High Resolution Crystal Optics

For Inelastic X-ray Scattering

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Background

Experience based on both BL35XU & BL43LXU at SPring-8

BL35XU

- meV non-resonant IXS
- Operating beamline for IXS
  - Design & commissioning 2000 -

BL43LXU

- New non-resonant IXS beamline for meV and 10 meV
  Commissioning

- *BL43 overview > Friday talk by A. Baron*

Contents

- ① Progress for High-Resolution analyzers
- ② Improving resolution with a T gradient
- ③ Plans for 10 meV (Medium-Resolution) analyzers
- ④ Time Permiting: Mirror Performance

BL43LXU

RIKEN Quantum Nano Dynamics Beamline

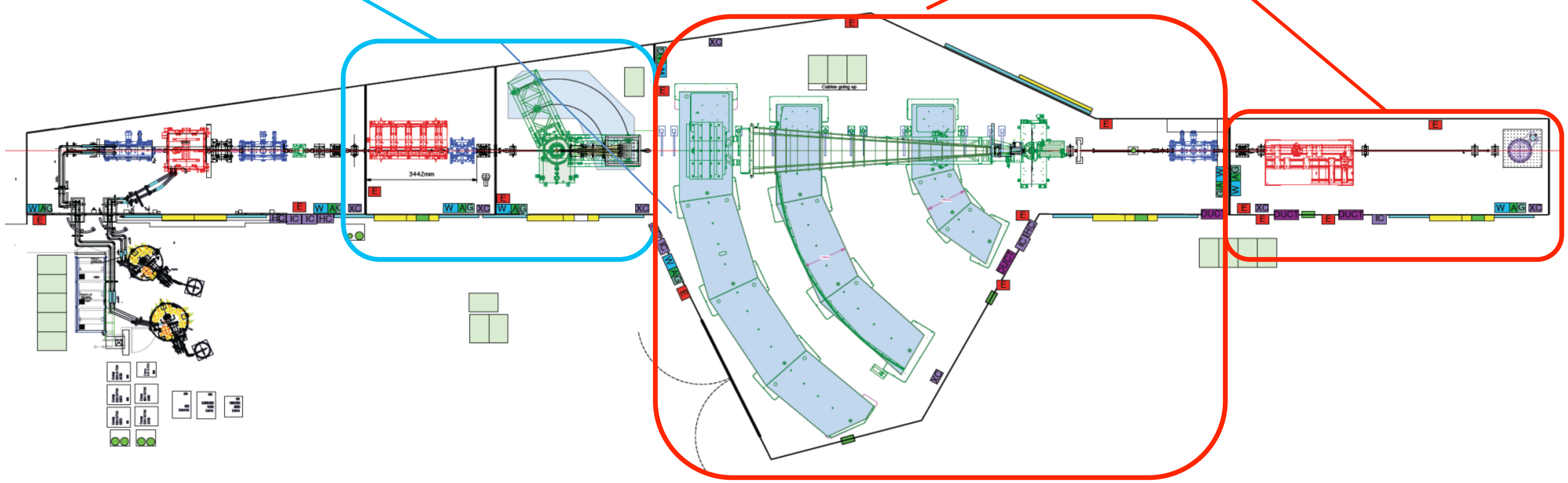
Non-Resonant Inelastic X-ray Scattering Beamline

Medium Resolution (MR) IXS

- Electronic excitation
- $\Delta E = 10 – 40$ meV
- $E\text{-}E_0 < 100$ eV
- $R = 1900$

High Resolution (HR) IXS

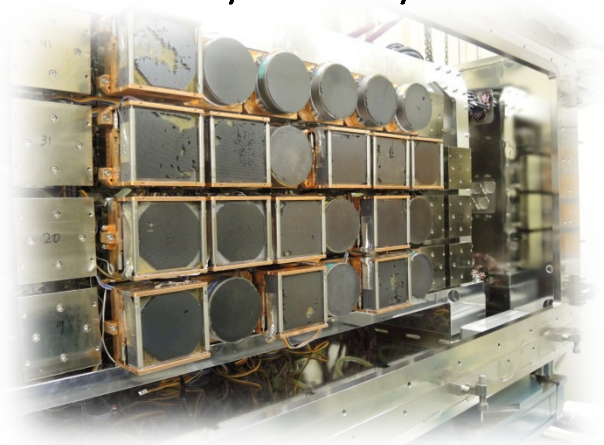
- Phonon (Electronic excitation)
- $\Delta E = 0.6 – 6.0$ meV
- $E\text{-}E_0 < 3$ eV
- $R = 9800$

