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Surface Characterization

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Review Contents

Reviews of surface characterization have appeared in *Analytical Chemistry* every two years since 1977 (*1−11*). The field continues to grow and diversify with new surface analysis techniques as well as new applications. This review is similar to the last one in this series, being written by multiple authors with different specialties in an attempt to highlight advances in each of these areas. This review begins with literature from January 1997 and ends with literature from approximately October, 1998.

PROXIMAL PROBES

A review of proximal probe techniques and applications relevant to the analytical chemist is presented, organized by type of investigation (rather than by SPM technique as in previous reviews). This review is not meant to be exhaustive, but rather highlights recent advances that have impact on surface characterization. Specifically, papers concerned solely with surface topography and structural determinations, as well as papers on superconducting substrates, will not be included. The complete proceedings of the Ninth International Conference of Scanning Tunneling Microscopy/Spectroscopy and Related Techniques are published as a supplement to *Applied Physics A* and papers from that conference are not included in this review (*A1*).

A number of review articles on SPM techniques and applications have appeared in the last two years. A set of reviews on many aspects of scanning probe microscopy was published as an issue of *Chemical Reviews* (*A2*). Lieber and co-workers reviewed fundamentals of the chemical force microscope (CFM) and its applications for probing the interaction strength between mol-

ecules (*A3*). Bottomley reviewed scanning probe microscopy for the 1998 *Analytical Chemistry* Fundamental Reviews issue (*A4*).

Scanning Probe Spectroscopies. Recording the vibrational spectra of isolated molecular adsorbates through inelastic electron tunneling spectroscopy (IETS) has been a long-standing goal in STM research. Ho and co-workers succeeded in recording IET spectra of acetylene and perdeuterated acetylene adsorbed on Cu(100) with a low-temperature STM (*A5*). They observed a peak in d^2I/dV^2 vs eV for acetylene at 358 meV corresponding to the C−H stretch. This peak is shifted to 266 meV for the C−D stretch in perdeuterated acetylene. Tuning the bias voltage to the energy of the vibrational mode and monitoring d^2I/dV^2 allows vibrational spectroscopic imaging. IETS with the STM allows unambiguous chemical identification of surface species and enables probing the effects of the local chemical environment of adsorbates.

Rotations of O_2 molecules adsorbed on Pt{111} at 8 K were driven by inelastic electron tunneling from an STM tip (*A6*). The threshold bias voltage for the transition between a single-electron and multiple-electron processes was determined. The potential energy barrier to rotation as well as the vibrational relaxation rates were obtained by comparing the experimental results with a theoretical model.

Stranick et al. developed a new near-field scanning optical microscope (NSOM) design based around an ellipsoidal cavity that enables simultaneous topographic, optical transmission and optical reflection data collection (*A7*). The reflected optical signal from the probe−sample junction is collected and reimaged by the ellipsoidal cavity, enabling coaxial light collection, which minimizes shadowing artifacts that often complicate interpretation of reflection measurements. A constant tip−sample separation is maintained by piezoelectric detection of shear force. Piezoelectric detection avoids the need for additional shear-force optics, which would cause alignment problems and block some of the reflected light.

A scanning near-field optical/atomic force microscope was used to probe *Escherichia coli* cells in both semidry and aqueous solution, simultaneously measuring topography and fluorescence (*A8*). This SPM has an optical resolution of 50−100 nm in fluorescence mode. Topographic and fluorescence images were acquired of transgenic *E. coli* with the gene for green fluorescent protein (GFP). Fluorescence images demonstrated that individual *E. coli* cells had different fluorescent intensities and also suggested that GFP oxidizes at the surface of the cell.

Emory and Nie used an integrated far-field optical and atomic force microscope to develop a method for screening and enriching Ag nanoparticles (*A9*). These Ag nanoparticles were reported to exhibit surface-enhanced Raman scattering enhancements of 14−

15 orders of magnitude, sufficient to be used for the optical detection and spectroscopy of single adsorbate molecules.

Ultrasonic force microscopy was used to image the local elastic properties of Ge quantum dots on a Si(001) substrate (A10). This probe has a lateral resolution of 5−10 nm and can determine areas of local strain variation.

A NSOM was used to collect photons from local second-harmonic generation (SHG) at a rough Ag surface (A11). SHG from s-polarized light is highly correlated to surface topography while SHG from p-polarized light is related to a SH field localization. Understanding the SHG dependence on excitation polarization remains necessary to develop a SH near-field microscope.

Photon emission STM (PESTM) was used to differentiate between regions of silicon carbide, Si(111)−(7 × 7), and Si(111)−(1 × 1)-W based on their photon emission efficiency (A12). Photon emission maps of the surface provided material contrast with a lateral resolution of ∼2−4 nm, in good agreement with the theoretical limit of 2 nm for plasmon localization.

A NSOM employing a tetrahedral tip was used to image a mixed gold and silver inverted film on an indium tin oxide (ITO) substrate with an edge resolution of 1 nm, at a wavelength of 635 nm (A13). Silver grains are more absorbing than gold grains, and this was used as a means to distinguish between the two materials. This technique can discriminate between different materials (simultaneous experiments were run with a coupled NSOM/STM).

Dual-mode NSOM was used for the characterization of the opaque Hf aryl(4-diethylphosphonate)-10-decylphosphonic acid ether nine-layer self-assembled multilayer on a silicon substrate (A14). This probe simplifies and increases the resolution in reflected-mode NSOM. Topography was shown to induce but be separable in this mode from NSOM contrast.

Differentiating and Identifying Adsorbates Using Scanning Tunneling Microscopy. Through an extensive study, the STM was used to discriminate between functionalized alkanes on HOPG under ambient conditions (A15). It was inferred that the trans-conductance of methylene protons could be used as a reference for the assignment of other functional groups. A HOMO ionization potential model was used to show that molecules with low ionization potential appear as protrusions. Topographic and electronic coupling factors are important in determining STM images. These experiments were treated with a simple model. Hartree−Fock calculations were used to simulate STM images (A16). The interactions between the molecules and graphite could be adjusted to study this effect.

Flynn et al. used STM to identify the R and S enantiomers of 2-bromohexadecanoic acid adsorbed onto the basal planes of graphite at the liquid−solid interface (A17). With the use of marker chemical groups and atoms, the conformations could be determined. Each molecule displays three different features: a large protrusion attributed to Br, next to a lower topographic region attributed to the carboxylic group, and a chain of small protrusions which are attributed to methylene protons.

Weiss et al. used the STM to measure the electron transport properties of single and bundled 2′-ethyl-4,4′-di(phenyleneethynylene)benzothioacetate molecules inserted and isolated in a 2D matrix of alkanethiol self-assembled monolayers (SAMs) (A18).

This study determined the contribution of the alkyl chains to tunneling and showed the dependence of the topographic contrast in STM on conductance and tunneling barrier.

Nanometer-Scale Structures. A gold quantum point contact the width of a single strand of Au atoms was created by making contact with a gold island on a Cu substrate with a STM tip (A19). The measured conductance for a single strand agreed well with the Landauer conductance of $G_0 = 2e^2/h$ (e is the charge on the electron and h is Plank's constant).

A magnetic force microscope (MFM) was used to study the magnetic switching behavior of Co dots on a GaAs surface (A20). A 3 × 3 array of Co dots was studied by placing a MFM within a Helmholtz coil pair. By applying external magnetic fields, the magnetization of different dots could be switched 180° at thresholds dependent upon the proximity of the probe tip.

Near-field photoluminescence and photoluminescence excitation spectroscopy were used to characterize the formation of single GaAs quantum wires grown on a patterned GaAs(311) surface at the side walls of 15−20-nm mesa stripes (A21). Photoluminescence was used to show that quantum wells and quantum wires are separated from each other and that carriers diffuse into the quantum wire from the quantum well. Photoluminescence and photoluminescence excitation spectroscopy in an applied magnetic field were used to investigate the spin relaxation of carriers of single InAs/GaAs self-assembled quantum dots. Controlling the polarization of the incident light enabled measurements of the spin-resolved spectra (A22).

Surface Adsorption, Diffusion, and Manipulation. In a study of the effects of strain on adsorption, a STM was used to provide evidence for the preferential adsorption site of oxygen atoms on Ru(0001) surface: on top of protrusions made by nanometer-size Ar bubbles (A23). This effect can be considered as the reverse of adsorbate-induced strain and can be used to provide a critical test of theoretical predictions.

STM was used to investigate ICl adsorption selectivity on the Si(111) surface (A24). Surprisingly, the formation of silicon iodide with the ejection of chlorine atoms back into the gas phase is the favored mechanism. This result was confirmed by careful comparison between STM data and Auger spectroscopy.

Diffusion of adsorbates on surfaces is typically due to random uncorrelated hopping events. Briner et al. showed that CO molecules on the Cu(110) surface diffuse at higher rates when bound as part of adsorbate chains than they do as isolated molecules (A25).

