RESEARCH USING SYNCHROTRON RADIATION

Ednor M. Rowe
Physical Sciences Laboratory - University of Wisconsin
Stoughton, Wisconsin

Summary

In a period of less than ten years, the use of synchrotron radiation in the study of the optical and electronic properties of solids, liquids, and gases has developed from a laboratory curiosity into a widely exploited technique applicable to the needs of investigators working in physics, chemistry, and biology. In this report we discuss the history, present state and future development of this phenomenon in far ultraviolet and soft x-ray research.

Introduction

It has been pointed out recently in an article in Particle Accelerators[1] that there is still much interesting and even exciting physics to be done with low energy accelerators. We would like to report on the use of electron machines of low, medium, and high energy by that "legion of patient spectroscopists" that Professor Weiskopf[2] spoke about with such affection at the 1971 International Conference on High Energy Accelerators, to explore a hitherto inaccessible portion of the electromagnetic spectrum.

The success of these machines as research instruments in this context results from the useful application of a normally unwanted by-product of their operation. This is, of course, synchrotron radiation, long looked upon by the accelerator builder as yet another manifestation of the essential perversity of nature: the very process which is involved in almost every experiment performed with high energy electron machines, bremsstrahlung radiation, also limits our ability to raise the energy of cyclic electron accelerators indefinitely.

However, after the prediction,[3] observation,[4,5] and theoretical treatment[6] of synchrotron radiation, Fano, and others, suggested that the unique properties of this phenomenon could be of great aid in the study of the electronic and optical properties of solids, liquids and gases. The application of modern optical techniques to investigations in the ultraviolet beyond the LiF cutoff at 1050 Å is difficult because of the lack of continuum sources of adequate intensity. This is particularly true of the wavelength range below 500 Å. In addition, this portion of the spectrum is a region of strong absorption, thus this range is also called the vacuum ultraviolet. Utilization of such sources as did exist at the time was made difficult by the fact that most of them were relatively high pressure gaseous discharge devices. Therefore, it was reasonable that synchrotron radiation, by virtue of its continuum nature and easily calculable spectral distribution, represented an attractive possibility for exploring the vacuum ultraviolet. There was, in addition, the fact that the electron machines that produced it operated at reasonable pressures, thus solving or at least easing vacuum separation problems between source and experiment.

Subsequently, pioneering work by Tomboulian and Hartman[7], and by Madden and Codling[8] verified the predictions. As a result, over the past ten years, vigorous programs to study the interaction of electromagnetic radiation with matter in this region of the spectrum, first in atomic physics then in solid state physics and more recently in chemistry and biology and have been instituted at electron machines located in Paris, Tokyo, Frascati, Hamburg, and Daresbury. In this country research is presently centered about two machines: the 180 MeV electron synchrotron located at the National Bureau of Standards and the 240 MeV electron storage ring at the Physical Sciences Laboratory of the University of Wisconsin. Use of the electron-positron storage ring SPEAR and CEA (operating in the storage ring mode) for this work has been proposed and small pilot programs have been initiated at both machines. A list of synchrotron radiation facilities both presently operating and proposed is given in Table I. Interestingly, in 1968, only the first four facilities listed were in operation.

Characteristics of Synchrotron Radiation

While the effects of synchrotron radiation on electron synchrotrons and storage rings are well-known to accelerator builders, it is important to include a discussion of the optical and spectral properties of the radiation that make it attractive to the investigators.

Electrons traversing the magnetic field of a circular accelerator radiate energy. This is the effect that is to be exploited. In their own frame of reference, they emit in a characteristic Larmor radiation pattern, and for highly relativistic electrons, this pattern becomes strongly distorted in the forward direction as viewed by an observer in the laboratory frame. Thus, if one looks in the orbital plane, in a direction opposite to the electron's motion and tangent to the orbit, the electron will be seen as a bright point of light (single electrons circulating at 240 MeV in Tantalus I can easily be seen). Even though the fundamental orbit frequency of the electrons and many higher harmonics (Fourier components) are present in the radiation, because of the distribution of synchrotron and betatron motions of the electrons a continuum spectrum is observed at the shorter wavelengths as is shown in Fig. 1. At wavelengths near the peak of the spectrum the root mean square angle of the emission cone is given by

$$\langle \phi^2 \rangle^{1/2} \approx \gamma^{-1} \tag{1}$$

where $\gamma = E/mc^2$, the ratio of electron energy to rest mass energy. Furthermore, the radiation in the plane of the orbit is polarized with electric vector oriented parallel to this plane. At longer wavelengths, the half angle is given approximately by

$$\langle \phi^2 \rangle^{1/2} \approx \frac{1}{\gamma} \left(\frac{\lambda_c}{\lambda} \right)^{1/3} \tag{2}$$

where λ_c is a characteristic wavelength given by

$$\lambda_c = 4\pi\rho / 3\gamma^3 \tag{3}$$

with ρ the radius of curvature of the electron path.

