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TEMPERATURE CONTROL OF CRYSTAL OPTICS FOR ULTRAHIGH-RESOLUTION APPLICATIONS*

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Abstract

The temperature control of crystal optics is critical for ultrahigh-resolution applications such as those used in meV-resolved Inelastic X-ray Scattering (IXS). Due to the low count rate and long acquisition time of these experiments, for 1-meV energy resolution at ~10 keV, the absolute temperature stability of the crystal optics must be maintained below 4 mK for days to ensure the required stability of the lattice constant, thereby ensuring the energy stability of the optics. Furthermore, the temperature control with sub-mK resolution enables setting the absolute temperature of the individual crystal, making it possible to align the reflection energy of each crystal's rocking curve in sub-meV precision thereby maximizing the combined efficiency of the crystal optics.

INTRODUCTION

The crystal optics employed in X-ray beamlines at synchrotron facilities utilize perfect single crystals such as silicon and diamond. Their performance in resolving power is governed by the perfection of their crystal lattice and dynamic X-ray diffraction: $\Delta E/E = -\Delta d/d$, where Δd is the lattice (d) variation across the crystal for the chosen reflection. When subjected to varying temperature environments, the changing temperature introduces changes in the lattice constant given by $\Delta d/d = \alpha\Delta T$, where α is the thermal expansion coefficient of the crystal. For Si ($\alpha = 2.6\times10^{-6}$ K^{-1}), therefore, a 40 mK temperature variation will shift the diffraction energy by ~1 meV for 10 keV X-rays. To achieve 1 meV resolution in a stable condition, a 4 mK temperature stability or better, corresponding to 1/10 of the energy resolution, is required.

In this article, we report the details of an EPICS temperature control system using PT1000 sensors, Keithley 3706A 7.5 digits sensor scanner, and Wiener MPOD LV power supply for the analyzer optics of the IXS beamline 10-ID at NSLS-II [1]. The crystal optics part of the analyzer is depicted in Fig. 1 schematically, where the dispersing (D) crystal has a total length of 1.2 m to achieve the required angular acceptance and is made up of 6 highly asymmetrically cut silicon crystals of 200 mm in length each. The lattice homogeneity and temperature stability of these D-crystals are the most critical for the performance of the analyzer. The temperature control system was applied to them. We were able to achieve absolute temperature stability below 1 mK and sub-meV energy alignment for these D-crystals. The EPICS ePID [2] record was used for the control of the power supplies based on the PT1000 sensor input that was read with a 7.5-digit resolution from the Keithley 3706A scanner. The system enhances the performance of the meV-resolved IXS spectrometer achieving a 1.4 meV total energy resolution with unprecedented spectral sharpness and stability for the studies of atomic dynamics in a broad range of materials.

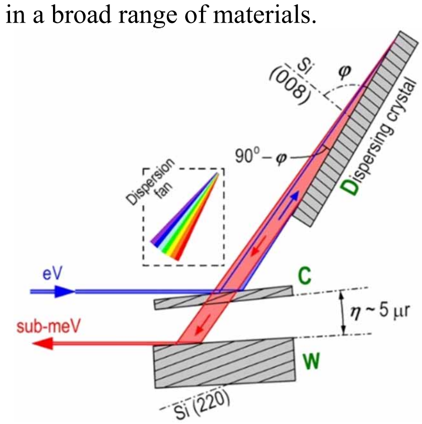

Figure 1: Schematic layout of the analyzer crystal optics employed at the meV-IXS spectrometer of the IXS 10-ID beamline at NSLS-II.

CONTROLLERS

Crystal temperature controls consist of a Keithley 3706A [Fig. 2 (top)] 7.5-digit resolution scanner. The Keithley Instruments Digital Multi-Meter (DMM) 3706A 6 slot system switch utilizes a 3724 dual 1/30 FET Card (Auto CJC with 3724-ST). Such a system allows us to measure up to 30 PT1000 sensors per 3724-ST card by scanning selected inputs. The 3706A takes input from the in-vacuum PT1000 temperature sensors attached to the copper base of each D-crystal of the analyzer optics. The copper base of each D-crystal is fitted with three PT1000 sensors, one at each end, and one near the heating element which is used for control. The control driver uses EPICS State Notation Language sequencer [3] which reads individual PT1000 inputs. The values read by the Keithley 3706A DMM are passed to EPICS PVs [2]. The Temperature PVs are processed by ePID EPICS PVs and provided as feedback to adjust the power output of the MPOD [Fig. 2 (bottom)] power supply channel providing heat to each D-crystal.

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Figure 2: Keithley 3706A (top), and MPOD power supply (bottom).

The raw temperature dependent PT1000 resistance values and values converted to temperature in Celsius, by EPICS calc records, are shown in Fig. 3. As a power supply we used a crate MPOD Micro 2 LX LV model MPB2C2303R1T, 800 W, LV, no HV, 2 slots, with MpodC controller. The MPOD crate is populated with a single MPOD LV module MPV8030I, providing 0 to 30V/2.5A max per 8 channels with 50 Watts per channel.

