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Historical Survey and New Opportunities in the VUV

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The recent development of synchrotron radiation science is reviewed in relation to results reported at conferences on vacuum ultraviolet radiation physics, and prospects for research in the near future are presented. Emphasis is placed on spectroscopy in the vacuum ultraviolet and soft X-ray regions. Other topics such as applications to engineering, biophysics, materials science and surface science are briefly discussed.

Keywords: vacuum ultraviolet spectroscopy; soft X-rays; photoemission.

Introduction

Synchrotron radiation science is now entering a third-generation development phase, brought about by the emergence of new light sources of high brilliance. The proposals for building such sources were made more than a decade ago. By the time these proposals had been approved and the construction started, accelerator physics and technology had progressed much faster than had been anticipated. The situation was more or less similar in the case of beamline and measurement technology. This progress inevitably altered the shape of planned projects: 15 years ago second-generation sources offered new opportunities in surface science and biophysics, whereas today the use of synchrotron radiation in elucidating the electronic structure of the surface of a given material is quite common. When we proposed the construction of a new light source in Tokyo (not yet realized) we intended to carry out photoemission measurements at very low temperatures, fluorescence measurements in the VUV and soft X-ray (SX) region, spin- and angle-resolved photoemission measurements, coincidence measurements by simultaneous irradiation with laser and synchrotron light, photoabsorption measurements under extremum conditions, such as very high pressures and very high magnetic fields, and photoemission measurements with very high resolution. Some of the proposed experiments have already been carried out using our second-generation light source.

Recent research trends appear to place more weight on materials science than on the basic physical processes met in spectroscopic studies. This implies more interest in the ground-state properties of matter than in optically excited states or dynamic processes of electronic transitions, and more emphasis on statistical mechanics than on quantum mechanics. Such a trend has been accelerated by the advent of oxide superconductors with high critical temperatures. Another important aspect of contemporary synchrotron radiation science is that demand for brilliant light comes from a variety of research fields. This is illustrated below by a review of reports presented in a series of international conferences on vacuum ultraviolet radiation physics.

VUV conferences

Early conferences

This series of conferences started in 1962 at Los Angeles. The second conference was held at Gatlinburg in 1968 and after this the conferences have been held regularly every three years.

Spectroscopic investigations of gases and solids using synchrotron radiation were reported at the Gatlinburg conference. This opened a new era in VUV and SX spectroscopy. In the following conference, held in Tokyo in 1971, more results of photoabsorption experiments using synchrotron radiation were reported. All experiments were carried out with first-generation light sources, *i.e.* electron synchrotrons in parasitic use. Many reports on the developments of other laboratory light sources were also presented at this conference. Photoemission experiments, both XPS and UPS, can be found in the Conference Digest (Nakai, 1971), as can preliminary results of photoelectron diffraction. At this stage, no measurements of photoelectron energy-distribution curves (EDCs) using synchrotron radiation as the excitation light had appeared, although scientists were aware of the importance of such experiments. Photoelectric yield spectra measured with synchrotron radiation were reported. In this period, synchrotron radiation facilities were not widespread.

Fourth conference, Hamburg, 1974

The fourth conference was held at Hamburg in 1974 (Kunz, Koch & Haensel, 1974). The use of synchrotron radiation in the VUV–SX region had become more common. Second-generation light sources, electron storage rings in dedicated use for synchrotron radiation research, were about to emerge. They were converted from machines designed and constructed for elementary particle or nuclear physics or designed from the beginning as light sources; they were in the construction or commissioning stages. At the conference, the use of VUV spectroscopy for investigations of solar, stellar, galactic and comet matter was reported. Synchrotron radiation made detailed analysis of the Rydberg series possible. Deviations from quantum defect theory were examined and the Lu–Fano diagram was used as a useful technique. In solid-state spectroscopy, exciting arguments (Dow, 1974, 1975; Mahan, 1974, 1975; Ohtaka & Tanabe, 1990) were made concerning the validity of the Mahan–Noziere–DeDominicis theory on the threshold anomaly in innershell excitation spectra of nearly-free-electron metals. This phenomenon, the appearance of spike features at the thresholds of absorption or emission spectra, had been considered as a typical example of the many-electron interactions associated with optical excitations in solids, and many theorists had been concerned with this intriguing problem.