Some SPMs are beginning to be designed to work over a range of temperatures and pressures in order to study reactions under reaction conditions. A STM was used for the structural determination of the molecular adsorbate CO, in equilibrium with the gas phase, on Pt(111) (A26). A hexagonal periodic structure (12 ± 2 Å with corrugation of 0.30 ± 0.05 Å) was observed at pressures between 200 and 750 Torr. The overlayer was incommensurate with the Pt(111) substrate forming a moiré-type structure. CO molecules in the overlayer were found to lie flat on the substrate. The differences between the low- and high-pressure conditions showed how important it will be to develop surface characterization techniques capable of working under realistic reaction conditions.

Meyer et al. demonstrated atom-by-atom lateral manipulation of Cu substrate atoms with a STM probe tip (*A27*). Cu atoms on the Cu(211) surface were moved parallel and perpendicular to step edges. The tunnel gap impedance had to be decreased (increasing the tip—atom interaction and decreasing tip—sample separation) from that for parallel manipulation to enable moving atoms perpendicular to step edges, revealing the asymmetric barriers to motion for this system. Manipulation of atoms in this manner allows creation of atom vacancy pairs as well as motion along close-packed rows. Lateral manipulation of single atoms, molecules, and dimers on a Cu(211) surface was also investigated (*A28*). Analysis of the tip—sample separation during lateral manipulation of adsorbates, along step edges of the Cu(211) surface, revealed two different manipulation modes. Attractive tip—adsorbate interactions (pulling) vs repulsive tip—adsorbate interactions (pushing) could be determined from tip—sample separation curve characteristics. Lead dimers were shown to make single, double, and triple lattice site hops as a function of tunneling resistance.

Surface Films. Berger et al. used the AFM to perform in situ measurements of the surface stress changes and kinetics during the self-assembly of alkanethiols on a Au surface on SiN_x cantilevers (*A29*). The compressive surface stress, caused by the self-assembly process, followed Langmuir adsorption kinetics up to monolayer coverage. It was found that the surface stress increased linearly with length of the alkyl chain. These sensitive measurements based on AFM cantilevers have potential applications as sensors for specific chemical and biological interactions.

Forster et al. studied the design of a surface Au/Ni alloy for steam reforming (abstraction of H from hydrocarbons) (*A30*). The catalyst consisted of a $MgAl_2O_4$—substrate-supported Ni catalyst alloyed with a 0.3 wt % Au. Density functional theory (DFT) calculations for the dissociation were used as a theoretical comparison. STM and molecular beam scattering was used to measure the dissociation probability of methane. Extended X-ray absorption fine structure spectroscopy (EXAFS) was used to confirm the surface alloy. Both pure Ni and the Au/Ni catalyst were first reduced in pure H and subsequently exposed to a diluted *n*-butane gas at 500 °C. The Ni catalyst deactivated quickly due to graphitic carbon contamination, while the alloy catalyst constantly converted *n*-butane. No graphitic carbon was observed on the Au/Ni sample.

Liley et al. used lateral force microscopy in the wearless regime to measure the friction characteristics of a lipid monolayer on a mica surface (*A31*). It was found that the friction anisotropy and asymmetry of a compliant monolayer was induced by a tilt angle as small as 15° within the monolayer. In agreement with predictions, the anisotropic friction depended on both the angular tilt and the tilt direction. The asymmetry could not be explained.

Hamers et al. showed that well-defined organic films can be formed on the reconstructed Si(001) surface with STM and IR characterization (*A32*). Si dimers can react with the π-bonds of the unsaturated organic species cyclopentene. STM was used to show that the molecules are ordered translationally. The dimers were spaced at integral values of 3.85 Å.

Tapping-mode near-field scanning optical microscopy (TM-NSOM) was used to characterize a Langmuir—Blodgett monolayer of L-α-dipalmitoylphosphatidycholine (DPPC)/0.25 mol % 1,1′-dioctadecyl-3,3,3′,3′-tetramethylindocarbocyanine perchlorate (di-IC_{18}) fluorescent dye (*A33*). Phase separation within the lipid monolayer was shown through fluorescence, topography and compliance images.

Monolayer Film Manipulation. The surface architecture of self-assembled monolayers can be controlled by such techniques as microcontact printing (μCP), "nanografting", local scanning probe electrochemistry, and nanometer-scale patterning of metals. In addition to being involved in manipulation, SPM measurements are used to determine the patterns formed.

Using STM and wettability measurements, Larsen et al. compared the surface structures of SAMs of dodecanethiol (DDT) on Au(111) formed by poly(dimethylsiloxane) (PDMS) μCP and by solution equilibration (*A34*). Using 100 mM DDT in ethanol solution to wet the PDMS press, the STM images of the resulting SAMs showed domains of $(\sqrt{3} \times \sqrt{3})R30°$ lattice with $(4\sqrt{3} \times 2\sqrt{3})R30°$ superlattice structures, indistinguishable from the STM images of the SAMs formed by solution- or vapor-phase deposition. The image similarities remained at all length scales examined (up to $4 \times 4 \mu m^2$). Subsequent measurements showed that μCP SAMs formed from more dilute solutions lost these structural similarities.

Xu et al. investigated the kinetics of the alkanethiol self-assembly on gold surfaces, specifically comparing SAMs formed by solution equilibration with SAMs formed under spatially constrained self-assembly (SCSA) conditions associated with nanografting (*A35*). Time-dependent in situ AFM imaging was used to monitor the rates of adsorption of the alkanethiols. SCSA was found to occur more than 10 times faster than unconstrained self-assembly and also showed fewer defects. The proposed mechanism suggested that the steric hindrance associated with SCSA prevented the initial laying-down configuration of the adsorbed alkanethiols, instead favoring the S down, alkyl chain up configuration. They argue that the initial presence of this laying-down configuration in unconstrained self-assembly slows the adsorption of subsequent alkanethiol molecules from solution.

Sugimura and Nakagiri used a combination of scanning probe lithography (SPL) and self-assembly to produce coplanar nanostructures consisting of two different types of organosilane monolayers (*A36*). A trimethylsilyl monolayer was prepared on the native oxide of Si. Line patterns were then "drawn" on this template by removing some of the monolayer using a SPM probe tip. As the exposed native oxide surface once again became hydrophilic, the SPM probe was connected to the surface through an adsorbed water column to anodize the exposed area. A different organosilane was then adsorbed onto the anodized area. The template was then labeled with aldehyde-modified fluorescent latex nanoparticles and viewed with a fluorescence optical micrograph. The nanoscale area-selective growth of the different organosilanes was thus confirmed.

Similar work was done by Zamborini and Crooks using an STM tip where an alkanethiol SAM on Au(111) was first etched with a positive bias through faradaic electrochemical process in humid environment (*A37*). A few layers of Au were also removed through the process. Ag was then deposited by reduction onto the scanned area using the Pt/Ir STM tip. The process was monitored via in situ STM imaging. Different size Ag deposits could be obtained.

Carbon Nanotubes. Since the discovery of carbon nanotubes, their peculiar electronic and mechanical properties have

attracted much attention. Scanning probe microscopies and spectroscopies have proven uniquely positioned to determine these properties as a function of nanotube structure. The electrical conductance of nanotubes was predicted to depend critically on the diameter and helicity of the tubes. Dekker and co-workers (A38) and Lieber and co-workers (A39) used scanning tunneling microscopy/spectroscopy to measure the electronic properties and atomic structure of single-walled nanotubes (SWNTs) deposited on Au(111). Both groups confirmed that the electronic structures of the tubes are a function of both diameter and helicity and fit theoretical predictions.

Carbon nanotubes are candidates for use as quantum wires. Dekker and co-workers used an AFM to find a single-wall nanotube, which was deposited (by chance) across Pt electrodes on a Si/SiO_2 substrate (A40). This enabled electrical transport measurements of what was then known to be a SWNT (A41).

The mechanical properties of nanotubes have also been investigated with SPMs. Avouris and co-workers used an AFM to investigate the deformation of isolated and crossed carbon nanotubes, deposited on H-passivated Si(100), due to surface van der Waals interactions (A42). The nanotubes deform elastically, and the extent of deformation depends on both the tube radius and number of shells that make up the nanotube.

Lieber and co-workers showed that carboxylic acid-terminated SWNTs could be functionalized through amide linkages to provide chemically sensitive imaging of surfaces (A43). In tapping mode, adhesive force measurements could discriminate between terminated CH_3 and COOH regions of the sample.

AFM was used for the manipulation and imaging of multiwalled nanotubes (MWNTs) on Si(100) (A44). Nanotubes were found to conform to the substrate topography. The nanotubes were manipulated under control and could be pushed into different shapes, positions, and alignments. Nanotubes deform elastically, and it may be possible to induce local chemistry by straining a nanotube.

