THIN FILM ANALYZERS

The term *thin films* has been applied to coatings whose thicknesses range from less than an atomic monolayer to films on the order of 1 mm thick. In addition to their well-known use as decorative or protective finishes, thin films of various types have found extensive use in the fields of electronics and optics.

This article describes a number of different thin film analyzers. Stated properly, since each film analyzer uses a number of different instruments, this article treats a number of different techniques of film *analysis* (i.e., methods and instruments used to determine experimentally such parameters as film thickness, structure, optical behavior, and material content of various types of films).

A very important application of thin films is in the field of semiconductor electronics. Although semiconductor circuits are generally fabricated on chips that are on the order of 0.5 mm thick, it is a combination of thin layers at the top of the chip that performs the electronic functions. Though structurally integral with the rest of the chip, these active layers can therefore be considered to be a set of thin films. Consequently, experimental methods of determining the structural and material content of such top layers are also those used in the analysis of thin films.

OPTICAL METHODS OF ANALYSIS

Reflectivity

The partial reflection of light at the interface between two materials of different index of refraction permits the measurement of the thickness of a transparent film. Specifically, when light of a range of wavelengths from a source in air strikes the front surface of a film of index of refraction n_1 and thickness t_1, some of this light is reflected, and the rest is transmitted. Similarly, if the slab is either free standing or mounted on a medium of refractive index different from n_1, some of the transmitted light is reflected from the back surface, reaches the front surface, and is again partially reflected and partially transmitted toward the source. This process is repeated ad infinitum, so that the total amount of light reflected toward the source is the sum of these individual waves, each with a phase determined by the index of refraction of the film, its thickness, and the frequency of the light. The result of this interference effect is a reflectance (i.e., the ratio of reflected to incident light), with peaks at frequencies governed by n_1, t_1, and the angle of incidence of the incoming light. The film thickness can be extracted by determination of several of these peak frequencies (1,2). By using a white light source and dispersing the reflected light with a spectrometer unto a diode array, the technique can be set up for automatic data acquisition (3,4).

Alternative methods exist, such as the use of interference of light reflected from a variable thickness reference with that from the film under test, the use of a Fourier transform spectrometer, and the use of a monochromatic light source with variable angle of incidence (5–7).

For visible light, the interference technique is effective for films of thickness greater than 2000 Å.

Because of the relation of the color of light to its wavelength, the interference technique is well known in the semiconductor industry, where the thickness of a film of SiO_2 or Si_3N_4 on a silicon wafer can be easily determined by observation of the color of the light reflected from the wafer.

Transmission

Because of wave interference effects, the transmittance of a free-standing transparent film oscillates with wavelength λ. Maximum transmission for normal incidence of light occurs when the thickness of the film is an integral number of half-wavelengths (in the film). As a result, for transparent films it is found that the film thickness t_1 is given by $t_1 = (1/2n_1)(1/\lambda_a - 1/\lambda_b)^{-1}$, where λ_a and λ_b are free-space wavelengths corresponding to two adjacent maxima of transmission in the plot of transmittance versus λ (1).

If the film contains materials that absorb energy at certain frequencies within the spectrum of electromagnetic waves, these absorption peaks manifest themselves as attenuation peaks of the transmitted signal. *Transmission spectroscopy,* a technique that is very popular in the chemical industry, is the method of detecting the presence and quantity of such materials by finding the presence and magnitude of such absorption lines. Methods for converting the attenuation to the density of the absorbing material are given in the literature.

While absorption lines can be found by scanning through the spectrum with a broad spectrum source and a monochromator, a preferred method, with a great improvement in signal-to-noise ratio, is Fourier transform infrared spectroscopy (FTIR), which uses a broad spectrum source (8–10). The spectral distribution of the transmitted signal is found by taking the fast Fourier transform of the interferogram that is obtained when the transmitted signal is used as the source of a Michelson interferometer. (The interferogram is the signal output versus location of the movable mirror of the interferometer.)

Ellipsometry

The technique of ellipsometry is suitable to measurement with great precision of the thickness and index of refraction of thin, nonabsorbing films on highly absorbing substrates.

