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SURFACE CHARACTERIZATION:
PRESENT STATUS AND THE NEED FOR STANDARDS*

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A summary is given of the present status and use of surface-characterization measurements in the United States. Attention is primarily devoted to those properties needed to characterize a solid surface, specifically the determination of surface composition, surface atomic structure, surface electronic structure, and atomic motions on surfaces; these characteristics directly affect many important surface properties or processes that occur on surfaces (e.g., electrical and optical properties, adhesion, bonding, catalytic activity, plating, durability, corrosion, decoration, segregation, lubrication, and reactivity). The above four forms of surface characterization are widely utilized in surface-science experiments while measurements of surface composition are routinely made to solve a wide variety of problems in the semiconductor, chemical, petroleum, and metals industries for applications ranging from process and device development, process control, process evaluation, to failure analysis. Surface-characterization measurements in government laboratories support a variety of agency missions. Surface science and surface technology have both grown rapidly in the past ten years, and further growth is expected. At this time, there is an almost complete lack of standard, standard procedures, and standard materials to support surface-characterization measurements. A new Committee on Surface Analysis has been recently formed by the American Society for Testing and Materials to develop standard for all methods of surface analysis in common use. Examples are given of the standards that need to be developed.

1. Introduction

Surface properties are not only an integral part of our culture ("beauty is only skin deep") but impact considerably on the properties of materials and the development of new technologies important for an industrial society. Processes and properties such as catalysis, adhesion, lubrication, durability, bonding, oxidation, passivation, plating, epitaxy, coating, doping, and crystal growth depend critically for their success or failure on surface characteristics. Likewise, the minimization of material or device failure (i.e., product reliability) can depend on the reduction of corrosion or wear, the absence of surface contaminants in the fabrication of semiconductor devices, or the nonoccurrence of embrittlement in metals caused by segregation of impurities to interfaces.

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* Invited article.

The present status of methods for surface characterization and the need for standards in the future have been described in a recent report [1]. This report is one of a series devoted to an analysis of the National Measurement System (NMS) in the United States. The concept of a NMS was introduced by Huntoon [2] to describe the vast, complex system of measurements that effects every sector of an industrialized nation. According to Huntoon, the essential function of the NMS is to provide a quantitative basis in measurement for (i) interchangeability and (ii) decisions for action in all aspects of our daily life – public affairs, commerce, industry, science, and engineering. The NMS is also a key element in a worldwide system which links nations in a consistent network that is required for communication and trade. To analyze the NMS in the United States, the Institute for Basic Standards of the U.S. National Bureau of Standards has conducted separate analyses of major areas of physical measurement ranging from the basic quantities of mass, length, time, temperature, electric current, and luminous intensity to various mechanical quantities, electrical quantities, optical quantities, thermal quantities, atomic properties, ionizing radiation, as well as surface properties [3].

The purpose of this paper is to summarize some of the findings of the report on the National Measurement System for Surface Properties [1]. The large number of solid-surface properties that can be measured have been arbitrarily divided into two classes. First, there are the properties which can be used to characterize a surface: surface composition (as a function of position), surface atomic structure, surface electronic structure, motion of the surface atoms, the nature and distribution of surface defects, surface topography, and the nature and area of exposed facets. Second, there are derived properties of the surface characteristics as just defined and measures of the processes that occur on surfaces: electrical properties, optical properties, adhesion, bonding, catalytic activity, plating, durability, corrosion, decoration, grain-boundary segregation, lubrication, and reactivity. The report discusses directly only those measurements that are used for surface characterization with particular emphasis being given to the most common measurement, the measurement of surface composition, and lesser emphasis to the measurement of surface atomic structure, surface electronic structure, and atomic motions.

We give in section 2 a brief synopsis of the development of surface science and technology and an assessment of its significance. Section 3 is a summary of the present status of surface-characterization measurements, while section 4 is a discussion of anticipated future trends. The need for surface-measurement standards is discussed in section 5.

2. Significance of surface science and surface technology

The history of surface sience and surface technology in this century is interwoven with major discoveries and developments in other branches of science and technology: the discovery of thermionic-, photo-, field-, and secondary-electron emission; the development of radio, X-ray, and special-purpose tubes; the discovery of electron diffraction; the development of electron optics and electron-optical instrumentation; the investigations of the interactions of electrons, ions, neutral atoms and molecules, and photons with matter; the development of vacuum science and technology; the investigations of discharges in gases; the development of lamps; the development of semiconductor devices; the investigations of factors affecting the joining, working, wear, and polishing of metals; the investigations of corrosion and oxidation processes and the development of protective coatings; the studies of the adsorption, desorption, and reactions of gases on surfaces; and the development and characterization of improved catalysts. Many significant developments in surface science and surface technology, however, have occurred within the last ten years. Surface science has reached a stage of development such that knowledge of fundamental surface parameters can now be obtained. This growth has enabled the development of a new class of surface instrumentation that has been successfully applied to failure analysis, quality control, process evaluation and process development in the electronics, chemical, petroleum, and metals industries. New needs and the demand for improved materials and for new technologies have led to increasing demand for improved and more sensitive methods for characterizing surfaces and for measuring surface properties.

A realization of the current importance and relevance of surface science and technology can be obtained from two comprehensive surveys concerning future directions in physics and opportunities in materials research conducted under the sponsorship of the U.S. National Academy of Sciences [4,5]. In "Physics in Perspective" an expert panel on physics of condensed matter has listed a number of topics of high current interest in that they are the concern of a large number of physicists and they represent areas in which major scientific breakthroughs appear likely. Surface physics was one such topic. We quote from this source [4]:

"Rapid advances are taking place in the characterization of the surfaces of solids, largely using new experimental techniques and the improved theoretical understanding of quantum mechanics as applied to interfaces. By characterization is meant a detailed specification of the chemical identity, geometrical position, and, ultimately, the electronic and vibronic structure of atoms at surfaces. The electronic, geometrical and chemical behavior of defects and impurities adsorbed at the surface is another aspect of this problem. Also included in this category are the phenomena of tunneling in metal–insulator–metal, metal–insulator–semiconductor, and metal–insulator structures. Because of new experimental techniques such as Auger spectroscopy, advanced electron microscopy, and ultrahigh vacuums, rapid progress is being made. Entirely new methods for the control and preparation of interfaces have emerged from the vast amount of engineering and development carried out in recent years to produce integrated circuitry and other devices. The present rate of growth in this area offers the possibility of advanced understanding and control of chemisorption and catalysis."