MWNTs and SWNTs have been attached to AFM tips to image amyloid-β 1−40 (Aβ40) fibrils produced in vitro (A45). Gentler imaging leads to adhesion forces that are on the order of 2−5 times lower than for conventional AFM tips. Fibrils and protofibrils are 3−8 nm smaller when imaged with MWNTs as compared to conventional probe tips (12−30% increase in resolution). Features in images recorded with SWNT tips are 10−15 nm smaller than those recorded with conventional Si tips.

Lieber and co-workers showed that MWNTs are terminated with carboxy groups by measuring a drop in adhesion force at pH ~4.5, in agreement with the deprotonation of a carboxylic acid (A46). Force titrations were measured for pH 2−9. It was shown that MWNTs could be derivatized to measure ligand−receptor interactions.

Other Subjects. Rief et al. used the AFM to unfold titin, the giant sarcomeric protein of striated muscles, to investigate its mechanistic properties (A47). It was found that, at high extensions, the restoring forces of the protein showed a periodic sawtoothlike pattern. Together with the directly measurable restoring forces, this kind of pattern reveals the unfolding domains of proteins.

Rief et al. also used the AFM to probe dextran filaments linked to a gold surface to measure the various intermolecular and intramolecular forces (A48). The polysaccharide filaments were stretched away from the surface by an AFM tip. As the AFM-induced stretching forces increased, the polysaccharide's deformation was at first dominated by entropic forces according to the Langevin function with a 6-Å Kuhn length and then by a twist of bond angles. At higher stretching forces, distinct conformational changes occurred within the molecule to cope with the elongation and the bending of bond angles dominated the elongation.

Roters et al. used the SFM cantilever and laser-induced thermal noise to probe a polymer brush (immobilized poly(styrene) on a SiO_2 substrate) (A49). Different solvent environments were investigated—pure toluene and pure methanol. In toluene, the polymer brush was swollen and its boundaries were difficult to determine. In methanol, the brush collapsed and gave a dramatic change in response as the probe came into contact with its boundary.

Gimzewski and co-workers showed that the STM could be used for conformational analysis of Cu−tetra(3,5-di-*tert*-butyl-phenyl)porphyrin molecules on a corrugated Au(110) surface (A50). Single-molecule conformation relies on the symmetric lobes of the porphyrin and their aspect ratios of 1.4:1 for the precursor state on top of 2 × 1 terraces and 1:1.6 for the final adsorption states on a 6 × 1 surface reconstruction. Conformational changes are driven by surface−molecule interactions.

Scanning polarization force microscopy (SPFM) can be used to determine the electrical forces of a surface by recording noncontact images in constant force mode (A51). SPFM was used to study the condensation of gylcerol from its vapor onto a mica surface (~50% RH, 21 °C, and dc biases of −6 to +6 V). Condensation begins in two dimensions with droplets being formed; the droplets grow vertically and then laterally before the film completely covers the surface.

Seeman and co-workers used double-crossover oligonucle-otides to make β-form DNA create two-dimensional crystals with uniform thickness that have been specified by Wang tiles as large as 2 × 8 μm^2; imaged by contact-mode AFM under 2-propanol (A52). This was done to create multicomponent DNA lattices on mica surfaces.

McKendry et al. used CFM to discriminate between molecules of distinct chirality, due to the differences of adhesion and frictional forces between the functionalized tip and substrate for the enantiomers of mandelic acid (A53). Mandelic acid has a single chiral center that can be attached as an ether, through the aromatic ring, or as an ester. It was shown that dissimilar chiral forms have an adhesive force of 1.1 nN, and similar chiral forms ~0.5 nN; racemic mixtures gave adhesion forces between these values.

Bard and co-workers used the amperometric mode of SECM for electrogenerated chemiluminescence (A54). By applying alternating pulses of +1.60 and −1.40 V vs SSCE, Ru(bpy)$^{3+}$ and Ru(bpy)$^{+}$were sequentially generated on an indium tin oxide substrate. (ECL generated due to annihilation reaction to produce Ru(bpy)$^{2+}$.)

Weisendanger and co-workers used lateral force microscopy to obtain contrast on the triglycine sulfate, TGS(010), and guanidinium aluminum sulfate hexahydrate, GASH(0001), cleavage faces due to the surface structures (A55). Since etch morphology can be used to determine surface polarity, different

sample orientations were used to show that the observed effect was not electrostatic. Several friction coefficients were observed on the surface, due to different chemical compositions of oppositely charged domains. Friction coefficients within the same image depended on the orientation of the crystallite relative to the scan direction.

Loiacono et al. demonstrated that a conducting probe AFM (CPAFM) could be used to measure the conductivity of sexithiophene on Au and SiO_2 substrates (A56). The modified probe consisted of a tapping-mode AFM that could be externally switched to contact mode for point contact electrical characterization.

Bard et al. used AFM for the study of the diffuse double layer at an n-type TiO_2 single-crystal electrode with a SiO_2 tip, as part of a three-electrode system (A57). The isoelectronic point of the TiO_2 electrode and a point of zero force were measured in KCl solution.

Low-temperature STM was used to investigate the roles that metal promoter atoms play in molybdenum disulfide (MoS_2) hydrodesulfurization (HDS) catalysis (A58). Kushmerick and Weiss determined that Ni promoter atoms adsorbed to the MoS_2 basal plane are extremely mobile; the barrier to diffusion for this system is so small that Ni adatom motion was too rapid to enable STM imaging at temperatures down to 77 K. At 4 K, isolated atoms and small clusters were stable and could be imaged with the STM; however, Ni adatoms could still be easily manipulated with the STM tip. Spectroscopic imaging of the Ni cluster revealed its favorable electronic structure for binding nucleophilic reactants and intermediates.

The rapid pace of advances in scanning probe microscopy continues to provide ever more tools and more interpretable information for surface characterization.

ELECTRON SPECTROSCOPY

X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) are widely used techniques for studying the surface composition and surface chemistry of solid materials. Over 5800 English language papers related to XPS and AES have been published since the last review. A wide variety of materials have been studied using these techniques, including polymers, semiconductors, ceramics, metals, composites, catalysts, and biomaterials. This report concentrates on recent trends in the use of these techniques to solve material problems. A review of the basic fundamentals of XPS and AES has been published recently by Turner and Schreifels (B1).

Several books and book chapters were published on applications of XPS and AES since the last review. Sherwood reviewed the characterization of nanostructural materials using XPS and AES (B2). Briggs published a book on the analysis of polymer surfaces using XPS and static secondary ion mass spectrometry (B3). Chourasia and Chopra reviewed the common applications of AES (B4). The proceedings of the 4th International Workshop on AES and Electronic Structure are included in a special issue of the *Journal of Electron Spectroscopy and Related Phenomena* (B5).

Standard procedures for collecting, analyzing, and sharing surface analysis data continue to be developed. Efforts have focused on development of reference materials, databases, and methods for sharing data. The development of standard reference materials for surface analysis is being driven by the ISO Technical Committee 201. Work of this group was summarized by Powell and Shimizu (B6). Gaarenstroom developed an aluminum oxide thin-film standard which was characterized using seven different analytical techniques (B7). Fukumoto et al. grew an SiO_2/Si multilayer film for use as a depth-profiling standard (B8). Thin films of TiN, Ti(C,N), and (Ti,Al)N have been proposed as reference materials for measuring elemental compositions by Stock et al. (B9). The binding energy shifts of a series of Mg(II) compounds have been cataloged by Ardizzone et al. (B10). Seah and Gilmore produced a database of digital AES data (B11). They used these data to develop accurate quantification procedures. Godowski has also produced a database of AES spectra (B12). A database of XPS spectra was cataloged by Crist (B13). Binding energies and full width at half-maximums (fwhm) from pure elements and metal oxides were listed. Yosihara and Yoshitake described a database of AES and XPS data that is shared over the Internet (B14). Successful methods for sharing data among laboratories require repeatable and reproducible data collection. Seah reviewed the results of several interlaboratory studies aimed at improving data reproducibility and repeatability (B15). Brandi and Stori developed MAXMIND, a standardized data-exchange format that can be used for several analytical methods (B16). Conny et al. developed and tested a set of standard test data for evaluating curve fitting methods (B17). The data are used to illuminate errors that occur when using curve fitting procedures to separate overlapping spectra.

X-ray Photoelectron Spectroscopy. XPS continues to be an important tool for measuring surface chemistry and surface composition in support of a wide variety of technical areas. Review articles of many applications of XPS have recently been published. The characterization of thin films by XPS was reviewed by Hartmann and Lamb (B18). The interactions of Cl_2 plasmas with Si were reviewed (B19). The electron properties of rare-earth systems were reviewed by Laubschat (B20). XPS analysis of poly-(tetrafluoroethylene) metallization was reviewed by Rye et al. (B21). The sampling depth of a few atomic layers for XPS makes it a good technique for characterizing SAM specimens. Progress in this area was reviewed by Herdt et al. (B22). The measurement of surface acid−base properties was reviewed by Johansson and Klier (B23). Characterization of protein surfaces was reviewed by Blomberg et al. (B24). The characterization of petroleum reservoir cores was reviewed by Perry and Swift (B25).

