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Improving Thermal Deformation of Double Crystal Monochromators by Cryogenic Cooling

Dongsu Lee[1], Seoung-Seun Yang[1], Kyunghan Chun[1], Bonghwan Kim[1,*]

[1]*Department of Electronic and Electrical Engineering, Catholic University of Daegu, Gyeongbuk 38430, Korea.*
**Corresponding author*

Abstract
We developed a double crystal monochromator with an indirect cryogenic cooling system for enhancing the beam line stability. This device shows significant improvements in vibration stability and high stability of parallelism between two crystals. The associated errors in accordance with variations in the internal pressure of the cooling lines are approximately 0.003° for pitch angle of the first crystal and 0.001° for the second crystal. In case of the stability of parallelism between the two crystals, there is about a 0.0005° angular difference between the pitch and roll of the two crystals. During the energy scan, the pitch angle error in parallelism of crystals is approximately 0.005° and the roll angle is approximately 0.002°. To reduce vibration, a special bellows was installed for discharging nitrogen vapour from the circulating liquid nitrogen in circulation system and a copper braid was used to cool the second crystal. Also, this device shows an excellent thermal stability.

Keywords: Double crystal monochromator, indirect cooling system, beam line.

INTRODUCTION

When an electron which accelerated to almost the speed of light changes its direction, it emits energy. The emitted energy is X-ray wave length. A synchrotron is a typical application of using this principle. X-ray double crystal monochromators (DCMs) which are major components of beam line at third-generation synchrotron are widely used in order to precisely select the required photon beam energies from white-beam X-rays[1]. X-ray diffraction (XRD) is commonly used to analyse structure of solid state. But, this measurement can be only used for crystalline solids which have long-range order. To overcome this weakness, X-ray absorption fine structure (XAFS) had been developed. One of major parts at XAFS is double crystal monochromators[2].

In DCMs, the purpose of cryogenic cooling is to limit the thermal deformation of the crystal lattice in the region of the beam footprint. The slope error of the crystal surface should be smaller than the intrinsic rocking curve width of the crystal. Liquid nitrogen (LN_2) cooling gives the silicon crystal to be maintained in a temperature range where its ratio α/κ (α = thermal expansion coefficient, κ = thermal conductivity) is very low, so that the deformation of the crystal can be limited[3].

In general, LN_2 circulation is pressurized (typically between 0.2 and 1 MPa) to prevent the appearance of vapour nitrogen. When LN_2 is refilled to the LN_2 tank, the pressure inside the closed-loop circulation can be increased. So, that pressure can lead to a small angular displacement of the crystal. Also, vibrations on the diffracting crystal may result from the circulation of LN_2,

if the flow is not completely laminar and/or by the nitrogen bubbles created during the heat-exchange process[4].

The thermal deformation of the crystal induces rocking curve broadening, which leads to a loss of monochromatic flux. The rocking curve broadening shows us two important facts: 1) there is a change of the shape of the crystal surface owing to the thermal deformation. 2) a heat load produces a distortion of the diffracting crystal volume (thermal stress) that may, in cases of high heat loads, significantly modify the diffracting properties of the crystal[5].

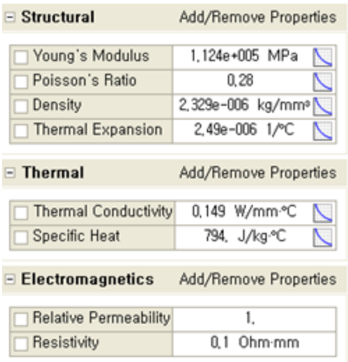
Almost all of the X-ray beam incident power is absorbed by the 1st crystal. In contrast, the heat load on the 2nd crystal is about 1.6% of the incident power. Therefore, it is very important to find a proper cooling method for the 1st crystal in order to prevent thermal deformation of the 1st crystal. In the process of deciding cooling method, we should consider three factors like ‘no thermal bump’, ‘no vibration’, and ‘optimal thermal stability’. And, cooling system is driven under ultra high vacuum (UHV)[6].

