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Low Energy X-Ray Spectrometer*

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ABSTRACT

A subkilovolt spectrometer has been produced to permit high-energy-resolution, time-dependent x-ray intensity measurements. The diffracting element is a curved mica ($d = 9.95\text{\AA}$) crystal. To preclude higher order ($n > 1$) diffractions, a carbon x-ray mirror that reflects only photons with energies less than $\sim 1.1\text{ keV}$ is utilized ahead of the diffracting element. The nominal energy range of interest is 800 to 900 eV. The diffracted photons are detected by a gold-surfaced photoelectric diode designed to have a very good frequency response, and whose current is recorded on an oscilloscope. A thin, aluminum light barrier is placed between the diffracting crystal and the photoelectric diode detector to keep any UV generated on or scattered by the crystal from illuminating the detector. High spectral energy resolution is provided by many photocathodes between 8- and 50-eV wide placed serially along the diffracted x-ray beam at the detector position. The spectrometer was calibrated for energy and energy dispersion using the Ni $L_{2,3}$ lines produced in the LLNL IONAC accelerator and in third order using a molybdenum target x-ray tube. For the latter calibration the carbon mirror was replaced by one surfaced with rhodium to raise the cut-off energy to about 3 keV. The carbon mirror reflection dependence on energy was measured using one of our Henke x-ray sources. The curved mica crystal diffraction efficiency was measured on our Low-Energy X-ray (LEX) machine. The spectrometer performs well although some changes in the way the x-ray mirror is held are desirable.

INTRODUCTION

A subkilovolt x-ray spectrometer having a high- and variable-energy resolution and good frequency response was needed for making time-dependent measurements of diffracted x rays. The nominal photon energy range of interest is 800 to 900 eV (15.5 to 13.8 $\text{\AA}$). We wanted a spectral energy resolution between 8 and 50 eV. The temporal response of the x-ray detector to a step function input should be a few ns. Figure 1 shows a cross section and Fig. 2 a photograph of the total spectrometer.

DIFFRACTION CRYSTAL

The photon energy range of interest dictated a reflection spectrometer to avoid the absorption of the diffraction crystal. Mica (002 plane) was selected as the diffracting crystal because it was readily available, easily curved without fracturing, and did not require special handling or environmental conditions. Alternatives to mica are crystals such as KAP, RAP, and lead stearate. Mica does have a serious drawback in this planned use; it has strong high-order diffractions, especially in the odd orders. The spectrometer would be used in

an environment where there would be photon energies at intensities sufficient to result in higher-order diffractions, adding significantly to the signal in the energy interval of interest. These higher energy photons had to be eliminated. To provide for this low-pass filter, an x-ray mirror was introduced in front of the diffracting crystal and is discussed in the following section.

It can be shown that there is a relationship between the range of photon energies to be observed, the radius of curvature of a bent diffraction crystal, and the width of a parallel x-ray beam incident on the crystal. The relationship is:

$$W_b = r (\cos \theta_h - \cos \theta_l)$$

where

- W_b = beam width (cm) of incident x rays,
- r = radius of curvature of bent crystal (cm),
- θ_h = Bragg angle of highest energy photon desired detected,
- θ_l = Bragg angle of lowest energy photon desired detected.

Figure 1. Simplified cross section drawing of spectrometer showing x-ray path along various internal components.

Figure 2. Spectrometer body showing ball and socket alignment/attachment flange, access and pump out ports, vacuum bellows, and x-ray detector.

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The Bragg angle is:

$$\theta = \sin^{-1} \left[6.20 \times 10^3 / (d) (E/n) \right]$$

where

d = diffraction crystal spacing ($\text{\AA}$),

E = photon energy (eV),

n = diffraction order.

It can also be shown that $\theta_h < 3\theta_n$, which precludes photons diffracted from the crystal at the high energy limit from intersecting the crystal.

For the spectrometer design, the mirror length and angle selected set $W_0 = 5.28$ mm. Other experimental parameters set are $d = 9.95$ $\text{\AA}$, E_h = photon energy associated with $\theta_h = 900$ eV, E_n = photon energy associated with $\theta_n = 800$ eV, and $n = 1$. Applying the above expressions, $r = 5.59$ cm. A radius of curvature of $r = 5.08$ cm was selected to allow detection of a somewhat larger energy range, and with the fixed beam width, the resulting energy range was $E_n = 794$ eV to $E_h = 903$ eV. The crystal depth selected was 3 cm.

