

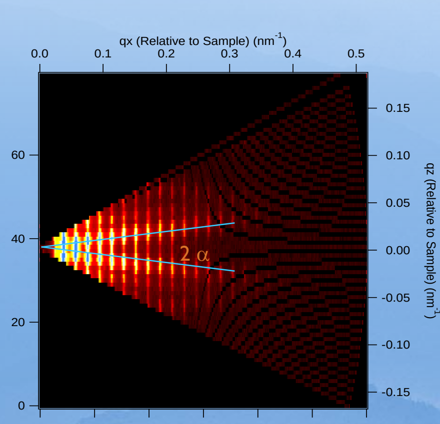

w
2q
0.01
0.005
0
-0.005
-0.01
0
0.005
0.01
0.015
0.02
1
1'
2
2'
3
3'
4
5
β
φ

cross section of a grating line

real space ⟷ Fourier space







IBM DOF p4
+0.4 micron

Period = (330.5 ± 0.5) nm

Linewidth = (160 ± 1) nm

Height = (460 ± 10) nm

Sidewall Angle = (5.6 ± 0.5)°

Random Deviation = 5 nm

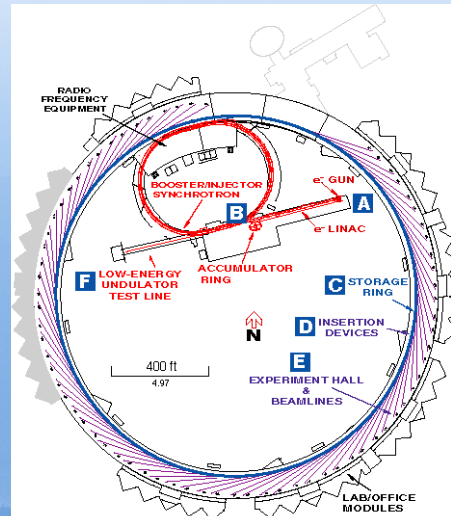
tSAXS Dimensional Metrology References

1. Wu, W.; Lin, E. K.; Lin, Q. H.; Angelopolous, M., Small angle neutron scattering measurements of nanoscale lithographic features. *J. Appl. Phys.* **2000,** *88* (12), 7298-7303.
2. Jones, R. L.; Hu, T.; Lin, E. K.; Wu, W. L.; Kolb, R.; Casa, D. M.; Bolton, P. J.; Barclay, G. G., Small angle x-ray scattering for sub-100 nm pattern characterization. *Appl. Phys. Lett.* **2003,** *83* (19), 4059-4061.
3. Hartschuh, R.; Ding, Y.; Roh, J. H.; Kisliuk, A.; Sokolov, A. P.; Soles, C. L.; Jones, R. L.; Hu, T. J.; Wu, W. L.; Mahorowala, A. P., Brillouin scattering studies of polymeric nanostructures. *J. Polym. Sci. Pt. B-Polym. Phys.* **2004,** *42* (6), 1106-1113.
4. Hu, T. J.; Jones, R. L.; Wu, W. L.; Lin, E. K.; Lin, Q. H.; Keane, D.; Weigand, S.; Quintana, J., Small angle x-ray scattering metrology for sidewall angle and cross section of nanometer scale line gratings. *J. Appl. Phys.* **2004,** *96* (4), 1983-1987.
5. Jones, R. L.; Hu, T.; Soles, C. L.; Lin, E. K.; Wu, W.; Casa, D. M.; Mahorowala, A., Preliminary evaluation of line-edge roughness metrology based on CD-SAXS. *Metrology, Inspection, and Process Control for Microlithography Xviii, Pts 1 and 2* **2004,** *5375*, 191-198.
6. Jones, R. L.; Lin, E. K.; Lin, Q. H.; Weigand, S. J.; Keane, D. T.; Quintana, J. P.; Wu, W. L., Cross section and critical dimension metrology in dense high aspect ratio patterns with CD-SAXS. *Characterization and Metrology for ULSI Technology 2005* **2005,** *788*, 403-406.
7. Jones, R. L.; Lin, E. K.; Qinghuan, L.; Weigand, S. J.; Keane, D. T.; Quintana, J. P.; Wen-li, W., Cross section and critical dimension metrology in dense high aspect ratio patterns with CD-SAXS. *AIP Conference Proceedings* **2005,** (788), 403-6.
8. Jones, R. L.; Lin, E. K.; Wu, W. L.; Weigand, S. J.; Keane, D. T.; Quintana, J. M., Cross sectional measurements of dense high aspect ratio patterns using CD-SAXS. *Metrology, Inspection, and Process Control for Microlithography XIX, Pts 1-3* **2005,** *5752*, 404-411.
9. Jones, R. L.; Soles, C. L.; Lin, E. K.; Hu, W.; Reano, R. M.; Pang, S. W.; Weigand, S. J.; Keane, D. T.; Quintana, J. P., Pattern fidelity in nanoimprinted films using CD-SAXS. *Emerging Lithographic Technologies IX, Pts 1 and 2* **2005,** *5751*, 415-422.
10. Jones, R. L.; Hu, T. J.; Soles, C. L.; Lin, E. K.; Reano, R. M.; Casa, D. M., Real-time shape evolution of nanoimprinted polymer structures during thermal annealing. *Nano Letters* **2006,** *6* (8), 1723-1728.
11. Jones, R. L.; Soles, C. L.; Lin, E. K.; Hu, W.; Reano, R. M.; Pang, S. W.; Weigand, S. J.; Keane, D. T.; Quintana, J. P., Pattern fidelity in nanoimprinted films using critical dimension small angle x-ray scattering. *Journal of Microlithography Microfabrication and Microsystems* **2006,** *5* (1).
12. Jones, R. L.; Wu, W. L.; Wang, C. Q.; Lin, E. K.; Kwang-Woo, C.; Rice, B. J.; Thompson, G. M.; Weigand, S. J.; Keane, D. T., Characterization of line edge roughness using CD-SAXS - art. no. 61520N. *Metrology, Inspection, and Process Control for Microlithography XX, Pts 1 and 2* **2006,** *6152*, N1520-N1520.
13. Knight, S.; Dixson, R.; Jones, R. L.; Lin, E. K.; Orji, N. G.; Silver, R.; Villarrubia, J. S.; Vladar, A. E.; Wu, W. L., Advanced metrology needs for nanoelectronics lithography. *Comptes Rendus Physique* **2006,** *7* (8), 931-941.
14. Chengqing, W.; Jones, R. L.; Lin, E. K.; Wen-li, W.; Villarrubia, J. S.; Kwang-Woo, C.; Clarke, J. S.; Rice, B. J.; Leeson, M. J.; Roberts, J.; Bristol, R.; Bunday, B., Line edge roughness characterization of sub-50 nm structures using CD-SAXS: round-robin benchmark results. *Proceedings of the SPIE - The International Society for Optical Engineering* **2007**, 65181O (9 pp.).
15. Ho, D. L.; Wang, C. Q.; Lin, E. K.; Jones, R. L.; Wu, W. L., A laboratory scale critical-dimension small-angle x-ray scattering instrument. *Frontiers of Characterization and Metrology for Nanoelectronics: 2007* **2007,** *931*, 382-386.
16. Ro, H. W.; Jones, R. L.; Peng, H.; Hines, D. R.; Lee, H. J.; Lin, E. K.; Karim, A.; Yoon, D. Y.; Gidley, D. W.; Soles, C. L., The direct patterning of nanoporous interlayer dielectric insulator films by nanoimprint lithography. *Adv. Mater.* **2007,** *19* (19), 2919-+.
17. Wang, C. Q.; Jones, R. L.; Lin, E. K.; Wu, W. L.; Ho, D. L.; Villarrubia, J. S.; Choi, K. W.; Clarke, J. S.; Roberts, J.; Bristol, R.; Bunday, B., Line edge roughness and cross sectional characterization of sub-50 nm structures using critical dimension small angle x-ray scattering. *Frontiers of Characterization and Metrology for Nanoelectronics: 2007* **2007,** *931*, 402-406.
18. Wang, C. Q.; Jones, R. L.; Lin, E. K.; Wu, W. L.; Villarrubia, J. S.; Choi, K. W.; Clarke, J. S.; Rice, B. J.; Leeson, M.; Roberts, J.; Bristol, R.; Bunday, B., Line edge roughness characterization of sub-50nm structures using CD-SAXS: Round-robin benchmark results - art. no. 65181O. *Metrology, Inspection, and Process Control for Microlithography XXI, Pts 1-3* **2007,** *6518*, O5181-O5181.
19. Chengqing, W.; Kwang-Woo, C.; Jones, R. L.; Soles, C.; Lin, E. K.; Wen-li, W.; Clarke, J. S.; Villarrubia, J. S.; Bunday, B., Linewidth roughness and cross-sectional measurements of sub-50 nm structures with CD-SAXS and CD-SEM. *Proceedings of the SPIE - The International Society for Optical Engineering* **2008**, 69221Z-1-8.
20. Chengqing, W.; Kwang-Woo, C.; Wei-En, F.; Ho, D. L.; Jones, R. L.; Soles, C.; Lin, E. K.; Wen-Li, W.; Clarke, J. S.; Bunday, B., CD-SAXS measurements using laboratory-based and synchrotron-based instruments. *Proceedings of the SPIE - The International Society for Optical Engineering* **2008**, 69222E-1-7.