Light from the electrons in orbit is unlike that from a point source, however, because of the narrow radiation cone of each electron. This natural vertical collimation of the radiation is often overlooked when comparisons between synchrotron radiation sources and conventional x-ray tubes are made.

The power radiated per second per unit wavelength by an electron of energy $\gamma\ mc^2$ with orbital radius R is

Table I. Existing Synchrotron Radiation Groups, October, 1972

Location	Designation	Machine Type	Energy (BeV)	Circulating Current (ma)	No. of Users
Gaithersburg, Md.	SURF	synch.	0.18	5	10
Stoughton, Wis.	Tantalus I	storage	0.25	10	30
Hamburg, Ger.	DESY	synch.	7.5	10	30
Tokyo, Japan	INS-SOR	synch.	1.3	50	100
Frascati, Italy		synch.	1.1	10	5
Daresbury, England	NINA	synch.	5	20	20
Moscow, Russia		synch.	.6	(10)	2
Orsay, France	ACCO	storage	0.7	50	20
Yerevan, Russia		synch.	6.5	(10)	2
Cambridge, Mass.	CEA	storage	3	15	2
Bonn, Ger.		synch.	2.3	10	5

Planned Synchrotron Facilities

Stanford, Calif.	SPEAR	storage	2	500	10
Hamburg, Ger.	DORIS	storage	3	300	50
Tokyo, Japan		storage	0.3	100	100
Orsay, France	D.C.I.	storage	2.2	250	20
Moscow, Russia		synch.-storage	1.3	(100)	---
Tomsk, Russia		synch.	1.36	(10)	---
Novosibirsk, Russia	VEPP-3	storage	2	(250)	---
Kharkov, Russia		storage	0.1	---	---
Stoughton, Wis.	Tantalus II	storage	1.76	100	Large
Gaithersburg, Md.	SURF-2	storage	.240	100	20

given by

$$\partial P/\partial\lambda = 3^{5/2}\, ce^2 \gamma^7\, G(y)/16\pi^2 R^3 \qquad (4)$$

where

$$G(y) = y^3 \int_y^\infty K_{5/3}(x). \qquad (5)$$

The function G(y) must be evaluated numerically. However, for wavelengths such that $y \ll 1$, G(y) is given approximately by[9]

$$G(y) \approx 2^{2/3}\, \Gamma\,(2/3) y^{7/3} \qquad (6)$$

while at wavelengths such that $y \gg 1$

$$G(y) \approx [\pi/2]^{1/2}\, y^{3/2}\, e^{-y} \qquad (7)$$

Here c and e have their usual meaning and $y = \lambda_c/\lambda$. The spectral function G(y) peaks at a wavelength $\lambda_p = 0.42\,\lambda_c$, reaching a value of ≈ 1.2. Instead of the power emitted, spectroscopists are more interested in the total photon flux per unit wavelength. This is given by

$$dI/d\lambda = (dP/d\lambda)\, N\, \lambda/hc \qquad (8)$$

where $N = 2\pi \overset{*}{R} J/ec$, $\overset{*}{R}$ is the average radius of the orbit and J is the average beam current density.

In general, synchrotron radiation is not coherent so that the radiated intensity just increases with the number of electrons in orbit. If, however, one could reach a very high beam density so that the distance between electrons becomes comparable with the main wavelengths of emission, coherent effects would appear. This can be seen in Eq. 4 which shows that the radiated power depends upon charge squared. In the extreme case, if n electrons are close enough together to behave as a single entity of total charge ne the power should increase as n^2.

Practical formulae for the characteristics discussed above are given in Table II. Curves showing the spectral characteristics of the radiation from a number of electron machines appear in Fig. 1.

No summary of the optical properties of synchrotron radiation would be complete without a discussion of source brightness. We will compare a representative machine, in this case Tantalus II, with a very nonrepresentative plasma discharge device.

