

ENCYCLOPEDIA OF MATERIALS CHARACTERIZATION

C. Richard Brundle

Charles A. Evans, Jr.

Shaun Wilson

A complex network diagram consisting of numerous green dots scattered across the upper left and middle sections, and a dense, intricate web of blue lines that dominates the right side and extends into the lower right. The background is white, and the entire diagram is set against a dark blue vertical bar on the left side of the cover.

MATERIALS CHARACTERIZATION
SERIES

SURFACES, INTERFACES, THIN FILMS

This book was acquired, developed, and produced by Manning Publications Co.

Copyright © 1992 by Butterworth-Heinemann, a division of Reed Publishing (USA) Inc.

All rights reserved.

No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by means, electronic, mechanical, photocopying, or otherwise, without prior written permission of the publisher.

Recognizing the importance of preserving what has been written, it is the policy of Butterworth-Heinemann and of Manning to have the books they publish printed on acid-free paper, and we exert our best efforts to that end.

Library of Congress Cataloging-in-Publication Data

Brundle, C. R.

Encyclopedia of materials characterization: surfaces, interfaces, thin films/C. Richard Brundle, Charles A. Evans, Jr., Shaun Wilson.

p. cm.—(Materials characterization series)

Includes bibliographical references and index.

ISBN 0-7506-9168-9

1. Surfaces (Technology)—Testing. I. Evans, Charles A. II. Wilson, Shaun. III. Title. IV. Series.

TA418.7.B73 1992

92-14999

620'.44--dc20

CIP

Butterworth-Heinemann

80 Montvale Avenue

Stoneham, MA 02180

Manning Publications Co.

3 Lewis Street

Greenwich, CT 06830

1 0 9 8 7 6 5 4 3 2 1

Printed in the United States of America

10.1 Dynamic SIMS

Dynamic Secondary Ion Mass Spectrometry

PAUL K. CHU

Contents

- Introduction
- Basic Principles
- Common Modes of Analysis and Examples
- Sample Requirements
- Artifacts
- Quantification
- Instrumentation
- Conclusions

Introduction

Dynamic SIMS, normally referred to as SIMS, is one of the most sensitive analytical techniques, with elemental detection limits in the ppm to sub-ppb range, depth resolution (z) as good as 2 nm and lateral (x, y) resolution between 50 nm and 2 μm , depending upon the application and mode of operation. SIMS can be used to measure any elemental impurity, from hydrogen to uranium and any isotope of any element. The detection limit of most impurities is typically between 10^{12} and 10^{16} atoms/ cm^3 , which is at least several orders of magnitude lower (better) than the detection limits of other analytical techniques capable of providing similar lateral and depth information. Therefore, SIMS (or the related technique, SALI) is almost always the analytical technique of choice when ultrahigh sensitivity with simultaneous depth or lateral information is required. Additionally, its ability to detect hydrogen is unique and not possible using most other non-mass spectrometry surface-sensitive analytical techniques.

Dynamic SIMS is used to measure elemental impurities in a wide variety of materials, but is almost new used to provide chemical bonding and molecular information because of the destructive nature of the technique. Molecular identification or measurement of the chemical bonds present in the sample is better performed using analytical techniques, such as X-Ray Photoelectron Spectrometry (XPS), Infrared (IR) Spectroscopy, or Static SIMS.

The accuracy of SIMS quantification ranges from $\pm 2\%$ in optimal cases to a factor of 2, depending upon the application and availability of good standards. However, it is generally not used for the measurement of major components, such as silicon and tungsten in tungsten silicide thin films, or aluminum and oxygen in alumina, where other analytical techniques, such as wet chemistry, X-Ray Fluorescence (XRF), Electron Probe (EPMA), or Rutherford Backscattering Spectrometry (RBS), to name only a few, may provide much better quantitative accuracy ($\pm 1\%$ or better).

Because of its unique ability to measure the depth or lateral distributions of impurities or dopants at trace levels, SIMS is used in a great number of applications areas. In semiconductor applications, it is used to quantitatively measure the depth distributions of unwanted impurities or intentional dopants in single or multilayered structures. In metallurgical applications, it is used to measure surface contamination, impurities in grain boundaries, ultratrace level impurities in metal grains, and changes in composition caused by ion implantation for surface hardening. In polymers or other organic materials, SIMS is used to measure trace impurities on the surface or in the bulk of the material. In geological applications, SIMS is used to identify mineral phases, and to measure trace level impurities at grain boundaries and within individual phases. Isotope ratios and diffusion studies are used to date geological materials in cosmogeochemical and geochronological applications. In biology and pharmacology, SIMS is used to measure trace elements in localized areas, by taking advantage of its excellent lateral resolution, and in very small volumes, taking advantage of its extremely low detection limits.

Basic Principles

Sputtering

When heavy primary ions (oxygen or heavier) having energies between 1 and 20 keV impact a solid surface (the sample), energy is transferred to atoms in the surface through direct or indirect collisions. This creates a mixing zone consisting of primary ions and displaced atoms from the sample. The energy and momentum transfer process results in the ejection of neutral and charged particles (atomic ions and ionized clusters of atoms, called molecular ions) from the surface in a process called sputtering (Figure 1).