There are two cooling methods, such as indirect and direct cooling. At present, indirect cooling method is most popular, because direct cooling does not result in a very decisive reduction of the surface shape errors and the technological difficulties to make it[3]. In case of indirect cooling method, LN_2 flows inside copper blocks, which are clamped to the sides of the crystals. The heat flow is evacuated from the crystal through the silicon-copper interface, with possibly some intermediate material like indium or indium-gallium, intended to improve the thermal contact. On the other hand, direct cooling method can be used for suppressing thermal resistance effectively. Channels are machined in the silicon crystal, and LN_2 flows directly inside the silicon channels. The silicon crystal is sealed at both ends to invar junction plates by indium seals. The junction plate should be made of a material with a linear expansion coefficient similar to silicon in the temperature range 300K - 80K. Finally, to optimize the parallelism between the two crystals, it is most important for crystals to minimize their thermal deformation using a proper cooling system.

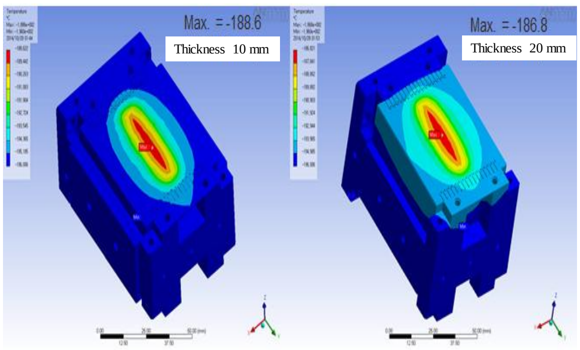
For this purpose, this paper will show the new concept of DCM which has cryogenic cooling system using a LN^2 and its improved beam line performances like thermal stability, angular variation in accordance with internal pressure of cooling line, and stability of parallelism between two crystals.

REVIEW OF COOLING CONDITIONS OF SILICON CRYSTAL THROUGH ANALYSIS BY ANSYS

Because the degree of deformation and the deformed structure of optical devices contacting incident energy give critical effects to the performance of beam light, it is very important to find precision requirements by cooling structures. To get information of temperature distribution during given period on a specific material, we usually do heat analysis. This method is essential to select material and to find thermal strain and heat stress.



(a)



(b) (c)

Figure 1. ANSYS simulation. (a) Input data of silicon and temperature distribution of crystals thickness of 10 mm (b) and thickness 20 mm (c).

The thermal conductivity κ of the silicon, the impinging X-ray beam power distribution, the crystal geometry and cooling condition effect the temperature distribution on the crystal mono chromator. The thermal expansion coefficient α is also related with thermal deformation of the crystal. Normally, the thermal deformation is inversely proportional to the thermal conductivity κ and proportional to the thermal expansion coefficient α. The ratio α/κ is often used to evaluate the thermal deformation. The ratio α/κ of silicon at LN_2 temperature (77K at 1 atm) is much lower than at room temperature. Because of that, LN_2 cooling can significantly reduce the thermal deformation of the silicon crystal compared with water cooling[5]. The effective cooling coefficient is strongly influenced by the thermal contact resistance at the interface between the crystal and the copper cooling block. The applied pressure (contact pressure) and the surface state of the contact bodies at the interface are the crucial factors on this thermal contact resistance[7].

Considering the above mentioned factors, we input physical characteristics of silicon as shown in Fig. 1(a). The simulation results of crystal thickness 10 mm and 20 mm showed that maximum temperature of 20 mm thickness was 2°C higher than 10 mm thickness, and in case of the temperature of the crystal body, 20 mm thickness was higher than 10 mm thickness as shown in Fig. 1(b) and Fig. 1(c).

From this result, we know that cooling from side body is more effective than bottom cooling. Also, the thinner thickness affects the higher cooling efficiency.