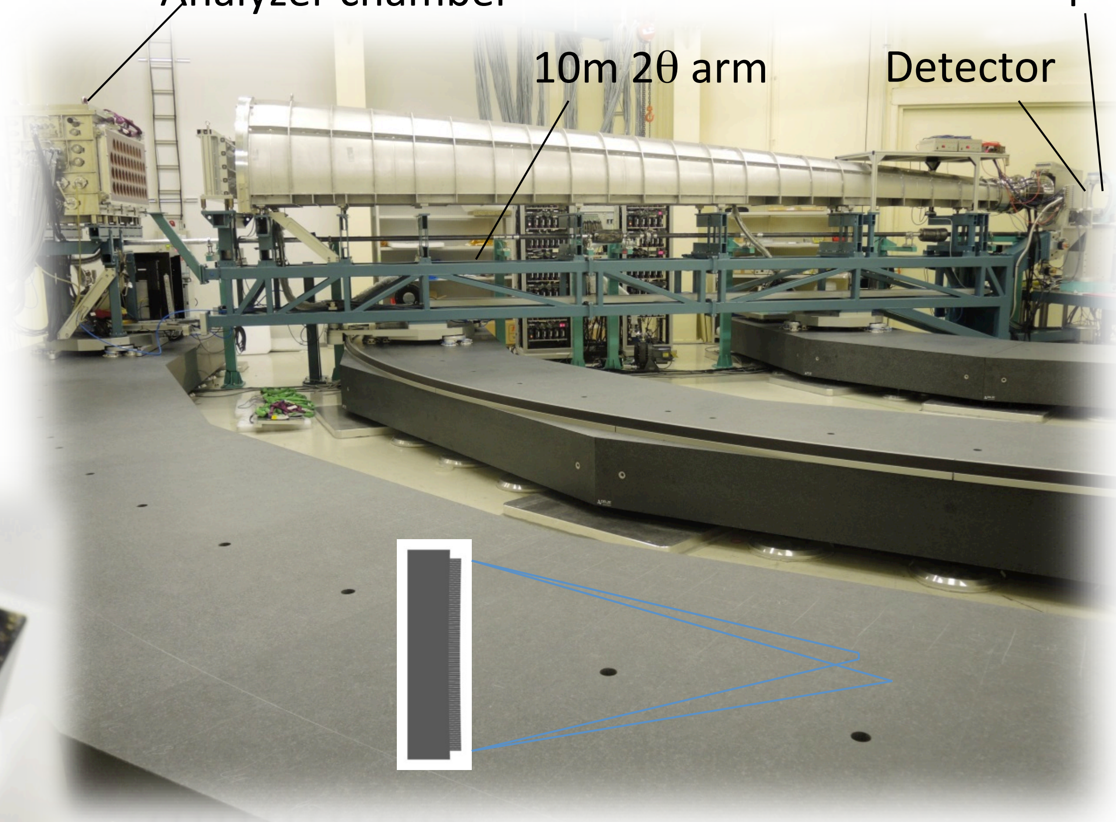


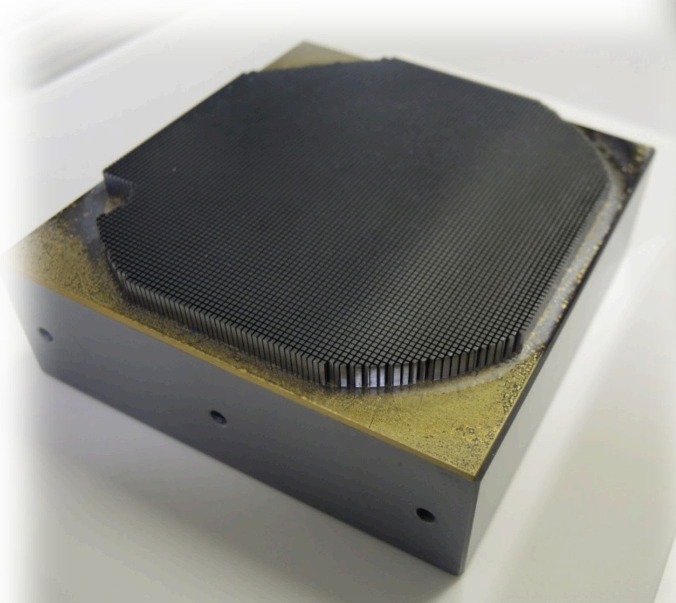
① HR analyzers

High-Resolution IXS

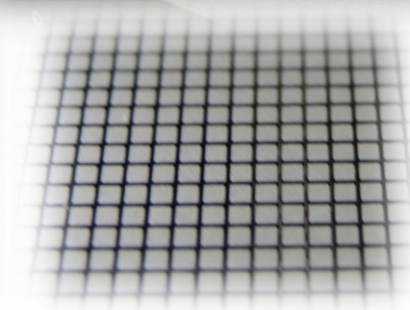
42 analyzer array







R9800 Analyzer



Arm 10 m
ΔE = 0.6-6 meV, E = 15.8 - 25.7 keV
E-E_0 < 3 eV, Q < 15 $Å^{-1}$
Phonon (Electronic excitation)

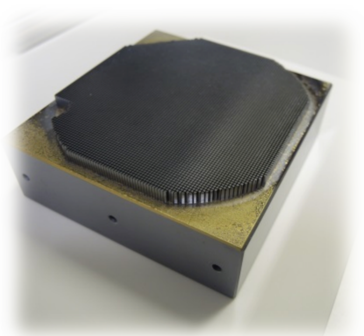
Progress of Analyzer Performance

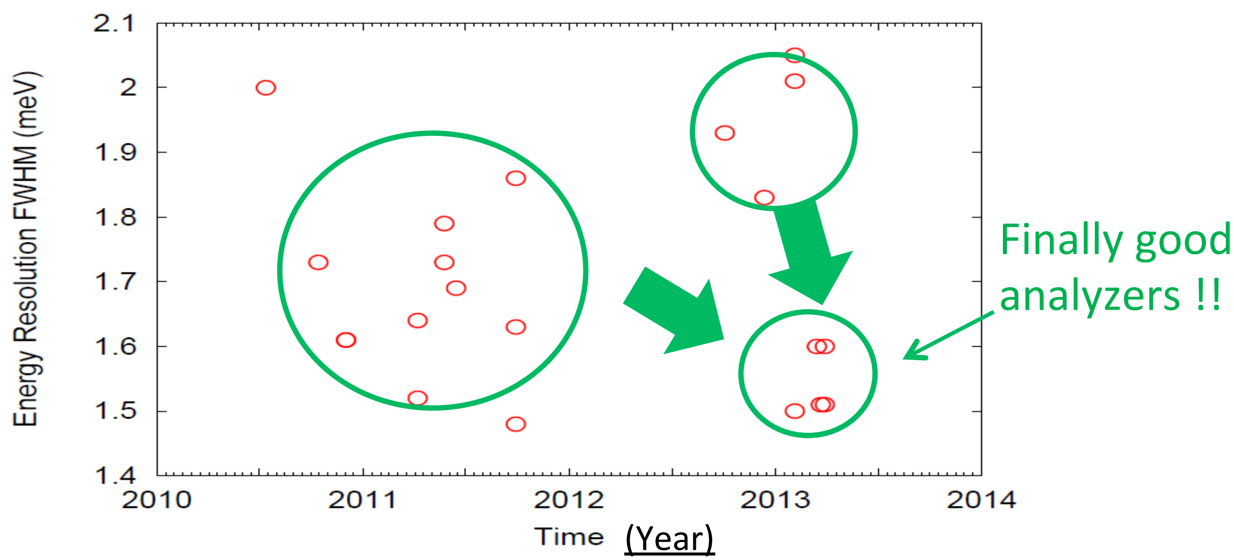
HR-Analyzers (R9800)

- NEC fabricated BL35XU analyzers, but they declined to make analyzers for BL43LXU
- Technology Transfer from NEC to a new company (SARTONWORKS)
- SARTONWORKS started from 2010 but first attempts failed. Learning Needed.

