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Surface Analysis: X-ray Photoelectron Spectroscopy, Auger Electron Spectroscopy, and Secondary Ion Mass Spectrometry

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The present review is on the subject of surface analysis and includes the fields of X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), and secondary ion mass spectrometry (SIMS) for the period of 1979–1981. This review will cover the literature abstracted in *Chemical Abstracts* between September 3, 1979, and November 2, 1981 (plus some important articles that have appeared in the latter part of 1981). The reviews of XPS, AES, and SIMS are written as separate sections for the reader's convenience.

XPS, AES, and SIMS are the most widely used techniques in surface analysis and are often used in combination. Although XPS (and UPS) and AES have been covered by earlier Fundamental Reviews in *Analytical Chemistry* (*1–5*), SIMS is only mentioned briefly in the Application Reviews on Surface Characterization (*6*). Considering the importance of surface analysis in today's technology, we thought that XPS, AES, and SIMS should be included in one review.

This review, although lengthy, is not an all-inclusive (2 year) bibliography of XPS, AES, or SIMS. We have tried to select the most important papers in each field that (in our opinion) will advance the "state of the art" of XPS, AES, and SIMS. We also decided to omit some topics such as the use of synchrotron radiation in XPS, for example, and to limit or ignore a large number of papers on applications in order to keep this

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review to a manageable size. Therefore, we apologize if we have overlooked some important paper(s). Researchers are also invited to send us reprints of papers that they believe should be included in future fundamental reviews on surface analysis.

The most difficult task in writing this review (other than the task of handling such a large number of papers) is trying to write coherent paragraphs while limiting the scope of the subject to only those papers published during the last 2 years. Therefore, we apologize if the paragraph structure seems "choppy", because it is.

X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

Introduction

X-ray photoelectron spectroscopy, XPS (also referred to as electron spectroscopy for chemical analysis, ESCA), has received wide acceptance in the scientific community. The highlight of the reporting period was the winning of part of the Nobel Prize in Physics by K. Siegbahn for his work in photoelectron spectroscopy. A large number of areas of research are actively using the technique, as can be seen by the many different journals cited in this report. The use of multiple techniques in a given study is increasing, and the combinations are too numerous to list.

Volume 4 of "Electron Spectroscopy: Theory and Technical Applications" (*A1*) appeared during the reporting period. Seah (*A2*) has reviewed the quantitative aspects and Roberts (*A3*) the surface chemistry uses of XPS. Much of the literature of the 1970s has been covered by Chaudhari and Cheng (*A4*). Briefer reviews on research on catalyst by Brinen (*A5*), catalyst and adhesion by Briggs (*A6*), and electrode surfaces by McIntyre et al. (*A7*) have appeared also. Electronic structure and bonding have been covered by Watson and Perlman (*A8*). Several other reviews will be listed in the individual sections.

In the next several sections various topics that affect XPS analysis will be discussed. The discussion will then turn to some specialized topics. Theoretical areas of research will conclude the XPS section of this review.

Factors Affecting XPS Analysis

Binding Energies. The measurement of binding energies and relative intensities is at the heart of most XPS experiments. From these measurements much information about the surface region and atomic environment is derived. Unfortunately, the reproducibility of such measurements between different laboratories has been shown to be poor. Powell et al. (*A9*) have conducted a round robin survey using Cu, Ag, and Ni with 38 different instruments manufactured by eight companies. Variations in binding energies of 2 eV and intensity ratios by a factor of 10 were reported; part of the differences were ascribed to poor instrument performance. More standardization in procedure is clearly needed in this regard.

Hazell et al. (*A10*) have prepared a chart of binding energies that would be encountered in normal XPS experiments. Such a chart is easier to use than the usual tabulated listings. Bakke et al. (*A11*) have listed the binding energies for approximately 800 different compounds. The determination of binding energies by the two X-ray wavelength method (Ag Lα,β and Al Kα) by Bird and Swift (*A12*) has led to changes for suggested binding energies of noble metals and Co compared to previous reports. Differences of almost 0.5 eV were found. Core level binding energies for Hf to Bi have been measured by Nyholm et al. (*A13*); differences with other workers in most cases were small.

Adventitious carbon, i.e., carbon from the background contamination, has often been used as a binding energy calibration. Jaegle et al. (*A14*) have found the peak position for this line can vary by about 1 eV after ion bombardment, depending on the time after sputtering and the substrate. Extra atomic relaxation of the overlayer was used to explain the observed variations. Lewis and Kelly (*A15*) have suggested a procedure to measure binding energies of insulators while using a monochromatic X-ray source and an electron flood gun. Their proposed model depends on a current balance that leads to the result that for insulators the surface potential should follow the electron flood gun voltage.

Wagner (*A16*) has investigated the use of a hot filament to reduce the effect of charging when a nonmonochromatic X-ray source is used with insulating samples. He noted that the observed binding energies are a function of the operation of the filament; he found also that mixing an insulator and conducting powder could not be used to eliminate charging.

Wagner (*A17*) has proposed that electron spectrometers should be calibrated via the use of the $2p_{3/2}$, Auger, and 3p lines from cleaned Cu. For insulating samples the adventitious C 1s line or the gold decoration method was recommended. The influence of surface atoms on observed binding energies has been investigated by Chadwick and Karolewski (*A18*). These workers concluded that materials chosen for standards should have a negligible surface atom core shift relative to the bulk, and for atoms in which this effect is large, the kinetic energy of the transition used should be large so as to minimize the surface effect. Thus, the use of the Cu $2p_{3/2}$ line suggested by Wagner is questionable. The use of gold deposition on nonconducting samples has been examined by Uwamino et al. (*A19*); a gold layer of 6 Å was found under their conditions to be an optimal value from the point of view of consistent binding energies of the nonconducting substrate (after setting the Au 4 $f_{7/2}$ peak at 83.8 eV) and of the fwhm of the various peaks.

Cross Sections. Another factor that has to be considered in the use of XPS as an analytical technique is the relative cross sections between various transitions. Nefedov and coworkers in a series of articles have investigated several aspects of this area. A Monte Carlo type calculation for several different types of sample configurations has shown that elastic scattering has a large effect on the absolute peak intensity and angular distribution of photoelectrons (*A20*). The mean free paths of the escaping electrons are affected also. Photoionization cross sections have been computed for H to Zn with Au Mα and Ag Lα X-ray lines (*A21*). Some comparisons with experiment were given.

Goldberg et al. (*A22*) have computed cross sections for a number of selected atoms. Comparisons to previous calculated values were good. Castle and West (*A23*) have measured the relative intensities of 45 elements with a silicon Kα X-ray source. (A description of the building of the Si X-ray source was also given.) A comparison to theoretical computations was made, and the results were usually in good agreement. Szajman et al. (*A24*) have measured subshell cross sections using Al Kα radiation for Li through Pb. After corrections for a contaminated surface, reasonable agreement with Scofield's cross sections was obtained for 1s, 2p, and 3d levels. The comparisons were not as favorable with other levels. Vulli (*A25*) has measured the asymmetry factor β (which takes into account angular emission factors of ejected electrons) and made comparisons to theory. The agreement was not very good; clearly more work of this nature is needed.

The use of chemical systems to test XPS has been carried out by several groups. Benziger and Madix (*A26*) have studied the observed intensities of adsorbates on a Fe(100) surface up to monolayer coverages as determined by LEED. For those elements studied (C, O, S, K), the agreement between experiment and computed photoelectron cross sections was very good. Garbassi et al. (*A27*) have employed mixed oxides of Te–Ni and Te–Ta. Agreement between previous sensitivity factors and experiment was satisfactory.

Inelastic Mean Free Paths. One approach to the determination of inelastic mean face paths (IMFP) has been to use thin overlayers. With this approach a wide variety of materials has been investigated. Vasquez and Grunthaner (*A28*) have developed a method to determine overlayer uniformity (in their case, an oxide layer on Si). They found that the IMFP can vary for nominally the same material; this was ascribed to strained regions that are structurally different. Szajman et al. (*A29*) have used CdTe overlayers on Ag and found IMFPs of 13–32 A for electron kinetic energies of 350–1439 eV.

The matrix effect on IMFPs has been investigated by Hirokawa and Danzaki (*A30*). Ni/Cu and Ti/Zr hydroxides of different compositions and with and without matrices of Fe or Mg hydroxides showed a matrix effect of 20% or less. The differences in the energies of the lines analyzed were less than 300 eV.

Organic overlayers continue to offer a wide range of reported IMFPs. Roberts et al. (*A31*) have determined IMFPs of 29 and 33 A for electrons of 1196 and 1328 eV with thin films of poly(methylmethacrylate) on SiO_2. Only a poor $E^{0.5}$ energy dependence was noted. Langmuir-Blodgett films of cadmium stearate, a cadmium salt of a diacetylene polymer, and 9-*n*-

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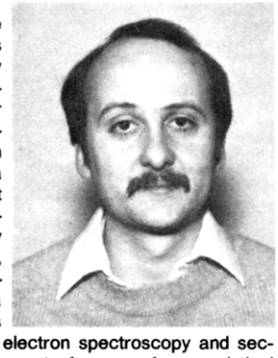

Richard Colton is a research chemist at the Naval Research Laboratory. He earned his B.S. and Ph.D. degrees from the University of Pittsbrugh in 1972 and 1976, respectively. He performed his graduate work under the direction of Dr. J. W. Rabalais in the areas of ultraviolet and X-ray photoelectron spectroscopy. In 1976, he was awarded a NRC Resident Research Associateship at the Naval Research Laboratory for a research proposal dealing with the secondary ion mass spectrometry (SIMS). In 1977, Dr. Colton joined the staff at NRL and conducts basic and applied research in the area of surface chemistry. His research interests include surface and materials analyses by electron spectroscopy and secondary ion mass spectrometry, the development of new surface analytical tools, and the study of the mechanisms of molecular and polyatomic ion emission. Dr. Colton is a member of the ACS and AVS.

butyl-10-anthrylpropionic acid have been used by Clark et al. (*A32*) to determine the IMFP at 1170 eV. Values for the respective polymers were 45, 57, and 70 A; the packing density of the various films appear to affect the IMFP. Hupfer et al. (*A33*) have employed polymerized layers of the Cd salt of diacetylene carbonic acid on various substrates to obtain IMFP's. They found a range of values from 83 to 121 A for electron kinetic energies of 788–1480 eV; due to scatter in the data, no energy dependence could be found. From these studies it continues to be apparent that the properties of the organic films used play an important part of the reported IMFPs.

Nonuniform Surfaces. The use of XPS for analysis of nonuniform surfaces, i.e., spherical particles, catalyst, and contamination, has to take into account the nature of the surfaces. Cross and Dewing (*A34*) have found that the equations for flat surface, when used with overlayers on spherical particles, could be in error by a factor of 2. Also noted was that changes in the takeoff angle did not alter the overlayer to substrate ratio. Ebel (*A35*) has concluded that at low takeoff angles, measurement difficulties predominate, while at large angles, roughness hinders the determination of the thickness of overlayers. A stacking model for nonporous particles has been tested experimentally for Mo oxides and Al_2O_3 with surfaces areas of 10–300 m^2/g by Defosse (*A36*). Takeoff angle dependence has been studied for oxide and contamination layers on Si by Yamada and Kuroda (*A37*). A simple angular dependent model could describe their results in terms of uniform layers. Ebel (*A38*) has developed a method to measure overlayer thickness that does not require a knowledge of the takeoff angle of photoemitted electrons. The method depends upon assumptions for the energy dependence of the IMFP, photoelectron cross sections, and the measurement of two different energy levels for the same element.

X-ray Induced Auger Transitions. The use of Auger transitions in conjunction with XPS lines has continued to be studied. Wagner et al. (*A39*) have investigated oxygen-containing organic and inorganic compounds by observing both the kinetic energy of the KLL Auger transition and the O 1s photoemission line. With the use of the modified Auger parameter (i.e., the addition of the Auger transition kinetic energy and the XPS binding energy), the data showed that the various classes of compounds had distinct locations in the chemical state plots. The Bremsstrahlung-induced Auger lines (which are above the X-ray photon energy) have been recorded by Wagner and Taylor (*A40*) for compounds of Al through S, Br, Mo, Ta, Au, and Pb. The Auger parameter that was computed showed differences of up to several electronvolts for the various compounds containing the same element. Castle and West (*A41*) have investigated several Si-containing compounds and minerals by the same procedure (these authors used a slightly different definition of the Auger parameter). The positions of Bremsstrahlung-induced Auger transitions and the Auger parameter for red P have been observed by Scharli and Brunner (*A42*). Bremsstrahlung radiation (with a Cu anode) has been used by Holton et al. (*A43*) to identify the Al 2p line and a plasmon peak for various thickness of Al on Cu. The intensity ratio of the Al 2p to $KL_{2,3}L_{2,3}:^1D_2$ line decreased as the film thickness increased; these results agreed with theory. Thomas (*A44*) has used a theoretical procedure to understand the relationship between the Auger parameter and the extraatomic relaxation energy. In certain instances the numberical evaluations can be somewhat in error. Note: Other examples of Bremsstrahlung-induced Auger transitions are in the Auger electron spectroscopy section of this review.

Satellite Effects. In addition to the main core lines observed in XPS, satellite lines due to shake processes and plasmons are observed often in many systems. Behstedt (*A45*) has shown that at the kinetic energies of electrons used in XPS, the relaxation effect of valence electrons is small and the observed spectrum depends upon the core hole. The shake-up spectra of the O and N 1s levels in para-substituted nitrobenzenes and nitrobenzenes has been investigated experimentally and theoretically by Distefano et al. (*A46*). The shake-up lines increased in intensity (up to 50% in some cases) with electron-releasing ability of the substituent. For *N,N*-dimethyl-*p*-nitroanaline both in the gas and solid phases, Freund and Bigelow (*A47*) have been able to explain theoretically the observed spectra. They suggest that their results were consistent with a negative shake-up energy concept. Loubriel (*A48*) analyzed the shake-up intensities and energies of $Cr(NO)_4$ by SCF-Xα-multiple scattering calculations. Good agreement with regard to intensity and energy of the main satellite peaks vs. experiment was achieved. Loubriel (*A49*) has used the same procedure to compute the shake-up intensity and energy for $Ni(CO)_4$. The satellite peak was assigned to a 1π to 2π shake-up transition. The satellite structure of the 2p and 3s levels of Fe(III) halides and $MnCl_2$ has been investigated by Scrocco (*A50*). It was concluded for the case of the 2p transitions that multielectron excitations were most predominant, while for 3s and 3p levels, multiplet splitting was the major component of the observed satellite structure. Somewhat similar conclusions have been made for the Ni halide 2p and 3p satellite structure by Scrocco (*A51*). Tse et al. (*A52*) have observed the shake-up spectra of the dimethyl compounds of Zn, Cd, and Hg for both the main metal and carbon photoemission line. From SCF-Xα-SW calculations, the energies and intensities of the metal shake-up peaks were in good agreement with experiment; for the C shake-up peaks, the calculations yield reasonable values for the energies, but the intensities were markedly underestimated. Cox (*A53*) has been able to explain the observed regularities in the 3s–3d and 4s–4f exchange splittings. Also it was predicted that correlation effects should remain proportional to the exchange integrals with changes in spin state and covalency.

Data Handling. Proctor and Sherwood (*A54*) have modified the central point smoothing process that many workers use to smooth XPS data with poor signal-to-noise ratios. The procedure involves the use of an estimation of values at the ends of the spectrum which allows a large number of smoothing calculations. Examples with noisy data for O 1s and Sn 3d spectra were given. The deconvolution of XPS spectra by the maximum entropy method has been described by Vasquez et al. (*A55*). The procedure can resolve peaks that could not be done by Fourier transform methods; however, line shape information is lost. Davis et al. (*A56*) have developed a deconvolution procedure for XPS spectra. The instrument/loss function includes a source function, a backscattered electron spectrum energy loss, and an analyzer function. Examples with GaAs, GaSe, and GeS were given.

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Radiation Damage. Changes in XPS spectra due to the X-ray source have been observed in a number of cases since the technique has become widespread. The number of such instances appears to be less than with the electron beam normally used with AES. Photon-stimulated desorption usually requires photons of a specific wavelength and therefore is observed most easily when a tuned source is used.

Copperthwaite (*A57*) has reviewed a number of systems where changes in the XPS spectrum with time of X-ray exposure have been reported. In addition, other methods of inducing changes (i.e., electrons, high energy radiation, and heating) that have been analyzed by XPS have been considered. The need for the coupling of different techniques in studies of this nature has been noted also. The effect of temperature and X-ray exposure time on the N 1s signal of $NaNO_3$ has been examined by Copperthwaite (*A58*). It was suggested from the XPS spectra that at least four reduction products of NO_3^- were present in the surface region. The X-ray-induced decomposition of gold(III) dethiocarbamates has been investigated by van Attekum and Trooster (*A59*). They have found that the decomposition rate could be reduced by employing a graphite substrate and lowering the sample temperature. The effect of UV radiation on graphite fluoride in various solvents has been monitored by Watanabe and Ueno (*A60*). They found that the C-to-F ratio increased and the relative number of CF_2 and CF_3 groups decreased with increasing exposure. Grunthaner et al. (*A61*) have used the electron flood gun source, that often is employed to reduce charging, to investigate changes in the Si/SiO_2 interface region. They have observed a decrease in the signal ascribed to Si(+3); they also noted the effect of the flood gun potential on the position and fwhm of the Si and O XPS peaks. Damage due to low energy ion bombardment on transition metal sulfides has been studied by Coyle et al. (*A62*). They observed large differences in beam dosages with Ar to induce formation of the metal and sulfur from Fe to Cu; an inverse relationship was noted with O_2^+ bombardment. Suoninen et al. (*A63*) have observed that XPS is somewhat less sensitive to changes induced by an electron beam on V_2O_5 compared to LEED or appearance potential spectroscopy. Electron beam interactions with selected halates and perhalates have been investigated by Sasaki et al. (*A64*). They have found that $LiIO_4$ was very resistant to damage, but $NaClO_3$, $LiClO_3$, and $LiBrO_3$ decomposed stepwise by losing oxygen to Cl^- or Br^-.