In "Materials and Man's Needs", a distinguished panel has pointed out that improved materials are needed for major goals, such as improved or new methods of energy generation, communication, transportation, health services, housing, management of the environment, and consumer goods [5]. The panel describes a number of frontiers in materials research that they believe will lead to improved materials and processes. Work on surfaces was one of the designated frontiers, and we quote the opinion of the panel to show the extensive impact of surface technology:

"Surfaces and interfaces are possibly one of the most fruitful research topics in materials science. Knowledge at the most fundamental level in this area can be expected to be relevant to almost all uses of materials, from the processing and performance of integrated circuits to the corrosion of structural components, from frictional wear and energy loss to catalysis and flammability, from crystal growth to adhesion. The variety and complexity of surfaces and surface layers are at least comparable to the variety and complexity of bulk properties, but our understanding of surfaces is, in contrast, in its infancy.

"The aim is to develop more sophisticated insight into the electronic and chemical properties of surfaces. These properties are very sensitive to the detailed ways in which atoms are positioned at the surface, however, and in general these positions are not known. Surface properties are related also to the properties of the underlying bulk material, but in ways that are not often clear. And though bulk properties, by-and-large, are understood in principle, if not always in detail, this is not true of many of the surface properties where the broad outlines of the phenomenology are only now being drawn. This phenomenology concerns, for example, the details and statistical mechanics of surface topology, local bond and electronic structures, the energy states of electrons at surfaces, and models for nucleation and growth."

The latter report clearly shows the diverse and pervasive role of surfaces in current technology. Claims have been made over the last thirty years, that "surface science" or a particular, then new, technique would "solve" major problems or lead to dramatic improvements in major areas of surface technology such as catalysis and corrosion. Although some of these claims have been overoptimistic, the optimism of the above two National Academy of Sciences reports is warranted by the following significant developments in the last decade:

(a) The routine attainment of ultrahigh vacuum.

(b) The development of techniques and instrumentation to prepare and characterize surfaces in order to solve surface-related problems.

(c) The extension of solid-state theory to surfaces.

(d) A greater understanding of the role of surfaces in various phenomena.

(e) The development of surface-related technologies (microelectronics, catalysis, controlled-thermonuclear-fusion reactors, corrosion) in response to material needs or problems (energy, pollution, defense, materials conservation, and productivity).

3. Present status of surface-characterization measurements

Advances in surface science and technology depend on the availability and quality of measurements of relevant surface characteristics. To adequately characterize a surface, it is often necessary to measure the following quantities [6]:

(1) The type of atomic species present on the surface.

(2) The arrangement or structure of the surface atoms.

(3) The electronic structure (principally of the valence electrons) of the surface atoms.

(4) The motion of the atoms (atomic vibrations, surface diffusion, diffusion to and from the bulk, and evaporation).

(5) The nature and distribution of defects on the surface.

(6) The surface topography.

(7) The nature and area of exposed facets.

(8) The spatial distribution of foreign atoms on the surface.

(9) The depth distribution of different atomic species in the vicinity of interfaces.

Complete characterization of a surface on the scale indicated above is rarely attemped on account of the cost and complexity involved, the belief that certain types of characterization may not be required for a particular application, or the dynamic nature of surfaces. This last factor is extremely important; a surface can change its structure and composition as a function of time, either after generation, after heat treatment, after or during a particular surface-characterization measurement, or as the result of probe–sample interactions during what was intended to be solely a surface-characterization measurement.

Measurements on surfaces are made for a variety of purposes ranging from scientific to technological. For scientific applications, a surface will often have to be characterized fairly completely to test a theory or to gain a fundamental understanding of a particular property or process. For technological applications, the surface will generally have to be characterized to the extent necessary to relate to a particular phenomenon or the measurement of other surface-related properties.

A large variety of measurement techniques has been proposed for the characterization of surfaces. A summary of these techniques, adapted from a recent review of surface physics by Murday [7], is given in tables 1 and 2. This list of techniques is extensive but is probably not comprehensive.

Assessments of the various surface-characterization techniques are given in many recent books and review articles [8–51]. Some of these characterization techniques are relatively new and/or undeveloped and, although they may give useful information concerning a surface, the interpretation of the data may be complex or the technique may be restricted to special classes of materials. We have therefore restricted attention here to the more important techniques that are in widespread or growing use and, in the case of measurements in surface technology, to those for which appropriate instrumentation is commercially available. On this basis, the determination of surface composition is found to be the most common form of surface characterization. Measurements of surface atomic structure, surface electronic structure, and of atomic dynamics on surfaces (e.g. diffusion, nucleation, desorption

Table 1
Outline of surface-characterization techniques adapted from the report by Murday [7]. The techniques are classified according to the particles or radiation used as incident probes (i.e., to excite the sample) and detected quantities (i.e., to obtain information about the sample). The entries in the table are identified in table 2.

Incident probe	Detected particle					
	photon	electron	neutral	ion	phonon	E/H field
photon	ATR COL ELL ESR EXAFS IRS LS MOSS NMR SRS XRD	AEAPS AEM AES PEM PES SEE SEXAFS UPS XEM XES XPS	LMP PD	LMP PD		
electron	APS BIS CL CIS EM SXAPS SXES	AEAPS AEM AES DAPS EELS HEED IS LEED SEE RHEED SEM SLEEP STEM TEM	ESDN SDMM	ESDI		
neutral	NIRS	SEE	AIM MBRS MBSS		HA	
ion	GDOS IIRS IIXS NRS	IMXA INS SEE	ISD SDMM	GDMS IMMA ISD ISS NRS RBS SIIMS SIMS		
phonon	ES TL	TE	FD	SI	ASW	
E/H field	EL	FEES FEM ITS	FDM FDS	FIM FIM-APS FIS		CPD SC