Issues

- Silicon Quality
- Polishing
- Etching (fast etching → slow etching)
- Bonding



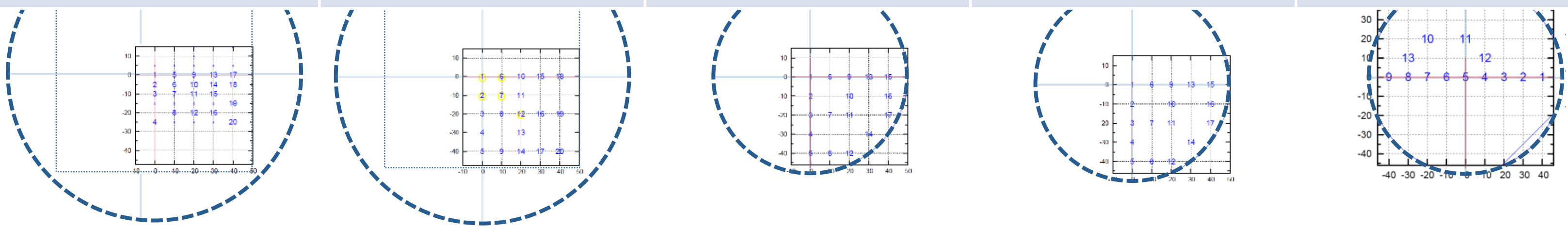


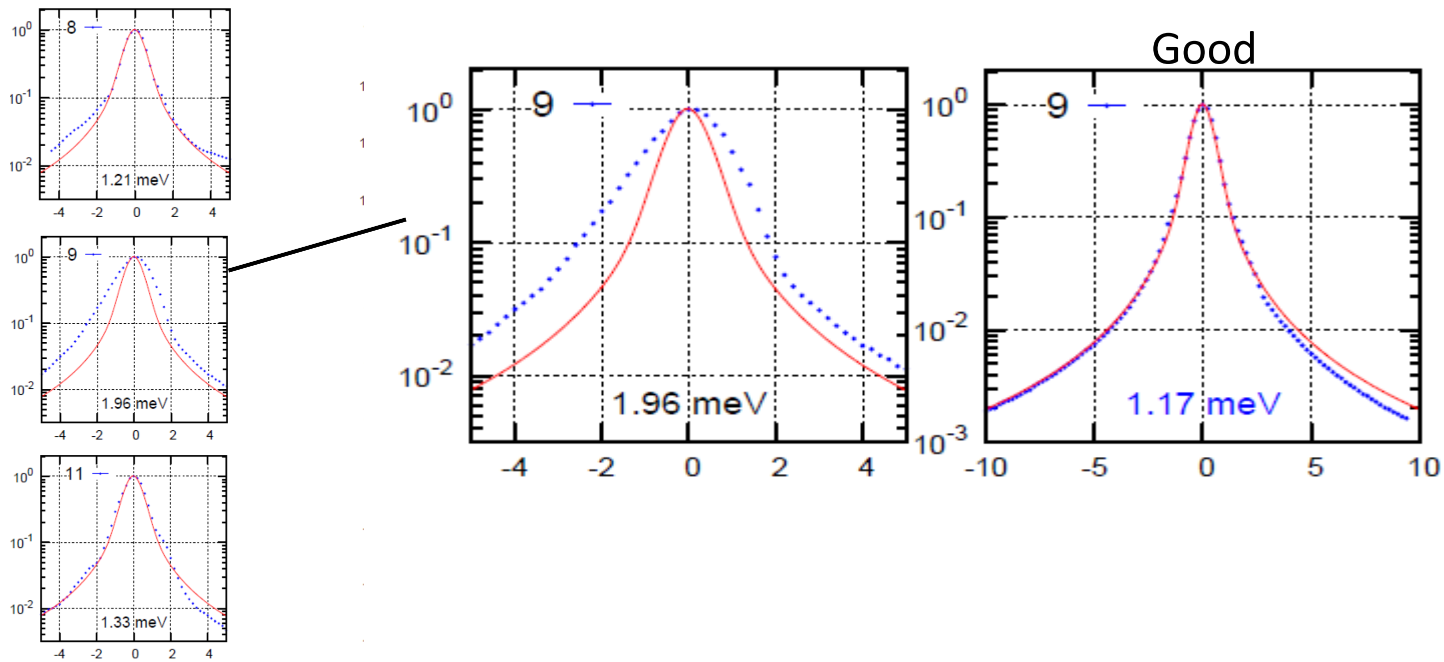
Silicon Quality Check

- Single crystal : ϕ6’, ϕ4‘ FZ<111> INGOT
- SHIN-ETSU 6 inch ingot had a bad quality !

Silicon Ingot
made by SHI-ETSU HANDOTAI COMPANY. LTD.,
Silchem/Wacker Chemie AG

SHIN-ETSU	SHIN-ETSU	SHIN-ETSU	SHIN-ETSU	Wacker
6 inch No.1	6 inch No.2	4 inch No.1	4 inch No.2	4 inch No.1





Some Bad Spots

Silicon Quality Check

SHIN-ETSU	SHIN-ETSU	SHIN-ETSU	SHIN-ETSU	Wacker
6 inch No.1	6 inch No.2	4 inch No.1	4 inch No.2	4 inch No.1

- First 6” Silicon -> Check the ends -> OK
- Problems: Check leftover -> Some bad points
- Another 6” Ingot: OK
- Went to 4” Wafers -> as all OK
- 2013: Return to known good 6” wafer

Expect Main Issue Probably polishing.

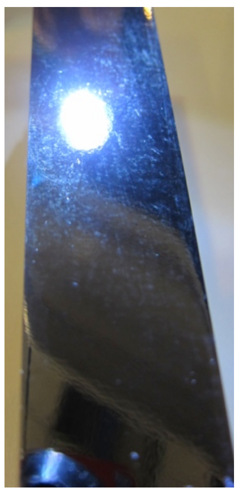
Issue with Initial Silicon Polish

- Company had some experience doing strain-free polishing.
- However, some local bad performance of other crystals -> Investigation

Mechanical = with abrasive powder
MCP = Mechano-Chemical Polishing (No abrasive powder)

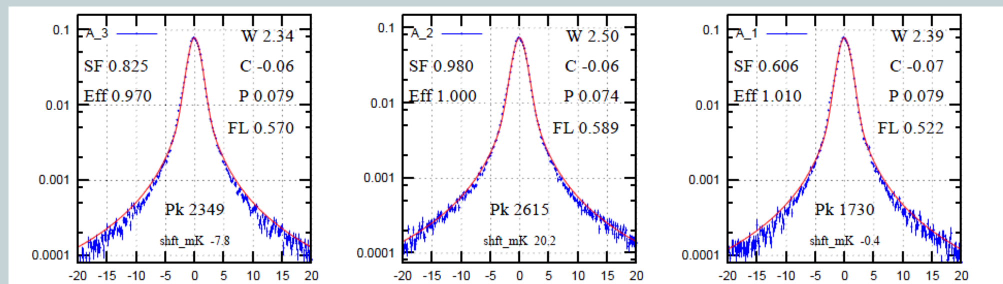
Silicon after a light etch

Polished Surface

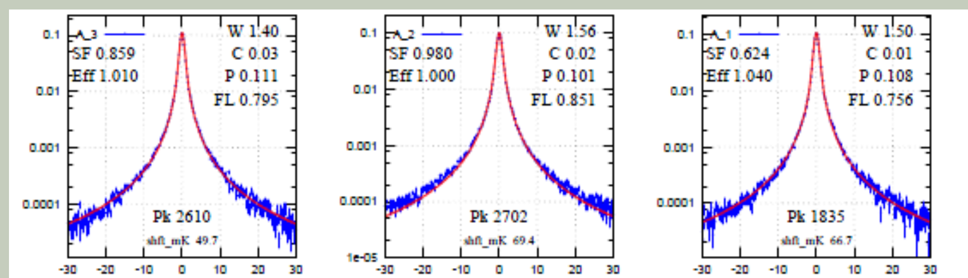


Back

Old: Etch & Mechanical & Some MCP



New: Etch & MCP (100um)

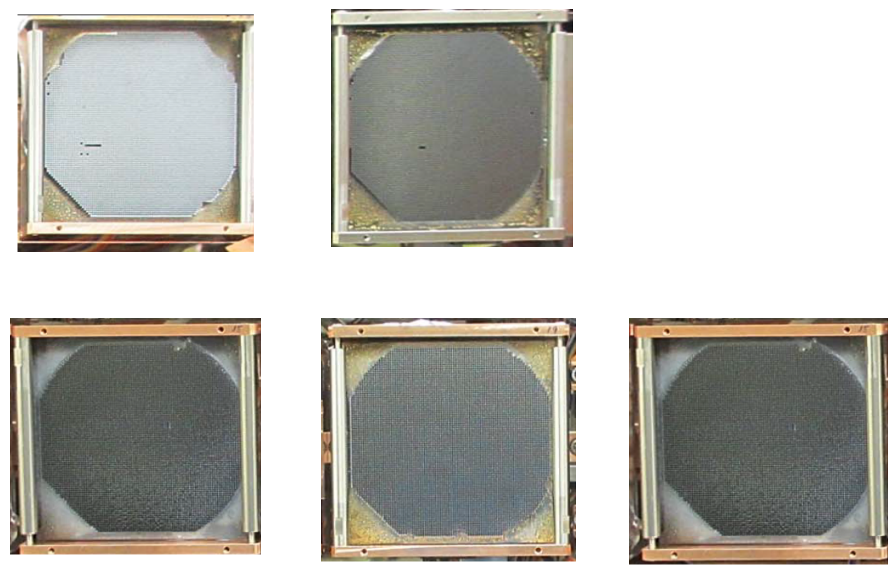


Now in progress: sufficiently flat MCP-> Now <6 urad rms

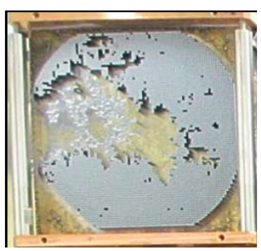
Bonding

- High Temperature Gold diffusion bond
  (not SARTONWORKS)
- Not always stable
- Process variation. Not understood. About 1 in 5 analyzers fails.

