HARRY K. CHARLES, JR., JOEL A. WEINER, and NORMAN A. BLUM

MATERIALS CHARACTERIZATION AND ANALYSIS: APPLICATIONS TO MICROELECTRONICS

The APL Microelectronics Group has been actively engaged in the design, fabrication, packaging, and testing of miniaturized electronic devices, circuits, and systems for use in space, avionics, biomedicine, and ocean science. Advanced techniques for the analysis and characterization of materials have significantly aided the production, delivery, and ultimate reliability of the circuitry. In particular, such inspection and characterization instruments as the scanning electron microscope, the scanning Auger microprobe, and the secondary ion mass spectrometer have allowed the development of new materials and processes applicable to hybrids, surface mounts, and other types of microelectronics and have also provided a way to ensure greater reliability and product safety. Other relevant tools and analyses include X-ray techniques (diffraction and spectroscopy), residual gas analysis, profilometry, atomic absorption, light and electron scattering methods, and a variety of mechanical, electrical, and environmental stress tests.

INTRODUCTION

Advanced methods for analyzing, characterizing, and testing materials have been developed to the point where they can be applied routinely not only in research and development but also in the day-to-day production of hybrid microcircuits and assemblies suitable for use in the fields of space research, avionic systems development, biomedicine, and ocean science. Basic tools (e.g., the scanning electron microscope equipped with various detectors, the energy dispersive X-ray spectrometer, and auxiliary current amplifiers) can be used daily for inspection, quality control, materials characterization, and failure analysis.[1]

The scanning electron microscope has been used in every aspect of medical microelectronics manufacture, from basic substrate patterning to lid sealing. Another important method that has had widespread use is surface profilometry[2] for thickness and dimension measurements, a technique that quantifies surface roughness and allows deposited layers and photolithographically patterned areas to be measured routinely (both in thickness and extent) in a nondestructive contact way. Beta-backscatter thickness measurement[3,4] is another nondestructive technique that has played a significant role in the screening of packages, deposited layers, and printed circuit board materials.

Various materials identification and analysis techniques such as energy dispersive X-ray spectroscopy, residual gas analysis, atomic absorption, scanning Auger microscopy, secondary ion mass spectrometry, and X-ray diffraction have been used productively in the development and testing of microelectronic devices and assemblies. Energy dispersive X-ray spectroscopy,[5] in conjunction with the scanning electron microscope, has been used to locate and identify impurities and to monitor the relative composition of various microelectronic coatings, deposited layers, and basic starting materials. Residual gas analysis[6] has been used to analyze, for example, gaseous products given off during charge and discharge cycles of potential implant capacitors and outgas products from curing epoxies and other organic materials. These analytical results contribute to specific component and materials selection as well as aid in the development of processing and techniques to minimize contamination.

Scanning Auger microscopy[7] has made several contributions to microelectronics development including contamination and cleaning tests, elemental materials identification, and depth profiling. X-ray diffraction[8] has aided in materials identification and in the determination of microstructure (crystalline versus amorphous) and alloy composition. Secondary ion mass spectrometry[9] has provided materials identification, impurity profiles, and the ability to look at both elemental and molecular compositions. Atomic absorption spectroscopy[10] has also been used for quantitative impurity and compositional determinations.

MICROELECTRONIC APPLICATIONS OF THE SCANNING ELECTRON MICROSCOPE

When a material is irradiated by a primary electron beam, secondary electrons are emitted with energies

between 0 and 30 electronvolts, with the upper energy limit set by the maximum primary beam energy. Secondary electrons produce excellent topographical images because of their abundance and ease of collection. Figure 1 is a secondary electron topographical image of an integrated circuit die in a ceramic chip carrier that is used in a biomedical implant. The depth of field of the scanning electron microscope and its obvious utility in inspection and quality assurance for integrated circuits and packaging are evident. The effect of the material work function (as shown by the brightness of the gold bonding pad image) on the contrast of the secondary electron image is also apparent. Backscattered electrons have energies distributed about the primary beam energy but are much less abundant than the secondary electrons, thus producing less intense topographic images. However, the yield of backscattered electrons increases with the atomic number of the material under analysis and therefore can be useful for material identification. Figure 2 is an example of how a backscattered electron image was useful in detecting an electrical problem (residual metal deposits) on a substrate used in a medical electronic system.

Figure 3 shows the utility of the scanning electron microscope in process development and also shows how it can be combined with other methods (such as impedance measurements) to produce structures suitable for APL system applications. The example shown is a platinum-gold electrode platinized to reduce impedance and improve electronic signal reception. Impedance reduction factors greater than 10 have been achieved by this method. Platinization techniques have been applied in both underwater conductivity sensors and in neural prosthesis electrodes used for recording neuronal activity from the brain of an awake, performing monkey.[11] Platinization of thin-film platinum-gold layers has been accomplished for multisensor neural probes.

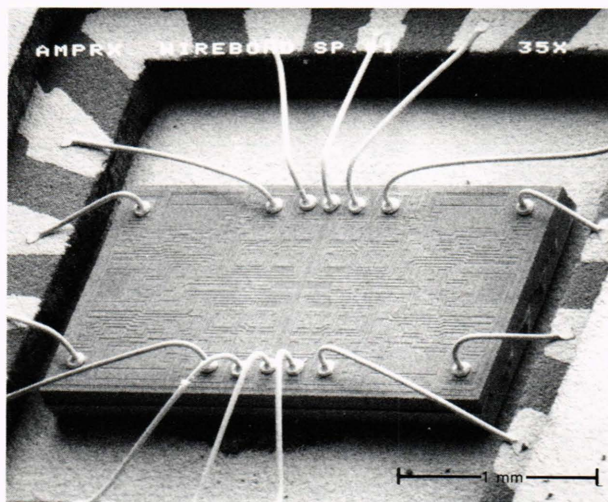


Figure 1—Secondary electron photomicrograph of an integrated circuit chip in a leadless ceramic chip carrier. Note the unlimited depth of field and the topographical nature of the scanning electron microscope image.

Fluorescent X rays are important for use in scanning electron microscope imaging. A scanning electron microscope equipped with a suitable energy-dispersive X-ray detector can be used in two modes for materials identification: full spectrum elemental analysis (atomic number 10 (neon) and above) and spectrum peak siting or dot mapping of a particular constituent over the entire sample surface. An example of an elemental X-ray spectrum (which revealed a cadmium impurity in an epoxy preform used in a spacecraft hybrid) is shown in Fig. 4. Figure 5 illustrates a material dot map of tin diffused into a capacitor end cap after solder reflow.

NONDESTRUCTIVE THICKNESS MEASUREMENTS

Thickness measurements play an extremely important role in evaluating high-reliability hybrids and

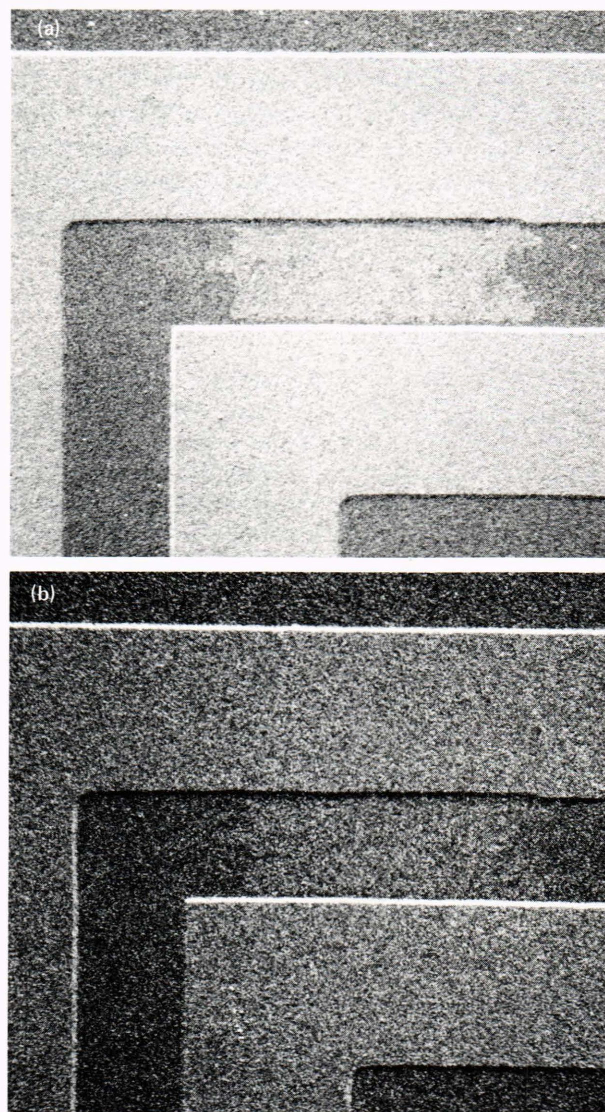


Figure 2—(a) Residual metal deposits on a substrate after photolithography using a high-resolution backscattered electron detector. The residual material produced a surface leakage problem. (b) A corresponding secondary electron image provided for comparison.