CD-SAXS Dimensional Metrology References

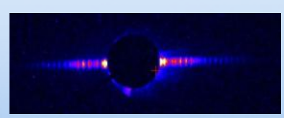
21. Chengqing, W.; Kwang-Woo, C.; Wei-En, F.; Jones, R. L.; Ho, D. L.; Soles, C.; Lin, E. K.; Wen-li, W.; Clarke, J. S.; Villarrubia, J. S.; Bunday, B., Linewidth roughness and cross-sectional measurements of sub-50 nm structures using CD- SAXS and CD-SEM. *2008 IEEE/SEMI Advanced Semiconductor Manufacturing Conference* **2008**, 142-7.
22. Hyun Wook, R.; Jones, R. L.; Huagen, P.; Hae-Jeong, L.; Lin, E. K.; Karim, A.; Yoon, D. Y.; Gidley, D. W.; Soles, C. L., Porosity characteristics of ultra-low dielectric insulator films directly patterned by nanoimprint lithography. *Proceedings of the SPIE - The International Society for Optical Engineering* **2008**, 69211M-1-11.
23. Ro, H. W.; Jones, R. L.; Peng, H.; Lee, H. J.; Lin, E. K.; Karim, A.; Yoon, D. Y.; Gidley, D. W.; Soles, C. L., Porosity characteristics of ultra-low dielectric insulator films directly patterned by nanoimprint lithography - art. no. 69211M. *Emerging Lithographic Technologies Xii, Pts 1 and 2* **2008,** *6921*, M9211-M9211.
24. Ro, H. W.; Peng, H.; Niihara, K. I.; Lee, H. J.; Lin, E. K.; Karim, A.; Gidley, D. W.; Jinnai, H.; Yoon, D. Y.; Soles, C. L., Self-sealing of nanoporous low dielectric constant patterns fabricated by nanoimprint lithography. *Adv. Mater.* **2008,** *20* (10), 1934-+.
25. Wang, C.; Choi, K. W.; Jones, R. L.; Soles, C.; Lin, E. K.; Wu, W. L.; Clarke, J. S.; Villarrubia, J. S.; Bunday, B., Linewidth roughness and cross-sectional measurements of sub-50 nm structures with CD-SAXS and CD-SEM - art. no. 69221Z. *Metrology, Inspection, and Process Control for Microlithography Xxii, Pts 1 and 2* **2008,** *6922* (1-2), Z9221-Z9221.
26. Wang, C. Q.; Choi, K. W.; Fu, W. E.; Ho, D. L.; Jones, R. L.; Soles, C.; Lin, E. K.; Wu, W. L.; Clarke, J. S.; Bunday, B., CD-SAXS measurements using laboratory-based and synchrotron-based instruments - art. no. 69222E. *Metrology, Inspection, and Process Control for Microlithography Xxii, Pts 1 and 2* **2008,** *6922* (1-2), E9222-E9222.
27. Wang, C. Q.; Choi, K. W.; Fu, W. E.; Jones, R. L.; Ho, D. L.; Soles, C.; Lin, E. K.; Wu, W. L.; Clarke, J. S.; Villarrubia, J. S.; Bunday, B., Linewidth roughness and cross-sectional measurements of sub-50 nm structures using CD-SAXS and CD-SEM. *2008 IEEE/SEMI Advanced Semiconductor Manufacturing Conference* **2008**, 142-147.
28. Chengqing, W.; Kwang-Woo, C.; Yi-Ching, C.; Price, J.; Ho, D. L.; Jones, R. L.; Soles, C.; Lin, E. K.; Wen-Li, W.; Bunday, B. D., Nonplanar high-k dielectric thickness measurements using CD- SAXS. *Proceedings of the SPIE - The International Society for Optical Engineering* **2009**, 72722M (8 pp.).
29. Wang, C. Q.; Fu, W. E.; Li, B.; Huang, H.; Soles, C.; Lin, E. K.; Wu, W. L.; Ho, P. S.; Cresswell, M. W., Small angle X-ray scattering measurements of spatial dependent linewidth in dense nanoline gratings. *Thin Solid Films* **2009,** *517* (20), 5844-5847.
30. Yokhin, B.; Krokhmal, A.; Dikopoltsev, A.; Berman, D.; Mazor, I.; Lee, B. H.; Ihm, D. C.; Kim, K. H., Compact X-ray Tool For Critical-Dimension Metrology. *Frontiers of Characterization and Metrology for Nanoelectronics: 2009* **2009,** *1173*, 364-368.

The Advanced Photon Source

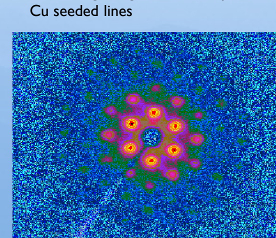


- APS is the first synchrotron to feature a constant x-ray flux.
- Most synchrotrons have decaying fluxes that are “refilled” every 10-12 hours

Lab Scale tSAXS Prototype



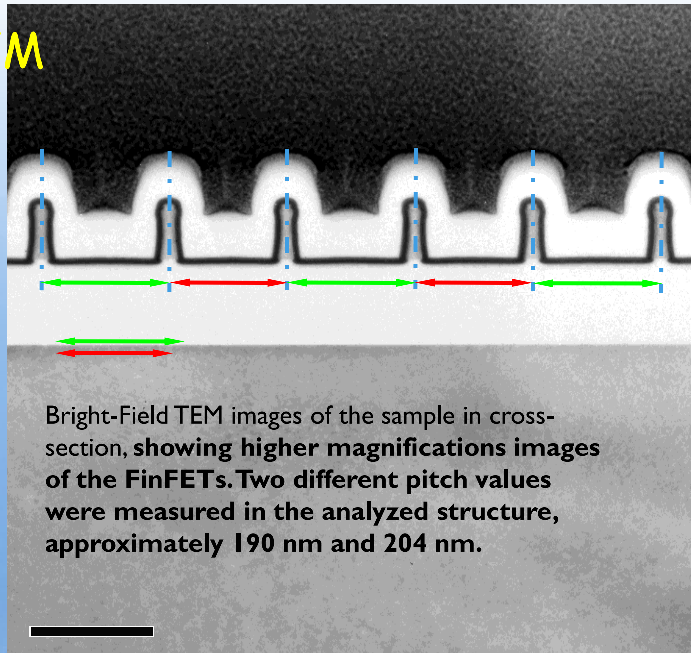
Intel test grating with 400nm pitch Cu seeded lines