Good



Bad



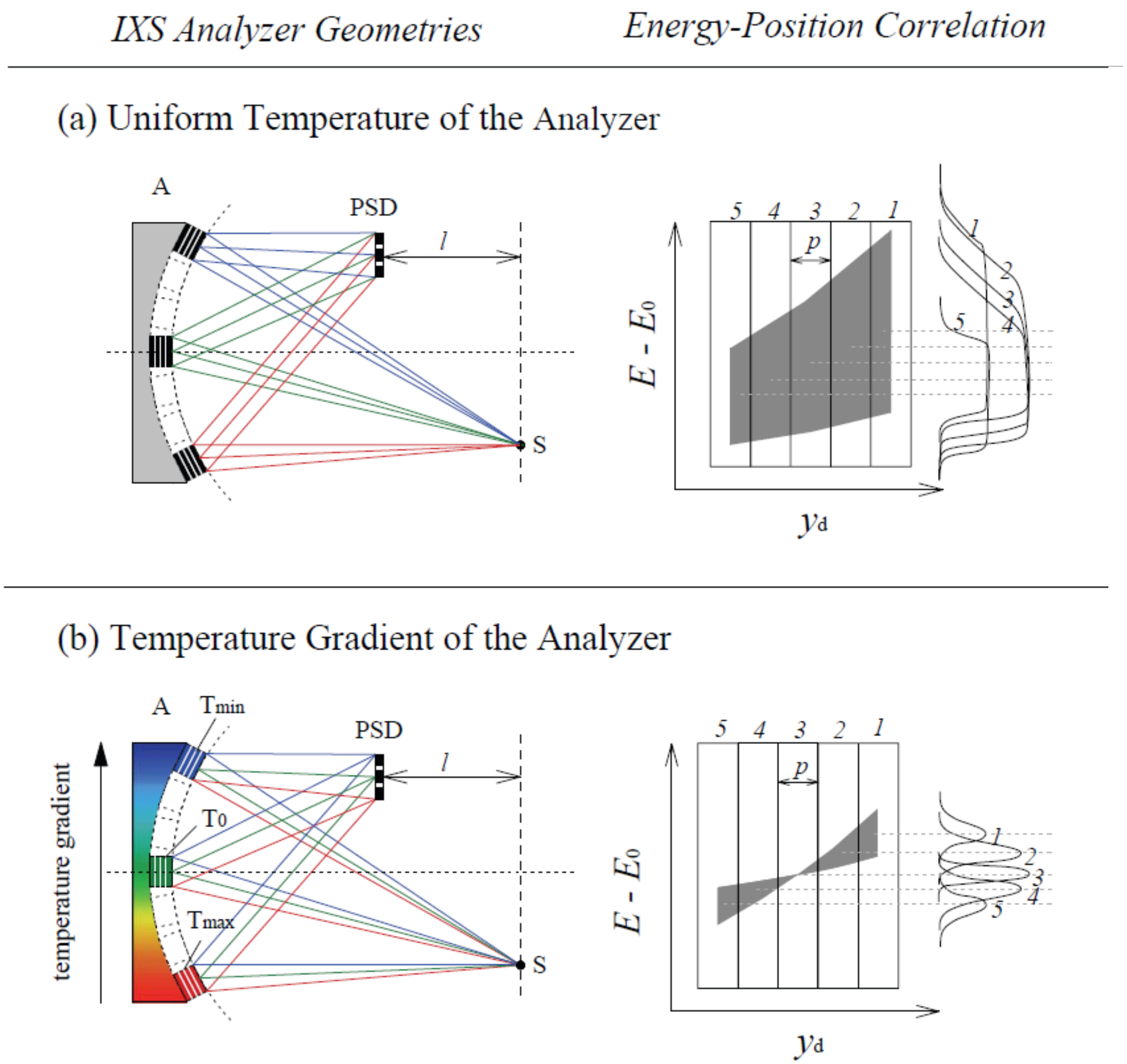
Present status

- NEC. Delivered 6 crystals in 2013
  4 are 1.6 meV or better and good efficiency
  1 poor (1.6 meV and low reflectivity)
  1 Failed due to bonding -> replaced with reasonable crystal (1.5 meV)

- SARTONWORKS relatively reliable after learning period.
  Note: very high transparency compared to NEC.

② Temperature-Gradient

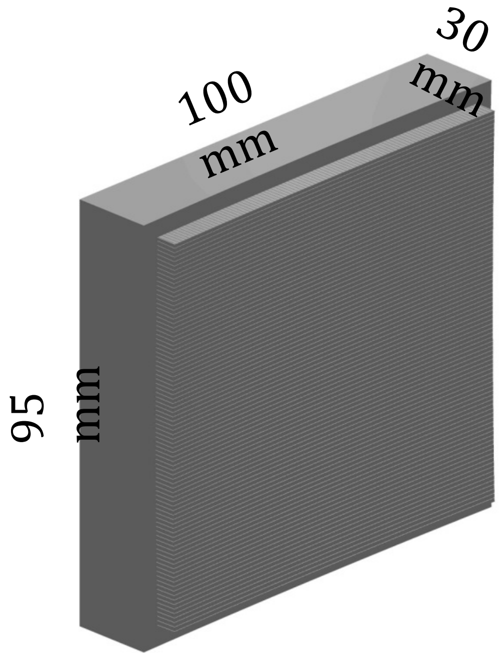
Temperature Gradient Analyzer



- T-gradient Idea -> mostly aimed at small radius 10 meV resolution but works large radius 1 meV resolution
- To obtain large sample area, detector must be moved away from sample
- Deviation from Rowland-condition causes chromatic aberration (demagnification contribution)
- T-gradient can correct Bragg angle of each crystallites and compensate energy-position correlation close to linear

D. Ishikawa and A. Q. R. Baron, J. Synchrotron Rad. (2010). ***17****, 12-24*
Temperature gradient analyzers for compact high-resolution X-ray spectrometers