Table 2
Key to acronyms and entries in table 1

AEAPS	Auger-electron appearance-potential spectroscopy
AEM	Auger-electron microscopy
AES	Auger-electron spectroscopy
AIM	Adsorption isotherm measurements
APS	Appearance-potential spectroscopy
ASW	Acoustic surface-wave measurements
ATR	Attenuated total reflectance
BIS	Bremsstrahlung isochromat spectroscopy
CIS	Characteristic isochromat spectroscopy
CL	Cathodoluminescence
COL	Colorimetry: IR, visible, UV, X-ray, and γ-ray absorption spectroscopy
CPD	Contact potential difference (work-function measurements)
DAPS	Disappearance-potential spectroscopy
EL	Electroluminescence
ELL	Ellipsometry
EELS	Electron energy-loss spectroscopy
EM	Electron microprobe
ES	Emission spectroscopy
ESDI	Electron-stimulated desorption of ions
ESDN	Electron-stimulated desorption of neutrals
ESR	Electron-spin resonance
EXAFS	Extended X-ray absorption fine structure
FD	Flash desorption
FDM	Field-desorption microscopy
FDS	Field-desorption spectroscopy
FEM	Field-emission microscopy
FEES	Field-electron energy spectroscopy
FIM	Field-ion microscopy
FIM–APS	Field-ion microscope – atom probe spectroscopy
FIS	Field-ion spectroscopy
GDMS	Glow-discharge mass spectroscopy
GDOS	Glow-discharge optical spectroscopy
HA	Heat of adsorption
HEED	High-energy electron diffraction
IIRS	Ion-impact radiation spectroscopy
IIXS	Ion-induced X-ray spectroscopy
IMMA	Ion microprobe mass analysis
IMXA	Ion microprobe X-ray analysis
INS	Ion-neutralization spectroscopy
IRS	Internal reflectance spectroscopy
IS	Ionization spectroscopy
ISD	Ion-stimulated desorption
ISS	Ion-scattering spectroscopy
ITS	Inelastic tunneling spectroscopy
LEED	Low-energy electron diffraction
LMP	Laser microprobe
LS	Light scattering
MBRS	Molecular-beam reactive scattering
MBSS	Molecular-beam surface scattering
MOSS	Mössbauer spectroscopy
NIRS	Neutral impact radiation spectroscopy
NMR	Nuclear magnetic resonance
NRS	Nuclear reaction spectroscopy
PD	Photodesorption
PEM	Photoelectrom microscopy
PES	Photoelectron spectroscopy
RBS	Rutherford backscattering spectroscopy
RHEED	Reflection high-energy electron diffraction
SC	Surface capacitance
SDMM	Scanning desorption molecule microscopy
SEE	Secondary-electron emission
SEM	Scanning electron microscopy
SEXAFS	Surface extended X-ray absorption fine structure
SI	Surface ionization
SIIMS	Secondary-ion imaging mass spectroscopy
SIMS	Secondary-ion mass spectroscopy

Table 2 (continued)

SLEEP	Scanning low-energy electron probe
SRS	Surface reflectance spectroscopy
STEM	Scanning transmission electron microscopy
SXAPS	Soft X-ray appearance–potential spectroscopy
SXES	Soft X-ray emission spectroscopy
TE	Thermionic emission
TEM	Transmission electron microscopy
TL	Thermoluminescence
UPS	Ultraviolet photoemission spectroscopy
XEM	Exoelectron microscopy
XES	Exoelectron spectroscopy
XPS	X-ray photoemission spectroscopy
XRD	X-ray diffraction (glancing incidence)

and evaporation) are also of considerable importance in surface science on account of their impact in surface technology (e.g., by understanding and controlling mechanisms of catalysis, bonding, and corrosion and through development of improved semiconductor, thin-film or electrochemical devices).

Based on an awareness of published literature, contacts with many of the manufacturers of surface-analysis instruments, contacts with users of these instruments in industrial, government and university laboratories, and a recent analysis of the analytical-instruments market [52], we have determined that most U.S. measurements of surface composition are now made by Auger-electron spectroscopy (AES) and to a lesser extent by X-ray photoelectron spectroscopy (XPS), secondary-ion mass spectroscopy (SIMS), and ion-scattering spectroscopy (ISS). It is estimates that about 60% of the U.S. surface-analysis instrumentation is in industrial laboratories, about 20% in government laboratories, and about 20% in university laboratories. The total U.S. capital investment is estimated to be about \$40M to \$50M while the annual sales volume for U.S. manufacturers is believed to be about \$10M per year now with an annual growth rate of 20 to 30% per year [1,52,53].

It is obvious, from the number of conferences, reviews, and books representing a variety of disciplines and technologies, that there has been a tremendous growth in surface science and surface technology over the past five to ten years [8–51]. Not so obvious is the extent to which surface-analysis instrumentation is used for industrial problem-solving. We now summarize some of the objectives of surface-characterization measurements in general and of surface-analysis measurements in particular.

(a) *Surface science*

Surface science is performed at universities, research foundations, government installations, and industrial laboratories. At this time, there appear to be two distinct but related thrusts in fundamental surface science.

First, considerable effort is being applied theoretically and experimentally to determine the relationships between surface composition, surface structure, surface electronic structure, and the motions of surface atoms. Experimental work of this type is often performed as far as possible on "ideal" surfaces in order to compare measurements with theoretical work. Research is also performed with "less ideal" or "real" surfaces, ranging from small particles, surfaces with various amounts and types of defects, to surfaces of metastable and amorphous materials. Many of the measurement techniques listed in tables 1 and 2 are used for these measurements.

Second, a large amount of effort is being devoted to obtain a fundamental understanding of various electrical, optical, mechanical and chemical properties of the many different kinds of surfaces just described and of processes which occur on such surfaces. Specific problem areas are: adhesion, adsorption, diffusion from or to the bulk, catalysis, contacts, corrosion, crystal growth, epitaxy, evaporation, interface properties and reactions, nucleation, oxidation, radiation effects, segregation, surface conductivity, surface diffusion, surface-phase changes, surface reactions, surface thermodynamics, and thin-film growth, structure, and properties. Experimental and theoretical data are being obtained concurrently in many of these areas and are being related, where applicable, to the properties of the constituent or interacting materials or surfaces. This scientific work is motivated by needs for improved materials or processes and is usually closely coupled with related developmental programs. Applications of surface physics to the computer industry, for example, are described in a recent article by Branscomb [54] while the applications of surface chemistry to problems in catalysis are described in articles by Yates [55] and Fischer [56].