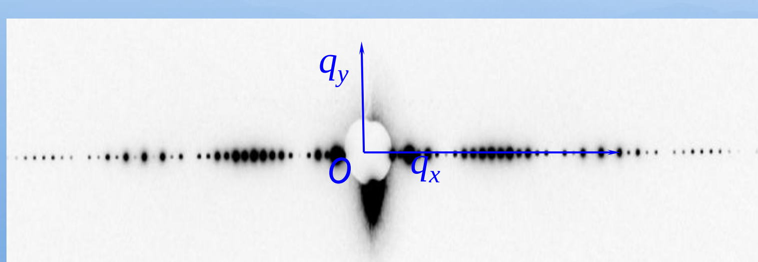
Nanoimprint array of 60 nm holes

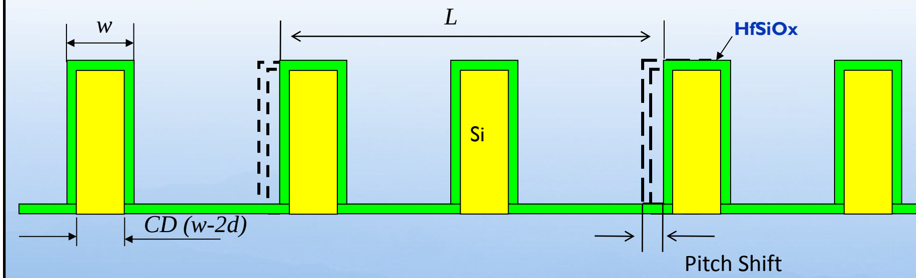
- Beam flux ~4 to 5 orders of magnitude below APS
- Installed November 2005

X-TEM



FinFET with high-k dielectric



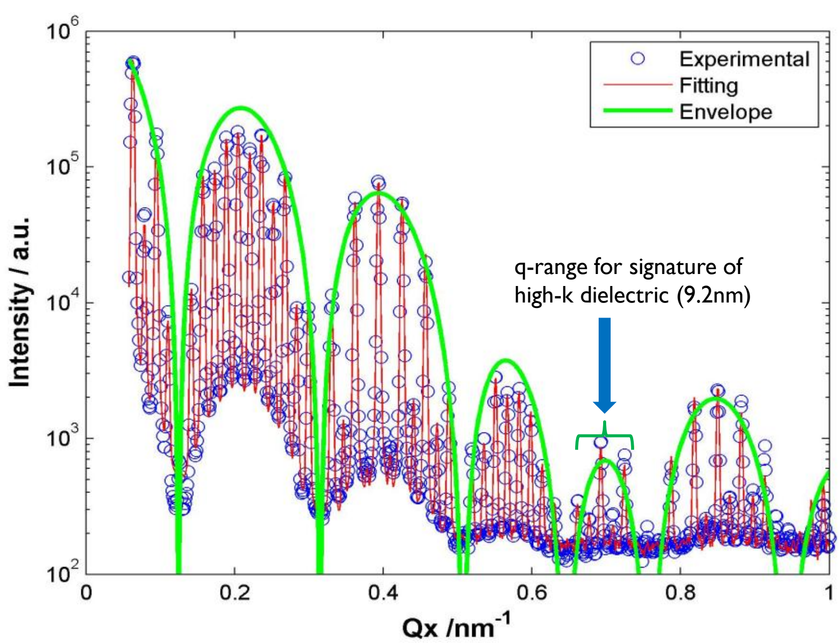


Definitions:
CD: silicon width
w: line width (CD) + High-k thickness
d: High-k thickness
L: effective pitch
ρ_1: electron density of High-k dielectric (2.012 e/Å^3)
ρ_2: electron density of silicon (0.699 e/Å^3)

Samples Descriptions:

Larger array/cell of ~ 300 x 700um
200nm Pitch

1) Si (CD = 50nm), no HfSiOx
2) Si (CD = 30nm), no HfSiOx
3) Si (CD = 30nm), 2 nm HfSiOx
4) Si (CD = 30nm), 4 nm HfSiOx
5) Si (CD = 30nm), 7 nm HfSiOx
6) Si (CD = 30nm), 10 nm HfSiOx

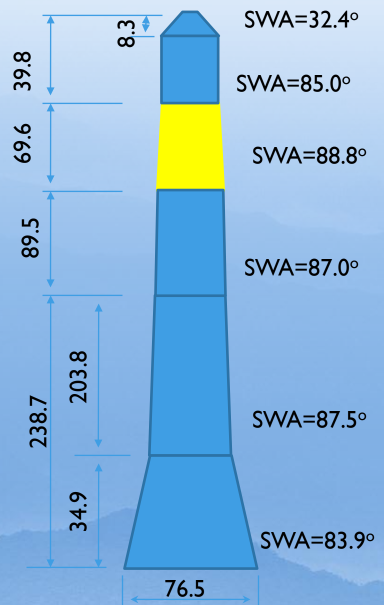


30

Six Trapezoids (Heights scaled to design value)

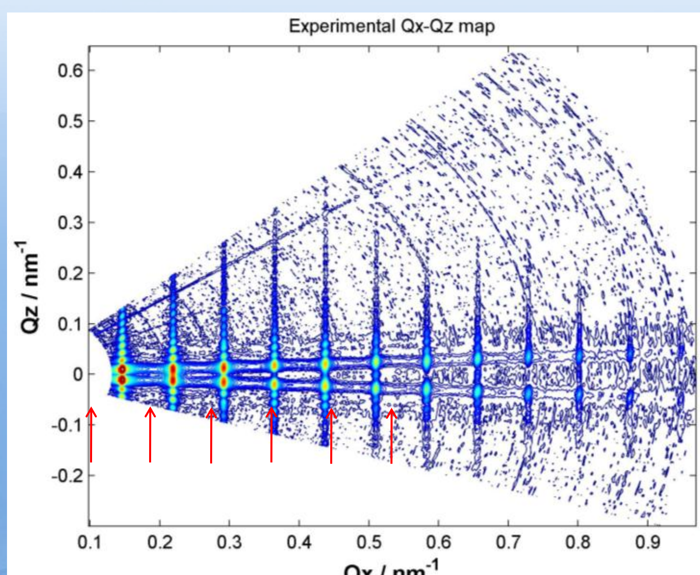
Wafer 1, cell 1

$\chi^2 = 185.8$



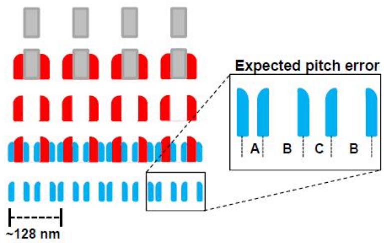
Total Height: 438.6

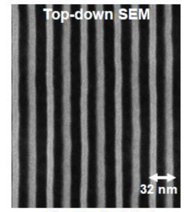
Wafer 1, cell 1



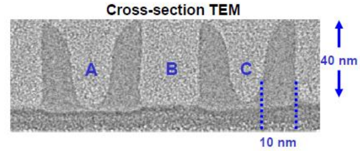
ME Dept. NTU / TSPE 105

Spacer-Assisted Quadruple Patterning (SAQP)