At the design energy of 1.76 GeV, the energy loss per turn in Tantalus II will be 180 keV. Thus at 100 mA circulating, the electrons will radiate energy at the rate of 18 kW. The expected beam cross section is 0.1 by 0.5 cm. Taking $\langle\phi^2\rangle^{1/2} = 10^{-3}$ radian we have a source brightness B of

$$B = 1.8 \times 10^4 \text{ watts } (5 \times 10^{-2} \text{ cm} \times 2\pi \times 2 \times 10^{-3})^{-1}$$

$$\approx 3 \times 10^7 \text{ watts } (\text{cm}^2 \text{ steradian})^{-1}$$

For comparison let us consider a theta pinch discharge device with the following parameters. Repetition rate, 1 Hz; time duration of discharge, 10^{-5} seconds; power level during discharge; 10^8 watts, volume of discharge; 1 cm radius sphere. We will assume a plasma temperature of 1.5×10^7 °K which gives the peak of the black body power spectrum from the discharge the same wavelength as the peak of the synchrotron radiation power spectrum. The brightness in this case is 60 watts $(\text{cm}^2 \text{ steradian})^{-1}$.

Table II. "Practical" Formulae

$$\lambda_c\,(\text{Å}) = 5.59\ \frac{R(m)}{[E(GeV)]^3}$$

$$\frac{dI}{d\lambda}\left(\frac{\text{photons}}{\text{sec Å mA mrad}}\right) = 7.9 \times 10^{11}\ G(y)\ J\ (mA)\ \frac{[E(GeV)]^7}{[R(m)]^2}\quad \lambda\quad (\text{Å})$$

$$\frac{dI}{dE}\left(\frac{\text{photons}}{\text{sec eV mA mrad}}\right) = 5.56 \times 10^{7}\ G(y)\ J\ (mA)\ \frac{[E(GeV)]^7}{[R(m)]^2}\quad \lambda^3\quad (\text{Å})$$

for $>> \lambda_c$:

$$\frac{dI}{d\lambda}\left(\frac{\text{photons}}{\text{sec Å mA mrad}}\right) = 9.35 \times 10^{13}\ J\ (mA)\ \frac{[R(m)]^{1/3}}{[\lambda\,(\text{Å})]^{4/3}}\ .$$

That these sources are extremely bright can be appreciated from Fig. 2. Here, synchrotron radiation from 4 milliradians of the orbit of CEA operating at 2.5 GeV and 15 mA circulating has been brought to focus with a grazing incidence ellipsoidal mirror at a distance of twenty meters from the source and allowed to pass through a beryllium window. The streamer of light comes from the recombination of air molecules ionized by the radiation.

Some Typical Synchrotron Radiation Facilities

The radiation facility at DESY used by Haensel and his colleagues is shown schematically in Fig. 3. The synchrotron radiation from a single port is split three ways by the plane mirrors labeled M_1, M_2, and M_3 after traveling a distance of forty meters to the bunker. Radiation may be supplied to two of the three beam lines simultaneously. However, particle radiation levels from DESY near the beam lines are too high to allow the investigators access to their instruments when the beam shutters labeled BS-1 and BS-2, are open.

Extensive use of the natural collimation and polarization of the radiation discussed in the second section has been made in the design of the instrumentation used at this facility. For example, the monochromators, labeled W_1, W_2, etc., used for selecting specific wavelengths from the continuum have no entrance slits: the electron beam in DESY itself performs that function. In addition, in instruments designed for the shorter wavelengths, dispersion and reflection are done in the vertical plane in order to both preserve the polarization of the radiation and to improve the transmission efficiency of the instruments.

All instruments are constructed so as to transmit directly the primary synchrotron radiation beam when not in use. Thus, typically, several experiments are installed on a single beam line as is shown in the figure.

The object labeled CH-1 is a chopper that cuts off the photon beam at times during the acceleration cycle when the spectral distribution of the synchrotron radiation makes it unsuitable for the experimenters. This can occur, for example, at high electron energy when the peak of the spectrum is in the x-ray range and would cause either higher order problems or radiation damage to the instrumentation.

Approximately thirty investigators are working at the DESY facility and this group has been extremely productive.[10] However, this group functions parasitically to the main operation at DESY which is, of course, dedicated to high energy physics. Further, DESY is a pulsed source and this makes some experiments difficult. The photon beam change in intensity, spectral distribution, position and profile during each machine cycle. To make matters worse, beam currents change from cycle to cycle.