One of the main advantages of XPS over other surface analysis techniques is the ability to measure the chemical state of elements. These measurements are often difficult due to peak overlaps since XPS peaks are rather broad and chemical shifts occur over a narrow energy range. However, great strides are being made using numerical methods to extract chemical-state information from XPS spectra with overlapping peaks. Splinter and McIntyre developed a maximum entropy method to enhance the spectral resolution of XPS data (B26). They applied this method to the determination of chemical states of a series of minerals (B27). Povstugar et al. employed a fast Fourier transform algorithm to analyze XPS spectra (B28). The total number of overlapping peaks could be extracted from complex spectra using this method. The XPS spectrum of an iron oxide thin film was analyzed by comparing the data to a spectrum that was reconstructed from

reference iron spectra (*B29*). Both film composition and thickness could be determined when the background was corrected for the contributions of inelastic scattering. Pattern recognition methods were used to characterize polycrystalline silicon layers (*B30*). The XPS spectrum of hydrogenated silicon was compared to a simulated spectrum which was constructed from a quantum mechanical modeling package (*B31*). The hydrogen content and type of Si–H bonding were determined. Quantum mechanical calculations were also used to determine the chemical shifts in metal alloys (*B32*). Baker and Hammer constructed a microstructure model of CN_x films based on data from other analytical techniques (*B33*). This model was employed to construct the C 1s spectrum, which agreed well with the experimental results.

Most of the chemical-state determinations made using XPS involve core-level data. However, scientists are increasingly using valence band (VB) spectra to determine the surface chemistry of materials. The increased popularity of VB data is driven by the proliferation of newer instruments which combine high spectral resolution with high sensitivity. Sherwood studied the electrochemical corrosion of nickel, molybdenum, aluminum, and carbon in an anaerobic cell (*B34*). Core and VB spectra were used to explain the surface chemical changes that occurred. The surface chemistry of La_2CuO_4 crystals was studied using VB photoemission by Zakharov et al. (*B35*). Use of the VB to characterize polymers is increasingly popular. Thomas and Fulghum characterized the surface of miscible and immiscible polymer blends using VB data (*B36*). Blends of polyolefin polymers were characterized using VD data by Galuska and Halverson (*B37*) and Schmitz et al. (*B38*). The interpretation of VB spectra is enhanced when molecular orbital (MO) calculations are used to simulate the spectra. The initial stages of Si oxidation were characterized using VB spectra and MO calculations (*B39*). The interactions between stearate-based lubricants and aluminum were measured by Havercroft and Sherwood (*B40*). Stearic acid and stearates gave characteristic VB spectra that were verified using MO calculations. VB spectra and MO calculations were used to characterize TiC, TiN, and TiCN specimens (*B41*). The VB spectra of 33 polymers were measured and compared to MO calculations (*B42*). Good agreement between theory and experiment was obtained.

As the probe size of XPS analysis shrinks, the problem of differential charging on insulating specimen increases. This is due to charges being concentrated into smaller areas, which results in larger charge gradients between charged and uncharged regions. Results of imaging XPS experiments on insulating specimens were reviewed by Coluzza and Moberg (*B43*). Tielsch and Fulghum examined differential charging effects on thin films grown on conductive and insulating substrates (*B44*). The nature of the substrate was the most significant factor influencing the differential charging of the coating. Larson and Kelly found that low-energy electron flood guns did not adequately control differential charging due to formation of a negative potential region surrounding the X-ray spot (*B45*). Addition of a low-energy positive ion flux eliminated this negative potential and improved the control of differential charging. Often, XPS data from insulating specimens must be charge referenced to allow meaningful comparison with reference data. Commonly, the C 1s spectrum from adventitious carbon is used for this purpose. Bhattachary et al. discussed an alternative approach to charge referencing using the O 1s

spectrum (*B46*). Superior results were obtained when compared to the C 1s referencing approach for data from a series of mixed metal oxide specimens. Decoration of the surface with 20-nm Au particles provided superior charge referencing when compared to C 1s results on aluminum oxide, hydroxide, and fluoride specimens (*B47*).

XPS is often used in the semiconductor industry to identify defects and to monitor surface and thin-film processing procedures. Most of this work is performed in a dedicated XPS instrument, but increasingly, XPS is being integrated into processing tools for on-line monitoring. Froeschle et al. integrated XPS into a rapid thermal oxidation tool (*B48*). The cleaning and oxidation steps were easily monitored and optimized using this approach. The optimization of a wafer dry-cleaning tool was facilitated using in situ XPS (*B49*). In this tool, an oxygen plasma removes fluorocarbon contamination while vapor-phase HF removes the resulting oxide leaving a clean surface.

Auger Electron Spectroscopy. AES is used to further materials research in many technical areas. AES characterization of optical materials (*B50*), photocathodes (*B51*), and biomaterials (*B52*) were reviewed. AES has made important contributions to the understanding of contamination segregation in metals. Measurements of antimony and tin segregation in steel were reviewed (*B53*). Walmsley et al. examined steel fracture surfaces using high spatial resolution AES (*B54*). They found tin segregated into small cavities while phosphorus was uniformly spread across grain surfaces.

One of the principle advantages of AES is high spatial resolution coupled with surface sensitivity. These attributes make AES an important technique for measuring contaminants and defects on semiconductor devices. Fillmore and Krasinski evaluated a defect review tool, an AES instrument designed to be integrated into a semiconductor processing line (*B55*). They found it to be a valuable tool for defect analysis and process monitoring. An in-line AES spectrometer was used to characterize the Si–SiO_2 interface in a plasma-assisted nitridation/oxidation process (*B56*). Kinsgley and Harris used Monte Carlo simulations to model the electron beam interactions with small particles (*B57*). Optimum experimental conditions for characterizing small particles were determined. Contamination buildup on side walls during resist etching was characterized by Ha et al. (*B58*). Various defects on magnetic hard disks were characterized using AES (*B59*). Cheng et al. combined high spatial resolution AES with a focused ion beam (FIB) to perform three-dimensional microanalysis of integrated circuits (*B60*). AES elemental maps were obtained after successive removal of thin layers with the FIB. Three-dimensional characterization of a W/TiN/Ti/Si contact structure was performed by Roberts et al. (*B61*). Werner et al. (*B62*) and Ide et al. (*B63*) developed methods for imaging p–n junctions in semiconductor devices. Small shifts in Auger line positions are caused by Fermi level differences between n- and p-doped Si. Arabczyk et al. developed a method for separating the surface and bulk components in an AES spectrum (*B64*). They take a weighted difference between spectra from a clean surface and a surface covered with a monolayer of absorbate.

Analysis of thin films and buried interfaces by sputter depth profiling continues to be an important application of AES. Recent work is aimed at improving the quantification and depth resolution

of the technique. Recent progress in sputter depth profiling was reviewed by Hoffman (*B65*) and Wetzig et al. (*B66*). Sulyok and Menyhard developed a sputter depth-profiling instrument with optimized depth resolution (*B67*). Grazing-incidence low-energy ion sputtering coupled with specimen rotation produces good depth resolution when characterizing buried layers. Hoffman and Rar employed SF_6 ions at low energy and grazing incidence to obtain high depth resolution profiles of GaAs/AlAs multilayers (*B68*). Sputter depth profiles were interpreted by comparing the data with dynamic TRIM simulations of model systems (*B69*). Werner et al. used a Ti_xC_{1-x} specimen with a concentration gradient to develop a calibration curve for quantification of sputter depth profiles (*B70*). The concentrations of unknown Ti_xC_{1-x} specimens could accurately be determined using this curve. Principle component analysis (*B71*) and Monte Carlo simulations (*B72*) were used to analyze depth profile data.

Analyses of line positions and line shapes in AES spectra continue to be used to determine the chemical state of elements on solid surfaces. Line shape changes in Si spectra were used to characterize surface hydrogenation of Si (*B73*) and plasma damage on ion-etched SiO_2 (*B74*). Zapata-Navarro et al. determined the oxidation states of Te in oxidized CdTe thin films based on changes in AES line shapes (*B75*). Nitration of GaAs was characterized by chemical shifts of Ga, As, and N Auger transitions (*B76*). Molybdenum silicides were detected at the Mo/Si interface using linear least-squares fitting and target factor analysis of depth profile data (*B77*).

OPTICAL CHARACTERIZATION OF SURFACES

Optical measurements have proved to be a valuable method of surface characterization. Raman scattering and infrared (IR) spectroscopy measurements of the vibrational modes have become the most used optical methods of characterizing surface properties. The application of these techniques to thin film and surface structures is often difficult because of sensitivity limitations of the techniques. Other techniques such as ellipsometry, reflectance difference spectroscopy, and nonlinear laser techniques are also finding application in the characterization of surfaces. Continued advancement of the optical measurements has been stimulated by the advantage of nondestructive and noncontact measurements which are often relatively straightforward to interpret. While disordered and microstructured surfaces are important for many applications, the fundamental processes are most often demonstrated at ordered surfaces, and as such, the structures at ordered surfaces will be emphasized here. This review will highlight new developments of optical characterization of surface processes on various semiconducting, insulating, and metallic surfaces.

