Double Crystal Monochromator Workshop
ESRF, Grenoble, 13 and 14 May 2014

ESRF | The European Synchrotron

Thermal, mechanical deformation and stability of Monochromator Crystals under high heat load

Outline

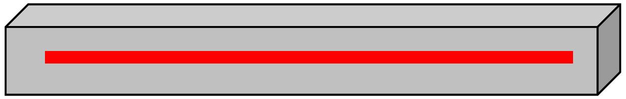
- **Cooling strategy**
  - Water or LN2, direct or indirect, crystal size and cooling scheme
- **Crystal material, properties**
  - Crystals, Silicon, doping, anisotropic elasticity, pure isotope
- **Thermal deformation**
  - Finite element analysis (FEA)
  - Measurement techniques
  - Comparison between measurements and simulations
  - Some extended FEA results
    - Power and power density, beam size, grazing angle, cooling coefficient,…
    - Focusing effects
- **Initial deformation of the crystal**
  - Manufacturing, mounting, and cooling down to LN2 temperature
- **Stability and vibration**
  - Some measurement results

Cooling strategy: mirror and monochromator

Thermal deformation - comparison of mirror and monochromator

- **For an incident beam at 30 m**
  - Power density P_{a0}=200 W/mm^2
  - Beam size HxV=2x1 mm^2

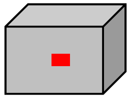
White beam mirror



typical length: 1000 mm

- typical **grazing angle**: 2 mrad
- footprint: 500 ~ 1000 mm
- Power density P_a ~ 1 W/mm^2
- **Topside cooling by water**

Monochromator crystal

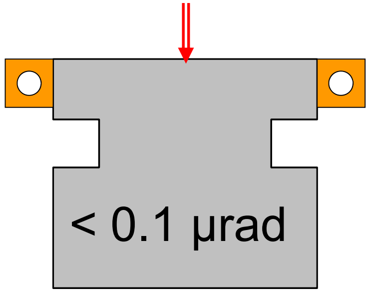


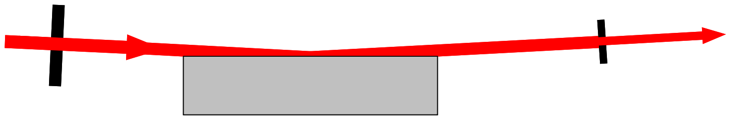
typical length: 100 mm

- typical **Bragg angle**: 12° (209 mrad)
- footprint ~ **1%** as long as for mirror
- Power density P_a ~ 50 W/mm^2
- **Cooling scheme ?**

Cooling strategy: white beam mirror

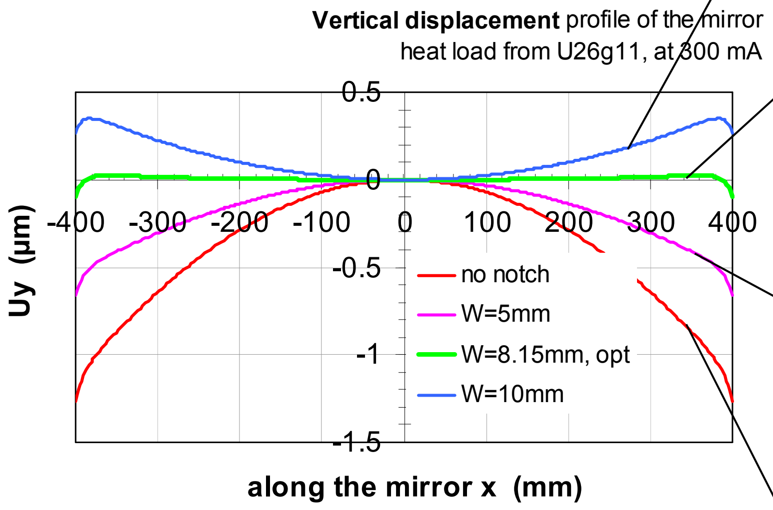
Solution to minimize thermal deformation for white beam mirror (smart shape + full illumination):

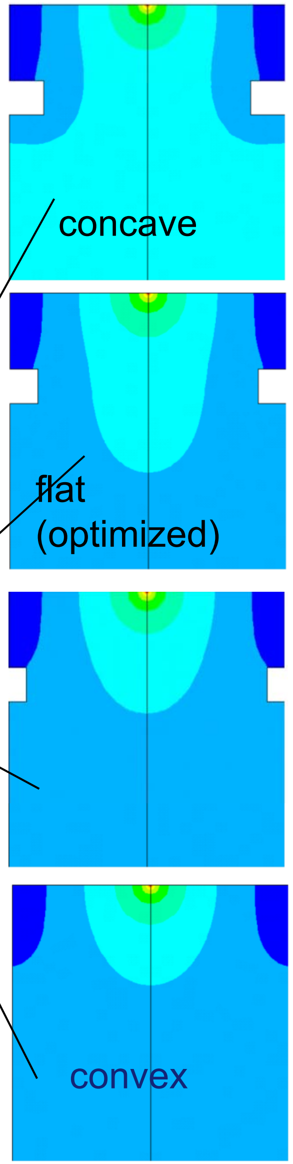




ESRF UPBL06 (ID16)

- Mirror size: 800x80x80
- Grazing angle 3.1 mrad
- 2~3 coatings
- Heat load: **834 W**
  Gaussian distribution
  σ =3.86 mm


Vertical displacement profile of the mirror heat load from U26g11, at 300 mA



Zhang L. et al., J. Phys.: Conf. Ser. ***425*** *(2013) 052029*
doi:10.1088/1742-6596/425/5/052029

Cooling strategy: mirror and monochromator

Can monochromator crystal be cooled as mirror (full illumination) ?

White beam mirror

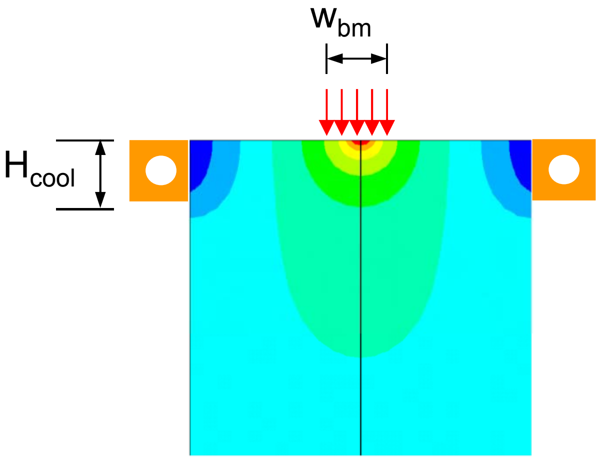
$P_a = 1\ \mathrm{W/mm^2}$

$W_{bm}=2\ \mathrm{mm}$, $H_{cool}=10\ \mathrm{mm}$

$h_{cv\text{-}eq} = 5000\ \mathrm{W/m^2/°C}$

$$\overline{\Delta T_{\min}} = \overline{T_{\min}} - T_f = \frac{P_a}{h_{cv-eq}} \frac{W_{bm}}{2H_{cool}}$$

$$\overline{\Delta T_{\min}} = 20°C$$



For Monochromator crystal

$P_a \sim 50\ \mathrm{W/mm^2}$

$$\rightarrow \overline{\Delta T_{\min}} = 1000°C \quad \text{!!!}$$

Impossible to cool the Monochromator as the mirror

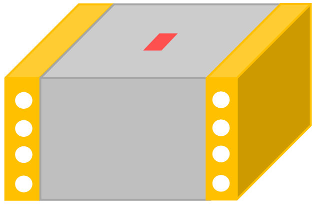
Cooling strategy for monochromator crystal

Full side cooling (to increase cooling surface area)

- $P_a \sim 50\ W/mm^2$
- $W_{bm}=2$ mm, $L_{bm}=5$ mm,
- $H_{cool}=t_{mono}=50$ mm
- $L_{cool}=L_{mono}=100$ mm >> L_{bm}
- $h_{cv\text{-}eq} = 5000\ W/m^2/^{\circ}C$

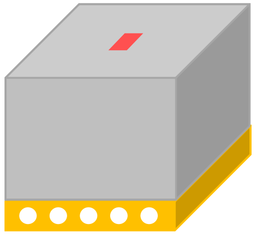
$$\overline{\Delta T_{\min}} = \frac{P_a * W_{bm} * L_{bm}}{h_{cv-eq} * 2 * H_{cool} * L_{cool}}$$

$$\overline{\Delta T_{\min}} = 10^{\circ}C$$



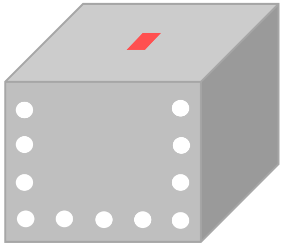
Cooling of monochromator needs crystal significantly longer than beam footprint

Bottom cooling



- Cooling surface area reduced

Direct cooling



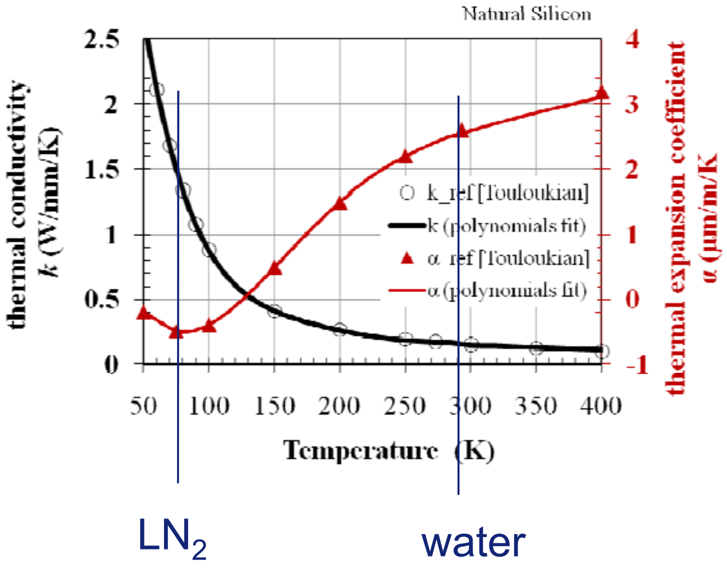
- No thermal contact resistant
- Sealing difficulty
- Sealing induced stress and deformation

Crystal material, Properties of Si

Materials for Monochromator crystal

- **Silicon:**
  - Perfect crystal
  - Large size Φ100x500
  - Very reasonable price (900€/kg, 9000€ for Φ100x500)
  - Interesting properties at low temperature
- **Germanium**
  - Less perfect
  - Medium size Φ100x75
  - 4 ~ 40 times more expensive than Si
- **HPHT Synthetic Diamond**
  - Imperfect
  - Small size 10x10x1
  - Expensive