A synchrotron radiation facility of somewhat different character has grown up about Tantalus I, the 240 MeV electron storage ring at the Synchrotron Radiation Center of the Physical Sciences Laboratory of the University of Wisconsin.[11] A plan view of the experimental area at the Center is shown in Fig. 4. Here, the basic philosophy followed has been to put more beam lines on the machine rather than to put more experiments on the beam lines, though, of course, some beam lines are split so as to serve two users simultaneously. As the machine now stands there are ten beam ports.

The electron beam in a storage ring, such as Tantalus I, is characterized by great stability in intensity, position and profile. Thus, electron beams of rather low intensity are quite usable photon sources because of their superior duty cycle. At Tantalus I, because of the low circulating beam intensity and energy, particle radiation from the ring is not a hazard and investigators can work at their equipment during storage ring operation. Because experiments are generally mounted close to the ring at this facility extensive use of grazing incidence mirrors is made to focus the synchrotron radiation on the entrance slits of monochromators. In some cases, the radiation from as much as fifty milliradians of the electron orbit can be gathered. These mirrors are generally mounted in chambers set on top of sputter ion and titanium sublimation pumps so that the assembly fulfills a second function, that of providing differential pumping between the storage ring and the experimenters.

Tantalus I is operated solely as a synchrotron radiation source. Thus, the investigators working at the facility can exercise considerable control over the operation of the machine. Changes in beam intensity, position, and energy can be and regularly are made at the request of the users. There are now fourteen groups from all over the country using the facility.

Synchrotron Radiation Research at Present

The range of investigations being carried out at synchrotron radiation facilities both here and abroad is wide and varied, and a complete description of the experimental program being carried out at any one of them would be far beyond the scope of this report. Thus, we will limit ourselves to the description of several experiments, presently being carried out at the Synchrotron Radiation Center of the Physical Sciences Laboratory of the University of Wisconsin, that best illustrate the ways in which the investigators working there make use of the storage ring. However, the experiments chosen for discussion here are representative of the investigations that have been going on world-wide for the past several years.

The University of Illinois group has worked from the first at the study of the transmission properties of a wide variety of solids and gases in 60-250 Å range. Using a highly modified Hilger-Watts 1 meter spectrograph in the setup shown schematically in Fig. 5, they have obtained absorption-transmission spectra on such diverse materials as the alkali halides, nickel oxide, pure nickel crystalline and amorphous silicon, aluminum, silene, and sulfur hexafluoride. The resolution obtained in these measurements has been remarkable. For example, shifts in the energy levels of the chlorine core electrons of a few tenths of an eV at 100 eV in the alkali halides KCl and NaCl due to the chemical environment of the chlorine atoms were easily observed. In Fig. 6 we show the transmission spectrum of silene (SiH_4) obtained by them. The structure shown pushes the resolution of the instrument, measured to be .025 eV at 100 eV.

In general, intricate and varied spectra have been observed in these studies. The broad features of the spectra obtained can be explained in terms of matrix elements and oscillator strength sum rules. However, the details which appear, which are due to density of states effects, excitons and collective electron effects, are only now being identified.

The Illinois group was the first at the Synchrotron Radiation Center to make use of the techniques of high energy physics in their studies, namely particle counting (in this case photons) and digital recording of data on tape for later computer analysis. These techniques are now standard at the Center and elsewhere.

Members of the Space Astronomy Laboratory of the University of Wisconsin have pursued a program aimed at the calibration of photo detectors to be shown in satellites (OAO II and others). These measurements depend on the fact that the wavelength distribution of the synchrotron radiation is exactly calculable provided that one knows the electron energy, orbit radius and the number of electrons circulating. The first two quantities can be known to high precision but the last quantity is rather difficult to measure exactly at very low circulating beam currents. The Space Astronomy group, in collaboration with the Storage Ring Operations group, took the following novel approach to the problem. Using a filter to define a segment of the spectrum and a series of absorbers calibrated for this segment of the spectral range, they were able to keep the input to their photo detectors in approximately the same intensity range as the circulating beam in the storage ring was reduced from a few thousand electrons (their calibration intensity) to approximately 100 electrons. This intensity reduction was accomplished by lowering the accelerating voltage so as to increase the loss rate of the circulating electrons due to quantum fluctuations. When approximately 100 electrons circulating was reached, changes in detector signal due to individual electrons leaving the machine were easily seen. Thus, the exact number of electrons contributing to the detector output signal during calibration could be determined, assuming only detector linearity over a limited range.