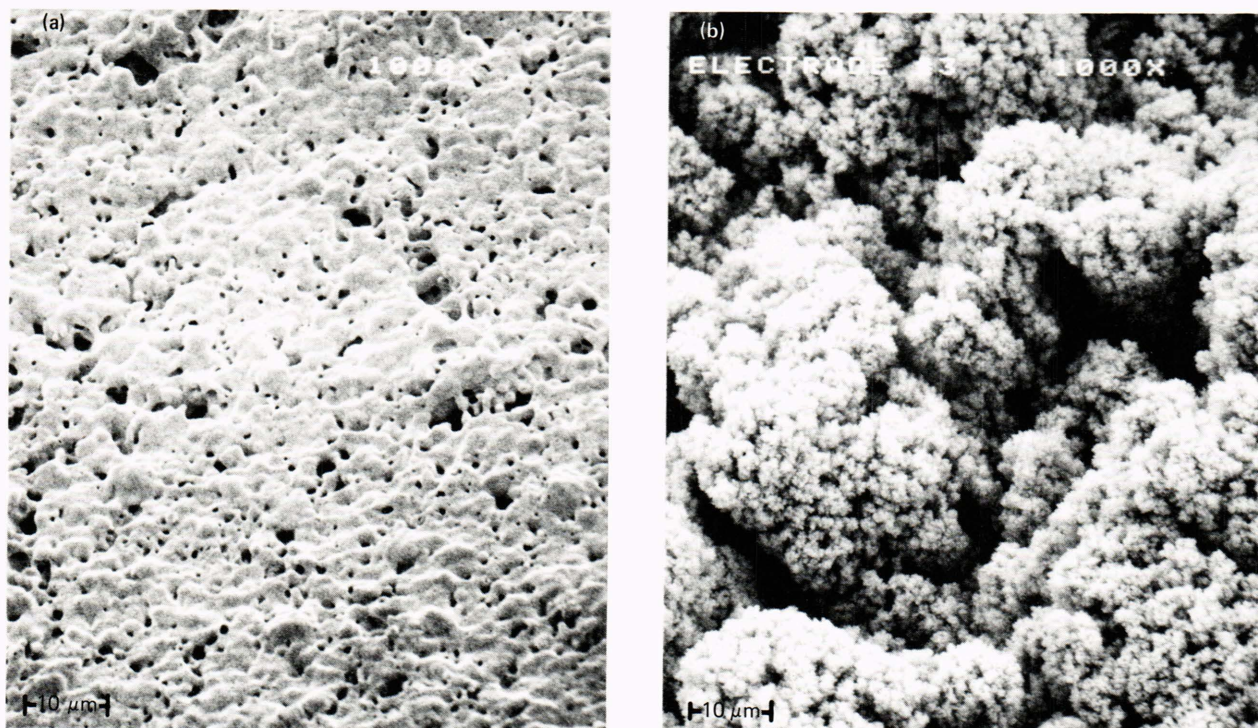


Figure 3—Scanning electron microscope morphology studies of thick-film platinum-gold electrodes platinized to reduce impedance. (a) Bare electrode; (b) converted to platinum black in a 3 percent platinum chloride solution at 1.65 amperes per square centimeter for 2 minutes.

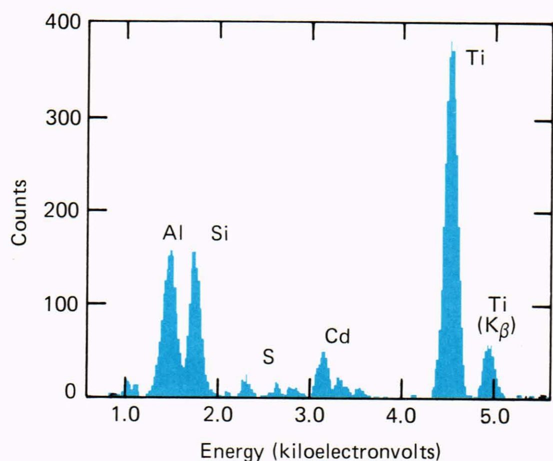


Figure 4—Energy-dispersive X-ray spectrum of an epoxy preform. The epoxy is Ablefilm® 517. The titanium peaks (Ti) are associated with the pigmented filler. The cadmium (Cd) impurity was later determined to be 2.2 percent by atomic absorption. The sulfur (S) is believed to be associated with the glass reinforcing mesh (as is the silicon (Si)); the aluminum (Al) is believed to be due to the sample mounting stud material.

surface-mount assemblies in microelectronics. Although they can be made using the scanning electron microscope (with proper image correction for foreshortening) and by standard optical interference techniques, such methods typically are destructive (requiring overcoating) and can be done only on a lot sample basis. Exact production control requires film thickness measurements of each substrate, component, or microcircuit package. Two important, nondestructive thickness-measurement techniques that can be applied to production samples are stylus profilometry and beta-backscatter.

Stylus profilometry was used, for example, to diagnose a plating problem in the fabrication of a spiral inductor. The problem, as depicted in Fig. 6, was inconsistent plating thickness across the width of the plated track—it varied as much as 1.8 micrometers (out of a nominal 10 micrometers total thickness). The variation shown by the stylus profiler trace was determined to be a function of track location, suggesting that it was caused by improperly positioned anodes in the plating bath rather than by a photolithography-related process as was originally believed. Modifying the plating technique and repositioning the electrodes solved the problem.

Figure 7 illustrates the thickness of gold plating on the inside of a ceramic chip carrier[12] as determined by the beta-backscatter technique. The carriers were to be used on a surface-mounted controller board[13] of a programmable implantable medication system. Thin plating could result in improper eutectic die attachment and an ultimate decrease in system reliability. The plating was specified to be 2.54 ± 0.25 micrometers thick. As seen in Fig. 7, only at the outer edges of the package bottom do the thicknesses meet the specified value. (Each thickness reading is the average of 10 measurements.) The absolute accuracy of the beta-backscatter technique has been shown to be better than ±5 percent.