New Instruments and Methods

An angular resolved electron spectrometer with a position-sensitive multidetection system has been described by Hansson et al. (*A65*). The analyzer had about twice the energy resolution and four times the angular resolution with the same count as an advanced commercial analyzer. Read (*A66*) has patented an analyzer that incorporates both a cylindrical mirror and a half-spherical analyzer. The analyzer can operate in a raster mode for spatial analysis, regional analysis about a given point, or entire sample analysis. Richter and Peplinski (*A67*) have studied the transmission function of an hemispherical analyzer in the fixed transmission and fixed retarding modes of operation. Over an energy region of 400–1470 eV, the ratio of fixed transmission energy to fixed retarding methods had a kinetic energy dependence of $E^{-1.25}$. Concurrently, Cross and Castle (*A68*) also have investigated the same problem with the same type of analyzer. These authors have suggested that above 550 eV, the kinetic energy dependence mentioned above should go as $E^{-0.8}$, with an increase in the exponent as the kinetic energy decreases. It is possible that the method of determining this ratio is dependent on the experimental procedures of the two groups. VanAttekum and Trooster (*A69*) have considered the resolution that can be obtained with an unmonochromatized Mg Kα X-ray source and have concluded that, under their experimental conditions, the reported line widths are not materially less when a monochromatic X-ray source has been used. Keast and Downing (*A70*) have shown that with a collimator tube, areas of less than 0.5 mm^2 can be analyzed by XPS. While most samples studied by XPS are either rigid solids or gaseous, other forms can be investigated. Hirokawa and Oku (*A71*) have considered the problems of the analytical analysis of powders. Calibration curves for individual transition metal oxides were made and the procedure given was tested on samples prepared by several different methods. Siegbahn et al. (*A72*) have made improvements on the analysis of liquids by XPS. By controlling the temperature of the liquid on a metal backing holder and the use of a monochromatic X-ray source, a 10-fold improvement in the signal-to-noise ratio, an extension of the number of possible solvents, and an improvement in line widths were achieved. Cazaux et al. (*A73*) have developed a technique for simultaneous bulk and surface analysis by electron spectroscopy. The material to be analyzed is an anode that is bombarded from behind by an electron gun; electrons are ejected toward an energy analyzer. Lateral resolution on the order of 15 μm was achieved.

Analysis of Various Materials

Depth Analysis and Layers. XPS usually is not used for depth profiles or layered materials analysis because most systems are not capable of making continuous determinations during sputtering. This is not a limitation with most AES systems. The use of XPS for measuring depth profiles by knowing the angle of the escaping photoelectron has been proposed independently by Nefedov (*A74*) and Pijolat and Hollinger (*A75*). Nefedov has considered several different concentration gradient profiles and has computed the variation in intensity with the escape angle. Typical errors were not greater than 20%. Pijolat and Hollinger have studied the effect of escape angle for three systems, Ag/Al_2O_3, SiO_2/Si, and Ni/Cu. From both experimental and computer simulation results, it was suggested that a depth resolution of approximately IMFP/3 could be achieved. Mizokawa et al. (*A76*) have investigated the energy shifts of Ga photoelectron and Auger lines and P photoelectron lines between an oxide layer and a substrate of GaP. They concluded that differences found for oxides formed under different conditions were due to changes in the Fermi level pinning position of the substrate and not to changes in the substrate structure or extra atomic relaxation charging or an interface dipole. Steiner and Hufner (*A77*) have investigated binding energy shifts for Ni on Au and Au on Ni overlayers. Comparisons with calculated binding energy shifts and surface segregation energies were made. The surface composition of several Zr–Ni compounds after heating in air and FeNi alloys on a SiO_2 substrate has been studied by observing the takeoff angle dependence of the core photoelectrons and ion sputtering by Nefedov et al. (*A78*). Gilding et al. (*A79*) have computed the intensity as a function of takeoff angle for a model C/H/O polymer, and protein adsorption and desorption from a methyl methacrylate/acrylic acid polymer. Good comparisons were made with radio-labeling techniques.

The effect of ion bombardment on a sample has been examined by Riviere (*A80*), who used XPS to study tin plate passivated by dichromate. Different profiles with respect to Cr(III) concentration were found; with 5-keV Ar ions, a rapid reduction of the Cr(III) ions was observed. The higher energy ions also removed material at a greater rate for a given dose.

Alloys and Solid Solutions. The investigation of alloys by XPS can lead to several useful areas of more practical applications such as catalyst and thermodynamic information. Steiner et al. (*A81*) have measured the binding energy shifts for a number of dilute alloys (<10%) and compared the data with the alloy heat of formation derived from a semiempirical model. Good agreement between the experimental and calculated values was achieved for most of the systems investigated.

The Mn $2p_{3/2}$ XPS spectra of Mn–Ag alloys, as observed by Steiner et al. (*A82*), have shown a peak 4 eV higher in binding energy than that of the metal even at low Mn concentrations. This finding was ascribed to a large hybridization of the Mn 3d electrons with the neighboring host d-electrons; no shifts or broadening in the Ag 3d lines was noted. Ando (*A83*) has concluded that Cu is monovalent for the solid solutions of $Fe_{1-x}Cu_xCr_2S_4$. Hirokawa et al. (*A84*) has found agreement for theoretical photoionization cross sections and mean free paths when applied to Cu–Ni alloy surfaces. From shifts in the Pd and Ag binding energies for Pd–Ag alloys, Steiner and Hufner (*A85*) have been able to make correlations for the heats of formation of the alloys with calorimetric measurements. Kleiman et al. (*A86*) have found for Pt–Cu alloys that only the core level valence band centroid of Cu shifts with changes in composition. The Cu structure suggested dehybridization and little charge transfer.

Polymers. XPS is being used extensively to study polymer surfaces. As might be expected, many investigations have been centered on materials of practical importance, biomaterials,

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

adhesion, etc. However, there are a number of areas where fundamental information is reported. Dilks (*A87*) has reviewed the use of XPS for the analysis of polymers with a number of applications. Most reports usually are concerned with core level spectra, but work on the X-ray-induced valence spectra of polymers has been summarized by Pireux et al. (*A88*). Multicomponent block polymer surfaces have been studied by O'Malley and Thomas (*A89*). The use of intensity ratios, variable takeoff angles, and shake-up satellites in the analysis of these surfaces was illustrated. Dilks (*A90*) has observed that peroxy linkages in polymers fall into two regions of binding energies, i.e., ~534 eV for peroxides and hydroperoxides and ~535 eV for peracids, peresters, oxetanes, dioxiranes, and malozonide. The peroxy features are unstable in the X-ray flux.

The use of chemical reactions to characterize polymers has been employed by several groups. Everhart and Reilley (*A91*) have derivatized the surface of plasma modified polyethylene. A vertical inhomogenity for N and O was noted via angular dependent measurements; also, decomposition was found in some cases. These workers also observed the effect of various solvents on treated polymers and noted that some functional groups were mobile on the surface (*A92*). Copolymers of vinyl chloride and vinyl acetate, with or without vinyl alcohol, have been investigated by Pennings and Bosman (*A93*) by hydrolysis followed by reaction with a fluorine-containing acid chloride. The extent of alcohol and acetate groups on the surface then was monitored by the amount of fluorine observed. Bigelow et al. (*A94*) have sulfonated polystyrene and found by angular resolution measurements that with more reaction S and O preferentially were below the surface. The photooxidation of polystyrene has been studied by Peeling and Clark (*A95*) who observed that several carbon–oxygen moieties were formed. From the disappearance of the C 1s shake-up peak, it was concluded that the oxidation occurred on the phenyl groups.

Andre et al. (*A96*) have reviewed the attempts to understand the XPS spectra of various polymers (e.g., block or alternating structure) via ab initio calculations. In addition, the calculations for bond alteration in linear chains (e.g., polyacetylene) were considered. Nonempirical LCAO-MO-SCF calculations have been made on a number of nitrogen-containing polymers by Clark and Harrison (*A97*). They found that for certain functional groups a simple additive model was inadequate to explain the experimental results. Examples of the use of XPS for analysis of polymer surfaces will be given in the section on applications.

XPS of Compounds. Listed in Table I are reports on the XPS spectra of various compounds. As can be seen, a wide variety of materials has been studied. Parameters such as binding energies, substituent, and structural effects have been reported. The information that is contained in these papers should be of use to those with interest in the listed or similar compounds.

Applications

XPS has been used to study a wide variety of materials in order to obtain practical information about the surface region and electronic structure. The articles cited below are not inclusive as to the uses to which XPS has been placed but do offer an indication of some of the applications that have been reported. These areas are polymers and organic compounds (*A98–A101*), polymers from plasma reactions (*A102–A110*), biological and related materials (*A111–A115*), adhesion (*A116–A119*), soils (*A120*), electrodes (*A121–A129*), ion selective electrodes (*A130–A133*), silica and glass surfaces (*A134–A139*), catalysis (*A140–A158*), thin films (*A159–A165*), atmospheric particles (*A166–A167*), corrosion (*A168–A173*), electronic materials (*A174–A176*), and ion implanted materials (*A177–A179*).

Theory

Theoretical work has been concerned with such topics as the prediction of binding energies, multiplet splittings, shake-events, and the density of states. Maksic (*A180*) has suggested that a weighted Lowdin orthogonalization procedure with proper factors could be used for construction of basis sets. Shung and Langreth (*A181*) have found that asymmetry due to the difference between the threshold and core energy follows a simple power law fairly well. This is in spite of the fact that the Hamiltonian used near the threshold does not appear to be valid at a first approximation.

Broughton and Bagus (*A182*) have computed the most common core levels shifts and relaxation energies for many metallic and semimetallic elements and the anions of the halogens and chalcogens. They have concluded that the effects of core–valence and valence–valence interactions can have an influence on core level shifts. The 1s photoelectron spectrum of Li has been calculated by Larkins et al. (*A183*) by Hartree–Fock or configuration interaction procedures. Good agreement with regards to binding energy and shake-off processes was achieved. A unified approach with a nonseparable core-hold conduction-electron scattering potential has been used by Bose et al. (*A184*) to compute the XPS spectra of Na and Al. The theoretical spectra were able to predict the asymmetry of the main peak, the plasmon satellite and the background; agreement with experimental work was good. Beck and Nicolaides (*A185*) have developed a procedure that selects the correlation effects and open-shell multiplet splittings most important in the XPS spectra of simple metals and insulators. Agreement with experimental results for alkali metals and halides was achieved. The prediction of binding energies by a Dirac–Fock program for a number of elements has been made by Key et al. (*A186*). For light elements, relativistic corrections were found to be significant; in most cases the calculated results were within 1 eV of experiment. Clark et al. (*A187*) has studied the effects of oxidization state and electronic environment for the first-row transition metal ions with a LCAO-MO-SCF procedure. They have found that most of the relaxation energy comes from the 3d-valence component. Feldkamp and Davis (*A188*) have used a simple model based upon the interaction of the 3d band with the core hole for Ni. Reasonable agreement for the shape of the $2p_{3/2}$ was found. Treglia et al. (*A189*) have concluded that correlation effects within the 3d band of Ni cannot be quantitatively explained by available theory with regards to satellite structure. Two different types of central field calculations (relativistic Hartree–Fock and Hartree–Fock–Slater) have been shown by McGilp and Weightman (*A190*) to give essentially the same binding energies for Zn, Cd, and Hg. Discrepancies between theory and experiment (usually a few electronvolts) were ascribed to many-electron effects. Cox (*A191*) has been able to include some correlation effects into the van Week equation to explain the 3s–3d and 4s–4f exchange splittings. The use of the splittings in XPS spectra to consider changes of spin state and covalency was discussed also.

Ohno (*A192*) has employed a many body approach to describe the 4s–4p XPS spectra of Pd to Xe. Much better agreement with experiment was achieved with this method than with a quasi-particle approach. Cox et al. (*A193*) has measured and computed the 4s and valence band spectra for the rare earth metals. An atomic configuration interaction model was used by Boring et al. (*A194*) to describe the XPS 5p spectra of several actinides. The atomic model could account for the satellite structure without the need of extraatomic relations for shake-up processes.

Several theoretical investigations of organic compounds have been reported. Ficker (*A195*) has used LCAO-MO-SCF calculations to compute the binding and relaxation energies for different configuration of ethane and ethylene. Agreement with experiment (where available) was within 1 eV. Nonempirical LCAO-MO-SCF calculations have been made on acetylacetone in various configurations by Clark and Harrison (*A196*). Reasonable agreement for the binding energies of O and C with different atomic environments was obtained; also considered was the enol form of the molecule and the shake-up features of the spectrum. Nokagaki et al. (*A197*) have characterized the energy difference and relative intensity for the N 1s line in a series of nitro-substituted aromatic compounds from the viewpoint of charge transfer. The nature of the shake-up process could be predicted from this approach. Bigelow and Freund (*A198*) have suggested that the N 1s and O 1s spectra of *p*-nitroaniline can be understood by intermolecular orbital coupling. Bossa et al. (*A199*) have tried to correlate the N 1s and S 2p binding energies with net atomic charges for substituted azolidiones by three different procedures, i.e., ab initio, CNDO, and EHT. Close agreement between the CNDO and ab initio results was achieved; poor correlations were found from the EHT procedure. Brant et al. (*A200*) have found from XPS spectra and semiempirical MNDO calculations a π-type bonding in bis(trifluoromethyl) oxides and CF_3SCF_3. This bonding was suggested to be im-

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

Table I. Compounds Studied by XPS

systems studied: major interest	ref
metals: binding energies data bank	*a*
selected metals: core level lifetimes	*b*
1,3,5-trinitro-1,3,5-triazocyclohexane, trinitrotoluene, and NH_4NO_3: effect of shock and structure	*c*
polyacetylene (doped and undoped): electronic structure	*d–i*
pyridines: binding energies and structural correlations	*j*
dithizone and related compounds: structural form	*k*
nitrogen-containing compounds: binding energies and structure	*l*
monomeric and dimeric Fe porphyrin compounds: electronic structure	*m*
metalloporphyrins: satellite structure	*n*
dibenzo crown complexes: structure	*o*
trithiapentalene and related compounds and Se and Te analogues: structural effects on chemical shifts	*p, q*
metal complexes of 2-mercaptobenzimidole and 2-mercaptobenzoxazole: binding energies and structure	*r*
cryptates complexes: binding energies	*s*
pyrimidine-thiones: binding energies and structure	*t*
Fe and Co nitrosyl complexes of *O*-phenylenebis(bimethylarsine): effect of ligands on binding energies	*u*
substituted acetylene compounds: binding energies and fit to theory	*v*
lithium graphite: π electron bands	*w, x*
alkaline earth oxides: binding energies and O KVV Auger line shapes	*y, z*
Te–Nb and Te–Ta oxides: binding energies and composition	*aa*
second and third row oxides: binding energies and spin-orbit splitting	*ab*
NaNCO: binding energies and theoretical fit	*ac*
Mg-montmorillonite: identification of different Mg sites	*ad*
binary metals: effect of impurities on properties	*ae*
Si–F alloys: chemical bonding	*af*
silicate and phosphate glasses: atomic environments	*ag*
phosphorus compounds: binding energy correlation to effective charge	*ah*
S_2N_2 and related compounds: binding energies	*ai, aj*
S_2N_2 ring derivatives: electronic structure	*ak*
Ca, Sr, and Ba: core level splitting	*al*
$Ca_2V_2O_3$: outer shell electronic structure	*am*
Ti to Zn: core level binding energies	*an*
transition metal borides: binding energies and electronic structure	*ao*
Ti, V, Cr, Mn, and Nb oxides: binding energies as a function of oxidation state	*ap*
V oxides: binding energies and electronic structure	*aq*
Cr_2O_5: valence states and binding energies	*ar*
Ti, Th, Ce, and La compounds: effect of screening	*as*
Ti, Zr, Nb, Mo, and HF: experimental and theoretical line shape	*at*
transition metal pyrites: core and valence band analysis	*au*
iron borides: composition effects	*av–ax*
K_2FeO_4: binding energies	*ay*
hydrous ferric oxides: binding energies and Auger parameters	*az*
iron fluorides: binding energies and bonding	*ba*
nickel halides: binding energies and satellite structure	*bb*
Ni complexes: binding energies, line shape, and structure	*bc*
Co, Ni, and Cu acetylacetonate: binding energies and satellite structure	*bd*
Ni and Pd complexes: binding energies	*be*
Ni and Pd alloys: valence band effects on core level line shape	*bf*
Cu_2O and CuO: primary and secondary peaks	*bg*
copper sulfides and selenides: valence states	*bh*
Cu(III) complexes: primary and secondary peaks	*bi*
Co complexes: binding energies and satellite structure	*bj*
GaN: binding energies and electronic structure	*bk*
$Ga_{1-x}Al_xSb$: core and valence band changes with varying composition	*bl*
AsF_3, AsF_5, and SbF_5: binding energies	*bm*
Y_2O_3-ZrO_2 solid solutions: electronic structure	*bn*
Nb, Mo, Ru, and Rh: analysis of line shape	*bo*
$[Mo_6Cl_8]Cl_4$: binding energies and comparison to theory	*bp*
Mo–S complexes: binding energies and structure	*bq*
hydrogen molybdenum bronze: conduction mechanism	*br*
MoS_3 and WS_3: structure and oxidation states	*bs*
Mo carbonyl complexes: binding energies	*bt*
ternary molybdenum sulfides: band structure	*bu*
mixed valence Ru compounds: electronic structure	*bv*
Rh oxides: electronic structure	*bw*
Rh and Pt complexes: binding energies and electronic structure	*bx*
PdO: binding energies and electronic structure	*by*
PdSb, PtBi, and AuSn: binding energies, intensities, and electronic structure	*bz*
Ag, Sn, $YbAu_2$, and Au: changes at vacuum-surface interface	*ca*
Ba and BaO: binding energies and Auger parameters	*cb*
BaS and $BaSO_4$: binding energies	*cc*
$LaCoO_3$: bulk and surface composition	*cd*
Ce compounds: binding energies and electronic structure	*ce*
$CeNi_2$: mixed valence states	*cf*
$CePd_3$: mixed valence states	*cg*
$CeBe_{13}$: change of valence with temperature	*ch*
rare earth oxyfluorides and orthouanadates: binding energies and electronic structures	*ci*
$EuCu_2Si_2$: valence states	*cj*

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

Table I (*Continued*)

systems studied: major interest	ref
Yb-Al films: mixed valence states	*ck*
Yb intermetallics: mixed valence states	*cl*
H_xWO_3: electronic structure vs. composition	*cm*
Pt thiourea complexes: mixed valence states	*cn*
Pt oxides: binding energies and bonding	*co*
Au compounds: binding energies	*cp*
Au cluster and phosphine compounds: binding energies and intensities	*cq*
Tl: binding energies	*cr*
Tl halides: changes in crystal structure	*cs*
Th: line shape	*ct*
Th, U, and several compounds: electronic structure	*cu*
Th and U intermetallics: binding energies and electronic structures	*cv*
U and UO_2: binding energies and satellite structure	*cw*
UO_2, US, and UAs: electronic structure	*cx*
UGa: satellite structure	*cy*
UAsSe: electronic structure	*cz*
UPd_3: electronic structure	*da*

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POSCO Future M v. TIAX LLC
POSCO Future M EX1020

Table I (Footnotes *Continued*)

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portant in lone-pair stabilization.