Thermal slope : $\Delta\theta \propto \frac{\alpha}{k}$



$$\frac{\frac{\alpha}{k}(T=295K)}{\frac{\alpha}{k}(T=77K)} \approx 100$$

Anisotropic elasticity of Si

- **Silicon: cubic diamond crystal structure**
- **Stiffness coefficient matrix**
  - 3 three independent elastic coefficients for Si (100)
  - Can be calculated for any crystallographic orientation
    - Analytically
    - By codes (MatLab, Python)

$$C_{100} = \begin{bmatrix} c_{11} & c_{12} & c_{12} & & & \\ c_{12} & c_{11} & c_{12} & & & \\ c_{12} & c_{12} & c_{11} & & & \\ & & & c_{44} & & \\ & & & & c_{44} & \\ & & & & & c_{44} \end{bmatrix}$$

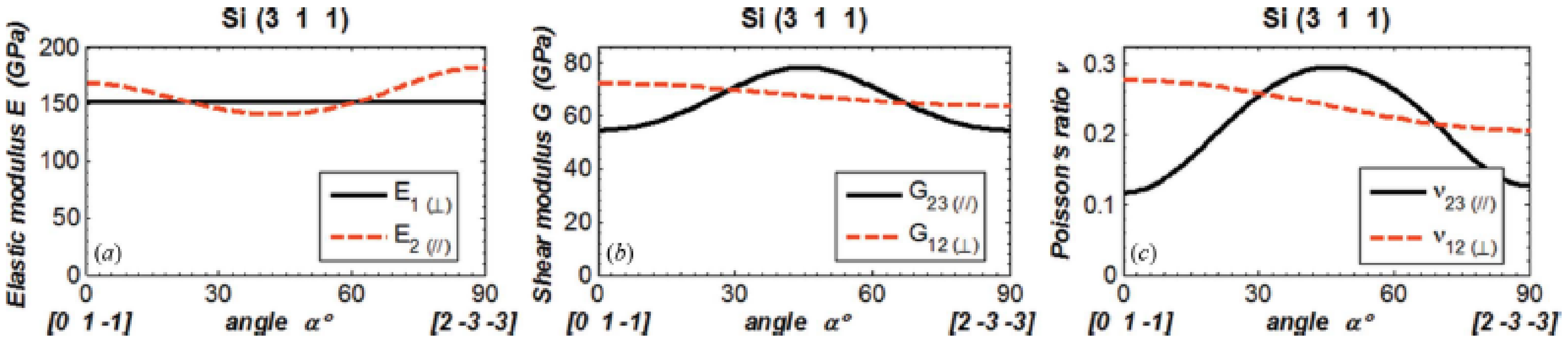
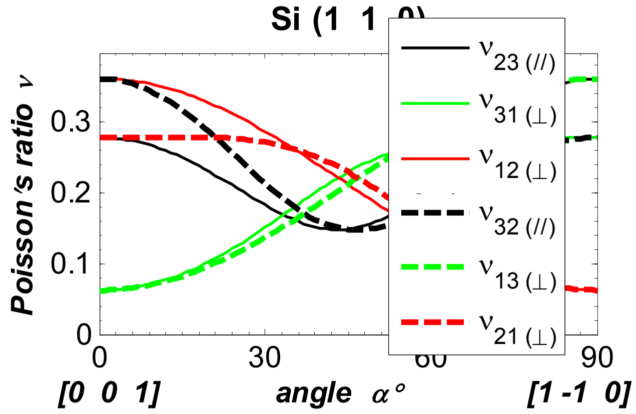


Figure 6
(a) Elastic modulus in the directions $\mathbf{e}_1'$ and $\mathbf{e}_2'$. (b) Shear modulus and (c) Poisson's ratio in the directions 12 and 23 for silicon (311). The vector $\mathbf{e}_1'$ is fixed in the normal direction [311], and the vectors $\mathbf{e}_2'$ and $\mathbf{e}_3'$ are in the crystal plane (311). The angle α is between the vectors $\mathbf{e}_2'$ and $[0\ 1\ -1]/2^{1/2}$ in the crystal plane: $\mathbf{e}_2'(\alpha=0°) = [0\ 1\ -1]/2^{1/2}$, $\mathbf{e}_2'(\alpha=90°) = [2\ -3\ -3]/(22)^{1/2}$.

- Important for bent silicon crystal
- For thermal deformation ?

L. Zhang et al., J. Synchrotron Rad. (2014). ***21****, 507–517*

Anisotropic elasticity of Si



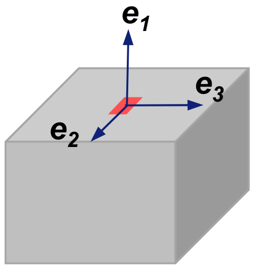
➢ Thermal deformation

- Depends on the Poisson's ratio:

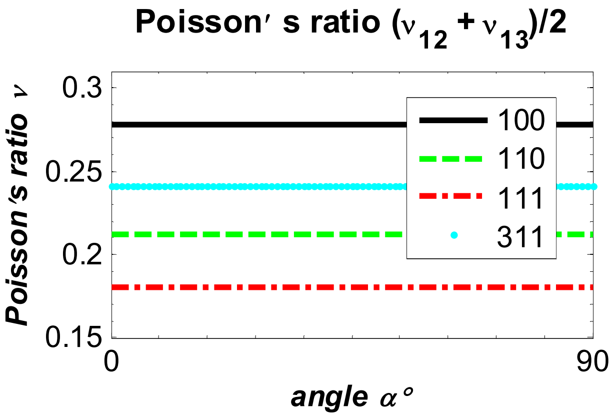
$$\Delta\theta \propto (1+\nu)\frac{\alpha}{k}$$

- Poisson's ratio depends on the crystal orientation
- Thermal slope error

$$\Delta\theta = \frac{\partial u_1}{\partial x_2} \propto (\nu_{12}+\nu_{13})/2$$



- But the average $\nu_{av} = (\nu_{12} + \nu_{13})/2$ is constant



➢ Thermal deformation with anisotropic elasticity of silicon → Simulation with isotropic and constant elasticity (ν_{av})

L. Zhang et al., J. Synchrotron Rad. (2014). ***21****, 507–517*

Si related Crystal material: Germanium doped silicon

- **Germanium doped silicon Si_{100-x}-Ge_x($x \le 2\%$)**
  - Ge doping decreases dislocation mobility, and modifies dislocation nodes in Si crystalline lattice

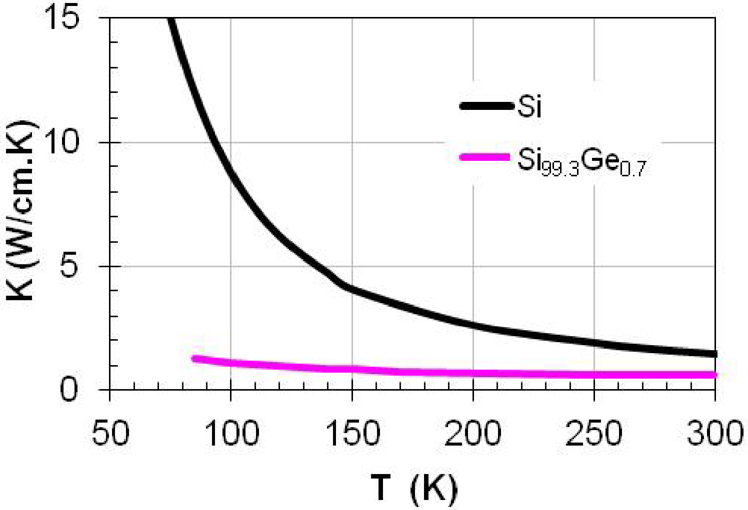
  →Increasing semi-conducting device efficiency: material strength, current carrier mobility

  - Application to DCM: Si_{100-x}-Ge_x for 1st crystal (LN_2), Si for 2nd crystal (water)
    - Vegard's law: $$\Delta d / d_{Si} = \mu x$$ ($\mu = 4.18 \times 10^{-4}$)
    - Concentration x ~ 0.7%

A. Souvorov and A. Snigirev, Rev. Sci. Instrum. ***68****, 1997*

- Ge doping reduces dramatically the thermal conductivity of Si especially at LN2 temperature

→Therefore the application of Si-Ge crystals to cryogenic cooling cannot be recommended



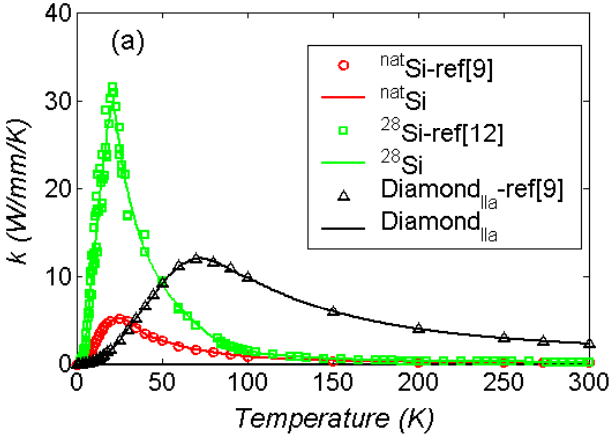
A. Freund, J.A. Gillet & L. Zhang, Proc. SPIE ***3448****, (1998); doi:10.1117/12.332526*

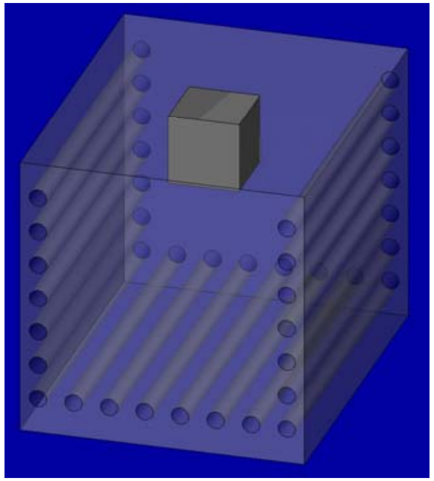
Si related Crystal material: pure isotope silicon

- **Three stable isotopes in natural silicon:**
  - Silicon-28 : 92%
  - Silicon-29 : 4.7%
  - Silicon-30 : 3.3%

Single-isotope silicon-28 crystal (99.9%)

- **Very high thermal conductivity (k = 30 000 W m^{-1} K^{-1} at 20 K, 6 times higher than natural Si)**
- **Available, used in semiconductor industry**
- **Small size, very expensive**
- **Technology challenge for effective cooling**
  - Huge size and high cost of cooling system for 500 W cooling power