The group from the University of Southern California, under the leadership of Professor Judge, is studying the extreme ultraviolet absorption cross sections of the atmospheric gases. The importance of these studies can hardly be overestimated in view of the fact that we live in that huge "heat engine" which is our atmosphere. We can never hope to understand the dynamics of the atmosphere without detailed knowledge of the processes by which energy from the sun is absorbed and transferred in it.

To illustrate the impact that access to a stable, continuum source has had on these studies, we show Fig. 7. The various experimental points shown represent the best data available up until 1972. It should be realized that each of the points represents days, weeks and, in some cases, months of careful measurement and remeasurement with line sources, some of which were quite weak. In contrast, the smooth curve represents data obtained by the USC group in one fifteen-minute scan using the storage ring as a source. The existence of the two Rydberg-like series that appear at 510 Å and 660 Å might have been inferred from the point by point data, but the detailed shape of these series would have been exceedingly difficult to deduce. The measurement of auto-ionization cross sections of the atmospheric gases which, curiously, are functions of pressure are also being undertaken by this group.

The accurate determination of the density of electronic states in metals and other solids is a problem almost as old as solid state physics. In the past, these measurements have been difficult to make, not only because of the lack of sources in the 100 to 1000 Å range, but also because of the problem of surface contamination of the sample: because of the extreme absorption of radiation in this wavelength range, the photo electrons to be analyzed can only come from depths equal to those of the first few crystal planes under the surface. Even with modern high vacuum techniques, the maintenance of sample cleanliness against back streaming gas from the monochromators and the windowless, high pressure discharge lamps commonly used for this work is still extremely difficult. The electron storage ring, which requires high vacuum for its operation, and is an intense continuum source, has proven to be a nearly ideal source for this work. In addition, advantage can be taken of the polarization of the photon beams to study anisotropy effects in the density of states.

The experimental method is rather simple and elegant. Photons of a specific energy are allowed to impinge on the sample and the energy distribution of the photoelectrons emitted is determined with an electron energy analyzer. The photon energy is then changed and a new distribution of photoelectron energies is determined. Taking photon energy increments of the order of one volt, data sufficient for the complete determination of the density of electronic states in a sample can be obtained in about four hours, a time period consistent with the surface contamination time at pressures of 10^{-11} torr.

Future Developments

Presently, the work being carried out at the various radiation facilities now in operation is confined to 50 Å and longer wavelength range. While much work has been done, far more questions have been raised than have been answered. Thus, this region of the spectrum will continue to be studied for some time. The higher intensities available in this wavelength range at the new high energy storage rings, assuming that higher order and radiation damage problems can be overcome, will allow second generation experiments. Here one thinks, for example, of modulation spectroscopy and derivative spectroscopy. As an example of the latter, we include Fig. 8. Here, the absorption spectrum of silicon and the first two derivatives of this spectrum are shown. With this technique, details hidden in the rather diffuse absorption spectrum can be enhanced. However, intensity is, as usual, a problem. In the example shown, the derivative curves were only obtained at the expense of considerable computer assisted smoothing of the raw data. High intensity photon beams of good duty cycle will make it possible to obtain these data far more directly.

The shorter wavelength range promises exciting developments. Up until the present time, of one wanted to explore the range between 1 Å and 10 Å, with any intensity, the only sources available were x-ray tubes with aluminum or magnesium anodes. But these are line sources with line widths of the order of an eV. Yet this is the energy range which has become tremendously interesting because of the development of ESCA (electron spectroscopic chemical analysis) and XPS (x-ray photoelectron spectroscopy) techniques. For this work the natural line widths of conventional x-ray sources mentioned are too great and monochromators must be used with an unavoidable reduction in the intensity. This, coupled with the fact that these sources are of low brightness to begin with, make it difficult to achieve adequate intensities for studies in this wavelength range. Work recently reported[12] from DESY indicates that orders of magnitude increase in photon flux can be obtained using crystals to monochromatize the synchrotron radiation from GeV range accelerators. This is a natural result of the high brightness characteristic of these sources.

Another area soon to be exploited at high energy electron machines is molecular biology. The availability of synchrotron radiation sources of enormous brightness used in conjunction with high efficiency monochromators, in effect "tunable" x-ray machines, is bound to have great impact on the spectroscopy of proteins and other complex organics. Work is now in progress at DESY and NINA to develop x-ray microscopes to study muscle cells in vivo, and preparations are being made to use the radiation from the electron synchrotron at Yerevan to study proteins.