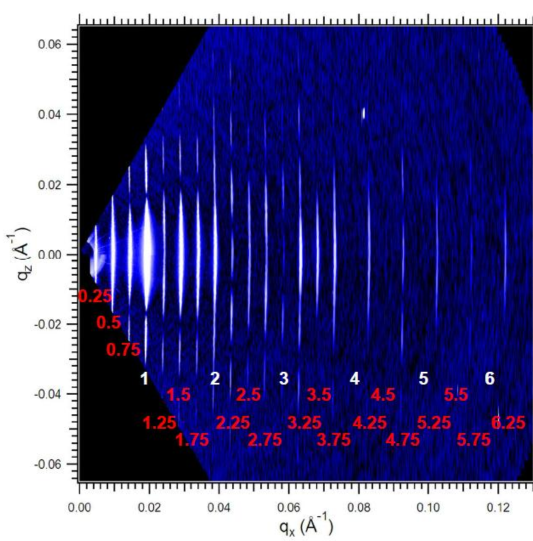


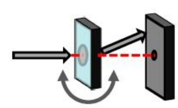
From Andras Vladar



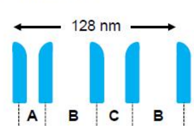
- Measurement goals
  - Average 2D shape, pitch error, and edge roughness
- Sample set with sub-nm controlled variation in pitch error and nominal 32 nm pitch

van Veenhuizen et al. Interconnect Technology Conference, 2012

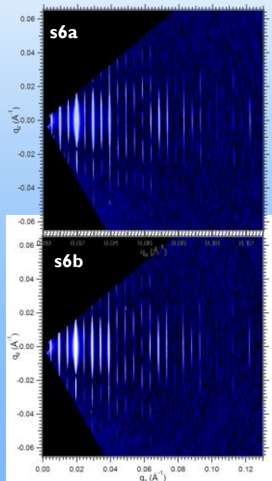




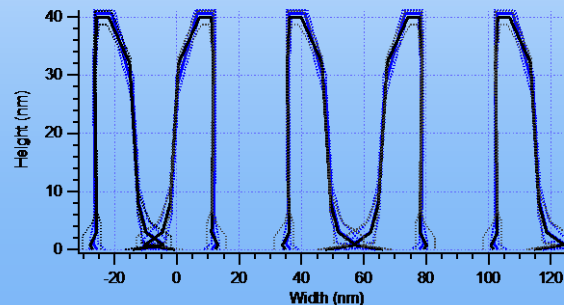
- Composite from 121 images
  - ±60° sample angle
  - 10 s/image = ~30 min/scan
  - Data highly oversampled
- 32 nm, 64 nm, and 128 nm pitches clearly visible
  - Non-integer peaks from pitch quartering error



Similar Sample Comparison

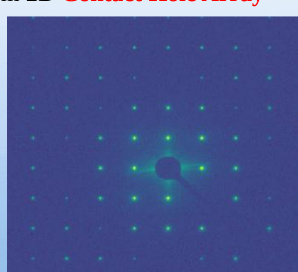


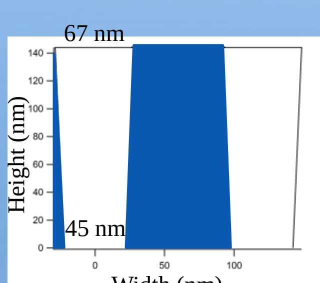
	S6a	s6b
H	40.4 ± 0.6 nm	39.9 ± 0.4 nm
$W_{1/2H}$	12.5 ± 0.2 nm	12.3 ± 0.1 nm
σ	1.0 nm	1.1 nm
SL	128.7 ± 1.3 nm	128.7 ± 1.3 nm



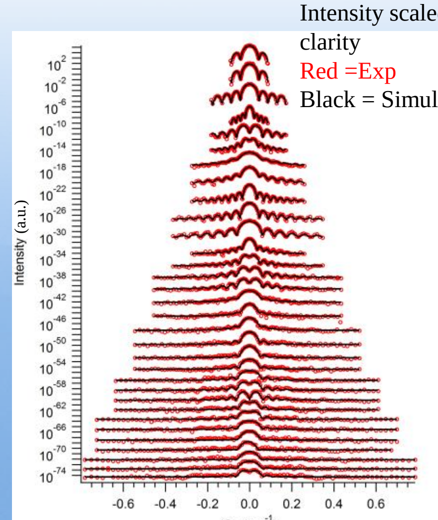
- Samples are on different die, but with same processing
- X-ray scattering patterns are almost identical
- Fits are identical within the uncertainty

Normal Incidence Scattering Pattern from 2D **Contact Hole Array**





Intensity scaled for clarity
Red =Exp
Black = Simulated



NIST: Daniel F. Sunday, R. Joseph Kline
CEA-LETI: Raluca Tiron, Florian Delachat

Summary - tSAXS

tSAXS has been used successfully to demonstrate its capability to measure pitch, line width, line height, sidewall angle, thickness of non-planar high-k dielectric layer on FinFET, line edge roughness, standing wave roughness in photoresist lines and some complicated cross sectional profiles.

OUTLINE

1. Transmission small angle X-ray scattering (tSAXS)
2. **Grazing incident small angle X-ray scattering (GISAXS)**
3. X-ray reflectivity (XRR)
4. CMS efforts to address/circumvent difficulty caused by the insufficient brilliance in current laboratory based X-ray sources

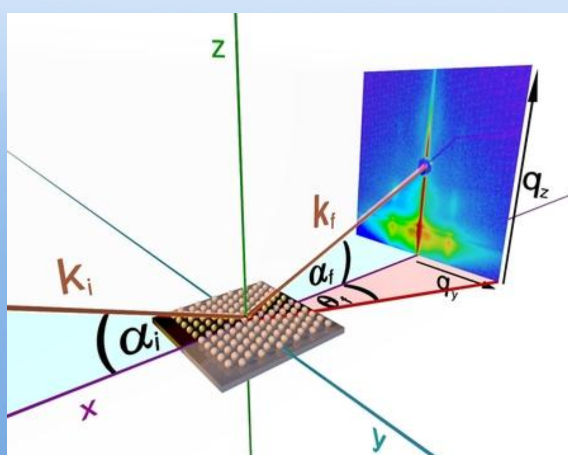
Grazing Incident X-ray Scattering/Reflectivity for Characterizing Nano-patterns on Flat Substrate

Wen-li Wu, Chengqing Wang, Ronald L. Jones, Christopher Soles, Hae-Jeong Lee, Kevin G. Yager#
Polymers Division, National Institute of Standards and Technology, Gaithersburg, MD
#current address: Brookhaven National Laboratory, NY

