Cryogenic cooling of monochromator crystals: Indirect or direct cooling?

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Abstract

At the ESRF, 17 Beamlines are equipped with liquid nitrogen cooled monochromators. Liquid nitrogen (LN2) enables to cool the silicon crystals down to a temperature range where the thermal properties of the Silicon are favourable, and, consequently to reduce the thermal deformation induced by the beam heat load. In most cases, indirect cooling is applied: liquid nitrogen flows inside copper blocks, which are clamped to the sides of the crystals. The heat flow is evacuated from the crystal though the silicon – copper interface, with possibly some intermediate material like indium or indium-gallium, intended to improve the thermal contact. In order to suppress this thermal resistance, some attempts have been made to implement direct cooling: channels are machined in the Silicon crystal, and the liquid nitrogen flows directly inside the Silicon channels. This requires a vacuum tight sealing between the silicon block and the metallic LN2 feeding pipes.

In a first part, calculations results are presented, enabling to assess the gain that can be expected from direct cooling, compared to the classical indirect side cooling technique.

In a second part, based on a few crystals assemblies in use at the ESRF, the critical points associated with indirect side cooled and with directly cooled crystals are described.

1. Influence of the cooling on the crystal deformation

1.1. Introduction

Before entering in the details of the cooling techniques, it is useful to try to assess the influence of the cooling on the crystal performances. The goal of cryogenic cooling is to limit the deformation of the crystal lattice in the region of the beam footprint. The slope error of the crystal surface must be small compared to the intrinsic rocking curve width of the crystal, which is of the order of 10µrad for Si 111 (it depends on the selected energy and on the crystal Miller indexes used for the Bragg reflection, and is much smaller for Si 311 for instance). Thanks to Liquid Nitrogen (LN2) cooling, the silicon crystal can be maintained in a temperature range where its ratio α/κ (α = thermal expansion coefficient, κ = thermal conductivity) is very low (see figure 1), so that the deformation of the crystal due to the beam heat load can be limited.

When the Beam heat load is increased, the crystal temperature increase depends on geometrical parameters and on the efficiency of the cooling. Figure 1 suggests that the cooling of the crystal should be efficient enough to keep the crystal temperature lower than 130K.

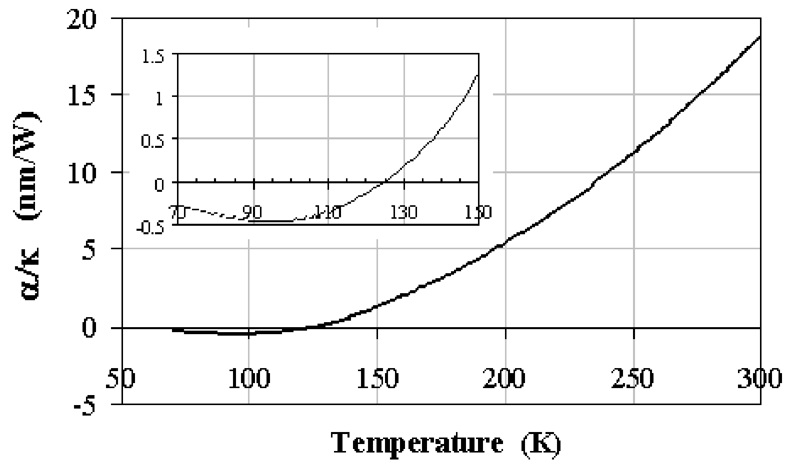

Figure 1: Ratio of thermal expansion coefficient and the thermal conductivity α/κ of Silicon versus temperature

1.2. Slope error vs cooling coefficient predicted by FEA calculations

In a research paper published in 2003 [1], Lin Zhang *et al.* have analysed the behaviour of the ESRF ID9 cryogenically cooled monochromator submitted to high heat load. Some of the results contained in this paper are shown in the present section in order to illustrate the influence of the cooling on the thermal slope error, for a given geometry. The ID9 monochromator is equipped with a monolithic crystal shown in figure 2, which receives a relatively large photon beam (Beam size: 10.35mm horizontal x 2.3mm vertical) with a power ranging from 20 to 520W. The crystal is indirectly cooled from both sides of its diffracting surface: LN2 circulates in two copper blocks that are clamped on the sides of the Silicon (cooled areas shown in dark blue in Figure 2).