The depth (thickness) of the mixing zone, which limits the depth resolution of a SIMS analysis typically to 2–30 nm, is a function of the energy, angle of incidence,

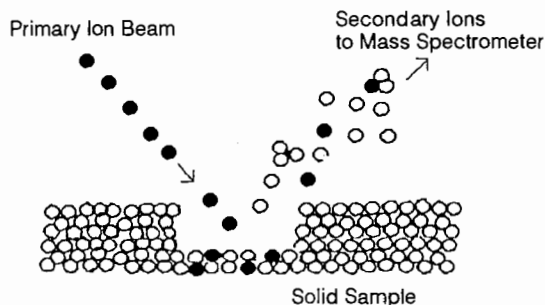


Figure 1 Diagram of the SIMS sputtering process.

and mass of the primary ions, as well as the sample material. Use of a higher mass primary ion beam, or a decrease in the primary ion energy or in the incoming angle with respect to the surface, will usually cause a decrease in the depth of the mixing zone and result in better depth resolution. Likewise, there is generally an inverse relationship between the depth (thickness) of the mixing zone and the average atomic number of the sample.

During a SIMS analysis, the primary ion beam continuously sputters the sample, advancing the mixing zone down and creating a sputtered crater. The rate at which the mixing zone is advanced is called the sputtering rate. The sputtering rate is usually increased by increasing the primary ion beam current density, using a higher atomic number primary ion or higher beam energy, or by decreasing the angle at which the primary ion beam impacts the surface. The primary ion beam currents used in typical SIMS analyses range from 10 nA to 15 μ A—a range of more than three decades.

The depth resolution of a SIMS analysis is also affected by the flatness of the sputtered crater bottom over the analytical area; a nonuniform crater bottom will result in a loss in depth resolution. Because most ion beams have a Gaussian spatial distribution, flat-bottomed craters are best formed by rastering the ion beam over an extended area encompassing some multiples of beam diameters. Moreover, to reject stray ions emanating from the crater walls (other depths), secondary ions are collected only from the central, flat-bottomed region of the crater through the use of electronic gating or physical apertures in the mass spectrometer. For example, secondary ions are often collected from an area as small as 30 μ m in diameter, while the primary ion beam sputters an area as large as 500 \times 500 μ m. Unfortunately, no matter what precautions and care are taken, the bottom of a sputtered crater becomes increasingly rough as the crater deepens, causing a continual degradation of depth resolution.

Detection Limits

The detection limit of each element depends upon the electron affinity or ionization potential of the element itself, the chemical nature of the sample in which it is contained, and the type and intensity of the primary ion beam used in the sputtering process.

Because SIMS can measure only ions created in the sputtering process and not neutral atoms or clusters, the detection limit of a particular element is affected by how efficiently it ionizes. The ionization efficiency of an element is referred to as its ion yield. The ion yield of a particular element A is simply the ratio of the number of A ions to the total number of A atoms sputtered from the mixing zone. For example, if element A has a 1:100 probability of being ionized in the sputtering process—that is, if 1 ion is formed from every 100 atoms of A sputtered from the sample—the ion yield of A would be $1/100$. The higher the ion yield for a given element, the lower (better) the detection limit.

Many factors affect the ion yield of an element or molecule. The most obvious is its intrinsic tendency to be ionized, that is, its ionization potential (in the case of positive ions) or electron affinity (in the case of negative ions). Boron, which has an ionization potential of 8.3 eV, loses an electron much more easily than does oxygen, which has an ionization potential of 13.6 eV, and therefore has a higher positive ion yield. Conversely, oxygen possesses a higher electron affinity than boron (1.5 versus 0.3 eV) and therefore more easily gains an electron to form a negative ion. Figures 2a and 2b are semilogarithmic plots of observed elemental ion yields relative to the ion yield of iron (M^+/Fe^+ or M^-/Fe^-) versus ionization potential or electron affinity for some of the elements certified in an NBS 661 stainless steel reference material. From these plots, it is easy to see that an element like zirconium has a very high positive ion yield and, therefore, an excellent detection limit, compared to sulfur, which has a poor positive ion yield and a correspondingly poor detection limit. Likewise, selenium has an excellent negative ion yield and an excellent detection limit, while manganese has a poor negative ion yield and poor detection limit. The correlation of electron affinity and ionization potential with detection limits is consistent in most cases; exceptions due to the nature of the element itself or to the chemical nature of the sample material exist. For example, fluorine exhibits an anomalously high positive ion yield in almost any sample type.

One of three kinds of primary ion beams is typically used in dynamic SIMS analyses: oxygen (O_2^+ or O^-), cesium (Cs^+), or argon (Ar^+). The use of an oxygen beam can increase the ion yield of positive ions, while the use of a cesium beam can increase the ion yield of negative ions, by as much as four orders of magnitude. A simple model explains these phenomena qualitatively by postulating that $M-O$ bonds are formed in an oxygen-rich mixing zone, created by oxygen ion bombardment. When these bonds break in the ion emission process, oxygen tends to become negatively charged due to its high ionization potential, and its counterpart

