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APPLICATIONS OF SYNCHROTRON RADIATION

Arthur Bienenstock*

Abstract

Following a brief review of its properties, discussions are presented of the application of synchrotron radiation to: (a) the determination of atomic arrangements using extended x-ray absorption fine structure (EXAFS); (b) the determination of atomic arrangements using x-ray anomalous scattering; (c) dynamic small angle x-ray scattering; (d) x-ray lithography and microscopy; (e) the determination of surface electronic states using vacuum ultraviolet radiation; (f) time resolved fluorescence from protein molecules.

I. Introduction

One of the most exciting by-products of high energy physics research has been the development of synchrotron radiation utilization by chemists, physicists, biologists, geologists and even electronics engineers. In this paper, I shall try to present an overview of those features of synchrotron radiation which make it valuable for these experimentalists, the types of experiments which synchrotron radiation makes possible and some idea of the impact of these experiments on the fields themselves. Since this is a brief overview, both the depth of description and the number of types of experiments described will be limited. Those desiring to delve deeper should see the more extended review article by Winick and Bienenstock[1] and the references contained therein.

II. Properties of Synchrotron Radiation

For the research to be described below, the most important feature of synchrotron radiation is its continuous, high intensity spectrum. The intensity rises quite slowly and smoothly with increasing photon energy until just below a critical energy given by $\varepsilon_c = 2.218E^3/R$, where ε_c is in KeV, the stored electron energy, E, is in GeV and the ring radius, R, is in meters. Usable photons are obtained for photon energies up to four or five times ε_c. At the highest values of E obtained in SPEAR, 4 to 4.5 GeV, ε_c ranges from 11 to 15.7 KeV. Hence, the usable photon energy range extends through the K-shell absorption edges of the elements through about atomic number 70, and includes the x-ray wavelengths normally used in x-ray diffraction. For photon energies up to ε_c, SPEAR produces more than 10^{12} photons per sec-mA-mrad in a 10% bandwidth.

This spectrum is in marked contrast to that produced by an x-ray tube, which consists of a small number of discrete lines characteristic of the anode above a weak continuum. Consequently, for many experiments in which the wavelength dependence of a phenomenon is being examined, or in which a broad spectral range is utilized or where a special wavelength which is not any anode characteristic wavelength must be utilized, synchrotron radiation provides 10^4 to 10^6 times the intensity produced by an x-ray tube.

For some experiments, the fact that the radiation is 100% linearly polarized with the electric vector in the plane of the electron orbit is of importance.

In addition, the radiation is extremely highly collimated with a vertical opening angle of the order of γ^{-1}, where $\gamma = E/mc^2$ and mc^2 is the electron rest mast. At 2.5 GeV, the opening angle is about one-half a milliradian.

Other experiments take advantage of the time structure. At SPEAR, for example, typical pulse lengths are of the order 0.2-0.4 nsec with pulse intervals of 780 nsec.

Finally, the storage ring offers a natural high vacuum environment which is extremely useful for surface studies.

III. Applications of Synchrotron Radiation

3.1 Extended X-ray Absorption Fine Structure.

The development of extended x-ray absorption fine structure (EXAFS) analysis as a means of determining the local coordination environment of individual atomic species in complex, polyatomic materials is one of the most important results of the availability of synchrotron radiation in the x-ray regime. Here, by coordination we mean the number and type of atoms surrounding a specific atomic species and the interatomic distances. This type of information is critical for modern attempts to understand at an atomic level the physical properties of liquids and solids.

EXAFS is fine structure on the x-ray absorption coefficient, μ, that appears for photon energies in the range of approximately 50-1500 eV above an absorption edge. Its origin can be understood through consideration of Eq. (1).

$$\mu(k) \propto |\langle f|H'|i\rangle|^2 \qquad (1)$$

Here, H' represents the electron-photon interaction and k is an effective free electron wave number for the photoejected electron given by

$$\hbar^2k^2/2m = E_{photon} - E_K \qquad (2)$$

where E_K is K-absorption edge of the element being studied.

The initial state, denoted i, is a K-shell state which is highly localized around a specific atomic nucleus. The final state, denoted f, is represented in Eq. (3) as an outgoing spherical wave plus waves which are back-scattered by the surrounding atoms.

$$\psi_f \propto \exp(ikr) + \text{back-scattered waves.} \qquad (3)$$

Hence, we can view Eq. (1) as indicating that the absorption coefficient samples the final state in the region of the nucleus of the absorbing atom. As the photon energy increases, so does k so that there are alternations between constructive and destructive interference between the outgoing spherical wave and the back scattered wave, as sampled at the original nucleus. It is this interference which gives rise to the fine structure.