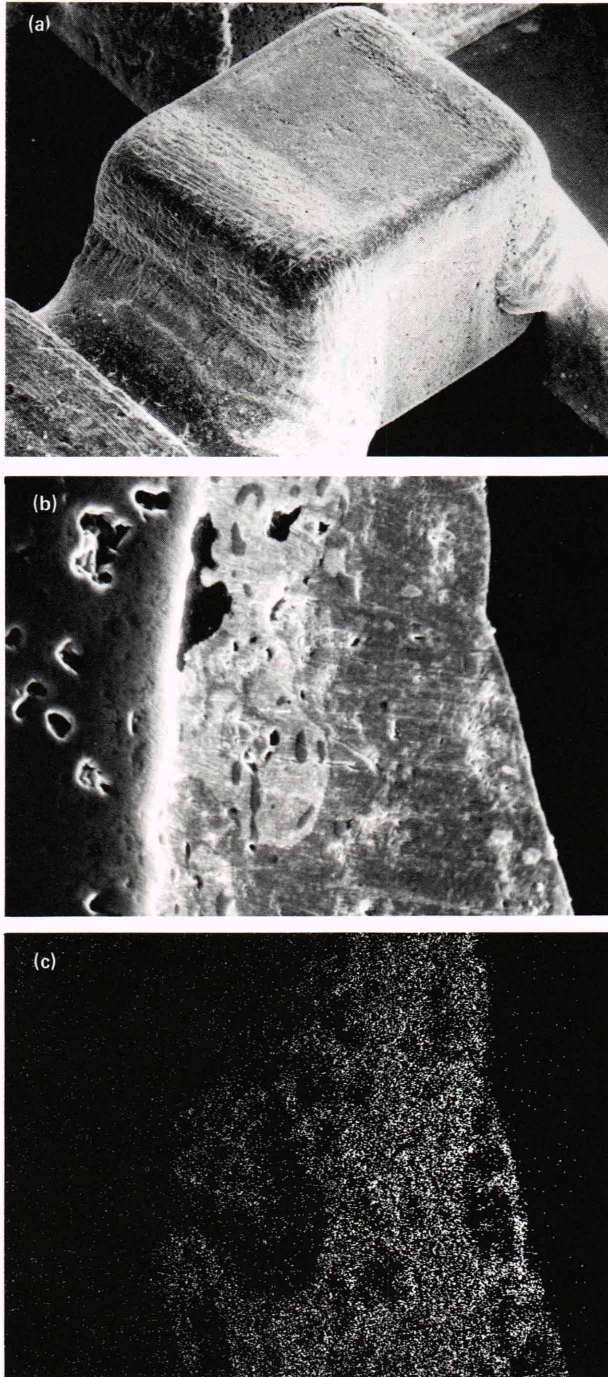


Figure 5—Capacitor with end caps subject to metallization leaching during solder reflow. Problems were experienced with several capacitors used in a spaceborne hybrid receiver system. (a) Secondary electron photomicrograph of the leached capacitor. (b) Secondary electron photomicrograph of the end-cap cross section after reflow. (c) X-ray dot map of tin penetration into the end cap.

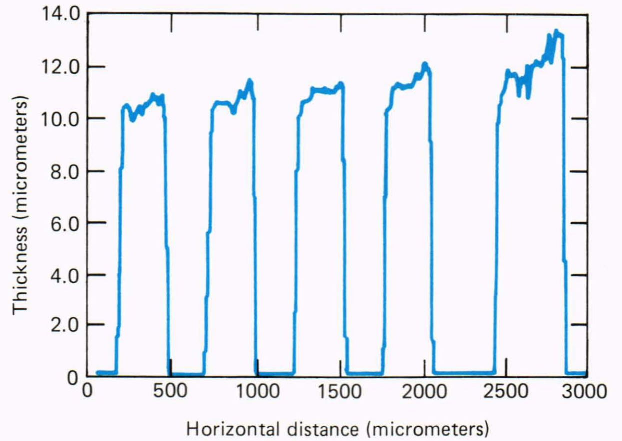


Figure 6—Stylus profile of a plated spiral inductor illustrating a variation in plating thickness of as much as 1.8 micrometers across the width of a track (approximately 300 to 500 micrometers wide). This variation indicates a plating problem.

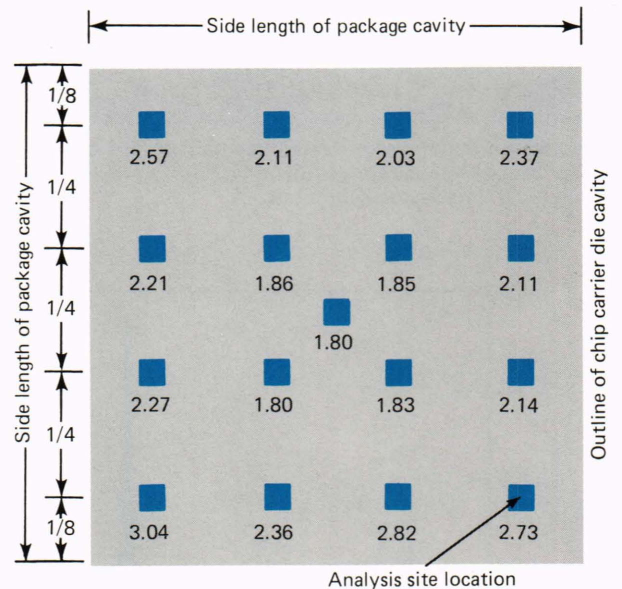


Figure 7—Thickness distribution of gold plating (in micrometers) on the inside of a leadless ceramic chip carrier of the type used in medical implant electronics.

RESIDUAL GAS ANALYSIS

Residual gas analysis[6] has been particularly useful in determining the outgassing of capacitors under rapid charge/discharge cycles (for potential use in an implantable defibrillator system) and the outgassing of various epoxies both during and after curing (for potential use in the assembly of microelectronic hybrids). Figure 8 illustrates the use of the residual gas analysis technique in determining both effective epoxy cure cycles and the proper solvent for postcure cleaning. Full details of this work were published previously.[14]

ATOMIC ABSORPTION SPECTROSCOPY

Atomic absorption spectroscopy has played an important role in the evaluation and quality assurance of space, medical, and underwater microelectronic devices. It has been used to identify and quantify impurities in support of energy-dispersive X-ray, Auger, and secondary ion mass spectrometry analyses. For example, the cadmium impurity (indicated in the energy-

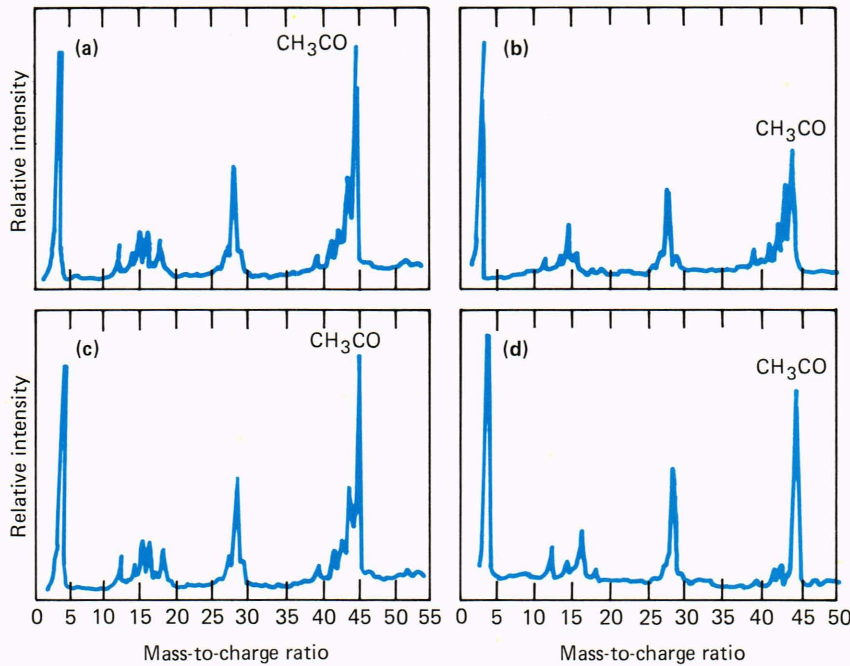


Figure 8—Postcure residual gas analysis spectra for Ablefilm 517 epoxy used in microelectronic circuits: (a) short cure process; (b) long cure process; (c) TCE solvent treatment after short cure; (d) alcohol solvent treatment after short cure.

dispersive X-ray spectrum, Fig. 4) was accurately determined by means of atomic absorption to be 2.2 percent.