While surfaces of silicon have been well studied, the importance of integrated circuit technology (IC) has led to renewed interest in the Si surface under various process-related conditions. One of the most common process steps in integrated circuit fabrication is the water rinsing of the Si wafer. Several studies have employed surface IR absorption spectroscopy to examine this process on Si(100) surfaces (*1−4*). For H_2O exposure and annealing, the results indicate that an oxide forms through initial insertion into the dimer bonds followed by insertion into the backbonds (*C1*). Detailed studies of the annealing behavior indicates that the wet oxidation process involves attack by atomic oxygen

and hydrogen desorption and that intermediate oxidation species were observed (*C2*). Other IR studies suggest that the initial oxidation in pure and ozonized water was not through a layer-by-layer process, but island formation was indicated (*C3*, *C4*). The step edge structure of H-terminated Si(111) vicinal surfaces was studied by IR spectroscopy for solution-prepared surfaces in which the electric field was controlled (*C5*). The results addressed conflicting prior studies. The vibrational dephasing dynamics on the Si(100)-2 × 1:H surface was studied, and the results indicated multimode dephasing (*C6*). The accuracy of contact angle measurements to determine H termination of HF-etched surfaces was determined through comparison with IR measurements (*C7*). It was found that the contact angle measurements consistently lagged behind the IR measurements of the surface concentration.

Light-emitting porous Si surfaces are typically produced by roughening in a fluoride solution. Recent IR measurements indicate that the SiH/SiH_2 ratio on the surfaces was constant and independent of doping, surface orientation, and roughening (*C8*). Deuterium incorporation may result in improved stability of Si surfaces and interfaces, which will be important for integrated circuit devices. Results of the D-to-H exchange rate have been obtained through IR spectroscopy (*C9*). New devices based on SiGe/Si heterostructures are being considered for several electronic applications. Results show that IR of H-terminated surfaces can be used to monitor the surface structure and surface concentrations (*C10*). IR measurements have also been used to examine the absorption of butadienes (*C11*), $H_8Si_8O_{12}$ clusters (*C12*), and benzene (*C13*) on Si surfaces.

The optical spectra of Si surfaces are employed in spectroscopic ellipsometry measurements. The observed critical point energy shifts in Si have been attributed to near-surface localization of excited electronic states which are related to the surface structure and surface termination (*C14*). Variable-angle spectroscopic ellipsometry can also be used to determine depth profiles of the free carrier concentration (*C15*).

Reflectance difference spectroscopy has been employed to distinguish the different reconstructions of cubic 3C−SiC(001) surfaces (*C16*). The results indicate the presence of surface states. IR spectroscopy has been employed to characterize the formation of SiC on Si through the carbonization of the Si surfaces by C_2H_2 in a gas source molecular beam epitaxy (*C17*). Raman spectroscopy has been employed to examine the strain and strain relaxation at the 3C−SiC/Si interface (*C18*).

Raman scattering (RS) is a key technique for the characterization of diamond thin films. Two studies have addressed issues related to the use of RS in this application. The RS of graphite edge planes showed shifts of the ∼1350-cm^{-1} disorder peak (*C19*). In another study, the RS of amorphous sp^3-bonded carbon (termed amorphous diamond) has been reported (*C20*). The reduced RS agrees closely with the calculated density of states of diamond. Confocal RS has been employed to examine the initial nucleation of diamond on Pt(111) and Ni substrates (*C21*, *C22*).

Raman scattering continues to be an important technique for materials and process characterization for compound semiconductors. RS has been employed to study the replacement of P by As for As-exposed InP surfaces (*C23*). Similarly, the structure of GaAs under the simultaneous presence of Sb and Bi was reported (*C24*). The free carrier concentration of Si-doped InP was determined

with micro-RS by examining the shift of the coupled plasmon-LO-phonon mode (*C25*). Using the same effect, the p-type accumulation zones have been studied for ZnSe/GaAs heterostructures (*C26*). Raman scattering has also been used to monitor the MBE growth of wide band gap semiconductors (*C27*). The interface modes of thin heterostructures can also be studied with RS. In particular, the RS and IR techniques were combined to examine the modes of GaN on GaAs (*C28*). RS has been used to monitor the deposition of *C60* on Si and GaAs surfaces and also to explore the phototransformation of the C_{60} thin films (*C29*, *C30*).

Optical spectroscopy is an important technique for the characterization of compound semiconductors. The far-IR spectra have been examined theoretically. The analysis explores the direct photon−phonon coupling at (001) surfaces of zinc blend crystals, and the calculated reflectance spectra display resonance structure (*C31*). The line shapes observed in reflectance−difference spectroscopy have been analyzed in terms of excited states localized near the surface (*C32*). The effectiveness of several optical techniques has been compared for measurement of the surface electric field in GaAs (*C33*). The technique of chemical modulation spectroscopy (CMS) has been applied to strain layers of GaAs on GaP (*C34*). The technique involves changing the coverage of the surface and measuring the change in reflectance. In other applications, reflectance anisotropy spectroscopy (RAS) was used to study C doping of GaAs (*C35*), photoreflectance was used to examine the growth mode of InAs−GaAs monolayer quantum wells (*C36*), and in situ UV ellipsometry was used to follow the decomposition at GaAs cathodes (*C37*). In related materials, the growth of thin Al films on various surfaces has been explored with in situ IR spectroscopic ellipsometry (*C38*).

In a unique study, polarized IR spectroscopy was employed to identify the formation of a two-dimensional quantum crystal of H_2 on NaCl (*C39*). Multichannel FT-IR spectroscopy has been applied to examine real-time crystal regrowth (after laser melting) (*C40*). The technique demonstrated millisecond time resolution.

The technique of reflection−absorption IR spectroscopy (RAIRS) has proved particularly useful for characterization of molecular adsorbates on metal and other surfaces. Technical advances have allowed measurement of RAIRS below 77 K (*C41*). For CO on Pd(100), a significant interaction between the physisorbed and chemisorbed layers is observed. The temperature dependence of the C−O stretch has been measured for CO on Ru(001) (*C42*). Results indicate that the anharmonic coupling of the C−O stretch mode is largely independent of structural properties of the surface. The IR spectra of CO and O coadsorbed on Ru(001) has been measured to determine the relative changes of the vibrational polarizability and the dielectric constant (*C43*). In studies of the adsorption of HCN on Pt(111) and Cu(100), significantly different chemical and surface transitions are observed, but the IR spectra are very similar for both surfaces (*C44*). In a study of CO on Ni(111), sum-frequency generation and RAIRS was used to explore the occupation of CO on both on-top and bridge sites (*C45*). IR measurements of CO adsorbed on hydrocarbon-covered Mo(100) indicated adsorption on the catalyst despite the thick carbonaceous layer (*C46*). Synchrotron FT-IR has been applied to study the adsorption of water on Ni(110) (*C47*). Several different transitions are observed as a function of temperature.

In a study to explore the growth of AlN materials, the interaction of ammonia with hydrogenated and clean Al(111) was studied by RAIRS (*C48*). Significant differences were found for the H-terminated surface.

IR reflection−absorption spectra were obtained in situ for adsorption in aqueous solutions. Results were also compared with UHV environments for CO on Ir(111) (*C49*). For CO on Pd(111) in the aqueous interface, evidence for an adsorbate-induced surface reconstruction was presented (*C50*). A change of the surface reconstruction was also observed for adsorbed phosphate species on Au(100) surfaces (*C51*).

IR spectroscopic techniques were also applied to larger molecular adsorbates. On Pt(111) surfaces, the following processes were studied: the melting of rodlike molecules (*C52*), the thermal decomposition of trimethylamine (*C53*), and the site preferences of CH_3Br (*C54*). For other systems, the adsorption of the following systems has been reported: 1-hexene on Ru(0001) (*C55*), methoxy decomposition on oxygen-covered Mo(110) (*C56*), acrolein on gold (*C57*), cyclopropane on Ni(111) and Cu(111) (*C58*), and glycine on Cu(110) (*C59*).

Surface-enhanced Raman scattering (SERS) has long been an important method for examining surface adsorption. The SERS technique has been used in nonresonant conditions to observe colorless, single biomolecules (*C60*, *C61*). The SERS process has been combined with the scanning probe technique to obtain Raman imaging of DNA with 100-nm resolution (*C62*−*C64*). While SERS has been useful for studies of adsorption on Ag, Au, and Cu, the combination of a confocal microscope and careful surface treatments can allow more general application of RS for studies of adsorbates on a wider range of surfaces (*C65*).