The EXAFS, χ(k), is defined by the relation

$$\chi(k) \equiv \{\mu(k)-\mu_o(k)\}/\mu_o(k) \qquad (4)$$

Here, μ(k) is the experimentally absorbed absorption coefficient, while $\mu_o(k)$ is the smooth, monotonically decreasing portion of it. For K-edges, the derivations of Sayers et. al.[2] as well as those of Ashley and Doniarh[3] have led to a semi-empirical equation for the χ(k) in terms of the atoms coordinating the specific species whose edge is being studied. It is

$$\mu(k) = - \Sigma_j(N_j/kR_j^2)|f_j(k)|\exp(-2k^2\langle u_j^2\rangle)$$
$$\times \exp\{-R_j/\lambda(k)\} \times \sin\{2kR_j+\alpha_j(k)\} \qquad (5)$$

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*Stanford Synchrotron Radiation Laboratory, Stanford University, Stanford CA 94305.

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Here, the summation is over coordination shells and atomic species within a shell, all denoted by the index, j. R_j is the average separation of atom j from the absorbing atom; u_j^2 is the mean square deviation of that distance. The photoelectron back scattering amplitude for the neighbors is $f_j(k)$, N_j is the number of atoms in the shell at average distance R_j, $\lambda(k)$ is an energy dependent mean free path, and $\alpha_j(k)$ is a phase shift.

Eq. (5) implies that it is possible to determine the N_j, R_j, and u_j^2 for each absorbing species in a complex, polyatomic material. These parameters constitute an almost complete description of the coordination of each species, and provide more information than is obtainable by any technique other than x-ray diffraction analysis of crystalline materials. The great strength of the technique is that each atom in such a complex material has its own absorption edge energy. Consequently, the coordination environment of each atomic species may be studied independently. No other structural technique has this general capability.

Although EXAFS has been observed for nearly half a century, its importance as a structural tool became apparent with the pioneering work of Lytle[4,5] and Sayers et. al.[6,2] in the mid 1960's and early 1970's. The availability of synchrotron radiation in the x-ray region during the past few years has led to dramatic progress in EXAFS data acquisition and analysis.

From the experimental point of view, the progress has been vastly accelerated because synchrotron radiation provides high intensity over a broad spectral range and thus is well suited for studies of the absorption coefficient as a function of photon energy over an extended energy range.

More detailed descriptions of EXAFS and its utilization are contained in the review article by Winick and Bienenstock[1]. Of particular interest is its utilization in a variety of molecules of biological interest. These uses may be characterized in the following manner.

Many biologically interesting molecules contain very small concentrations of metal atoms like Fe and Mo. An example is Fe in hemoglobin. It is believed by some that the biological activity of these molecules is related to changes in very local atomic arrangements around the metal atoms. With EXAFS, one has the capability of examining these local atomic arrangements even if the biological material cannot be crystallized. Hence, there have been a number of studies of such systems with the metals in, for example, different oxidation states. These have not tended to support the local distortion pictures described above.

Another interesting use of EXAFS is in the study of heterogeneous catalyst. Many catalysts consist of very small metallic particle clusters on substrates which are silica or alumina. Prior to the utilization of EXAFS, it was virtually impossible to determine the atomic arrangement within the metallic clusters because x-ray diffraction patterns were dominated by the substrate scattering. With EXAFS, one can look directly at the absorption edge of the metals in the metallic cluster and, therefore, examine the metal coordination directly.

As indicated above, the development of EXAFS as a structural tool came as a result of theoretical understanding which indicated that it could be used in that manner and the availability of synchrotron radiation - a high intensity source of radiation with a smooth intensity versus wavelength dependence. In the initial stages of experimentation, much effort was devoted to critical examination of the theory and utilization of the technique in a variety of systems. In recent years, the capabilities of multi-GeV storage rings to produce high intensity synchrotron radiation are being pushed to the limits so that EXAFS may be pushed to its limit. In particular, EXAFS analysis lends itself to determinations of the coordinations of extremely dilute species.

This coordination is not measured by measuring the absorption coefficient directly. Instead, another manifestation of Eq. (1) is used. Eq. (1) describes the probability of an electron being excited from an initial state to a final state. Any process whose amplitude is controlled by the same probability will also exhibit EXAFS. One example of this is the emission of K_α or K_β radiation which accompanies the electron-hole recombination process. The importance of this for examining the environments of very dilute species can be understood in the following way.

