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CONSIDERATIONS FOR THE USE OF
SYNCHROTRON RADIATION SOURCES TO MEASURE
SUB-keV X-RAY PHOTOABSORPTION
CROSS SECTIONS IN TRANSMISSION

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Considerations for the use of synchrotron radiation sources
to measure sub-keV x-ray photoabsorption cross sections in transmission

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ABSTRACT

Sub-keV x-ray photoabsorption cross section measurements in transmission have been made using synchrotron radiation beam lines on the VUV storage ring at the National Synchrotron Light Source (NSLS) and on the SPEAR storage ring at Stanford. The experimental considerations associated with making absolute measurements are reviewed along with techniques for resolving difficulties. Suggestions for future measurements are included.

1. INTRODUCTION

A program to measure absolute sub-keV x-ray photoabsorption cross sections using synchrotron radiation (SR) has been described in these proceedings.¹ In this paper we discuss in detail the experimental considerations involved in obtaining reliable sub-keV x-ray photoabsorption cross sections in transmission based on our experience using the U14A plane grating monochromator (PGM) beam line on the VUV ring at the National Synchrotron Light Source (NSLS) and more recently the new Beam Line VIII toroidal grating monochromator (TGM) and spherical grating monochromator (SGM) at the Stanford Synchrotron Radiation Laboratory (SSRL/SLAC) SPEAR storage ring. One of the experimental arrangements that we have used is shown schematically in Fig. 1. This consists of a bending magnet SR source, a grazing incidence grating monochromator (SGM) and its focusing optics,² a prefilter, a beam flux monitor, a transmission sample, a transmission detector, and a data acquisition system. An actual experimental arrangement is somewhat more complicated as shown in Fig. 2, since very fragile, thin, free-standing, solid samples must be interchanged and accurately positioned under ultra high vacuum (UHV) conditions. Our sample preparation method based on sputtered multilayer technology that features carbon inner and outer layers, is described in these proceedings. Thorough sample characterization measurements using ion bombardment techniques are also presented.

Figure 1. Schematic representation of a photoabsorption measurement arrangement using the Beam Line VIII SGM on SPEAR.

In order to take into account variations in SR source output, some investigators have obtained transmission results by ratioing sample in versus sample out measurements taken over rather short time periods.^{3,4} As will be discussed in detail in the next sections, we have chosen to compare transmission ratios obtained from rather lengthy (but monitored) grating wavelength scans for multilayer samples and the corresponding carbon-only samples. To account for source intensity variations during these long scans, we rely rather heavily on I_0 monitor data taken simultaneously with transmission detector data.

After a discussion of SR source considerations for these measurements in section 2, we consider sub-keV monochromators and optics in section 3, x-ray flux monitors and detectors in section 4, and prefilters in section 5. Following sample issues in section 6, we discuss experimental considerations based on our preliminary results for uranium, beryllium, and carbon obtained using the NSLS PGM and the new SSRL TGM. We conclude with recommendations for future measurement improvement.

Figure 2. Instrumentation set up in the NSLS U14A UHV target chamber for photoabsorption measurements. The Cu-mesh monitor is near the entrance slits. The two prefilterers are interchangeable and removable. Samples are translated into position horizontally. The x-ray diode features Au, Al, and Cr cathodes that are translated into position vertically.

2. SOURCE CONSIDERATIONS

Synchrotron radiation sources have several obvious advantages for sub-key photoabsorption cross section measurements in transmission. Although it ordinarily takes more than one set of diffracting/reflecting elements per beam line, SR sources can cover the entire sub-keV range continuously, thus permitting scans over sample absorption edges of interest. SR beam line output is monoenergetic (at least to first order since the first order greatly dominates) with moderate to very good energy resolution (0.05 to 2 eV) and with intensities at the sample that are 2 to 3 decades higher than our laboratory line sources.

Low SR output angular divergences and small source sizes in principle permit good focusing of well collimated beams at the sample and hence very good, "narrow beam", transmission geometries. For example, SR source vertical divergences are usually less than three mrad vertically. Bending magnet horizontal acceptance angles are usually less than one degree. Source sizes are roughly $1/2 \times 5 \text{ mm}^2$ FWHM at SSRL SPEAR and much smaller at the NSLS VUV ring.