Modern experimental surface science, as described above, must be performed with materials that are well characterized. The degree of characterization may vary, depending on the nature of the work, but in most cases involves the surface composition and often surface structure. In other cases, particular properties (e.g., adsorption, oxidation, and nucleation) are being correlated with the electronic structure at the surface or the motions of the surface atoms. Thus, part of the complexity (and cost) of surface science and technology is due to the many facilities that must be provided: surface generation and processing equipment, surface-characterization instrumentation, and components to measure the particular surface properties or processes of immediate concern.

(b) *Surface technology*

Applied research and development work on surfaces, surface properties, and surface processes is being performed at many industrial and government laboratories. Measurements of surface composition are frequently made as a function of position on a sample surface and as a function of depth. Surface atomic structure and surface electronic structure are measured at some laboratories in order to relate these parameters to other desired surface properties (e.g., reactivity, photocathode response, stability, oxidation, diffusion, electrical properties, etc.) for the development of new materials and processes. We give examples of the use of surface-composition measurements (predominantly by AES, XPS, SIMS, and ISS) to solve materials problems or to develop new processes and materials in industrial and government laboratories.

(i) *Semiconductor industry.* Devices of increasing density and complexity and of smaller size (microminiaturization) are being developed, but this trend has led to severe problems of reliability and yield. Typical yields of MOS/LSI devices (metal–oxide–semiconductor devices with large-scale integration), for example, are 5 to 8% of final assemblies. Failure rates can be alarmingly high [57,58] whereas reliability is crucial in large-scale civilian applications and in defense and space applications. In a recent analysis [57], 39% of device failures were attributed to surface-related defects and 18% of failures were attributed to metallization, oxide and bond defects. Many of these failures are caused by problems at surfaces or interfaces in the fabrication and processing of devices, such as residual contamination, adsorption of impurities, differential solubility, diffusion, void formation, electromigration, film dewetting, modification of surface electronic properties, and localized surface defects (e.g., dislocations, excessive roughness, and steps) [58,59]. The poor yields often found in the manufacture of complex LSI devices can be attributed to inadequate quality control during device manufacture; in particular, surface contamination during processing has been identified as a critical problem [57].

Auger-electron spectroscopy and secondary-ion mass spectroscopy are being frequently used to solve a variety of processing and production problems such as: (a) identification of stains, remnants of a previous processing step, corrosion products, compounds formed at interfaces by diffusion, and dopants and contaminants in semiconductors at various stages of processing; (b) analysis of failures in lead bonding, hermetic seals, and metallization adherence; and (c) establishment of adequate cleaning procedures [60]. These two surface-analysis techniques are also being employed for the development of new classes of devices and of new methods of fabrication and processing [54,61].

(ii) *Chemical industry.* The development of improved catalysts with greater selectivity and activity or lower cost was found to be a common objective in many laboratories. X-ray photoelectron spectroscopy is frequently used to correlate surface composition with catalytic activity and in at least one company is used for acceptance testing of catalysts prior to use. The techniques of AES and XPS are used for a variety of applications such as reactions at fuel-cell electrodes, adhesion phenomena, the bond strengths of composites, corrosion studies, process cleaning and wettability, structure determinations of complex molecules, detection of trace impurities, the migration of ions following irradiation, and analysis of surface coatings on particles in pigments. Other examples of industrial applications of XPS have been described in recent articles by Brinen [62] and by Riggs and Beimer [63].

(iii) *Petroleum industry.* The development of improved catalysts for process applications is an important goal in this industry and XPS is again frequently used to identify the surface species on active catalysts, to study the stability and regenerability of catalysts, to investigate the role of promoters, and to identify poisons. XPS and AES have also been used for investigations of corrosion, wear, fracture, and lubrication and for the characterization of metal, plastic and composite materials.

(iv) *Metals industries.* Measurements of surface composition are used for a variety of purposes in the laboratories of steel, automobile, and aerospace companies. Examples of applications of AES, XPS, SIMS and ISS are investigations of the diffusion and segregation of impurities in alloys, grain-boundary phenomena in alloys, failures of welds associated with residuals from cleaning baths, poisoning of catalytic converters in automobile exhausts, corrosion and wear of surfaces at higher temperatures, coverage of solid-state lubricants, adherence and blistering of coatings, bonding of fibers in composites, temper embrittlement, pickling rates, coatings to reduce eddy-current losses in transformer steels, catalytic activity as a function of alloy surface composition, elimination of poisons on catalyst surfaces, effects of surface films on brazing, lubrication, wear, adhesion, electrical contact-resistance, strength and durability of adhesive joints, and stress-corrosion cracking.

(v) *Other industries.* Surface-composition measurements by AES, XPS, SIMS, and ISS are used for a diversity of purposes in manufacturing industries ranging from electronic instrumentation and equipment, to business machines, communications equipment, and glass products. Examples of applications are quality control and failure analysis of incandescent lamps, lamp materials and phosphor coatings, diffusion studies of impurities in cladding, corrosion of pipes, identification of corrosion products of reactor fuel pellets, process development for thin-film devices, investigations of organic and molecular solids, design of catalysts for chemical synthesis, energy conversion, and pollution abatement, passivation of surfaces, thin-film nucleation and growth, diffusion of species in semiconductor devices, photocathode development, growth of oxide layers, and investigations of the strength and durability of glasses.

(vi) *Government applications.* The research and development laboratories of civilian and defense agencies of the U.S. government have widely different missions and activities. There are nevertheless many surface-characterization activities despite the diversity of objectives (e.g., space materials, energy programs, environmental programs, and defense). Examples of the application of AES, XPS, SIMS, and ISS are investigations of the migration of sodium ions in silicon dioxide under ion bombardment, segregations of impurities at grain-boundary interfaces, fracture and embrittlement, oxidation protection, activation and poisoning of cathodes, rocket-engine catalysts, optical properties, hydrogen permeation and hydriding, gettering processes, adhesive bonding, electroplating, improved corrosion resistance, electrochemistry of thin films, reliability of components, contamination of electrical contacts, cleaning and lubrication, surface reactions on pollutant particles, metal-polymer bearing systems, superconducting materials, damage to lenses, windows and mirrors of high-power laser systems, and contaminants from the first wall of a Tokamak fusion device.