By tuning a monochromator to accept only the radiation emitted by the atomic species whose coordination is being studied, one may achieve orders of magnitude increases in the signal-to-noise ratio compared to a direct measurement of the absorption coefficient. These increases arise from the fact that a very dilute species makes a very small contribution to the total absorption coefficient even near its own absorption edge, since all the other elements in the sample will have some finite absorption in that photon energy region. The fluorescence radiation at the wavelength of the characteristic of the dilute species, on the other hand, is dominated by that from the dilute species. Hence, its EXAFS may be measured directly. Clearly, however, the fluorescent intensity will be relatively low. Using sophisticated monochromatization techniques, Hastings et al[7] have measured the EXAFS associated with iron atoms present in copper in concentrations of only 50 ppm and also in extremely dilute protein solutions.

The recombination processes which lead to x-ray fluorescence also lead to the emission of Auger electrons whose energies are characteristic of the particular absorbing species and which reach the detector only if emitted from atoms within a few angstroms of the surface. Hence, the photon energy dependence of Auger emission may be used to determine the EXAFS from the surface of a material and, therefore, to determine the coordinations of atoms on or near the surface. Compared to the LEED technique, surface EXAFS is easier to interpret, is more accurate and does not require that the surface atoms be in a periodic array. Using this approach, Citrin et al[8] have examined the coordination of iodine atoms absorbed on a single crystal phase of silver. In related work, Stöhr et al[9] as well as Bianconi and Bachrach[10] have measured surface EXAFS through the measurement of inelastically scattered photoelectrons as a function of photon energy.

Both of the preceding techniques, dilute atom EXAFS and surface EXAFS, place extreme demands on measurement capabilities. In particular, they demand extreme enhancement of specific signal to noise ratios. Such enhancements are accompanied by extremely low signal levels. Future developments of such techniques, which are of extreme importance in understanding the roles of impurities in a variety of physical processes and surface interactions in physical problems like catalysis or corrosion, will be enhanced markedly when sufficient time on the high energy storage rings is dedicated directly to the generation of synchrotron radiation. Still further extreme advances may be anticipated when undulators produce two to four order of magnitude increases in the x-ray flux density incident upon the samples.

3.2 The Anomalous Scattering of X-rays.

Most of us are familiar with the vast increases in understanding of basic biological phenomena which have been achieved through x-ray diffraction studies of atomic arrangements in proteins and other macromolecular systems. In fact, however, the crystal structures of only a small number of such molecules are known for two reasons. The first is that it has not been possible to crystallize many proteins. In such circumstances, EXAFS may provide the only mechanism for determining parts of the structure. In other cases, however, one faces the classical limitation of x-ray crystal structure analysis. That is, the intensities of x-ray diffraction peaks are directly linked to the Fourier transform of the electron density. With complete knowledge of this transform, one could obtain the electron density which is the most complete information obtainable from x-ray diffraction. In fact, however, the diffraction intensity yields only the amplitude of the Fourier transform and not its phase. While there are a variety of methods of overcoming this problem in simpler systems, it is difficult to deal with in the complex macromolecular biological systems. One method which has proven particularly effective is known as multiple isomorphous replacement. In this method, three different crystals of the material to be studied are grown. The crystals are assumed to be identical in atomic structure except that each contains a different heavy metal atom at a specific molecular site. Through the differences in intensity associated with these different metal atoms, one may determine the phases associated with the Fourier transforms and perform the inversion to obtain the electron density.

Unfortunately, often it has not been possible to grow three different such crystals. In addition, it is not always apparent that the atomic structures are identical in the three different crystals. As discussed below, the tunability of the x-ray wavelength made possible by synchrotron radiation makes possible the avoidance of this approach for many protein systems. The same tunability also makes possible more detailed understanding of atomic arrangements in metallic and glassy alloys.

The scattering of x-rays by an atom, j, is describable by the atomic scattering factor, $f_j(k)$. When the x-ray photon energy is far above any atomic absorption edge energy, the scattering factor is simply the Fourier transform of the atomic electron density measured relative to the center of the atom. Here, k represents the Fourier transform variable and the scattering vector.

When, however, the incident photon energy is close to atoms in absorption edge of an atom in the sample, the atomic scattering factor is given by

$$f_j = f_j^o + \Delta f_j' + i\Delta f_j'' \quad (6)$$

Here, f_j^o is the atomic scattering factor at high photon energies while $\Delta f_j'$ and $\Delta f_j''$ are correction terms which are real functions of both photon energy and scattering vector magnitude, k. $\Delta f'$ is large and negative for photon energies just below the edge energy while $\Delta f''$ becomes large and positive for photon energies just above the edge energy so that large changes in f_j can be achieved through variation of the x-ray wavelength near the absorption edge.