DESIGN OF COOLING SYSTEM

A. 1st Crystal Cooling Block

The 1st crystal block is made of brazed copper with internal LN_2 channel as shown in Fig. 2. In many cases, the silicon crystal is clamped between the two copper cooling blocks. The clamping pressure is normally set to 5 - 10 bar. In addition, clamping screws, spring washers, and invar are needed to fasten it. But, we used fingers for fixing silicon crystal, in order to reduce the deformation of silicon crystal due to the clamping pressure and to facilitate fastening. To obtain a good thermal contact at the interface between the silicon crystal and the copper block, 125 μm thick indium foil coated with eutectic indium-gallium-tin alloy is inserted. To prevent the crystal block from being corroded by the metallic eutectic, the crystal block is coated with nickel. Since the thermal conductivity of the silicon crystal is very high at 80K (i.e. twice as high as copper), it is useful to oversize the crystal height to increase the heat exchange surface area between copper block and silicon crystal.

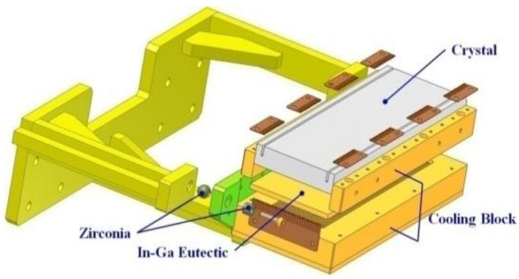


Figure 2. Schematic design for the 1st crystal assembly.

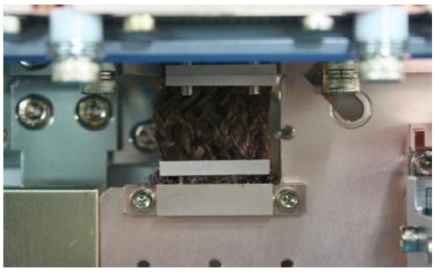


Figure 3. Copper braid for the 2nd crystal.

B. 2nd Crystal Cooling Block

For reducing vibration, the 2nd crystal is indirectly cooled by copper braid, which is connected to the 1st crystal holder, instead of using plate as shown in Fig. 3. However, the 1st crystal is fixed on the main plate and connected to the cooling pipe. Thus, the 1st crystal holder is cooled by LN_2 directly.

C. Insulation between the Crystal Block and the Main Structure

Reducing the heat transfer between the main structure and the crystal block which is cooled by LN2, zirconia rings and balls with low thermal conductivity are used in in the mechanical connection between the two structures as shown in Fig. 4.

D. Liquid Nitrogen (LN_2) Circulation System

The tubes for supply and return of LN_2 are installed along the central of rotary feed-through in the vacuum side, by way of cooling chamber, which is installed at the end of the rotary feedthrough. The cooling chamber is an interface of LN_2 route for supply and return (Fig. 5). In order to curb vibration and align the contact surface between cooling line and crystal block, bellows were installed after vacuum coupling radiation (VCR) fitting. Also, special bellow was equipped for discharging nitrogen gas which is generated during circulation. Between the cooling chamber and the VCR fittings, the tubes have flexible joints. LN_2 is designed to flow in a certain direction.

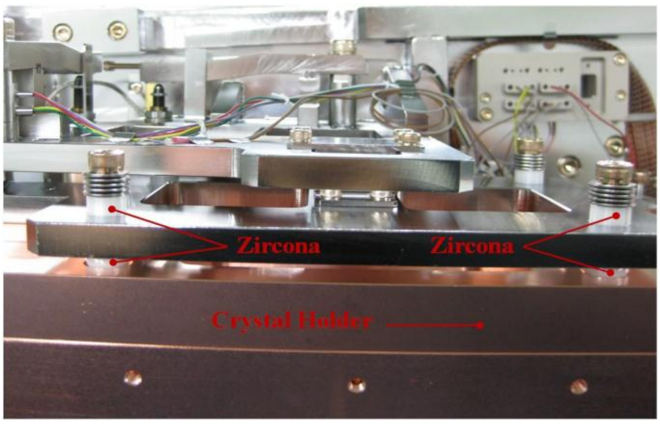


Figure 4. Insulation between the two structures.