Work supported by NIST Office of Microelectronics Programs

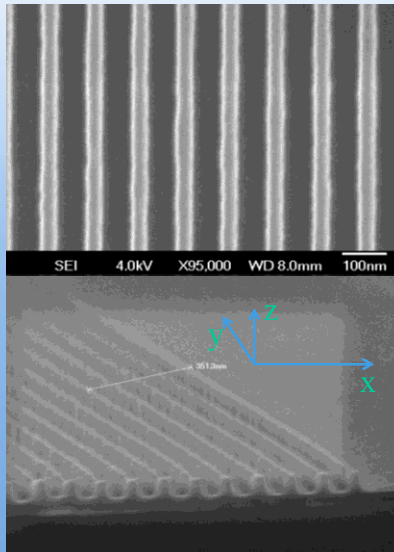
39

Grazing Incident X-ray Scattering

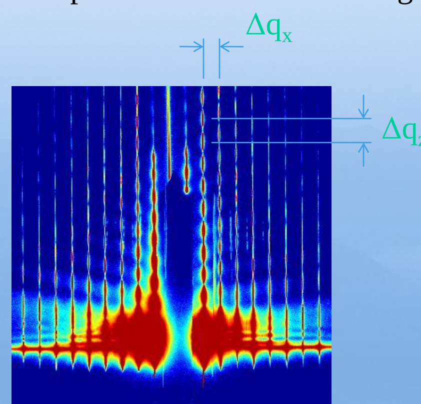


Advantage: *(1)* high scattering intensities due to large beam path of the incident beam, *(2)* depth resolution possible

Disadvantage: large test area needed & **difficult data analysis quantitatively**

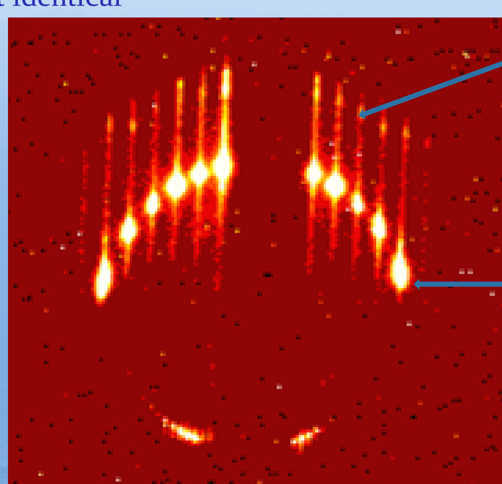


Quantitative information on pitch and line height can be straightforward; Line gratings with 88nm pitch and ~ 45nm line height



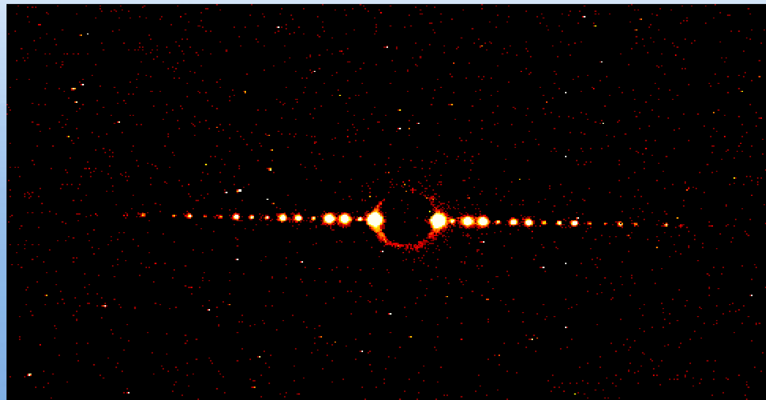
$\Delta q_z : \Delta q_x = 88:45$

GISAXS result from an **Intel line gratings** made of low-k materials; $\Delta q_z > \Delta q_x$ or pitch is larger than the line height? In fact, the pitch (399nm) and the line height (400nm) is almost identical



(2) Scattering of the surface enhanced beam

(1) Scattering of the reflected beam



tSAXS data from the same Intel sample

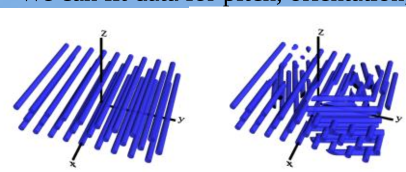
New Data Analysis Effort

- Calculate reflected scattering based on an arbitrary reference function $V_0 = V(z)$, the xy-averaged potential:

$$\mathbf{R}(\mathbf{k}_i, \mathbf{k}_x) = \tilde{\mathbf{R}}(-\mathbf{k}_s, -\mathbf{k}_i) + \frac{1}{2ik_1 a\sqrt{\sin\theta_i \sin\theta_s}} \iiint (V - V_0)\tilde{\psi}\psi dv$$

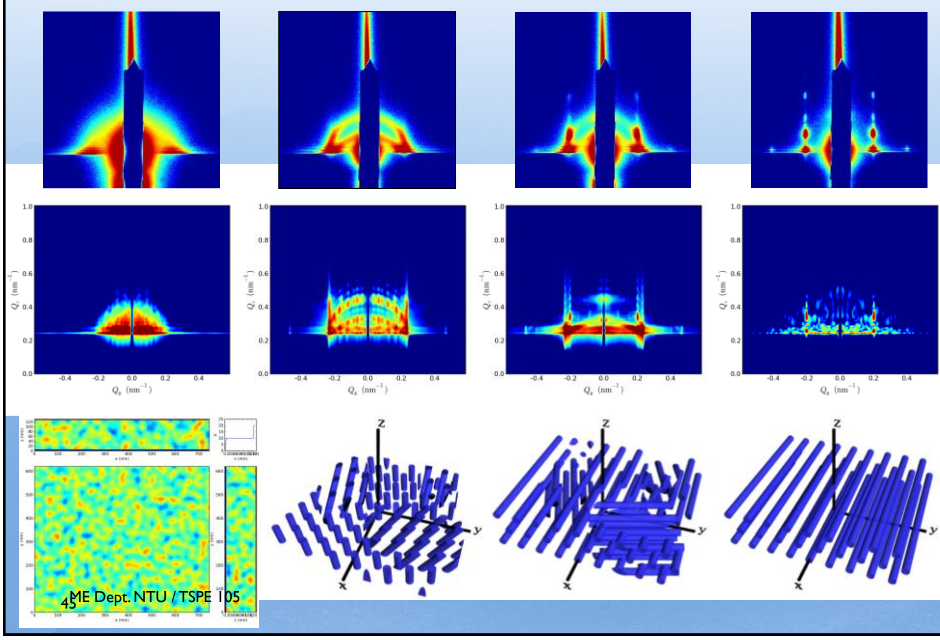
$$\iiint (V - V_0)\psi\tilde{\psi}dv = \iiint (V - \overline{V}(z)) e^{ik_1(\cos\theta_s \cos\phi_s + \cos\theta_i)x} e^{ik_1 \cos\theta_s \sin\phi_s y} \psi_z \tilde{\psi}_z dxdydz$$

- By inputting the x-ray reflectivity curve, we account for reflection effects (including multiple internal reflections)
- We can fit data for pitch, orientation, order/disorder, etc.



Wen-li Wu, *J. Chem. Phys.*, **1993**, *98*, 1687
Wen-li Wu, *J. Chem. Phys.*, **1994**, *101*, 4198

Evolution of Order



•Additional information exists in the GISAXS data due to its **complex and multiple** origins of scattering.