X-RAY DIFFRACTION

Identification of the polycrystalline phases present in a sample is facilitated by X-ray diffraction analysis.[8] Each distinct phase or crystalline form of an element or compound produces a distinct X-ray diffraction pattern that may be used to identify the presence of that particular phase. X-ray diffraction responds to the crystalline structure as well as to the chemical composition of a material, in contrast to chemical analyses or other advanced materials analysis methods that are often insensitive to the crystalline structure. For example, if a sample contains microcrystallites of the compounds AB, A_2B_3, and A_3B_2 (where A and B are chemical elements), most forms of chemical analysis would show the presence and relative amounts of the elements A and B but would give no information concerning the crystalline structure of the compounds or the relative abundance of the microcrystallite phases in the sample.

X-ray diffraction patterns were obtained on thin-film couples of aluminum and gold. Interplanar spacings (*d*-values) derived from the patterns were compared directly with values listed in the Powder Diffraction File, thus enabling the phases present in the films to be identified (Table 1). Wire-bond reliability, particularly the formation of uncontrolled aluminum-gold intermetallic growth, has been of great concern in microelectronic hybrids. Studies of bonding methods and the formation of intermetallic structures, especially in the form of thin-film reaction couples, have aided materially in improving wire-bond reliability[16]

Table 1—Prominent lines present in an X-ray diffraction pattern of aluminum-gold couple compared with standard strong lines[15] for Al_2Au_5, Al_2Au, $AlAu_4$, and AlAu. Al_2Au_5 appears to be the principal phase component in the sample.

Observed d-Values				
AlAu Couple	Al_2Au_5	Al_2Au	$AlAu_4$	*AlAu*
3.87	3.71			4.60
3.50	3.37	3.46		3.19
3.23		3.00	3.09	3.16
3.12	2.97		2.83	2.94
2.82	2.81			
2.74	2.73			
2.54	2.52			
2.46	2.45			
2.42	2.42			
2.34	2.33		2.31*	2.31
2.27	2.26*			
2.22	2.23*			
2.18		2.12	2.19	2.19*
2.10	2.10			2.14
2.09			2.09	2.03
1.93	1.93			1.97
1.88	1.86	1.81*		
1.85	1.78	1.73	1.63	1.66
		1.50	1.55	1.59
				1.48
		1.38	1.36	
		1.34	1.28	
		1.22		
		1.15	1.17	

*Indicates strongest lines.

with applications to space, biomedical, and underwater microelectronic systems.

AUGER MICROPROBE ANALYSIS

Auger analysis has been crucial in developing cleaning processes and evaluating residual contaminants from curing epoxies. For example, Fig. 9 shows Auger spectra of aluminum-metallized silicon substrates exposed to vapors produced during the cure of Ablefilm® 517 and 550 (Ablefilm is a tradename of the Ablestick Corp., California). The substrates were placed near curing epoxy samples that consisted of two sandwiched stacks of 10 single layers of epoxy between 11 alumina (polycrystalline Al_2O_3) substrates (7.5 by 25 millimeters). The metallized substrates were subjected to a 10-minute ultraviolet/ozone cleaning treatment[17] before the experiments. No absorbed species were detected during the cure of Ablefilm 517 (the carbon signal was essentially at the noise level), but a large carbon signal and a nitrogen trace were detected on the substrate exposed during the cure of Ablefilm 550. Previous studies[18] have shown that Ablefilm 550 outgasses more hydrocarbon species than Ablefilm 517, results that agree with the Auger analysis. (The nitrogen species probably originates from the amine curing agent.) Auger analysis has also shown that surface diffusion of epoxy-related species[6] on metallized microelectronic circuits may prove to be a serious problem for subsequent processing and ultimate reliability.

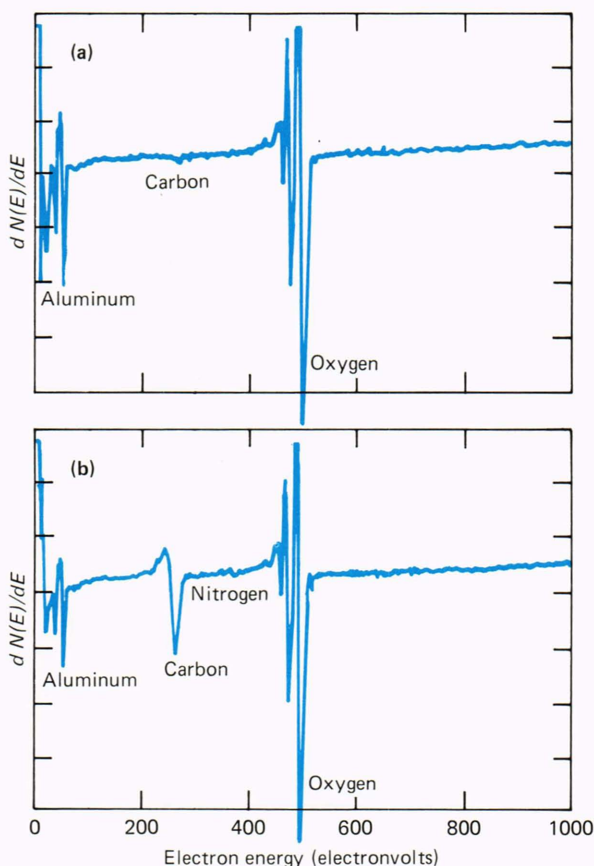


Figure 9—Auger spectra of aluminum on silicon substrates placed near epoxy samples during cure. The substrates were cleaned with ultraviolet/ozone prior to the experiment. (a) Exposed during cure of Ablefilm 517. (b) Exposed during cure of Ablefilm 550. *dN(E)/dE* is the energy derivative of the number of Auger electrons.

SECONDARY ION MASS SPECTROMETRY ANALYSIS

Secondary ion mass spectrometry has been an important tool in evaluating medical implant systems and in selecting components and materials for use in hybrid production. Moreover, it has provided materials identification (it is sensitive to all elements, even hydrogen, and to complexes to some degree) and depth profiles in intermetallic studies. This method has also shed light on package plating problems, various failure analyses, and cleaning processes. Figure 10 is an example of profile results for a series of cleaning and bonding studies. A capacitor end-cap leaching problem[19] was solved this way.

Table 2 presents the relative compositions of the end-cap metallizations from various vendors and processes. The two units prone to leaching (from two different vendors) showed high zinc content. Such an impurity could easily be screened by secondary ion mass spectrometry on a lot sample basis to improve the product assembly yield and hence overall microelectronic system reliability.

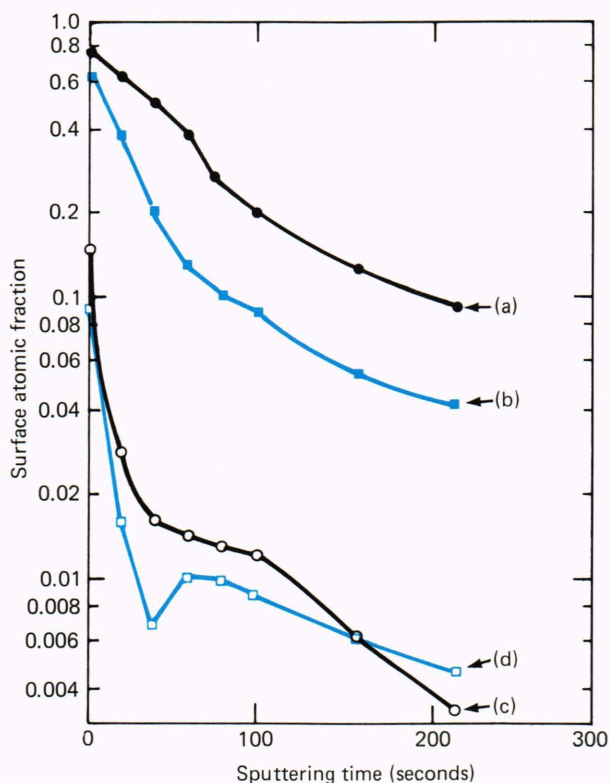


Figure 10—Secondary ion mass spectrometry of carbon on photoresist contaminated aluminum metallization (on silicon): (a) contaminated; (b) solvent-cleaned (TCE/acetone/alcohol); (c) ultraviolet/ozone cleaned (10 minutes); (d) uncontaminated control.