The main disadvantage of sub-keV SR sources for photoabsorption measurements is spectral contaminations from higher orders and/or specularly reflected light. Most sub-keV beam lines feature general purpose grating monochromators with one operating diffracting element and limited capability for higher-order suppression. Depending on the details of the photoabsorption cross section, these measurements can be very sensitive to the presence of even low amounts of spectral impurities. A contributing factor to this problem is that detectors suitable for sub-keV SR experiments have very poor energy resolution. See section 4.

Some beam lines have critical energy cut-off mirrors designed to suppress higher orders. Our first SR measurements were conducted using the NSLS U14A beam line because of this advantageous feature discussed in the next section.¹⁰

Another significant problem for sub-keV SR measurements is that samples and sample holders must be UHV compatible. The SR sample chamber needs to be kept at pressures below 5×10^{-9} torr (unless, of course, differential pumping methods are used). This complicates and lengthens sample introduction times because of the need for cleaning and bake out of components and chambers.

Another disadvantage of SR sources is the variation of output with time. These variations are due both to short term instabilities and to storage ring decay times. Decay times for SPEAR are 2 to 30 hours and for the NSLS VUV ring are 1.5 to 6 hours. This leads to the requirement for well-behaved x-ray flux monitors, as will be discussed later, or to an alternative means of comparing sample in versus sample out wavelength scans. In addition, SR sources are clearly not as conveniently located as are our laboratory sources, nor do they always operate. They are subject to planned as well as unexpected shutdowns, both short term and long term.

3. MONOCHROMATOR AND FOCUSING MIRROR CONSIDERATIONS

Sub-keV monochromators generally utilize gratings for wavelength dispersion. There are relatively few sub-keV double crystal monochromators.^{11,12} Crystal monochromators don't operate below 800 eV due to lack of suitable natural crystals with large d-spacings. A few single and double multilayer monochromators are now in operation throughout the sub-keV range.^{13,14} We are fortunate to have obtained confirmation measurements on one of the new layered synthetic microstructure (LSM) systems, although these results are discussed elsewhere.^{1,14}

Grating monochromators tend to have one diffracting element covering a moderate range and interchangeable gratings and/or mirrors. They usually have fixed exit angles. As the grating is rotated in order to scan over its range from low to high energies, it approaches a practical high energy limit. This limit is due to a gradual decrease in first order efficiency along with a gradual increase in scattered zero-order spectral contributions that are rather difficult to quantify.

Spectral contributions from higher orders are a problem for transmission measurements particularly at the low energy end of a given grating range. The grating grazing angle decreases with decreasing x-ray energy. The critical energy cut off due to the grating reflectivity increases and becomes less effective in cutting off higher-order contributions.¹⁵ The higher-order suppression capability over a given monochromator range (based on a single grating rotation) depends strongly on the combined reflectivity of the mirrors in the system. Figure 3 shows mirror reflectivity calculations for each of the grating monochromators that we have used for our measurements. The NSLS PGM has the advantage of separate exit mirrors with different wavelength cut-off parameters for each range. The PGM characterization work of Williams, et al., has shown that higher-order suppression is very effective, but that there are still small second-order contributions at the low energy end of each range.¹⁶ For the case of each of the two SSRL grating monochromators, the highest energy range has significantly better higher-order rejection than the respective other two lower energy ranges. Note that the SSRL BL-VIII TGM mirror roll-off behavior compares favorably with the PGM Range 3 mirror-grating characteristics with regard to suppression of higher orders between 100 and 200 eV.

Figure 3. Reflectivity of focusing mirrors calculated (a) for the three ranges of the NSLS U14A PGM line and (b) for the SSRL BL-VIII TGM and SGM lines, respectively.

In conclusion, we thus emphasize that each SR grating range has a central energy region where it is most effective, a low energy region with moderate or serious higher-order contributions, and an upper energy limit characterized by increasing contributions due to specularly reflected light. It is thus very important to get photoabsorption data at more than one SR beam line and several grating ranges.

4. SUB-KEV X-RAY DETECTION CONSIDERATIONS

Sub-keV detectors and secondary monitors used for SR photoabsorption experiments must be capable of responding at high count rates with adequate time response. In addition, they must be linear over a wide dynamic range with an adequate signal-to-dark current ratio. They must be UHV compatible. A suitable smooth response over the energy range of interest is required preferably with high quantum efficiency and good long and short term stability (in air and high vacuum). Uniformity of response over the detecting surface is important, and a tendency to suppress higher orders is desirable. Neither the detector nor the monitor needs to be absolutely calibrated in order to derive absolute photoabsorption cross sections from transmission data. This is not the case for cross sections derived from reflectivity measurements.