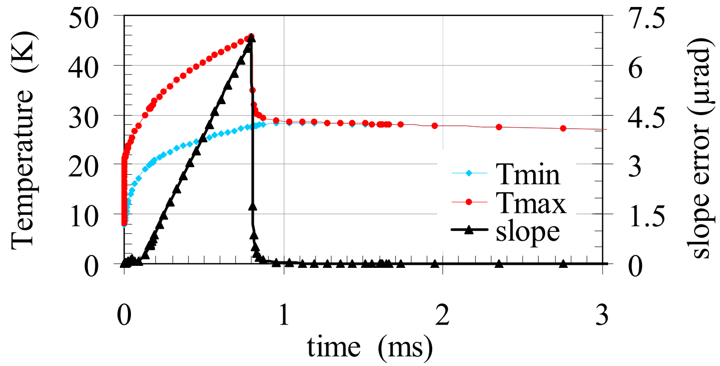


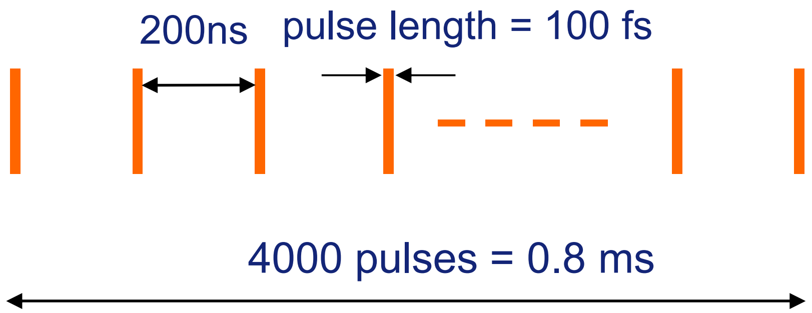
*L. Zhang et al., AIP Conference Proceedings, **705**, pp.639-642 (2003)*

^{nat}Si, ^{28}Si and Diamond for very high heat-load monochromator

Macro-pulse train effects (f=10Hz)

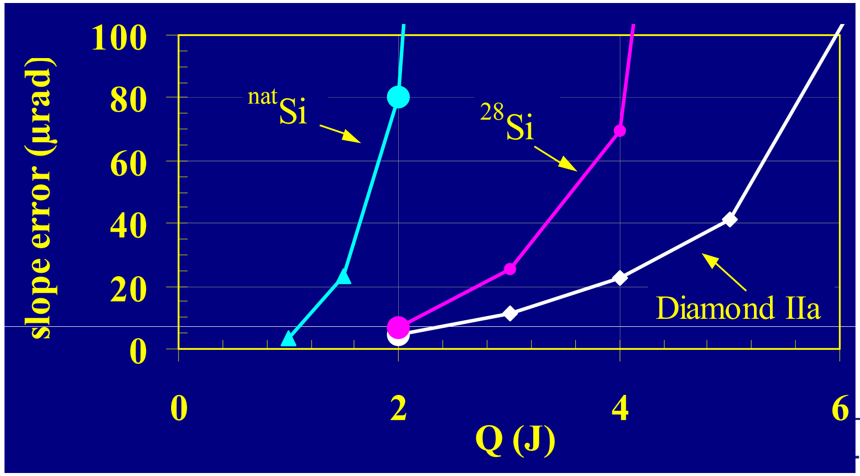
- LN2 cooled diamond crystal (20mm x 20mm x 20mm)
- **LHe cooled single-isotope silicon-28 crystal** (20mm x 20mm x 20mm)
- LHe cooled natural silicon crystal (120mm x 60mm x 60mm)





200ns pulse length = 100 fs

4000 pulses = 0.8 ms



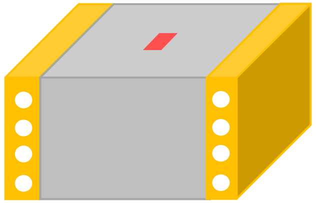
Time-structure proposal of TESLA X-FEL
(repetition rate 10 Hz)

*L. Zhang et al., AIP Conference Proceedings, **705**, pp.639-642 (2003)*

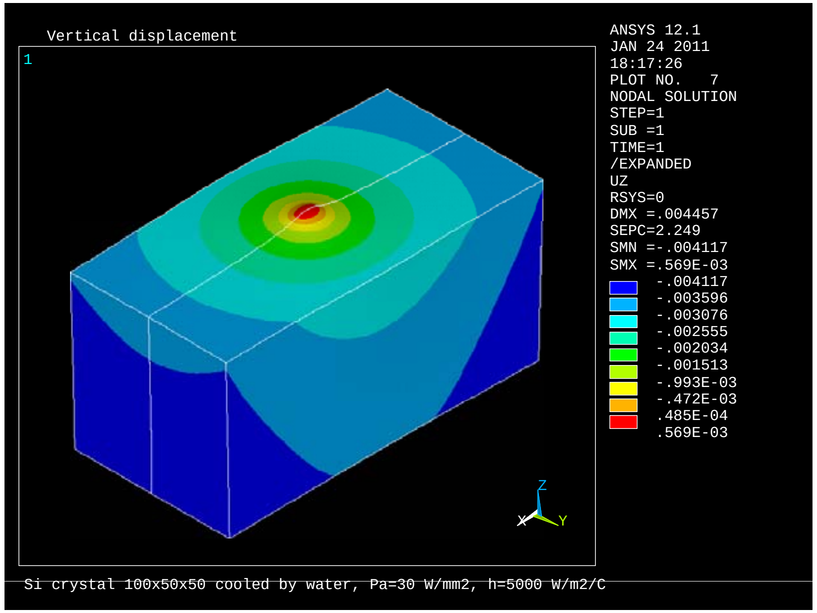
Thermal deformation of the monochromator crystal

For monochromator crystal

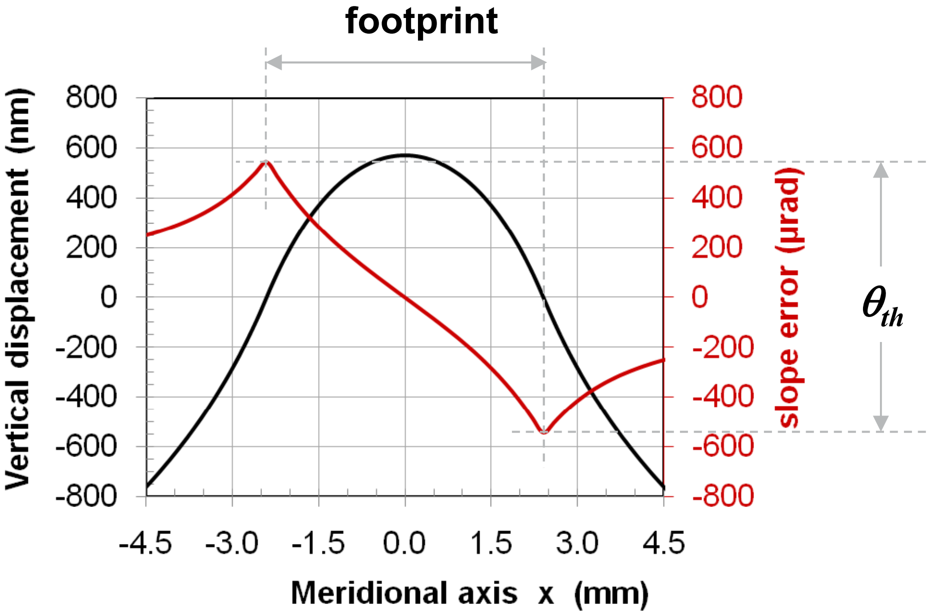
- 3D temperature and deformation
- Non-linear material properties (k, α)
- Finite Element Analysis (FEA) for the modeling



Example of water cooling

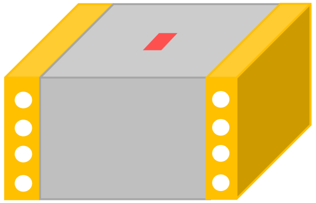


```
Vertical displacement
1
ANSYS 12.1
JAN 24 2011
18:17:26
PLOT NO.   7
NODAL SOLUTION
STEP=1
SUB =1
TIME=1
/EXPANDED
UZ
RSYS=0
DMX =.004457
SEPC=2.249
SMN =-.004117
SMX =.569E-03
     -.004117
     -.003596
     -.003076
     -.002555
     -.002034
     -.001513
     -.993E-03
     -.472E-03
     .485E-04
     .569E-03
Si crystal 100x50x50 cooled by water, Pa=30 W/mm2, h=5000 W/m2/C
```

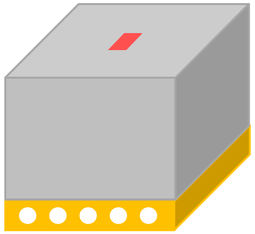


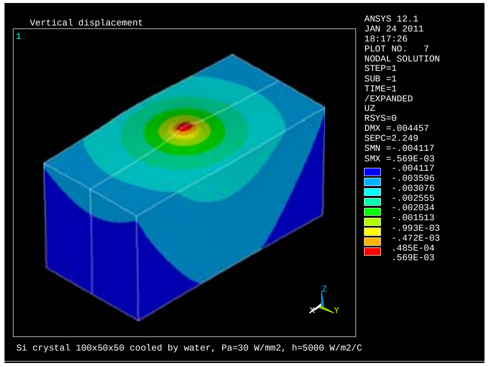
Thermal deformation : side cooling versus bottom cooling

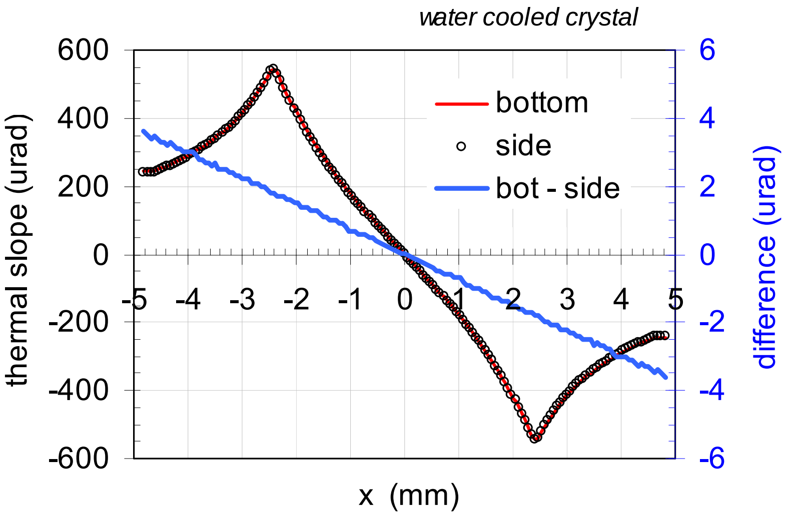
Side cooling **(by water)** **bottom cooling**

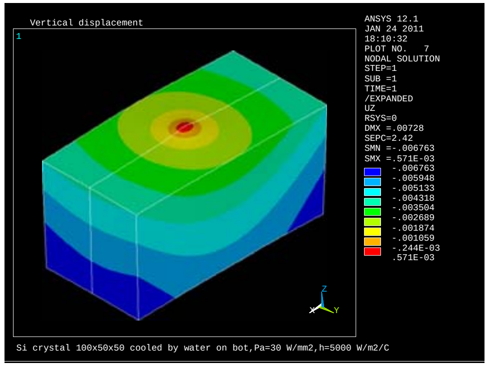


- **Similar temperature distribution**
  but low temperature with side cooling
- **Very comparable thermal deformation**:
  0.7% lower thermal deformation with side cooling
  → Thermal bump deformation predominant !