The experience with Tantalus I, the first storage ring to be used for far ultraviolet and soft x-ray investigations, suggests strongly that in the future, this field of research will be dominated by those groups working at the new high energy, high current storage rings such as SPEAR, DORIS, DCI, and VEPP-3. In all probability, in the future, at least one electron storage ring, with energy in the GeV range, such as Tantalus II[13], will be constructed to be a dedicated light source. Investigators having access to such a machine will have an immense advantage over those working parasitically at high energy physics facilities.

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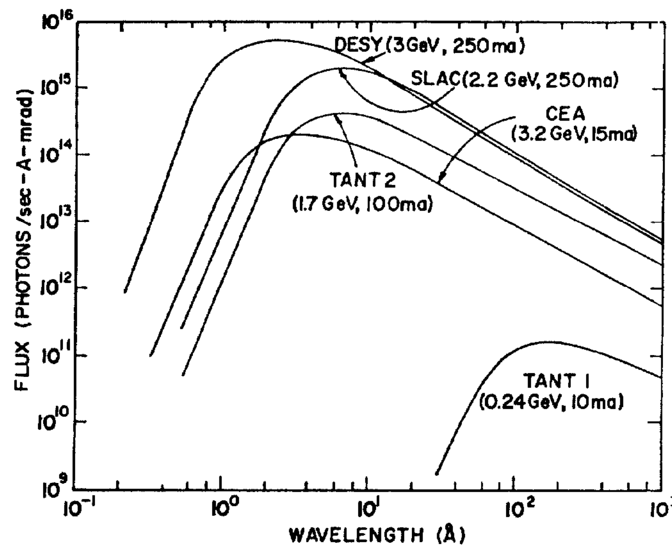

Fig. 1 Spectral properties of the synchrotron radiation from several electron machines.

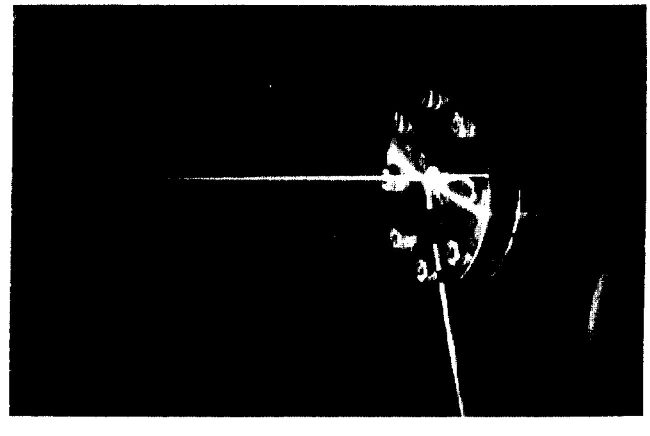

Fig. 2 Focused synchrotron radiation in air from the Cambridge Electron Accelerator. Courtesy Professor Paul Horowitz, Harvard University.

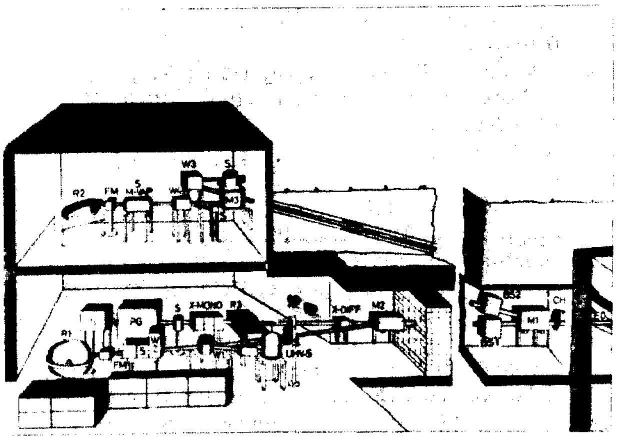

Fig. 3 The DESY Synchrotron Radiation Facility shown schematically. Courtesy Professor Ruprecht Haensel, Deutsches Elektronen-Synchrotron, Institute for Experimental Physics.