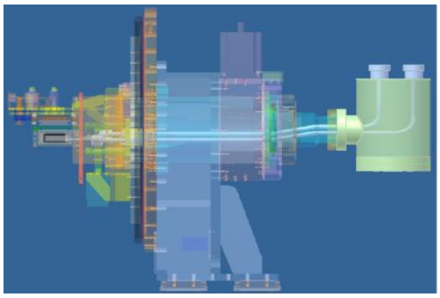


Figure 5. Schematic design for LN_2 cooling chamber.

RESULTS OF BEAM PERFORMANCE

A. Thermal stability

The thermal stability of the 1st crystal has a strong influence on the stability of the energy resolution of the beam line. Consecutive energy scanning experiments have been performed at the Cu K-edge for Cu foil using X-ray absorption near edge structure (XANES)[8]. Figure 6 shows that all ten scans have almost same shape[9]. That is to say, each of ten spectra has very good reproducibility. However, red line around 9,000 eV shows little difference among spectra, because of vibration from cooling devices. When pumping speed reduces, this phenomenon is disappeared. From this result, we notice that thermal deformation of crystal surface is very minimal, even though consecutive scanning.

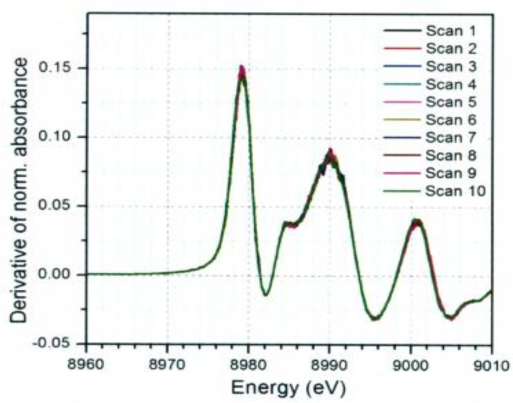


Figure 6. Consecutive scans of Cu K-edge XANES.

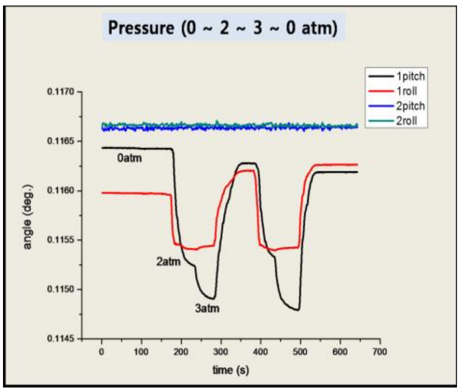


Figure 7. Result of angular variations of two crystals.

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B. Angular variation in accordance with internal pressure of cooling line

In order to cool the 1st crystal, the LN_2 cooling line is directly integrated with the 1st crystal block. And, to eliminate the deformation in the cooling pipe due to thermal expansion-contraction of the cooling line, a bellows is inserted in front of the VCR fitting. Because of that, a certain amount of LN_2 pressure is applied to the 1st crystal and this causes a torque to be applied to the 1st crystal block, which is fixed to the rotating plate. The angular variations of the two crystals caused by this torque were measured.

After the test instruments were configured, gaseous nitrogen was used for the test, instead of using LN_2. The method of measurement called for the autocollimator to be set up for a continuous measurement (every second), and under these conditions a nitrogen regulator was varied from 0 atom to 2 atom and then again from 2 atom to 3 atom and in this condition the finally the nitrogen was vented all the while the variations in the pitch and roll of the 1st and the 2nd crystals were measured. The resulting data is shown in Fig. 7. The associated errors in the pitch and roll of the 1st & the 2nd crystals in accordance with variations in the internal pressure of the cooling lines are approximately 0.003 for the pitch angle of the 1st crystal and, for the 2nd crystal, the pitch angle error are 0.001. Since the 2nd crystal is connected to the 1st crystal via the copper braid for indirect cooling, the angular errors are close to being zero.