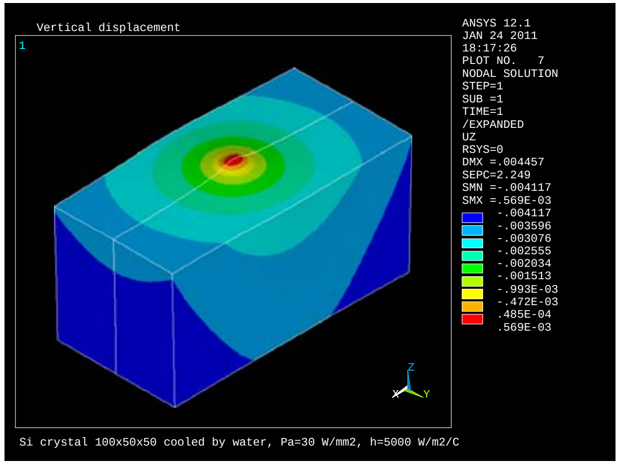




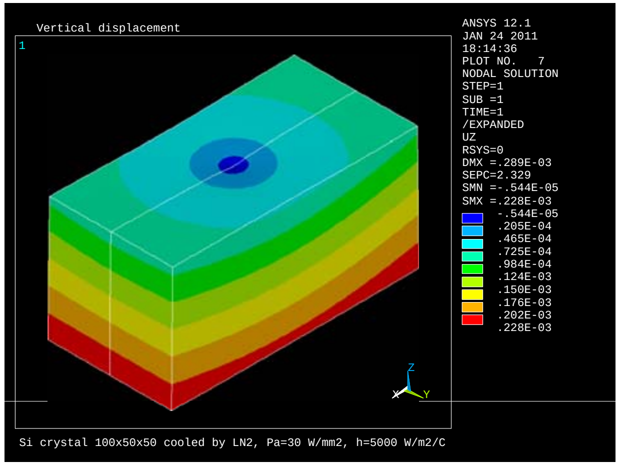
How to reduce this huge thermal slope error θ_{th}= 1085 µrad ?

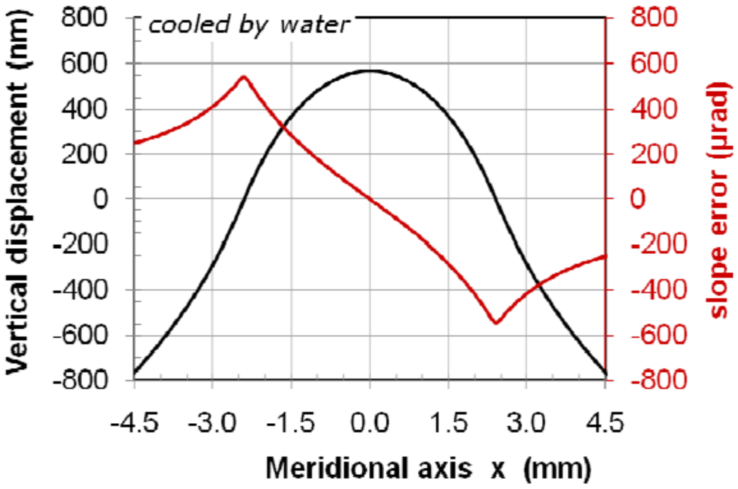
Thermal deformation : water cooling versus LN2 cooling

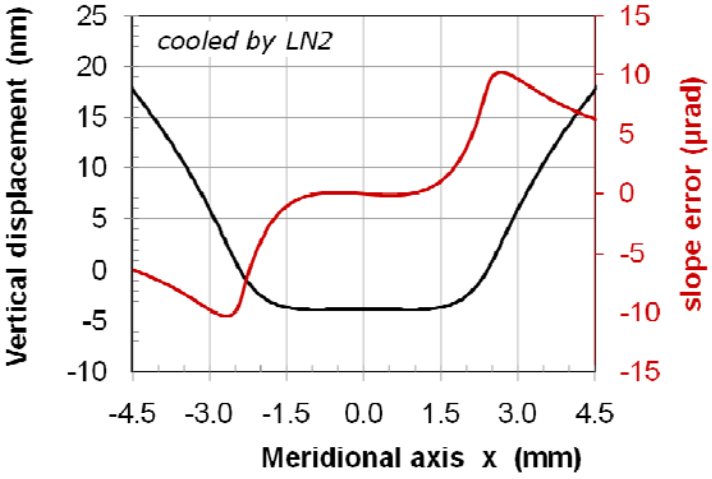
Water cooling vs LN2 (Liquid Nitrogen) cooling



For LN2 cooling
θ_{th}=18 µrad
θ_{RMS}=3 µrad
Reduced by a factor of 61 for θ_{th}
(96 for θ_{RMS})



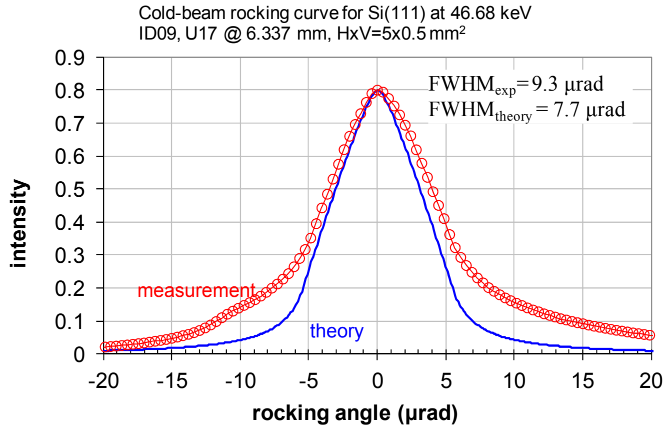




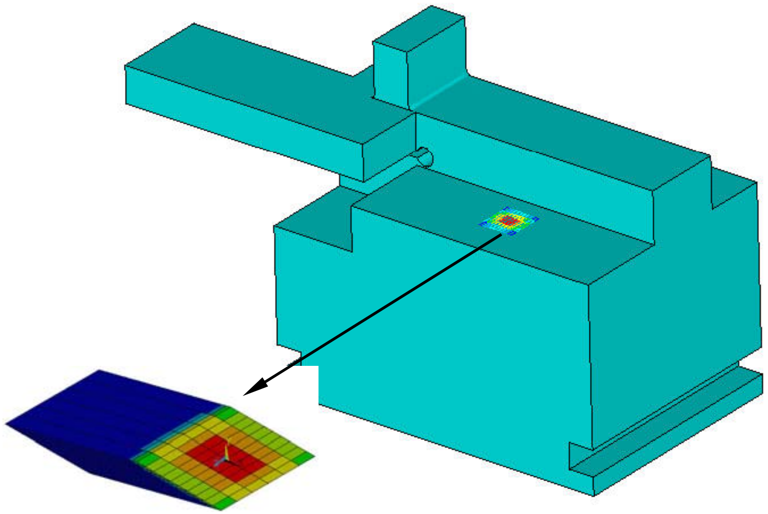
Thermal deformation: indirect measurement technique

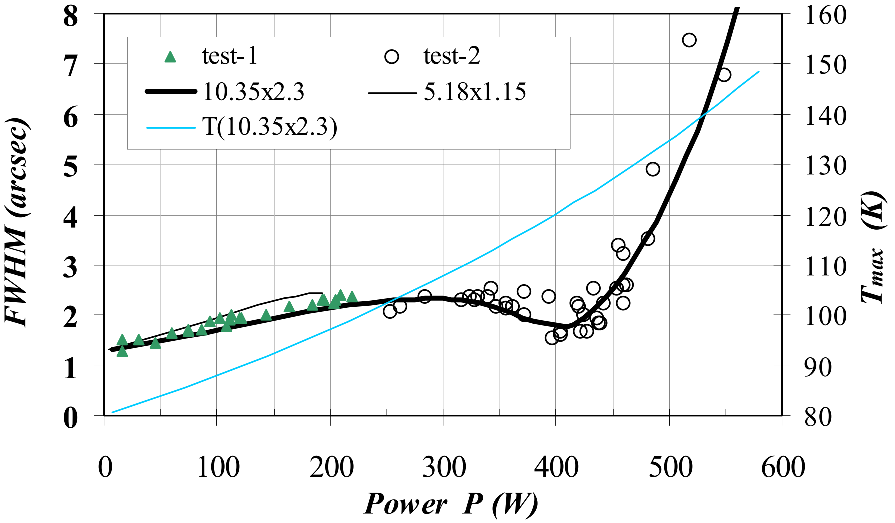
- **Thermal deformation → Rocking-curve broadening**
- **Rocking-curve width:**

$$FWHM_c = \sqrt{(\theta_{th} + \theta_0)^2 + FWHM_{\text{intr}}^2}$$



- Comparison of **test and FEA results** for ID09 LN2 cooled Si crystal (Channel-Cut Monochromator)



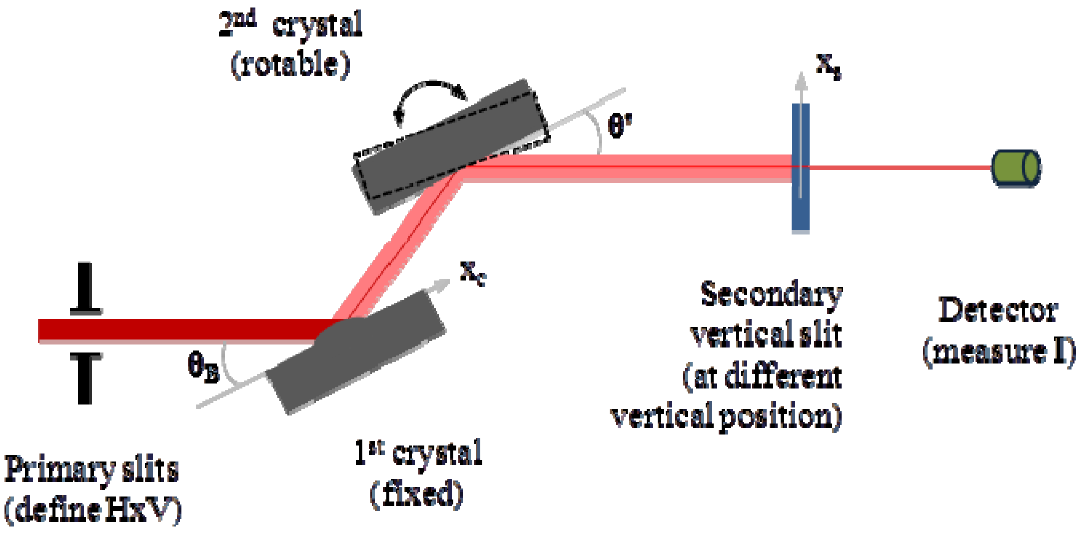


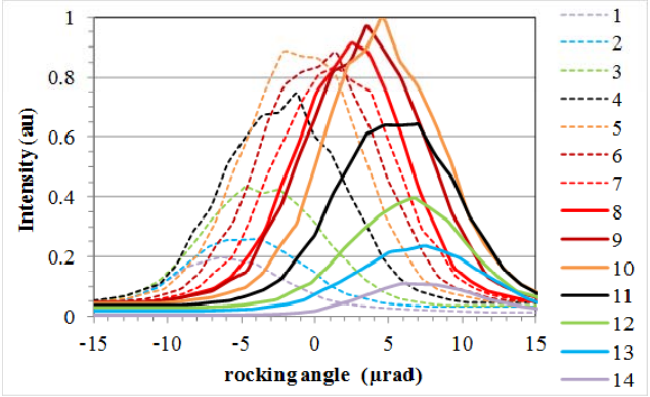
*Zhang L. et al., JSR (2003). **10**, 313-319*