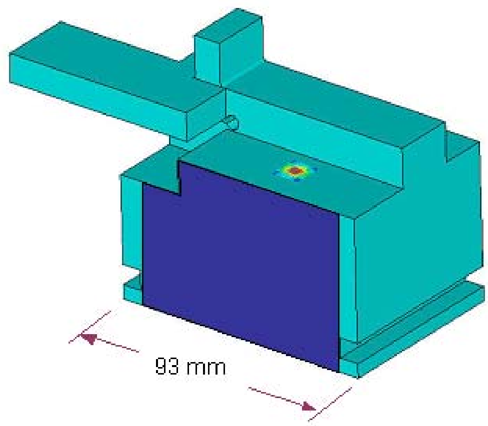

Figure 2: ESRF ID9 channel-cut Si crystal monochromator, showing the beam footprint (red) and the cooled areas (dark blue, on both sides)

The cooling can be modelled by applying a heat transfer coefficient hcv on the cooled areas of the silicon crystal. Typical values of hcv are given below:

hcv = 3000 W m^{-2} K in the case of fair thermal contact between the copper cooling blocks and the silicon crystal.

hcv = 5000 W m^{-2} K in the case of good thermal contact

hcv = 8000 W m^{-2} K in the case of excellent thermal contact

hcv = 18000 W m^{-2} K side cooling coefficient equivalent to direct cooling with an enhanced cooling area.

For these four cooling coefficient values, FEA calculations have been done to predict the crystal surface slope error as a function of the beam power. Results are plotted in figure 3.

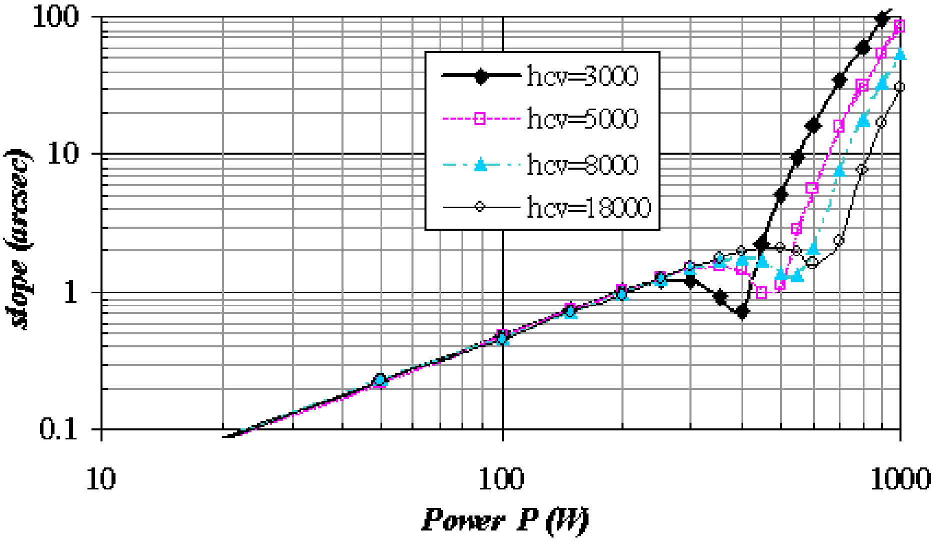

Figure 3: ESRF ID9 channel-cut Si crystal: FEA calculated thermal slope error versus absorbed power for four different cooling coefficients.

Figure 3 raises the following comments: In the “linear”region (P < 300 W), the slope error is independent of the cooling coefficient. This can be explained by the fact that the curve of α/κ vs Temperature (figure 1) is quite flat for temperatures below 125 K (calculations also show that, in the “linear” region the crystal temperature is lower than 125 K). For P > 300 W, the slope error first decreases (“transition” region, corresponding to temperatures where α/κ is minimum), and then increases very rapidly. In the “non-linear region” (P >> 300 W), the slope error is strongly dependent on the cooling coefficient. In this region, the maximum crystal temperature is higher than 125K and, as shown in figure 1, the ratio α/κ increases very rapidly with the temperature, and the temperature depends on the cooling coefficient.
However, the slope error in the “non-linear”region is too high compared to the crystal acceptance, and safe operation is only guaranteed in the “linear” and “transition” regions.
This clearly shows that for a fixed geometry, enhancing the cooling by direct cooling is beneficial in only a limited number of cases. In the studied case, direct cooling would give better results than indirect cooling only for P > 450 W, where the slope error, in all cases, is relatively high (> 1.5 arcsec, i.e. 7.5 μrad). Considering that the acceptable slope error is often less than 10 μrad, the applications where direct cooling would bring a decisive advantage are quite limited.