New aspects of VUV–SX spectroscopy at this conference were the introduction of the EXAFS technique and photoelectron spectroscopy using synchrotron radiation. Both of these later promoted research activity in VUV–SX spectroscopy enormously. At the same time, surface science research and biological materials science entered the field for the first time.

Fifth conference, Montpellier, 1977

The fifth conference, held at Montpellier in 1977 (Castex, Pinchaux & Pouey, 1978), appears to have been quite influential in that VUV–SX research emerged in the shape which we now see as a contemporary research field. The use of synchrotron radiation from an electron storage ring became common and the resulting spectroscopy occupied most of VUV–SX research. In atomic and molecular spectroscopy, correlation effects associated with innershell excitations were an important issue. The use of intense and stable light with a continuous spectrum was essential to obtain experimental data suitable for detailed quantitative discussions. Experimentally, angle-resolved photoemission measurements were carried out both for photoelectrons and photoions.

Various techniques using synchrotron radiation became established, namely, photoemission experiments on solid materials, the method of energy-band mapping, the elucidation of intrinsic surface states and the states of adsorbed molecules by angle-resolved photoemission. These methods opened the way to quantitative elucidation of the electronic structure of condensed matter. Synchrotron radiation also brought about new methods of photoemission research: constant-initial-state (CIS) and constant-final-state (CFS) spectroscopy and resonant photoemission. Later, resonant photoemission and related analyses of CIS and CFS spectra were found to be quite effective for clarifying the states of strongly correlated electron systems and the associated electronic transitions. The observation of the famous two-hole bound state in Ni was reported for the first time (Petroff, 1978; Guillot *et al.*, 1977). In surface science research, the method of photoelectron diffraction emerged as a tool in the VUV–SX region. Measurements of fluorescence, particularly lifetime measurements, were also reported.

EXAFS research appeared to be expanding continuously, as was biological research using synchrotron radiation. Apart from the applications of synchrotron radiation, the important issues of the conference were line sources from plasma and the search for VUV lasers. In spite of the growing importance of synchrotron radiation, there was still interest in lasers as an 'ideal' light source. A specific aspect at the meeting was the report on X-ray microlithography (Spiller, Feder & Topolian, 1978); synchrotron radiation had not yet been used there, although its importance was emphasized.

Sixth conference, Charlottesville, 1980

At the sixth conference held in Charlottesville in 1980 (Howard, 1980) the results were largely a continuation and extension of those of the Montpellier conference. However, the experiments were more sophisticated and refined. They included energy-band mapping and the elucidation of surface states by means of angle-resolved photoemission, studies of correlation effects in innershell excitations, high-resolution spectroscopy for the Rydberg series, measurements of the fragmentation of molecular ions, time-resolved measurements of fluorescence in solid rare gases, and, finally, soft X-ray emission measurements on very clean surfaces of metals.

In addition, there were new features. The characteristics and operation of VUV noble-gas excimer lasers and some of their applications were presented. Harmonic generation and frequency mixing to produce coherent VUV light was explained. Population inversion in a stationary recombining plasma was also reported. This program for laser-based work was quite appropriate. It was very important to know the state-of-the-art development of VUV–SX lasers that would replace inconvenient and expensive synchrotron radiation sources! It appeared to be a dream for a scientist working in the field of VUV–SX and X-ray research to have compact, conventional and laboratory-size tunable lasers for short-wavelength light. As an application of lasers, multiphoton spectroscopy was discussed in detail. The techniques of multiphoton ionization including those using supersonic molecular beams provided a field of spectroscopy complementary to VUV–SX spectroscopy. The accurate high-resolution data obtained presented detailed information on atomic or molecular excited states, although the energy range attainable was limited.

EXAFS techniques had been developing steadily. Photoelectric yield spectra were used instead of absorbance for materials that could not be made into thin films and thereby were unsuitable for absorption measurements. This enhanced the applicability of EXAFS methods to a considerable extent. Spin-resolved photoemission experiments on atoms and molecules were reported. Although this was a novel feature of VUV–SX spectroscopy, synchrotron radiation was not yet used. With respect to instrumentation, the possibility of an undulator as a light source was discussed. Beamline optics were attracting concern as an important technology in synchrotron radiation applications that needed development.