Green's function methods have been suggested by Born and Ohrn (*A201*) for use to compute binding energies in simple molecules. For H_2O and C_2H_2, good agreement for the valence band region energies was obtained; but, much poorer results for the core level energies were found. Agren and Muller (*A202*) have used two different Frank–Condon calculations to compute the O spectrum for H_2O; both methods yielded similar results. Ab initio Hartree–Fock calculations have been employed by Agren et al. (*A203*) to understand the vibration excitations in the XPS core and valence spectra of NH_3 and H_2O. Overall, the agreement between theory and experiment was good. Tse and co-workers (*A204*) have used the SCF-Xα-SW method to compute the shake-up spectra of the isoelectric series CH_4 to Ne and CO. Identification of the major components of the shake-up spectra and predictions of the energies of these transitions to within a few electronvolts was made. The intensities of the shake-up structures were computed also, and it was found that a touching sphere approximation was better than that for an overlapping sphere. Palma et al. (*A205*) have combined features of the sudden approximation with the plane wave method to compute the spectra of CO, H_2O, and CH_4. Reasonable agreement for both the energies and relative intensities features for the spectra of these compounds was achieved. An ab initio full valence configuration interaction procedure has been used by Hanjou et al. (*A206*) to compute the energies and intensities of the XPS spectra of O_2 and NO. The results were in reasonable agreement with experiment.

A self-consistent charge molecular orbital computation method has been used by Maksic and Rupnik (*A207*) to find binding energy shifts for N 1s for 15 different compounds. An average deviation of ~0.5 eV was obtained and this method can be extended to large molecules. Ghatikar and Padalia (*A208*) have used a semiempirical approach to compute the effective charge on B, C, N, O, F, P, and S in a large number of compounds from XPS spectra. Comparisons to other calculational procedures were made. Sasaki and Adachi (*A209*) have employed a discrete variation Xα cluster method to compute the XPS valence spectra of several oxyanions; good agreement with experimental results was found. An SCF-Xα-SW calculation of 1s and 2p binding energies and the KLL Auger energies for SiH_4, $SiCl_4$, and SiF_4 has been made by Hartmann and Szargan (*A210*); good agreement was obtained with experiment where data were available.

An analysis of chemical shifts in oxides with a point charge ionic model has been studied by Broughton and Bagus (*A211*). The limitations of the Madelung potential were shown, as it was found that the model worked well for closed shell and low oxidation state oxides, but not for high oxidation state systems. Bagus and Bauschlicher (*A212*) have concluded that the restricted Hartree–Fock method cannot give reasonable results for a free O_2 anion, such as would be found in ionic oxides. If a Watson sphere is used as an external potential, then agreement to approximately 3.5 eV for FeO was obtained. Nakamatsu et al. (*A213*) has used a SCC DV-Xα approach to compute with good agreement the XPS valence spectra for several alkali earth titanates. Several perovskite compounds have been examined with the SCF-MS-Xα method by Michel-Calendini et al. (*A214*). The density of states, binding energies, and other properties were computed. The surface electronic state of ZnO has been calculated from XPS data by Ivanov and Pollmann (*A215*). While there were no surface states in the band gap, distinct surface ionic O 2p and Zn 4s surface state features were found.

Broclawik et al. (*A216*) have carried out SCF-SW-Xα calculations on various molybdenum oxide clusters to interpret the experimental XPS spectra. The results were used to explain the stages of reduction and structure for the oxides considered. A self-consistent Madelung potential has been combined with the DV-Xα cluster procedure by Tsukada (*A217*) and applied to a ReO_6^{6-} cluster. Improvements over previous calculations with comparison to XPS valence spectra were claimed. Thornton and Dempsey (*A218*) have used a multiple scattering Xα and intermediate coupling calculations to evaluate the 3d and 4d spectra of CeO_2. The effects of inter- and intraatomic coexcitations and multiplet coupling made comparisons to experiment difficult.

The energy level populations for the valence band region of several uranium compounds have been calculated by Shiokawa et al. (*A219*). Reasonable agreement with the experimental XPS spectra was achieved. Several transition metal ions and halide clusters have been treated by the mass operator approximation by Pavao and Leite (*A220*). This approach gave a reasonable description of electron exchange correlation effects. Ab initio calculations have been made by Larsson et al. (*A221*) to investigate the satellites in the core level spectra for $CuCl_4^{2-}$ clusters. The strongest satellite was ascribed to a Cu 3d–Cl 3p excitation. Folmer and deBoer (*A222*) have developed a simple procedure to account for the observed asymmetry in the core lines of several different anions. The asymmetry could be ascribed to density of states of the anion. Maksic and Rupnik (*A223*) have used the same method noted earlier (*A208*) for seven Ge compounds; comparable deviations in binding energy shifts (~0.5 eV) to those of N were found. Nguyen et al. (*A224*) have calculated the electronic structure of ZrS in NaCl-type and WC-type structures, and the results were in good agreement with experiment. A relativistic Dirac–Fock calculation and a simple electrostatic model were used by Mathews et al. (*A225*) to compare the experimentally observed binding energies for the 3 $d_{5/2}$ lines of Cs and I in CsI. The Dirac–Fock energies were

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

in better agreement for the Cs than the I. Relativistic Hartree–Fock calculations to predict the 4f binding energies of several rare earth compounds due to different configuration have been made by Herbst and Wilkins (*A226*). Agreement with experiment to within 2 eV was achieved.

AUGER ELECTRON SPECTROSCOPY (AES)

Introduction

Auger electron spectroscopy has advanced on many fronts over the past 2 years. Experimental and theoretical improvements have extended the types of analysis possible with AES. The basic principles and some applications of AES have been reviewed by Holloway (*B1*), and for surface analysis by Wild (*B2*).

Auger Line Shapes

Elements. The line shapes of Auger spectra of numerous elements have been studied in detail during the reporting period. When a valence electron is involved in the transition studied, information concerning the density of states (DOS) of the valence state and the mechanism of the Auger process can be obtained. Gavriljuk and Lifshits (*B3*) have analyzed the AES C KVV spectrum and concluded that the bonding structure of the surface is similar to that of the bulk. Lasser and Fuggle (*B4*) have studied the KLV spectra of Na, Mg, Al, and Si and concluded that the effect of core holes on the ionized atom was greater on the energy distribution of s states than p states. Gunnarsson et al. (*B5*) investigated the effect of plasmon gain peaks on the Auger spectra of Na, Mg, and Al. They concluded that a Lorentizan convolution was inadequate to explain these peaks and that interference effects between different plasmon gain peaks broaden the final gain peak. The Auger spectra of Si continues to receive attention. Brockman and Russer (*B6*) have investigated the $L_1L_{2,3}V$ and $L_{2,3}VV$ transitions for Si(111). They claimed that they were able to find evidence for surface dangling bonds states. Agreement with the DOS for the $L_1L_{2,3}V$ spectra, but only with a partial DOS, could account for the $L_{2,3}VV$ spectra. Morgen and Onsgaard (*B7*) could explain the $L_{2,3}VV$ spectra from their study of Si(111) on the 7×7 superstructure from the DOS. These workers also investigated the effect of the adsorption of O_2, H_2O, and CO, and Ar^+ bombardment on the $L_{2,3}VV$ spectra.

Aksela and Vayryner and co-workers have studied the Auger spectra of a number of different elements in gas and solid state. The high-resolution $L_{2,3}M_{2,3}M_{2,3}$ and $L_{2,3}M_{2,3}N_1$ spectra of free K were interpreted from optical data (*B8*). For Mn Vayrynen (*B9*) has noted large differences in the intensities ratios of the various $L_{2,3}MM$ transitions of Mn between the metallic and atomic state. Inner shell transitions for the metal were more atomiclike while outer shell transition were bandlike. The $L_2L_3M_{45}$ Coster–Kronig transitions have been shown to be responsible for the low intensity of the L_2MM transitions. Also $M_{2,3}M_{4,5}M_{4,5}$ super-Coster–Kronig transitions were observed (*B10*). Jach and Powell (*B11*) have found shifts of near 1 eV in the L_3VV peak positions for Cu and Ni when the incident electron energy was varied from just above the L_3 binding energy. Aksela (*B12*) has determined the kinetic energy shifts between the atomic and solid-state AES $L_3M_{4,5}M_{4,5}$ spectra for Cu to Se. A thermochemical model for the Auger process gave good agreement between the experimental and calculated values. Hilaire et al. (*B13*) have concluded that the $M_{4,5}N_{4,5}N_{4,5}$ transitions of Pd are bandlike and that the fine structure of these peaks depends on surface structures. Aksela (*B14*) et al. studied the LMM transitions of Br_2 and Kr. An agreement between calculated (jj coupling for the initial state and intermediate coupling for the final state) and experimental spectra for the $L_{2,3}M_{4,5}M_{4,5}$ and $L_3M_{2,3}M_{4,5}$ transitions was found; disagreement was noted for the $L_2M_{2,3}M_{4,5}$ cases.

Aksela et al. (*B15*) and Pessa et al. (*B16*) have studied the $M_{4,5}N_{4,5}N_{4,5}$ spectra of Ag through Te, both in the vapor and solid phase. The relative energies and intensities were approximately the same for both phases; however, the solid phase line widths were almost 1 eV wider. The lines are also shifted to higher kinetic energies (7–12 eV) for the solid vs. the vapor state. Two theoretical models (thermochemical and self-consistent-field density formalism) were found to give good agreement for the free atom–metal shifts. The $M_{4,5}N_{4,5}N_{4,5}$ and $M_{4,5}N_{4,5}O$, spectra of silver vapor have been studied via optically known energy levels by Vayryner et al. (*B17*). Kaindl et al. (*B18*) have found differences for the NOO Auger line shapes of Xe adsorbed from single layers to multilayers on Pd(001) and on preadsorbed Kr. Shifts in the main Auger peaks between unoxidized and oxidized Sm and Fr have been reported by Netzer et al. (*B19*). Also, the spectra were characterized by broad, asymmetric profile and distinct onsets. Matthew et al. (*B20*) investigated the $N_{4,5}N_{6,7}N_{6,7}$ and $N_{4,5}$-$N_{6,7}O$ transitions for Ir through Au and observed that discrepancies between earlier theory and experiment may be due to sensitivity of super-Coster–Kronig and Coster–Kronig transitions to configuration interaction.

Compounds, Alloys, and Complexes. The line shapes observed in the Auger transitions of various compounds, alloys, and gas–solid reactions have been studied extensively. Madden (*B21*) has reviewed the use of AES for obtaining chemical information in a large number of systems. Line shape analysis for the study of molecular surface reaction products has been reviewed by Netzer (*B22*). Bermudez and Ritz (*B23*) showed that the core–valence (F center) Auger transition for LiF yielded a bandlike final state. Bacon (*B24*) observed shifts in the derivative AES spectra of elemental B, Si, and Zr and compounds of these elements. Also noted were changes in the energy minima among the various materials investigated. Numerous carbon compounds have been studied by AES. The AES C KVV spectrum of graphite intercalated with the alkali metals (Cs and Rb) have showed a transition not present in pure graphite (*B25*). However, the selection rules for such transitions proposed by Oelhafen et al. (*B25*) were challenged by Bader (*B26*). Rye and co-workers have investigated the linear alkanes C_1 to C_6 and C_3 through C_6 cycloalkanes (*B27*, *B28*). Fingerprint spectra were obtained for the linear C_1 to C_4's and neopentane C KVV spectra. A one-electron theory could explain the spectra for methane and ethane, but discrepancies began to appear for propane. Differences were found also for the various cycloalkanes (*B28*). Several metal carbonyls gave essentially similar C KVV spectra (*B29*) and they were compared to a theoretical analysis which suggested that some π back-bonding was involved in the observed transitions. The Auger spectrum of CH_3CN (*B30*) has indicated that the contribution from each C atom is independent for the total C KVV spectra. Also, the intensity depends on the electron intensity of the orbitals, and the hybridization sensitivity for group orbitals is similar to that in simpler compounds. The C and O KVV spectra of CO and CO_2 have been determined (*B31*) and compared to a calculated one-electron theory with regards to intensities and energies. Some disagreements were noted.

The monitoring of the $L_{2,3}VV$ spectra during the oxidation of Si(111) has been employed by several groups. Munoz et al. (*B32*) have proposed the detection of a molecular oxygen state, while Lang et al. (*B33*) have investigated the onset of silica formation. The possibility of detection of incompleted oxidization was discussed. Wildman et al. (*B34*) have used differences in the Si LVV spectra to monitor deviations in stoichiometry of thin oxide films on Si. Hezel and Lieske (*B35*) showed differences in the Si LVV spectra between amorphous SiO_2, Si_3N_4, and Si oxinitrides with various O/N ratios. In addition, a correlation of energy levels determined from electron energy less spectra with the observed transition energies gave good agreement. Taylor (*B36*) has observed differences in the X-ray excited LVV and KLL spectra of Si and Si_3N_4; the effects of various preparations and ion sputtering were studied also. The ability to detect the influence of hydrogen on the $L_{2,3}VV$ spectra of Si in Si_3N_4 has been reported by Madden (*B37*). Ho et al. (*B38*) has found a large difference in the $L_{2,3}VV$ spectra of Pd_2Si compared to Si, while the KLL spectra were very similar. The spectra were analyzed on the basis of a partial DOS. A partial density of states model was found by Bader et al. (*B39*) to explain the Si $L_{2,3}VV$ line shape of Pd_4Si, and oxygen chemisorption altered the line shape. Energy shifts in the LVV spectra of transition metal silicides ion implanted with P were observed by Wang et al. (*B40*). The Ar^+ induced Auger LVV spectra of Si and several metal silicon alloys have been found to be different by Iwami et al. (*B41*). They suggested that the observed electrons were from atoms just below the surface. Muria (*B42*) has investigated the LVV spectra of a number of sulfur-containing compounds. For sulfides the spectra were analyzed by a cross transition and the final state. With sulfates the effect of the molecular orbitals on the final line shapes was noted.

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

Weissmann et al. (*B43*) have reported differences in the $M_{2,3}VV$ transitions of the fluorides of Mn, Co, and Ni compared to the pure elements. Two peaks with different relative intensities for the fluorides were observed, with one peak suggested to be a cross transition from the F 2p level. The Auger line shapes of GeS(001) and GeSe(001) have been studied by Davis et al. (*B44*); the spectra were interpreted via a site-specific densities of state. Both anion and cation affects could be detected. Bahl et al. (*B45*) have compared calculated and experimental AES spectra of Se and Se compounds, both in terms of energy and intensities. A method for calculating energies proposed by Shirley was found to be better than a method based upon Koopmans theorem. The Pd $M_{4,5}N_{4,5}N_{4,5}$ spectra for various alloys have been studied by Weightman and Andrews (*B46*). The differences noted between the pure metal and the alloys were suggested to be due to the influence of the 4d band on $4d^2$ two-hole final states. This explanation does not appear to be valid for the Ag alloys also investigated by these authors (*B46*); in this case the Cini model could explain the differences in the Ag $M_{4,5}N_{4,5}N_{4,5}$ spectra. Mariot et al. (*B47*) also have studied AgPd alloys and reached similar conclusions. The $M_{4,5}N_{4,5}N_{4,5}$ spectra of In and In chlorides in the vapor have been studied by Aksela et al. (*B48*); they found that their results could be explained from known optical energy levels. Egelhoff and Tibbetts (*B49*) were able to show a large effect due to the core hole in the core–valence–valence spectra between Yb and a Yb–Al alloy.

Factors Affecting Auger Analysis

During analysis of AES spectra the possibility of various physical factors influencing the observation must be considered. Especially important are alterations due to the effect of the electron beam used in the determination of many AES spectra and changes arising from ion beams used for sputtering. These, and other effects considered below, i.e., backscattering and the mean free path of the ejected electron, are of extreme importance when quantification is desired.

Sputter Effects. Morgen and Ryborg (*B50*) have observed the effect of sputtering on the (111) face of Si by monitoring changes in $L_{2,3}VV$ spectrum. Structural changes (observed by LEED) were observed at ion doses of approximately the equivalent of a monolayer ($\sim 10^{15}$ cm^{-2}). With doses of $\sim 10^{17}$ cm^{-2} the Auger line shape had been altered markedly. Queirolo and Pignatel (*B51*) have found that the effect of the electron beam on the P signal in doped SiO_2 could be reversed by 1-keV Ar ions. Kny (*B52*) has observed a buildup of carbide type C on Si during Ar ion bombardment. CH_4 was the source of the C. Differences in the sputter yield in a Cu–Ni alloy have been noted by Rehn and Wiedersich (*B53*); changes in temperature did not alter the relative sputter yield. Solute segregation while sputtering at elevated temperatures for O in Nb has been demonstrated by Hofmann (*B54*). Thus, it is possible that diffusion effects can alter the apparent depth profile and that measurement of diffusion can be made. Preferential sputtering has been found by Frankenthal and Siconolfi (*B55*) in a Sn–Pb solder; the Pb-rich phase was removed at a greater rate than the Sn phase.

Electron Beam Effects. The damage due to the electron beam used for most AES analysis has been reviewed in some detail by Pantano and Madey (*B56*). The effects of charging, beam heating, and electron excitation have been discussed in some detail. Roll (*B57*) has examined theoretically the heating effect of an electron beam upon a thin film over a substrate. Under certain conditions, temperature increases of several hundred degrees are possible; comparisons were made with experiments by Röll et al. (*B58*) on multilayer Ni–Cu films. Ohunchi et al. (*B59*) have studied the migration of Na in thin films of soda–silica glass deposited on stainless steel. The diminution of the Na Auger signal with time was found to be strongly dependent upon the electron beam energy and to a lesser extent upon the current density. Ashley and Anderson (*B60*) have developed a model that describes the energy loss mechanism of cascading electrons in SiO_2. In addition, electron inelastic mean free paths that were computed agreed well with experiment where data were available. The effect of the electron beam on the Auger line shapes of various sulfur species have been noted by Miura (*B42*) and Turner et al. (*B61*).

Backscattering. The change in the observed yield of Auger electrons due to backscattered electrons has been studied by several groups. Ichimura et al. (*B62*) have made Monte Carlo calculations to account for backscattered electrons; good comparisons to experimental results were obtained for Al, Cu, and Ag with a somewhat poorer result for Au. Jablonski (*B63, B64*) has used a Monte Carlo method to compute backscattering for V through Ge, and several alloys. Differences of up to 10% between two definitions of backscattering were found with the largest discrepancies at lower electron beam energies. Gergery et al. (*B65*) have studied several materials (with a wide range of atomic number) and found that backscattering increases strongly with *Z*. LeHeriey et al. (*B66*) have considered the effect of both forward and backscattered electrons along with differences in atomic density to determine atomic sensitivity factors.