In basic ellipsometry, a beam of light that is linearly polarized at an angle with respect to the plane of incidence impinges on the film/substrate system. The components that are vertically and horizontally polarized with respect to the plane of incidence are reflected with different intensities and phases, thereby combining into an elliptically polarized beam. If the complex index of refraction of the substance is known, the reflection coefficients (i.e., the complex ratio of reflected to incident electric field for each of the two polarizations) can be used to compute the index of refraction and thickness of the film. Films as thin as 10 Å, as well as others thousands of angstroms thick, have been measured by this ellipsometric method (11,12).

A convenient method of ellipsometry uses calibrated computer-controlled stepping motors to vary polarizations until a

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null output is found. Another system, the rotating analyzer ellipsometer, has high speed and high accuracy but makes more stringent demands on the optical system (13,14).

In a dynamic application of the technique, ellipsometry carried out at several frequencies has been used to monitor and control growth rate, layer thickness, substrate temperature, and ternary alloy composition in a III–V semiconductor molecular beam epitaxy system (15).

STRUCTURE

Profilometry

For some applications of thin films it is important to know the degree of flatness or the contours of a surface. An instrument used to carry out such measurements is called a profilometer.

Profilometer. A mechanical instrument that has been in use for a number of years plots a contour map by running a stylus along a surface with constant force. As an example of the performance of such an instrument, following are specifications given for the Dektak IIA Surface Profile Measuring System, distributed by Sloan Technology Corp., Santa Barbara, California:

Vertical resolution: 5 Å
Horizontal resolution: 500 Å
Stylus force: 50 mg (adjustable)
Scan length: 25 μm to 30 mm

Scanning Tunneling and Atomic Force Microscopes. A great advance in resolution occurred with the invention of the scanning tunneling microscope (STM) (16) and atomic force microscope (AFM) (17). (The latter is also known as scanning force microscope.)

In the STM a sharp metal tip is scanned a few angstroms above an electron-conducting surface. With a few volts applied between tip and surface, an electron tunneling current flows, of magnitude exponentially dependent on the separation. As the tip is scanned over the surface, a feedback circuit maintains constant tunneling current by adjusting the tip height via a piezoelectric transducer on which the tip is mounted. A plot of the voltage applied to the transducer results in a contour image that has atomic resolution.

Alternatively, with the feedback turned off and scanning performed at constant height, variations in the tunneling current can result in an atomic resolution “current image.”

Differences in the contour heights can be revealed to better than 1/100 of an atomic diameter. The lateral resolution is determined by the radius of protrusions at the tip. (A tip terminating in a single atom will have atomic lateral resolution.)

Since an STM yields a very detailed image of a surface, it can be used for surface metrology, such as measuring the line widths of lithographic patterns.

An atomic force microscope is an ultrasensitive profilometer that can produce contour images of both conducting and insulating surfaces with atomic resolution. A tip of a material such as silicon or silicon nitride that has been fastened to the end of a cantilever of a light material, such as aluminum foil, is placed in contact with the surface to be scanned. As the surface is moved under the tip, the cantilever deflects with the topography. In one configuration, a laser beam reflecting off the cantilever falls on a two-element photodetector, which provides a feedback signal to a piezoelectric transducer that controls the tip height so as to maintain a constant (small) force between tip and surface. A plot of the piezoelectric voltage necessary to maintain this constant force is a contour map of the surface, with atomic resolution.

Extensions of the STM and AFM have resulted in a magnetic force microscope that is capable of mapping the domain pattern of thin magnetic films (18) and in an electrostatic force microscope that can measure potential or doping variations in semiconductor circuits (19–21).

Low-Energy Electron Diffraction

Low-energy electron diffraction (LEED) uses the wave nature of electrons to reveal the crystal structure of the very surface of a film (22). In LEED an electron beam of energy range 20 eV to 300 eV and of narrow energy spread is shot normally at the film. A fraction of the incident electrons is backscattered (reflected). Those that are backscattered elastically from the top few atomic layers form an electron diffraction pattern that reveals the geometry of the surface. To display this pattern in a LEED instrument, all of the backscattered electrons are first caused to pass through a set of retarding grids, to remove those that have lost some energy. The remaining ones are accelerated, to strike a fluorescent screen and display the desired diffraction pattern. Since changes in the surface, such as by adsorption of foreign molecules, often introduce new periodicities in the surface placement of atoms, these changes can be detected by observation of the displayed pattern.