Table 2—Composition of various chip capacitor end caps determined by secondary ion mass spectrometry. Data listed are the relative atomic fractions. Lots CO and PL displayed severe leaching.[18]

Species	*Capacitor Lot*				
	CO	*CN*	*PO*	*PN**	*PL*
Ag	0.09	0.13	0.061	0.009	0.044
Pd	0.10	0.15	0.047	ND**	0.038
Zn	0.21	0.042	0.057	ND	0.18
Cd	0.014	0.021	0.074	ND	0.082
Sn	0.003	0.009	ND	0.25	ND
Pb	0.10	0.091	ND	0.3	0.005
Zr	0.004	0.026	0.002	0.001	0.006
Bi	0.32	0.39	0.62	0.28	0.57
B	0.079	0.10	0.084	0.042	0.049
Al	0.079	0.066	0.051	0.013	0.019
K	0.001	0.001	0.0003	0.001	0.0001
Ni	ND	ND	0.001	0.10	ND
Co	0.001	0.002	0.001	ND	0.0001

*Lot and caps were solder dipped.
**ND = not detected.

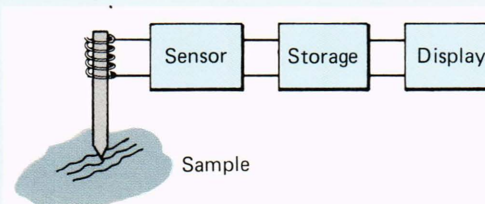
SUMMARY

Various advanced materials analysis, inspection, measurement, and characterization techniques have been used in the last few years to aid in evaluating high-reliability microelectronic systems. The ability to view specimens clearly in three dimensions, such as is provided by the scanning electron microscope with its nearly unlimited depth of field, has been invaluable. Materials identification provided by wavelength and energy-dispersive X-ray techniques, Auger electron spectroscopy, and secondary ion mass spectrometry have aided in the development of new materials and processes. This is particularly true in the areas of organic adhesives, substrate and component cleaning, and photolithography. The ability to track impurities as a function of distance from a sample surface (regression Auger and secondary ion mass spectrometry) has allowed us to distinguish surface-adsorbed species from bulk impurities.

The techniques of atomic absorption (which provide accurate quantitative analysis for selected materials in trace quantities), X-ray diffraction (for crystallographic structure identification), and residual gas analysis (with its ability to identify volatile species) have played significant roles in evaluating sophisticated microelectronic systems. Surface profilometry and beta-backscatter thickness techniques allow the needed checks on microcircuit dimensions and layer thicknesses to ensure high yields and ultimate device reliability.

NONDESTRUCTIVE THICKNESS MEASUREMENTS BY STYLUS PROFILOMETRY

Stylus profilometry is conducted with a microprocessor-controlled measurement apparatus in which a diamond stylus of small radius (typically 5 micrometers in diameter) is loaded with a downward force (nominally 5 to 25 milligrams) and is drawn across the surface to be profiled. Any vertical displacement of the stylus is detected by a linear variable differential transducer. The transducer signal is electronically amplified and digitized. In modern machines, the digitized scan signal can be stored, expanded, smoothed, and sent to a hard-copy printer or mainframe computer for further processing. The digitized data can be displayed on a monitor and can be used to measure the height of a step, line width, average surface roughness, cross-sectional area maximum height, or average height. This technique is able to resolve vertical details as small as 1 nanometer and horizontal dimensions down to 0.1 micrometer. The use of stylus profilometers is covered by ISO Standard 4518.



Block diagram of the stylus profilometer.

Sloan Dektak IIA stylus profilometer.

SCANNING ELECTRON MICROSCOPY

Elementary atomic physics predicts that when an electron with enough energy strikes and penetrates a solid, it will be scattered by a core electron in an atom on or near the material's surface. In a simplified view of the process, the incident (primary) electron is backscattered, a core electron is ejected from the atom (the emitted electron is called a secondary electron), and the atom is left in an excited state. The excited atom can decay by emitting an X ray, the energy of which depends on the spacing of the electron energy levels of the atom in question and, thus, can be used for element (atom) identification. This is the basis for the energy dispersive X-ray spectrometer. The excited atom can also decay in a two-step process in which an excited state electron decays to a lower energy state, and a second electron (usually a valence bond electron) simultaneously is emitted with a specific energy that is indicative of the particular element. The emitted electron is called an Auger electron. Detection and energy analysis of Auger electrons form the basis of scanning Auger microscopy. Auger transitions are more prevalent in the lighter elements, while X-ray transitions dominate in materials with high atomic numbers.

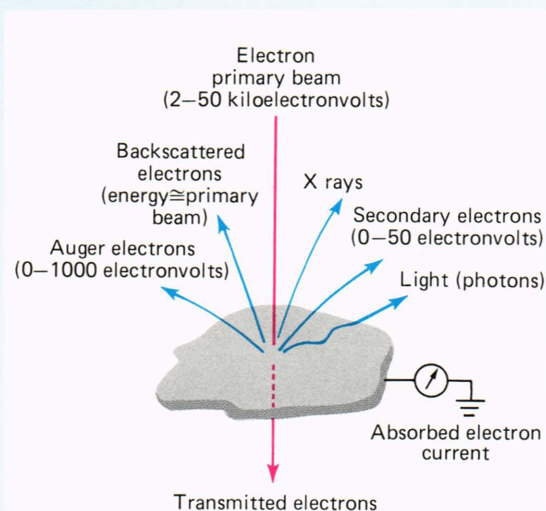
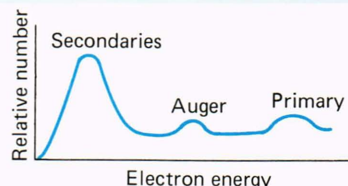


Diagram of possible electron beam interaction with a sample.

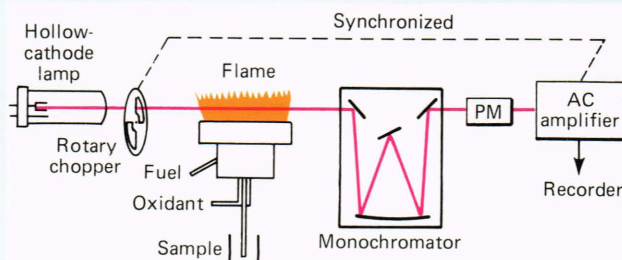


Energy distribution of response electrons

The Prince Gamma Tech System 4 energy dispersive X-ray system. The screen illustration is a digital dot map of hybrid metallization.

ATOMIC ABSORPTION

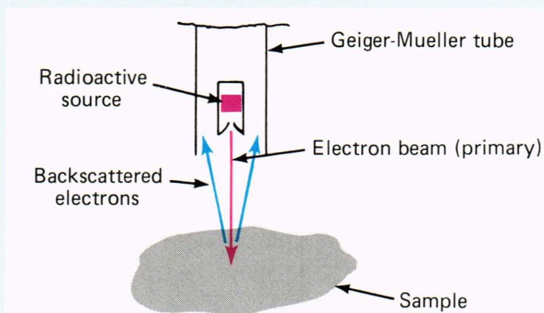
In an atomic absorption spectrometer, a specimen to be analyzed is dissolved in a suitable liquid and vaporized by passing it through a suitably hot flame. The burning causes some of the specimen material to dissociate into its individual constituent atoms (or ions). If the dissociated atoms are irradiated with light of the proper wavelength, the intensity of the light is reduced as a result of absorption caused by the excitation of electrons in the free atoms. This reduction in intensity of the light beam is directly proportional to the concentration of the specimen in the flame. Each material absorbs at a different wavelength so that a light source with a different wavelength output is required for each material to be identified and quantified. A spectrophotometer is used to measure the intensity of the transmitted light at the appropriate wavelength (with no sample present) and to quantify the intensity loss or absorption produced by introducing the specimen into the flame. Detection levels in atomic absorption spectroscopy are in the range of less than one part per million, and overall absolute quantitative analysis can be obtained with an accuracy of ± 2 percent.