The mica crystal stock received from the vendor was initially checked for diffraction efficiency. This was done with a flat crystal in each of four different orientations 90° apart on both sides of the crystal. By using the same orientation that gave the highest efficiency when the crystal was flat, it was felt that one could optimize the chances of getting the highest diffraction efficiency when the crystal was curved. The crystals were mounted in a picture frame crystal holder appropriately designed to achieve the desired radius of curvature and allow for finite crystal thicknesses. Figure 3 shows the bent crystal and associated holder. The thinner crystals ($\sim 5 \times 10^{-2}$ mm) appeared to have a more uniform radius of curvature when bent than those twice as thick. After mounting, each crystal was checked in third order with a molybdenum X-ray source to assure the range of energies desired was achieved.

We experimentally determined the diffraction efficiency of each bent crystal by the method discussed in Ref. 1. The subkilovolt diffraction efficiency measurements were made on the LLNL IONAC (2) accelerator and the 1- to 4-keV determinations were made on the Low-Energy X-ray machine (3). Efficiency measurements were thus made at two energies for second, third, and fourth orders;

Figure 3. Crystal/collimator assembly attached to stand-off housing.

at four energies for first order; and at a single energy for fifth order. Typical results can be seen in Fig. 4 for the various orders, and Fig. 5 shows the efficiency for two different crystals in first order in the photon energy range of interest. The data points in Figs. 4 and 5 are connected with straight lines for ease of reading and inferring trends but are not authoritative on intermediate values.

Figure 4. Curved mica crystal (roc = 5.08 cm) diffraction efficiencies for $n = 1, 2, 3, 4$, and 5.

Figure 5. Curved mica crystal (roc = 5.08 cm) diffraction efficiencies for two different crystals at subkilovolt photon energies, $n = 1$.

X-RAY MIRROR

The mirror material chosen for the x-ray mirror was vitreous carbon. Beryllium would also have been satisfactory, but because the mirrors required handling and exposure to air, carbon was selected. The cut-off energy (~60% reflection) selected was 1.1 keV. At the low energy end of the range of interest (800 eV), we had to avoid possible second-order contributions at 1.6 keV, and at the high energy end (900 eV) we wanted to have a mirror reflection like that for the rest of the range of interest (~70%). The cut-off energy choice was biased toward the 900 eV as the reflection generally falls off rapidly but has a tail. We wanted the reflection to be as near to zero as possible at 1.6 keV, while still having a reasonable reflection at 900 eV.

The 1.1 keV cut-off energy engendered a calculated 1.3° angle between a parallel incident beam of x rays and the mirror surface. The vitreous carbon mirror stock was available in lengths up to 30.48 cm. It was considered too much of a problem to properly align the mirrors in series to get an effective length of multiples of 30.48 cm, so a single mirror of that length was used. This decision influenced the rest of the design of the spectrometer because the mirror length and angle determine the maximum x-ray beam width incident on the diffracting crystal; here that would be 6.92 mm. A beam width of 5.28 mm was selected to allow some latitude in mirror positioning, i.e., ± 0.82 mm.

The spectrometer was mechanically designed to allow us to remove and replace the mirror assembly with a high degree of assurance that its physical position was reproducible; this allowed us to easily align the whole spectrometer to the source of x rays. The vitreous carbon was attached to a glass substrate to provide for mechanical rigidity. The mirror was positioned by three registration screws in the protective cover plate of the mirror holder. The mirror was held against the registration screws and kept centered in the holder by ball plunger screws. This method of positioning was not completely satisfactory. The ball plunger screws did not always keep the mirror in position. During routine handling on two occasions, the mirror was found jammed out of position. Adjustment of the ball plunger screws centering the mirror in the holder proved very critical. If these screws are too tight and the mirror assembly is jarred, the mirror can jam out of position and the ball plunger screws on the mirror back do not restore the mirror surface to its position against the registration screws. The mirror angle was set optically utilizing a transit and appropriately positioned fiducial cross-hairs on an alignment fixture at the end of the spectrometer. Figure 6 is a photograph of the mirror and mirror holder. Figure 7 shows the mirror assembly being placed in the spectrometer body.

Figure 6. Carbon mirror mounted in holder with beam defining collimators attached to each end.

Figure 7. Technician installing mirror holder and collimator assembly into spectrometer body.

Figure 8. Experimental and theoretical reflectivity vs energy for a carbon mirror at 1.3° .

The mirror was lapped with 3- and 1- μ m diameter diamond polishing compound to a satisfactory flatness to achieve a 70% x-ray reflection (4).

The mirror reflectivity was experimentally determined utilizing one of the Henke x-ray machines. The mirror was placed on a goniometer and the direct and reflected spectrum from a copper anode x-ray source determined. The reflected spectrum was then divided by the direct spectrum to determine the x-ray reflectivity as a function of photon energy. The mirror reflectivity is also predicted theoretically using the REFLACT code (5). Figure 8 shows the experimental and theoretical mirror reflectivity as a function of energy for a typical carbon mirror at a 1.3° angle.