•This poses challenge on quantitative data analysis

Summary - GISAXS

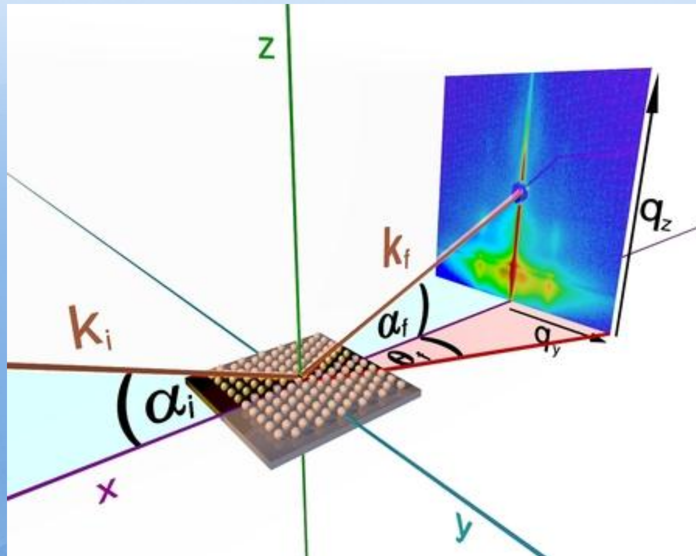
- Grazing incident X-ray scattering enables data with enhanced S/N over transmission measurements when large sample size is available
- Quantitative data analysis (beyond pitch and line height in simple line gratings) requires additional developments, a logic starting point is specular X-ray reflectivity

47

OUTLINE

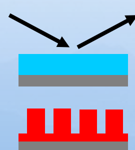
1. Transmission small angle X-ray scattering (tSAXS)
2. Grazing incident small angle X-ray scattering (GISAXS)
3. **X-ray reflectivity (XRR)**
4. CMS efforts to address/circumvent difficulty caused by the insufficient brilliance in current laboratory based X-ray sources

Grazing Incident X-ray Scattering and X-ray Reflectivity



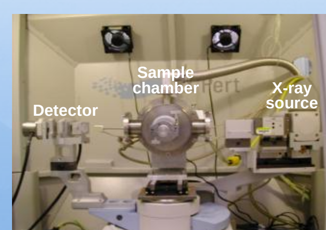
Why X-ray reflectivity (SXR)

Extending applicability from planar films to line gratings



- Planar films
  film thickness
  electron density through film
  interfacial roughness
- Patterned (imprinted) films
  pattern height
  line-to-space ratio (*vs.* pattern height)
  residual layer thickness
  fidelity of pattern transfer

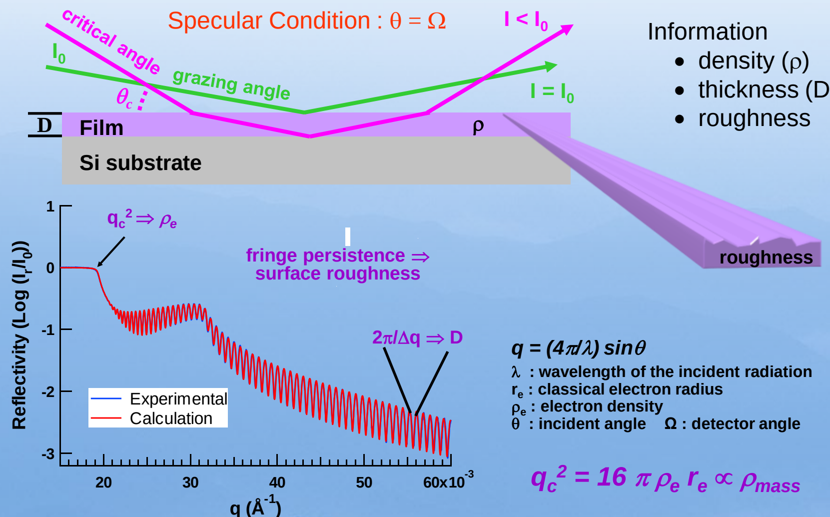
High resolution X-ray reflectometer



High precision settings

X-ray optics
goniometer control

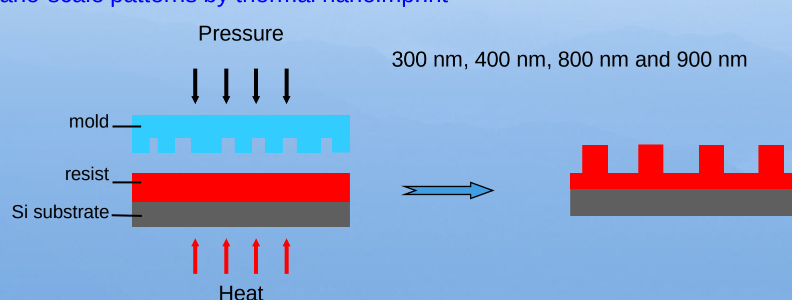
Principle of Specular X-ray Reflectivity (SXR)



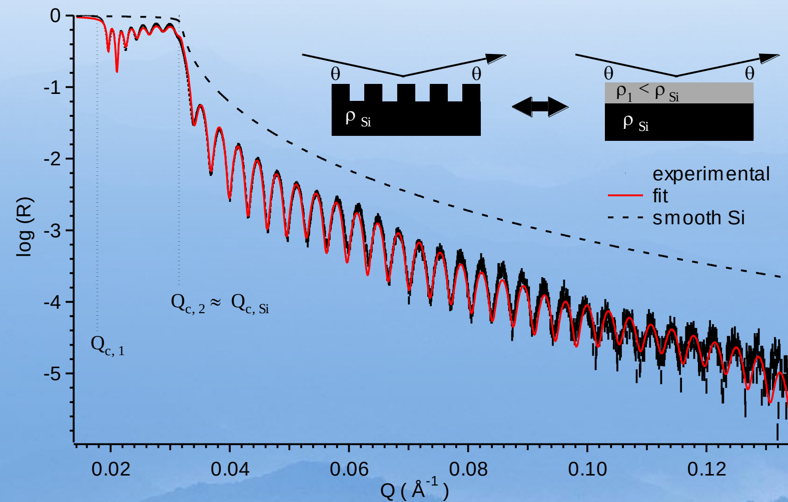
If atomic composition is given, q_c is converted into ρ_{mass}

Samples preparation: line-space gratings via nanoimprint

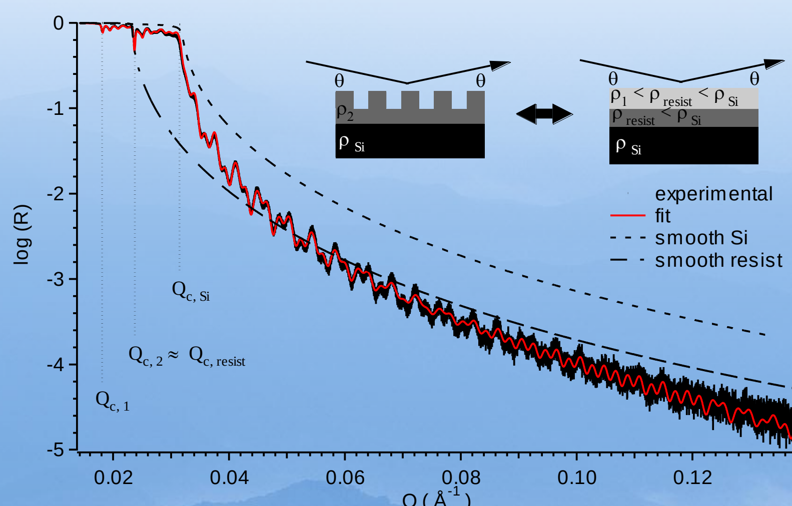
Nano-scale patterns by thermal nanoimprint



SXR of Mold

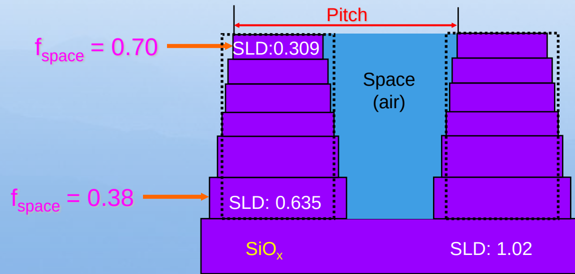


SXR of Imprinted Resist



Line to space ratio

Comparing SLD in the patterned region to the fully dense material



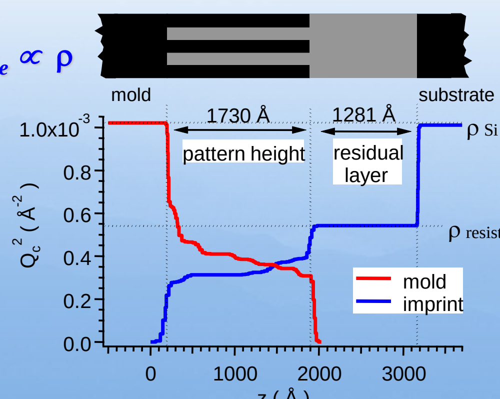
$$SLD\ (layer) = SLD\ (Si/SiO_2) \times (1 - f_{space}) + SLD\ (air) \times f_{space}$$

SLD (layer): SLD of each layer in the patterned region
SLD (Si/SiO_2) and SLD (air): SLD of Si substrate and air, respectively
f_{space}: lateral fraction of space between the lines in the pitch.