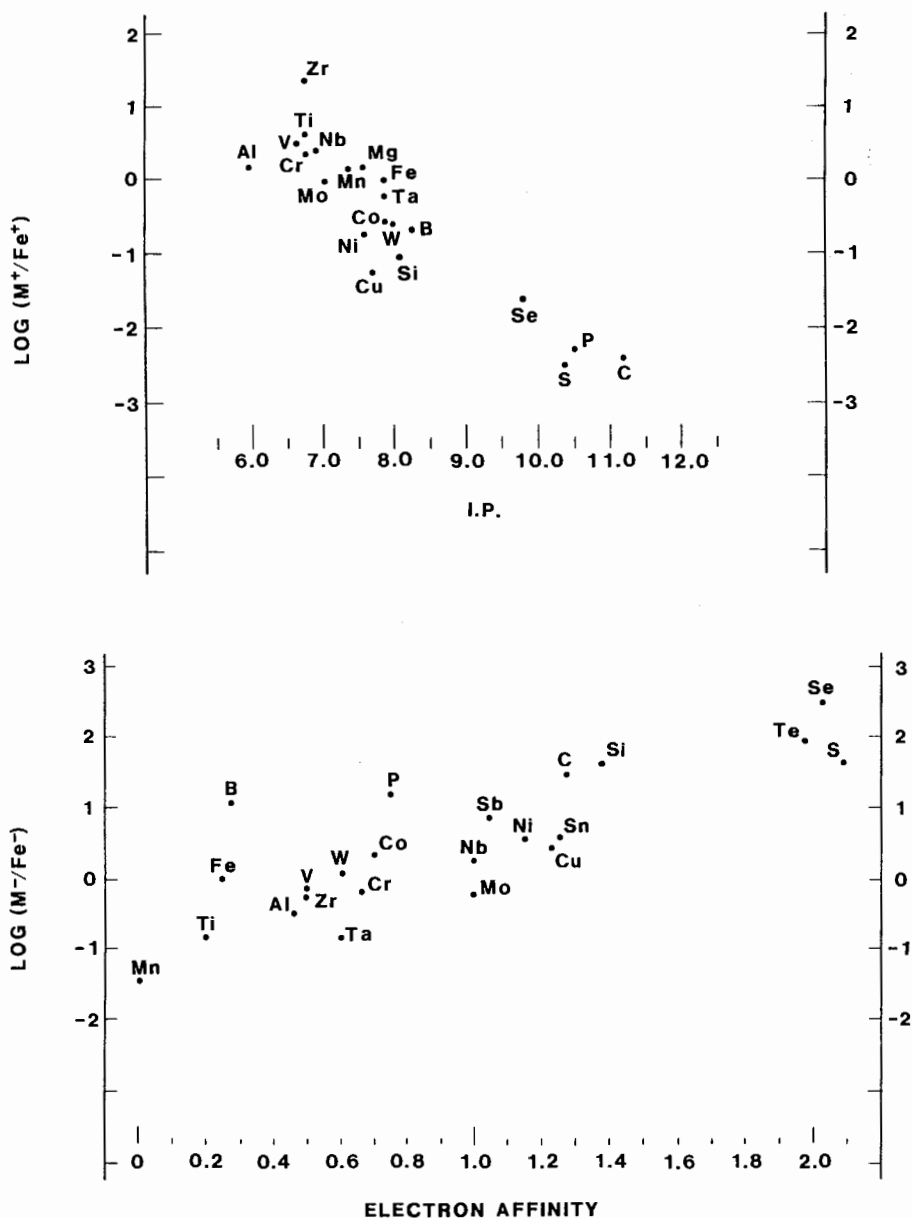


Figure 2 (a) Semilogarithmic plot of the positive relative ion yields of various certified elements (M^+ / Fe^+) in NBS 661 stainless steel reference material versus ionization potential. (b) Semilogarithmic plot of the negative relative ion yields of various certified elements (M^- / Fe^-) in NBS 661 stainless steel reference material versus electron affinity.

M dissociates as a positive ion.¹ Conversely, the enhanced ion yields of the cesium ion beam can be explained using a work function model,² which postulates that because the work function of a cesiated surface is drastically reduced, there are more secondary electrons excited over the surface potential barrier to result in enhanced formation of negative ions. The use of an argon primary beam does not enhance the ion yields of either positive or negative ions, and is therefore, much less frequently used in SIMS analyses.

Like the chemical composition of the primary beam, the chemical nature of the sample affects the ion yield of elements contained within it. For example, the presence of a large amount of an electronegative element like oxygen in a sample enhances the positive secondary ion yields of impurities contained in it compared to a similar sample containing less oxygen.

Another factor affecting detection limits is the sputtering rate employed during the analysis. As a general rule, a higher sputtering rate yields a lower (better) detection limit because more ions are measured per unit time, improving the detection limits on a statistical basis alone. However, in circumstances when the detection limit of an element is limited by the presence of a spectral interference (see below), the detection limit may not get better with increased sputtering rate. Additionally and unfortunately, an increase in the sputtering rate nearly always results in some loss in depth resolution.

Common Modes of Analysis and Examples

SIMS can be operated in any of four basic modes to yield a wide variety of information:

- 1 The depth profiling mode, by far the most common, is used to measure the concentrations of specific preselected elements as a function of depth (z) from the surface.
- 2 The bulk analysis mode is used to achieve maximum sensitivity to trace-level components, while sacrificing both depth (z) and lateral (x and y) resolution.
- 3 The mass scan mode is used to survey the entire mass spectrum within a certain volume of the specimen.
- 4 The imaging mode is used to determine the lateral distribution (x and y) of specific preselected elements. In certain circumstances, an imaging depth profile is acquired, combining the use of both depth profiling and imaging.

