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A double crystal X-ray monochromator for the SpLine diffraction and absorption synchrotron bending magnet beamline at the ESRF

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Abstract. The CRG BM25-SpLine Beamline is located at bending magnet 25 of the ESRF. The beamline, which is split in two branches, is devoted to XAS, XRD and HAXPES. The photon energy covered by both branches range between 5 and 45 keV. The beamline double crystal monochromator (DCM) uses two parallel Si(111) crystals in (+, -) configuration to produce a monochromatic exit beam parallel to the incident white X-ray beam. It accepts 2mrad of radiation from the bending magnet. The DCM has been recently upgraded. Several special features concerning the cooling of the first crystal, second crystal positioning and sagittal focusing mechanism has been improved. In this work a detailed description of the performed modifications is presented.

1. Introduction

The main goal of the Spanish Collaborating Research Group (CRG) BM25-SpLine X-ray beamline is to satisfy the needs of the Spanish scientific community with a broad range of interests across very different research areas: physics, chemistry, material science, biology, environmental sciences and cultural heritage. Hence, the CRG BM25-SpLine beamline has been conceived as interdisciplinary and multipurpose scientific infrastructure. The beamline is installed at the D25 bending magnet of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. It is split in two branches, A and B, with independent optics and experimental hutches. The Spanish CRG BM25-SpLine beamline is dedicated to structural investigations using hard X-ray scattering mostly in materials science, specialized on X-ray absorption spectroscopy, X-ray diffraction techniques and hard X-ray photoemission spectroscopy [1].

Such a broad range of experimental techniques imposes many technical requirements to the X-ray monochromator. Mainly, high stability, in terms of photon energy, intensity and position, during photon energy scans and for fixed photon energies is strictly mandatory. Small beam sizes are also needed for a wide range of photon energies which should be maintained during photon energy scans. All these accomplishments are fulfilled by the double crystal monochromator installed at SpLine. Recently, the monochromator has been upgraded by the inclusion of a new cooling system and new piezoelectric devices for the pitch, roll, yaw and bender movements of the monochromator second crystal.

2.1 First crystal cooling system based on Ethanol

The monochromator is a pseudo channel-cut type with two fixed Si(111) crystals moved together by a simple goniometer circle, in the (-n,+n) configuration. Considering the thermal conductivity and thermal expansion coefficient of Si, the ideal cooling temperature is around 100K [2,3]. Generally this condition is achieved by using pressurized liquid nitrogen (LN2), which supposes a relative complex and expensive pumping system. An additionally difficulty is the poor heat transfer properties of LN2 compared to water or ethanol which limit the heat per unit area that can be dissipated. At SpLine we have developed a less expensive alternative to the standard LN2 cooling used at the ESRF. This novel cooling system is based on ethanol, which has similar [4] cooling properties as water [5,6] but can be operated at much lower temperatures. The new cooling system use as coolant liquid ethanol at -80^{o} C to cool down the monochromator first crystal while the second crystal is kept at room temperature. Although the water cooling is well adapted to the power received by the radiation emitted by the ESRF bending magnet, it imposes severe limitations that degrade the quality of the monochromatic beam delivered for the experiments. Lowering the cooling temperature from 20^{0}C for water to -80^{0}C for ethanol, the thermal conductivity (k) is enhanced and the thermal expansion coefficient (α) is reduced. The combination of these two effects produces a decrease of the ratio α/k and consequently an improvement in α/k *ratio* of 75%. The cooling system consists of a circulation pump, transfer tubes and ultra-low refrigerated circulators (refrigerators) with ethanol accumulation tanks. A picture of the two (one for each beamline branch) ultra-low refrigerated circulators with the insulated transfer tubes is shown in Figure 1. The ethanol is pressurized by the pump and cooled to -80^{0}C. The low temperature liquid cools the first crystal of the fixed-exit double crystal monochromator and returns to the accumulation tank. The total cooling capacity of the system is 800 W at -80^{0}C, which includes heat loads from the crystals, the pump, the pipes and the vacuum chamber. Thus, the net capacity to cool the crystals is estimated to be about 500 W. Figure 1 shows a picture of the monochromator first crystal. The heat absorbers consist of two copper cooling jackets clamped to the crystal side faces via an indium/gallium liquid film, in order to ensure a good thermal contact and to relax the thermal differential dilatation strains with silicon. A copper circulation pipe of 6 mm diameter is brazed to the absorber at the upper part. This part is close to the beam impact point on the silicon crystal, which in this way reduces the head path from the hot point to the copper absorber.

