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Chapter 3.40

Special considerations for insertion-device beamlines

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Some special considerations that are needed for X-ray absorption fine-structure measurements at insertion devices are introduced. These include radiation damage, beam stabilization during energy scans and effects due to beam coherence.

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Related chapters

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1. Introduction

X-ray absorption fine-structure (XAFS) measurements using insertion devices (IDs), especially undulator sources, have some unique challenges compared with the more gentle bending-magnet sources. The high intensity exacerbates problems with radiation damage. This can become even more critical if, as is often the case, the high-brilliance ID source is focused to a small spot. In addition, a small focus will enhance issues with sample non-uniformity. For undulator sources the narrow spectrum also needs special consideration. Either the undulator gap needs to be changed in concert with the monochromator energy or the undulator gap needs to be tapered to broaden its spectral width. Finally, the increased coherence of a high-brilliance source can become problematic during energy scans. This chapter will look at how these issues can affect the quality of XAFS measurements.

2. High intensity and radiation damage

Insertion-device beamlines will have high intensity. A focused wiggler line or an unfocused undulator beam can have an intensity of the order of 10^{13} photons s^{-1} in a millimetre-sized beam. The flux density on microfocused lines can be even higher. It is well known that high-intensity undulator beams can damage room-temperature protein samples in less than a second (Ascone *et al.*, 2003; Davis *et al.*, 2012). This often shows up as photoreduction with an accompanying edge shift. For this reason, biological samples are often run at cryogenic temperatures. Cryogenic temperatures extend the lifetime of samples, but radiation damage will still occur and needs to be monitored by running repeated short scans or by some other form of spectroscopy. Similar to proteins, wet samples will often show radiation damage. Even for an unfocused undulator beam, bubbles can be observed in aqueous solutions on the minute timescale. These are visual indication of interaction of the beam with the water to form various radical species and H_2 gas (George *et al.*, 2012). This can be mitigated by freezing the solutions or using a flow system to maintain fresh sample in the beam.

While samples containing water appear to be especially sensitive to the beam, radiation damage can be observed in a variety of samples. When using high-intensity insertion devices, it is always good practice to monitor for beam damage and to minimize the exposure time. Cryogenic temperatures will help, but cannot be relied on to eliminate damage.

In addition to radiation damage the high flux of ID sources can lead to nonlinearities in detectors, even for the commonly used ion chambers (Calvin & Nariyama, 2024).

3. Microbeams and sample uniformity

With their high brilliance, ID beamlines often provide microbeams. These offer special challenges for measuring XAFS. Few samples are perfectly uniform on the micrometre scale, and the measurements rely on high levels of beam and sample stability. This is emphasized in extended XAFS (EXAFS) measurements, where it is necessary to measure absorption changes of the order of 10^{-3} or better. If a 1 µm beam is incident on a 1 µm size particle, relative motions on the nanometre scale can cause changes in the signal similar to the EXAFS at high k. Such stability is challenging when scanning over the wide energy ranges needed for EXAFS.

Microbeams can be similar in size to the size of individual crystalline grains in a sample. The sample then appears to the beam as a collection of single crystals, and Bragg reflection interference can be an issue. As the energy is scanned there can be specific energies where the Bragg reflection condition is satisfied for a grain. These will show up as unusual bumps or dips in the data. When large, they are obvious. However, smaller distortions can be mistaken for a real signal. Since they depend strongly on the orientation of the grain, moving the beam to a different part of the sample is a good way to identify any suspected distortions. This same issue can be observed on a larger scale if the grain size of the sample is large. For example, frozen solution samples can have large grains from the freezing process. Bragg effects can then be observed with millimetre-size beams.

4. Energy scanning with an undulator

Wigglers have a smooth spectrum like a bending magnet. Undulators, however, have a sharply peaked spectrum that is much narrower than the typical XAFS energy range. For maximum signal, the undulator energy needs to be scanned in synchrony with the monochromator. Since undulator structures need to deal with strong magnet forces, energy changes may limit the scanning speed. Modern quick-scanning monochromators can change energy much faster than most undulators. For faster data collection, the undulator can be tapered to broaden its energy range. Tapering can have two deleterious effects. The brilliance is reduced, and the beam shape will be distorted and have an energy dependence. Reduced brilliance may be acceptable if the signals are strong enough. When the energy is scanned with a tapered undulator the spatial distribution of the flux will vary. For an unfocused undulator beam this will cause problems if the sample is not uniform. This problem can be minimized if the undulator beam is focused, as all of the energies are then brought back to a common focus.

In the future, superconducting undulators may improve the energy scanning speed. In their case the energy can be scanned by changing the current, which is potentially much easier than a mechanical scan.

5. Beam coherence

Undulator beams have a significant coherent fraction. As more rings convert to advanced multibend achromat lattices, the coherence of the beams will increase. While increased coherence is beneficial for many experiments, it can be detrimental for spectroscopy. Coherence makes the beam more sensitive to imperfections in windows and optics (Espeso *et al.*, 1998). The phase change at these imperfections can cause interference fringes. Similar fringes can appear at the edges of slits or other apertures. Since the interference is wavelength-dependent, these can cause energy-dependent changes in the beam profile even for a very stable beamline. If the sample is not perfectly homogeneous then these changes can show up in the data. Similarly, an inhomogeneous sample can generate its own interference with a partially coherent beam, modifying the intensity profile within the sample and ultimately changing the absorption of the beam. Again, these changes in absorption will be energy-dependent. With current ID sources these effects are likely to be small and for XANES experiments they can usually be ignored. For high-quality EXAFS measurements to high k where the signal changes are small they can become important, especially at future more coherent sources.

6. Summary

This chapter has discussed a number of issues that can complicate measurements with ID sources. They illustrate the advantage of using less intense sources such as bending magnets when high beam brilliance is not needed. For most transmission XAFS measurements it will be easier to obtain high-quality data using a less intense source. Measurements on an ID source are best reserved for those cases where the higher intensity and brilliance is essential.

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