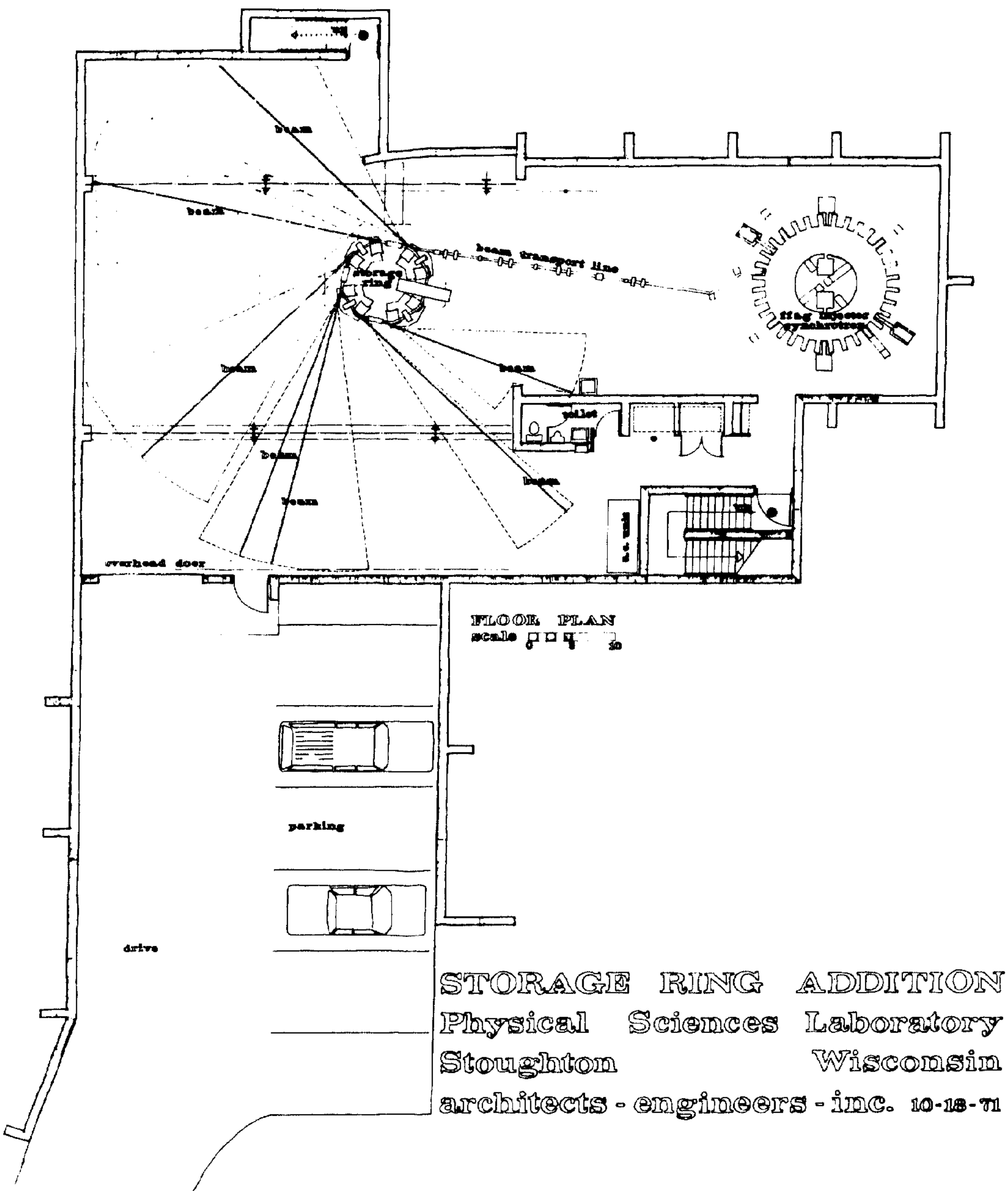

Fig. 4 Floor plan of the Synchrotron Radiation Center of the University of Wisconsin.

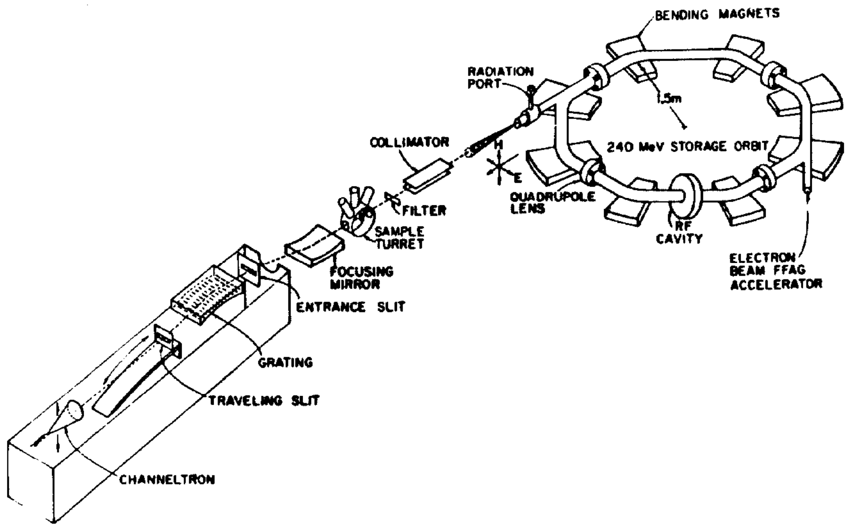

Fig. 5 Typical arrangement of apparatus for x-ray absorption spectroscopy. Courtesy Professor F. C. Brown, University of Illinois.