Schematic diagram of an atomic absorption spectrometer.

NONDESTRUCTIVE THICKNESS MEASUREMENTS BY THE BETA-BACKSCATTER TECHNIQUE

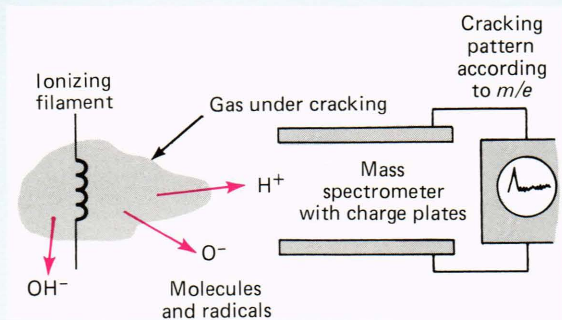
In the beta-backscatter technique, a beam of energetic electrons (nominally 100 kiloelectronvolts) from a radioactive source (typically promethium-147, carbon-14, or thallium-204) is allowed to impinge upon the sample. The total number of electrons backscattered during a specified sampling time is measured using a Geiger-Mueller tube that is placed coaxially in line with the radioisotope source and the material being tested. That number is a function of thickness and the atomic number of the material being bombarded. If a heavier element (high atomic number, e.g., gold) is plated or deposited on a lighter element substrate (low atomic number, e.g., aluminum), the number of backscattered electrons is substantially greater than the number from the uncoated substrate because of the higher atomic number of the atoms in the coating film. If the lighter element is plated on the heavier element substrate, the number of backscattered electrons is reduced (compared with the number from the uncoated substrate) because of the lower emission of the lighter element material and the absorption of substrate emissions by the coating film. If the detected signals from the unknown are compared with those from known thickness reference standards (pure substrate material, pure coating material, and two samples of known coating thickness on the substrate material), the number density of the coating element and, hence, the coating thickness can be determined. The system can also be used to determine the composition of certain alloys (e.g., tin-lead solder). The use of beta-backscatter measurement techniques is covered by ASTM Standard B567-79a and ISO Standard 3543.



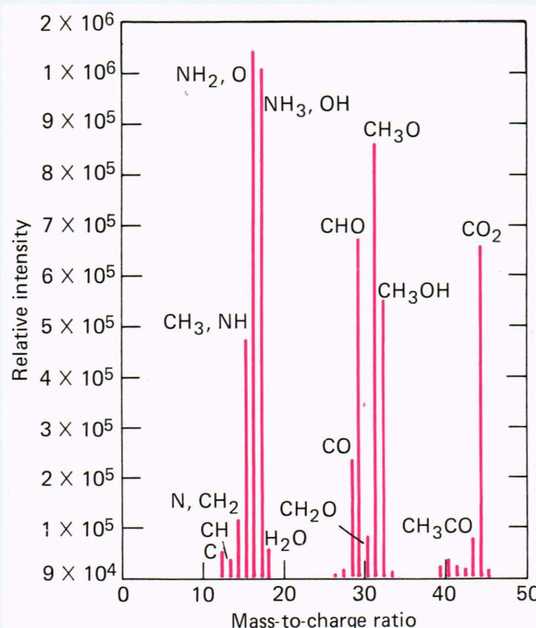
Schematic diagram of beta-backscatter analysis system.

RESIDUAL GAS ANALYSIS

In residual gas analysis, a hot filament is used to dissociate and ionize species of a gas specimen. The ionized particles are separated according to the mass-to-charge ratio (m/e) in either a monopole or a quadrupole mass analyzer. The relative intensity of each m/e peak (i.e., the number of ions at a particular mass-to-charge ratio) is then displayed to yield a spectrum or "cracking" pattern for the particular gas spectrum.



Schematic diagram of a residual gas analysis system.

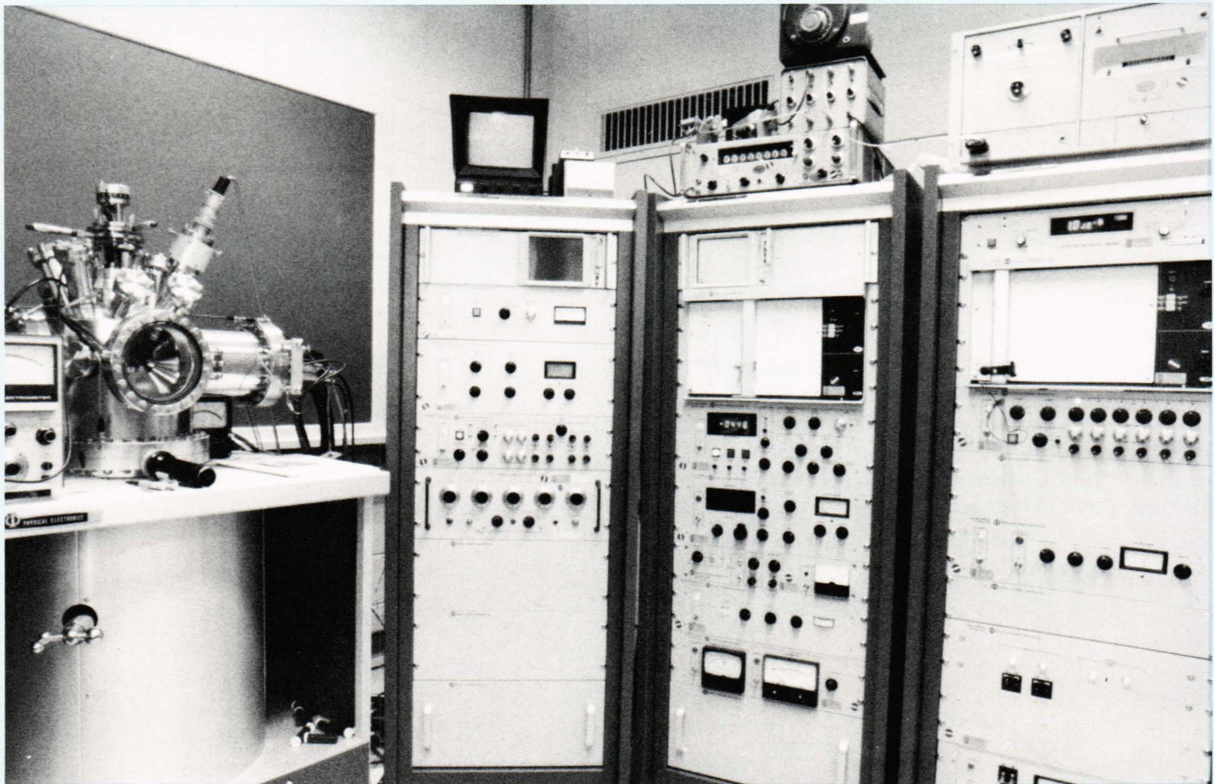


Residual gas analysis cracking pattern (0 to 50 atomic mass units) for a typical epoxy gas evolved from epoxy resins.

AUGER ELECTRON SPECTROSCOPY

If the number of response electrons resulting from the bombardment of a solid with a beam of primary electrons of energy (*Ep*) is plotted as a function of energy, three distinct regions result. The regions correspond to (a) a peak in the curve at *Ep* representing elastically scattered (backscattered) primary electrons, (b) a peak at 0 to 50 electronvolts representing the secondary electrons (as described under scanning electron microscope theory), and (c) a region between the two peaks that has low intensity and slope (Fig. 2). With sufficient amplification, the small peaks of that region (50 to 1000 electronvolts) can be detected. They correspond to the Auger electron emission. By taking the derivative of the number of response electrons with respect to energy, the Auger peaks are more readily identified. The specific energy locations for the Auger peaks of a given element are unique, and thus material can be identified.