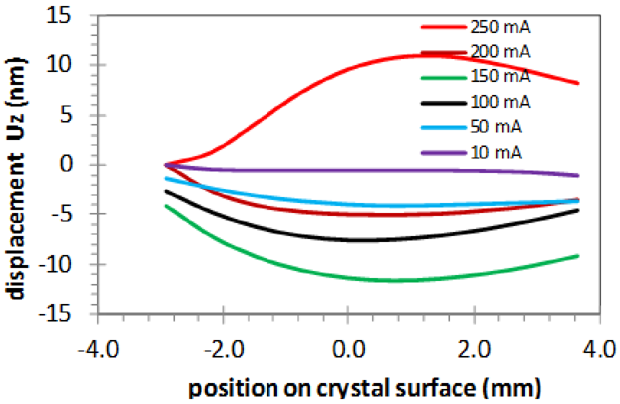
Thermal deformation: direct measurement technique

Applied to ID06, ID18 and ID26 LN2 cooled Si crystal

- **Multiple angular scans across the Bragg peak (rocking curve) at various vertical positions of a narrow-gap slit downstream from the monochromator**







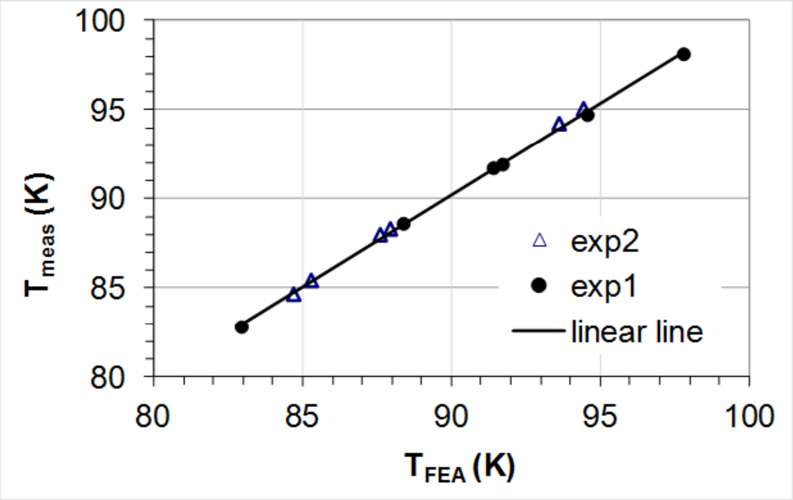
- **Thermal slope:** $\Delta\theta_{th} = \theta'_{peak} - \theta_B$

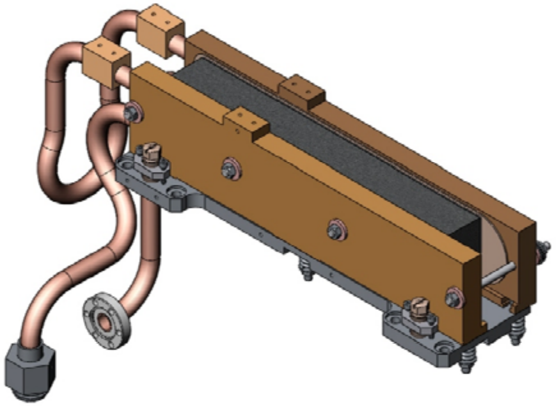
Zhang L. et al., J. Synchrotron Rad. (2013). ***20****, 567–580*

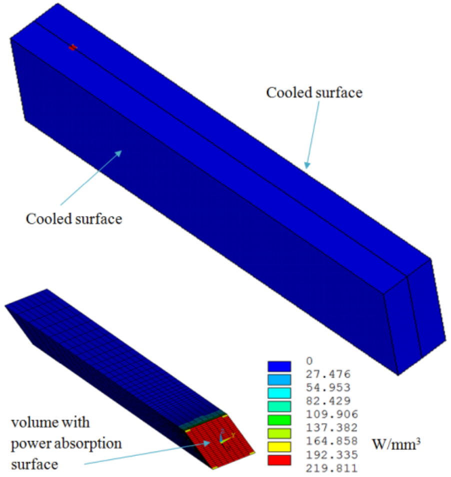
Thermal deformation: direct measurement technique

ID06 LN2 cooled Si crystal (DCM)

- **FEA (**Gaussian distribution and volume power absorption, $\mathbf{h_{cv}}$ determined by fitting temperature **in only one case)**
- **For various other cases (I, HxV)**







- **Excellent agreement in Temperature**

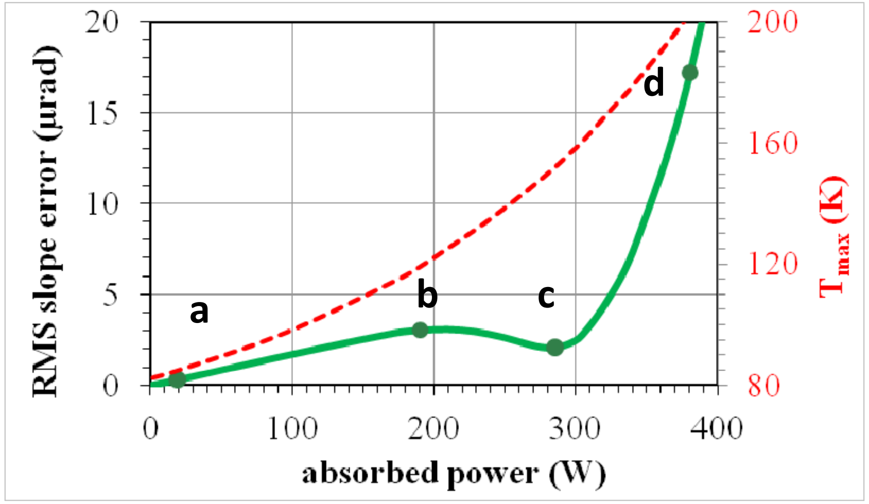
Zhang L. et al., J. Synchrotron Rad. (2013). ***20****, 567–580*

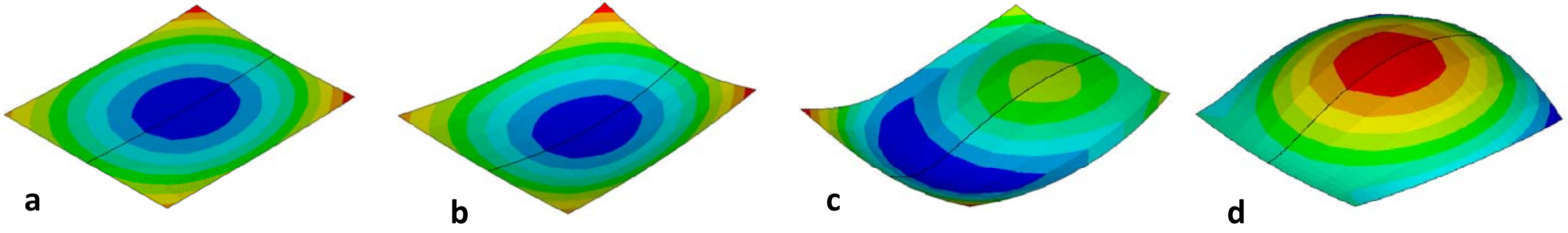
Thermal deformation: direct measurement technique

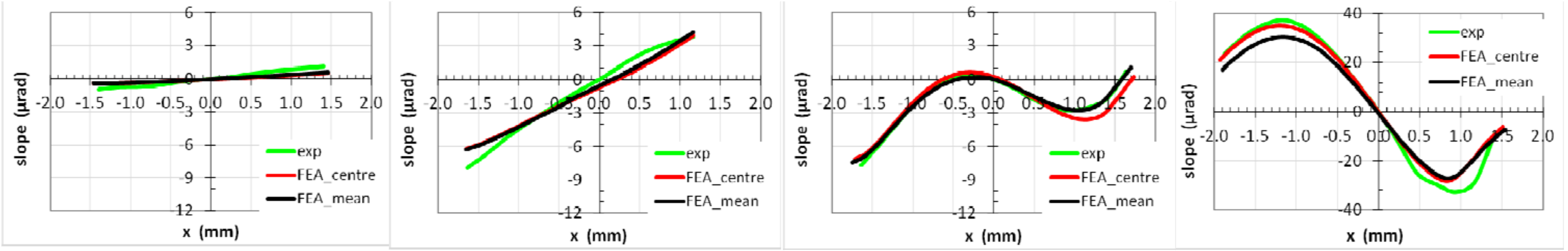
ID06 LN2 cooled Si crystal (DCM)

- (1st) Direct comparison of test results and FEA results

Zhang L. et al., J. Synchrotron Rad. (2013). ***20****, 567–580*





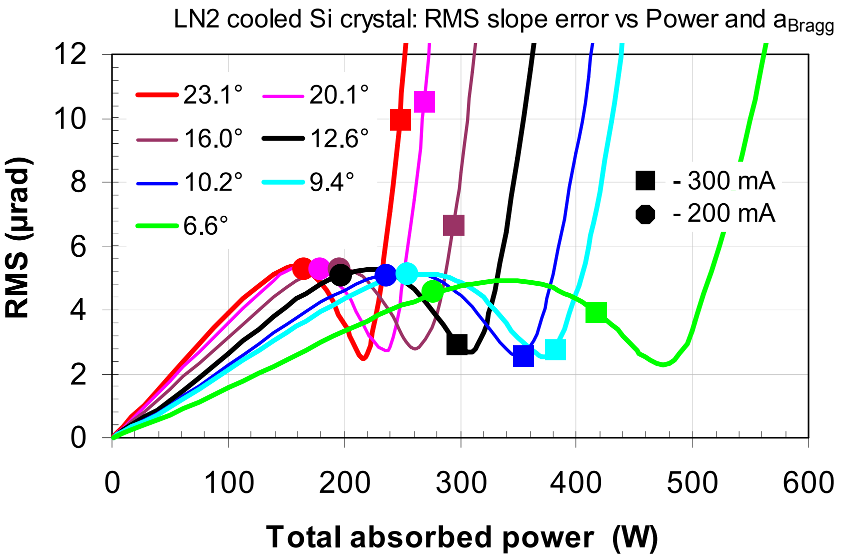


Thermals slope versus Power and Bragg angle

For UPBL06 LN2 cooled monochromator crystal

- Si 111, 5~20 keV
- LxWxT = 100x60x80 mm^3
- White beam mirror used to reduce the heat load
- Beam size HxV=2x1 mm^2
- **Indirect cooling h_{cv}=4000 W/m^2/K**