4. Anticipated future trends

We have seen in the previous section that surface-characterization techniques in general and surface-analysis methods in particular are being widely used for a great variety of scientific and technological purposes. The relevance and impact of these activities and support for the belief that there will be continued growth in surface-characterization measurements can be appreciated from table 3 which contains selected portions of the recent report "Materials and Man's Needs" sponsored by the U.S. National Academy of Sciences [5]. The panel that prepared this report solicited the views of many experts on current needs in applied materials research and engineering. Table 3 gives a selection of the needs rated as "very high priority" or "high priority" that relate to surface properties and processes in nine major areas of public concern and societal need: (1) communications, computers, and control; (2) consumer goods; (3) defense and space; (4) energy; (5) environmental quality; (6) health services; (7) housing and other construction; (8) production equipment; and (9) transportation equipment. Clearly other measurement techniques than those discussed here will be required to develop improved materials, products, or processes for the various specific areas of applications. It is obvious, however, from this table and the specific examples of surface measurements and applications given in section 3 that surface-characterization measurements are now having and will continue to have a considerable impact on programs and policies of major public concern.

There are additional reasons for expecting further growth of surface-characterization measurements.

(a) *Energy*

The energy crisis facing the industrialized nations of the world is a complex matter of high public concern. Surface technology will be required in the following areas.

(i) *Controlled thermonuclear reactors.* Whatever the type of reactor that may eventually be developed as an alternate source of energy, there will be a significant problem in the design of the "first wall" that will be exposed to the hot plasma [64–66]. Radiation from the hot plasma will strike the surfaces of the first wall (and perhaps other components, such as diverters and limiters) and will cause wall damage and erosion and the release of gas and impurity atoms into the plasma, thereby affecting the performance and efficiency of the device.

A study group of the Americal Physical Society has recently reported that [64] "surface effects in CTR devices are strongly influenced by impurities, by adsorbed surface films, and by surface topography. Scrupulous attention must be paid to these points in all investigations in this area". Surface analysis is therefore required to analyze the topmost layers of the wall surface in order to gauge the efficiency of initial cleaning operations and to identify the nature of the equilibrium surface for other performance criteria (e.g., sputtering, diffusion, and wear).

Table 3
Impact areas of applied research and engineering that involve surface properties, processes, or measurements, selected from the recent National Academy of Sciences report "Materials and Man's Needs" [5]

1. Communications, computers, and control

(a) Properties

Electrical: memories; solid-state circuitry, large-scale integration, display devices, Josephson devices, charge-coupled devices; miniaturization; reliability

Atomic structure: perfection; quality of crystals; surface effects; electromigration; ion implantation

Microstructure: defects in III–IV and II–VI semiconductors, defects in crystals; films and epitaxy; interface imperfections; electromigration; yields; metallization

Optical: optical properties; displays, solid-state lasers; optical communications; optical storage

Dielectric: surface effects at semiconductor/insulator interfaces; encapsulation; substrates

(b) Materials

Elemental and compound semiconductors: for electronic circuits; large-scale integration; displays; semiconductor memory; variable bandgap

Thin films; Large-scale integration; control of metallization; thin-film memories; thin-film integrated optical devices; epitaxy; perfection of thin films

Ceramics: substrates, oxide layers, dielectrics; integrated optics; encapsulation; laser windows

Glasses and amorphous materials; optical transmission; integrated optics; laser windows; radiation-hard switches; glass for passivation

Inorganic, nonmetallic elements and compounds: electro-optic microelectronics; displays; modulators; detectors

(c) Processes

Vapor and electrodeposition, epitaxy: yield and processing of large-scale integrated circuits; thin-film quality; epitaxy; greater miniaturization; control of metallization

Chemical: corrosion; compatibility in environment; contacts; connectors; doping; distribution of dopants; etching

Extraction, purification, refining: purification; synthesis; characterization

Synthesis and polymerization: encapsulants; conducting adhesives; coatings; seals

Radiation treatment: ion implantation; radiation damage

Plating and coating: encapsulation; environmental protection

2. Consumer goods

(a) Materials
Plastics: stronger plastics; wear resistance; less brittleness; nonflammable plastics; impact-resistant plastics; biodegradable plastics

Table 3 (continued)

2. Consumer goods (continued)

Adhesives, coatings, finishes, seals: Resistant polymers and rubbers; corrosion protection; enamels; hot-water tank coatings; self-cleaning coatings for ranges; reduce permeability of packaging films; bonding; fastening

(b) Processes

Plastics extrusion and moldings: reinforced plastic; composites; colloid properties; improved fibers

Synthesis and polymerization: composite processing; biodegradable polymers; improved cross-linking; molecular architecture for special properties; improve fiber strength by controlling molecular orientation

3. Defense and space

(a) Properties

Mechanical and acoustic: mechanical properties of composites; high-temperature materials; fatigue; corrosion fatigue; crack propagation; high-temperature fatigue; creep resistance; fracture toughness; impact resistance; fatigue resistance; undersea equipment; materials for pressure hulls

Microstructure: dispersion hardening; microstructural stability; corrosion pitting; uniformity of mechanical properties; radiation-resistant materials; hydrogen compatibility

(b) Materials

Composites: composites for ship construction; structural designs for composites; improved fracture toughness of composites; fatigue-resistant composites; dispersion-hardened alloys; reliability of composites

Adhesives, coatings, finishes, seals: high-temperature coatings; fabrication of metal-nonmetal systems; integrity of polymer adhesives; degradation of adhesive bonds; antifouling coatings for ships; coatings to reduce corrosion; low drag and low contamination paints; room-temperature curing adhesives; thermal-control coatings; ablation materials; cements; sealants for deep-sea equipment

Joining: welding of titanium; weldable aluminum alloys; welding of dispersion-hardened alloys; joining of composites; adhesion mechanisms, seals for undersea repeaters

Testing and nondestructive testing: failure analysis; service life; failure prediction; nondestructive testing for welds

4. Energy

(a) Properties

Chemical: batteries; higher energy density; improved electrodes; lower weight; longer life; catalysts for batteries; new contained materials for batteries; corrosion of cables, of heat exchangers, of turbine blades; radiation effects on corrosion; high-temperature corrosion