Rectangular Analyzer T-control



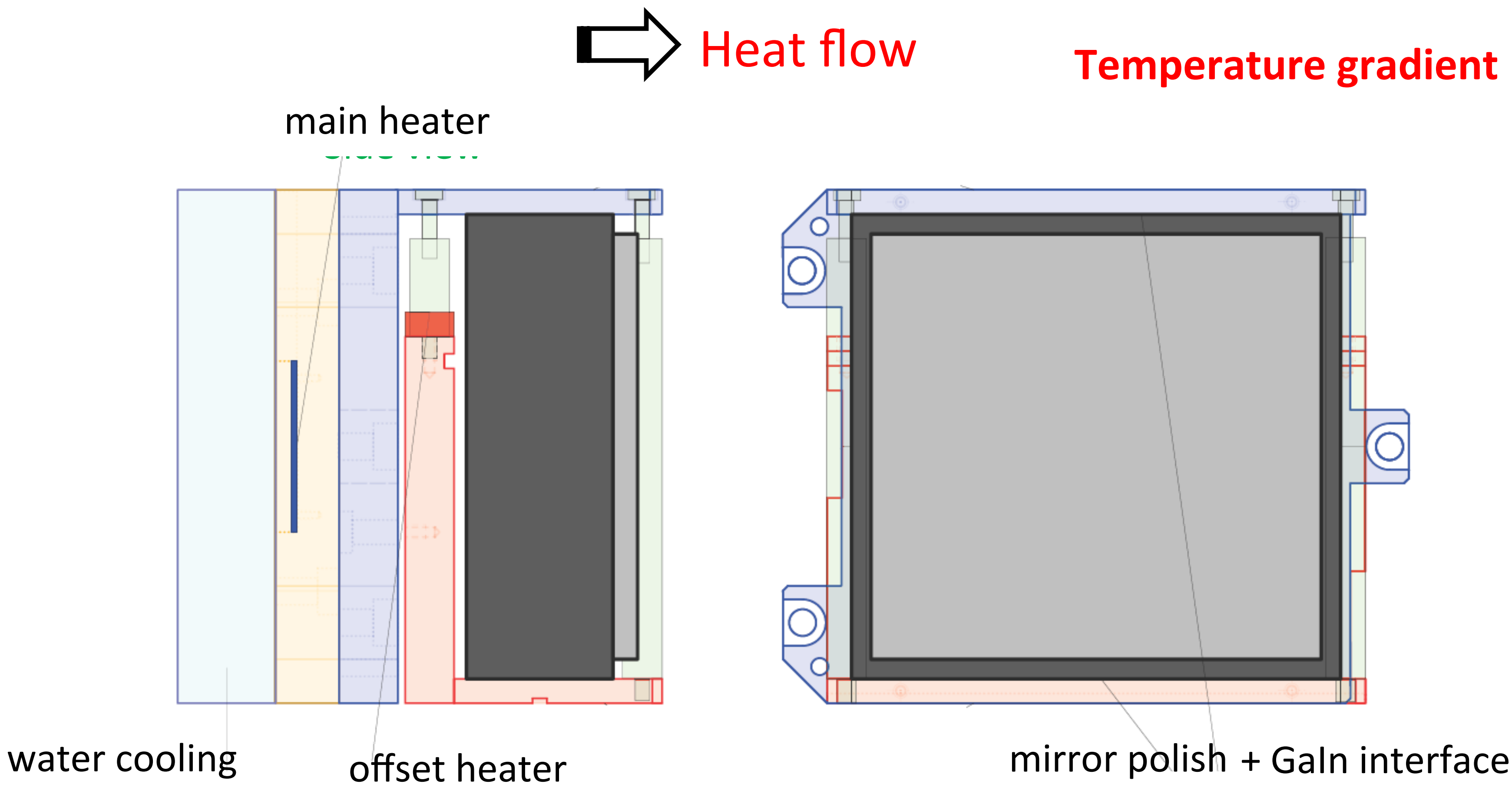
$Q = \lambda \cdot \Delta T \cdot S / l$

- **One-dimensional** temperature gradient
- Temperature *offset* **ΔT = ~ 10 mK**
- Required power **Q = ~ 50 mW**

		Silicon
λ	[W/m·K]	148
S	[mm2]	30 x 100
l	[mm]	95
ΔT	[mK]	10
R	[Ω]	50
V	[V]	1.5
Q	[W]	0.047

- Q : Conductive *heat transfer*
- λ : *Thermal Conductivity*
- S : Heat transfer area
- l : *Heat transfer* distance
- ΔT : Temperature offset

Analyzer Holder (thermal circuit)

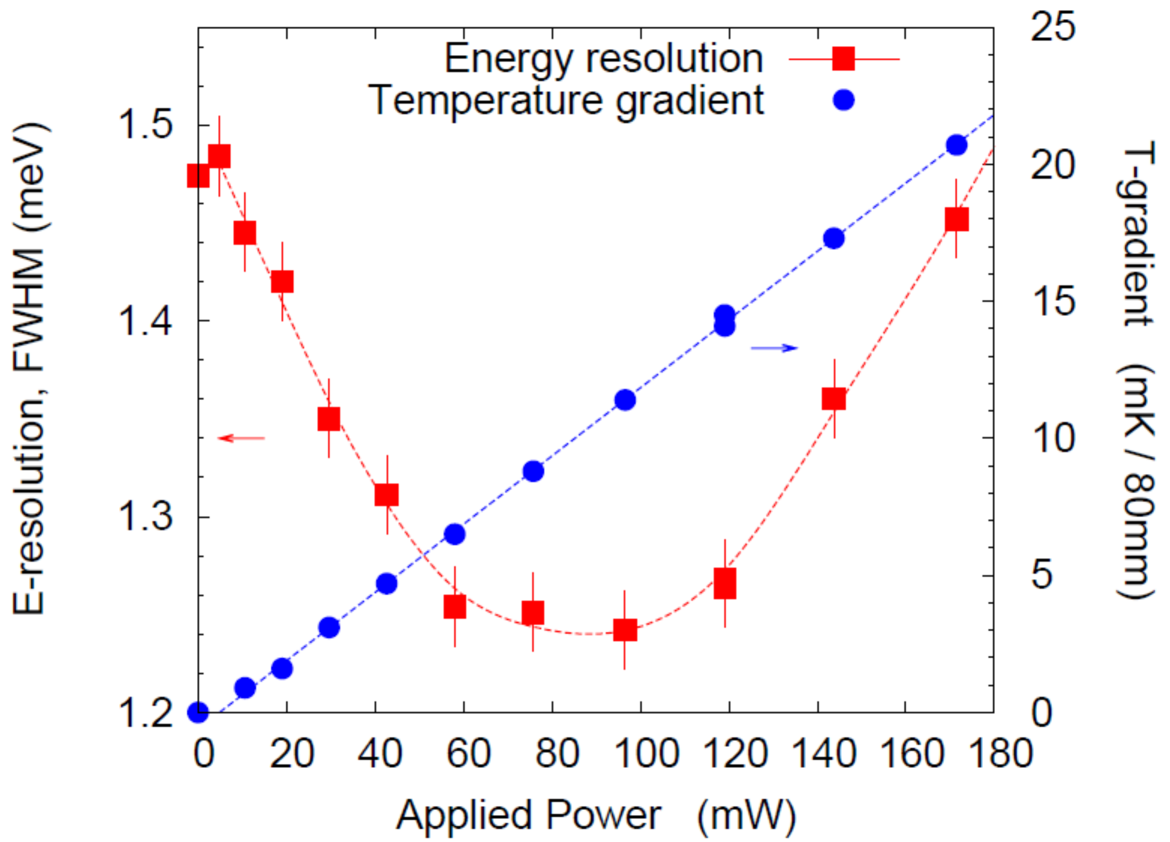


- Both uniform temperature and T-gradient are possible.
- Thermal circuit to do it practically
- After equilibrium state, temperature of each vertical position is fixed and stabilized

T-gradient Application

For High-Resolution Analyzer

- T-gradient can be applied proportionally to applied power
- Resolution has minimum
- Resolution improved from 1.5 to 1.25 meV <Si(11 11 11) reflection>
- For higher-order reflection T-gradient will be more important