2. Liquid Nitrogen circuit and flow parameters at the ESRF

Each ESRF LN2 cooled monochromators is connected to a closed loop LN2 circuit. A cryogenic unit including a pump and an heat exchanger maintains the LN2 temperature at 78 K at the entrance of the monochromator. The LN2 flowrate can be adjusted by varying the pump frequency. In normal operation, the typical LN2 flowrate is 240 l/hour (45 Hz pump frequency), the pressure is set to 3-4 bar, and the typical pressure drop across the circuit (including the crystal cooling blocks and the connecting lines) is 0.4 bar.
For 500 W evacuated by the liquid Nitrogen, with a flowrate of 240 l/hour, the expected temperature rise of the LN2 is 4.5 K and the maximum LN2 temperature is therefore 78 + 4.5 = 82.5 K. At 3 bar, the boiling temperature of LN2 is 88 K, which provides a sufficient margin as compared to 82.5 K.

The LN2 is injected in the copper cooling blocks through 8 or 10 mm inner diameter pipes. The corresponding fluid velocity is 1.35 – 0.85 m/s and Reynolds number is 55000 – 43000, indicating a strongly turbulent flow. However, the pressure drop is relatively low (1.9 times lower than for water at the same velocity), and the energy transferred to the cooling circuitry is therefore limited, which explains that vibrations induced to the crystal can be kept within acceptable limits.
The optimum LN2 velocity is finely adjusted by varying the LN2 pump frequency and simultaneously monitoring the vibrations induced on the crystal, in order not to generate a fluid induced vibrations spectrum exciting the natural frequency of the crystal support (see detailled procedure in [2]).

3. Indirect cooling by the sides: Example of ESRF design - Critical points

3.1. Description of some ESRF crystal assemblies

An example of first crystal assembly of a Double crystal monochromator is shown in figure 4.

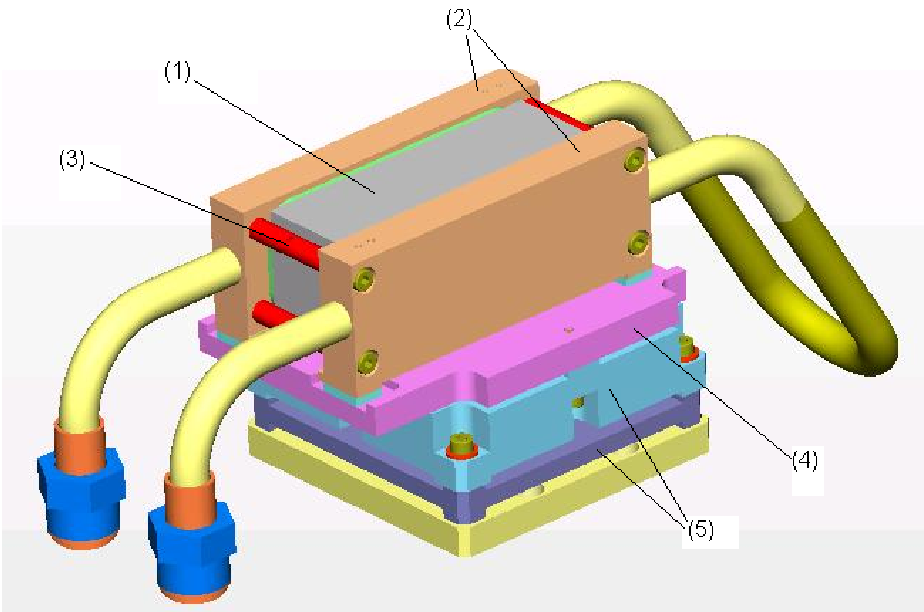

Figure 4: Example of ESRF first crystal assembly: (1) silicon crystal; (2)copper cooling blocks with internal fins;(3) invar clamping rods;(4) invar base plate; (5) ceramic insulating plates