By varying the x-ray wavelength at which the diffraction studies are performed, one may make a single metal atom appear like three different metal atoms through appropriate choices of the x-ray wavelength. Consequently, all the changes in scattering which are achieved through the chemical method of multiple isomorphous replacement can be achieved through variation of the x-ray wavelength at which the diffraction experiment is performed. This approach is most effective if very large changes in the f_j are achieved through the wavelength variation. This, in turn, implies the ability to tune the wavelength very close to the particular absorption edge being utilized. This is only possible with the intense continuous synchrotron radiation. With x-ray tubes, the useable wavelengths are determined completely by useable anode materials and not by the sample being studied.

The wavelength shift method offers immense advantages over the method of chemical multiple isomorphous replacement. First of all, only one crystal need be grown. In addition, one knows with certainty that the experiment is being performed on a crystal with a unique atomic arrangement.

It has been employed by Phillips et al.[11] in the synchrotron radiation study of the protein rubredoxin. This paper contains details of the analysis as well as a large bibliography dealing with the theory of the use of anomalous scattering for this purpose.

The availability of synchrotron radiation has also made it possible to measure $\Delta f'$ and $\Delta f''$ very close to absorption edges. Measurements by Templeton et al[12] as well as Fukamachi et al[13] utilizing synchrotron radiation have shown that the magnitude of $\Delta f'$ for photons just below the edges of Cs and Cu are significantly larger than had been anticipated previously. This is particularly important because large shifts make the method utilizing anomalous scattering much more effective than would small shifts.

In a related approach, anomalous scattering is being used to learn more about atomic arrangements in binary (A-B) amorphous materials. Normally, x-ray diffraction from amorphous materials yields a weighted atom-atom pair distribution function. That is, one determines the probability of finding two atoms separated by distance R. From monotonic system, this pair distribution function yields information like the average number of near and next nearest neighbor atoms as well as the associated interatomic distances.

For binary systems, however, the information obtained is much more ambiguous. While one may obtain a well defined near neighbor peak in the atom-atom pair distribution function, it is not possible to determine a unique coordination number from the area of this peak because A-A, A-B and B-B atomic pairs may contribute to it.

As discussed extensively by Bienenstock[14] and references contained therein, it is possible to utilize anomalous scattering to obtain separate A-A, A-B and B-B pair distribution functions. This approach is presently being explored by Fuoss and co-workers at SSRL.

Finally, anomalous scattering offers the possibility of obtaining detailed information about the ordering of metal atoms in a binary metallic alloys which undergo order-disorder transformations. In such systems, the important x-ray diffraction information is proportional to $(f_A - f_B)^2$. Unfortunately, alloys which show order-disorder transitions usually contain atoms which are adjacent on the periodic table. Cu-Zn and Co-Fe are examples. Consequently, the difference of their scattering factors is extremely small and it is extremely difficult to obtain reliable information about the state of order through x-ray diffraction. This difference may be made extremely large by utilizing x-rays whose energy is just below the absorption edge of the lighter atom and reducing the magnitude of the smaller atomic scattering factor. This approach is being utilized by Sparks and co-workers at SSRL.

3.3 X-ray Small Angle Scattering.

Thus far, this paper has dealt with techniques which utilize, for the most part, the combination of high intensity and continuously varying intensity versus wavelength of synchrotron radiation. In small angle x-ray scattering and in x-ray lithography, one utilizes the high intensity coupled with the extreme collimation of the x-ray beam.

As indicated above, x-ray diffraction intensities are directly linked to the Fourier transform of the electron density. For small values of the scattering vector or transform variable, one is observing long wavelength fluctuations in the electron density. These may occur because the natural periodicities of the system are extremely long, such as in muscle fibers, or because one has two phases mixed together, such as in a protein in solution. Small angle x-ray scattering is a method commonly employed to study such systems. In general, when employed with an x-ray tube which provides a highly divergent x-ray beam, rather precise slit systems must be employed to select out a very narrow pencil of the emitted radiation. As a result, only a very small portion of the x-rays emitted by the tube can be utilized in the experiment and exposure times are extremely long.

The natural collimation of x-rays produced as synchrotron radiation makes it a natural for such studies since one can frequently use virtually all of the x-rays produced in the desirable wavelength region. As a result, many orders of magnitude decreases in exposure times have been obtained and an entirely new field, the study of dynamic phenomena utilizing small angle x-ray scattering has been initiated. For example, Kulipanov and Skrinskii[15] have described a small angle diffraction study of a frog muscle contraction cycle (approximately 64 msec) performed at Novosibirsk on the Vepp-3 storage ring. Similarly, Webb[16] has reported work performed at SSRL in which, by the same sort of signal averaging, ten msec x-ray diffraction patterns have been obtained from stimulated garfish olfactory nerve.