The escape depth of electrons in Ge has been investigated by AES by Grant and Monch (*B67*) and their values were in reasonable agreement with other studies. Nishmori et al. (*B68*) have studied escape depths for Cr on Ti and Fe substrates with AES and disappearance potential spectroscopy. They have concluded that the electron mean free path depends upon both the electron energy and the material under investigation.

Angular Effects. Angular effects on the Auger electron yield with single crystals have been considered both experimentally and theoretically by several groups (*B69–B74*).

Secondary Electrons. A correction for the Auger electron yield due to secondary electron emission has been proposed by Sickafus (*B75*), which involves a two-step process. Integrated Auger lines are first corrected for the cascade background and are then narrowed.

Instrumental Effects. LeHericy and Langeron (*B76*) have discussed the values and influence of various physical parameters upon equations used in quantitative Auger analysis. Seah (*B77*) has recommended that all electrodes in an energy analyzer should have the AC modulation applied in phase. This is very important with analyzers designed for both AES and XPS and in the energy region below 200 eV. Seah (*B78*) also has suggested for quantitative AES analysis that the negative portion of the derivative spectra with a modulation of 5 eV be used; thus, the loss of resolution tends to cancel the effects of line shape, background, etc. Changes in peak-to-peak height at submonolayer coverages due to alterations in the background for the cylindrical mirror and the low energy electron diffraction analyzers have been computed by Anderson (*B79*). The simple model proposed gave good results in the energy regions considered (30–200 eV). Ingrey and Westwood (*B80*) have observed that the relative peak-to-peak height ratio of the Cd to Se in a CdSe single crystal varied by up to 15% as the electron beam diameter was changed; this effect was due to the earth's magnetic field. The use of computers for data acquisition and treatment has been discussed by Strausser (*B81*) and Baun (*B82*). Barthes and Rhead (*B83*) have proposed that diffraction effects could be important for the poor reproducibility that was observed in the case of Pb overlayers on Au single crystals.

Specific AES Analysis

Quantitative. A wide variety of specific systems have been studied by AES in which procedures are given that could be extended to other materials. Kelemen and Wachs (*B84*) have observed that half a monolayer of carbon on a Ag single crystal did not change markedly the ratio of the 266 to 304 eV peaks of the Ag derivative AES spectra. This problem was solved by subtracting the direct energy distribution curves for a surface with a carbon overlayer from a clean Ag surface. The differences in the S AES line shapes for sulfide and sulfates has been utilized by Turner et al, (*B61*) to determine the ratio of mixtures of compounds containing these anions. A simple addition of the pure compound line shapes (after corrections, for background and electron beam effects) was found to be sufficient to make quantitative analysis. The location of the interface in Cu_xS/CdS solar cells has been analyzed by Matysik and Ramos (*B85*) by the differences in the S LVV spectra of Cu_xS and CdS. Duc et al. (*B86*) have developed a quantitative method for the determination of the ratio of the depth of an overlayer to the mean free path for a homogeneous overlayer; a homogeneous rutile film was used for verification of the procedure. Tokutaka et al. (*B87*) have derived a method for a quantitative calibration curve for an Auger signal vs. film thickness for an overgrowth thin film by determining secondary electron coefficients. Thin films of Ag on Au and Be on Cu gave reasonable agreement. From changes in the Cu MVV line shape for a Cu epitaxial layer on NaCl, Namba et

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

al. (*B88*) have proposed that changes in surface topography and electronic structure could be observed. These workers (*B89*) also have used the same procedure for Pd layers on Cu. In this case both Cu and Pd MVV line shapes were employed. The M_1VV transitions for Cu and Ni (90–110 eV) have been employed by Goto et al. (*B90*) to determine alloy compositions with these metals. Computer simulations gave good agreement with experiment. Mitchell et al. (*B91*) have been able to achieve quantitative analysis to ±0.3 atomic % of iron oxides during sputtering. Better results were obtained when only the negative portion of the peak-to-peak signal was used. Paterson et al. (*B92*) have compared the peak-to-peak heights for various sputtered Fe–Cr and Fe–Ni–Cr alloys to the bulk chemical composition and have found good agreement between the analysis. The effect of backscattering, escape depth, and atomic density appeared to have varied little between the various samples. An iterative method to correct matrix effects has been developed and tested by Sekine and Mogami (*B93*) for alloys; reasonable agreement was achieved with comparison to electron beam microprobe analysis (EMPA). Jablonski (*B94*) has found that the thin model film for EMPA is equivalent to expressions for AES backscattering. Oxide films on Fe–Cr alloy surfaces have been investigated quantitatively by Ishiguro and Homma (*B95*); the effects of sputtering have been considered also. Hartsough et al. (*B96*) have observed large differences in depth analysis using AES and Rutherford backscattering; these results may be used to provide information about sputtering yields and quantitative AES analysis. Garrenstroom (*B97*) has developed a simple data matrix technique for depth profile analysis that relies upon Auger line shape differences of the materials under investigation. The method was applied to two different thin film systems. The variation of the peak-to-peak height of the derivative spectra vs. the total Auger current has been used by Rawlings et al. (*B98*) to monitor changes in the chemical state of NO adsorption on W. The sensitivity for detection of chemical changes was compared to XPS results. Several different analytical methods have been used by Buitrago (*B99*) to study Nb–Ge alloys. The best results were obtained by the use of elemental standards. Low-energy ion implantation with known doses of P into Si has been used for calibration purposes for AES analysis by Thomas (*B100*). In addition, sputter yield information was obtained.

Scanning Auger Analysis. Spatial resolution on the order of 50 nm combined with scanning capability is now available commercially with scanning Auger microprobe (SAM) equipment. This specialized use of AES has been reviewed by Johnson with an emphasis on metallographic applications (*B101*). Zalar and Hofmann (*B102*) have used SAM to analyze the crater edge profile from sputtering Ni–Cr multilayer sandwich films. Large lateral magnification factors ($>10^3$) were obtained under these conditions. Moulder et al. (*B103*) have demonstrated the capability of studying the elemental compositions of thick films (72 μm) with SAM. Care in sample preparation was emphasized by these workers for this application. A model to compute the spatial resolution of an electron beam due to interactions with the material under study has been developed by Durek (*B104*); the results agreed with experiment for Ti with a beam diameter of 0.1 μm and a beam energy of 10 and 50 keV. A computer program has been developed by Schaffer (*B105*) with FORTRAN programs to control a SAM. The use of SAM in practical problems has included stress corrosion cracking and the effect of heat treatment on solute concentration at grain boundaries of 7075 Al alloy (*B106, B107*) and grain boundary features in Fe and Ni alloys (*B108*). Os/Ru-coated and noncoated andoes (*B109*) and Au–Ni–Cu thin films used for electronic devices (*B110*) have been analyzed by SAM.

Gas–Solid Reactions. The review of gas–solid reactions has been treated in the previous review by Larabee and Shaftner (*B111*). However, several studies of gas–solid reactions have reported interesting effects with regards to AES analysis. For example, Reuter and Wittmaack (*B112*) have found an enhanced oxygen uptake on sputter-cleaned Si surface under electron or argon ion bombardment. A similar enhancement effect for CdTe and CdSe has been displayed by oxygen on surfaces exposed to an electron beam by Ebina et al. (*B113*). Lieske and Hezel (*B114*) have combined ion energy electron spectroscopy and AES to analyze the various types of Si bonds, i.e., Si=O, Si–O and Si–C that were observed during the oxidization of Si by O_2. These authors used changes in Si LVV Auger spectra with oxidization. Lang et al. (*B115*) also have employed this Auger transition in their study of Si oxidization. With the reaction of N ions and neutrals on Si, Delord et al. (*B116*) have observed changes in the Si LVV spectra, but none for the N KLL. This result suggests that the nitride formed on the surface has N in a single atomic environment. By measuring intensity ratios of various LMM transitions for several transition metal oxides and sulfides, Rao et al. (*B117*) have been able to take determinations of the number of valence electrons and the oxidization state of metal. Thus, in the case of oxidization studies, the O KLL intensities did not have to be considered. Dolle et al. (*B118*) have suggested that changes in the Cr Auger line shapes observed during the oxidization of Cr could be explained by an interatomic transition with the 2p levels of oxygen.

AES Analysis. AES has been utilized to investigate numerous systems of practical importance. Some examples are given in Table II.

Depth Profile Analysis. AES has found wide use as a method for depth profiling with numerous systems of commercial importance having been investigated. The topography of the surface under investigation has received attention recently. Makh et al. (*B119*) has made calculations on the artifacts that result with the sputtering of nonflat surfaces, e.g., wires, fibers, etc. The production of a lap taper to facilitate depth profiling has been considered by Lea and Seah (*B120*); in many instances such a procedure can reduce analysis time. Such factors as the material under study and the distance of an interface from the surface were considered. Kempf (*B121*) has developed an interference technique that is capable of depth resolution of <1 nm. The procedure is based upon measuring the phase difference between two laser beams; one beam is focused onto the area being sputtered and the other on an unsputtered portion of the sample. Guglielmacci and Gillet (*B122*) have developed relationships to understand the concentration gradient effect and to convert sputtering times to depths. Examples were given with Ag/Pt and Ag/Au epitaxy compounds. The Si–SiO_2 interface has been studied by Helms et al. (*B123*), in which models for escape depth and ion knock-on broading were developed. Microtopographical features for depth profiles of porous Al anodes have been investigated by Sun et al. (*B124*). Such factors as reduction of the Auger signal, changes in the depth scale, loss of depth resolution, and changes in surface topography had to be considered. Malherbe and Hofmann (*B125*) have studied the depth profiles of low energy N implants into Cr and compared these results to a theoretical model. Berneron et al. (*B126*) has compared depth profile results of passivated ferritic steels by AES, glow discharge spectroscopy, and the ion microprobe. The latter techniques were able to identify Mo in small amounts, whereas AES could not; the major elements were observed with all of the techniques. Listed in Table III is a limited number of examples of sputter profile studies.

Theory of the Auger Effect

Work has continued in an effort to gain a greater theoretical understanding of the Auger process. Such knowledge will increase the usefulness of Auger spectroscopy in routine analytical situations and also will lead to more information on chemical bonding. However, the problem of interpretation is not simple, since the Auger process is a two-electron process and no easy and clear set of selection rules exist. In addition, multiplet splitting further complicated the evaluation of experimental spectra. The next few years should show even greater understanding of the Auger process.

Feibelman (*B127*) has proposed that the rate of reneutralization of the two holes on a single atom is of the order of 10^{-13} to 10^{-14} s. This "bottleneck" can then account for the desorption of an ion from the surface. Schulman and Dow (*B128*) have used a mean-field model to compute the Auger line shape of Li. They have found that many body effects contribute to the line shape in addition to band effects as has been suggested by Jennison. Jennison et al. (*B129*) has analyzed the Auger line shape for Be and found that valence band screening effects can be seen in the observed spectra for this metal. The analysis of Auger line shapes for several simple hydrocarbons and Cu and Be has been made with some success by a simple one electron theory with uncorrelated final state hole motion by Jennison (*B130*). However, initial state

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

Table II. Examples of the Use of AES

system studied	ref
B-Si compounds	*a*
diamond surfaces	*b*
lubricant additives	*c*
Zeolites	*d*
vapor deposited SiO_x	*e*
Si oil contamination	*f*
glass surfaces	*g*
semiinsulating silicon films	*h*
dissolution of TiO_x	*i*
Ti alloy films	*j*
sputter-deposited Cr_2O_3 films	*k*
black chrome films	*l*
passivated Cr surfaces	*m*
Cu(I) and Cu(II) oxides	*n*
Ag-glass interfaces	*o*
Fe/Cr oxide solid solutions	*p*
Fe/Ni oxide films	*q*
Ni-Th alloy oxidization	*r*
steel grain boundaries	*s, t*
laser melted Cu alloys	*u*
ion implants of GaAs	*v*
Pd/Si interfaces	*w*
Pd-Au alloys	*x*
Ag/Pd/Ti solar cells	*y*

[a] G. U. Pignatel and G. Queirolo, *Thin Solid Films* **1980**, *67*, 233; *J. Electrochem. Soc.* **1979**, *126*, 1805. [b] S. V. Pepper, *Appl. Phys. Lett.* **1981**, *38*, 344. [c] H. Montes, A. Gauthier, G. Blane, and J. Brissof, *Vide, Couches Minces* **1980**, *201* (Suppl. Proc. Int. Vac. Cong., 8th V2) 475. [d] S. L. Suib, G. D. Stucky, and R. J. Blattner, *J. Catal.* **1980**, *65*, 174, 179. [e] K. Maki and Y. Shigeta, *Jpn. J. Appl. Phys.* **1981**, *20*, 1047. [f] C. A. Haque and A. K. Spiegler, *Appl. Surf. Sci.* **1980**, *4*, 214. [g] D. E. Clark and E. L. Yen-Bower, *Surf. Sci.* **1980**, *100*, 53. [h] T. Adachi and C. R. Helms, *J. Electrochem. Soc.* **1980**, *127*, 1617. [i] T. N. Wittberg, W. E. Moddeman, L. W. Collins, and P. S. Wang, *Vide, Couches Minces* **1980**, *201* (Suppl. Proc. Int. Vac Cong., 8th V2) 562. [j] W. L. Baun, *Surf. Technol.* **1980**, *11*, 421. [k] B. Bhushan, *Thin Solid Films* **1980**, *73*, 255. [l] C. M. Lampert, *Thin Solid Films* **1980**, *72*, 73. [m] M. Seo, R. Saito, and N. Sato, *J. Electrochem. Soc.* **1980**, *127*, 1909. [n] G. Benndorf, H. Caus, B. Egert, H. Seidel, and F. Thieme, *J. Electron Spectrosc. Relat. Phenom.* **1980**, *19*, 77. [o] R. Bastasz, *Solar Energy Mater.* **1980**, *3*, 169. [p] M. C. Kung and H. H. Kung, *Surf. Sci.* **1981**, *104*, 253. [q] T. N. Wittberg, J. R. Hoenigman, W. E. Moddeman, and R. L. Salerno, *Appl. Surf. Sci.* **1980**, *4*, 531. [r] C. Berry, D. Majumdar, and Y. W. Chung, *Surf. Sci.* **1980**, *94*, 293. [s] T. Ogura, A. Makino, and T. Masumoto, *Scr. Met.* **1980**, *14*, 887. [t] D. Roptin and M. Cailler, *Scr. Metal.* **1980**, *14*, 1139. [u] C. W. Draper, S. P. Sharma, J. L. Yeh, and S. L. Bernasek, *Surf. Interface Anal.* **1980**, *2*, 179. [v] Y. S. Park, W. M. Theis, and J. T. Grant, *Appl. Surf. Sci.* **1980**, *4*, 445. [w] G. W. Rubloff, P. S. Ho, J. F. Freeouf, and J. E. Lewis, *Phys. Rev. B* **1981**, *23*, 4183. [x] D. D. Eley and P. B. Moore, *J. Chem. Soc., Faraday Trans. 1* **1980**, *76*, 1388. [y] E. N. Sickafus, J. Tabock, J. L. Bomback, S. M. Lee, and M. S. Sundaram, *Thin Solid Films* **1981**, *78*, 49.

Table III. Examples of AES Profile Investigations

system studied	ref
$Si/SiO_2/Mo$	*a*
Si/SiO_2 reacted with H_2	*b*
passivated Inconel and Incoloy alloys	*c*
NiCr alloys	*d*
stainless steel oxides	*e*
Co alloy oxides	*f*
effects of sputtering on Cu-Ni and Ag-Au alloys	*g*
Cu-Ni alloys	*h*
ZnS reaction with H_2O	*i*
Cu_2S-CdS heterojunction	*j*
Pt-Si interface	*k*
Passive layers of tinplate	*l*

[a] A. J. Bevolo, G. J. Campisi, H. R. Shanks, and F. A. Schmidt, *J. Appl. Phys.* **1980**, *51*, 5390. [b] S. R. Jost and W. C. Johnson, *Appl. Phys. Lett.* **1980**, *36*, 446. [c] M. Seo and N. Sato, *Corrosion* **1980**, *36*, 334. [d] A. Zalar, *Vide, Couches. Minches* **1980**, *201* (Suppl. Proc. Int. Conf. Solid Surf., 4th and Europ. Conf. Surf. Sci., 3rd, V2) 1311. [e] D. R. Baer and M. D. Merz, *Metall. Trans.* **1980**, *11A*, 1973. [f] P.-E. Nilsson-Jatko and S.-E. Karlson, *Vide, Couches Minces* **1980**, *201* (Suppl. Proc. Int. Vac. Cong., 8th, V2), 527. [g] M. Yabumoto, H. Kakibayashi, M. Mohri, K. Watanabe, and T. Yamasina, *Thin Solid Films* **1979**, *63*, 263. [h] H. Shimizu, N. Koyama, and Y. Ishida, *Jpn. J. Appl. Phys.* **1980**, *19*, L671. [i] A. Okada and T. Oka, *J. Appl. Phys.* **1980**, *50*, 6934. [j] J. Morimoto, *Jpn. J. Appl. Phys.* **1980**, *19*, L296. [k] I. Abbati, L. Braicouich, B. DeMichelis, O. Bisi, and R. Rovetta, *Solid State Commun.* **1981**, *37*, 119. [l] A. P. Grande and J. S. Johannesson, *Vide, Couches Minces* **1980**, *201* (Suppl. Proc. Int. Vac. Cong., 8th, V2) 449.

relaxation and screening for metals have to be considered for detailed fits. Hartree–Fock–Slater transition state calculations by Sen (*B131*) of the Auger KLL transition energies were within a few electronvolts of experiment for Na and Mg. The breakdown of the one-electron model to describe the $M_{4,5}N_{2,3}N_{4,5}$ Auger transitions for Pd to Te has been considered by Ohno and Wendin (*B132*). A Coster–Kronig fluctuation was ascribed to the level shifts and broadening observed.