UV LIGHT BARRIER

As the x-ray detectors use a gold cathode, which is sensitive to ultraviolet as well as x rays, there was concern that UV emanating from x rays absorbed in the diffraction crystal and UV reflected off the

crystal and associated hardware would illuminate the detector. To mitigate this concern, a UV light barrier of aluminum was placed between the diffraction crystal and some internal collimation ahead of the detector. The barrier was 0.75 μm thick and had a transmission of 0.65 at 852 eV as measured on the IONAC. The aluminum barrier was glued to a stainless steel frame and can be seen in Fig. 3. It is shown being positioned behind the crystal in Fig. 9.

Figure 9. Installation of ultra-violet light barrier behind bent crystal on collimator/crystal assembly.

X-RAY DETECTOR

The detector that sensed the diffracted x rays was designed to accommodate different criteria on spectral resolution over the energy range under study and to be a high frequency response detector.

Achieving the desired energy resolution was a matter of having the x-ray detector sufficiently far from the crystal consistent with a detector geometry that could easily be fabricated. The detector cathodes vary from 8 to 55 mm in width, and the distance from the point of diffraction on the crystal to the detector cathodes ranges from 46 to 50 cm giving an energy dispersion at the detector of 0.65 to 1.0 eV/mm.

Two detector types were built, one with three cathodes and another with seven. Both detector types had a background cathode that would sense x rays scattered from the crystal but would be out of the pattern of diffracted x rays.

Gold was selected as the cathode surface because of its large photoelectric cross section at the energies of interest and because of ease of plating.

The detector, of necessity, had to be windowless and have a vacuum common to that of the spectrometer; because of this, it was desirable to minimize the number of vacuum feedthrough electrical connectors. It was decided to operate the cathodes at a negative high voltage requiring but one connector per cathode; however, an isolation capacitor is necessary to record the signal. The anode was at ground potential and was made of a nickel screen.

A prototype detector was made to verify the design and make measurements of frequency response, voltage standoff, etc.; refinements to the initial design were made. We finally built a detector that had two layers of cathodes and two anode screens. The two layers of cathodes, one behind the other, permitted differing spectral resolution and

continuous energy coverage where desired. One anode screen was in front of all cathodes, and the second was between the two cathode layers. The anode screen had a transmission of ~81% (the open area ratio). The cathodes were operated at -4 kV and, with the electrode spacing used, gave a calculated Child's Law space charge limited current of ~40 A/cm². Figure 10 is an exterior view of the seven-cathode detector; Fig. 11 is a view of the cathodes looking in the direction from which the x rays would be incident. Figure 12 shows the three-element detector mounted on the standoff connecting the detector and the crystal holder/collimator section.

Figure 10. Multi-cathode x-ray detector mounted on adapter flange with electrical and vacuum feedthroughs shown on body.

Figure 11. Interior view of multicathode x-ray detector showing individual signal cathodes and one background cathode.

Figure 12. X-ray detector and crystal/collimator section attached to stand-off housing. X rays are incident from right.

Figure 13. A typical x-ray detector cathode sensitivity in the subkilovolt photon energy range as measured by EG&G and LLNL.

The detailed design and fabrication of the final detectors was done by the Detector Group at EG&G's Las Vegas Atlas facility. They calibrated the various cathodes for a variety of photon energies. The detectors were also calibrated on the IOMAC. Figure 13 shows typical calibration results determined by the two laboratories. The actual data points are plotted and connected by straight lines to illustrate the trend of sensitivity with energy.

The detector time responses were measured utilizing bremsstrahlung from a tungsten target on the LINAC located at EG&G's Santa Barbara facility (6). While not entirely appropriate for determining the time response in the manner the detectors are used, the LINAC radiation pulse (FWHM ~70 ps) and the detector's response to that pulse were believed to indicate the detector's temporal response. To the LINAC pulse, the largest cathode gave a rise time of ~1.1 ns while the rest of the cathodes varied in rise time between 0.7 to 0.3 ns.

SPECTROMETER

Internal collimation in the spectrometer first defines the x-ray beam incident on the mirror and then inhibits x rays scattered from the mirror, diffraction crystal, or other internal parts from reaching the detector. The internal collimation is shown schematically in Fig. 1, and parts of the collimators can be seen in Figs. 3, 6, 7, 9, and 12 attached to the crystal and mirror holders.