Depth Profiling Mode

If the primary ion beam is used to continuously remove material from the surface of a specimen in a given area, the analytical zone is advanced into the sample as a function of the sputtering time. By monitoring the secondary ion count rates of selected

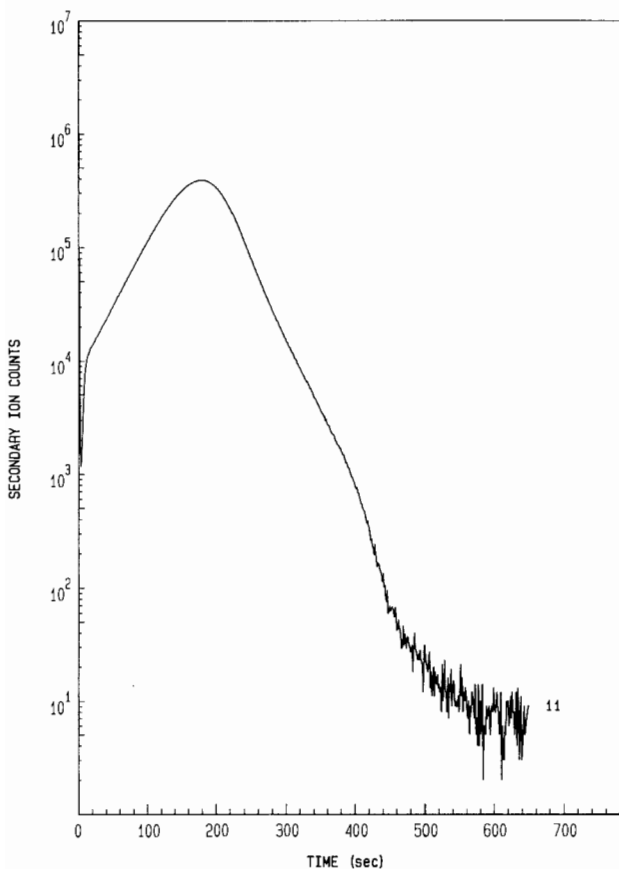


Figure 3a Unprocessed depth profile (secondary ion intensity versus sputtering time) of a silicon sample containing a boron ion implant.

elements as a function of time, a profile of the in-depth distribution of the elements is obtained. The depth scale of the profile is commonly determined by physically measuring the depth of the crater formed in the sputtering process and assigning that depth to the total sputtering time required to complete the depth profile. A depth scale assigned in this way will be accurate only if the sputtering rate is uniform throughout the entire profile. For samples composed of layers that sputter at different rates, an accurate depth scale can be assigned only if the relative sputtering rates of the different layers are known. A typical SIMS depth profile is collected as secondary ion counts per second versus sputtering time (typically one second per measurement) and converted to a plot of concentration versus depth by using the depth of the sputtering crater and comparing the data to standards. Figure 3a is an unprocessed depth profile of a silicon sample containing a boron ion implant.

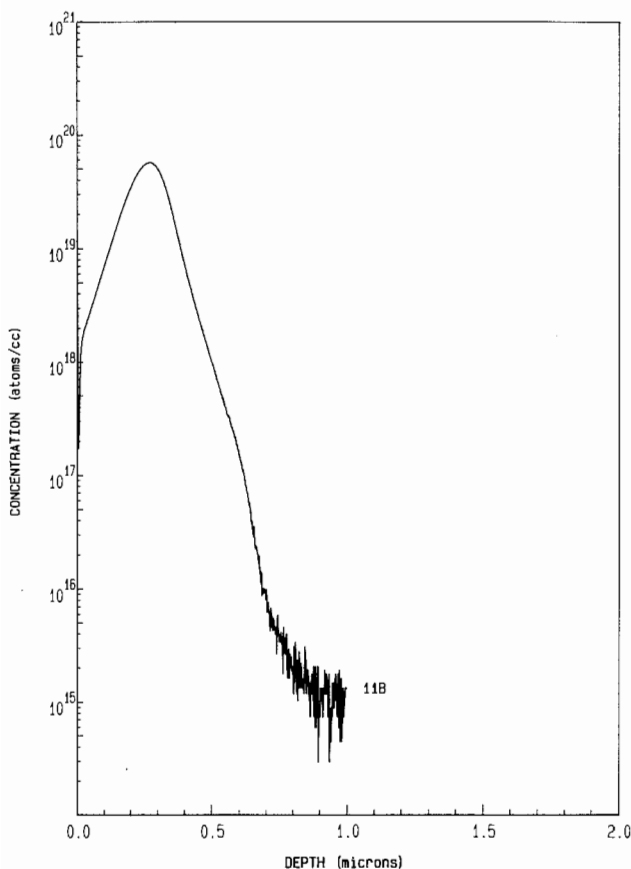


Figure 3b Depth profile in (a), after converting the sputtering time to depth and the secondary ion intensities to concentrations.

Figure 3b shows the same depth profile after converting to depth and concentration. Depth profiles can be performed to depths exceeding 100 μm and can take many hours to acquire; a more typical depth profile is several μm in depth and requires less than one hour to acquire.