The Silicon crystal is clamped between the two copper cooling blocks. The clamping pressure is set to 5-10 bar, by fastening the clamping screws in the rods (3) to the correct torque with a torque limiting screw driver. In order to maintain this clamping pressure constant at low temperature, invar is used for parts (3) and (4) (the thermal expansion of invar and silicon are similar over the 300 K – 80 K temperature range). In addition, spring washers are installed on the clamping screws. This clamping pressure of 5-10 bar is sufficient to obtain a good thermal contact (see [3], [4], [6]) and does not induce significant deformation of the diffracting planes: FEA calculations show that for a 50 x 50 x 25 mm (height x length x width) crystal clamped with 10 bar pressure on its 50 x 50 mm sides, the slope error induced on the diffracting surface is 0.6 μrad.
At the interface between the silicon and the copper, two different techniques are used to obtain a good thermal contact: On some monochromators, a 0.5mm thick indium foil is inserted between the copper and the Silicon, and this foil may be coated with eutectic Indium – Gallium alloy (liquid at ambient temperature). The copper cooling blocks need to be nickel coated in this case in order to avoid the In-Ga to diffuse inside the copper. This foil improves the thermal contact and, thanks to its relatively high thickness, absorbs the differential expansion between the copper blocks and the Silicon and limits the resulting stress and strain transmitted to the silicon at low temperature. On other monochromators, the copper and the silicon surfaces are finely polished to optical quality. Good monochromator

performances have been reported in both cases; a quantified comparison would require dedicated heat transfer tests in similar conditions and dimensions (see discussion in 3.3). The flatness of the silicon and copper surfaces is essential in both cases, and the cooling blocks need to be reinforced to limit their deformation when submitted to the 4 bar LN2 internal pressure.
Inside the cooling blocks, fins are machined in the copper part in contact with the crystals (typical dimensions of fins: 1 mm thick, 8 mm high). The typical total channel section in the fins chamber is 120 mm^2, corresponding to an average LN2 velocity of 0.55 m/s.
The support and thermal insulation of the crystal-cooling blocks assembly is done either by ceramic plates, as sown in figure 4, or by stainless steel plates and intermediate ZrO_2 balls.
Crystal dimensions (first crystal of Double crystals monochromator): Length: 40 to 150 mm; width: 25 to 35 mm; height: 40 to 70 mm. Note that since the thermal conductivity of the silicon is very high at 80 k (~900 $W . m^{-1} . K^{-1}$, i.e. twice as high as copper), it is useful to oversize the crystal height in order to increase the heat exchange surface area between copper and silicon.

3.2. Cooling coefficient

As expressed in [1], in the case of indirect cooling with cooling blocks clamped on the crystal, the effective cooling coefficient hcv on the silicon contact surface can be calculated as:

$$hcv = \left[\frac{1}{fa \times hcv0} + R(Cu) + R(In) + Rc \right]^{-1} \quad (1)$$

Where fa is the ratio of the cooling channel surface area and the contact surface area, hcv0 is the cooling coefficient of the LN2 in the cooling channels, R(Cu) and R(In) are the thermal conduction resistances in the copper and in the indium foil, respectively, Rc is the thermal contact resistance at the interface.

With fa = 5 (case where fins are machined inside the copper), hcv0 = 2000 $W.m^{-2}.K^{-1}$, Thermal conductivities k(Cu) = 400 $W.m^{-1}.K^{-1}$ and k(In) = 80 $W.m^{-1}.K^{-1}$ at 80K, thickness t(Cu) = 3 to 11mm, and t(In) = 0.5mm, and Rc estimated according to data from [3], [4], [6] and [7], the different resistive terms of equation (1) take the following values:

$$\left[\frac{1}{fa \times hcv0} \right] = 10 \, . \, 10^{-5} \, m^2 . K . W^{-1}$$

R (Cu) = 0.75 to 2.75. 10^{-5} $m^2 . K . W^{-1}$ R (In) = 0.62. 10^{-5} $m^2 . K . W^{-1}$

Rc = 1 to 20 . 10^{-5} $m^2 . K . W^{-1}$ (Rc is quite sensitive to the quality of the surface contact)

=> hcv = 3000 to 8000 $W. m^{-2}. K^{-1}$

This shows that R(Cu) and R(In) are generally negligible. The two predominant factors are the thermal contact resistance and the cooling efficiency in the copper channels.