Thomas and Weightman (*B133*) have examined the ratio of the Coulomb repulsion to energy separation between single particle bonding and antibonding states for simple diatomic molecules. The effect of this ratio on the multiplet structure on small diatomics will be small, but for larger molecules the effect should be larger. Agren (*B134*) has used ab initio techniques to describe the molecular valence spectra of several fixed gases. The assignment of the various transitions was aided by the fact that the spectra could be divided into three separate regions; configuration interaction was found to be important in this analysis. Jennison (*B135*) has concluded that inclusion of initial state core hole projected on the final state reduces the error in peak intensities from up to 70 to an average of 20% in the calculation of the KLL Auger spectra of NH_3. Jennison et al. (*B136*) have concluded that configuration interaction is important to compute the Auger spectra of unsaturated polar molecules while one electron results can agree with experiment for saturated and nonpolar unsaturated molecules. Kosugi et al. (*B137*) have developed a simple empirical approach to describe the Auger spectra of simple gas molecules that is based upon single ionization potentials. However, the testing of the procedure involved transitions in which the two holes of the final state are different. Dunlap et al. (*B138*) has tested the possibility of using ab initio $X\alpha$ type calculations to analyze Auger spectra. Qualitative agreement was obtained for O_2, and suggestions for improving agreement between experiment and theory were made. Kualheim and Faegri (*B139*) have concluded that correlation mixing must be included to adequately describe the experimental spectra of HF; it was not as important for Ne.

Ramaker (*B140*) has investigated final state correlation effects on the Auger O KLL and Si $L_{2,3}VV$ spectra in SiO_2. The O KLL spectra suggested strong localization around a single SiO_2 cluster, while the Si L_3VV spectra had both local and nonlocal contributions. Also obtained were good values for hole–hole repulsion and bandwidths. Dunlap et al. (*B141*) has extended the procedure to analyze the N KVV spectra of $NaNO_3$ and the S $L_{2,3}VV$ spectra of Li_2SO_4. In these cases also, there is an intermediate level of localization, along with some shake-up features. Kunjunny and Ferry (*B142*) have used a semiempirical model to compute the Auger line shape of the Si/SiO_2.

Riddoch and Jaros (*B143*) have tried to use an effective mass model to determine the Auger cross section for Auger transitions that involved two deeply bound electrons; however, this approach was not very successful for GaP interface. Reasonable agreement between initial chemisorbed oxygen spectra and the theory was achieved. Aitken et al. (*B144*) has investigated initial and final state effects on electronegativity

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

for a series of Cl-containing compounds. Their results obtained from Cl KLL and 2p ionization energies agree with experiment and simple theory substituent effects.

Combined XPS-AES Topics

The similarity between and the ability to do both XPS and AES in the many commercial systems has led to a combined approach in many studies. The combination of these techniques has enabled investigators to obtain much more information on a particular problem than could be provided by only one of these procedures. Also many of the problems are common to both of these techniques. Most of the papers in the individual sections strongly favored one procedure over the other when multiple procedures were used. Those listed in this section have a more balanced point of view. The topics covered will be roughly the same as those indicated for the individual techniques. The reader should examine those portions of interest (e.g., mean free paths, etc.) in all of the sections in order to have a full picture of the area.

The use of combined techniques has been reviewed by Shemenski (*B145*) for quality control purposes, environmental particle analyses by Natush (*B146*), surface quantification by Powell (*B147*), polymer technology by Holm and Shorp (*B148*), and glass technology by Fox (*B149*). Chang (*B150*) has compared the techniques with regards to sensitivity, speed, spatial and depth resolution, chemical bonding information, sample damage, quantitative analysis, and charging problems. He has concluded at present that AES scores higher than XPS, but if theoretical limits could be achieved, XPS would be superior. Any specific problem, of course, would have its own score card. Powell (*B151*) has reviewed the progress of the ASTM E-42 committee on Surface Analysis.

Depth Profiles. Quantitative depth profiling has been reviewed by Hofmann (*B152*) and by Mathieu and Landot (*B153*). Seah et al. (*B154*) has concluded for depth profiles that statistical contributions are small when compared to atomic mixing, diffusion and instrument effects. Substrate roughness, incident ion beam angle, and sputter yield are important factors also in depth profile analysis (*B155*, *B156*). Several procedures to minimize these effects have been suggested by Holloway and Bhattacharya (*B157*). Examples with reactive and multiple ion beams, sample rotation and special mounting, and varying the incidence angle and ion energy were given. Preferential sputtering with argon of oxygen for niobium oxides has been observed by Karulkar (*B158*). Also, lower ion energy appeared to result in greater reduction of the oxides.

Electron Mean Free Path. The energy dependence of the inelastic electron mean free path plays an important part in quantitative analysis with XPS and AES. There have been different values proposed for this energy dependence, $\lambda \propto E^n$, λ is the inelastic mean free path (IMFP), E is the electron kinetic energy, and $n \sim 0.5$–1. Szajman et al. (*B159*) have derived an analytical expression to compute electron mean free paths in free electron metals and by extension to semiconductors, insulators, and nonfree electronlike metals. Good agreement with experimental results was reported for a number of materials at energies greater than 200 eV. This group then has proposed that the electron mean free path has the form $AE^{0.75}$, where A is a constant dependent upon the material for energies above 200 eV (*B160*). The mean free paths ranged from about 10 to 30 A for energies of 200–1500 eV. Seah and Dench (*B161*) have examined IMFP's from a large number of materials. Their analysis indicated that there were three separate classes of materials: elements, inorganic, and organic compounds. All of the materials followed a general equation of the form $\lambda = (A/E^2) + BE^{1/2}$, where A and B are constants (B is proportional to atomic size for the elements and inorganic compounds). Wagner et al. (*B162*) have suggested that the energy dependence at energies above 100 eV should more closely follow a power dependency of 0.65–0.75. For Ge, Gant and Monen (*B163*) have concluded that the power dependency is about 0.77 from 30 to 1250 eV. However, the comparisons with previous investigations was poor.

Satellite Structure. Correlations in the structure of XPS and features in Auger spectra have been observed in several studies. Martensson et al. (*B165*) have detected that the energy shift in the Pd $3d_{3/2}$ satellite is nearly the same as that for the main Auger peaks with Cu Pd alloys. The main 3d line did not shift by the same amount and this leads to the conclusion that single and double holes may undergo different chemical shifts. Van der Laan et al. (*B166*) have analyzed the XPS and AES spectra of copper dihalides and have shown that the satellite structure of the 3d lines comes from the same final state as the main LMV Auger lines. Also from these spectra, the valence band could be assigned and the type of bonding could be suggested. The relative shifts between the Pt $4f_{5/2}$ XPS and NNN AES and the O KLL lines for different thickness of a Pt layer on $SiTiO_3$ have been used by Bahl and co-workers (*B167*) to determine changes in the total relaxation energy. Girvin and Penn (*B168*) have investigated satellites in filled d state systems from the initial excitation to final deexcitation for both photoemission and photoinduced Auger transitions.

Examples. In Table IV are examples where AES and XPS have been used mutually for analysis for a wide variety of materials of a basic or practical importance.

SECONDARY ION MASS SPECTROMETRY (SIMS)

Introduction

Bombarding a solid surface with energetic (low kiloelectronvolt) ions or neutrals results in the emission of secondary particles, namely, positive and negative ions, neutrals, electrons, and photons. This phenomenon known as sputtering is dependent on several important parameters such as the energy, mass, and angle of the incident beam and the mass, structure, and binding energy of the atoms which form the surface of the target. Mass analysis of the sputtered secondary ions forms the basis of secondary ion mass spectrometry (SIMS).

As a surface analytical tool, SIMS has several distinct advantages over X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) in that SIMS is sensitive to all elements and isotopes of the periodic table, whereas XPS and AES cannot detect H or He, and SIMS has a lower detection limit $\sim 10^{-5}$ atomic % compared to 0.1 atomic % and 1.0 % for AES and XPS, respectively. SIMS, however, has several disadvantages in that its elemental sensitivity varies over 5 orders of magnitude and differs for a given element in different sample matrices, i.e., SIMS demonstrates a strong matrix effect. This matrix effect makes SIMS measurements difficult to quantify. Recent progress in developing methods for quantitative SIMS, however, is significant and will be discussed later in this review.

SIMS methodology has evolved along two distinct lines. The first and original method showed SIMS as an analytical tool for in-depth and micro/trace analysis. These instruments require microscope or microprobe capabilities that are ideally suited for depth profiling, ion imaging, or trace elemental analysis. The SIMS method is commonly referred to as "dynamic SIMS" since it uses high primary ion beam current densities (>1 $\mu A/cm^2$) to generate sample sputter rates >50 Å/min. The high sputter rates, in effect, lower the detection limit of the method. The dynamic SIMS method is applied primarily to studies in electronics technology and material science.

The second SIMS method was pioneered by A. Benninghoven (University of Munster, West Germany) in the late 1960s and is capable of analyzing monolayer quantities of materials on surfaces. In order to detect surface monolayers, one must first lower the sample sputter rate by lowering the current density of the primary ion beam. Then, in order to compensate for the corresponding loss in signal intensity (due to the lower current densities), the analysis area is increased by broadening or rastering the primary ion beam. The SIMS method known as "static" or low damage SIMS is applied to the study of gas–surface interactions and, most recently, the ionization of nonvolatile and thermally labile molecules. New results even include the study of biological and polymeric materials. Since most of these latter studies deal with the emission of polyatomic or molecular ions from the surface, the name "molecular SIMS" has been used.

The application of SIMS as a sensitive ionization source for nonvolatile and thermally labile molecules compares favorably with other ionization methods in mass spectrometry, such as field desorption (FD), californium-252 plasma desorption (PD), and laser desorption (LD), or laser microprobe mass analysis (LAMMA) in that the molecules not only survive the energetic excitation process but commonly appear

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Table IV. Example of AES and XPS Combined Analysis

system studied	major emphasis	ref
Mg_2Cu, Mg_2Ni, and Mg_2Ni-Mg	surface vs. bulk composition of hydrogen storage material	*a*
alkali earth metals and oxides	solid-state reaction products	*b*
XeF_2 and SiF_4 on Si	formation of surface species	*c*
$Si_{1-x}C_x$:H films	polymer structure	*d*
mirror surfaces	results of environmental exposure	*e*
sulfur compounds	reference spectra	*f*
TiH_x and $TiD_{0.9}$	changes in spectra with composition	*g*
black chrome and cobalt	composition and chemistry of solar absorbers	*h*
Fe-Ni-Cr alloys	oxide film formation	*i*
iron oxides	formation of oxides under controlled conditions	*j*
ion implanted stainless steel	chemical state of implanted species	*k*
stainless steels	surface composition and adhesive bonding	*l*
NiSi and Pd_3Si	phase characterization	*m*
Ni-Cu alloy	composition profile	*n*
Ni/Al catalysts	surface compositions	*o*
Cu-Be	surface composition vs. secondary electron yield	*p*
Zn_3P_2 solar cells	surface composition and depth analysis	*q*
GaAs	oxide layers composition	*r*
GaAs	Schottky barrier interfaces	*s*
Pd-Au alloys	interactions of hydrogen and oxygen	*t*
Pd layers on Si	composition of interface region	*u*
CdTe and InP	interfaces with metal overlayers	*v*
oxides on InP	composition and depth profile	*w*
antimony oxides	composition	*x*
$Hg_{1-x}CdTe$	oxide and interface properties	*y*
Au particles	changes in atomic environment with particle size	*z*
Pu compounds	correlation with phase diagram and reactions	*aa*

[a] Th. von Waldkirch, A. Sieler, P. Zurcher, and H. J. Mathieu, *Mat. Res. Bull.* **1980**, *15*, 353; A. Seiler, L. Schlapbach, Th. von Waldkirch, D. Shaltiel, and F. Stucki, *J. Less-Common Metals* **1980**, *73*, 193. [b] J. Verhoeven and H. van Doveren, *Thin Solid Films* **1981**, *77*, 367. [c] T. J. Chuang, *J. Appl. Phys.* **1980**, *51*, 2614. [d] W.-Y. Lee, *J. Appl. Phys.* **1980**, *51*, 3365. [e] L. R. Pederson and M. T. Thomas, *Sol. Ener. Mater.* **1980**, *3*, 151. [f] D. Lichtman, J. H. Craig, Jr., V. Sailer, and M. Drinkwine, *Appl. Surf. Sci.* **1981**, *7*, 325. [g] B. C. Lamartine, T. W. Haas, and J. S. Solomon, *Appl. Surf. Sci.* **1980**, *4*, 537. [h] G. Zajac, G. B. Smith, and A. Ignatien, *J. Appl. Phys.* **1980**, *51*, 5544; G. B. Smith, A. Ignatien, and G. Zajac, *J. Appl. Phys.* **1980**, *51*, 4186. [i] G. R. Connor In "Applied Surface Analysis, ASTM STP 699"; T. L. Barr and L. E. Davis, Eds.; American Society for Testing and Materials: Philadelphia, PA, 1980; p 54. [j] D. A. Stout, G. Gawell, J. B. Lumsden, and R. W. Stachle In "Applied Surface Analysis, ASTM STP 699"; T. L. Barr and L. E. Davis, Eds.; American Society for Testing and Materials: Philadelphia, PA, 1980; p 42. [k] I. L. Singer and J. S. Murday, *J. Vac. Sci. Technol.* **1980**, *17*, 327. [l] M. Gettings and A. J. Kinloch, *Surf. Interface Anal.* **1979**, *1*, 165. [m] P. R. Webber, C. E. Rojas, P. J. Dobson, and D. Chadwick, *Surf. Sci.* **1981**, *105*, 20. [n] V. Atzrodt, Th. Wirth, and H. Lange, *Phys. Stat. Sol.* (*a*) **1980**, *62*, 531. [o] J. C. Klein and D. M. Hercules, *Anal. Chem.* **1981**, *53*, 754. [p] Y. Fujii, F. Kanematsu, T. Koshikawa, and E. Sugata, *J. Vac. Sci. Technol.* **1980**, *17*, 1221. [q] L. L. Kazmerski, P. J. Ireland, and A. Catalano, *J. Vac. Sci. Technol.* **1981**, *18*, 368. [r] L. L. Kazmerski, P. J. Ireland, S. S. Chu, and Y. T. Lee, *J. Vac. Sci. Technol.* **1980**, *17*, 521. [s] L. L. Kazmerski and P. J. Ireland, *Surf. Interface Anal.* **1979**, *1*, 144. [t] L. Hilaire, P. Legare, Y. Holl, and G. Maire, *Surf. Sci.* **1981**, *103*, 125. [u] J. H. Thomas III, and D. E. Carlson, *J. Electrochem. Soc.* **1981**, *128*, 415. [v] T. P. Humphreys, M. H. Patterson, and R. H. Williams, *J. Vac. Sci. Technol.* **1980**, *17*, 886. [w] L. L. Kazmerski, P. J. Ireland, P. Sheldon, T. L. Chu, S. S. Chu, and C. L. Lin, *J. Vac. Sci. Technol.* **1980**, *17*, 1061. [x] F. Garbassi, *Surf. Interface Anal.* **1980**, *2*, 165. [y] T. S. Sun, S. P. Buchner, and N. E. Byer, *J. Vac. Sci. Technol.* **1980**, *17*, 1067. [z] L. Oberli, R. Monot, H. J. Mathieu, D. Landot, and J. Buttet, *Surf. Sci.* **1981**, *106*, 301. [aa] D. T. Larson, *J. Vac. Sci. Technol.* **1980**, *17*, 55; D. T. Larson and J. M. Haschke, *Inorg. Chem.* **1981**, *20*, 1945.

as protonated, cationized, and deprotonated molecular-like ions.

Fast atom bombardment (FAB) and other neutral beam sources are also being used in SIMS. The FAB technique, in particular, has made considerable progress in the analysis of biological/macromolecules. Recent developments in FAB are therefore included in this review.

This fundamental review of SIMS (and related techniques) is divided into four sections. The first section reviews the theoretical aspects of sputtering and the mechanisms for secondary ion formation and emission. The second section reviews various instrumental developments. The third section discusses the methods used in quantitative SIMS analysis including depth profiling. The last section deals with the formation and emission of polyatomic and molecular ions in SIMS and includes a discussion on analyzing nonvolatile and thermally labile molecules.

Books, Conferences, Reviews. A source book on SIMS is not yet available. SIMS, however, has been the subject of several book chapters, review articles, symposia, international conferences and workshops during this review period. Werner (*C1*) discussed "New Developments in Secondary Ion Mass Spectrometry" as part of a symposium on Applied Surface Analysis at the 29th Pittsburgh Conference on Analytical Chemistry in 1978. The paper (published in 1979) includes a discussion of in-depth profiling, depth resolution, high mass resolution, qualitative/quantitative analyses, detection limits, element mapping, nonplanar surfaces, and quadrupole/magnetic mass spectrometers in SIMS.

Two international SIMS conferences (SIMS II and III) were held. SIMS II convened at Stanford University on August 27–31, 1979. The complete proceedings of the conference contain over 80 invited and contributed papers on fundamental aspects (13 papers), quantitation (10 papers), semiconductors (11 papers), static SIMS (8 papers), metallurgy (8 papers), instrumentation (13 papers), geology (5 papers), biology (7 papers), combined techniques (8 papers), and three postdeadline papers (*C2*). The proceedings present an excellent survey of SIMS.

The Third International SIMS Conference was held in Budapest, Hungary, on 30 August–5 September 1981. The proceedings of this conference are not yet published.

Several pointed-workshops were also held during this review period. One workshop (*C3*, *C4*) dealt with "Ion Formation from Organic Solids" and compared spectral similarities between SIMS, field desorption, plasma desorption and laser desorption mass spectrometry. The proceedings of this workshop will be published in early 1982 by Springer.

The Middle Atlantic Mass Spectrometry Laboratory sponsored two symposia on fast atom and ion-induced mass

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POSCO Future M EX1020

Table V. Recent Review Articles on SIMS and Related Topics

	ref
ion beam methods	C9
SIMS principles	
fundamentals	C10-C13
quantitative analysis/depth profiling	C14-C18
instrumentation	C19-C26
molecular or polyatomic ions	C27-C29
SIMS bibliography	C30, C31
SIMS applications	
surface analysis	C32-C37
surface reactions	C38-C40
surface structure	C38, C41
biological/biomedical	C42-C45
corrosion science	C46
earth/planetary science	C47
environmental	C48, C49
metallurgy/materials science	C50-C52
polymers	C53, C54
semiconductors/electron devices	C16, C55, C56
related techniques	
(for nonvolatile/thermally labile solids)	
field ionization (FI), field desorption (FD), and in-beam techniques	C57, C58
fast-atom bombardment (FAB)	C59
laser desorption (LD)/LAMMA	C57, C60-C62
plasma desorption (PD)	C63, C64
mass spectrometry	
30 years of mass spectrometry	C65
ion chemistry	C66
ion energetics	C67
organic mass spectrometry	C68, C69
high-temperature mass spectrometry	C70
mass spectrometry/mass spectrometry	C71-C79

spectrometry of nonvolatile organic solids. One of the meetings is discussed in a recent article (*C5*).

The Annual Conference of the American Society for Mass Spectrometry (ASMS) presents a vareity of papers on SIMS, FDMS, LDMS, and PDMS (*C6*). The 29th Annual Conference held in Minneapolis on 24–29 May 1981 conducted a special FAB symposium in which over 15 FAB papers were presented.