Mass Scan Mode

A mass scan is acquired in cases when a survey of all impurities present in a volume of material is needed. Rather than measuring the secondary ion count rates of pre-selected elements as a function of sputtering time the count rates of all secondary ions are measured as a function of mass. Because a mass scan is continuously acquired over a mass range, no depth profiling or lateral information is available while operating in this mode. Figure 4 shows a mass scan acquired from a zirconia

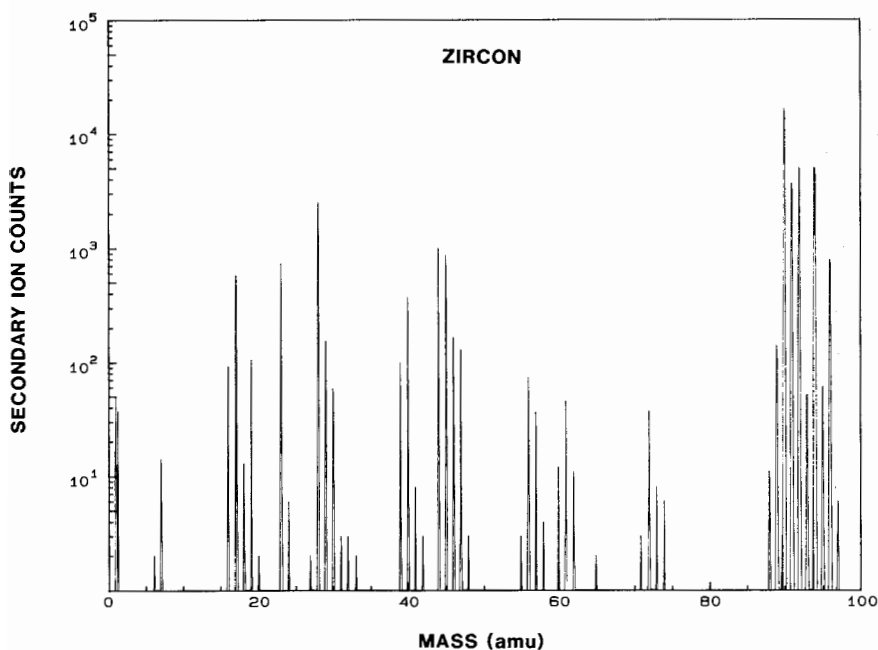


Figure 4 Mass scan acquired from a zirconia crystal.

crystal (geological sample). It shows peaks for many elements and molecules, but provides no information concerning the depth or lateral distribution of these impurities.

Bulk Analysis Mode

Bulk analysis mode is typically used to obtain the lowest possible detection limits of one or several elements in a uniform sample. This mode of operation is similar to a depth profile with the sputtering rate set to the maximum. This causes the crater bottom to lose its flatness and allows impurities from the crater walls to be measured, thereby sacrificing depth resolution. Therefore, accurate measurement of impurities is obtained only when they are uniformly distributed in the sample. This method of measurement usually results in at least a factor-of-10 improvement in detection limits over the depth profiling mode. As an example, the detection limit of boron in silicon using the bulk analysis mode is 5×10^{12} atoms/cm³, several orders of magnitude better than the boron background acquired using the depth profiling mode (6×10^{14} atoms/cm³), as shown in Figure 3b.

Imaging Mode

SIMS imaging is performed using one of two methods. The first, called ion microscopy or stigmatic imaging, is only possible using specially constructed mass spectrometers capable of maintaining the x - y spatial relationships of the secondary ions. These mass spectrometers are typically specially configured double-focusing magnetic-sector spectrometers and are actually better termed secondary ion *microscopes*. The lateral resolution of microscope imaging is typically no better than 1 μm . The second method, scanning imaging, is performed by measuring the secondary ion intensity as a function of the lateral position of a small spot scanning ion beam. The lateral resolution of this type of imaging is largely dependent on the diameter of the primary ion beam, which can be as small as 50 nm.

Figures 5a and 5b are mass-resolved secondary ion images of gold (Au) and sulfur (S) in a cross-sectioned and polished pyrite (gold ore) sample acquired using the microscope imaging method. The gray level is proportional to the secondary ion intensity measured at each location, i.e., more gold or sulfur is found in darker locations. These images show that the gold, the geologist's primary interest, is localized in the outer few μm of the sulfur-containing pyrite grain.

By acquiring mass-resolved images as a function of sputtering time, an imaging depth profile is obtained. This combined mode of operation provides simultaneous lateral and depth resolution to provide what is known as three-dimensional analysis.

Sample Requirements

Most SIMS instruments are configured to handle samples less than 2.5 cm in diameter and 1 cm in thickness. The surface of the sample must be as smooth as possible because surface roughness causes a significant loss in depth resolution; cross sections and other cut samples must be well polished before analysis. In SIMS instruments capable of stigmatic imaging, the sample should be planar, because it effectively is part of the secondary ion optics. Nonplanar samples are better analyzed using a quadrupole SIMS instrument (discussed below) in which the sample shape does not affect the results as strongly. Samples composed of materials that are dielectric (nonconducting) must be analyzed using special conditions (see below). Quadrupole SIMS instruments are also less affected by sample charging and are often used to analyze dielectric samples.

Artifacts

Although SIMS is one of the most powerful surface analysis techniques, its application is complicated by a variety of artifacts.