3.3. Thermal contact resistance

References [3] to [6] can be consulted to get some values and optimum conditions for the thermal contact resistance in vacuum at ambient temperature. Thermal contact resistance studies at 80K are not so common. References [6], [7] and [8] suggest that the thermal contact resistance is twice higher (i.e. worse) at 80 K as compared to the resistance at ambient temperature.

The results published in these papers show a significant improvement of the thermal contact with an indium intermediate foil at a contact pressure of 5-10 bar, but important dispersions in the results are also mentioned, due to surface effects which are difficult to control. Some heat transfer measurements on a real crystal-cooling blocks assembly would therefore be necessary in order to get reliable quantified data applicable to our case.

4. Direct cooling: Critical points – The ESRF ID1 crystal design

4.1. Critical points of directly cooled crystals design

The main technological challenge in designing a directly cooled monochromator is to achieve a leak tight junction between the crystal and the LN2 supply pipes, down to 80 K, without inducing unacceptable shape distortions of the diffracting surface.

The common practice is to weld or brase the supply pipes to a junction plate, which then needs to be sealed to the silicon block. The junction plate should be made of a material with a linear expansion coefficient similar to silicon in the temperature range 300 K – 80 K. Either silicon, or invar 36 can be used. The sealing between the junction plates and the silicon block may be done by brazing,or by gluing, or by metallic seal. Both techniques have drawbacks: brazing might induce stresses and crystal distortions due to the differential expansion between the materials over the brazing thermal cycle; the glue must resist to the radiation level in the monochromator; metallic seals require quite high clamping forces which might distort the silicon lattice. In addition, in the three cases, the differential expansion of the materials over the temperature range 300 K – 80 K may also result in shape errors.

4.2. Example: The ESRF ID1 directly cooled crystal

The first crystal assembly of the double crystal monochromator of ID1 is shown in figure 5.