It is anticipated that these time dependent small angle scattering techniques will also be employed in Materials Science to observe, for example, the phase separation of amorphous and crystalline binary alloy as they are cooled from the melt. Such phase separation has been the subject of considerable theoretical work in recent years.

3.4 X-ray Lithography and Microscopy.

The extremely small divergence of the x-ray beam also makes synchrotron radiation ideal for x-ray lithography and microscopy. In both these techniques, the x-ray beam is transmitted through a sample onto photoresist. In lithography, the "sample" is a mask which is used to define an electrical circuit which is to be printed on a chip. In microscopy, the sample is a material in which the absorption variations which arise from thickness or composition variations, are used to obtain contrast. In both cases, the low divergence of the synchrotron radiation beam as compared to that from an x-ray tube provides for a great increase in resolution while the high intensities allow for shorter exposure times. For x-ray lithography, this high resolution enables replication of electric circuits consisting of extremely fine (0.1 micron) lines and patterns for very large scale integrated (VLSI) circuits.

In this context it should be noted that the extremely high intensities coupled with the extremely low divergences which are obtainable through the employment of an interference wiggler or undulator on a multi-GeV storage ring make possible consideration of an x-ray microscopy technique in which the sample would be scanned by an extremely fine x-ray beam and the absorption pattern reported electronically. Such a development awaits the appearance of an appropriate undulator which is presently being planned at SSRL.

3.5 Surface Electronic States Studied by Vacuum Ultraviolet Radiation.

Thus far, the examples chosen have been taken from techniques which employ x-rays, since the author is most familiar with these. The availability of tunable vacuum ultraviolet radiation has made possible, however, an entire new class of photoemission studies of electronic states at the surface of materials which has already provided a great deal of information about how molecules are absorbed on surfaces. In the simplest of these experiments, one has monochromatic radiation incident upon a sample and measures the energy distribution of the photoemitted electrons. This energy distribution peaks when the electron energy is equal to the photon energy minus some core binding energy, so that the latter may be determined. This core binding energy, in turn, provides information about the nature of the bonding of the photoemitting atom to its surrounding atoms.

The availability of tunable radiation makes this technique a relatively precise surface bonding study technique. The surface sensitivity arises from the fact that the mean free path of 100-200 eV photoejected electrons in the solid are of the order of 5 angstroms. Hence, one may study the effect of an absorbed layer on the atoms at the surface of a substrate by tuning the photon energy so that the electrons emitted from a specific core state of the substrate have energies of the order of 100 eV.

3.6 Time-resolved Fluorescence from Protein Molecules.

In the final example, let me turn to very recent studies of the dynamics of protein residues in various protein systems. These utilize the previously described time structure of the synchrotron radiation at SSRL, as well as the polarization of the synchrotron radiation light. In these experiments, a polarized light beam is passed through a protein system which contains a fluorescent residue. The absorption of the light is highly preferential. That is, absorption is large for those residues which have a specific orientation with respect to the electric vector of the radiation. The fluorescent radiation from these residues, in turn, is polarized in a well defined direction relative to the orientation of the residue. As time progresses, the polarization anisotropy of the fluorescent radiation decreases because the active residues rotate. Consequently, one may use the time dependence of this anisotropy to determine the rotational relaxation times of the fluorescent residues in specific protein systems. Using SPEAR as a source, the exciting pulses are of the order of 0.2 to 0.4 nsec and the spacing between pulses is 780 nsec. Consequently, one may examine dynamics on this time scale.

This approach has been utilized by Munro et al[17] to study the motion of tryptophan residues in a variety of proteins and to show that elementary steps in functionally significant conformation changes may take place in the subnanosecond time range. Sabersky and Munro[18] have shown that picosecond dynamics may be studied in this way using phase shift measurements.

IV Conclusion

In this paper, I have concentrated on a small number of fields which have originated or been changed markedly through the availability of synchrotron radiation. Another author might have chosen completely different examples, such as angle-resolved photoemission which yields detailed information about one-electron energy levels in solids or dynamic and high resolution x-ray topography, which shows imperfections and their motion in nearly perfect single crystals. The list continues to grow steadily as more scientists avail themselves of these unique radiation sources. What's more, we may anticipate whole new generations of experiments as undulators increase brightness by two to four orders of magnitude. For all of this, we who use the radiation are most indebted to the machine physicists for the accelerators and storage rings which produce it.

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