X-RAY TECHNIQUES

Appearance Potential Spectroscopy. For the study of the valence band states of atoms in the surface region, analytical techniques typically employ particles of low enough energy to probe only a few tenths of a nanometer into the solid. Among the techniques used to study the electronic structure of the surface, X-ray absorption (XAS), Bremsstrahlung Isochromat (BIS), inverse photoemission (IPE), and appearance potential (APS) spectroscopies have proved to be the most promising approaches. This review focuses on the various aspects of APS that apply to materials characterization of surfaces.

Appearance potential spectroscopy measures the probability of excitation of core electrons of the surface atoms as a function of incident electron energy. Unlike photoemission techniques, which utilize a one-electron excitation process to give the density of occupied states, APS is based on the two-electron excitation/relaxation process and provides information on the convoluted density of unoccupied states. The experiment consists of measuring the changes in the total X-ray fluorescence or secondary electron yield by a potential-modulation technique. When the incident electron energy equals the threshold of the excitation of a core level, both the incident and core electrons are scattered to unoccupied states above the Fermi level, E_F, with the creation of an inner hole. The excitation process is followed by the filling of the core hole, which causes X-rays or Auger electrons to be emitted. When the intensity of the emitted X-rays is measured, the method is called soft X-ray APS (SXAPS). Measuring the total

secondary electron current is called Auger electron APS (AEAPS). In another modification of the technique, the current of elastically reflected electrons is measured. At the threshold energy of core-level excitation, the number of electrons that undergo inelastic scattering disappear from the measured current. The method is appropriately called disappearance potential spectroscopy (DAPS). The excitation process is the same for all APS spectra. It seems that differences in the decay step are responsible for the differences in the features among SXAPS, AEAPS, and DAPS. Based on this model, the information obtained from DAPS is free of the relaxation complications. SXAPS and AEAPS, however, include additional information dependent on their respective decay steps. Because of the threshold energies used in APS, the inelastic interaction excludes the incident electron from the complications of excitation and transport processes owing to many possible electron interactions. Another important aspect of APS is that it reveals a localized density of states (DOS) because the matrix element governing the core hole production involves the very short range wave function of the initial core electron state. The intensity of the signal depends on the density of conduction band states. If the DOS is low, the signal obtained in the spectrum will be poor. Therefore APS cannot be effectively employed as a universal technique for surface analysis. The applicability of AEAPS as an analytical tool for determining the surface composition and depth profiling in a manner similar to AES is therefore limited. APS provides information regarding the elemental identification, chemical bonding, density of unoccupied states, nearest-neighbor configuration in the surface layer, and mechanism of excitation and transport processes occurring in the near-surface region of the sample. In this respect, APS, AES, and XPS are complementary techniques. Several review articles have been published highlighting the applications of APS (D1).

Ranglov et al. (D2) describe a high-performance detector for soft X-ray appearance potential spectroscopy. It consists of an alkali halide photon-to-electron converting layer evaporated onto a self-supporting amorphous C foil. This combination absorbs the low-energy photons thereby increasing the signal-to-noise ratio. Zhang et al. (D3) studied the sensitivity of the appearance potential spectroscopy for lanthanides, actinides, and 3d transition metals. The results indicate that higher sensitivity is correlated with large unoccupied density of states in these elements. Stanica and Osiceanue (D4) used the deconvolution method to derive the density of unoccupied states in Ti, Cr, Fe, and Ni from the experimental soft X-ray appearance potential spectra of their $2p_{3/2}$ core levels. The higher 4s states exhibit a bandlike character. The data processing is based on a least-squares method using spline function formalism. Although APS essentially probes the localized density of states, there is a good agreement with single-particle band structure calculation. Osiceanu and Vass (D5) obtained the L-shell appearance potential spectra of Fe, Cr, and Ni of the surface of "304" stainless steel on an "as-received" sample and after "in situ" thermal treatments in the range of 250−1200 °C. The results suggest a surface composition that is depleted in Cr, emphasizing the observation that stainless steel surfaces subject to high temperature in a vacuum tend to lose their corrosion resistance. The spectra deconvolution reveals a 3d band structure of the unoccupied states nearly unaffected by alloying.

Reinmuth et al. (D6) studied the electronic structure of magnetic compound $FeNi_3$ by spin-resolved soft X-ray appearance potential spectroscopy. The spectra reflecting the local densities of empty states on the Fe and Ni sites in this compound are compared with results for the pure elements. The experimentally observed energetic shifts and spin-asymmetry changes between the pure elements and the compound are understood as a consequence of changes in the spin-dependent local densities of states and in the core-level binding energies. The changes in binding energies for the $2p_{3/2}$ states of Fe and Ni in the compound with respect to the pure elements were calculated. Henig et al. (D7) studied the one- and two-electron excitation of core electrons into unoccupied states of MnO (100) by appearance potential spectroscopy at the metal 2p and ligand 1s thresholds. The number of the structures in the APS spectra at the O 1s threshold exhibits no direct influence of the electron correlation, because all dominant peaks can be explained by a one-electron transition of the O core electron into unoccupied Mn 3d, 4s and 4p states. But in the APS spectra at the Mn 2p threshold, the authors find two-electron excitations which are absent in the one-electron excitation spectra. Tilinin et al. (D8) studied the elastic contribution of Auger electrons to appearance potential spectroscopy both experimentally and theoretically. They used the overlayer technique to study the mean escape depth of Auger electrons escaping from a target without losing a considerable amount of their kinetic energy. It was found that due to the attenuation of the primary beam the mean escape depth of the elastic APS signal electrons turned out to be smaller, by a factor of 2, than that of the same Auger electrons in the usual AES. As a result, the surface sensitivity of the high-energy APS may be higher by a factor of ~2 compared to AES.

Rangelov and Dose (D9) studied the unoccupied density of states of diamond films by appearance potential spectroscopy. The experimental spectra show reasonable agreement with the spectra calculated using a theoretical unoccupied density of states. The gradual transformation of the diamond films to graphite by sputtering and annealing was monitored by APS allowing a clear differentiation between sp^3-hybridized C (diamond) and sp^2-hybridized C (graphite). Gorovikov et al. (D10) studied the C 1s-level appearance potential spectra of Yb/C_{60} system to obtain the unoccupied C-derived density of states. Step-by-step deposition of Yb onto the C_{60} film at room temperature results in the formation of a fulleride-like compound in the surface region, and the evolution of unoccupied density of states of C_{60} under doping by the Yb atoms can be described from the positions of charge transfer from Yb into the LUMO-derived band of C_{60}. Slezak et al. (D11) investigated the contributions of electrons with different energies to Auger electron appearance potential spectroscopy. The effective dimensionless coefficient σ_{eff}, representing the number of slow electrons emitted at the disappearance of one elastically reflected electron at the threshold potential of Ti, Cr, Mn, and Ni, ranges from 2.7 to 3.5 for $L_{2,3}$ subshells and from 9 to 11 for L_1 subshells. These results have been accounted for in terms of fast, highly probable Coster−Kronig transitions which turned out to be the only possibility for relaxation of the L_1 subshells of the materials under investigation. Ebert and Popescu (D12) present a theoretical description of spin-resolved appearance potential spectroscopy based on a single-particle description of the underly-

ing electronic structure. The final expression for the signal intensity is a cross section-weighted self-convolution of the density of states above the Fermi energy. Application of the formalism to bcc Fe and fcc Ni leads to results in good agreement with corresponding experimental data. Reinmuth et al. (D13) also studied the temperature-dependent magnetic properties of the FeNi$_3$(111) surface by spin-resolved appearance potential spectroscopy in the temperature range from 100 to 1100 K. Two phase transitions occur in the bulk: a compositional order−disorder phase transition between 770 and 785 K and a ferromagnetic−paramagnetic phase transition with a Curie temperature of 863 K. The first one does not show up in the appearance potential signals of Fe and Ni while the latter is reflected in the spin asymmetry signals. Both the Fe and the Ni signals follow the bulk magnetization curve of FeNi$_3$. The spin asymmetry of the appearance potential signal is observed to be proportional to the magnetization.

Glancing-Angle X-ray Diffraction. X-ray diffraction is a versatile analytical technique for determining the atomic structure in crystalline solids, which include metals, ceramics, geological materials, and organics (D14). The near-surface crystal structure of materials is characterized by positioning X-ray sources at a glancing angle with respect to the sample. Glancing-angle X-ray diffraction, which enhances the surface sensitivity of materials under investigation, is used to examine polycrystalline films typically no thinner than 500 Å. The glancing angle affects the depth to which the X-ray beam penetrates the sample. Glancing angle X-ray diffraction thus differs from the conventional X-ray diffraction in that it provides information about crystallinity for materials at surfaces.

Dudchick et al. (D15) describe the calculation procedures and experimental results from glancing-angle X-ray fluorescence from thin films on a flat substrate. A new X-ray tube unit with a super-smooth-surface anode and built-in waveguide collimator is described.