Figure 3: Temperature PVs read from Keithley 3706A for each PT1000 sensor.

The Temperature values read by the Keithley 3706A are processed by EPICS ePID (Fig. 4) record as input, with tuned values of PID parameters. The output of ePID shown in Fig 4, feeds the value of the voltage of the MPOD power supply. We have intentionally chosen a long scan period of 10 seconds, to minimize wear of the mechanical contacts of the Keithley 3706A DMM scanner. We observed and had to ensure that the ePID value was larger than zero since zero input of the MPOD power supply turns it off and disables it for further control. This is shown in Fig. 4, as the minimum value of ePID output calc was set to 0.01 C.

The MPOD power supply control screen shown in Fig. 5 allows EPICS control of individual power supply channels to be turned on/off and the voltage output (heating power) to be adjusted. Each of these channels controls the heater power of one of the D-crystals of the analyzer optics. When the analyzer temperature control is enabled, each respective power supply voltage is adjusted by the ePID record to stabilize the temperature.

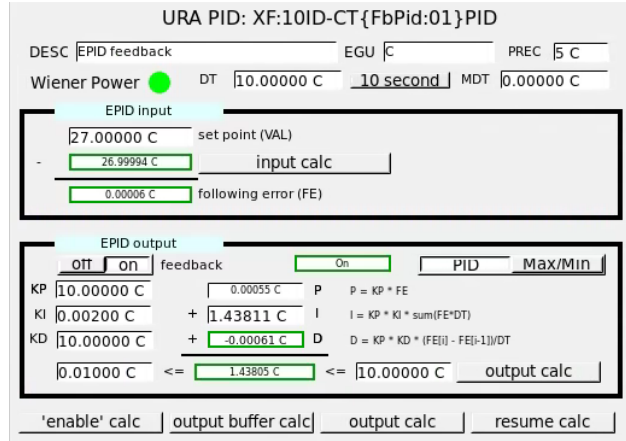

Figure 4: ePID EPICS record computes output power to specific MPOD power supply channel.

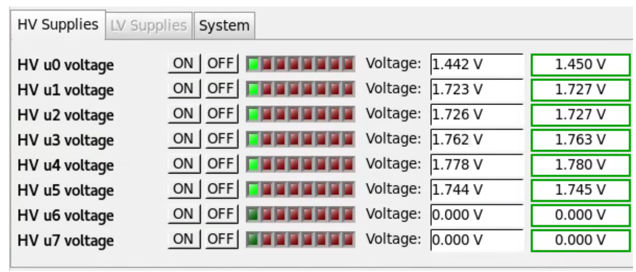

Figure 5: MPOD Power supply voltage fed from the ePID EPICS record controls the power delivered to the D-crystals.

MECHANICAL

The heater element and temperature sensors are attached to the Cu base supporting the D-crystal (Fig. 6). The D-crystal is then supported through two indium stripes at its Airy points on the Cu base to minimize lattice bending by gravity, and is enclosed with a Cu enclosure attached to the Cu base. The entire mechanism is installed in a low vacuum environment providing sufficient thermal sensitivity to control the temperature of the crystals to achieve a stability of <1 mK/24hr and to align and maintain their diffraction energy to optimize reflectivity.

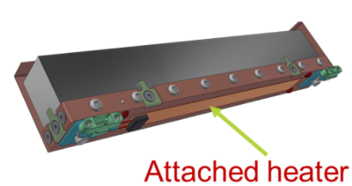

Figure 6: Crystal housing assembly showing the heater and sensor attachment to the D-crystal.

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RESULTS

Using the EPICS ePID control we exceeded the required 4 mK temperature stability of the meV-IXS spectrometer analyzer, which resulted in an overall energy resolution exceeding 1.5 meV at an incident photon energy of 9.1 keV.

The energy resolution of each of the D-crystals, aligned in energy, is shown in Fig. 7. Each of the six D dispersive silicon crystals exhibits small energy variation along the length of the crystal due to intrinsic lattice inhomogeneity and/or gravity-induced bending, which limits the overall resolution of the analyzer. However, all crystals are aligned in energy and stabilized for extended period of time for data collection.

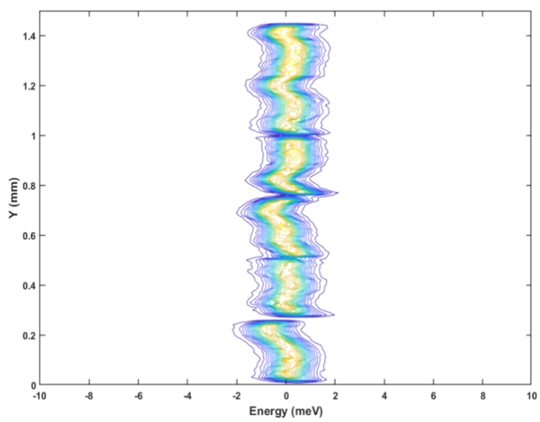

Figure 7: The reflectivity contour of the temperature-stabilized and energy-aligned D-crystals vs. energy resolution. The Y axis corresponds to the beam height for each of the 6 D-crystals in the analyzer.