We have used metallic photodiodes, either directly or amplified with a channel electron multiplier (CEM), since they meet nearly all of the above detector criteria. Due to the low SR current per pulse and high duty cycle, it is not practical to operate these diodes in the pulsed mode. We therefore measure either the current leaving an isolated cathode or, in other instances, the current from a grounded cathode amplified with a CEM. Figure 2 shows the photodiode that we designed and used in the NSLS U14A UHV target chamber. A translator allowed selection of pure gold, aluminum, or chromium metal cathodes used to cover a wide energy range. The anode was a nonintercepting stainless steel cylinder biased to 90v. This instrumentation is based partly on the design of an aluminum oxide diode designed and calibrated at the National Bureau of Standards (NBS) that we have used for measurements on the SSRL BL-VIII TGM line.¹⁷ The main disadvantage of direct current measurements is low quantum efficiency. This is acceptable if the dark current is low. The cathode thus needs to be shielded and electrically isolated. (In our NSLS design, the cathodes are mounted on quartz disks to accomplish this). Low dark current is also important for the Type 1 monitor shown in Fig. 4. In this case, the monitor current is measured directly from a mesh that intercepts only about 15% of the incident flux. Thus, the monitor signal output is reduced accordingly. We have found that signal-to-noise levels are generally acceptable. However, for best results the dark current has to be measured during a small portion of each scan. We have observed significant dark current changes with time during these SR measurements and have noted interference from ion pumps in some cases.

Figure 4. Two types of flux monitors used for our transmission measurements. In Type 1, a picoammeter measures current from an isolated Cu mesh. In Type 2, current from the mesh, coated with gold in situ, is amplified by a CEM. Gain is adjusted by changing the parameter V_1 from 50 to 250. The adjustable battery box is very well isolated for low noise. A typical battery voltage is 1800v.

The second arrangement shown in Fig. 4 utilizes a CEM with 10^2 to 10^6 gain to amplify the cathode current. This has been used frequently at SSRL¹⁷ and NSLS¹⁸ to provide better signal-to-noise ratios. The dynamic range obtained using a CEM is lower than that of Type 1 configurations, but it is still adequate. We have used CEM arrangements for both detector and monitor recently on the new SSRL BL-VIII SGM beam line. The capability of controlling the gain is a significant advantage particularly for high resolution work requiring narrow slits and reduced throughput. It is not as easy to shield the more open cathode, however, and backgrounds due to stray electron pickup can be more of a problem. The offsets caused by amplification of background electrons may not be easy to understand and hence to correct.

Since the wavelength dependence of the quantum efficiency is characteristic of electron emission from the cathode material, it is the same for both types of detectors and monitors. Figure 5 shows this dependence modeled assuming pure metal cathodes. As previously noted, gold is the metal cathode of choice between 250 and 1200 eV. In this range the gold response is relatively smooth, has a large amplitude, and tends to reject higher-order contributions (assuming a two keV or lower mirror roll off). The aluminum response is more effective below 250 eV down to about 100 eV. (The NBS detector features a very stable aluminum-oxide cathode calibrated between 10 and 250 eV.)¹⁷ We have used Cr below 100 eV where the Al L-edges tend to degrade performance. Copper has been chosen for its moderately smooth response over a wide energy range below the Cu L₃-edge (932 eV). Copper and gold have the advantage that they can be readily vapor deposited in situ, at least for the more open cathode arrangement. This is an advantage for near-edge carbon and oxygen work where it is important to minimize detector surface impurities that degrade the response. Freshly evaporated gold on both the monitor and detector can produce very satisfactory, smooth, and similar responses over the entire SGM highest energy range including the oxygen K-edge (543 eV).

Figure 5. X-ray diode detector response versus photon energy for pure metal cathodes used in our transmission measurements. Optimum ranges for detection are indicated by brackets. Relative quantum efficiencies are modeled using f_2 form factor data from Henke, et al.¹⁹

5. PREFILTER CONSIDERATIONS

Prefilters are used to preferentially reduce the undesirable photon background contributions outside of our specific range of interest. Absorption edges of these prefilter materials provide additional information required for wavelength calibrations. Agreement observed between transmission data recorded with and without prefilters provides a test of the validity of the data. General transmission data for some prefilters used at SSRL and NSLS are shown in Fig. 6. At NSLS we were constrained by limitations of space in the UI4A target chamber to use only two prefilters. (See Fig. 2). We therefore chose molybdenum for its transmission window between 80 and 240 eV and copper for its transmission below the copper L_3 edge at 932 eV.²⁰ Larger arrays have been installed on our two SSRL beam lines.²¹ In general, molybdenum (1500Å), boron (3000Å), and aluminum (1500Å) have been useful for the lower energy ranges (40-500 eV) whereas copper (2300Å) and magnesium (20,000Å) have been useful for the higher energy range (500-1300 eV).