The spectrometer was designed so it could be disassembled after internal alignment and calibration and reassembled with assurance that the alignment and/or calibration was still valid. This was achieved by having all critical alignment mating faces dowel-pinned. As was mentioned earlier, the spectrometer is aligned to the source with the mirror removed; this makes for easy setup as one does not have to establish an offset for the alignment transit to view the reflected source off of the mirror or try looking through the diffraction crystal. There was sufficient concern about the reproducibility of the mirror replacement that the mirror was cycled in and out while the spectrometer was attached to the IOMAC and the position of the nickel $L_{2,3}$ line at the

detector location was determined with a proportional counter. There was no significant shift in the line position recorded, within the resolution of the measurement, when the line was located after the mirror was reinstalled.

Copper gaskets are used at all joints where vacuum seals are required. The spectrometer is rough pumped with a cryogenic absorption pump, and then a valve is opened connecting the spectrometer to an ion pump. Typical operating pressures are 10^{-7} torr.

The calibration of the completed spectrometer for energy vs position at the detector location is made with a molybdenum anode x-ray tube. The calibration is done in third order, necessitating the use of a rhodium x-ray mirror rather than the carbon mirror in order to get the cut-off energy above 2.8 keV for the 1.3° mirror angle. The molybdenum lines were recorded on film. A reference wire casting a shadow in the bremsstrahlung continuum and keyed to the alignment dowel pins and to scribe marks on the detector allowed us to cross reference the energy calibration with the various hardware parts for positioning the detector to the desired energy. The developed calibration film was read with a scanning microdensitometer.

Data processing resulted in three-dimensional plots of density vs position along the diffracted x-ray beam and depth. The density was averaged over depth, and the best line position determined by fitting a polynomial to the average density vs diffracted beam position. The line energy was then compared with the energy calculated utilizing the appropriate parameters of the spectrometer for that same position with a code written to model spectrometer performance (7). A first-order least-squares fit in energy space was made between the standard line energies (8) and theory, and the theoretical energy values adjusted by the first-order fit to determine a continuous set of calibrated energy vs position at the detector location. The detector was positioned according to this calibrated set of energy values. Figure 14 shows the theoretical and calibrated energy vs position. Figure 15 shows the same information on a larger scale so it is easier to see the difference between the two curves; one can see that the calculation

Figure 14. The calibrated and theoretical diffracted photon energy vs position at the x-ray detector location.

Figure 15. The calibrated and theoretical diffracted photon energy vs position at the x-ray detector location; repeated here to better illustrate the difference.

would predict a photon energy of ~1 eV higher at a given position than the calibrated energy value. Figure 16 is the energy dispersion calculated at the detector location. No attempt was made to change this curve to reflect the calibration. The dispersion is useful in sizing cathode lengths along the direction of the diffracted x rays for the varying spectral resolutions and for predicting detector signals.

Using the information given here and some assumptions, it is possible to estimate the uniform intensity per keV of x rays at the spectrometer entrance that would give 1 A of detector current.

Figure 16. Theoretical photon energy dispersion vs position at the detector location.

ASSUMES:

Average photon energy = $\bar{E} = 850$ eV
 Mirror reflectivity = $R = 0.7$
 Crystal diffraction efficiency = $\epsilon = 5 \times 10^{-5}$ cm
 Transmission of UV light barrier = $T = 0.65$
 Energy dispersion at detector = $dE/dx = 0.8$ eV/mm
 Detector cathode area (1 cm wide $\times$ 3 cm deep) =
 $A_d = 3$ cm²
 Detector Sensitivity = $S_d = 90$ A/W
 Current = $i = 1$ A

Now the detector current is:

$$i = \left[\frac{1}{E} \left(\frac{\text{keV}}{\text{cm}^2 \cdot \text{s} \cdot \text{keV}} \right) \right] (R) [\epsilon (\text{cm})] (T) \left[\frac{dE}{dx} (\text{eV/mm}) \right] \times \left[A_d (\text{cm}^2) \right] \left[S_d \left(\frac{\text{A}}{\text{W}} \right) \right] = 1 \text{ A}$$

$$\frac{1}{E} = \frac{1(\text{A})}{(0.7) \left[5 \times 10^{-5} (\text{cm}) \right] (0.65) \left[8 \times 10^{-3} (\text{keV/cm}) \right] \left[3 (\text{cm})^2 \right]}$$

$$\times \frac{1}{\left[9 \times 10^{-5} (\text{A/W}) \right] \left[1.6 \times 10^{-14} (\text{W} \cdot \text{s/keV}) \right]}$$

$$= \sim 10^{24} \frac{\text{keV}}{\text{cm}^2 \cdot \text{s} \cdot \text{keV}} \quad \text{or} \quad \sim 2 \times 10^{10} \frac{\text{W}}{\text{cm}^2 \cdot \text{keV}}$$

CONCLUSION

The spectrometer described has performed satisfactorily; however, any future version should have an alternate means of x-ray mirror positioning and support. The detector has a better than expected temporal response.

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