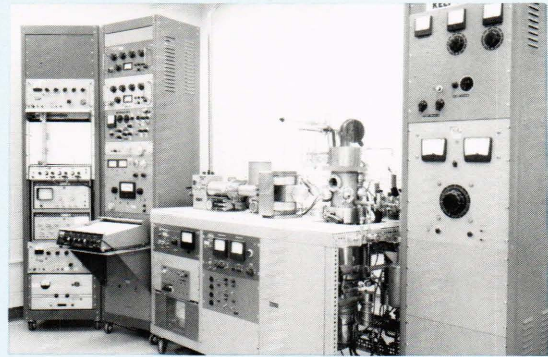
Because of the large inelastic scattering cross section for electrons in solids, Auger electrons can be detected only from atoms within about 1 nanometer of the surface, a true surface analysis technique. Although the short inelastic mean free path causes Auger electron spectroscopy to be very sensitive to the surface composition of solids, it also complicates the analysis at greater depths. To determine composition at depths greater than or equal to 10 nanometers, Auger analysis must be combined with a technique for material removal, such as inert gas ion sputtering. As the surface is regressed because of sputtering, continuous Auger analyses are performed on the freshly exposed surface. Depth profiling is an important Auger technique and has been applied in many microelectronic activities along with more conventional top monolayer surface analysis.



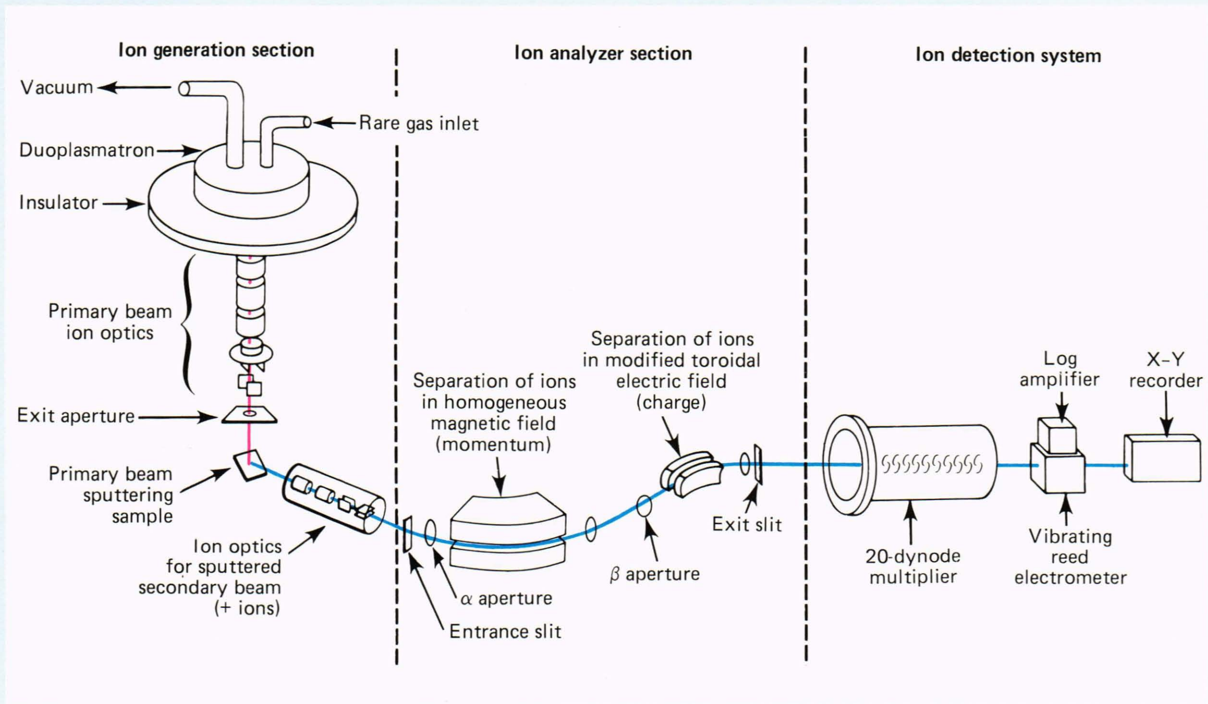
APL's Perkin Elmer scanning Auger spectrometer. The ultrahigh vacuum sample chamber, detector, and sputtering source are shown at the left.

SECONDARY ION MASS SPECTROMETRY

When an energetic ion (typically 1 to 20 kiloelectronvolts) collides with a solid surface, atoms and molecular species from the material under analysis are ejected with a certain distribution in energy and angle. Depending on the impact energy, angle of incidence, and nature of the bombarding ion, some fraction of the sputtered atoms and molecules escapes from the sample as positive or negative ions (other escaping material corresponds to neutral atoms and atom clusters). These secondary ions, which are representative of the composition of the outermost atomic layers of the materials under analysis, can be collected by a mass spectrometer and separated and detected on a mass-to-charge ratio (m/e) basis. They provide a qualitative and, with proper standards, quantitative analysis of the near-surface region (one to three monolayers thick). The detection and identification of the sputtered secondary ions constitute a secondary ion mass spectrometry analysis. The method analyzes only the material removed from a surface by sputtering, while Auger electron spectrometry analyzes the outer few atomic layers present at the sample surface without substantial material removal. Hence, the former method is inherently destructive to the specimen while the latter is nondestructive (when used in the nonprofiling mode). However, this method provides much more information than the Auger method about compounds, molecules, and other complexes present at the sample's surface. Surface depth regression occurs automatically as a function of time, and depth profiles are easily obtained by monitoring a particular m/e peak with time. Under proper circumstances and for some atomic species, the sensitivity of secondary ion mass spectrometry can be at the parts per billion level.



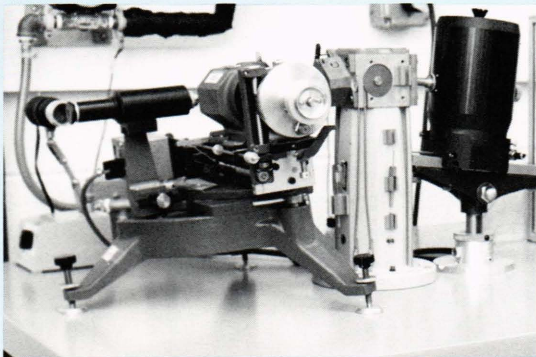
APL's GCA Corp. IMS 101-B secondary ion mass spectrometer.



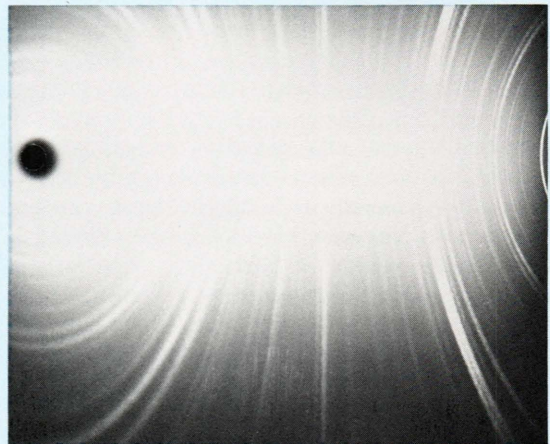
Schematic diagram of a secondary ion mass spectrometer.