*Line-to-space ratio can be determined via XR **BUT not** the dimension of line or space*

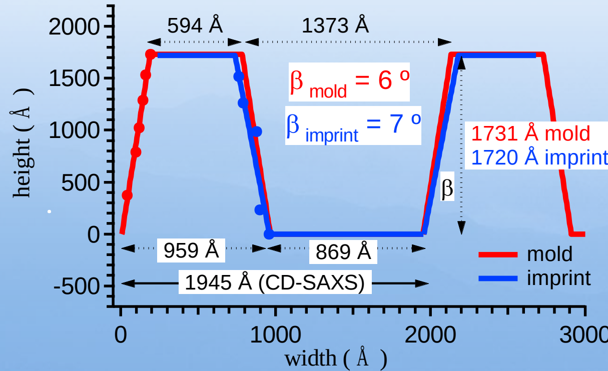
X-ray Scattering Length Density Profiles

$$Q_c^2 = 16\,\pi\, r_o\, \rho_e \propto \rho$$



- Residual layer thickness is 1281 ± 10 Å
- Imprint pattern height is 1720 ± 10 Å
- Mold pattern height is 1730 ± 10 Å
- Excellent mold filling!

Profiling real pattern Structure



<u>average line widths</u>

mold = 1120 Å
imprint = 777 Å

<u>CD-SAXS model: mold</u>

W_{gap} = 772 ± 10 Å
W_{line} = 1173 ± 10 Å
H = **1650 ± 50** Å
β_{right} = 5.4 ± 0.5 °
β_{left} = 6.3 ± 0.5 °

- CD-SAXS pitch & SXR line-space ratio defines absolute length scale
- Full line shape profile as a function of pattern height are quantified
- Excellent fidelity of pattern transfer
- Side wall angles and line/space ratio are consistent with CD-SAXS
- Less agreements w/ CD-SAXS line height; SXR is probably superior

Summary - XRR

- **XRR** is utilized as a powerful methodology to quantify **relative** line-to-space ratio as a function of pattern height in periodic patterns
- For periodic patterns an lateral length scale from **tSAXS** can be used to convert the relative line-to-space ratios into absolute values
- SXR complements CD-SAXS for 3D dimensional metrology in nano-structures

Conclusion

The application of X-ray for nanostructure characterization, via tSAXS, GISAXS and XRR or a combination of all the above, is now well accepted by academia and industrial world as well. For its applications in the process development and the process control for semiconductor fabrication **high brilliance X-ray sources** are needed to fulfil the stringent requirement in speed.

59

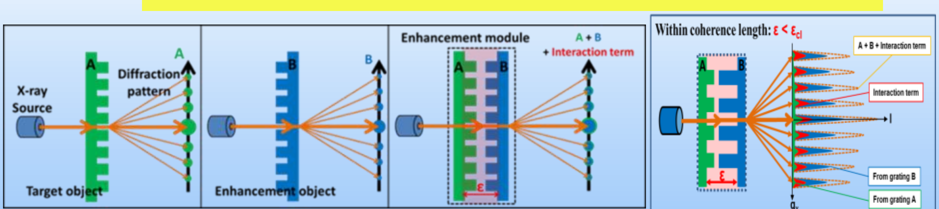
OUTLINE

1. Transmission small angle X-ray scattering (tSAXS)
2. Grazing incident small angle X-ray scattering (GISAXS)
3. X-ray reflectivity (XRR)
4. **Efforts at CMS to address/circumvent the difficulty caused by the insufficient brilliance in current laboratory based X-ray sources**

To enhance the tSAXS signal by 10x -100x

Apparatus for amplifying intensity during transmission small angle—X-ray scattering measurements
US 9297772 B2, Mar 29, 2016
Inventors: Wei-En Fu, Wen-li Wu
Original Assignee: Industrial Technology Research Institute

Enhancement Module (EM) for CD & Overlay

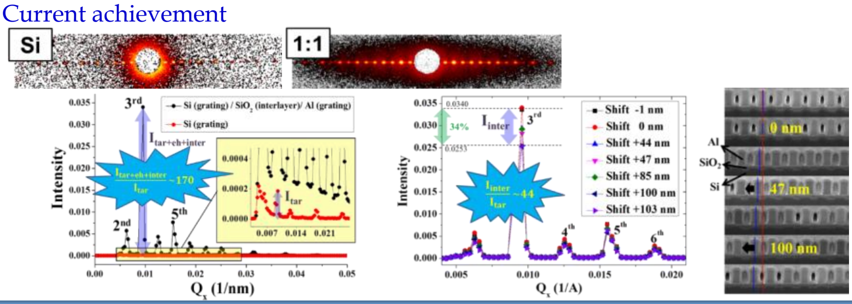


$$I(q) \propto \Delta b_1^{\,2} \times F_1^{\,2}(q) + \Delta b_2^{\,2} \times F_2^{\,2}(q) + 2\Delta b_1 \Delta b_2 \cos(q\eta)|F_1(q)F_2(q)|$$

Target object term — Enhancement module term — Interaction term

Within X-ray longitudinal coherent length
10x to 100x in signal

Current achievement



Why Small Angle Electron Scattering (SAES)

To circumvent the difficulties encountered by GISAXS;

- Large footprint
- Lack of small X-ray source with sufficient brilliance

US Patent 9390888: Apparatus and method of applying small-angle electron scattering to characterize nanostructures on opaque substrate

Inventors: Wu; Wen-Li, Chien; Yun-San, Fu; Wei-En, Chen; Yen-Song, Ho; Hsin-Chia (H
Original Assignee: Industrial Technology Research Institute

Small Angle e-beam Scattering (SAES)

- e-beam has the following **advantages** for scattering applications in comparison to X-ray;
  - EM lens and other optical components have been well developed
  - High intensity focus beam available with a focal spot size of **a few nanometers** – footprint can be much smaller than X-ray (a major problem for GISAXS)
  - Scattering cross section is ~10^4 greater than that of X-ray for all materials – incident beam intensity is no longer a show stop for SAES
- E-beam has also the following disadvantages;
  - Complicate interactions with matters including elastic, inelastic scattering, secondary electron and multiple scattering – data interpretation can be challenging
  - **Low** penetration power – SAES has to be operated in a **reflective** mode or back scattering mode; **a complementary** but **NOT a complete replacement for X-ray based metrologies**
  - Not applicable for studying buried structures
  - Extreme short wavelength, often in picometers. This results in very small scattering angles

$$\lambda = h \Big/ \sqrt{2m_0 eV\,(1 + eV / 2m_0 c^2)}$$

Short wavelength of e-beam → a great spatial resolution

V / kV	Non rel. λ / pm	Rel. λ / pm	m x m_0	v / 10^8 m/s
100	3.86	3.70	1.20	1.64
200	2.73	2.51	1.39	2.09
300	2.23	1.97	1.59	2.33
400	1.93	1.64	1.78	2.48
1000	1.22	0.87	2.96	2.82

Table 1: Properties of electrons depending on the acceleration voltage.