➢ **Bragg angle: 5.6 ~ 23.1°**



Thermals slope versus Power and Cooling coefficient

For UPBL06 LN2 cooled monochromator crystal

- **Bragg angle: 10.4,**

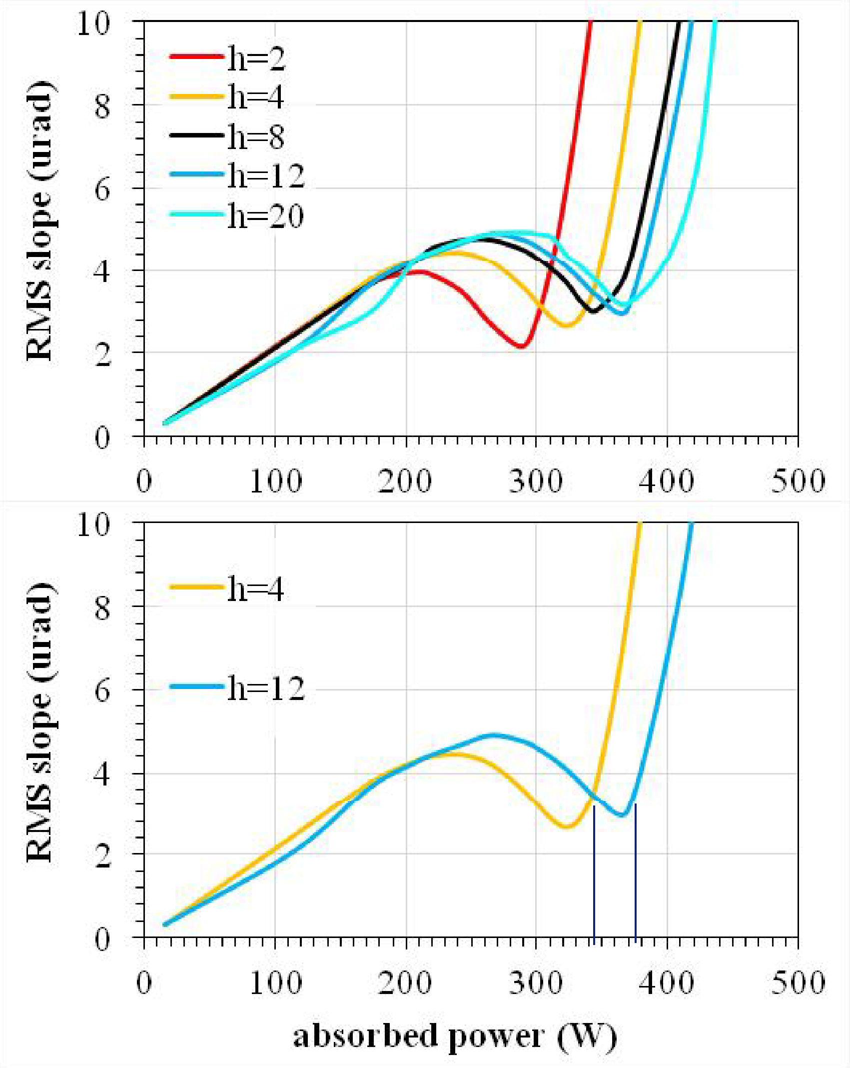
➢ **Effective cooling coefficients:**

- h_{cv} (W/m²/K)
- 2000 poor contact
- 4000 correct contact
- 8000 excellent contact
- 12000 direct cooling
- 20000 enhanced direct cooling

➢ **Indirect cooling vs direct cooling**

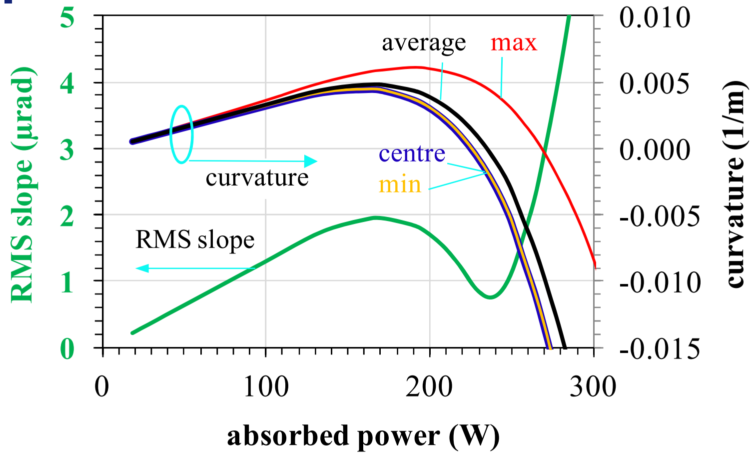
- P_{limit} (Indirect cooling) = 345 W
- P_{limit} (direct cooling) = 375 W
- Direct cooling is interesting for the heat load in a small range (345, 375) W

➢ **Good contact between cooling block and silicon crystal is needed**



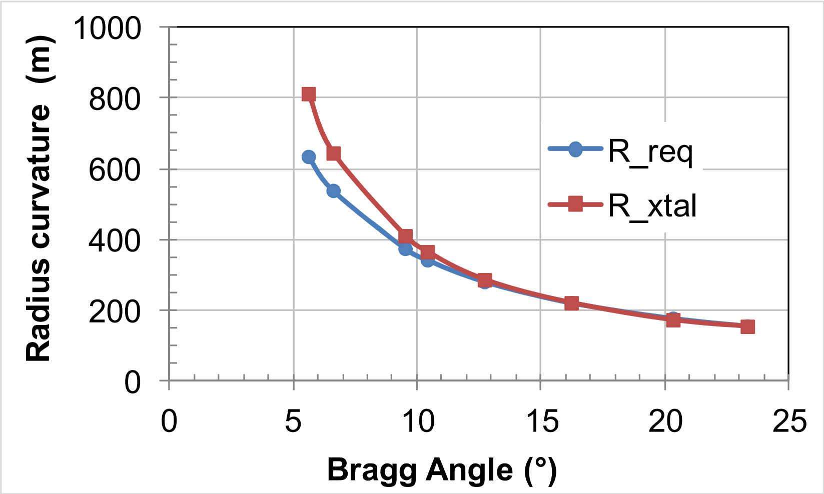
Focusing effects of the monochromator crystal

For UPBL06 (ID20) LN2 cooled Si crystal



- Silicon crystal at *p*=31 m
- Beam size: HxV=(**1.8~2.8**)x0.8 mm^2 at 27m
- Bragg angle: 5.6 ~ 23.1°
- Variable absorbed power
- Gaussian power distribution
- Thermal deformed crystal shape calculated by FEA: radius R_xtal
- Required radius R_req for beam collimation (q→ ∞):

$$R_{-req} = \frac{2p}{\sin(\theta_{Bragg})}$$

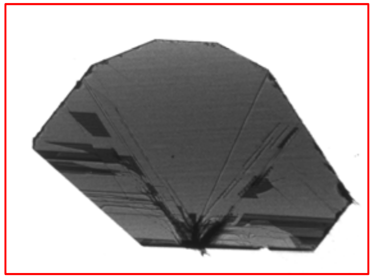


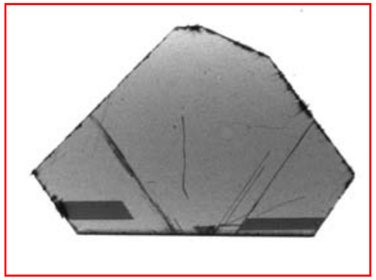
- Beam collimation is achievable by using only monochromator and by monitoring primary slits opening

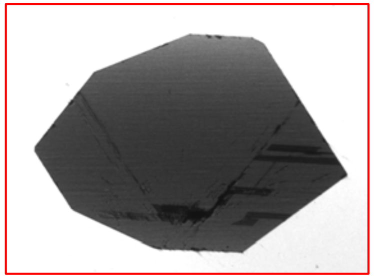
Zhang L. et al., J. Phys.: Conf. Ser. ***425*** *(2013) 052008*
doi:10.1088/1742-6596/425/5/052008

Diamond crystal monochromator

ID28 SS Diamond monochromator, U32g15



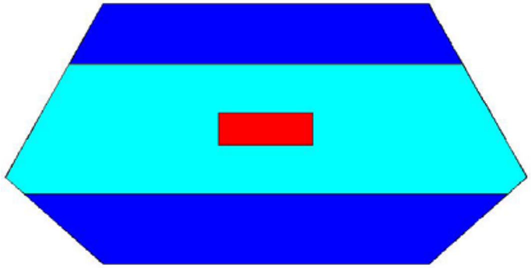




Parameters

- e_{ph} > 12 keV
- At 28 m from the sources
- Incident angle: 26 degrees
- Beam size: HxV = 1.3 x 0.5 mm^2
- Water cooling (indirect)
- Diamond crystal size: 4x8x0.3 mm^3
- Darwin width at 311: **2 µrad**

Recommendations for $\Delta\theta < 0.4$ µrad, $T_{max} < T_{melt}$(In)

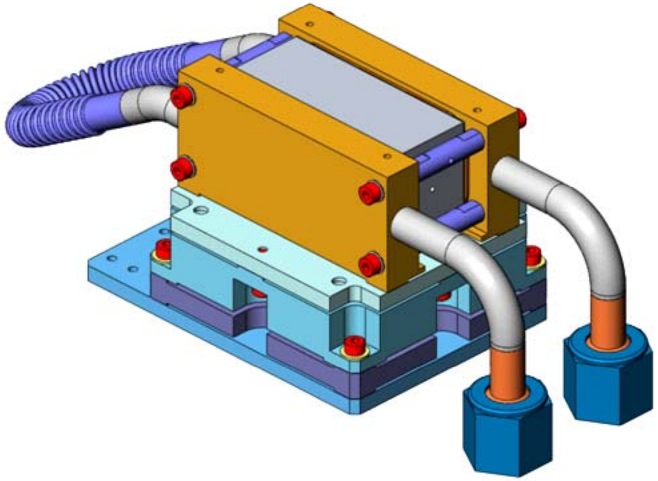


- Beam size reduced to just cope with centre cone HxV = 1.3 x 0.5 mm^2
- 0.8-mm (0.3+0.5) thick diamond attenuator in front
- Maximize contact surface area
- Thermal Contact Resistance (TCR) > **7000 W/m²/°C** (Indium foil to be used)