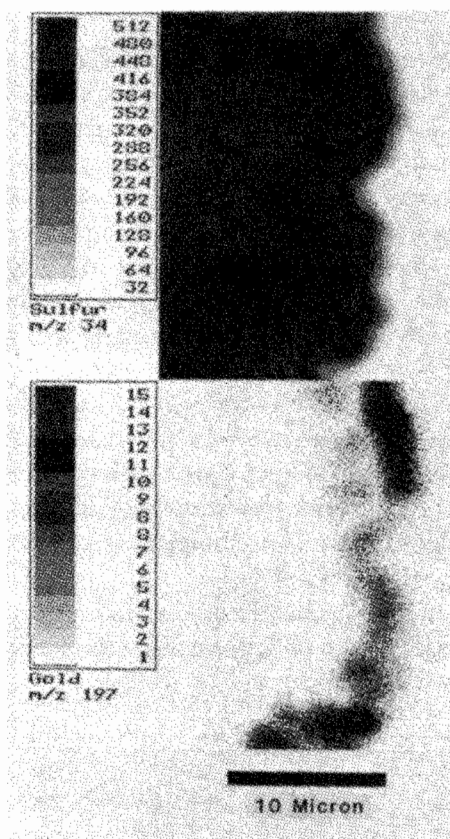


Figure 5 Mass-resolved secondary ion images of sulfur and gold in a pyrite ore sample. A comparison of the two images clearly shows the gold is found in the outer several μm of the sulfur-containing pyrite grain. These images were acquired using a magnetic-sector mass spectrometer in the microscope-imaging mode.

Mass Interferences

The most frequent artifacts arise from interferences in the mass spectrum, that is, ionized atomic clusters (molecular ions) or multiply charged ions whose nominal mass-to-charge ratio equals that of the elemental ions of interest. Such interferences can cause erroneous assignment of an element not present in the sample or simply can degrade the detection limit of the element of interest. Figure 6 is a mass spectrum obtained from high-purity silicon, using oxygen ion bombardment. In addition to the $^{28}\text{Si}^+$, $^{29}\text{Si}^+$, and $^{30}\text{Si}^+$ isotope peaks, there exist numerous other peaks of atomic and molecular ions typically composed of primary ion species (oxygen), ions

THE MASS SPECTRUM

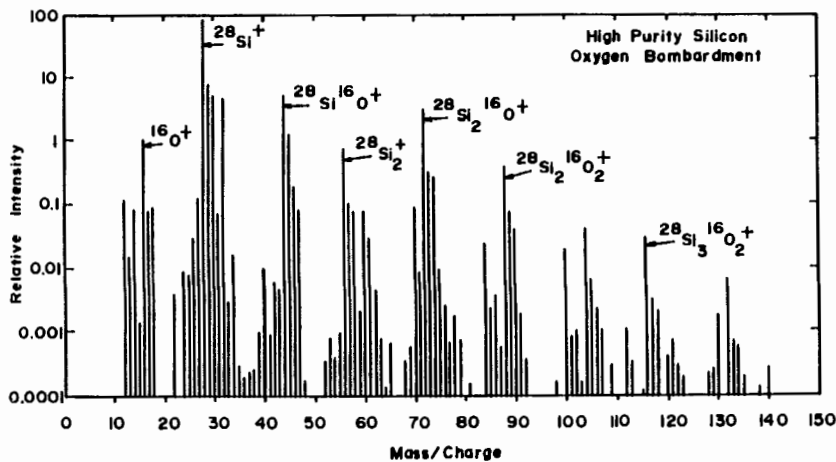


Figure 6 Typical secondary ion mass spectrum obtained from high-purity silicon using an oxygen ion beam. Major ion peaks are identified in the spectrum.

of the major components of the sample (silicon), or atmospheric species (hydrogen, carbon, oxygen, nitrogen, etc.) remaining in the high-vacuum sample chamber. Many of these peaks are sufficiently intense to produce a measurable background, which may preclude determination of a specific element (impurity), even in the ppm range.

Once identified, voltage offset and high mass resolution techniques may be used to reduce the detrimental effect of these interfering ions. In the voltage offset technique, the mass spectrometer is adjusted to accept only ions in a certain (usually higher) kinetic energy range. This technique is effective in discriminating against molecular ions because the energy distribution of atomic ions (typically the ions of interest) is broader than that of molecular ions at the same nominal mass. Figure 7 shows two SIMS depth profiles of the same silicon sample implanted with arsenic (^{75}As). These depth profiles were obtained under normal conditions (0-V offset) and under voltage offset conditions (50-V offset). The improvement in the detection limit of arsenic with the use of a 50-V offset results from discrimination of the $^{29}\text{Si}^{30}\text{Si}^{16}\text{O}$ molecular ion also at mass 75.

High mass resolution techniques are used to separate peaks at the same nominal mass by the very small mass differences between them. As an example, a combination of ^{30}Si and ^1H to form the molecular ion $^{30}\text{Si}^1\text{H}^-$, severely degrades the detection limit of phosphorous (^{31}P) in a silicon sample. The exact mass of phosphorous (^{31}P) is 31.9738 amu while the real masses of the interfering $^{30}\text{Si}^1\text{H}$ and $^{29}\text{Si}^1\text{H}_2$ molecules are 31.9816 amu and 31.9921 amu, respectively. Figure 8 shows a mass

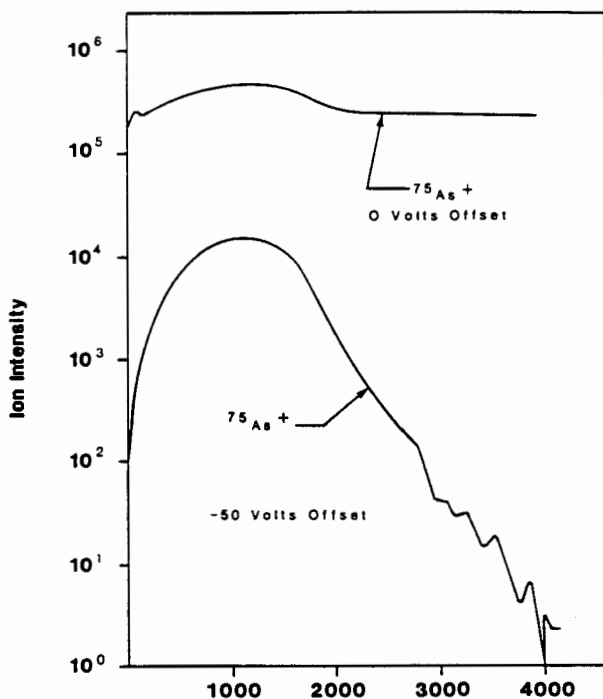


Figure 7 Depth profile of an arsenic (^{75}As) ion implant in silicon with and without use of voltage offset techniques. Voltage offset provides an enhanced detection limit for As in Si.