Since even at a relatively good vacuum of 10^{-6} torr a surface will become contaminated in seconds, LEED measurements must be carried out with the surface that is being probed in an ultrahigh vacuum (10^{-10} torr or better).

Reflection High-Energy Electron Diffraction

In reflection high-energy electron diffraction (RHEED), also called HEED, electrons of energy of around 10 keV to 100 keV are shot at a surface at a glancing angle. The resulting diffraction pattern of these forward-scattered electrons provides information about the smoothness of a surface. Since electrons are scattered forward, it is possible in some cases to use RHEED for continuous monitoring during controlled growth of a film, such as in the deposition of thin films by molecular beam epitaxy (23).

Scanning Electron Microscopy

In scanning electron microscopy (SEM) an electron beam of approximately 10 Å to 50 Å diameter is scanned across the surface of the film that is to be analyzed. The incident electrons cause secondary electrons to be emitted. By collecting these secondaries or the backscattered incident primary electrons and displaying their intensity on a monitor, an image that relates to the film’s surface structure is obtained (24–26). Since an electron’s DeBroglie wavelength can be much smaller than wavelengths of visible light, the so-called far field resolution of an electron microscope is much finer than that of an optical microscope. (The wavelength of a 10 keV electron is 0.000012 μm, while the wavelength of visible light is in the range 0.4 μm to 0.7 μm.)

Transmission Electron Microscopy

In transmission electron microscopy (TEM), electrons are accelerated to energies of 100 keV to 500 keV, for transmission through and imaging of structures of films of thickness from a single monolayer up to a few thousand Ångstroms (27). TEM is therefore analogous to optical transmission microscopy (OTM), but with a higher degree of resolution. However, unlike in OTM, where images are formed principally because of absorption, in TEM image formation is due to electron scattering or diffraction by the film.

The electrons that pass through a film in TEM form a diffraction pattern in the so-called focal plane and an image in another plane, the corresponding image plane of the electron lens system. Both can be displayed on a fluorescent screen with additional electron lenses, to provide information on crystalline phases, amorphous regions, and crystalline imperfections.

To obtain detailed local information, electrons are focused to a small spot and scanned across the film. This method is thus the transmission equivalent of SEM.

MATERIAL ANALYSIS

The preceding text has discussed what can be called the mechanical structure of a film or its surface. The following deals with determining the material composition or chemical structure of films.

Photoluminescence

Photoluminescence (PL) is a nondestructive technique that can detect the presence of certain materials, such as impurities in semiconducting films (28). To carry out PL, the specimen of interest is cooled to near liquid helium temperature and illuminated with light whose photon energy exceeds the semiconductor's bandgap, to create electron–hole pairs. When recombination takes place, the determination of the wavelengths of the recombination radiation produced identifies the impurities.

Raman Spectroscopy

In the analysis technique based on the Raman effect, a film is illuminated with a laser, the "pump," and the radiation emitted by the sample is examined for the presence of light whose frequencies differ slightly from the pump frequency (29). The amounts of the frequency shifts between the laser light and the emitted light, which are due to the parametric conversion of some of the pump energy into energy of phonons, reveal information about content and structure of the film. Spectroscopic examination of the emitted light and comparison to known shifts allow the determination of composition, degree of crystallinity, strain, alloy fluctuations, and other imperfections, such as damage due to ion implantation. This technique has been found most successful in detecting organic contaminants and in detecting stress in materials.

Electron Beam Analyzers

Auger Electron Spectroscopy. In Auger electron spectroscopy (AES), electrons of 1 keV to 25 keV are injected into the surface of a film, and the kinetic energies of electrons emitted from the surface are measured, to form a plot of $N(E)$ versus E. Here E is the electron energy and $N(E)$ is the number of electrons emitted per unit energy increment per second. The display of the derivative of this plot, $d[N(E)]/dE$, will generally have one or more outstanding features, such as nearly vertical sharp lines. These are due to the so-called Auger electrons that are characteristic of and reveal the presence of elemental species (identified by the value of energy at the position of the sharp line) lying within the first 50 Å of the surface (30,31). In addition, slight shifts in the positions from those expected for elemental atoms or changes in line shape indicate chemical binding information.