Seventh conference, Jerusalem, 1983

In the seventh conference held at Jerusalem in 1983 (Weinreb & Ron, 1983), spectroscopy using synchrotron radiation and studies closely related to this type of spectroscopy appeared to be a standard research field in VUV–SX spectroscopy. Work was also continuing on developing new VUV light sources. However, the major issues were spectrometers and optical components. This was because new facilities such as BESSY, super ACO and the Photon Factory had been built but experiments on new beamlines were either at the construction or commissioning stages. New instruments had also been installed in older facilities. By this time, the main area of spectroscopic research had become photoelectron spectroscopy. Thus, the design and performance of electron spectrometers were discussed in detail. A toroidal grating monochromator (TGM), and a sophisticated monochromator with an elliptical mirror focusing system, SX-700, were introduced and discussed. A TGM had been known to give an excellent performance in a beamline for photoemission experiments since the success of angle-resolved photoemission in the ACO ring reported in the Montpellier meeting.

Most of the scientific issues were developments of those presented at the preceding conference: ionization of atoms and molecules, dissociation, the electronic states of adsorbed molecules, analyses of intrinsic surface states, energy-band mapping, photo-stimulated desorption and multiphoton excitation were all topics that were discussed again. A new trend was an increased emphasis on correlation effects in condensed matter. These effects were investigated by analyzing the satellite spectra of photoemission lines. The results of spin- and angle-resolved photoemission experiments carried out on both gases and solids by synchrotron radiation excitation were reported. Magnetic circular dichroism (MCD) spectra were also presented. Fluorescence measurements on both organic and inorganic materials were also important, although the photon energy was limited to a comparatively low-value region. A novel feature of the Jerusalem conference was that synchrotron radiation microlithography aimed at industrial applications was reviewed. The corresponding experimental work was carried out by the IBM group (Grobman, 1983). This area of research was then later developed by Japanese industrial companies and BESSY as well as synchrotron radiation facilities in the USA.

Eighth conference, Lund, 1986

The eighth conference was held at Lund in 1986 (Nilsson & Nordgren, 1987). In this conference the main new trends were in the spectroscopy of condensed matter. Discussions became more like those of materials science than of pure solid-state physics. This indicated that experimental methods had been established to an appreciable extent, and that the basic physical processes involved in electronic transitions and the principles of formation of the related electronic structures, which together give rise to observed spectra, had been understood more quantitatively. In fact, this was not necessarily the case: novel materials showing exotic properties were later discussed; experimental techniques were further refined. The materials science trend appeared to be distinct in the photoelectron spectroscopy of semiconductor surfaces, metal–semiconductor interfaces, organic molecular solids, organometallic compounds, and localized electron systems such as compounds of transition metals, rare-earth metals and uranium. Reported experiments of photoelectron diffraction, soft X-ray absorption and EXAFS were also in this direction. In atomic and molecular spectroscopy, experiments and theoretical analyses were more detailed. Increasing amounts of advanced data were available from new facilities (BESSY and super ACO in LURE), making an important contribution to VUV–SX science.

In addition to synchrotron radiation science, studies of multiphoton ionization processes, spectroscopic investigations of cool stars, and spectroscopy associated with fusion research had progressed steadily. A new distinctive aspect was the great advance in data processing that was possible with advanced microcomputer technology. The investigation of microclusters was predicted to grow quickly. In molecular spectroscopy, experimental coincidence measurements such as PIPICO (photoion–photoion coincidence) and PEPICO (photoelectron–photoion coincidence) had shown themselves to be promising.

A state-of-the-art review was presented for inverse photoemission (Smith, 1987), destined to be an important technique complementary to VUV–SX spectroscopy. Regarding yield spectroscopy, the applications of Auger-yield spectra to XANES and of fluorescence-yield spectra to EXAFS were discussed. Time-resolved fluorescence spectroscopy was also evolving: the energy resolution was increased and a device to enhance time resolution was installed. In particular, fluorescence decay data on biological materials obtained with the MAX synchrotron suggested a new direction for molecular dynamics and molecular structure research. Spin- and angle-resolved photoemission spectroscopy was more refined for both gaseous and condensed matter samples.

An application that attracted interest was X-ray microscopy developed in the BESSY storage ring by the Göttingen group (Rudolph *et al.*, 1987). In the report concerning this, the beamline optics were presented and images of biological specimens with 500 times magnification were reported.