Table 3 (continued)

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4. Energy (continued)

Atomic structure: solid-state electrolytes; hydrogen embrittlement; superconducting materials for power transmission

Microstructure: radiation resistance; swelling; void formation; blistering; stability under high neutron fluxes; radiation-hard control equipment

Thermodynamic: combustion efficiency; thermoelectric power converters; magnetohydrodynamic conversion systems; electrohydrodynamic conversion systems

Mechanical and acoustical: high-temperature materials for reactors, both for fuel containers and converters; lightweight conductors; high-temperature alloys for turbines; high-temperature bearings; creep; fracture toughness; high strength; thoughness; fracture propagation in pipeline materials

(b) Materials

Ceramics: high-temperature materials for burners; plasma containment; high-voltage insulators; ceramics for turbine blades

(c) Processes

Testing and nondestructive testing: failure criteria; lifetime prediction; nondestructive testing of reactor components

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5. Environmental quality

(a) Properties

Chemical: catalysts for automobile exhausts; pollution detection of control systems; improved, cleaner extraction processes; improved beneficiation of ores; recovery processes

Biological: handling corrosive, toxic, and dusty materials; biodegradable plastics

(b) Materials

Plastics: wear

(c) Processes

Extraction, purification, refining: improved extraction methods; improved incineration methods; control of pollution and environmental degradation caused by mining and extraction

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6. Health services

(a) Properties

Chemical: corrosion of implants; microbial corrosion; stress corrosion

Biological: biological response to implants; biocompatibility; rejection; toxicity; immunological response

Microstructure: adhesion; prosthesis/tissue interface; adhesion between implants and tissue

Table 3 (continued)

6. Health services (continued)

Mechanical and acoustical: artificial bone, teeth, tissue, membranes, and organs; better filling material for teeth; fatigue; wear; alloys for joints

(b) Materials

Plastics: membranes; artificial teeth; dental adhesives; artificial heart valves; encapsulants for implants; containers for blood

Prosthetic and medical materials: implants; artificial organs, bones, teeth, tissue and membranes; compatibility and biological response

Fibers and textiles: membranes, fine wires, organ replacements

Rubbers: artificial organs, tissue, membranes

Composites: for implants; bone and tooth replacements; joints; pins

Organo- and organometallic compounds: prosthesis-tissue interface; adhesion between bone and tissue; for implants

(c) Processes

Synthesis and polymerization: dental adhesives; compatibility; interface between tissue and prosthesis

Plastics extrusion and molding: precision forming; controlled porosity; artificial organs; heart valves; membranes

Testing and nondestructive testing: quality control; methods to evaluate compatibility; characterization of properties of implants

7. Housing and other construction

(a) Properties

Chemical:corrosion; atmospheric degradation

8. Production equipment

(a) Materials

Ferrous metals and alloys: harder dies; better cutting tools; better saws; rust resistance

Nonferrous structural metals and alloys: improved wear and fatigue properties

Lubricants, oils, solvents, cleansers: tribilogy-lubricants; wear and abrasion resistance

(b) Processes

Testing and nondestructive testing: quality control; fatigue failures

9. Transportation equipment

(a) Properties

Mechanical and acoustical: fatigue; crack propagation; temperature cycling; better bearings

Table 3 (continued)

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9. Transportation equipment (continued)

Microstructure: super alloys for engines; corrosion resistance; stress corrosion

Chemical: corrosion resistance; stress corrosion; corrosion fatigue; high-temperature oxidation; catalytic converters for automotive exhaust

(b) Materials

Adhesives, coatings, finishes, seals: Adhesives and sealants for aircraft; adhesives for automobile bodies, frames. and repairs; coatings for automobile mufflers; coatings for turbine blades; seals for gas turbines; seals for Wankel engines; refractory coatings

Lubricants, oils, solvent, cleansers: wear, abrasion resistance

Ferrous metals and alloys: improved high-temperature properties; corrosion resistance

Nonferrous structural metals and alloys: superalloys; high-temperature materials for turbine engines

Plastics: composites

Composites: develop composites for use in engine and bodies of automobiles and aircraft; joining metals

(c) Processes

Metal deformation and processing: nondestructive testing evaluation

Heat treatment: improved strength; high-temperature properties

Material removal: improved shaping methods

Joining: fasteners and bonding systems for aircraft and for automobiles; joining methods for composite materials

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(ii) *Laser fusion.* Laser-induced fusion is another potential technique for energy generation. In addition to the "first wall" problems noted in (i) above, damage can occur to the optical components (mirrors, windows, etc.). Damage thresholds are now strongly limited by surface defects (roughness, morphology of films) and by surface impurities (contaminants introduced by fabrication and by surface treatments) [67].

(iii) *Liquefaction and gasification of coals.* Improved catalysts (with the necessary efficiency, stability, selectivity, and cost) need to be developed to obtain liquid and gaseous synthetic fuels from coal [56,68]. Quantitative surface analysis will enable improved catalysts to be scientifically engineered so that the desired reactions can be promoted and undesired components (ash, sulfur, nitrogen) removed [69–73]. Identification of the nature and measurements of the rates of specific surface (catalyzed) reactions are also required so that improved (e.g., alloy) catalysts can be designed.

(iv) *Hydrogen.* Hydrogen has been proposed as a major source of energy in the future [74,75]. There are substantial surface problems in hydrogen storage (as hydrides), transport (due to hydrogen embrittlement), and efficient use (e.g., in fuel cells) [76].

A summary of critical materials problems associated with the production of energy (including fusion devices, magnetohydrodynamic systems, solar-conversion devices, coal-conversion systems, and energy-storage devices) is given in a recent book [74]. The pervasiveness of surface science and of surface-related problems in materials research for many energy technologies has also been emphasized in the reports of a recent series of workshops sponsored by the U.S. Energy Research and Development Administration [77].

(b) *Defense*

Surface technology is required for the development of rocket-engine catalysts, laser optical components, oxidation and corrosion protection, night-vision devices, lubricants, microelectronic devices, avoidance of fracture and embrittlement, development of high-performance composite and other materials, ablation materials, adhesion and welding of materials, failure analysis, and nondestructive testing.

(c) *Advanced materials*

Needs for advanced materials that require surface technology are shown in table 3. Similar needs have been highlighted in a recent series of articles on high-technology materials [69,78,79].