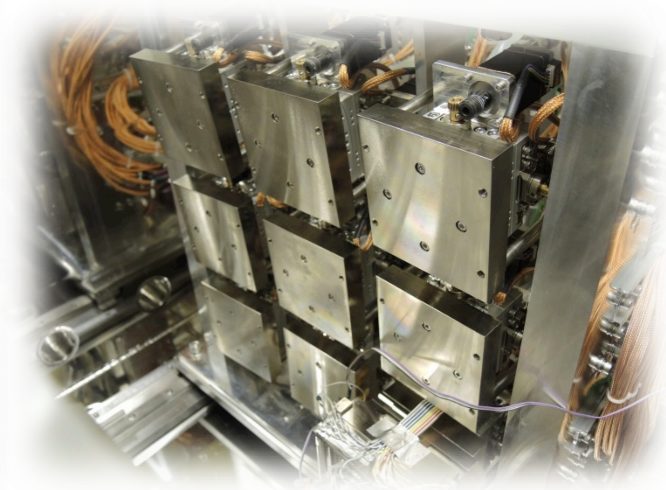


③ Plans for MR analyzers

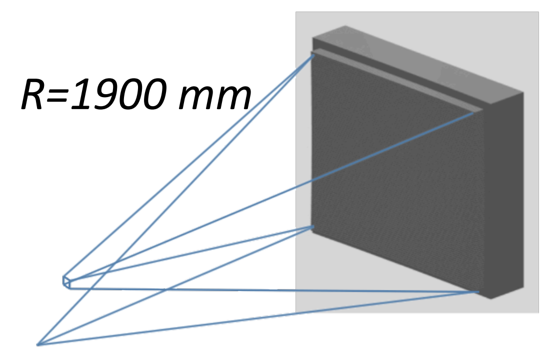
MR-IXS Spectrometer

commissioning

2 m - 2θ arm



3 x 3 Analyzer array

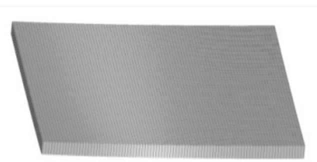


ΔE = 10 meV, E = 15.8 keV
$E\text{-}E_0$ < 100 eV, Q < 15 $Å^{-1}$
Electronic excitation

- Under Development

Plans for MR-IXS analyzers

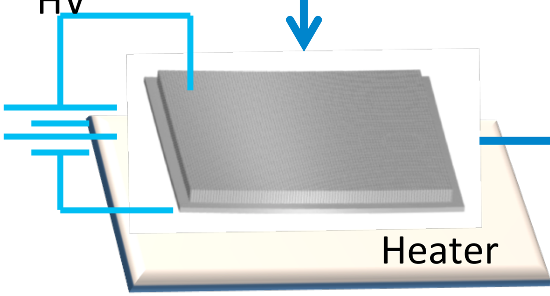
- SARTONWORKS failed
- **Move work in-house** after difficulties with HR-Analyzer
- **Anodic bonding**: Acquiring pieces to do it. (Advice from A. Said)
- Collaboration with JASRI optics group (Ohashi, Goto, et al)



Diced cut Si wafer

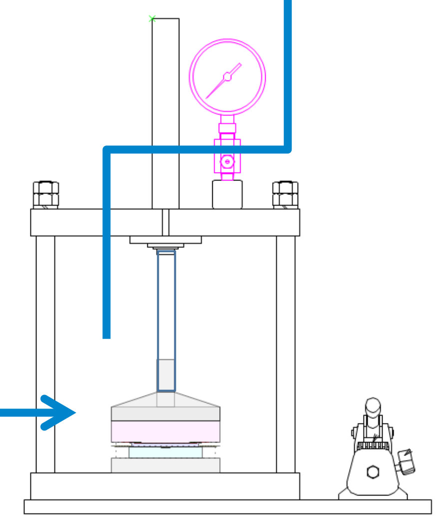
+

Glass wafer

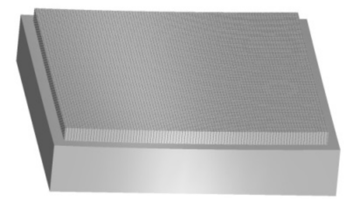


Anodic bonding

Spherical Analyzer substrate
Invar (R=1900)



Forming along spherical surface + Fixing by adhesive bonding

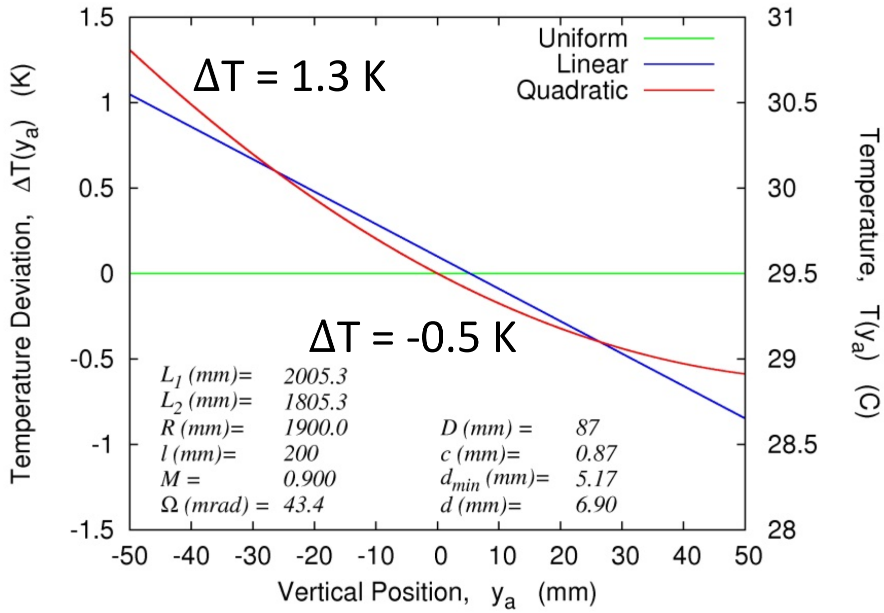


Etching + Polishing

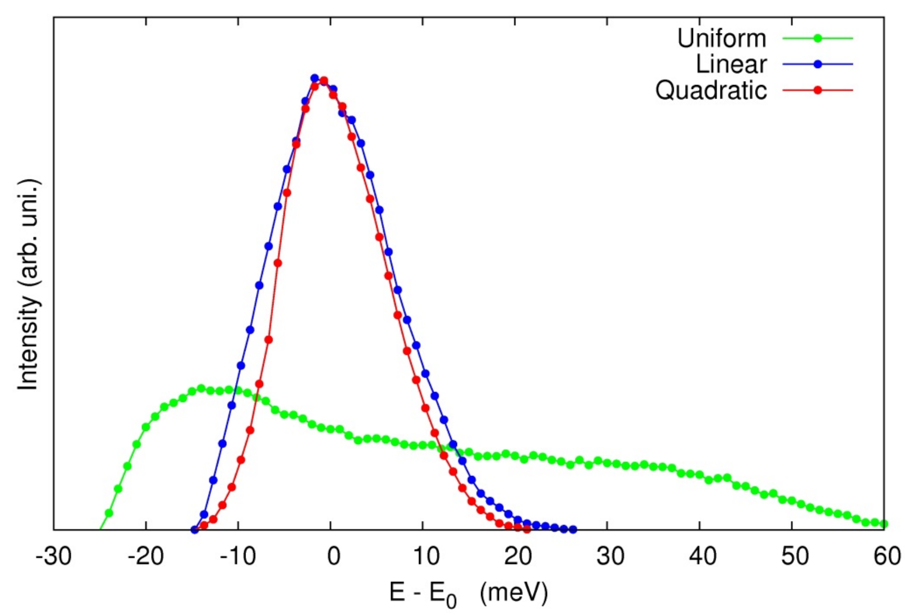
Finish

Simulation of MR-IXS Analyzer

Temperature Gradient



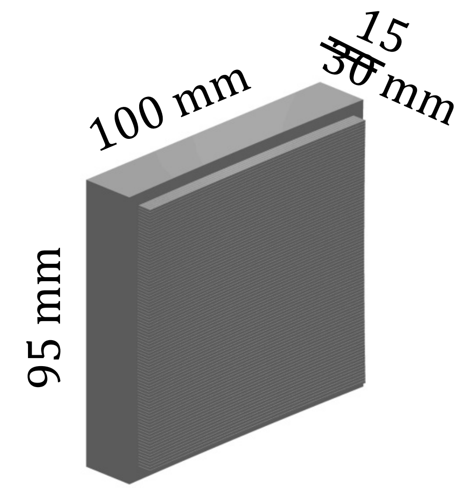
Resolution Function



- Uniform Temperature cannot obtain good energy resolution
- Linear T-gradient reduce energy resolution
- Quadratic T-gradient go to minimum and symmetric line-shape.