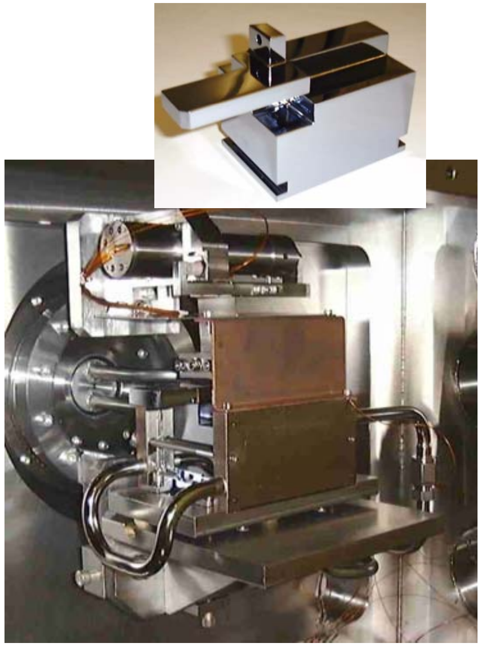
Initial deformation of the crystal

Heat load tests of the LN2 cooled monochromator → initial deformation of the crystal due to

- Monochromator components **manufacturing**, crystal **cutting**, **mounting** and **assembling**, **cooling down** from T_{room} to T_{LN2}



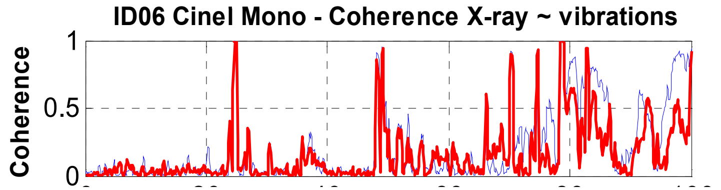
* Chumakov A.I. *et al.*, J. Synchrotron Rad. (2014). **21**, 315–324

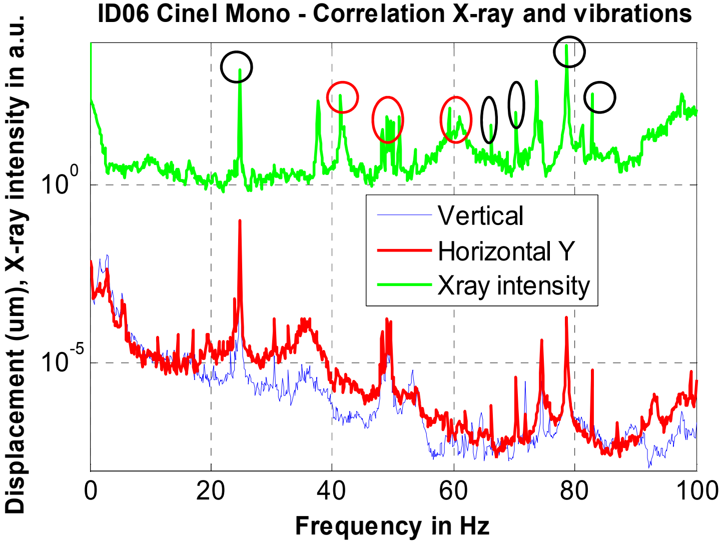


	ID06 DCM	ID18 DCM	ID09 CCM	
θ_0 =	**0.45**	**1.0***	**5.5**	μrad
h_{eff} =	2900	3500	1400	$W/m^2/K$

Stability and vibration of the monochromator

Example of ID06 Cinel mono

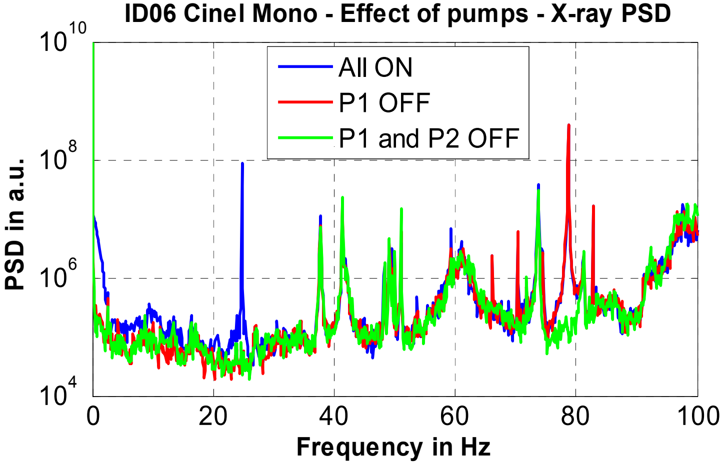




Correlation between X-ray intensity fluctuation and mechanical vibration

- F=24.8, 66.2, 70.3, 78.7, 82.8 Hz
- 1st peak due to vacuum pump 1, other 4 peaks due to pump 2

Remaining peaks ○ probably due to mechanics → room for improvement

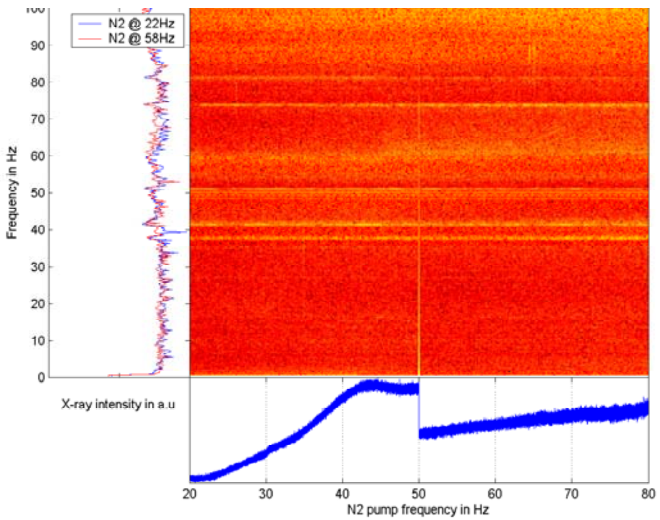


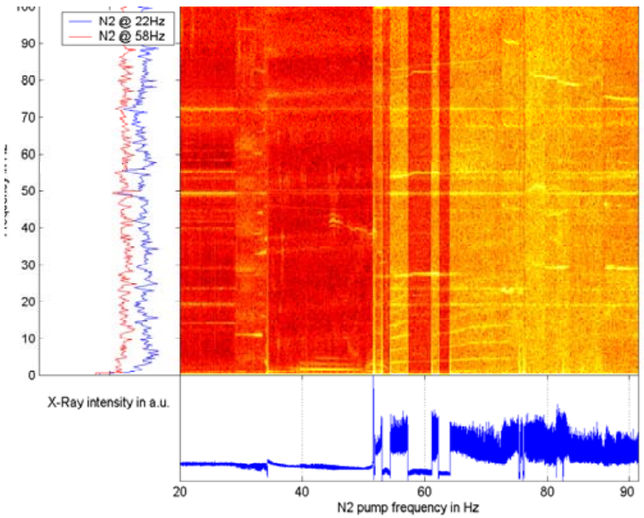
DCM vibration tests in ID06 (2008)

Cinel

Oxford

Beam intensity fluctuation ΔI versus the cryo-cooler pump frequency





- For Oxford mono, high ΔI for f_{pump} > 45 Hz is due to the cooling scheme and mechanical structure of the mono

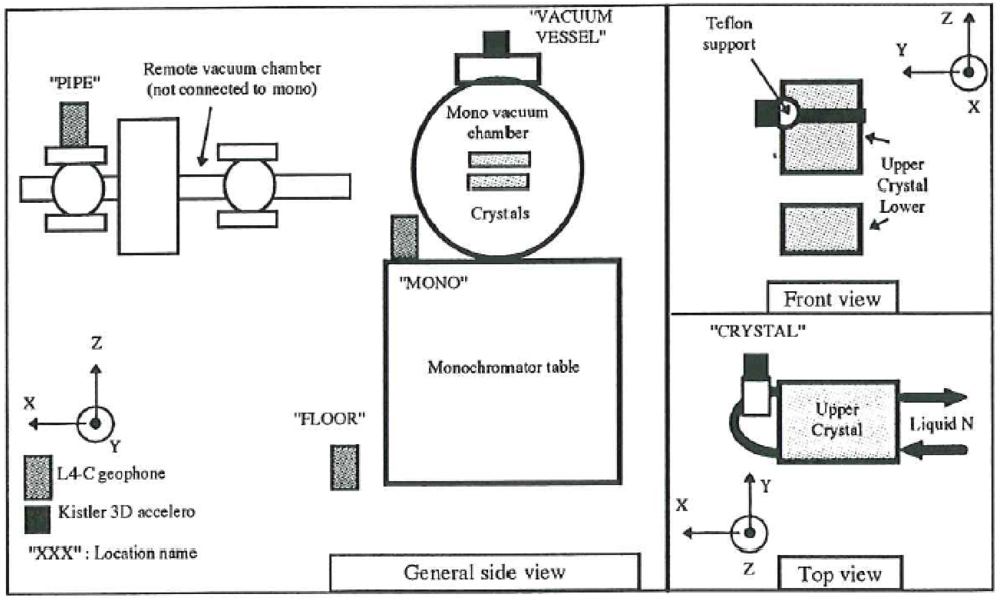
DCM vibration tests in ID22 (1997)

1997, ID22, 3D accelerometer

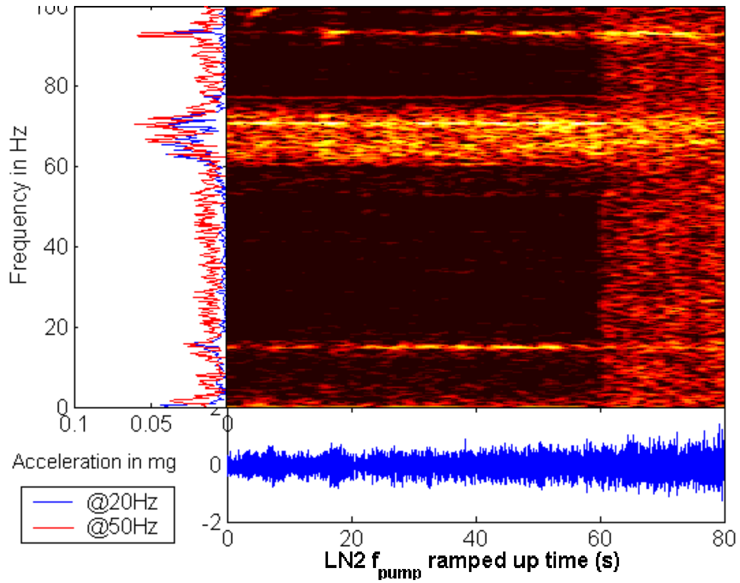
1st direct in-situ measurement

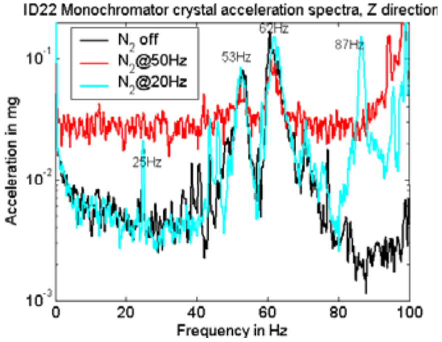
on the LN2 cooled crystal

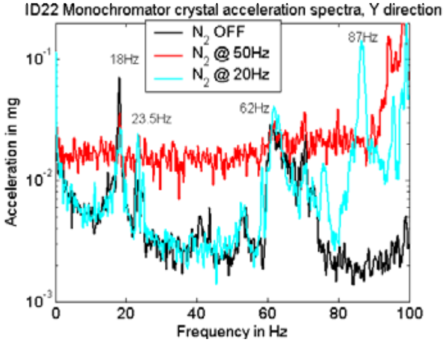
SET-UP



HP: 100Hz, 800 lines, tcap: 80sec.
Matlab: high pass 0.5Hz, twin/ovlp: 8/0sec.
Spectrogram: twin/ovlp: 2/1sec.