X-RAY DIFFRACTION

The quantitative theory of X-ray diffraction by crystalline solids is quite complicated. Diffraction is a physical phenomenon that exists because of the wave nature of X rays. Because the spacing between atomic lattice planes in a crystal is of the same order of magnitude as a wavelength of X rays (a few tenths of a nanometer), reflection of the X rays from various lattice planes can interfere constructively or destructively, depending on the direction of the X-ray beam relative to the lattice planes. When many crystallites are randomly oriented with respect to a collimated X-ray beam, the X rays are reflected (diffracted) in such a way as to exhibit intensity maxima at certain angles that are characteristic of the lattice spacing. If the sample is a three-dimensional solid, the diffraction maxima are along conical projections having their apexes at the points where the X-ray beams strike the samples and with characteristic vertex angles. A photographic film that is sensitive to X rays intercepts them, and the projection of the conical segments on the plane film produces a series of exposed arcs. The set of arcs is called a powder diffraction pattern; the spacing and intensities (for a known configuration of X-ray beam, sample, and film plane) are the "fingerprints" by which crystalline phases are identified. Identification is accomplished by comparing the measured diffraction pattern with those of over 30,000 phases that have been identified and tabulated in the Powder Diffraction File.



X-ray diffraction equipment. A Seifert 180-1000 stabilized X-ray generator with a chromium target (vanadium filter). The Read X-ray camera is visible in the upper center.



X-ray diffraction pattern of an aluminum-gold couple (approximately 2 parts aluminum to 5 parts gold) on glass fired at 250°C for several hours.

REFERENCES

[1] H. K. Charles, Jr., B. M. Romenesko, and D. O. Shapiro, "Inspection and Failure Analysis of Hybrid Microelectronics Using Scanning Electron Microscope Techniques," *Int. J. Hybrid Microelectron.* **4**, 139 (1981).

[2] J. M. Bennett and J. H. Dancy, "Stylus Profiling Instruments for Measuring Statistical Properties of Smooth Optical Surfaces," *Appl. Opt.* **20**, 1785 (1981).

[3] J. A. Weiner, O. M. Uy, H. K. Charles, Jr., B. M. Romenesko, and R. vonBriesen, "Beta-Backscatter Thickness Measurement Applications in Microelectronic Fabrication and Control," *Int. J. Hybrid Microelectron.* **6**, 462 (1983).

[4] R. L. Meek, "Plating Thickness Measurements," *Plating Surf. Finishing*, 528 (1975).

[5] D. B. Holt, M. D. Muir, P. R. Grant, and I. M. Boswarva, eds., *Quantitative Scanning Electron Microscopy*, Academic Press, New York (1974).

[6] R. C. Benson, B. H. Nall, F. G. Satkiewicz, and H. K. Charles, Jr., "Surface Analysis of Adsorbed Species from Epoxy Adhesives used in Microelectronics," in *Proc. 6th Symp. on Applied Surface Analysis* (1984).

[7] A. W. Czanderna, ed., *Methods of Surface Analysis*, Elsevier Scientific Publishing Co., New York (1975).

[8] L. Azaroff, *Elements of X-Ray Crystallography*, McGraw-Hill, New York (1968).

[9] H. Liebl, "Secondary-Ion Mass Spectrometry and Its Use in Depth Profiling," *J. Vacuum Sci. Tech.* **12**, 385 (1975).

[10] D. R. Thomerson and K. C. Thompson, "Recent Developments in Atomic Absorption Spectrometry," *Am. Lab.*, 53 (1974).

[11] H. K. Charles, Jr., J. T. Massey, and V. B. Mountcastle, "Polyimides as Insulating Layers for Implantable Electrodes," in *Polyimides: Synthesis, Characterization and Application*, K. L. Mittal, ed., Vol, 2, p. 1139, Plenum Press, New York (1984).

[12] H. K. Charles, Jr., and B. M. Romenesko, "Ceramic Chip Carrier Soldering, Cleaning and Reliability," in *Proc. 32nd Electronic Components Conf.*, IEEE, p. 369 (1982).

[13] R. E. Hicks, H. K. Charles, Jr., G. D. Wagner, and B. M. Romenesko, "Trends in Medical Electronics Using Surface Mounted Components and Hybrids," *Int. J. Hybrid Microelectron.* **6**, 283 (1983).

[14] E. S. Dettmer, H. K. Charles, Jr., R. C. Benson, B. H. Nall, F. G. Satkiewicz, C. B. Bargeron, and T. E. Phillips, "Epoxy Characterization Using Mechanical, Electrical and Surface Analysis Techniques," *Int. J. Hybrid Microelectron.* **6**, 375 (1983).

[15] Powder Diffraction File, Joint Committee on Powder Diffraction Standards, 1601 Park Lane, Swarthmore, Pa. 69081.

[16] G. V. Clatterbaugh, J. A. Weiner, and H. K. Charles, Jr., "Gold-Aluminum Intermetallics: Ball Bond Shear Testing and Thin Film Reaction Couples," in *Proc. 34th Electronic Components Conf.*, IEEE, p. 21, (1984).

[17] J. A. Weiner, G. V. Clatterbaugh, H. K. Charles, Jr., and B. M. Romenesko, "Gold Ball Bond Shear Strength: Effects of Cleaning, Metallization and Bonding Parameters," in *Proc. 33rd Electronic Components Conf.*, IEEE, p. 208 (1983).

[18] H. K. Charles, Jr., B. M. Romenesko, G. D. Wagner, R. C. Benson, and O. M. Uy, "The Influence of Contamination on Aluminum-Gold Intermetallics," in *Proc. Int. Reliability Physics Symp.*, IEEE, p. 128 (1982).

[19] H. K. Charles, Jr., and J. A. Weiner, "Capacitor End Cap Testing and Analysis," in *Proc. 4th Capacitor and Resistor Technology Symp.*, Components Technology Institute, Inc., p. 180 (1984).

ACKNOWLEDGMENTS — The authors acknowledge the efforts of D. O. Shapiro for scanning electron microscope microphotography, energy dispersive X-ray spectra collection, and atomic absorption analysis; B. H. Nall for Auger spectroscopy; F. G. Satkiewicz for secondary ion mass spectrometry data collection and analysis; and R. C. Benson for residual gas analysis. Special thanks are extended to J. I. Bittner for her assistance.

THE AUTHORS

HARRY K. CHARLES, Jr. (right), received the B.S.E.E. degree from Drexel University and the Ph.D. degree in electrical engineering from The Johns Hopkins University in 1972. After completing a post-doctoral research appointment in the Research Center of APL, he joined APL's Microelectronics Group, which he now supervises. His current research and development interests include the study of contacts and interfacial phenomena in hybrids and surface-mounted microelectronic assemblies. He has published over 55 papers in these and other fields.

Dr. Charles is a senior member of the IEEE and a member of the American Physical Society, the International Solar Energy Society, the Electron Microscopy Society of America, the Microbeam Analysis Society, and the International Society for Hybrid Microelectronics.

NORMAN A. BLUM (left) was born in Boston in 1932. He received the A.B. in physics from Harvard in 1954 and the M.S. and Ph.D. degrees in physics from Brandeis University in 1959 and 1964, respectively. He served in the U.S. Navy as a line officer from 1954 to 1957. He was a staff member at MIT's Francis Bitter National Magnet Laboratory from 1960 to 1966 and a visiting scientist there from 1966 to 1970. Dr. Blum was a member of the senior scientific staff at NASA's Electronics Research Center from 1966 to 1970. Since 1970, he has been a senior physicist at APL, where he is supervisor of the Microelectronics Substrate Processing Section.

Dr. Blum is a member of the American Physical Society, the IEEE, and the International Society for Hybrid Microelectronics.

JOEL A. WEINER (standing), born in Brooklyn, N.Y., received B.S. and M.S. degrees in chemistry from The City University of New York (1970) and from Rutgers University (1974).

Since 1982, he has been a senior staff chemist at APL where he is supervisor of the Inspection and Quality Assurance Section for the Microelectronics Group. Before joining APL, Mr. Weiner was involved in ion beam and plasma processing, autocatalytic metal deposition and surface chemistry, and thin-film deposition and analysis. He is a member of the International Society for Hybrid Microelectronics and Sigma Xi.