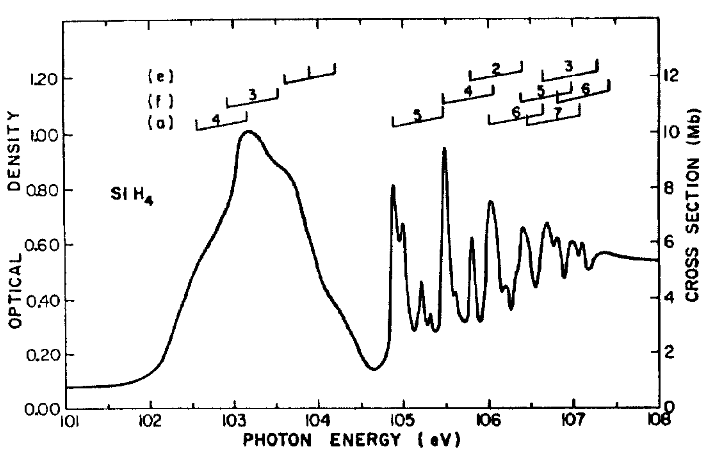

Fig. 6 Absorption spectrum of silene. Three separate Rydberg-like series can be identified. Courtesy Professor F. C. Brown.

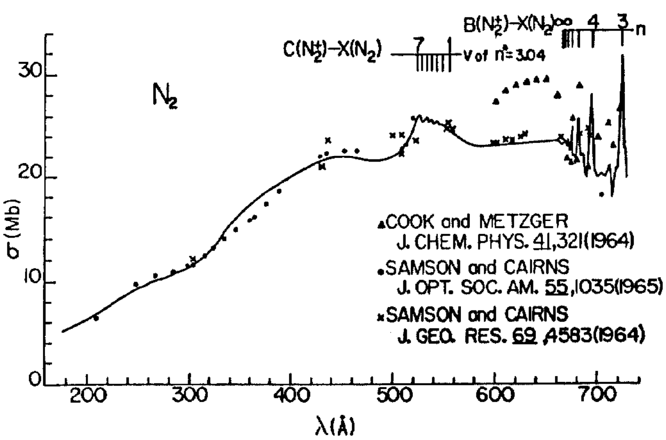

Fig. 7 Absorption spectrum of nitrogen. Smooth curve represents data gotten in one 15-minute scan at the Synchrotron Radiation Center of the University of Wisconsin. Courtesy Dr. Robert Carlson, University of Southern California.

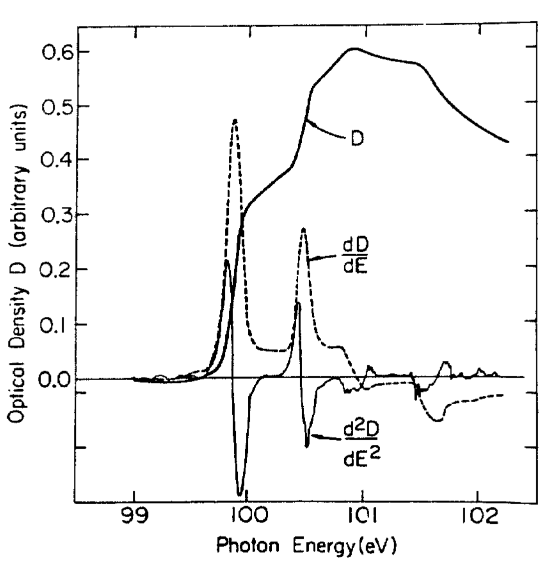

Fig. 8 Experimentally measured absorption spectrum of silicon. First and second derivative curves were calculated. As can be seen the derivative curves become progressively more noisy. However, features of the rather diffuse experimental curve that would normally be hidden are brought out clearly. Courtesy Professor F. C. Brown.