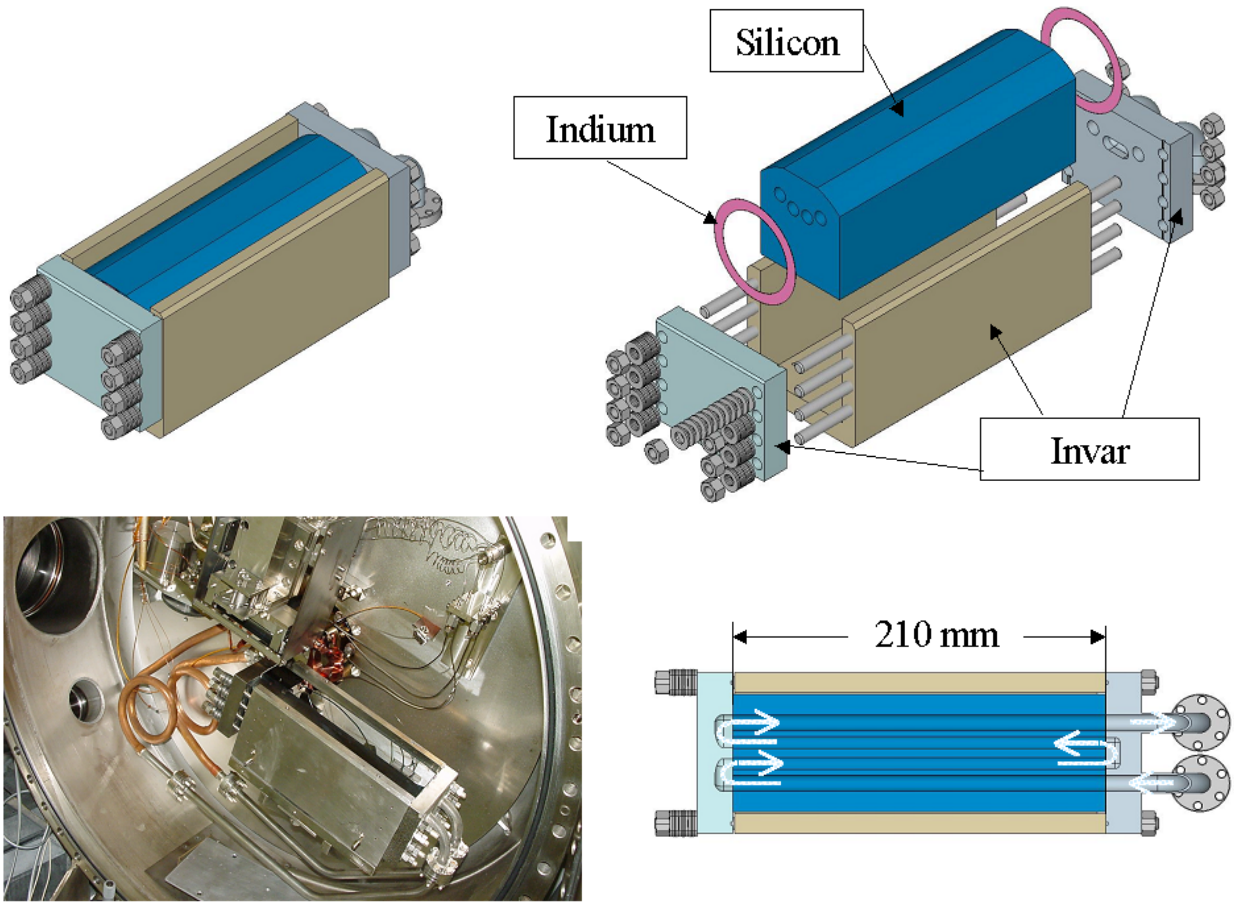

Figure 5: The first crystal assembly of the double crystal monochromator of ID1

The silicon crystal is 210 mm long and drilled with four cooling channels diameter 10 mm. It is sealed at both ends to invar junction plates by indium seals. The indium seals are made of an indium wire diameter 1.5 mm, shaped over a 60 mm diameter circle, mounted between two silicon and invar flat faces. From [9], the total force required to obtain a vacuum tight sealing is 22200 N, i.e. 118 N per mm of seal, corresponding to a residual thickness and width of respectively 0.3 and 5.9 mm for the idium after fastening. The U-shaped part linking the junction plates is made of invar and spring washers of adequate stiffness are installed under the clamping nuts to maintain a constant force within +/- 5% along the temperature cycle.
Reliable vacuum tightness has been observed over several years and temperature cycles with this set up. Figure 6 shows the silicon block displacements induced by the 22200 N force applied to fasten the indium seal, calculated by FEA.