Figure 1. (Left) Picture of the two (one for each beamline branch) ultra-low refrigerated circulators with the insulated transfer tubes, and (right) picture of the monochromator first crystal.

The efficiency of the ethanol based cooling system was tested at the Branch B of the SpLine beamline for different radiation powers, i.e., different primary slits apertures, and compared with the values obtained with the water based cooling system for identical conditions. The test was performed at

Branch B where the radiation power is higher than in Branch A (315 W compared to 150W for a ring current of 200 mA). The photon energy used was 20 keV which corresponds to the critical energy of Branch B. For the tests, no optical element was placed before the monochromator so the radiation power was directly transferred to the monochromator. It can be clearly seen in Figure 2a, for the case of water as cooling liquid, that the rocking curve of the monochromator second crystal gets broader as the radiation power is increased evidencing the departure from the monochromator ideal conditions, (rocking curve FWHM = 17 µrad). For the highest values of radiation power (above 50% of the maximum delivered power) the rocking curve is completely distorted showing the presence of two peaks. The FWHM of the rocking curve, as shown in Figure 2c, changes from 18 µrad to 41 µrad for 5% and 100% of the maximum radiation power, respectively. On the other hand, for the case of ethanol as cooling liquid, the rocking curve also gets broader with the increase of radiation power (see figure 2b) but the FWHM of the rocking curve for the maximum radiation power is 21 µrad (Figure 2c). An enhancement of the monochromator efficiency is clearly obtained. In real working conditions, a mirror, which is coated with Rh and operated at 2.5 mrad (cut-off=26.8 KeV, vertical angular acceptance=100 µrad), is placed before the monochromator reducing considerably the power received by the first crystal of the monochromator. The rocking curve obtained with the ethanol based cooling system for the mentioned working conditions is shown in Figure 2d. A value of 17.9 µrad is obtained for the rocking curve FWHM being very close to the ideal value.

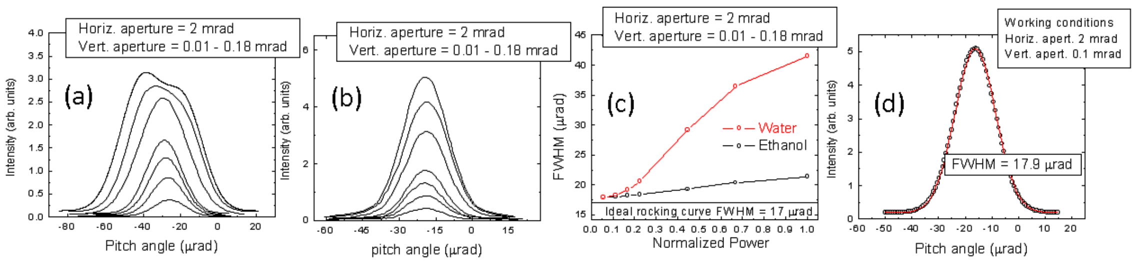

Figure 2. Rocking curves for different radiation powers for water (a) and ethanol (b) as coolant for a photon energy of 20keV. (C) FWHM of the rocking curves. (d) Rocking curve in working conditions. A mirror is placed before the monochromator reducing considerably the power received by the first Si crystal. The obtained value for the FWHM of the rocking curve (17.9 µrad) is close to the ideal conditions.

2.2 Large course piezoelectric for pitch, roll, yaw and bender

The second crystal is equipped with four special piezo stepping motor drivers that allow controlling the parallel setting of the double crystal monochromator. The positioning of the second monochromator crystal is performed by three independent (pitch, roll and yaw) special devices developed for SpLine by Physics Instruments (PI), which combines a step walk and an analogue movement. The Nexline PI piezo motors (N_PI) offer much higher resolution and forces than ultrasonic piezo motors. Such devices can perform a total course of 2 mm with 5 nanometer resolution. In open-loop operation a resolution of 25 pico-meters can be achieved. The N_PI generates a push/pull force up to 50 N with 70 N holding forces. The piezoelectric devices are placed in a configuration in which the 2 mm course produces an angular rotation in three perpendicular directions (pitch, roll and yaw) of the crystal of 20 mrad. Such a large angular movement allows recovering the parallelism between the two monochromator crystals for a wide range of Bragg angles. In this way, the intensity of the monochromatic beam is maximized during energy scans of more than several keV. The inclusion of such positioning devices offers a fast response maintaining high stability. Figure 3 shows a scheme and a real picture of the second monochromator crystal where the piezoelectric devices can be seen.
The sagittal focalization is performed by mechanical bending of the second crystal. A novel bender curves sagittally the Si(111) crystal in order to focus the beam dynamically at different sample