Ninth conference, Honolulu, 1989

The ninth conference held at Honolulu in 1989 (Shirley & Margaritondo, 1990) reflected important advances made since the preceding conference. Three new facilities, SRC at Stoughton with the Aladdin storage ring, NSLS at Brookhaven, and the MAX Laboratory at Lund, equipped with high-performance experimental stations, had come into routine operation. Undulator beamlines were operating in a few facilities and producing data of high quality. X-ray microscopy data using undulator radiation were presented (Kirz *et al.*, 1990). A report was also presented on the photochemical reactions of alkali halides induced by undulator radiation (Watanabe *et al.*, 1990). Soft X-ray fluorescence spectra obtained by undulator excitation were also reported (Nordgren & Wassdahl, 1990). In this report, fluorescence spectra caused by excitations with high-energy photons followed by recombination of valence electrons with innershell holes were made measurable with a well optimized optical system by irradiation with intense monochromatic light. Fluorescence measurements in the high-photon-energy region had been carried out before, but these were for yield spectroscopy applied to EXAFS experiments. Fluorescence spectroscopy in the innershell excitation region was believed to open up novel VUV–SX radiation physics. The design and optics of a new type of undulator were also discussed. Discussions on insertion devices were concerned with the realization of third-generation light sources.

A second distinctive aspect of the conference was the opening of the era of high-resolution spectroscopy. This was brought about by high-performance monochromators, such as the Dragon at NSLS and SSRL, and SX-700-II at BESSY. Since the resolving power was enhanced almost by an order of magnitude, the molecular spectra improved dramatically. In the case of SSRL, the monochromator was installed in a wiggler beamline. A third development was that there were reports on spatially resolved XANES spectra (Harp, Han & Tonner, 1990). The spectra were obtained by the combination of electron microscopy and photoelectric yield spectroscopy. This method was expected to expand the usefulness of XANES analyses enormously. A fourth new aspect at the meeting was photoelectron spectroscopy by means of double excitation with synchrotron radiation and laser light (Meyer *et al.*, 1990). This is a type of multiphoton process but the excitation is not a single coherent excitation with two photons; laser light is used as a pumping source. Such a coincident excitation opened up a way of analysing the excited states inaccessible by ordinary single-excitation processes.

Other topics discussed were advances in inverse photoemission, compact storage rings for microlithography, solid-state physics of newly discovered materials, circular dichroism, the spin polarization of photoelectrons, microclusters and metallic superlattices. In inverse photoemission, emitted-light spectra were also measured and these made the observation of resonant inverse photoemission possible. Oxide superconductors with high critical temperatures were newly discovered materials. Photoemission and inverse photoemission data on these materials presented a good deal of information about the electronic structures, which were crucially important for understanding the physical background of the properties of these materials. Experimentally, photoemission experiments at very low temperatures using single crystals were carried out for the first time. Similar research was performed on heavy fermion materials. The elucidation of electronic structures was one of the 'hot' topics in solid-state physics at the time. Photoemission spectroscopy by the use of synchrotron radiation was obviously quite a powerful method for investigations of the problem. In the remaining fields of VUV–SX physics and related science including atomic and molecular spectroscopy, surface and interface research, space and plasma physics, and multilayer optics, progress was also distinct.

Tenth conference, Paris, 1992

The tenth conference, the most recent, was held in Paris in 1992 (Wuilleumier, Petroff & Nenner, 1993). In this conference, dramatic progress in experimental techniques was not apparent. In this sense, the three years since the preceding conference seemed to have been spent in refining earlier investigations. The use of undulators had become more common. Monochromators and electron spectrometers with high resolving powers were employed in many facilities. The high flux of light from an insertion device compensated for the loss of signal intensities inevitable in high-resolution measurements. In the case of condensed matter, measurements at low temperatures were found to be crucial for obtaining highly resolved spectra. Advanced computer techniques altered the way in which experiments were controlled and the data obtained were processed. This enhanced the number of parameters describing spectra, and data were illustrated more or less in a stereographic manner. In photoemission measurements, spatially resolved measurements became more common. This technique was applied to observe magnetic domains (Stöhr, 1992). The excellent characteristics of synchrotron radiation, particularly the time structure and polarization, were fully used. In particular, circularly polarized light was conveniently used in observing spin-dependent phenomena.