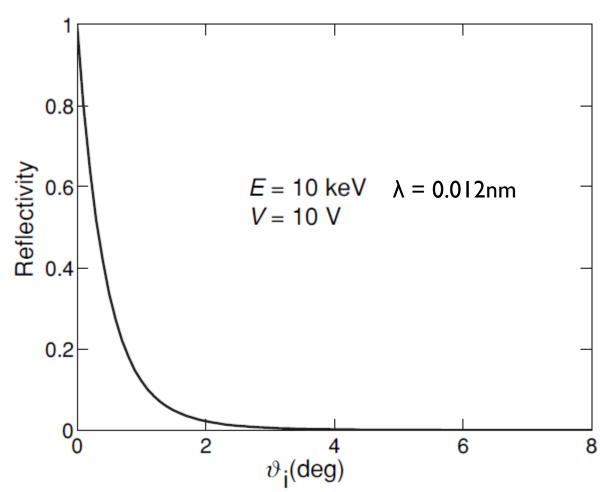
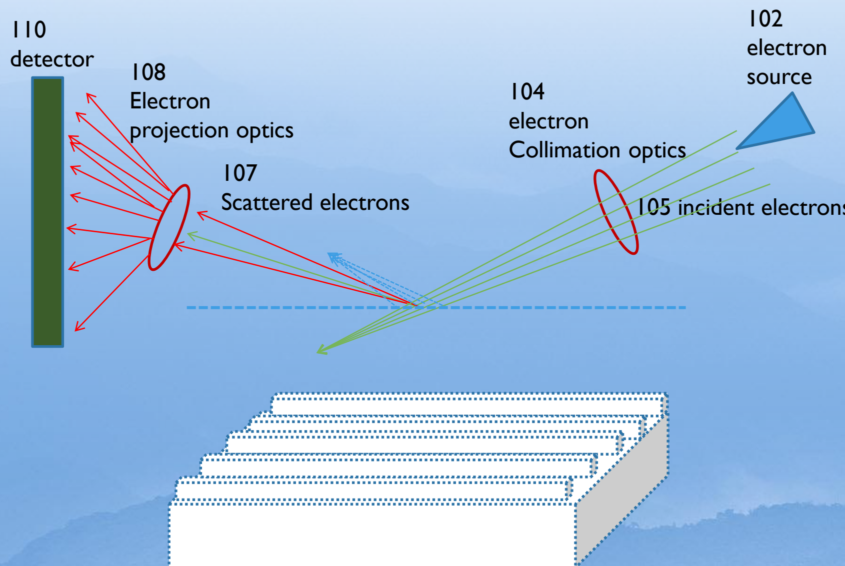
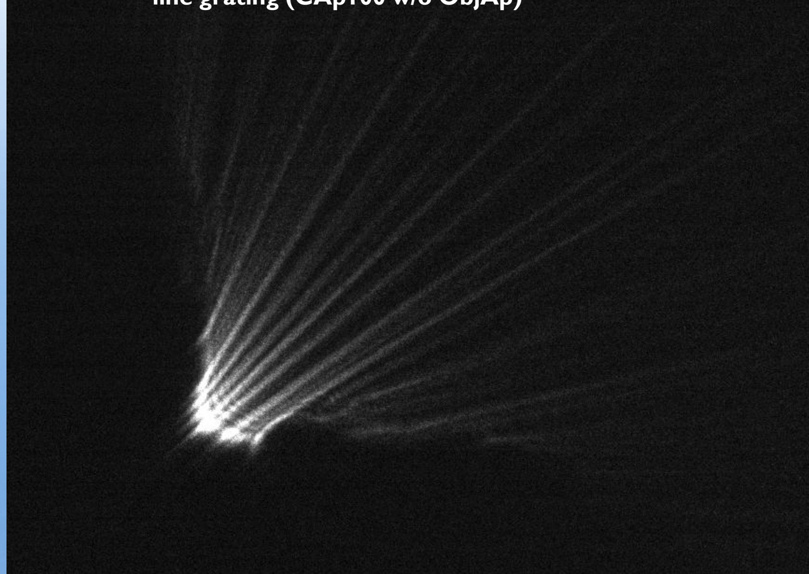


Figure 4.4 Reflectivity vs external incident angle from the Fresnel equation.

Multiple lenses are needed after sample for SAES measurements

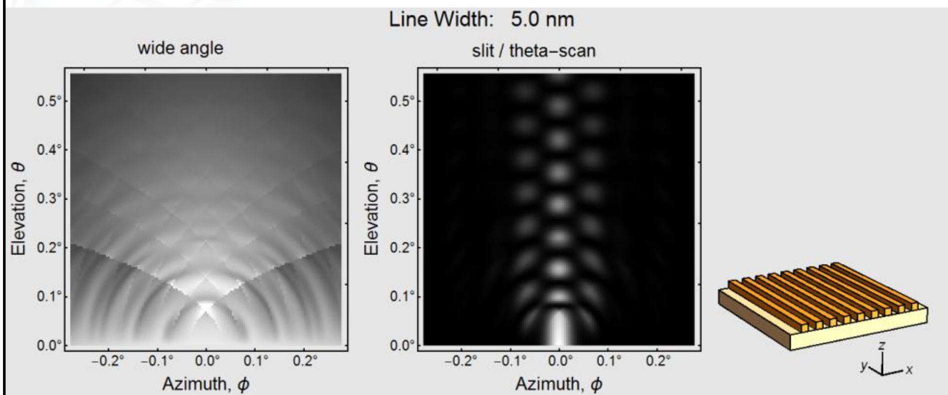


A preliminary SAES results from a Copper line grating (CAp100 w/o ObjAp)



Lawrence Friedman,
W.L. Wu

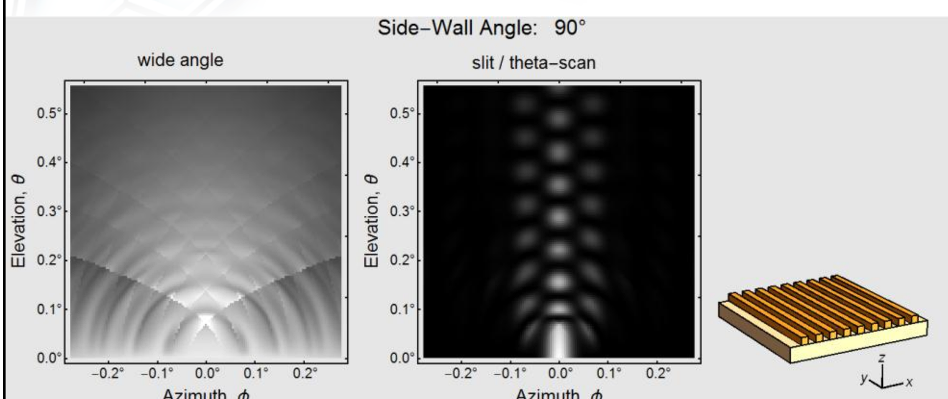
Line Width Sensitivity



MATERIAL MEASUREMENT LABORATORY

Lawrence Friedman,
W.L. Wu

Shape Sensitivity



MATERIAL MEASUREMENT LABORATORY


Happy 30th Anniversary