MR-IXS Analyzer Substrate

- **ΔT = 1 ~ 6 K** (much higher than HR case)
- **Silicon substrate requires high power** to apply large T-gradient
- High power operation needs much cooling and may cause instability of temperature
- **Low thermal conductive material is needed**



$Q = \lambda \cdot \Delta T \cdot S / l$

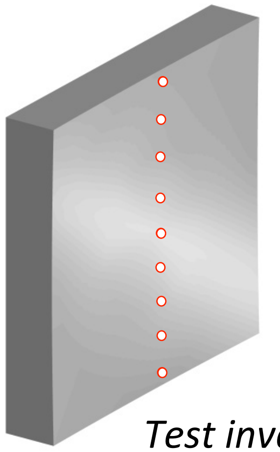
- Q : Conductive *heat transfer*
- λ : *Thermal Conductivity*
- S : Heat transfer area
- l : *Heat transfer* distance
- ΔT : Temperature offset

		Silicon	Invar
λ	[W/ m·K]	148	13.4
S	[mm2]	30 x 100	15 x 100
l	[mm]	95	95
ΔT	[K]	2	2
R	[Ω]	50	50
V	[V]	21	6
Q	[W]	9.3	0.42

< 1/20

MR-IXS Analyzer T-control

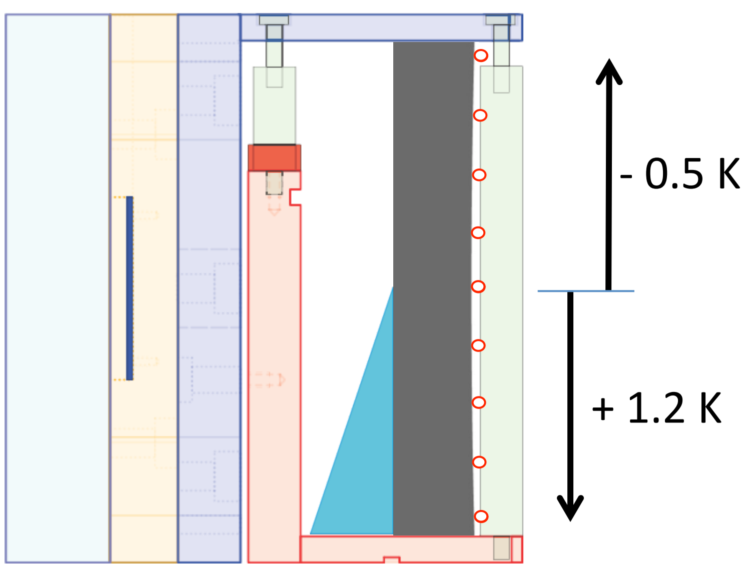
Off-Line test : linear and non-linear T-gradient

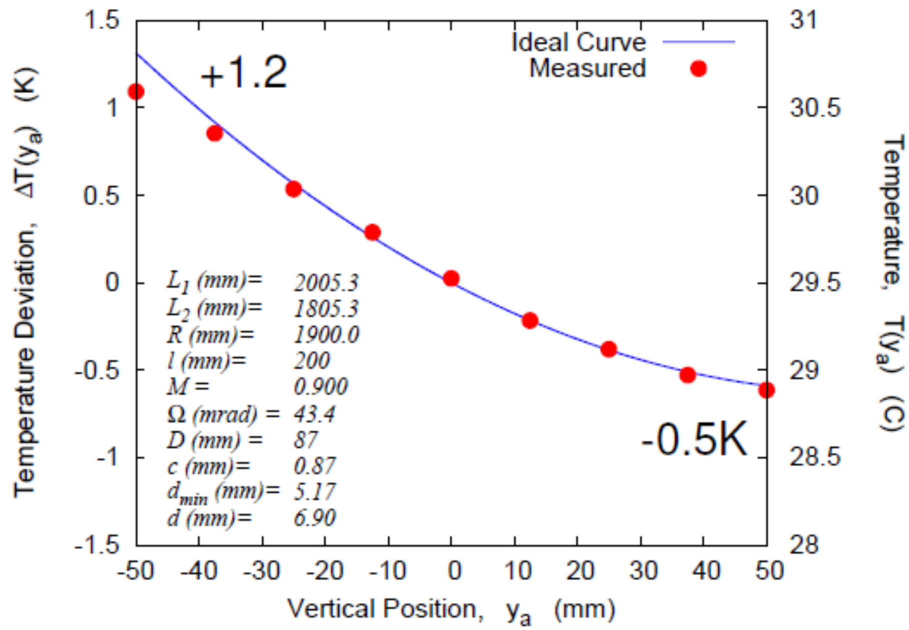


Test invar substrate
106 x 106 x 15 mm

○ T-sensor

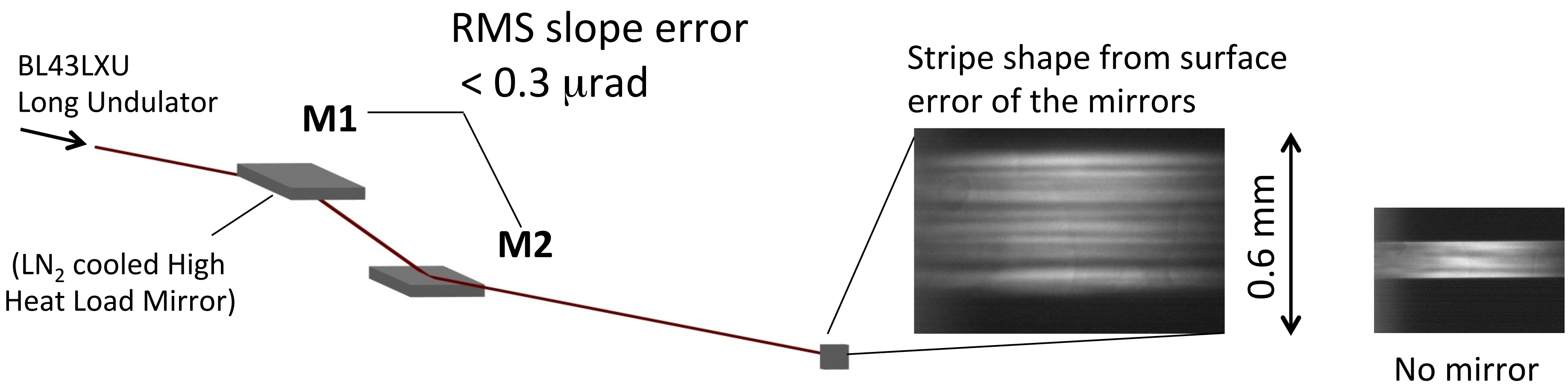
- Linear T-gradient **ΔT = 1 ~ 6 K /100 mm** is possible
- In reality, **quadratic T-gradient** is more desirable.
- Non-linear T-gradient can be controlled by changing thermal flow rate