New materials, C_{60} and its relatives, were investigated. More detailed investigations were carried out on high-T_c superconductors and the Kondo model of heavy fermions was discussed. Interesting solid-state research was found in a report on the observation of changes in EDCs associated with phase transitions (Maltere, Dardel, Grioni, Weidel & Baer, 1993; Takahashi, Suzuki, Kusonoki & Katayama-Yoshida, 1993; Onellion, 1993) such as the Mott transition and the Peierls transition, and with the occurrence of the BCS gap. In molecular spectroscopy, coincidence experiments like PIPICO, PEPICO and PEPIPICO (photoelectron–photoion–photoion coincidence) were the highlight of the conference (Morin, Lavollée & Simon, 1993). The investigations of clusters appeared to attract considerable interest. Fluorescence measurements became more important. Angle-resolved free-photoion spectra measured using undulator light were reported. Developments of insertion devices and optimized beam lines reported in the conference appeared to be aimed at third-generation facilities. Surface science had progressed steadily.

Summary of the VUV field to date

So far, this paper has given a brief overview of the research appearing in the past conferences on VUV radiation physics. This may be summarized as follows: vacuum ultraviolent radiation physics was changed when synchrotron radiation was introduced! The combination of synchrotron radiation and photoelectron spectroscopy opened up new fields. Second-generation light sources, electron storage rings, promoted the investigation of surfaces and interfaces where ultrahigh vacuum was necessary. The second phase of progress in research was initiated with the advent of high-resolution spectrometers, advanced computer technology and the discovery of novel materials.

Future prospects

Finally, we consider the prospects of VUV research in the near future. Microlithography and microengineering research for industrial applications will proceed and progress steadily, although there are some barriers to be overcome. As we have seen above, dramatic advances in basic science can occur when equipment leading to considerable refinements in experimental technique is developed or if novel materials with exotic properties are discovered. Concerning advanced devices, we expect rapid progress in the near future. This movement will be accelerated by the use of third-generation light sources. Resolving powers of the order of 10^4 will be commonplace, as will measurements on condensed matter at very low temperatures. In photoemission, many experiments will be made in the angle-, space- and spin-resolved mode, and the polarization of synchrotron radiation will be fully utilized. In atomic and molecular spectroscopy, multi-coincidence experiments such as PEPICO, PEPIPICO and PEPIPI...CO will be a major area of research. Double excitation with synchrotron radiation and laser light will also be applied to condensed matter studies.

It is unlikely that the pulse width will be reduced drastically in time-resolved experiments. The number of fluorescence experiments, particularly those related to innershell transitions, will be considerably increased. The recently developed method of virtually reducing lifetime broadening (Hämäläinen, Siddons, Hastings & Berman, 1991) will be widely adopted. The resonant scattering of high-energy photons is a second-order process and will provide new information. New experiments that are expected to be carried out in the near future are measurements under extreme conditions such as very high magnetic fields. In this way, the emergence of third-generation light sources will stimulate arguments concerning methodology. Thus, new opportunities allow novel scientific questions to be addressed and new methods to be devised.

In atomic physics, while interests in correlation effects and other higher-order processes will increase constantly and while elements capable of being handled easily and presenting this sort of information will be used further, new elements will also be exploited. For this exploitation, new techniques for producing atomic vapors will be developed. In molecular spectroscopy, experiments on adsorbed molecules will increase. Studies of free molecules in coincidence experiments will increase slowly, and activity will be concentrated on technical developments and arguments concerning the data obtained.

In condensed-matter physics, it is difficult to imagine what kind of new materials will emerge. This will occur quite accidentally. Other materials which we are able to obtain are those for studies of materials science. Artificial systems like superlattices and metallic overlayers will increase. The objects of surface and interface studies must be increased. The experimental data will be more detailed and displayed in at least two dimensions. In the area of bulk materials, the electronic structures of alloys should be explored more by using spectroscopic methods. The elucidation of the relation between various phase transitions, particularly first-order transitions and changes in the electronic structure around the critical points, is also important.

Finally, the number of investigations on organic materials and biological materials will be enhanced in the future. Experiments are not easy. Problems we shall meet are ultrahigh vacuum conditions and the damage of samples by radiation. If they are overcome, the area opening up to us is very promising.

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