Figure 6. Data from Henke, et al.¹⁹ showing the useful ranges of several prefilters used for our photoabsorption experiments. The form factor, f_2 , is proportional to the absorption cross section times the photon energy.

6. SAMPLE CONSIDERATIONS

Sample requirements for accurate absolute sub-keV cross section measurements have been described previously in these proceedings.¹ Survivability is a major consideration for these ultra-thin (1000-25,000Å), free-standing samples. Low yields in preparation and breakage during transportation and installation are common despite our use of the most delicate handling procedures. Our sample design and fabrication method, based on sputtered multilayer technology,² has been described elsewhere in these proceedings.⁴ Our samples feature 500-1000Å carbon outer layers and thinner, inner carbon layers alternating with randomized 20-1000Å inner metallic layers to improve sample mechanical strength and to greatly inhibit surface oxygen contamination. Storage in dry argon is routinely used. The use of alternating carbon and metallic layers is also intended to improve the uniformity of these samples, to reduce porosity, and hence to reduce pin hole formation.³ Uniformity is also enhanced by using a good deposition geometry and by depositing materials on smooth glass slides with suitable releasing agents. This is particularly important for SR measurements, since SR beams tend to be somewhat less uniform at the target than typical laboratory sources. It is important to insure that the measured region is a good representation of the total sample that is weighed.

Thorough sample characterization is required for good absolute measurements. It is essential to measure the mass-per-unit area of each sample with the most accurate microbalance available. As described in these proceedings,⁵ additional characterization measurements using Rutherford back scattering (RBS) and particle induced x-ray emission (PIXE) yield vital information about both the target element and the included carbon layers. The mass-per-unit area values of the separate components of a two (or multi-element) target determined from these ion bombardment methods are totaled and compared with the corresponding values measured with a microbalance. Typical results have shown agreement of 5% or better for the metallic component derived using the separate measurement techniques.⁵ Overall uncertainties for absolute cross sections derived from such characterization measurements were less than 10%. In addition, RBS and PIXE results showing the occasional presence and distribution of undesirable impurities, particularly oxygen, have led to improvements in the deposition environment and hence to the fabrication of purer targets.

The use of carbon overlayers requires the comparison of sample transmission data with that of a second sample containing carbon only, the second sample being fabricated during the sputtering operation. Moderate corrections for RBS-measured differences in carbon content are made routinely. These differences are attributed to slight variations in sticking rates between carbon-only layers and carbon-metal layers and small differences in the deposition geometry. We contend that corrections for carbon yield more accurate results than the results obtained with *in vacuo* deposition techniques used to minimize oxygen contamination. These *in situ* techniques do not allow full characterization of the target and tend to produce significant errors in the mass-per-unit-area determination.

7. DISCUSSION OF PRELIMINARY RESULTS

In this section examples are presented to illustrate important considerations for analyzing SR transmission data in order to obtain reliable absolute sub-keV x-ray photoabsorption cross sections. In particular, we emphasize the need for validating our measurements based on the self consistency achieved by using more than one monochromator and more than one prefilter. Further refinement and interpretation of these data are in progress.

Figure 7 shows experimental results for Be and U photoabsorption cross sections obtained using various prefilters on the recently dedicated SSRL Beam Line VIII TGM. These results show good monochromator resolution and scanning repeatability required to register the rapidly varying structure near thresholds. [The measured TGM resolution is better than 0.25 eV at 100 eV.] A necessary but not sufficient condition for valid photoabsorption

Figure 7 Preliminary photoabsorption cross section results obtained using different prefilters on the SSRL Beam Line VIII TGM for (a) beryllium and (b) uranium. The nominal operating range of this grating is indicated.