The Fourth International Conference on Ion Beam Analysis was held in Aarhus, Denmark, on June 25–29, 1979. The proceedings of the conference published in early 1980 (*C7*) contain most of the 126 contributed papers. The major topics are stopping power and straggling (16 papers), cross sections for ion-beam analysis (13 papers), methods and apparatus (23 papers), radiation damage, defects, and diffusion (13 papers), sputtering profiling and SIMS (17 papers), applications to arts and archaeology (6 papers), new applications of ion-beam analysis (24 papers), and surface studies (14 papers). Some of the papers pertaining to depth profiling and SIMS will be discussed later in this review.

The Eighth International Mass Spectrometry Conference was held in Oslo, Norway, in August 1979. The proceedings of the conference published in 1980 (*C8*) fill two 3 in. thick volumes with 250 papers on state-of-the-art mass spectrometry. The volumes contain a short section (10 papers) on SIMS and an excellent section (20 papers) on the ionization of nonvolatile compounds. Many of these papers are discussed individually throughout this review.

A large number of review articles on SIMS appeared in 1979–81. Table V lists the recently published review articles on SIMS and related topics. The articles that are particularly noteworthy include two SIMS bibliographies written by Yin (*C31*) covering the period 1958–1975 and by Baun (*C30*), an extensive review article by Conzemius (*C60*) with 462 references covering the applications (to solids) of the laser ion source in mass spectrometry, and an excellent article by Werner (*C32*) on "Modern Methods for Thin Film and Surface Analyses" comparing the analytical features of laser optical emission spectroscopy, XPS, AES, electron microprobe analysis, scanning and transmission electron microscopy, low and high energy ion scattering spectroscopy, and SIMS.

Applications. Some recent applications of SIMS and related techniques are listed by category in Table VI. The individual references contain a few key words that describe the subject of each paper.

Table VI. Recent Applications of SIMS and Related Techniques

	ref
SIMS	
adsorption/oxidation/reaction	C80-C96
analysis - combined techniques (XPS, AES, etc.)	C97-C104
archaeology	C105, C106
biology/plants	C107
biomedical	C108-C112
corrosion	C113, C114
diffusion/glasses	C115-C127
electrochemistry	C128, C129
environmental	C130
geology	C131-C135
metallurgy/materials science	C136-C146
polymers	C147-C149
semiconductors/electronic devices	C150-C160
FAB, fast atom bombardment	C161-C167
LD/LAMMA, laser desorption/laser microprobe mass analysis	C168-C189
PD, californium-252 plasma desorption	C190-C194

Sputtering—Theory and Mechanism

Atomic and molecular particles are ejected from solid surfaces by the impact of energetic particles. The phenomenon known as sputtering is important in today's science and technology. Sputtering phenomena are fairly well understood for simple elemental targets but become much more complex for multicomponent or molecular systems. An understanding of the basic processes associated with the formation/emission of ions during sputtering is critical to the application of SIMS for the chemical analysis of surfaces. To what extent the ejected secondary ions represent the virgin surface is a primary concern in SIMS.

Constructing a comprehensive theory of the secondary ion emission process based on SIMS results alone will be very difficult without placing into proper perspective the interrelation of bombardment-induced ion, neutral, electron and photon emission. The ion emission process can be treated in two steps: collisional sputtering and particle ionization.

Collisional Sputtering. Most of today's knowledge about the experimental and theoretical aspects of sputtering has been recently collected and summarized. One source consists of the published proceedings of the Second International Workshop on Inelastic Ion-Surface Collisions held at McMaster University (Hamilton, Ontario) on 14–16 August 1978 (*C195*). The volume contains 28 papers consisting of review articles on radiation damage (*C196*), ion-induced electron emission (*C197*), photon-induced sputtering (*C198*), thermal effects in sputtering (*C199*), total current spectroscopy (*C200*), bombardment-induced light emission (*C201*), gas-phase collisions (*C202*), ionization processes in secondary ion emission (*C203*), energy dependence of the ionization probability (*C204*), electron and photon desorption mechanisms (*C205*), sputtered ion emission (*C206*), and other contributed papers.

The proceedings of the Third International Workshop on Inelastic Ion-Surface Collisions held at Feldkirchen-Westerham, Fed. Rep. of Germany, on September 17–19, 1980, contains 23 papers on the topics of electron emission, electron and photon impact, electron transfer, polarized light emission and excited particle emission during sputtering (*C207*). The volume contains 14 major review articles on the sputtering process including theoretical aspects of electron emission during ion bombardment (*C208*), ion-induced Auger electron emission (*C209*), interaction of metastable atoms with surfaces (*C210*), angle-resolved electron and photon stimulated desorption (*C211*), Auger-initiated desorption (*C212*), electron capture (*C213, C214*), electronic excitation via collisions (*C215*), charge fractions (*C216, C217*), polarized light emission (*C218*), outer-shell excitation (*C219, C220*), theory of charge states (*C221*), ion and excited state formation (*C222*), and velocity measurements (*C223*).

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POSCO Future M EX1020

The Eighth International Conference on Atomic Collisions in Solids was held in Hamilton, Canada, on 13–17 August 1979. The proceedings of the conference (*C224*) contain 105 papers: 7 papers on coherent radiation, 11 papers on molecular ion break-up, 10 papers on channeling and blocking, 6 papers on ranges, 8 papers on energy loss, 8 papers on projectile and target states, 10 papers on high density cascades, 8 papers on sputtering, 11 papers on radiation damage, 6 papers on trapping, adsorption, and emission, 7 papers on surface scattering, 6 papers on surface charge exchange, and 7 papers on electron and photon emission. Some of these papers will be discussed in detail in the following sections of this review.

The Ninth International Conference on Atomic Collisions in Solids was held in Lyon on July 6–10, 1981. The proceedings are to be published in *Nuclear Instruments and Methods*.

Springer-Verlag will publish three volumes in their Topics in Applied Physics series on "Sputtering by Particle Bombardment". The first volume (*C225*) deals with the physical basis for sputtering of single element solids. Chapter 1 by R. Behrisch gives a general overview and introduction to sputtering yields, calculations, and applications. Chapter 2 by P. Sigmund begins with a historical survey of the different sputtering theories and the classification of sputtering events as knock-on sputtering or elastic collisions and electronic excitations including chemical sputtering. The remainder of the chapter deals with the theory of atomic collision and penetration phenomena pertaining to knock-on sputtering and collision cascades. Results from elastic-collision theory and linear cascade theory are also presented as well as the angular and energy distributions of emitted particles. Sigmund also discusses related effects such as the excitation states of sputtered particles, ion-induced desorption, recoil implantation, ion-beam induced atomic mixing, and sputtering from multicomponent targets. Chapter 3 by M. T. Robinson deals with theoretical aspects of monocrystal sputtering including effects due or caused by the structure of surfaces, radiation damage, and surface binding energies. The ideas of crystal transparency and channeling aid in understanding the effects of orientation on the sputtering yields. A more comprehensive model of the sputtering process is based on computer simulations. Several examples are given that include displacement cascades in stable, metastable and quasistable dynamical modes and with the binary collision approximation. In Chapter 4, H. H. Andersen and H. L. Bay present an overview of all reported results about different methods to determine total and differential sputtering yields and their dependence on ion mass, energy, and angle of incidence and on target structure and temperature. The sputtering yield data are depicted on a set of graphs and some are compared to Sigmund's sputtering theory. In the fifth and final chapter, H. E. Roosendaal discusses sputtering yields of single crystals and the dependence on crystallographic orientation of the target relative to the incident beam direction. Particular attention is made of the channeling model.

The remaining two volumes are not yet published. The second volume will deal with the sputtering of multicomponent targets such as alloys and compounds, chemical sputtering, and sputtering by electrons and neutrals. The third volume will present information about the angular, energy, mass, and charge-state distribution of sputtered particles.

Other papers dealing with collisional aspects of sputtering include theoretical studies on the depth of origin of sputtered atoms (*C226*), atomic mixing by ion beams (*C227*), sputtering from elastic-collision spikes in heavy-ion-bombarded metals (*C228*), low yield sputtering by backscattered ions (*C229*), the emission of atoms and electrons from high density collision cascades in metals (*C230*), threshold studies of secondary electron emission induced by macro-ion impact of solid surfaces (*C231*), transport theory for kinetic emission of secondary electrons from solids by electron and ion bombardment (*C232*), the influence of adsorption energies on the ion-impact desorption of adsorbed layers on various metals (*C233*), and the influence of ion beams on the adsorption process (*C234*).

Several researchers have also measured the velocity and energy distribution of sputtered atoms and molecules. Husinsky, et al. (*C235*) using the laser-doppler method measured the velocity of sputtered particles. Their data showed the influence of slow thermal processes, thermal spikes, and collision cascades to the sputtering process. Gruen and co-workers (*C236*) used laser-induced fluorescence (LIF) spectroscopy to study the sputtering of uranium atoms. They showed that the LIF method could be used to obtain velocity distributions of sputtered neutral atoms, and possibly, of sputtered ions and excited atoms and ions. Snowdon and Macdonald (*C237*) measured the population distribution of the rotational and vibrational states of CH created by both gas phase collisions and sputtering of an adsorbed hydrocarbon layer. They found that the process leading to the ejection of the electronically excited molecule is governed by a direct projectile–adsorbate interaction but not by the conditions of the thermodynamic or random cascade models. (Taglauer et al. (*C233*) also describe ion-impact desorption as a direct projectile-adsorbate impact phenomenon.) Tsong and Yusuf (*C238*) measured the velocities of sputtered excited atoms and found that excited atoms are formed early in the sputtering process, i.e., after the first few collisions. Thompson and co-workers (*C239*) discuss the effect of ion mass and target temperature on the energy distribution of sputtered atoms.

Sputtering Simulation. Other research on sputtering that has received much attention during this review period is the computer simulation of the sputtering process by classical molecular dynamics models (*C240, C241*). These models are used to predict sputtering yield, energy and angular distributions of ejected particles, and mechanisms for cluster formation. Several other important results for the simulated sputtering of pure elemental (single crystal) targets show that cluster species form by recombination in a region above the surface (*C242*) and that their formation is strongly dependent on crystal orientation of the target and on the site geometry of atomic or molecular adsorbates (*C243–C245*). Another result indicates that molecular adsorbates such as CO and C_6H_6 are ejected intact from the surface and form clusters with substrate atoms above the surface (*C246, C247*).

Additionally, the angular distribution of higher energy particles (>20 eV) exhibit strong anisotropies (*C248, C249*) which can be used to determine the adsorbate positions (*C250–C252*). Recent theoretical studies of the angular distributions of atomic adsorbates include the effects of bonding site geometry (*C253*) and of interaction potential (*C254*). Harrison (*C255*) discusses the ion–atom potential function dependence of simulated sputtering and finds that a wide choice of potential functions will reproduce the experimental yields.

Garrison discusses the related theory of ion scattering from single crystals (*C256*).

One example of computer simulated sputtering from multicomponent systems is the molecular dynamics study of KCl. The study concluded that "the sputtered clusters are most likely to represent the undisturbed surface as the primary beam collides at smaller angles" (*C257*).

Ionization/Ion Emission Models. The molecular dynamics calculations do not currently consider the ionization probability for a given sputtered particle. In fact, a comprehensive ionization theory for sputtered particles does not exist although several ionization models for intrinsic (*C203*) and chemical (*C206*) ion emission have been proposed. One promising theory previously developed for ion scattering (*C258*) explores the probability for ion neutralization near the surface by Auger neutralization, resonant tunneling, and radiative or inelastic transition processes (*C259*). Murray and Rabalais have also revamped an ionization–neutralization mechanism for molecular ion emission (*C260*) where resonance and Auger charge-exchange transitions occur between the surface and the outgoing particle.

Winograd and co-workers (*C261*) have recently added an image force to their classical dynamics calculations for Ni atoms. Their calculations show excellent agreement with the experimentally measured angle and energy distribution of Ni^+ ions. Two important conclusions of the study are that the ionization probability is nearly isotropic and that it is weakly dependent on particle velocity.

The velocity or energy dependence of the ionization probability has also been considered by several other researchers. Lundquist found a simple power dependence on energy for the ionization probability for clean Cu, Ni, W, and Ti surfaces (*C262–C264*). Blaise and Nourtier (*C203*), and independently, Norskov and Lundquist (*C265*) calculated the ionization probability of secondary ions emitted during sputtering and found an exponential dependence on the normal component

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POSCO Future M EX1020

of the emission velocity. Sroubek, Zdansky, and Zavadil recently argued against such a velocity dependence (*C266*). Yu found experimentally, however, that the ionization probability P (of sputtered O^- from chemisorbed oxygen layers on V and Nb) was dependent on the normal component of the emission velocity, which suggests that the ionization process is an ion–surface interaction and not an ion–atom binary interaction (*C267*). Yu used the exponential dependence of P on the sample work function to measure the velocity dependence. The sample work function changes when alkali metals are deposited on the target surface (*C267–C270*).

In addition to the ionization probability, other atomic properties of the emitted ions such as their stability, energy, etc., must be predictable in order to model their formation and emission, particularly if the ions are polyatomic. Plog and Gerhard recently expanded the "valence model" used to calculate polyatomic ion yields from oxidized metal surfaces and metal oxides (*C271*). Ganjei et al. correlated negative secondary ion intensities with anion stoichiometry for a series of oxyanions following a modified-Poisson distribution model for dissociation of the parent anion (*C272, C273*).

Several other recent studies address various aspects of the ion formation/emission process. Wittmaack (*C274*) observed a direct correlation of the ionization probability of Si^{2+} with the Si $L_{2,3}VV$ ion-excited Auger electron emission. These results signify the importance of resonance ionization in SIMS. The fundamental importance of the Auger process is also observed in the mechanism for electron- and photon-induced desorption (*C275, C276*). Coles (*C277*) proposed a surface plasma model in which electron-impact excitation and ionization occur just above the surface producing secondary ion and photon emission; two related papers still question the existence of local thermal equilibrium (*C278, C279*).

Other studies used the emission of ions, photons, and/or electrons to indicate the intermediate states in a surface reaction (*C280*), to determine excited state populations and energy distributions of sputtered species (*C281–C288*), or to compare the absolute yields between excited neutrals and positive ions (*C289*).

Instrumentation

The basic components of a secondary ion mass spectrometer are a primary ion source, a target holder, and a mass spectrometer. The primary ion beam is produced typically from common or inert gases by a conventional ion gun. The ions are extracted from the (differentially pumped) source, focused, and accelerated to energies between 1 and 20 keV before they impact the target. The primary ions sometimes pass through a mass filter to eliminate any fragment or impurity ions that are present in the source. Methods to remove the neutral component of the primary ion beam are also used to improve the homogeneity of the ion beam. Rastering the ion beam increases the sampling area needed to increase the sensitivity in static SIMS or to produce flat-bottom craters for depth profiling studies. Once the ion beam strikes the target, secondary ions are ejected with discrete angular and energy distributions. These secondary ions are then collected (by appropriate ion optics), energy-filtered, and mass analyzed. The energy filters commonly used are simple deflection electrode, parallel plate, electrostatic mirror, cylindrical mirror, and grided retardation/acceleration analyzers. The mass analyzers include quadrupole, magnetic (single and double focusing), and time-of-flight analyzers. As the ions emerge from the mass analyzer, they impinge upon a collector producing secondary electrons. The secondary electrons are multiplied, counted, or measured by pulse-counting or analog devices and stored as a permanent record (on magnetic tape/disk or graph paper). Some instrumentation produces a visual image of the ion (secondary electrons) signal. The processing of the data reflects the needs of the researcher.

Instrumentation for field desorption (FD), laser desorption (LD)/LAMMA, and plasma desorption (PD) mass spectrometry differ in the way the secondary ions are produced. In FD, for example, the ions are emitted from a heated tip or needle placed in a high electric field. Recent developments in FD permit ions to be generated at relatively low energies and velocities so that quadrupole mass spectrometers can be used (*C290*). For LD/LAMMA, a continuous wave or pulsed laser irradiates the solid sample to produce the secondary ions (*C291*). PD mass spectrometry uses a californium-252 radioactive source whose MeV fission fragments desorb secondary ions from the surface of the target as the MeV ions pass through the target (*C292*). PD experiments usually use a time-of-flight mass spectrometer to analyze the secondary ions.

Ion Source. The flux density of the primary ions determines the conditions for "static" or "dynamic" SIMS. The ion current densities used in static SIMS are typically $<1 \times 10^{-8}$ A/cm^2 whereas in dynamic SIMS they are usually $>1 \times 10^{-6}$ A/cm^2. Since these and other ion beam factors such as size, energy, etc. usually require using different types of ion sources, most SIMS instruments are optimized for only one mode of operation. The recently developed pulsed ion source can, however, be used in static SIMS even though its current density is $>1 \times 10^{-6}$ A/cm^2 (*C293, C294*). Kloeppel and Seidel have also developed an extremely stable plasma ion source (*C295*) which produces current densities $\sim 10^2$ μA/cm^2 for ion energies between 0.9–6.0 keV. The plasma source also has a beam profile which is planar.

Bombarding a target with energetic ions causes secondary ions to be emitted. If the target material is nonconducting, its surface can accumulate an electrostatic charge as it neutralizes the incoming ions and emits secondary ions. Several methods have been developed to reduce or neutralize the surface charging. One method bombards the target with cesium ions (*C296*) and another floods the target surface with cesium atoms from an evaporator source (*C297*). Depositing cesium on the target surfaces changes their electronic properties by reducing the surface work function (while enhancing the yield of negative secondary ions). The cesium evaporator has an advantage over the cesium ion source since it can be used on instruments that already have conventional ion sources (*C297*).

Another method for analyzing nonconducting targets in SIMS is using neutral beams. One simple design of a neutral beam source uses a perforated metal plate at the end of a conventional ion gun (*C298*). The ions are neutralized by charge exchange reaction with the metal surface. Another neutral beam SIMS source neutralizes the ion beam by passing it through a high-pressure gas (*C299*).

Flooding the target surface with low energy electrons is also effective in neutralizing the surface charge (*C300*). A procedure for calibrating an electron flood source has been described (*C300*).

The purity of the ion beam can have a profound effect on the SIMS experiment and study. Wittmaack and Clegg (*C301*), for example, extended the dynamic range to 10^6 for their depth profiling instrument by eliminating the neutral components of their ion beam and by placing the target in ultra-high vacuum. Mass analyzing the primary ion beam is also advantageous (*C302*). One commercial ion source, the Colutron, uses a Wien-type linear mass filter. The Colutron ion sources have been used recently for depth profiling studies (*C302*) and to simulate ion impact desorption of impurities from Tokamak walls (*C303*).