To determine concentrations of the various species, peak-to-peak heights of the features in the derivative spectrum are used, corrected for elemental sensitivity factors.

Several ways of measuring the energy of collected electrons can be used. A preferred instrument is the cylindrical mirror analyzer spectroscope (CMA). Its basic structure consists of segments of two coaxial cylinders, across which a variable dc voltage is applied, to select electrons of the desired energy that are to be collected (32).

Depth profiling can be carried out by alternating Auger analysis with removal of layers of material by sputter etching with an inert ion beam. Scanning AES has been carried out with electron beams focused into spots as small as 130 Å in diameter. All elements other than hydrogren and helium can be detected.

Electron Microprobe/Electron Probe Microanalysis. In electron microprobe analysis (EMP), electrons of energy 5 keV to 20 keV are injected into the surface, and identification of the elemental species is by measurement of the spectrum of emitted X-rays (33,34). In addition to a continum in this spectrum one finds one or more sharp X-ray "lines." The latter are characteristic of the elements emitting them. For example, the presence of copper is revealed by its K-alpha line of energy 8.048 keV, corresponding to a wavelength of 1.54 Å.

Two types of X-ray spectrometers are in use: (1) energy dispersive (EDS) and (2) wavelength dispersive (WDS). Energy analysis in EDS is by a cooled lithium-drifted silicon diode that generates electron–hole pairs (ehp's). These are converted into voltage pulses, which are stored in the "bins" of a multichannel analyzer. Since the number of ehp's generated per X-ray pulse is a function of its energy, the output of the multichannel analyzer is the desired spectrum.

In WDS one or more crystals diffract(s) the X-rays to angles determined by their wavelengths. Detection is by a Geiger-counter-type of gas-filled tube.

EDS is more rapid than WDS and is therefore used more frequently for quick surveys or spatial maps. However, it has poorer energy resolution (e.g., 150 eV versus 5 eV).

Spatial resolution of EMP is on the order of 1 μm to 10 μm. While therefore not used for detecting surface contaminants, it is well suited for locating impurities within a film and determining alloy composition.

Electron Energy Loss Spectroscopy. The transmitted electrons in scanning transmission electron microscopy (STEM), in which a fine electron beam is raster-scanned across a film, can be analyzed for energy loss by sensing the current collected for different retarding voltages applied to the electrode that collects the transmitted electrons. This technique, electron energy loss spectroscopy (EELS), has been used to locate precipitates containing elements that are not readily located by other techniques such as EMP (35,36).

Secondary Ion Mass Spectrometry/Sputtered Neutral Mass Spectrometry

The term *secondary* refers to particles emitted from a surface because this surface has been struck by other, or "primary," particles.

In contrast to most other methods of analysis in use, secondary ion mass spectrometry (SIMS) and secondary neutral mass spectrometry (SNMS) are destructive methods of analysis, for in these techniques ions of energy in the 1 keV to 20 keV range bombard and erode the surface of a film. Most of the products of this erosion are electrically neutral particles; a small fraction is ionized.

In SIMS these ions are sent through a mass spectrometer that reveals their charge-to-mass (Q/M) ratio and therefore identifies the composition of the emitted material. SNMS makes use of the neutral particles by ionizing them either with a strong laser beam or by sending them through a plasma chamber prior to directing them to the mass spectrometer. Progressive erosion and analysis provide information of relative density of the elements and isotopes versus depth of the film (37–43).

In an instrument called an *ion microprobe,* spatial analysis is carried out by focusing the bombarding beam to a small spot and scanning the sample, while simultaneously displaying on a screen a signal indicative of the spatial distribution of the relative density of a particular ion. To carry out the ion analysis, electrostatic quadrupole ion spectrometers, magnetic sector spectrometers, and time of flight tubes are in use. The quadrupole spectrometer comprises four parallel rods to which both a dc voltage V_{dc} and an ac voltage V_{rf} of a certain frequency are applied. Ions of a desired Q/M that will pass through the spectrometer are selected by adjustment of V_{dc}, V_{rf}, and the frequency.