spectrum obtained from a phosphorus doped amorphous silicon thin film using high mass resolution techniques. The two mass interferences, $^{30}\text{Si}^1\text{H}^-$ and $^{29}\text{Si}^1\text{H}_2^-$, are completely separated from the $^{31}\text{P}^-$ peak. Quadrupole instruments are not usually capable of such high mass resolution.

Primary Ion Beam Sputtering Equilibrium

As explained above, the mixing zone contains a mixture of atoms from the primary ion beam and the solid sample. In the case of oxygen or cesium ion bombardment, these primary species become part of the material in the mixing zone and can significantly alter the ion yields of elements in the sample. However, when sputtering is first started (at the beginning of a depth profile), the mixing zone contains very few atoms from the primary ion beam, causing ions ejected from the mixing zone to be less affected by the enhancement process.

Crater Bottom Roughening

In polycrystalline solids or samples consisting of various phases, each grain may sputter at a different rate producing extensive roughness in the bottom of the cra-

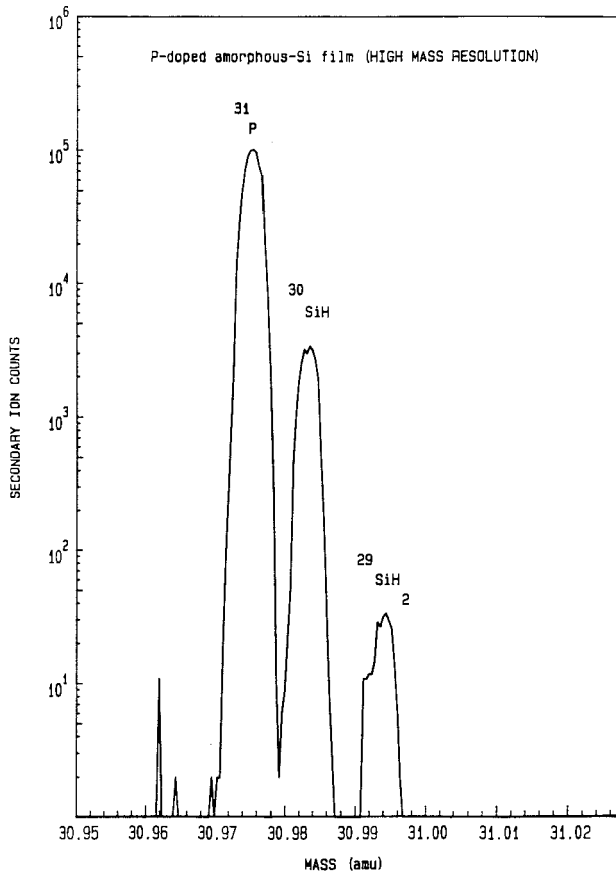


Figure 8 High mass resolution mass spectrum obtained from a phosphorus-doped amorphous silicon hydride thin film using a magnetic sector ion microanalyzer. The ^{31}P peak is well separated from the hydride interferences.

ter.³ The roughness of the crater bottom will result in a loss in depth resolution and cause the depth profile to appear smeared in depth.

Surface Oxide

By enhancing the positive ion yield of most elements, the presence of an oxide on the surface of a sample can cause the first several points of a SIMS depth profile to be misleadingly high. Exceptionally large secondary ion signals for most elements are observed while profiling through the surface oxide, even though their concentrations are not higher. In these cases, the first several data points are corrected or simply disregarded.

Sample Charging

The charge carried by positive primary ions can accumulate on the surface of a non-conducting sample, causing the primary ion beam to be defocused or to move away from the analytical area of interest, thus preventing continued analysis. In addition, the accumulated charge can change the energy of the ejected ions, thereby affecting their transmission and detection in the mass spectrometer. This effect, called *sample charging*, is eliminated or reduced by flooding the sample surface with a low energy electron beam, providing compensation for the build-up of positive charge. As a general rule, samples with resistivities above 10^8 ohm/cm² require the use of electron flooding. In highly insulating samples, the use of a negative primary ion beam may also alleviate this charging problem.

Adsorption of Gaseous Species

During the sputtering process, residual atoms and molecules in the vacuum above the sample surface (typically containing hydrogen, carbon, nitrogen, and oxygen) are incorporated into the mixing zone by absorbing onto newly exposed and unsputtered reactive ions and molecules of the sample. The incorporated atmospheric species are eventually ejected from the mixing zone as elemental and molecular ions and detected as if they were originally present in the sample, complicating SIMS detection of these species and adding interfering molecular ions to the secondary ion mass spectrum. As an example, a mass interference between ³¹P, and ³⁰Si¹H and ²⁹SiH₂, all having mass 31, can be caused by hydrogen from the atmosphere in the sample chamber. The detrimental effects of these atmospheric species can be reduced by improving the vacuum in the sample chamber, but no matter how good the vacuum is, some adsorption will occur.