The unit is capable of providing narrowly collimated beams of X-ray radiation with a microfocus line. Liu et al. (D16) developed the total-reflection-angle-X-ray spectroscopy system for reflection high-energy diffraction experiments. Applying the glancing-angle X-ray diffraction to the surface layer analysis for the SrTiO$_3$(001) single crystal, they found that the critical angle of total reflection can be used to determine the composition of the surface atomic layer. Emato et al. (D17) developed a X-ray diffraction technique to measure the strain field near a solid surface. The glancing angle of the X-rays can be set near the critical angle of total reflection by tuning the X-ray energy. The widths of the rocking curves for Si surfaces were used to distinguish the strain field corresponding to each surface structure. Fujii et al. (D18) report the development of a compact ultrahigh vacuum X-ray diffractometer for surface glancing scattering using a rotating-anode source. The apparatus is best suited to the in situ observations of growing crystal structures during the deposition. A preliminary experiment of the glancing angle scattering of polished Ag polycrystal surface gave an order of 10^3 counts s^{-1} of scattered X-ray intensity, which is enough to derive a significant interpretation of the surface structure. Noma and Iida (D19) employed the grazing-exit conditions to optimize the spatial resolution and the surface sensitivity of Pd, Pt, and their layered structure. Using a small

exit angle, the signal-to-background ratio was improved due to a shallow escape depth. Under the grazing-exit condition, the refraction effect of diffracted X-rays was observed, indicating the possibility of surface sensitivity.

Kapil et al. (D20) characterized the diamond thin films using glancing angle X-ray diffraction and micro Raman spectroscopy techniques. The relative growth of various polytypes was found to be a strong function of acetylene to O flow ratio, speed of rotation, and substrate materials. Dekempeneer et al. (D21) deposited transparent boron nitride coatings of hardness values ranging from 2 to 12 Gpa on glass and Si substrates by varying the plasma conditions. A combination of glancing-angle X-ray diffraction measurements shows that the coatings consist of hexagonal-types BN crystallites with different degrees of disorder. Losbichler et al. (D22) characterized the Ti−B−N films deposited on austenitic steel and molybdenum sheets by using glancing-angle diffraction, scanning (SEM) and transmission microscopy (TEM), and other related techniques. The composition of the films was found to lie on the quasi-binary section TiN−TiB$_2$ within the ternary system Ti−B−N. Coatings consisted of noncrystalline fcc and hcp Ti−B−N phases. Microstructure as well as chemical composition of the films does not seem to be influenced by varying the deposition parameters used in the investigation. Contrarily, the hardness was strongly influenced by varying the ion bombardment. Gust et al. (D23) have grown the barium titanate (BaTiO$_3$) thin films onto (100) Si substrates by spin coating. The crystalline behavior of the amorphous-gel films was characterized using glancing-angle diffraction and other techniques. Amorphous gel crystallized at a temperature of ∼600 °C to an intermediate nanoscale barium titanium carbonate phase, presumably BaTiO$_2$-CO$_3$, that subsequently transformed to noncrystalline BaTiO$_3$. Random nucleation in the bulk of the gel film was observed on all substrates. Sengupta et al. (D24) characterized the thin films (oxide III composites with barium strontium titanium oxide) using glancing-angle X-ray diffraction, Fourier transform Raman spectroscopy, and Fourier transform IR spectroscopy. The amount of the oxide additive in the BSTO matrix was varied from 1 to 60 wt %. The thin-film study has applications in higher microwave frequency antennas.

Bassani al. (D25) studied the properties of noncrystalline Si/CaF$_2$ multilayers, grown by MBE by glancing-angle X-ray diffraction. The materials exhibit visible luminescence at room temp. The dimensions of the Si grain within the Si layers were determined to be ≤1.5 nm. Tamisier et al. (D26) studied Au (47 Å)/Ni(31 Å) 12-layer films by glancing-angle X-ray diffraction, high-resolution TEM, and EXAFS. The film growth is accompanied by strong deformation of the Au and Ni crystal lattice, implying strong compressive stresses for the Au and tensile stresses for the Ni films. Ni diffusion in the Au films was detected. Cantalini et al. (D27) investigated the microstructure and the electrical properties of thermally evaporated WO$_3$ thin films by glancing-angle X-ray diffraction and other techniques. The as-deposited films were amorphous. After annealing at 500 °C in dry air, the films exhibited marked crystalline structures with preferential orientations of WO$_3$ in the (200) direction. The 24-h annealed film yielded a O/W ratio close to 2.9 which is in good agreement with the theoretical one. The film annealed at 500 °C for 24 h in the presence of NO$_2$ gas showed better electrical properties. Perry

et al. (*D28*) studied the residual stress and strain distribution in the surface of the ground cemented carbide before and after metal ion implantation using glancing-angle X-ray diffraction. The grinding operation introduces tensile stresses into the WC and cubic carbide phases extending down to a depth of ~0.2 μm, with parallel increase in the strain distribution. The effect of a Ni−Ti dual metal ion implantation has been extensively studied.

Liu et al. (*D29*) used glancing-angle X-ray diffraction to study the phase transition temperature and structure parameter of the surface layer, which differs from those of the bulk layer in polycrystalline $PbTiO_3$ ferroelectric thin film. The surface layer was characterized as fine crystal and low stressed; the bulk layer was characterized as large crystal and high stressed. Masion et al. (*D30*) studied the nucleation and growth mechanisms of iron oxyhydroxides in the presence of PO_4 ions by small-angle X-ray scattering. At a hydrolysis ratio R ([OH]/[Fe]) of 1.0, 60% of Fe are Fe monomers and 40% are edge-sharing dimers. At higher R, the aggregates consist essentially of edge-sharing Fe dimers (>84%). The Fe speciation derived from the modeling of the scattering curves confirmed and refined the results obtained by EXAFS technique.

Venugopalan et al. (*D31*) analyzed the interfacial reactions between nickel thin films and GaN using glancing-angle diffraction and Auger depth profiling. On healing at 600° for 1 h, Ga was dissolved in the fcc Ni film. After annealing at 750 °C for 1 h in N_2 or Ar, the intermixing increased. The reaction product was either Ni_3Ga or fcc Ni with dissolved Ga. Annealing at 900 °C gave the B2 phase NiGa. Calais et al. (*D32*) employed X-ray diffraction and related techniques to determine the crystallite sizes of MOS_2 catalyst particles and compared the results. The samples were prepared by thio salt decomposition or molten salt synthesis. These solids presented different morphologies; the latter preparation method provided samples with higher surface area.

Shimizugawa et al. (*D33*) investigated the structure of TeO_2−ZnO glasses with three different ZnO contents by X-ray diffraction using synchrotron radiation. The study suggests that the weak Te−O bond at 0.289 nm seen in α-TeO_2 crystal does not exist in glass structure.

Extended X-ray Absorption Fine Structure. The phenomenon of fine structure in the absorption coefficient in the first few hundred electronvolts energy range above the absorption edge has been exploited to determine atomic structure of ordered and disordered materials. Structures near the absorption threshold are known as X-ray absorption near-edge structures (XANES), while the features at higher energies are EXAFS. EXAFS techniques have had a major impact on the understanding of the structure of biological molecules, the structure of amorphous semiconductors and insulators, the structure of supported catalysts, and local structural arrangement in complicated crystals. There are many aspects of EXAFS techniques, relative to X-ray and neutron diffraction techniques, that should yield unique information about materials.

Core-level ionization in an atom by absorption of a photon with sufficient energy creates a photoelectron that propagates as a spherical wave. Scattering of this wave by the neighboring atoms causes interference effects that depend on the local geometry and the incident X-ray energy. This interference is reflected as a modulation in the absorption coefficient on the high-energy side

of the absorption discontinuity in the X-ray absorption spectrum. The term extended X-ray absorption fine structure refers to these oscillations, which may have a magnitude of ~10% of the absorption coefficient in the energy region above the edge. Fourier transform analysis of EXAFS yields structural information regarding the nearest-neighbor distances, coordination numbers and types of nearest neighbors. Contrary to XRD and low-energy electron diffraction (LEED) methods, which are only applicable to a structure with long-range order periodically, the EXAFS can be used for structure analysis in short-range order and disordered systems such as real catalysts. EXAFS probes out to ~6 Å in the immediate environment around each absorbing species. Information from the atoms near the surface can also be obtained with surface EXAFS (SEXAFS) for systems exhibiting a large surface-to-volume ratio, e.g., small catalyst particles. SEXAFS has been used to determine the location and the bond length of the absorbed atom on clean single-crystal surfaces.

An EXAFS experiment requires a very accurate determination of the X-ray absorption coefficient. High-intensity and tunable X-ray sources must be utilized in order to achieve this determination. The advent of synchrotron radiation and rotating anode X-ray tubes has speeded up the progress in the field. Synchrotron radiation is used in EXAFS measurements because it provides an intense beam of variable-energy, monoenergetic photons. The ability of EXAFS to determine the local structure around a specific atom has been used in the study of catalysis, multicomponent alloys, disordered and amorphous solids, metastable systems, and dilute systems having a very low concentration of elements of interest. As the availability of synchrotron sources increases, EXAFS will take its place alongside X-ray, neutron, and low-energy electron diffraction techniques as another powerful tool for determining atomic structure of ordered and disordered materials. Several authors have recently reviewed the application of EXAFS to study the geometric and electronic structures of materials (*D34*).