Figure 8. Configuration of test instrument.

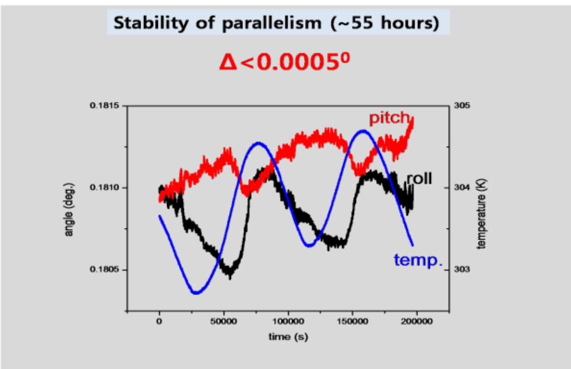

Figure 9. Stability of parallelism over time.

C. Stability of parallelism

To verify the stability of parallelism between the two crystals, after test instruments were configured (see Fig. 8), angular variation was measured in a steady state without any movement for over 55 hours in atmosphere while monitoring the ambient room temperature. After setting the Bragg angle to 10°, a laser beam is injected from the optical head located on the Beam-In and this reflects off the 1st crystal and then the 2nd crystal. Due to the 2nd crystal, a light is emitted at the beam off-set of 25 mm, and, in order to detect the angle of the light coming out of the Beam-Out, a detectable optical head is installed and the angle was measured. The result, as shown in Fig. 9, ascertains variations in the angle in accordance with the temperature[10]. The angle of the emitted light beam is determined by parallelism of the two crystals. As shown in Fig. 9, there is about a 0.0005° angular difference between the pitch and roll of the two crystals.

D. Angular Variation during Energy Scan

To measure parallelism of the two crystals in the energy scan mode, a measurement setup was configured as shown in Fig. 8. During energy scan, as the Bragg angle varies, the 2nd crystal moves rapidly in the Z direction to maintain the beam offset. The method of measurement calls for the autocollimator to be set up for a continuous measurement (every second). Under these conditions, the energy scan is performed by going from 8.0 KeV to 18 KeV and then back down to 8.0 KeV, while measuring the laser beam angle coming out of the Beam-Out. Figure 10 shows that during the energy scan the pitch angle error in parallelism of the two crystals is approximately 0.005° and the roll angle error is approximately 0.002° [10].

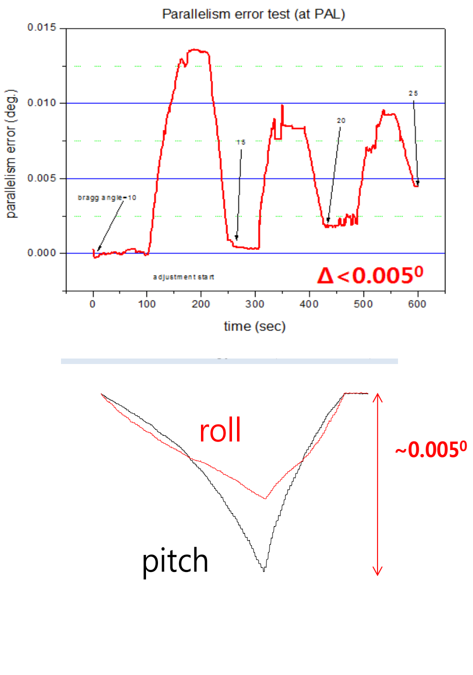

Figure 10. Test result of the parallelism during energy scan (8~18~8Kev).

CONCLUSION

The purpose of this study was to design a double crystal monochromator for maximizing the beam line stability thru improving thermal deformation on the surface of crystal. For this purpose, we have investigated several designs and developed this device which has indirect cryogenic cooling system. This study shows our cooling system was very suitable for reducing vibration. Due to improved vibration stability, high stability of parallelism between two crystals, especially during energy scan, could be acquired. We think this kind of easier cooling structure will cause the cost reduction of manufacturing and operating also.

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