The prediction of the durability of building materials and components, for example, is becoming increasingly important with the present interest in life-cycle benefit–cost analysis. Many building materials (e.g., concrete and paints) are composites in which internal interfaces have an important bearing on performance. In addition, surface characteristics are of vital importance in a wide range of building materials (e.g., adhesives, building-joint sealants, built-up rooling, electrical terminations, paints, and coatings) for which degradation occurs primarily by surface processes.

New bio-compatible and nondegradable materials are required for prostheses and implants (table 3). The development of such materials will require the use of surface-characterization measurements to assess corrosion rates in the body environment. It is anticipated that surface-measurement techniques will be developed to characterize lipid membranes and to determine how the transport of drugs and carcinogens through such membranes can be controlled.

(d) *Environment*

Improved catalysts and control methods are required for automobile exhausts, power-station exhausts, and factory exhausts. Improved measurement methods for determining the surface composition of pollutant particles in the atmosphere (as a result of the discharges from fossil-fueled power plants) and for determining the nature and rate of chemical reactions on the surfaces of these particles are urgently required.

(e) *Productivity*

Many industries are currently using surface-characterization instrumentation and surface technology to improve products, develop new products or to improve yield (section 3). It is believed, based on the present activity and the recent growth of activity, that this use of surface technology will continue and probably intensify. Specific major areas of development will probably include:

(i) the development of catalysts with improved performance (efficiency, stability, and selectivity) for existing and new processes in the petroleum and chemical industries;

(ii) the development of improved coatings and processes that can better withstand corrosion and wear (e.g., by ion implantation [80,81]);

(iii) the development of semiconductor devices, particularly large-scale integrated circuits, with greater yield and greater reliability;

(iv) the development of new components (devices, memories, displays, optical systems) for improved performance in the communications and computer industries; and

(v) the development of metal products with improved strength and service life.

In addition to the needs and developments described above, it is believed that the following factors will further stimulate growth in surface-measurement capability.

(f) *Surface science*

The development of surface science has reached the point where the properties of "ideal" surfaces can be measured with reasonable confidence and related to modern theories of surfaces. It appears likely that new and improved measurement techniques and methodologies will be developed to characterize "real" surfaces. Developments are anticipated in the following areas:

(i) methods to determine the local atomic structure of real surfaces, perhaps with the SEXAFS technique [82] (tables 1 and 2), from the angular distributions of photoelectrons or of excited ions (originating from chemisorbed species), or by electron energy-loss spectroscopy;

(ii) characterization of small particles or clusters (diameters $\gtrsim 10$ Å) that are used as practical catalysts [20];

(iii) methods, perhaps optical, to characterize surfaces in high-pressure environments (e.g., working catalysts) where electron- and ion-spectroscopic methods are generally unsuitable [83];

(iv) solution of other basic problems limiting the performance of industrial catalysts [71];

(v) methods to develop and characterize ultra-thin-film and microminiaturized semiconductor devices;

(vi) determination of the relationship of surface atomic structure and surface electronic structure for "ideal" and "real" surfaces to surface properties, such as reactivity, bonding, corrosion, durability, wear, and lubrication; and

(vii) determination of biophysical properties at biological surfaces and interfaces.

(g) *New instrumentation*

The commercial instrumentation now used for surface characterization has directly evolved from previously established scientific and technical knowledge. It is anticipated that instrumentation with improved capabilities for surface characterization will likewise be developed in the future; for example, the recent development of a photoelectron microscope for imaging biological surfaces has benefitted from earlier work on electron optics and surface physics and will probably stimulate new applications in biophysics [84].

(h) *Quantification*

Many present methods of surface characterization are qualitative rather than quantitative. Although qualitative determinations of surface composition have been extremely useful for the solution of practical problems (section 3), it seems intuitively clear that the various types of instrumentation will not be used to their full potential until the results of the measurements can be expressed in absolute terms with known uncertainty.

(i) *Legal*

As surface-characterization measurements are made more routinely and with greater (i.e., known) accuracy, it is believed that these measurements will be used for the prosecution and defense of patent suits where surface properties are involved. For example, measurements of surface composition will be used to specify the prepared or working surface of a proprietary coating or product (e.g., catalysts).

(j) *General analysis*

As the unique sensitivity of instruments that determine surface composition becomes more widely known in the technical community, it seems likely that the instruments will be used to solve a wider range of problems than those discussed in section 3, that is, there is no evidence of "saturation" in the present range of applications. It is therefore thought likely that within a decade most major laboratories will have surface-characterization instruments routinely available for analytical work, much as electron microscopes, mass spectrometers, chromatographs, X-ray analyzers, and spectrophotometers are available and in common use now. It is also believed that the surface sensitivity of the instrumentation discussed here could be valuable in forensic applications.

5. The need for standards

In most fields of science and technology, there are well-established "systems" by which useful measurements are made [2,3]. Each system is based on accepted scientific concepts which indicate that particular quantities can be determined; it is then a matter of convenience to select appropriate units. With this base, it is possible to construct instruments which, with associated specifications, reference data, and reference materials, give the capability to make measurements over a certain range of the quantity of interest with known precision and accuracy. Trained people, often with the aid of professional societies and standards groups, can then expect to make useful measurements (i.e., of the desired quality) with known costs and benefits.

The situation with surface measurements is now qualitatively different: there is no established measurement system of the type just described [1]. At this time there is an almost complete lack of standards, standard procedures, and standard materials to support surface-characterization measurements. Each experimenter is expected to do the best he can based on his own training and experience and using the sometimes conflicting data, spectra, and procedures suggested by others. This problem is made more severe by the variety of applications of surface-characterization measurements (sections 3 and 4) and the corresponding diversity of professional backgrounds of the practitioners. The greatest need now is for standards for surface analyses although it is believed that standards for other types of surface characterization (section 2) will also be required in due course.

The American Society for Testing and Materials (ASTM) has recently formed a new Committee E-42 on Surface Analysis [85]. Subcommittees have been established for the four surface-analysis techniques in common use (AES, XPS, ISS, and SIMS) and for nomenclature, ion-beam sputtering, standard reference materials, new technologies, and editorial processing. The development of documentary standards is now under way.