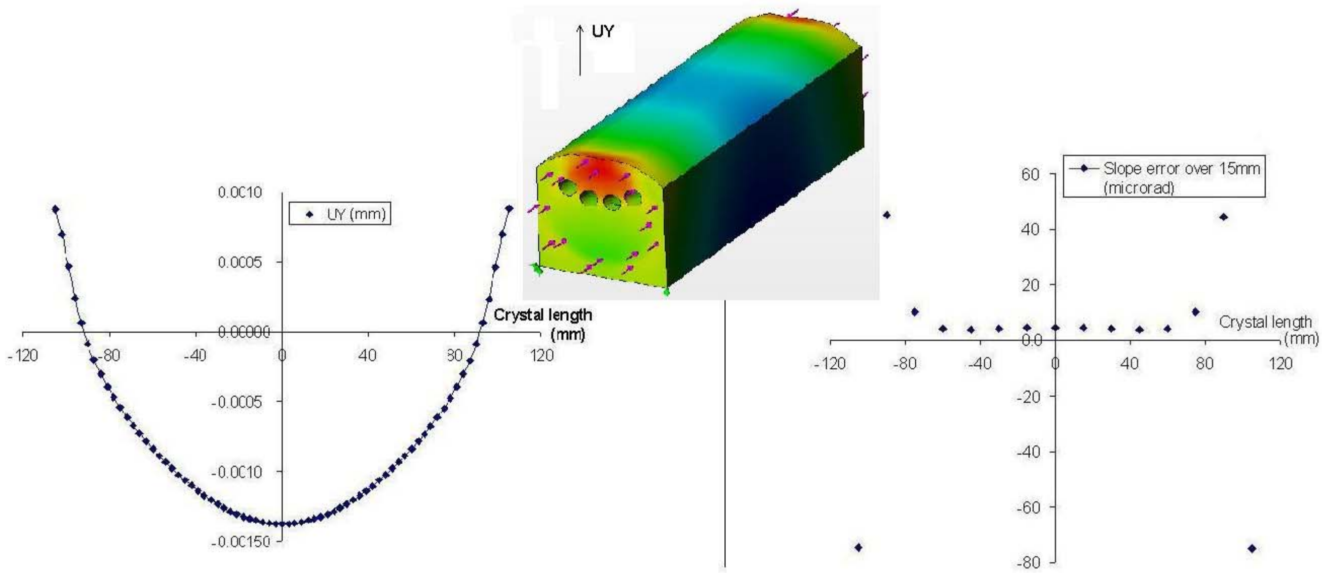

Figure 6: ID1 first crystal distortion of the top surface due to the 22200 N applied to fasten the indium seal, calculated by FEA: displacement normal to the diffracting surface UY vs crystal length; slope error over a 15mm long section vs crystal length.

The slope error graph indicates that over the 130 mm central part of the silicon block, the slope error between points included inside a 15 mm long section is less than 5 μrad (15 mm is the beam footprint at the lowest Bragg angle used on this monochromator). The crystal end sections close to the junction plates, where the slope error is too high, are not useable.

5. Conclusion

FEA calculations show that for a fixed crystal geometry and in the common operating range of the monochromators, enhanced cooling by direct cooling does not result in a very decisive reduction of the surface shape errors. FEA is a very useful tool to compare and predict the efficiency of different possible designs.
An indirectly cooled crystal assembly in operation at the ESRF has been described, underlining the critical factors for an efficient cooling. An example of a directly cooled crystal assembly in operation at the ESRF has been described, and the difficulties associated with this type of technique have been discussed and illustrated by calculation results.

References:

[1] L. Zhang, W.-K. Lee, M. Wulff, L. Eybert, “The performance of a cryogenically cooled monochromator for an in-vacuum undulator beamline” J. Synchrotron Rad. (2003). 10, 313-319.

[2] A. Chumakov, R. Ruffer, O. Leupold, J.-P. Celse, K. Martel, M. Rossat, W.-K. Lee, “Performance of a silicon monochromator under extereme heatload” J. Synchrotron Rad. (2004). 11, 132 - 141.
[3] P. Marion, L. Zhang, L. Vallet, M. Lesourd, “Thermal contact resistance study”, Proc. of MEDSI 2004, May, 2004, Grenoble, France.
[4] A.M. Khounsary, D. Chojnowski, and L. Assoufid, “Thermal contact resistance across a copper-silicon interface” Proc. of SPIE Vol. 3151 , pg 45-51, 1997. 0277-786X/97
[5] B. Fell, K. Fayz, “Thermal contact resistance investigation on the cooling of Phoenix-UV mirror M1” Proc. of MEDSI 2000, 13-14 July 2000, Wurenlingen, Switzerland.
[6] E. Gmelin, M. Asen-Palmer, M. Reuther and R. Villar, “Thermal boundary resistance of mechanical contacts at sub ambient temperatures” Review article in Journal of Applied Physics, 32 (1999) R19-R43
[7] June Yu, A.L. Yee and R.E. Schwall, “Thermal conductance of Cu/Cu and Cu/Si interfaces from 85K to 300K” Cryogenics, 1992, Vol. 32, N^{o} 7, pp.610-615.
[8] M.J. Nilles and S.W. Van Sciver, “Effects of oxidation and roughness on Cu contact resistance from 4K to 290K” Advances in Cryogenic Engineering 34 , pp.443-450, 1988
[9] Loys Goirand, “Les joints indium” Tutorial at the SFV, 10-11 December 2002, Noisy le Grand, France