Yiwata et al. (*D35*) carried out the V K-edge EXAFS measurements of crystalline V_2O_5 and four types of $Li_xV_2O_5$ with different insertions levels x. The data show no remarkable difference in the V−O distances in the five compounds, showing that the structure of VO_5 square pyramids changes only slightly as Li atoms are intercalated into the V_2O_5. Polcik et al. (*D36*) performed the polarization-dependent surface-extended X-ray absorption fine-structure experiments on Cu (21O)−(1 × 2)-O. The analysis shows that oxygen chemisorbs in long-bridge sites along the [001] direction with nearest-neighbor and next-nearest-neighbor O−Cu distances of 1.81 and 1.91 Å, respectively.

EXAFS measurements were performed at the In K edge, on devitrified $Ge_5Se_{80}In_{15}$ and on a series of vitreous Ge−Se−In alloys by Ledru et al. (*D37*). The results indicate that the In atoms have essentially the same local surroundings in all the samples investigated. The In atom is surrounded by three Se atoms at distance of ~0.259 nm and form In_2Se_3 monoclusters. Yao et al. (*D38*) studied the perovskite oxide with the composition $La_{1-x/2}Y_xSc_{1-x/2}$ crystal structure by the Rietveld method and EXAFS measurements. Nearly even distribution of the Y^{3+} ion between a-site and B-site was confirmed by EXAFS analysis. Y−O bond lengths measured by the Rietveld method deviated from those obtained by EXAFS. The results suggest that BO_6 octahe-

drons are the principal skeleton of the perovskite crystal lattice and doping of Y^{3+} causes distortion of arrangements of the BO_6 octahedrons. Roubin et al. (D39) investigated the EXAFS at the K edge of S atoms of OCS isolated in an Ar matrix. The Ar atoms are located at a mean distance of 3.78 Å from the S atoms, with a root-mean-square variation of the S−Ar distances of ∼0.08 Å. Results indicate that the host atoms occupy a statistical set of positions around the molecule, rather than well-defined sites. Saplekin et al. (D40) studied the structure of bulk amorphous GaSb using the EXAFS technique to explore the chemical disorder over the temperature range from 80 to 273 K. The configurational contribution to the Debye−Waller broadening of the distances has been extracted from the EXAFS data. The bond-angle distribution for a-GaSb was calculated from the EXAFS analysis and the mean bond angle has been calculated from the Sb K-edge data to be 108.9°. Choy et al. (D41) systematically performed EXAFS studies on the new mercuric halide intercalated super-conductors, $(HgX_2)_{0.5}Bi_2Sr_2CaCu_2O_7$ with X = Br and I, to study the electronic and crystal structures of these compounds. According to the I L_1- and Br K-edge XANES spectroscopic analyses, a small amount of electron is transferred from the host lattice to the intercalant HgX_2, resulting in the negatively charged slate of $(HgX_2)^{\delta-}$. The Cu K-edge EXAFS analysis reveals that the bond distance of (Cu−O_{axial}) is slightly shortened upon intercalation, reflecting the oxidation of CuO_2 layer, which is consistent with the I L_1- and Br K-edge XANES results. The Hg L_{111}-edge EXAFS results indicate that the Hg is coordinated with two bromide and iodide ligands with bond lengths of 2.46 and 2.65 Å, respectively. This is the first example of linear molecular salt HgX_2 stabilized in the solid state. Ridgeway et al. (D42) determined the structural parameters of stoichiometric, amorphous GaAs using EXAFS experiments performed in the transmission mode at 10 K. Amorphous GaAs exhibited increased nearest-neighbor bond length and a Debye−Waller factor relative to a crystalline sample. In contrast, the coordination numbers about both Ga and As atoms in the amorphous phases decreased to ∼3.85 atoms from the crystalline value of 4. All structural parameters were independent of implantation conditions. Woicik et al. (D43) employed the high-resolution EXAFS to determine the bond lengths in strained, buried $Ga_{1-x}In_xAs$ thin alloy films grown coherently on InP (001). Comparison with random-cluster calculation demonstrates that the external in-plane epitaxial strain imposed by pseudomorphic growth opposes the natural bond-length distortions due to alloying. Gota et al. (D44) used the SEXAFS to determine the growth law of subanometric particles. They demonstrated the radius range of 2−10 Å in the case of copper deposited on alumina. The scaling is explained by the coalescence of copper clusters, as in breath figures due to condensation of liquid droplets.

Pintar et al. (D45) characterized the γ-Al_2O_3-supported Pd−Cu bimetallic catalysts by EXAFS, AES, and XRD. The catalysts were tested for the selection hydrogenation of aqueous nitrate solutions to N. The reaction selectively depended on the catalyst preparation procedure, which affects the spatial distributions of metallic Cu and Pd phases. The EXAFS investigation confirms no significant structural differences among catalyst samples synthesized, which results in identical activity for nitrate removal. Lu (D46) studied the structure of liquid GaSb and InSb by EXAFS. The atomic pair distributions and the statistical distribution of the

coordination numbers were obtained. The results demonstrate that for GaSb and InSb melting destroys the covalent network, and this structural change causes the metallic behavior of liquid GaSb and InSb. Zubavichus et al. (D47) studied the local structures of host and guest layers of MoS_2 intercalated with $M(OH)_2$ (where M = Mn, Co, Ni) by EXAFS. The electronic structure and atomic environment of the M atoms in the intercalates are found to be similar to that of crystalline hydroxides $M(OH)_2$. In the Ni intercalate, Mo K-edge EXAFS revealed a structural change of the host MoS_2 layer similar to that reported for water dispersions of MoS_2 single layers. S K-edge XANES data indicate that the change is associated with increased electron density on the S atoms in the matrix. Wei et al. (D48) investigated the local structure of Fe^{3+} in $FeCl_3$ solutions by EXAFS. The results show that there are evident differences in the radial distribution functions of Fe^{3+} for various concentrations. The analysis of the data indicate that Fe^{3+} in 0.2, 0.8, and 3.0 mol/L $FeCl_3$ solution is coordinated by six O, one Cl, and five O atoms and two Cl and four O atoms, respectively. Cobalt silicide thin films prepared on Si(100) wafers were studied by EXAFS and other related techniques at the Co K edge by Naftel et al. (D49). XANES and EXAFS provide information about the electronic structure and the morphology of the films. The films studied have essentially the same structure as bulk $CoSi_2$. Both the spectroscopy and materials characterization aspects of EXAFS are discussed. Gehanno et al. (D50) studied the long-range order and short-range order of magnetic $Fe_{0.5}Pd_{0.5}$ epitaxial thin film by XRD and polarized fluorescence EXAFS. The alloys were prepared by co-deposition in the temperature of 10−700 K. The (001)-oriented thin films have a face-centered tetragonal structure flattened along the growth direction. The results indicate that the best ordered sample (both on a long-range and short-range scale) is the one co-deposited at 600 K. Zhu et al. (D51) used EXAFS, XRD, and XPS techniques to study the chemical forms of nitrogen and iron species during iron-catalyzed formation of N_2 during carbonization of polyacrylonitride. A solid-phase reaction mechanism was predominant, in which the interaction between iron species and hetrocyclic nitrogen structures results in the formation of iron nitrides, which then decompose to form N_2. Pyridinic rather than pyrrolic nitrogen is preferentially converted to N_2. There was countermigration of iron and nitrogen between the surfaces and bulk which favors their full contact and interaction and subsequent formation of N_2.

Schutz et al. (D52) demonstrated the potential of EXAFS to study magnetic and crystallographic structures and the low-energy exchange scattering process quantatively. The studies are hampered by the occurrence of strong magnetic multielectron effects and low frequent atomic contributions as well as the strongly enhanced magnetic multipole scattering. The authors suggest that by introduction of an additive spin-dependent part of the complex scattering amplitude for a spin-polarized outgoing photoelectron wave, a reasonable interpretation of the experimental data is possible in a first approximation.

Wang et al. (D53) report the first spin-polarized SEXAFS of an antiferromagnetically coupled system using K_β detection. The K_β spin-polarized SEXAFS effect is much larger than the K-edge XMCD spin-polarized SEXAFS because of the greater spin polarization of the excited photoelectrons. This internally refer-

enced technique is suited for antiferromagnetically coupled systems, as well as ferromagnetic and paramagnetic systems. Ahlers et al. (*D54*) obtained additional strong dispersive structures at the Gd, Tb, and Ho L edges within 200 eV above the absorption threshold on the magnetic or spin-dependent EXAFS. These features are interpreted as magnetic multielectron excitations. The authors find it feasible to separate magnetic multielectron excitation structures from the overlapping strong magnetic EXAFS for the first time and therefore obtain new insight into the physical origin of these effects.

ACKNOWLEDGMENT

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A19900159