Target. Several novel approaches for the handling and preparation of nonconventional samples have been reported. First, two groups have successfully developed a liquid chromatograph (LC)–SIMS interface using a moving metal ribbon (*C304, C305*). Second, a new electrospray system and procedure for the preparation of thin films of nonvolatile molecules for PD analyses has been reported (*C306*). This procedure can also be applied in LD and SIMS studies. Third, a commercial SIMS instrument was recently modified to handle highly radioactive specimens (*C307*). Last, a UV/O_3 method was developed for both surface cleaning and sample storage after cleaning by conventional techniques (*C308*).

Secondary Particles. Bombarding the solid surface with energetic particles causes secondary particles (electrons, ions, neutrals) and photons to be emitted. Since the neutrals outnumber the ions by $\sim$1000 to 1, several methods have been developed that postionize the neutrals prior to mass analysis. One method uses a microwave plasma (*C309*), while another uses a thermal ionization source (*C310*). The third method ionizes the sputtered neutrals by charge-exchange reaction with Ar ions from an ion source (*C311*). An electron bombardment-type ion source has been used to ionize evaporated particles (*C312*).

The emitted secondary particles also have discrete energy and angular distributions. Winograd and co-workers (*C313*) recently designed an energy- and angle-resolved SIMS in-

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

strument. The instrument incorporates a quadrupole mass filter mounted on a flange which can be rotated under UHV conditions in such a way as to vary the polar collection angle of secondary ions; rotating the sample crystal about an axis parallel to the primary ion beam varies the azimuthal angle.

Energy filters are usually used with quadrupole mass analyzers to filter out high-energy ions, neutral species, photons, etc. The 3M Co. recently patented a new prefilter-ionizer apparatus for SIMS use (*C314*). Energy filtering techniques are also used to suppress the intensities of molecular ions (in favor of atomic ions) in order to overcome mass spectral interferences (*C315*). Energy discrimination effects in SIMS are, however, noted and troublesome (*C316*).

In depth profiling the target sputter rate is usually determined by measuring the depth of the crater with a Talystep or interference light microscope. A new electrooptical technique is used for on-line measuring of the sputtered depth during SIMS and Auger electron spectroscopy (*C317*).

Instrumentation for ultra high sensitivity SIMS has been proposed where single atom detection is possible using dc accelerators (*C318*).

Mass Analyzer. The mass analyzers used in SIMS instruments include quadrupole, magnetic (single and double focusing), and time-of-flight (TOF) analyzers. The quadrupole mass filter (QMF) has several distinct advantages over the other analyzers because of its relatively low cost, its compactness, its rapid mass-scan rate, and its ability to operate at or near ground potential. In SIMS, the QMF has been marketed as an "add-on" capability to complement other surface analytical tools such as XPS and AES. Several combined UHV systems have been described (*C319–C322*). In addition, recent developments in QMF theory and design give a better understanding of ion transmission properties (*C323*) and of ways to collect ions from a large range of initial displacements and/or angles of emission (*C324*).

The quadrupole mass filters have, however, several problems; the most important ones being severe energy and mass discrimination effects. The magnetic analyzers (and particularly the double-focusing instruments), on the other hand, do not have these problems, and in addition, they have better ion transmission and mass resolution and a greater mass range. Recent use of magnetic analyzers in SIMS experiments has produced some astonishing results. Spectral mass resolution of 8000 was achieved by adapting a secondary ion source to a Varian 311A double-focusing mass spectrometer (*C325*). Several CEC-110 double-focusing (Mattauch-Herzog) mass spectrometers were converted to high-performance SIMS (*C326*) and mass-analyzed ion kinetic energy (MIKE) (*C327, C328*) instruments. The high-performance SIMS instrument demonstrated a mass range of 18 000 amu (*C329*). Other related (but non-SIMS) instrumental developments include the design of a double-focusing, static, axisymmetric mass spectrometer (*C330*) and of a high-performance instrument with mass resolution of 10 000–40 000 (*C331*). Several new scanning techniques for double-focusing mass spectrometers have also been developed (*C332*) and include a microprocessor controller for linked scans (*C333*), a novel mass marker for metastable ion scans (*C334*), and a rapid scanning magnet power supply (*C335*).

Time-of-flight (TOF) mass analyzers have also recently been used in SIMS (*C336*) and PD (*C337, C338*) experiments. Klots describes his TOF mass spectrometer as "a poor man's SIMS device" (*C339*). Traum and Woodruff describe TOF measurements with a CMA for simultaneous energy and mass determination of desorbed ions (*C340*).

The earlier ion microprobes or ion microscopes were designed from magnetic sector instruments. Recent design advances in dynamic SIMS instrumentation (*C341*) includes an ion microprobe mass analyzer of the Liebl-type (*C342*), a dissector ion microscope microanalyzer (*C343*), a direct-imaging scanning ion microprobe (*C344*), and a quadrupole-based scanning ion microprobe (*C345*). Levi-Setti and Fox (*C346*) give a detailed discussion of modern day high-resolution scanning ion probes. They examine the prospects for obtaining high resolution (<100 Å) with low-energy ion probes using either heavy ions for "destructive" imaging microanalysis or protons for "nondestructive" scanning transmission ion microscopy (STIM).

Various data acquisition processing systems for ion microscopes/microprobes have been described (*C347–C349*). A computer program for peak identification of elemental, molecular, and cluster ions in SIMS is also available (*C350*).

Quantitative Analysis

Since secondary ion yields for the elements vary over several orders of magnitude and change with matrix composition and since sputtering and instrumental artifacts also affect elemental sensitivities, SIMS measurements are difficult to quantify. Progress has been made, however, in developing methods for quantitative analysis that convert the measured secondary ion intensities to atomic concentrations. These methods for quantitative SIMS have been discussed recently in the excellent reviews by Wittmaack (*C351*) and by Werner (*C352*). The methods that we will review here use enhanced secondary ion yields, calibration curves and sensitivity factors, and glass, metal and ion-implanted standards to determine the true atomic concentrations of the sample. These methods are also applied to the quantitative analysis of depth profiles.

Sputtering Yields/Ion Yields. Sputtering yields in single-component systems are readily predicted and measured and are known to depend on the angle, energy, and mass of the primary ion beam and on the structure, orientation, and temperature of the target. Sputtering yields, nevertheless, do not usually vary by more than a factor of 10 for any element. Yield data for the pure elements were obtained recently using 500–1000 eV Ar^+ ions (*C353*). The data compare favorably with predicted values from Sigmund's theory.

A second study obtained sputtering yields for pure metals using 20-keV O_2^+ ions (*C354*). Since bombarding with O_2^+ ions causes oxygen to be implanted into the sample, sputtering yield varies with the concentration of oxygen. Warmoltz et al. (*C355*) derived the relation for the steady-state sputtering yield and the implanted oxygen surface concentration for different angles of incidence.

Other sputtering experiments include sputtering rates of minerals (*C356*), sputtering studies in Ar and O_2 glow discharges (*C357, C358*) and sputtering yield measurements of SiC by a volumetric method as a function of target temperature (*C359*). In the latter study, the yield dependence on temperature is attributed to different sputtering processes.

The secondary ion yields for pure elements vary by several orders of magnitude due mainly to differences in the atomic properties of the elements. While earlier studies correlated the positive and negative ion yields with the element's ionization potential and electron affinity, later studies showed ion yield enhancements which depend on the composition of the sample (or "matrix effect") and the sampling conditions of the experiment (*C352*). The presence of oxygen or cesium on the sample surface, for example, enhances the positive or negative ion yields, respectively.

In several recent studies, the yield enhancements are correlated with the sputtering yield of the matrix (*C360*), with the partial pressure of reactive gases like O_2 around the sample (*C361–C364*), with the concentration of surface impurities such as H, N, O, Cs, etc. (*C365, C366*), and with the use of reactive ion beams like O_2^+ and Cs^+ (*C367, C368*). In particular, Katz et al. (*C360*) found a linear correlation between sample sputtering yield and average sample mass. With this relationship the authors could predict ion yields and detection limits of related compounds. In another study, Deline (*C367*) found the near-surface concentration of the ion yield-enhancing species to be inversely proportional to the sample sputtering yield. However, whether "a unified explanation for secondary ion yields" exists or not remains debatable (*C369–C372*).

Rudat and Morrison (*C363, C364*) studied the effects of oxygen and nitrogen adsorption on secondary ion energy spectra obtained with reactive ion beams. They conclude that although the energy spectra resemble those obtained from the sputtering of pure elements, a better correlation between different matrices is possible when sputtering in the presence of a high partial pressure of O_2 (*C364*). They also observe abrupt changes in the energy spectra for Ni, Co, and W at high O coverage, apparently due to the incorporation of O into the surface lattice (*C364*).

Still other "matrix effect" studies try to decouple the sputtering and ionization processes by comparing changes in the ion versus neutral yields. Oechsner and co-workers (*C373–C375*) measured the changes in the ion yields (or ionization efficiencies) as a function of surface oxygen concentration by comparing ion vs. neutral yields.

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

For the analysis of multicomponent systems, Yu and Reuter caution that one component of a binary alloy bombarded with an O_2^+ ion beam (or an Ar^+ ion beam with adsorbed O_2) may be preferentially enhanced while the other component is suppressed (*C368*, *C376–C379*). Likewise, Modler and Beske (*C380*) found no simple linear relation between the secondary ion intensity and element concentration for CuNi alloys bombarded by O_2^+ ions. In another study, CuNi alloys bombarded with Ar^+ ions give ion yield ratios (Cu^+/Ni^+) that are unchanged from that of the pure metals (*C381*).

A model of preferred sputtering has been applied to the sputtering of binary alloys and compounds (*C382*). Betz in an excellent article on alloy sputtering (*C383*) found preferential sputtering for alloys and intermetallic compounds to be the rule.

Slodzian and co-workers (*C384*) studied the films formed on Fe, Ni, and Cr under ion bombardment in the presence of oxygen. They showed that the surface of each sample is covered with an oxide film whose composition is the same as that developed on Fe_2O_3, NiO, and Cr_2O_3, respectively, when sputtered under the same experimental conditions.

Empirical Methods/Physical Models. Several methods are used to convert measured secondary ion intensities to atomic concentrations. These methods can be grouped into two major categories: methods using various physical properties (i.e., ionization potential, binding energy, etc.) of the sample, physical models, and methods using calibration samples, empirical methods. Although many of the physical models such as the kinetic model, the autoionization model, the surface effects models, and the thermodynamic models (*C352*) were developed earlier (and continue to be applied), the empirical methods consistently give the best results. The empirical methods use calibration standards which are typically made from glass or iron alloys that are chemically doped with elements of known concentration. (NBS supplies a number of these characterized standards.) In quantitative SIMS, relative sensitivity factors for given elements in the standard are determined by comparing the secondary ion intensities for the given and matrix elements with their known concentrations. The elemental sensitivity factors can then be used to determine the unknown concentrations of given elements in samples that have the same type of matrix. Two recent experiments using sensitivity factors to study borosilicate glasses have been discussed (*C385*, *C386*). Sensitivity factors and practical ion yields are given for Mg, Si, Mn, Fe, and Cu impurities in Al (*C387*).

The relative sensitivity factor for a given element can also vary with the surface properties of the sample, particularly, if the surface is exposed to or bombarded with oxygen (*C351*, *C352*). Therefore, a new method was introduced recently which uses changes in the matrix ion species as an internal indicator for indexing the matrix surface properties (*C352*, *C388–C390*). The method is known as MISR or the matrix ion species ratio method. Using the indexed relative sensitivity factors has greatly improved the precision of the analysis (*C352*). Scilla et al. (*C391*) recently used the MISR method to correct for matrix effects in the SIMS analysis of high arsenic (>5%)/selenium alloys.

Using calibration standards poses several problems in quantitative SIMS. One problem associated particularly with iron alloys or steel standards is the homogeneity of the standard. A recent SIMS study concluded that the NBS steel standards 461-468 and 662-664 are unsuitable as standards in SIMS because all of the elements are not homogeneous within the sampling area of 100 Å (*C392*, *C393*). Therefore, researchers must be careful in both their choice of standard and what elements in each standard are suitable calibrants.

One way to overcome the sample homogeneity problem has been to develop standards by ion implantation (*C394–C399*). Here, the concentration and distribution of the dopants can be controlled more accurately, thereby forming standards with better homogeneity. The results with semiconductors, however, have been much more reliable than with ion-implanted metal standards (*C397*).

Leta and Morrison (*C397*) have described a new empirical method for quantitative SIMS analysis. They use the method of solid-state addition in which they implant their samples with a known concentration of the element of interest. Since the depth profile of the implanted species has a characteristic Gaussian shape, it is easily distinguished from the element originally present in the sample. Therefore, the known concentration of the implanted element is used as an internal standard to determine the concentration of the unknown. Bertrand et al. (*C400*) have used the standard addition method to detect Pb in lubricating oils.

Another problem in quantitative SIMS is caused by variations of the instruments. To evaluate these instrumental factors, Newbury (*C401*, *C402*) conducted comparative SIMS studies of selected glasses and steels with laboratories in the U.S., Japan, and Europe. He had each laboratory calculate relative sensitivity factors for several elements under a variety of experimental conditions. The results were astonishing and showed that a given relative sensitivity factor varied from 5 to 60. He also compared the measured concentrations with predicted values from physical models (*C401*, *C402*).

As mentioned earlier, physical models such as the kinetic model, the autoionization model, the surface effects models, and the thermodynamic models (*C352*) are also used in quantitative SIMS analysis. One thermodynamic model, the local thermal equilibrium (LTE) model, has become very controversial because the values for the temperature and electron density of the assumed plasma (used to solve the Saha-Eggert equation) are unrealistic and nonphysical. The model nevertheless continues to be used in SIMS and for some cases the analysis can be quite good. It is recommended, however, that the model be tested against suitable calibration standards prior to analyzing unknowns (*C392*, *C401–C403*).

Other researchers have modified the LTE model producing simplified versions (*C352*, *C404*). Morgan (*C405*, *C406*) recently described a versatile one-fitting-parameter LTE model with accuracies between factors of 2 and 3. The method is general and not restricted to certain elements in a certain matrix. Several researchers have applied this model to the study of doped GaAs (*C407*, *C408*).

The newest thermodynamic model, the local thermal nonequilibrium (LTNE) model, was applied recently to study both pure surfaces and those covered by oxides and other reactive layers (*C409*).

Depth Profiling/Microanalysis. As the sputtering process in SIMS removes successive atomic layers from the solid, the in-depth composition and distribution of elements can be determined by first recording the secondary ion intensity for each element as a function of sputtering time, thus producing a sputtering profile. Only after the ion intensities are converted to their respective atomic concentrations by the quantitative algorithms discussed above and after the time axis is converted to a depth (distance) scale, does the plot become the true depth profile.

The principles of quantitative depth profiling are given in the excellent review by Hofmann (*C410*). His review is divided into three parts. The first part covers the calibration of the depth scale and ion intensity. The second part discusses the problems associated with changes in the surface composition and topography due to sputtering, as well as their influence on depth resolution. The last part summarizes the basic requirements for optimum sputter profiling.

In depth profiling, the depth resolution of the sputtering experiment determines the quality of the profile. Hofmann (*C410*) identifies seven factors that contribute to the depth resolution, and other researchers have explored in detail ways to minimize their effects (*C411*). The first factor deals with instrumental effects that require keeping the ambient pressure of reactive gases below 10^{-8} torr (*C412*, *C413*) and maintaining the homogeneity of the primary ion beam. The primary ion beam should, for example, be uniform, have a constant ion flux (*C414*), and be rastered across the sample surface (*C415*) in order to have well-formed craters. (Tsong, et al. (*C415*) recently developed an approach to correct for crater-edge effects thereby eliminating the need to know the ion beam intensity distribution.) The ion beam should also be composed of low energy (~1 keV) ions (*C416*), preferably reactive (*C351*, *C417*), and be free of all impurities including neutrals (*C413*). Finally, the ion beam should have a glancing angle of incidence. Even though all of these beam conditions are met, surface shapes can form on the target surface during sputtering (*C418*, *C419*) and are dependent on the angle of incidence of the ion beam (*C420*, *C421*). These shapes can be suppressed, however, by using two ion guns incident at different angles (*C422*).

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

The second factor deals with initial surface roughness. A recent study found that substrate roughness contributes to the loss in depth resolution due to the orientation of the microplanes making up the rough surface (*C423*). In another study, researchers found increased surface roughening of Cu bombarded by reactive or noble ions due to "lattice-orientation-dependent sputtering" (*C420*) and to reorientation effects (*C424*).

The third factor called statistical surface erosion deals with the original sequential layer sputtering (SLS) model used to predict profile broadening associated with the statistical nature of sputtering. The most recent study incorporates site-dependent sputtering rates into the original SLS model, changing the depth resolution from a square root dependence to one that is constant and of the order of three monolayers (*C425*). Thus, the statistical sputtering effects are now considered to be minor when compared with atomic transport or instrumental effects (*C425, C426*).

The fourth factor deals with crystal orientation and imperfections and are largely associated with dependencies of the sputter yields.

The fifth factor on information depth is pertinent for higher energy primary ions only. For these ions, the mean escape depth of the sputtered particles increases with energy.

The sixth factor deals with preferential sputtering. Betz (*C383*) recently reviewed the sputtering phenomena for alloys and compounds. Although preferential sputtering does not usually occur in dynamic SIMS (for steady-state reasons), the steady-state surface layer sometimes takes longer to form (*C427*). Preferential sputtering can also form surface shapes (*C421*), particularly if a low-sputter-rate impurity is present on the surface. As a special case of preferential sputtering, ion-induced chemical reactions occur when sputtering compounds (*C428–C431*) or when sputtering with reactive ions (*C417*).

The seventh factor deals with knock-on and cascade (or atomic) mixing caused by the primary ion beam. A number of theoretical (including model calculations) (*C227, C415, C432–C436*) and experimental (*C437–C442*) studies including an isotope effect study (*C443*) have been reported. One conclusion of the work is that the influence of knock-on and cascade mixing is reduced by using heavy ions at low energy (<1 keV) and at glancing incidence.

The eighth and final factor deals with atomic transport effects such as bulk and surface diffusion, segregation (*C444*), etc., which can be induced by bombardment or radiation damage. Hofmann (*C410*) points out that although atomic transport effects are temperature dependent, it is surprising that little information is known about the temperature dependence of depth profiling (*C426, C444–C446*). As an answer to this question, Rabalais and co-workers (*C447*) studied the temperature dependence of the positive secondary ion yields from Fe, Co, Ni, Cu, and CuNi alloys. They observed strong temperature dependencies which are caused by the migration of minor amounts of sample impurities. Myers (*C448*), in another paper, discusses the mechanisms by which ion bombardment influences atomic transport as viewed by cascade mixing, by enhanced diffusion via mobile point defects, by rapid diffusion along extended defects, and by defect trapping. Among the four processes, Myers indicates that defect trapping of interstitials proved most important for high-energy ion beam analysis.