In the *ion microscope,* a primary ion beam is used to flood a surface. The emitted secondary ions are passed through a "magnetic sector spectrometer." This unit preserves information of their spatial distribution while also permitting only ions of the Q/M selected by the intensity of an applied magnetic field to reach a so-called microchannel plate detector. The electric output signal of this distributed detector is used to generate a display on a fluorescent screen that is a map of the spatial distribution of secondary ions of the selected charge-to-mass ratio emitted by the surface.

To carry out depth profiling, in *static SIMS* a sputtering rate of about 1 Å/h will generally record a complete mass spectrum of the top 5 Å of a film. In *dynamic SIMS* a depth profile of the relative content of a particular ion is carried out at the rate of around 10 μm/h.

Lateral resolution of SIMS is generally around 100 μm, though it can be made as small as 1 μm. Depth resolution is on the order of 50 Å to 100 Å.

Rutherford Backscattering Spectrometry

Another technique for determining atomic composition and depth information is Rutherford backscattering spectrometry (RBS), also called high-energy (back)scattering ion spectrometry (HEIS). In RBS a film is bombarded with a beam of 1 MeV to 3 MeV He^+ or He^{++} ions, generally accelerated to this energy by a tandem accelerator, and an energy analysis is carried out on the small fraction that undergoes elastic collisions with atomic nuclei within the film and are returned with a velocity that has a component in direction opposite to the primary beam (37–44).

The energy of a backscattered ion can be measured by a silicon surface barrier detector, which generates voltage pulses of amplitude proportional to its energy. These pulses are fed to a multichannel analyzer, which stores them in different "bins," depending on their height. The number of pulses in a particular bin is an indication of the relative density of the particular species of atom near the surface of the film and permits calculation of the actual area density.

For atoms on the surface of the film, the ratio of the energy of a backscattered ion to the energy it had when incident on the film at a specified angle depends only on the ratio M_1/M_2. Here M_1 is the mass of the bombarding ion, and M_2 is that of the nucleus of the atom in the film that caused it to be backscattered. Ions backscattered by atoms below the surface generally lose some energy during their passage to the surface. When these ions leave the surface, the curve of their yield versus energy is a series of quasi-rectangles, whose positions and widths provide information of the depth below the surface at which the detected atoms that backscattered the incident ions are located. It has been found that for film thicknesses less than 2000 Å the typical depth resolution is around 100 Å to 200 Å.

Applications of RBS are measurements of film thickness, uniformity, stoichiometry, and distribution of impurities. RBS has been found particularly suitable for detecting heavy elements in an environment of light elements. RBS can also be useful in determining crystallinity, through the phenomenon of *channeling.* For when a crystal is aligned so that an incident ion passes through it without colliding, the backscattering yield can decrease by as much as two orders of magnitude.

X-Ray Fluorescence

X-ray fluorescence (XRF), also known as X-ray fluorescence spectroscopy (XRFS), X-ray fluorescence analysis (XRFA), and X-ray secondary emission spectroscopy, is a nondestructive technique that can rapidly give average sample composition, including impurities. It can also be used for measuring film thickness (as thin as 100 Å) by comparing the intensity of X-ray fluorescence with that from a known standard (45,46).

In XRF a primary X-ray source illuminates the specimen, causing secondary X rays of lower energy to be emitted. As in EMP, the energy of the emitted X rays can be measured by either EDS or WDS systems. The energies identify the emitting substances; the relative intensities of emitted X-ray lines provide the information for calculating the concentrations of the emitting species. XRF can analyze areas as small as 10^{-6} cm^2.

X-Ray Photoelectron Spectroscopy

X-ray photoelectron spectroscopy (XPS), also known as electron spectroscopy for chemical analysis (ESCA), is a method for identifying chemical (not just elemental) species within a film (47–49). It is based on the well-known photoelectric effect, except that the incident radiation used is in the X ray, not the visible range of the electromagnetic spectrum.

In XPS a source of 1 keV to 2 keV X rays of narrow line width, obtained by using light elements (Al, Mg) as targets in the X-ray tube, illuminates a film, causing electrons to be ejected. Since the kinetic energy of an ejected electron depends on the energy that bound the electron to the atom whence it originated, measurement of this kinetic energy reveals the type of atom or compound of origin. The resulting curve of collected electron current versus energy provides the information of the density of the various chemical species present.