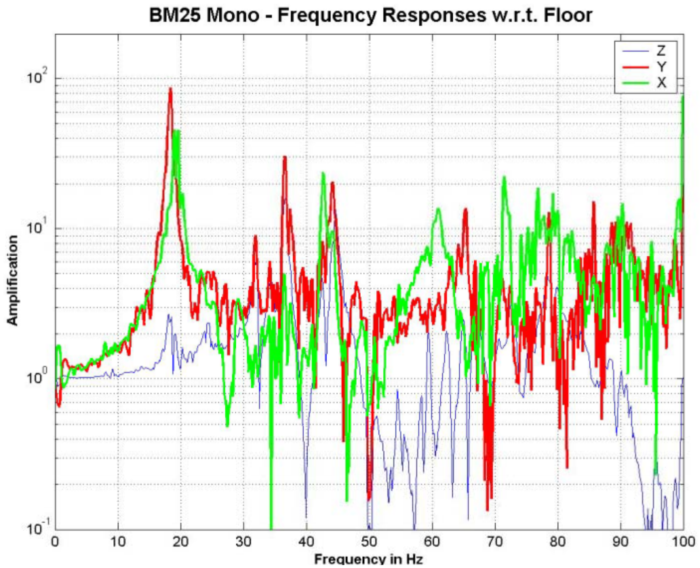




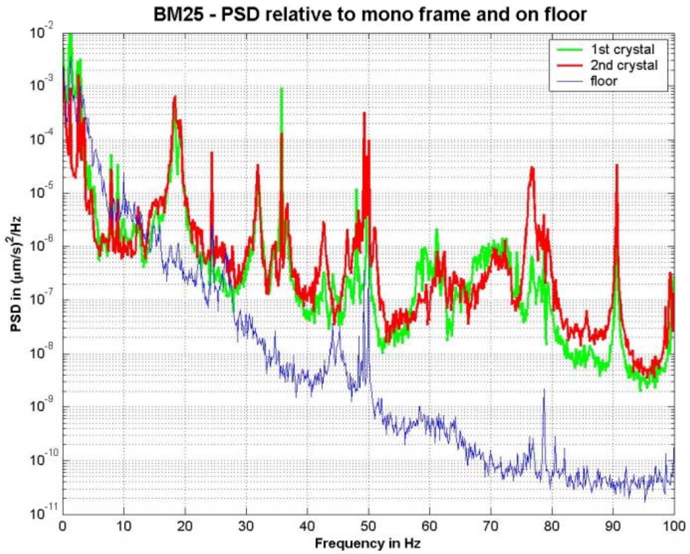
DCM vibration tests in BM25 (2004)

BM25 mono

FrF

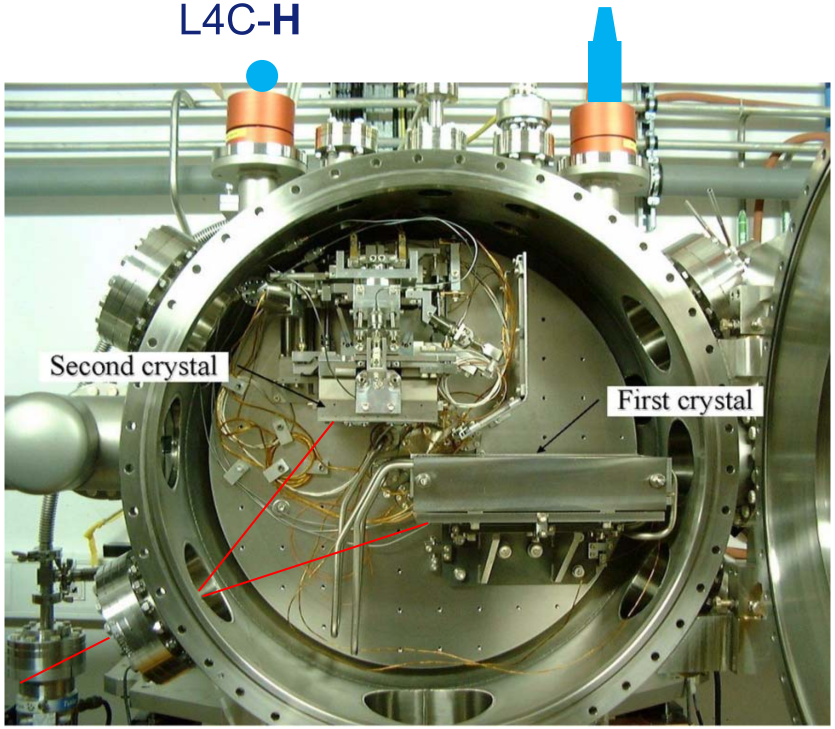


PSD (µm/s)²/Hz



Geophone L4C-**H**

Geophone L4C-**V**



Laser beam

Duo-beam Laser Vibrometer

Summary

- Thermal deformation can be accurately modeled
- Crystal monochromator has focusing effects (R~ 200 m)
- Thermal deformation depends on Poisson's ratio *v:*

  $$\Delta\theta \sim (1+ v)$$

  but Anisotropic elasticity of the silicon can be taken into account by use of an average Poisson's ratio in a simulation with isotropic and constant elasticity
- There are rooms for the improvement in terms of stability, and initial deformation of the crystal monochromator

Acknowledgment

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- *Co-authors of cited papers*

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  (* *Left ESRF*)

- *For diamond crystal monochromator*

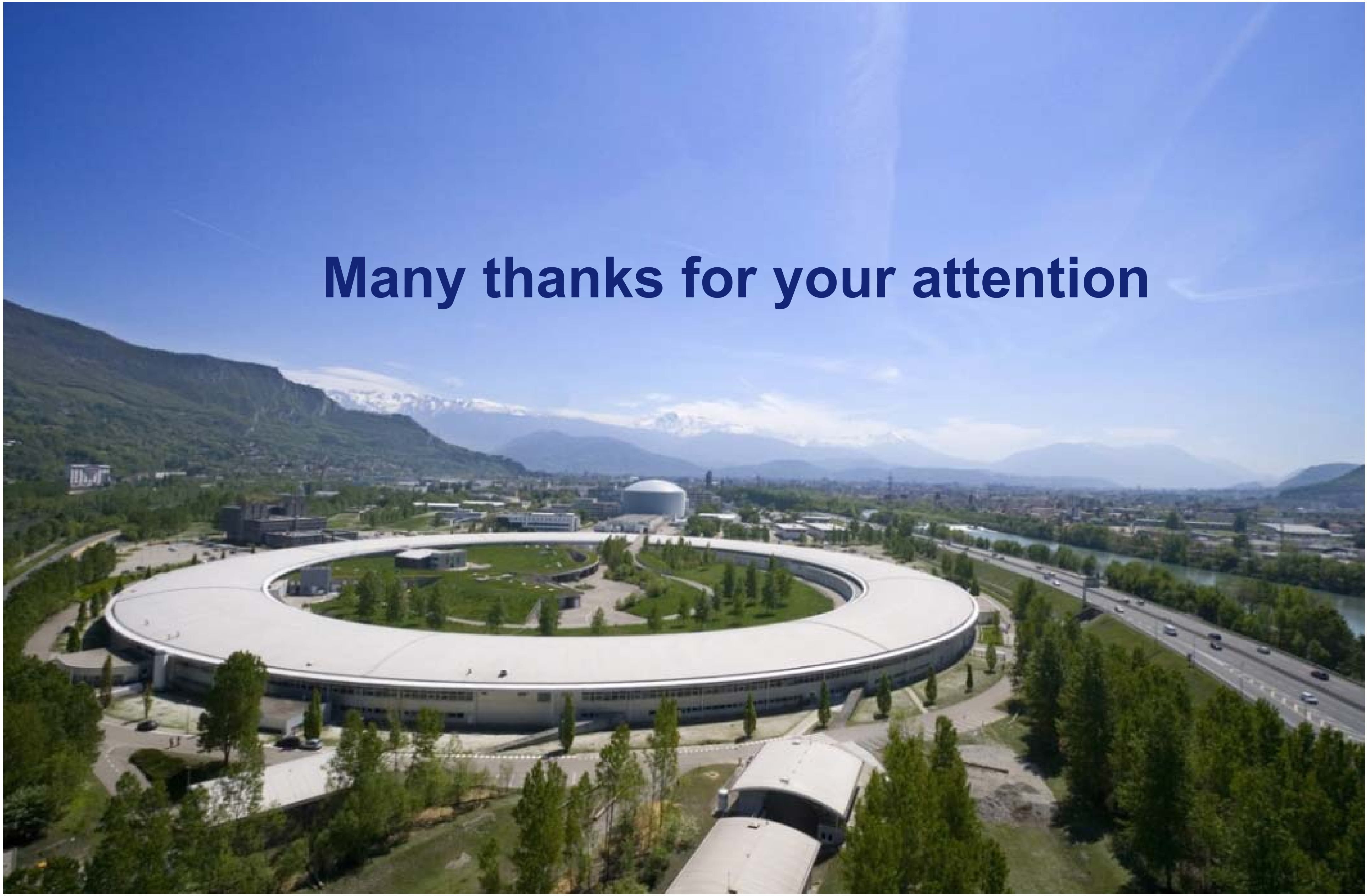
  A. Bosak, J. Härtwig, P. Van Vaerenbergh

- *Many other ESRF colleagues*

  J. Susini, Y. Dabin, M. Lesourd,…

Other light sources *(Co-authors of cited papers)* :

W.K. Lee, H. Schulte-Schrepping, T. Tschentscher



Many thanks for your attention