From these considerations, Hofmann (*C410*) lists the basic requirements to obtain optimum sputtering depth profiles: (1) The ambient environment needs a low residual reactive gas pressure. (2) The sample should be flat and preferably polished. It should also be amorphous and composed from materials of similar sputtering yield. Sufficient electrical and heat conductivities are also important so that the sample does not change or decompose when bombarded with ions. (Reuter, et al. (*C449*) observed unusual charging from targets containing low-conductivity regions.) (3) The primary ion beams should be constant and have a uniform current density. Ion-induced artifacts are reduced when the ion beam is low in energy (<1 keV), reactive, and rastered and has a glancing angle of incidence.

Many of these depth profiling principles are exemplified by several extraordinary depth profiling studies of implanted hydrogen (*C450–C455*), deuterium (*C450, C451*), carbon (*C456*), nitrogen (*C412*), oxygen (*C412, C456–C458*), and phosphorus (*C459*). These elements are among the most difficult elements to detect, particularly at trace levels of 10^{18} atoms/cm^3. Magee and co-workers have successfully profiled H and D in various Si samples with a sensitivity of $<5 \times 10^{17}$ atoms/cm^3 and a depth resolution of <100 Å, but only after the partial pressure of H_2O was below 1×10^{-10} torr (*C450, C451*). Similar conditions are needed to detect P and B in the low 10^{16} atom/cm^3 range (*C459*). Wach and Wittmaack detected $<10^{18}$ atoms/cm^3 of N in Si (*C412*).

Other depth profiling studies detected B in Si in the range of 10^{14}–10^{19} atoms/cm^3 (*C460*) and n-dopants in GaAs (*C461*). Clegg (*C462*) measured the detection limits for important impurities in GaAs ranging from 10^{14} atoms/cm^3 for Cr and Mn to 10^{16} atoms/cm^3 for Si, Zn, and Sn. Several researchers studied the effects of laser annealing on the distribution of ion-implanted species in Si (*C463, C464*). Leta et al. (*C399*) measured the projected ranges for 114 individual species/substrate combinations of ion-implanted materials. Slusser and Sluttery (*C465*) gave some helpful hints for profiling layered structures. And Svec and co-workers (*C466*) using scanner laser mass spectrometry studied the migration of trace level solutes in solids.

Two laboratories report depth profiling studies of compounds. Pena et al. (*C467*) profiled chemically treated TiO_2 electrodes, and Evans et al. (*C468*) profiled thin organic and organometallic films bonded to glassy C and semiconducting SnO_2.

Ion Imaging. Besides obtaining the depth distribution of elements in a sample, it is often necessary to know the spatial distribution of the elements on the surface. Several instruments such as the electron microprobe, the scanning Auger microprobe, and the ion microprobe and ion microscope currently provide an elemental image or map of the surface. The microprobe-type instruments produce their elemental image by rastering a sharply focused beam across the surface and recording the ion intensities as a function of beam position. The ion microscope, on the other hand, images the surface directly by maintaining a 1-to-1 correspondence between the location of the emitted ion and its final position on the detector.

Several research groups have recently attempted to quantify ion imaging. Schilling (*C469*) used image-processing techniques to convert the sputtered ion intensities into atomic concentrations. Ruedenauer and co-workers (*C470*) have developed computer algorithms to remove artifact contrast effects such as topographic, chromatic, or matrix contrast from secondary ion images. Several groups have used modified empirical or physical approaches for quantitative image analysis. Steiger and Ruedenauer (*C471*) used a modified one-parameter local thermal equilibrium (LTE) model requiring one internal standard to compute an image correction factor. Drummer and Morrison (*C472*) used both the modified LTE method and an empirical sensitivity factor approach to quantify their ion images. They also employed ion-implanted internal standards and "imaging standards" to evaluate their quantitative procedures. Furman and Morrison (*C473*) have developed a multidimensional imaging capability known as MIDAS or microscopic image digital acquisition system. MIDAS consists of an ion microscope, a low light level TV camera, a video color graphics system, and computer algorithms for digital image processing and allows for the rapid multidimensional image acquisition and digitization. These developments now permit secondary ion mass spectrometric–image-depth profiling (SIMS-IDP) studies for three-dimensional elemental analysis (*C474*).

Polyatomic and Molecular Ions

Polyatomic or molecular ions predominate the secondary ion emission pattern of most substances whether the substance is metallic, organic, or inorganic. Many researchers, who do quantitative SIMS or depth profiling studies, find polyatomic ions a nuisance because they overlap important spectral regions where atomic ions occur (*C315*). These researchers have developed, in fact, ways to eliminate (or filter-out) the polyatomic ions. Other researchers, however, use the polyatomic or molecular ions to conduct a detailed *chemical* analysis of the surface (*C475–C479*).

The formation mechanism of molecular or other polyatomic ions or cluster species by ion bombardment can be broadly viewed either as direct emission of species originating at the surface or the recombination of sputtered species to form new

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

structures. The direct emission model includes processes such as ejection through momentum transfer in collision cascades, thermal spikes, thermal evaporation, and even agglomeration in a selvedge region or any other unrealized process that ejects species from contiguous sites. The recombination model, on the other hand, includes processes like agglomeration, nucleation, or association reactions which form species containing constituents from nonadjacent sites. Experimental evidence for the direct emission or the recombination mechanism is ambiguous except for some clear-cut cases for organic and small molecular adsorbates which emit intact (*C475*). From the available experimental results, one might, however, generalize that the relative inter- and intramolecular bond strength of the solid may dictate the nature of the emission process where species that have strong covalent or ionic bonds may undergo direct emission and those that have weak metallic or van der Waals bonds may undergo recombination (*C475*).

Cluster Ions. One way to elucidate the sputtering and the secondary ion emission processes in SIMS is to understand the formation and emission of cluster ions. An elemental solid M, for instance, generally emits cluster ions $M_n^{\pm}$ whose intensity decreases as n increases. Other experimental features associated with cluster ion emission are reviewed by Blaise and Nourtier (*C203*). Fuerstenau and Hillenkamp (*C480*) recently described the characteristic features for the emission of cluster ions from thin foils of metals and semiconductors when irradiated with high-energy pulses of UV laser.

Although much theoretical and experimental information is already known about the emission of elemental or atomic ions $M^{\pm}$, little is known about the formation of the dimer, trimer, etc. Harrison, Garrison, Winograd, and co-workers, however, have recently used classical molecular dynamics calculations to model the sputtering process for elemental solids (single crystals) and simple atomic or molecular adsorbates (*C240–C242*). A principal conclusion of their model is that cluster species do not leave the surface of the solid as intact species but form in a region above the surface by a recombination mechanism. They also conclude, in fact, that it is rare for an emitted metal dimer to be formed from nearest neighbor atoms.

Rabalais and co-workers (*C481–C483*) also conducted several clustering experiments which concluded a recombination mechanism. One experiment determined the clustering distances (or the distances or region over which atoms could combine during sputtering) critical to form metal dimers. For Cs_2^+ dimers sputtered from a KCl–CsCl solid solution in which the [Cs]/[K] ratio varied from 10^{-1} to 10^{-5}, the clustering distance is ~200 and ~400 Å for He^+ and Ar^+ primary ions, respectively (*C481, C482*). In another study, Rabalais and co-workers observed sputtering-induced recombination of nitrogen isotopes on tungsten (*C483*).

The sputtering of multicomponent systems such as oxides and alloys is not understood as well as the sputtering of elemental targets, Wittmaack (*C476*) and Snowdon (*C484*) discuss the characteristic changes that occur in the secondary ion spectrum of metals during oxygen adsorption and during oxidation. (Atomic and cluster ion emission from silicon, silicon oxide, and dopants in silicon have been recently discussed (*C485*).) Wittmaack also presents some generalized rules for the emission of cluster ions from metal oxides (*C476*). Plog and Gerhard (*C486*) discuss the physical aspects of the parameters used in the "valence model" to determine the ion yields from oxidized metal surfaces and metal oxides. Wittmaack (*C476*) gives a different approach to the model.

Cluster ions are also formed by sputtering rare-gas solids (*C487*) and molecular solids (*C488–C493*). Michl and co-workers studied the SIMS of neat solid Ar, Kr, and Xe as a function of the type and energy of the primary ion, He^+–Xe^+ (*C487*). The relative abundance of higher cluster ions increases with the primary ion momentum.

In the case of frozen hydrocarbons such as methane (*C488*), pentane (*C489*), cyclohexane (*C490*), and benzene (*C490*), cluster ions appeared to form by ion–molecule reactions (*C475*) with no discrete repeating unit. If the frozen organic molecules, on the other hand, have a permanent dipole such as acetone (*C489*) or are hydrogen-bonded such as formic acid (*C491*), then the cluster series consists of $[H(M)_n]^+$ ions. A similar situation is observed for ice where a long series of $[H(H_2O)_n]^+$ ions occurs for n = 1 to 50 (*C490*).

Several other solids composed of small molecules give cluster ions of regular composition, i.e., $[(N_2)_n]^+$ and $[N(N_2)_n]^+$ from solid nitrogen (*C488, C492*), $[C(CO)_n]^+$ and $[(CO)_n]^+$ from solid CO (*C492*), $[NO(N_2O_3)_n]^+$ and $[NO(N_2O_3)_m(N_2O_4)_n]^+$ from solid NO, N_2O, N_2O_3, and N_2O_4 (*C493*), and $[O_{3n+2}]^+$ from solid O_2 (*C493*). Although the high degree of clustering and fragmentation complicates the analytical application of SIMS, Michl and co-workers are able to simplify the mass spectra by diluting the neat matrix with rare gases or by reducing the momentum of the primary ions.

Cluster ions are also formed by sputtering ionic solids such as the alkali halides. SIMS spectra of alkali halides, MX, show intense ions of the type M^+, M_2^+, $[MX]^+$, $[M(MX)_n]^+$, X^-, and $[X(MX)_n]^-$ (*C475, C482, C494–C496*). Researchers at the Naval Research Laboratory (NRL) recently observed positive cluster ions from CsI extending to the $[Cs(CsI)_{70}]^+$ ion at m/z 18 320 (*C496*). These high-mass cluster ions are the largest mass-resolved ions ever produced and detected using a conventional mass analyzer (*C496, C497*) and more than 20 times larger than ions reported with other SIMS instruments.

Although the original alkali halide cluster ion data were interpreted by a recombination mechanism (*C482*), the NRL group interpreted their data by a direct emission mechanism of sputtering. Their conclusion is based on several results: (1) the most stable cluster ion configurations correspond to bulk "cubiclike" structures, and (2) from a two-dimensional molecular dynamics study of metal halides (*C257*), cluster ions are more likely to represent the unarranged surface as the primary ion beam impinges at smaller angles relative to the surface.

The structure of the ionic clusters have also been studied by various bond-breaking (*C496*) and thermodynamic (*C498*) models.

The formation of cluster ions of alkali halides in field desorption studies are attributed to the effect of a field- and temperature-dependent charging of salt layers by alkali ions (*C499*). The melting point of the solid influences the ion formation mechanism.

Inorganic Salts. Secondary ion emission patterns from other alkali metal salts such as the alkali sulfates, nitrates, perchlorates, etc., which contain covalently bonded oxyanions within an ionic lattice, appear differently from the clustering patterns found for the alkali halides (*C475*). Ganjei et al. (*C267, C268*) correlated the negative secondary ion intensities with the stoichiometry of the anion. For example, the negative ion pattern (O^-, S^-, $[SO]^-$, $[SO_2]^-$, $[SO_3]^-$, and $[SO_4]^-$) can be represented by a modified-Poisson distribution if dissociation of $[SO_4]^-$ and $[SO_3]^-$ is assumed for sulfate and sulfite, respectively. A recombination model did not fit the observed intensity distribution (*C268*).

Other researchers studying similar inorganic systems found that the fragmentation of a given oxyanion is dependent on the nature of the cation, i.e., $AgNO_3$ vs. $NaNO_3$ (*C500*) and that the formation of species such as $[Na_3SO_4]^+$ from Na_2SO_3 could be explained by a gas-phase statistical recombination mechanism (*C501, C502*).

Nonvolatile and Thermally Labile Molecules. The recent development of several new ionization methods in mass spectrometry has significantly improved the analysis capability for nonvolatile and thermally labile molecules (*C57*). Several of these methods, namely, in-beam techniques, field desorption (FD), laser desorption (LD or LAMMA), fast-atom bombardment (FAB), and SIMS ionize molecules directly from the solid state thereby reducing the chance of thermal degradation. (Two other ionization techniques have been recently developed; one by Cotter (*C503*) using laser desorption chemical ionization mass spectrometry and another by Vestal and co-workers (*C504*) using a combination of partial vaporization by very rapid heating of a liquid solution containing the sample and final vaporization by impact of an aerosol containing the sample on a heated metal plate. The latter method requires no ionizing source such as an electron beam or strong electric field.) Although these methods use fundamentally different excitation sources, similarities in their mass spectra suggest a related ionization process (*C3, C5, C475, C477, C505, C506*).

Benninghoven and co-workers reported the first systematic study of some thermally sensitive molecules by SIMS. Their results for amino acids showed abundant $[HM]^+$ and $[M - H]^-$ molecular-like ions as well as the characteristic fragment

POSCO Future M v. TIAX LLC
POSCO Future M EX1020

ion $[HM - HCOOH]^+$ resulting from decarboxylation of the protonated molecular ion. These mass spectral features are due to ion/molecule reactions and unimolecular dissociations which are also common in other forms of mass spectrometry, e.g., chemical ionization mass spectrometry (*C475, C477, C478, C507*). The basic fragmentation process involves the loss of neutral molecules from the even-electron protonated or deprotonated molecular ion. Several detailed studies of the SIMS fragmentation process have been recently reported for amino acids (*C475, C508–C511*), organic acids (*C512*), various biomolecules (*C477, C513*), and organic amines and amides (*C477, C514*). Other recent organic SIMS studies include the analysis of sugars, nucleotides, nucleosides, and peptides (*C515–C517*), and mixtures of amino acids, barbiturates, opiates, and/or stimulants (*C518–C521*). Similar results are reported in recent FAB studies of amino acids, peptides, nucleotides, alkaloids, saccharides, and many other biomolecules (*C161–C167*). In fact, most FAB applications to date deal with biomolecules (*C5, C497*).

In addition to the protonated molecular ion, $[HM]^+$, the SIMS spectra of organic molecules contain cationized species formed between the molecule and metal (alkali, transition or noble) ions from the substrate or impurities (*C475, C477*). Cooks and co-workers tested the cationizing ability of several metals with polar and nonpolar organic molecules and found that (1) the cationization yield varied for different metals and for metals in different charge states, (2) the site of attachment is dependent on the nature of the metal, and (3) the degree and type of fragmentation is dependent on the attachment site and the strength of the interaction between the metal atom and the organic molecule (*C475, C477*). Recent examples of cationization in SIMS include studies on amino acids (*C475, C508–C511*), organic acids (*C512*), and organic amines and amides (*C477, C514*).

The polarity of the organic molecules affects the fragmentation reaction. While polar molecules form intense cationzed species and exhibit fragment ions characteristic of even-electron ions, nonpolar molecules form intense $M^{+\cdot}$ ions (by another type of ionization process, see below). Subsequent fragmentation of the $M^{+\cdot}$ ion is comparable to EI (electron impact) results. Hence, the chemical properties of molecules affect the formation of molecular ions. Empirical rules for cationization can perhaps be based on known principles of coordination chemistry (*C522*).

Cationization in other forms of mass spectrometry such as field desorption (FD), plasma desorption (PD), and laser desorption (LD) usually occurs by alkali ion attachment (*C168–C194*). Cooks and co-workers (*C523*), however, combining for the first time, laser desorption with mass spectrometry/mass spectrometry show cationization of sucrose by Ag^+. Cationization in thermal desorption (*C524*), with in-beam techniques (*C525, C526*), by heavy ion-induced desorption (*C527*), and by surface ionization (*C528*) have also been reported.

Cooks and co-workers (*C515*) have demonstrated that the capabilities of organic SIMS are greatly extended if the organic sample is mixed with ammonium chloride. The ammonium chloride acts as a room-temperature isolation matrix from which organic molecules can be sputtered, and since the matrix is not inert, it can protonate molecules more basic than ammonia. The ammonium chloride is usually mixed with the organic and then burnished into a metal foil. If sodium chloride is purposefully added to the ammonium chloride matrix, cationization by sodium is greatly increased.

In addition to cationization (and protonation), Cooks and co-workers (*C477*) have discovered two other ionization processes for organic molecules in SIMS. The first process involves an *electron transfer* to form intense $M^{\pm\cdot}$ molecular ions. This process has been observed primarily for nonpolar molecules. The proposed mechanisms are charge-exchange transitions between sputtered ions and the neutral organic molecules or electron attachment of low-energy secondary electrons to the neutral molecule. The fragmentation reactions of the $M^{\pm\cdot}$ ions follow the dissociation pathways for odd-electron gas-phase ions.

The second distinct type of ionization process in organic SIMS involves the *direct emission* of intact organic cations or anions from the solid state as $(M - \text{anion})^+$ and $(M - \text{cation})^-$ ions (*C477*). SIMS studies of organic salts give intense cationic and anionic species with little fragmentation. The SIMS spectra of quaternary ammonium salts, for example, show the direct emission of the quaternary ammonium ion $[R_4N]^+$ (*C529*). Similar ion emission patterns for the quaternary ammonium salts have also been reported for plasma desorption (*C505*), laser desorption (*C503, C505*), thermal desorption (*C530–C532*), and in-beam electron-impact (*C533*) techniques. A recent paper by Schuler and Krueger (*C505*) compares the mass spectra of tetrabutylammonium iodide produced by FD, LD, PD, and SIMS.

Cooks and co-workers also reported the direct emission of molecular dications from some diquaternary ammonium salts (*C534*). Their paper constitutes the first observation of multiply charged organic molecular ions in SIMS. Whether the dications are emitted or not depends on the intercharge separation of the two nitrogen groups in the molecule.

The secondary ion intensity for the organic salts is generally around 2 orders of magnitude higher than that observed in cationization. The efficiency of the various ionization processes in organic SIMS, therefore, falls in the order direct emission > cationization > electron transfer. The higher efficiency of the direct emission process lowers the detection limits for organic salts in SIMS such that picogram quantities can be detected (*C535*).

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