cross section results is good agreement between measurements when prefilters are used to suitably modify the monochromator output spectrum. In each case, results agree at intermediate energies as shown, but disagree at the low and high energy ends outside the grating's nominal operating range. Since the grating's first-order diffraction efficiency drops significantly with decreasing energy below 100 eV, the apparent rise in each sub-100 eV cross section is due to the presence of second-order radiation. The disagreement between data at low energies is due mainly to prefilter induced differences in first and second order contributions to the TGM output. The molybdenum prefilter, for example, is more effective at transmitting x rays in the 80-240 eV range while preferentially absorbing photons below 80 eV and above 240 eV. A contributing effect is that the TGM beam line mirror roll off is much less effective in suppressing second and higher order backgrounds for primary energies below 100 eV than for those above 100 eV. For the Mo filtered results in each case, the observed peak centered near 60 eV is thus due nearly entirely to second-order photons at twice the plotted x-ray energy. This peak closely resembles the peaked distribution in the central part of the monochromator range scaled by a factor of two in energy. Beryllium results above 240 eV are also in moderate disagreement. The disagreement in uranium cross section data as a function of prefilter is not as significant. The gradual rise in output is due to increased zero-order scattering contributions. These contributions are in a broad wavelength distribution where the average Be cross sections are higher than those corresponding to the first-order photons.

In Fig. 8 we compare preliminary TGM uranium and carbon results with a composite of the best results obtained at the NSLS U14A beam line using two different PGM ranges. The PGM data consists of Range 3 data joined to Range 2 data at 140 eV. A 3 eV shift in energy brings both sets of uranium data in Fig. 8a into much better agreement in the region of the main peak. This supplies additional motivation for a review of energy calibrations in both sets of measurements. There is reasonable agreement in the central uranium peak where the TGM performance is expected to be good and disagreement in both wings where the rise in TGM results implies data of questionable validity. The rise in the U cross section at 140 eV suggested by the TGM data is in the region of overlap between the PGM ranges where the U14A data is not quite as reliable. Efforts to resolve this slight discrepancy and the disagreement below the main peak are now in progress.

Figure 8 Comparisons of composite photoabsorption cross section results obtained using the NSLS U14A PGM with preliminary results obtained using a Mo prefilter on the SSRL TGM. Measurements are for (a) uranium (b) carbon.

Figure 8b shows TGM results obtained for a representative carbon sample using a Mo prefilter compared to a composite of NSLS PGM carbon results. There is a seven percent offset of the PGM results at 140 eV where Range 3 and Range 2 data are joined. Based on the shape of these two curves, the TGM and PGM data agree very well relatively between 90 and 190 eV except at the offset point. Nearly complete relative agreement is obtained if we assume a modest stray light correction and hence a reduction in the PGM Range 3 data at the upper end of its range near 140 eV. The two curves diverge rapidly in shape below 90 eV and above 190 eV again confirming the useful operating range of the TGM's grating. The roughly 18% disagreement in absolute results suggests sample-related problems associated with determining the weight-per-unit area for our thinnest (1000Å) carbon samples. The uncertainty in each thin carbon sample (our worst case) is about 10%. Analysis of other carbon data is underway to resolve this discrepancy.

8. CONCLUDING REMARKS

Synchrotron radiation sources are valuable in providing continuous coverage of the desired wavelength range with high flux and adequate to very good resolution. In this paper we have discussed in some detail the problems and pitfalls of making absolute photoabsorption cross section measurements in transmission using these SR sources along with our approach to overcoming these difficulties. We have stressed that, for reliable results, it is very important to get data at more than one SR beam line, for several grating ranges, and with several samples of varying thicknesses. We have shown that both relative and absolute cross section measurements can be affected by SR source spectral contaminations. To counteract this difficulty, agreement observed between data recorded with and without prefilters provides a useful test of validity. Our future plans include improving detector resolution by means of additional wavelength dispersion. We believe this will minimize the effects of spectral contaminations. We are considering a transmission grating arrangement and a multilayer-grating spectrograph for this application.²²

In addition, we recognize the need for liberal use of sum rule constraints to further test the reliability of our measurements. Furthermore, we need better models for the photoabsorption and photoionization processes to interpret the implications of our measurements particularly near inner-shell thresholds.

Despite conventional wisdom, determining absolute photoabsorption cross sections with reasonable accuracy from SR transmission measurements is not an easy task. To verify this point one need only scrutinize the significant discrepancies between various experimental determinations over the entire periodic system as presented in the latest data compilation.²³ A careful study of this compilation convinces us that the previous experimental determinations of the photoabsorption cross sections, particularly those below 1.5 keV, must be reassigned in the light of the difficulties enumerated above or, if necessary, remeasured.²⁴ That is the goal of our program.

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