The temperature response and settling of the system are shown in Fig. 8, each curve showing the temperatures at the ends of the D-crystals and at the heater control point. The far end of the crystal [middle violet curve], has a smaller impulse response and reaches a stable value of temperature after a small delay.

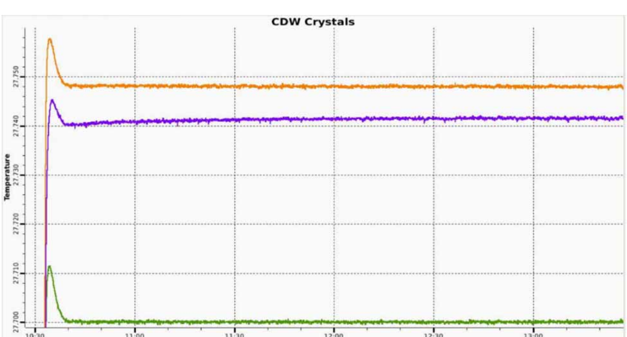

Figure 8: Temperature response of one of the D-crystals. The three PT1000 sensors were mounted at the control point, and each of the two crystal ends respectively.

Further studies shown in Fig. 9 capture the response of the system of all six D-crystals when temperature feedback is turned on and off.

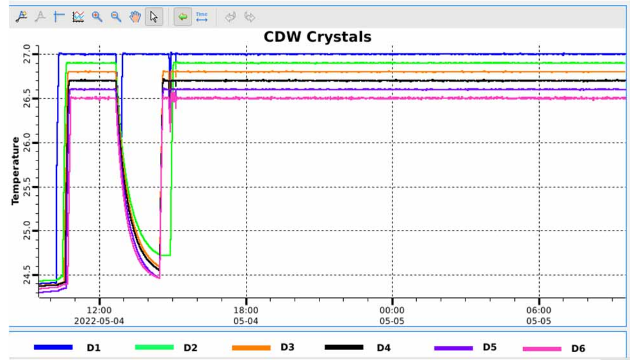

Figure 9: Temperature response of the 6 D-crystals with and without ePID temperature control.

Figure 10 compares the temperature stability of the D-crystals with and without feedback. Not shown is the observation that the temperature of the crystals without feedback fluctuates and follows the day/night cycle over longer periods of weekly/monthly weather patterns.

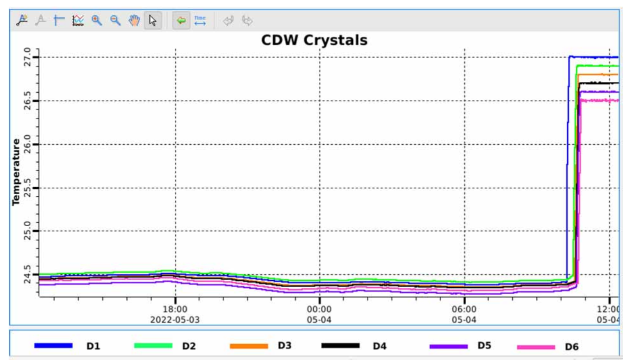

Figure 10: Temperature stability with and without ePID feedback.

Figure 11 shows details of the temperature stability of a typical D-crystal stabilized using the EPICS ePID record method presented here. As there was no active cooling implemented, the temperature of the D-crystals is maintained slightly above the temperature of the environment, to allow effective control.

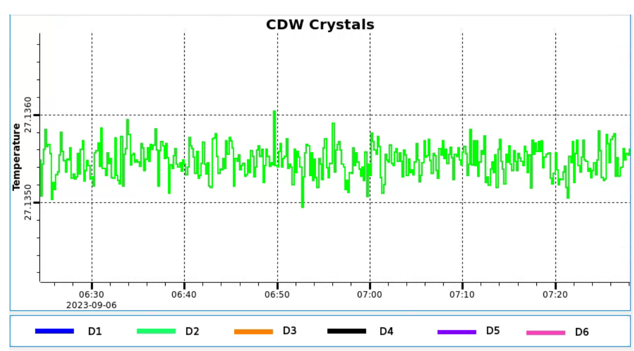

Figure 11: Temperature stability of one of the D crystals with ePID feedback, showing a stability of below 1 mK.

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SUMMARY

Using EPICS ePID control we achieved the required 4 mK temperature stability of the D-crystals for the analyzer crystal optics of the meV-IXS spectrometer for the NSLS-II IXS 10-ID beamline. The temperature stability is less than 1 mK during a typical one-week measurement. Without ePID EPICS temperature control, the temperature stability has been within 100-500 mK. The resulting energy resolution of the IXS 10-ID beamline is now limited by the perfection of crystal optics to about 1.4 meV.

ACKNOWLEDGMENTS

We thank Rick Green for help with the mechanical aspects of the beamline components.

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[3] EPICS sequencer (snc/seq), http://epics.web.psi.ch/software/sequencer/