Since the "escape depth" of electrons is around 50 Å, XPS is a surface analysis technique. By combining XPS with sputter erosion of a surface, a film may be depth profiled.

An advantage of XPS over methods based on the use of electron beams is that there is less danger of charging a surface with X rays, so that XPS is often a preferred choice for analysis of insulators.

X-Ray Topography

In X-ray topography (XRT) X rays are used to detect faults in the structure of single-crystal wafers or films. Basically, the film is placed at an angle with respect to the X-ray beam, to form a diffraction pattern on a photographic plate. Both film and plate are now moved perpendicular to the direction of the X-ray beam. A change in the diffraction pattern indicates a change in the crystal structure (50,51).

Neutron Activation

Neutron activation analysis is a method that can be used to find certain trace elements in a film. The method consists of placing the film into a nuclear reactor for several hours, to allow the nuclei of the trace elements to reach an excited state. After removal from the reactor, these excited nuclei "relax" by giving off gamma rays. Measurement of the energy and intensity of these with a germanium detector provides information on the presence and density of the atoms of these trace elements.

For this method of analysis to be successful, it is necessary that the host elements of the film have a half-life that is much shorter than that of the trace elements (52). An example of the use of the neutron activation technique is the detection and measurement of gold in silicon.

MACROSCOPIC FILMS

Although the term *thin film* generally refers to films whose thickness is a few microns or less, it has also been used to describe films of thickness on the order of 1 mm. Such films can be analyzed by the use of some of the techniques discussed in the previous sections, as well as by common microscopic and macroscopic techniques. Very rapid measurement of the height of a macroscopic film of liquid or solid, on the other hand, may require some special techniques, such as those discussed next.

Ultrasonic Systems

The methods of using mechanical waves of ultrasonic frequencies to measure the thickness of thin sheets or thick films are based on either "resonance" or "time of flight" measurements (53).

Techniques based on resonance utilize the fact that when an acoustic (ultrasonic) transducer is coupled to the sheet whose thickness is to be measured and the electrical input signal supplied to the transducer is swept through a frequency range, the electrical input impedance "seen" by the transducer exhibits resonance effects. If, for example, frequencies f_n and f_{n+1} are two adjacent resonances of the same type, the thickness of the sheet is found to be $c/2\Delta f$, where c is the velocity of the ultrasonic wave in the material and Δf is the difference in frequency of the two adjacent resonances.

In time-of-flight-based instruments an ultrasonic transducer is mounted to send ultrasonic pulses through the film. If the velocity of propagation in the film is known, a measurement of the time interval between echo pulses received from the discontinuities of ultrasonic characteristic impedance at the bottom of the film and at the top of the film reveals the film thickness.

Microwave Microstrip Method

A noncontacting system for measuring the height of a film on a metal plate is based on the fact that v, the velocity of propagation of microwaves along a transmission line, depends on the dielectric constant of the medium in the line. The effective dielectric constant of a modified microstrip transmission line that has the metal plate as one conductor and a metal strip some distance above the film as the second conductor depends on the height of the film. Consequently, the determination of v by measurement of the wavelength at a fixed frequency or of the resonant frequency of a section of this line, after calibration with a "standard," provides information on the film height. In a refinement that eliminates the need for an external source of microwave energy, a section of this modified microstrip line is used to control the frequency of a transistor oscillator, thereby creating a compact, inexpensive instrument (54).

Fluorescent Dye System

To measure the instantaneous local height of a film of water, a fluorescent dye is mixed into the water. The dye is excited with a light source, causing it to fluoresce. The height of the film is determined by measuring the relative amount of light emitted from the film (55).

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Reading List

To prepare this article, extensive use was made of the material of Chapters 9 and 10 of the text by D. K. Schroder entitled *Semiconductor Material and Device Characterization,* New York: Wiley, 1990. The writer gratefully acknowledges the support and initial review of the manuscript by Dr. Schroder.

In addition to this text, as well as texts dealing with various specialized topics as listed in the Bibliography, material dealing with the analysis of thin films will be found in the following:

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