It is difficult to prepare standards (specifications, test methods, definitions, classifications, or practices) when the field is growing rapidly. Although there are a lot of unanswered questions concerning the basic interactions in the various types of surface-analysis measurements and concerning the choice of procedures, it is believed that interim standards should be drafted on the basis of current knowledge with the expectation that they may be revised in the future. Standards for depth profiling, for example, could be prepared to reflect the current state-of-the-art even though the magnitude of various perturbing effects [13] (e.g., rates of ion-beam damage, differential-sputtering effects, topographic changes) are not known for many materials.

Table 4 contains a list of areas where standards are believed to be needed. The classification is to some extent arbitrary and there is clearly overlap in a number of instances. The list was prepared, however, in order to provide a basis for discussion and action and to delineate topics of small enough size that could be tacked by individuals or small groups on a reasonable time scale. Table 4 contains specific examples of the types of standards needed for Auger-electron spectroscopy but there are analogous needs for XPS, ISS, and SIMS.

It is clear from table 4 that a considerable amount of work will have to be done to develop appropriate standards. This effort will only be successful if concerned individuals, both in the USA and elsewhere, will support the new ASTM Committee in its attempts to upgrade the quality of surface analyses.

Table 4
Summary of areas where standards are needed for surface analyses (with examples given for the case of Auger-electron spectroscopy)

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1. Nomenclature

Definitions of terms and units

2. Specifications, methods and practices

Instrument performance (e.g., specifying the electron energy analyzer, measurement of current densities of electron and ion beams, detector efficiency, resolution, overall response of the instrument in a particular measurement situation, sensitivity)

Instrument calibration (e.g., energy and intensity scales)

Sample preparation (e.g., handling, cleaning, heating, cutting, mounting)

Sample characterization (e.g., type of sputtering, annealing, roughness, crystallinity, morphology, defect structure)

Experimental conditions (e.g., details of surface cleaning and processing, sputtering, annealing, temperature of measurements, conditions of measurement, pressure, geometry, beam energy, beam current or current density, beam size, angle of incidence, correction for charging, analyzer type and orientation, aperture of analyzer, modulation, energy resolution, detector efficiency, alignment of sample)

Depth profiling (e.g., type of ions, ion energy, ion current or current density, beam size, rastering, type of crater, sputtering yield, differential sputtering, annealing, conditions of multiplexing, depth resolution)

Data presentation (e.g., preferred form of presentation for energy distribution, derivative of energy distribution, and depth profiles; necessary information, as listed in experimental conditions; units; measurement of peak positions)

Data analysis (e.g., chemical shifts, peak line shapes and areas, level of contamination, surface structure, surface phases, morphology, thermal equilibrium, source of data for quantification, different types of analytical situations)

Measurement reliability (e.g., procedures to ensure that representative regions of a sample are being measured, homogeneity of sample, transverse and depth variations of composition, spatial resolution, procedures to ensure meaningful calibration and operation of equipment)

3. Reference materials

Selection (e.g., stability, homogeneity, kind of analytical use, energy range of peaks, ease of characterization, chemical properties when exposed to various ambients, physical properties such as grain size, crystallinity, and roughness)

Characterization (e.g., adequacy and availability of techniques at the point of supply and/or by the user)

Conditions of use (e.g., convenience; need for additional facilities for preparation or regeneration such as heating, cooling, sputtering, and evaporation; stability; checks to prevent accidental misuse; cost)

Types of applications (e.g., round robins, calibrations of energy and/or intensity scales, thin-film sputtering rate standards, sensitivity standards)

Table 4 (continued)

4. Reference data – tabulations and reviews

Spectra

Line shapes and line positions for elements and compounds

Chemical shifts in particular compounds or in local environments

Relaxation effects

Radiation damage to sample (e.g., electron, ion, and X-ray induced damage such as desorption, decomposition or polymerization)

5. Quantification

Definitions (e.g., specific analytical situations, sensitivity, surface structure, angular anisotropies, topography)

Methods (e.g., background subtraction, correction for inelastic scattering, line shapes and areas)

Procedures to convert intensities to concentrations (e.g., models)

Data (e.g., electron attenuation lengths, ionization cross sections, backscattering data)

Procedures to test for surface segregation

Precision and accuracy in different analytical situations.

6. Conclusion

There has been a rapid growth of activity in surface science and surface technology in the past ten years. Surface science and technology are both widely regarded as "frontier" areas in which major scientific breakthroughs will occur and major improvements in materials and processes will be developed.

Most of the measurement methods now in use for surface characterization have become commercially available within the last ten years. At this time there is an almost complete lack of standards, standard procedures, and standard materials to support surface-characterization measurements. It is hoped that needed standards can be developed soon under the auspices of the recently formed ASTM Committee on Surface Analysis.

In a year in which the fiftieth anniversary of the discovery of electron diffraction is being celebrated, it is appropriate to conclude with a quotation from a review published forty years ago [86].

"Already, at the tenth anniversary of G.P. Thomson's first experiments in 1927, the new technique is being successfully applied to such varied surface problems as thermionic and photoelectric emission, conduction of electricity in thin films, surface catalysis, electrodeposition, crystal growth, the colloidal state, corrosion, wear and lubrication; and is, in fact, affording for the first time a direct insight into the small-scale structure and properties of surfaces." It is clear from this review and the book on electron diffraction by Thomson and Cochrane in 1939 [87] that high-energy electron diffraction had already had significant impact on surface physics and chemistry. This technique and low-energy electron diffraction have been successfully applied in the subsequent forty years but it is also clear that many problems in surface science and surface technology still remain to be solved. One could reasonably ask whether the newer methods of surface characterization can be expected to "solve" problems (e.g., in catalysis and corrosion) which remain despite decades of efforts by many talented scientists using a great variety of experimental techniques [88]. It is believed by the present author that there are reasonable grounds for optimism because the newer or more recently developed surface-characterization techniques have either much greater surface sensitivity or give more detailed information (often on an atomic scale) about a surface. Scientific and technological problems can now be tackled with greater specificity than was possible even ten years ago. The need for a greater understanding of surfaces and surface properties has been well documented as has the need for improved materials and processes [1,4,5,88], as discussed in sections 3 and 4. The present level of activity in surface science and surface technology should lead to significant progress in the next decade.

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