positions. The bender, shown in Figure 3d, consists of a monolithic link mechanism [7] with six notch flexure hinges which assures a symmetrical action on the opposite edges of the thin crystal. The bender is expanded with Nexline PI piezo driven. A picture of the whole system is shown in Figure 3c. The curvature radius is varied between 1.1 and 22 m with a mechanical effort between 5 and 60 N. Such curvature radius enables focussing at a distance of 17 m from the monochromator for photon energies between 5 keV and 40 keV. The measured beam size (FWHM) at the sample position (knife-edge method) is 330 μm. It should be stressed that the horizontal acceptance divergence of the optics is 2 mrad so a beam size of 100 mm is obtained at the sample position (50 m from the source) if no sagittal bending is performed.

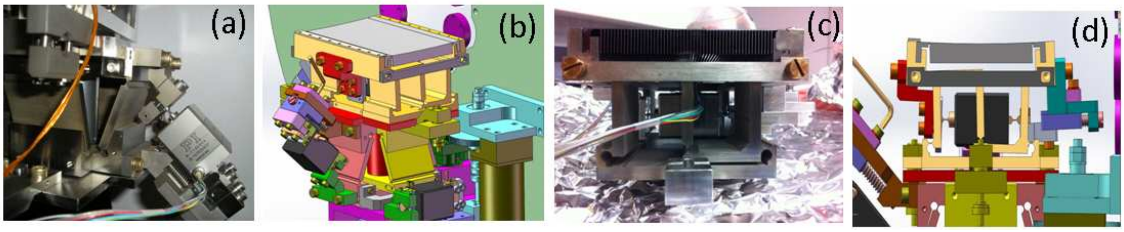

Figure 3. Picture (a) and scheme (b) of the second monochromator crystal. The pitch, roll and yaw angular movements are performed with piezo stepping motor drivers. Picture (c) and scheme (d) of the monolithic link mechanism that holds the second monochromator crystal. The bender is expanded with piezo stepping motor drivers.

3. Conclusions

We have recently performed an upgrade of the double crystal monochromator of the Spanish CRG beamline at the ESRF. A new monochromator cooling system based on ethanol at -80^{0}C as thermal exchange liquid has been installed and tested. An enhancement of the monochromator efficiency has been obtained. Also, the independent movements of the second crystal (pitch, roll and yaw) are now performed by three Nexline PI piezo motors which offer a total course of 20 mrad with a positioning resolution of 0.025 μrad. A fourth Nexline PI piezo device is used to bend the second monochomator crystal so to sagitally focalize the beam at different positions. A beam size of 300 micrometres has been obtained at the sample position. The performed monochromator upgrades resulted in an enhancement of the quality of the monochromatic beam delivered for the XAS, XRD and HAXPES experiments

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References

[1] Castro G R 1998 Journal of Synchrotron Radiation 5 657
[2] Bilderback D H, Freund A K, Knapp G S and Mills D M 2000 Journal of Synchrotron Radiation 7 pp. 53-60
[3] Carpentier P, Rossat M, Charrault P, Joly J, Pirocchi, Ferrer J-L, Kaikati O and Roth M 2001 Nuclear Instruments and Methods in Physics Research A 456 pp. 163-176.
[4] Lange's Handbook of Chemistry 10th ed, pp 1522-1524.
[5] Ramires M L V, Nieto de Castro C A, Nagasaka Y, Nagashima A, Assael M J and Wakeham W A 1995 J. Phys. Chem. Ref. Data Vol 24 No 3 pp 1377-1381
[6] Kestin J, Sokolov M and Wakeham W A 1978 J. Phys. Chem. Ref. Data Vol 7 No 3 pp. 941-948
[7] Tanaka M 1983 Japanese journal of applied physics 22 (1) pp. 193-200