Impurity Mobility—Ion Beam-Induced Diffusion

Another difficulty is ion beam-induced diffusion of extremely mobile ions, such as lithium and sodium, in dielectric thin-film samples. This effect is normally observed when depth profiling a dielectric thin film on a conducting substrate with a positive primary ion beam. Diffusion occurs because the primary ion beam deposits a charge on the sample surface, creating a large electric field across the thin film, thereby driving the mobile ions away from the surface, to the interface between the thin film and substrate. In bulk insulators, this problem may be less severe because the electric field gradient is smaller. Nonetheless, the acquired depth profile no longer reflects the original composition of the sample. This effect is reduced or eliminated by flooding the sample surface with a low energy beam of electrons during sputtering. The current of electrons striking the sample surface must be carefully balanced against the build-up of charge due to the primary ion beam. Otherwise, distortion of the depth profile will still occur. As a general rule, quadrupole mass spectrometers have much less difficulty with impurity mobility artifacts

than do magnetic sector spectrometers, and they are almost always used in these applications.

Quantification

Ion yields of different elements vary by several orders of magnitude and depend sensitively on the type of primary beam and sample. Accurate quantification requires comparison to standards or reference materials of similar or identical major element composition that must be measured using the same analytical conditions, especially using the same type of primary ion beam. For example, an aluminum sample with a known content of copper is not a good standard to use for quantification of copper in stainless steel. Similarly, a standard analyzed using a cesium primary ion beam must not be used as a standard for quantification of an unknown sample analyzed using an oxygen ion beam. In some cases, semiempirical ion yield systematics are successfully used to quantify certain analyses; this method of quantification is accurate only to within an order of magnitude.

Ion implantation is often used to produce reliable standards for quantification of SIMS analyses.⁴ Ion implantation allows the introduction of a known amount of an element into a solid sample. A sample with a major component composition similar to that of the unknown sample may be implanted to produce an accurate standard. The accuracy of quantification using this implantation method can be as good as $\pm 2\%$.

Instrumentation

SIMS instruments are generally distinguished by their primary ion beams, and the kinds of spectrometers they use to measure the secondary ions. Several types of primary ion beams—typically, oxygen, cesium, argon, or a liquid metal like gallium—are used in SIMS analyses, depending on the application. Nearly any SIMS instrument can be configured with one or more of these ion-beam types. The majority of SIMS mass spectrometers fall into three basic categories: double-focusing electrostatic or magnetic sector, quadrupole, and time-of-flight. Time-of-flight analyzers are primarily used for surface and organic analyses (especially for high molecular weight species) and are mentioned in the article on static SIMS.

A double-focusing, electrostatic or magnetic-sector mass spectrometer achieves mass separation using an electrostatic analyzer and magnet. Secondary ions of different mass are physically separated in the magnetic field, with light elements making a tight arc through the magnet and heavy elements making a broad arc. Ions of different charge-to-mass ratios are measured by changing the strength of the magnetic field in the magnet to align the ions of interest with a stationary detector. Magnetic-sector systems provide excellent detection limits because of their high transmission efficiency, and are capable of high mass resolution. Some of these

spectrometers are capable of stigmatic imaging (also called ion microscopy) which is used to acquire mass-resolved ion images with a resolution as good as 1 μm .

In quadrupole-based SIMS instruments, mass separation is achieved by passing the secondary ions down a path surrounded by four rods excited with various AC and DC voltages. Different sets of AC and DC conditions are used to direct the flight path of the selected secondary ions into the detector. The primary advantage of this kind of spectrometer is the high speed at which they can switch from peak to peak and their ability to perform analysis of dielectric thin films and bulk insulators. The ability of the quadrupole to switch rapidly between mass peaks enables acquisition of depth profiles with more data points per depth, which improves depth resolution. Additionally, most quadrupole-based SIMS instruments are equipped with enhanced vacuum systems, reducing the detrimental contribution of residual atmospheric species to the mass spectrum.

The choice of mass spectrometer for a particular analysis depends on the nature of the sample and the desired results. For low detection limits, high mass resolution, or stigmatic imaging, a magnetic sector-based instrument should be used. The analysis of dielectric materials (in many cases) or a need for ultrahigh depth resolution requires the use of a quadrupole instrument.

Conclusions

SIMS is one of the most powerful surface and microanalytical techniques for materials characterization. It is primarily used in the analysis of semiconductors, as well as for metallurgical, and geological materials. The advent of a growing number of standards for SIMS has greatly enhanced the quantitative accuracy and reliability of the technique in these areas. Future development is expected in the area of small spot analysis, implementation of post-sputtering ionization to SIMS (see the articles on SALI and SNMS), and newer areas of application, such as ceramics, polymers, and biological and pharmaceutical materials.

Related Articles in the Encyclopedia

Static SIMS, SALI, SNMS, and Surface Roughness

References

- 1 G. Slodzian. *Surf. Sci.* **48**, 161, 1975.
- 2 C. A. Andersen. *Int. J. Mass. Spect. Io Phys.* **3**, 413, 1970.
- 3 E. Zinner, S. Dnst, J. Chaumont, and J. C. Dran. *Proceedings of the Ninth Lunar and Planetary Sciences Conference.* 1978, p. 1667.
- 4 P. Williams. *IEEE Trans. Nucl. Sci.* **26**, 1809, 1979.