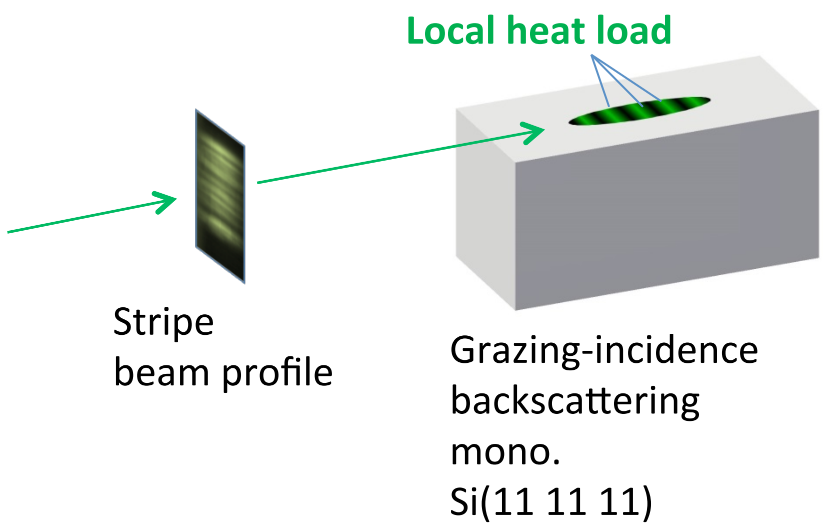




④ Heat Load from Mirrors

Heat Load from Mirrors

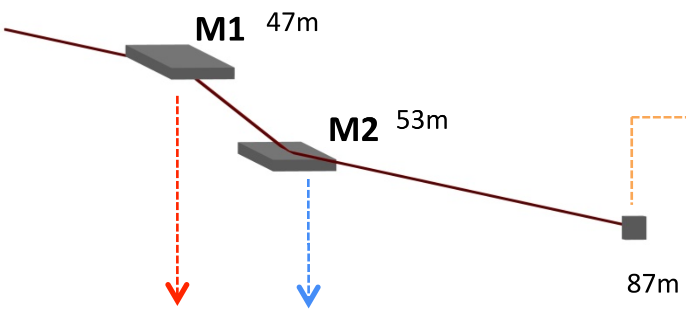




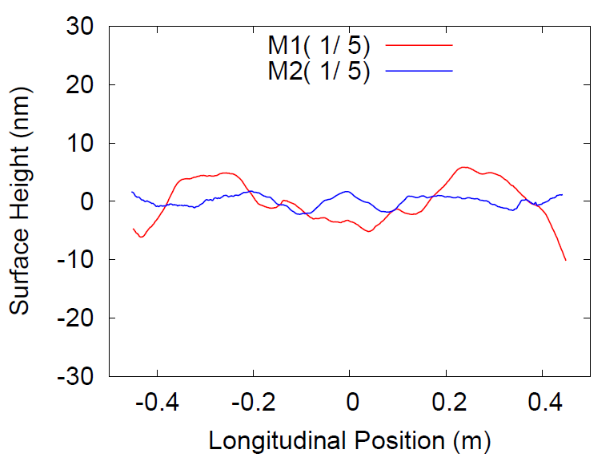
- For collimating mirrors, surface error is more severe than focusing mirror
- **Local heat load** makes resolution **worse !!**
  1.5 meV ---> 1.7 – 3 meV (FWHM)
- To reduce heat load onto downstream optics (Backscatt. Mono.) grazing incidence is used, but it is not enough
- **Additional mirror polishing is needed**

Allowable Surface Error of Mirrors

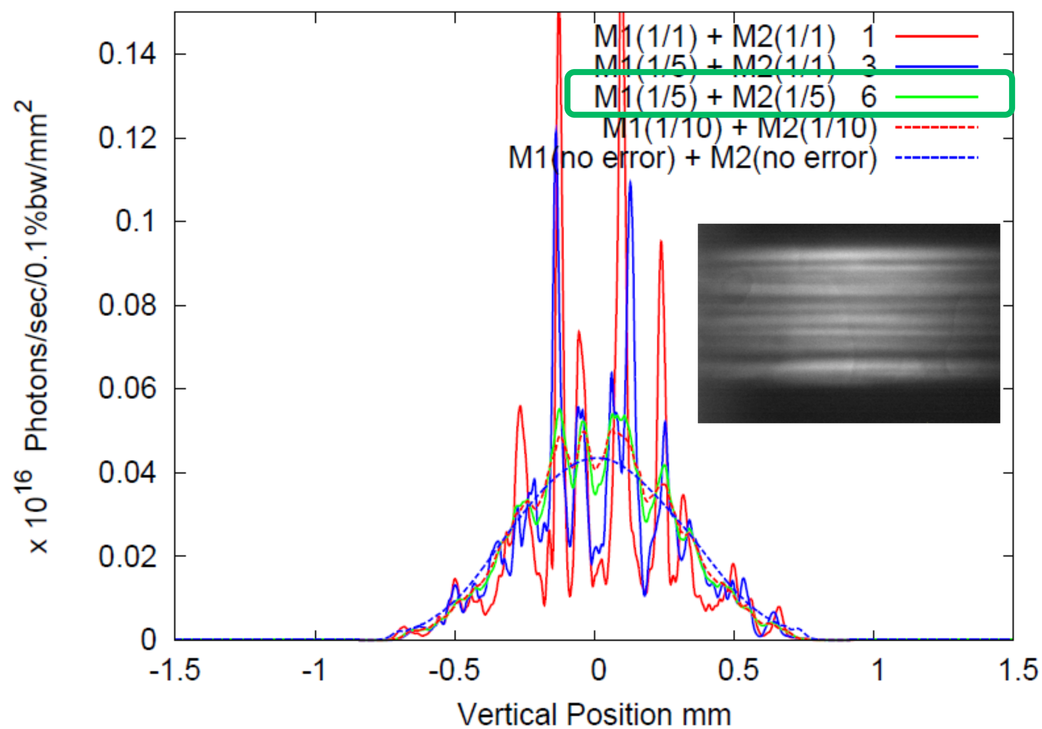
- How much must I polish present mirrors ?
- Calculations by SRW were performed
- Both mirror must be polished



surface error



beam profile (vertical plane)



Used by SRW, Tchoubar, Oleg, Simulating emission and propagation of partially-coherent undulator radiation

Summary

BL43LXU High-Resolution Crystal Optics

- Present status of HR-Analyzer (R9800)
- Concept of T-gradient optics and it's application
- Plans of MR-Analyzer (R1900)
- Heat load on high-resolution monochromators coming from collimating mirrors

Collaborators

BL43LXU (RIKEN Quantum Nano Dynamics Beamline)

- BL Commissioning

  Materials Dynamics Laboratory, RIKEN SPring-8 Center

  A. Q. R. Baron, H. Uchiyama

- MR-Analyzer in-house fabrication

  JASRI optics group

  (H. Ohashi, S. Goto, H. Yamazaki, Y. Shimizu, T. Miura)

- Mirror performance

  JASRI optics group

  (H. Ohashi, H. Yamazaki, Y. Senba)

Thank you for your attention.