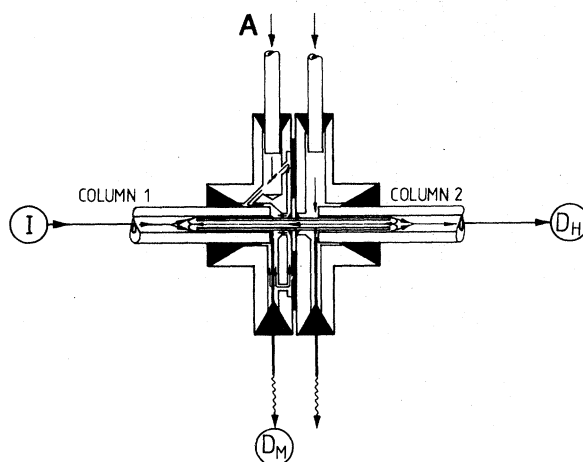


mated. The GC/9630 and the Sichromat 2 have two ovens whose temperatures can be independently controlled; the Series 500 has a third oven for accessories. The Sichromat comes with a coupling piece and the Series 500 comes with a mechanical valve, but the GC/9630 can be ordered with either a coupling piece or a mechanical valve. An IBM spokesman contends that the valve is satisfactory for more than 80 percent of potential applications.

All three GC's can be equipped with two sets of injectors and detectors so that the two columns can be operated in parallel (when, for example, it is desirable to separate a mixture under two sets of conditions to provide a definitive identification of components) or completely independently. The price of the GC/9630 ranges from \$14,000 to \$22,000, depending on the options selected; the base price of the Sichromat 2 fully equipped for MDGC is slightly under \$30,000; the price of the Series 500 ranges from \$15,000 to \$25,000.

MDGC can also be carried out with three or four columns. E. G. Boeren of Packard Instrument B.V. (Delft, the Netherlands), for example, described a three-column system for analyzing paraffins, olefins, naphthenes, and *iso*-aroma-



#### "Live" switching

The coupling piece devised by David Deans has no moving parts; the direction of flow of eluant gas is controlled by small differences in air pressure. Changes in pressure through inlet A can shift the flow of eluant from column 1 either to a detector ( $D_M$ ) or to column 2 and its detector ( $D_H$ ) [Source: Siemens AG]

tics in naphtha. The naphtha is first passed through a polar precolumn that separates it into two groups, one containing aromatics and the second containing saturated hydrocarbons and olefins.

The second fraction is passed through an Olefin Trap that captures all olefins containing more than five carbons. The saturated hydrocarbons pass through the trap and are carried to a column in a heated zone above the oven where they are separated according to number of carbons. The flow of carrier gas through the trap is then reversed and the temperature raised to release the olefins;

they then pass through a reactor which hydrogenates them for separation on the column in the heated zone. Meanwhile, the aromatics are separated by number of carbons on another column in the oven. A GC incorporating all these features and known as the model 412A PiANO Analyzer was displayed at the conference by Packard Instrument Company (Downers Grove, Illinois). This device is designed for petroleum refiners and for ink and paint manufacturers who must know the precise composition of naphtha, and sells for just under \$35,000.

—THOMAS H. MAUGH II

## Ion Beams for Compositional Analysis

Many analytical techniques can be classified according to the kind of particles (photons, electrons, atoms, ions, and so on) that irradiate an unknown and the kind of particles that emerge from the sample to be detected. Secondary ion mass spectrometry (SIMS) is a highly sensitive, surface-specific technique that makes use of ions (although not the same ones) in both roles. This year's Pittsburgh Conference saw the introduction of two other ways to use ions to quantitatively determine concentrations of elements in solid samples, as well as continued progress in SIMS instrumentation itself.

In SIMS, the bombarding or primary ion beam, which can be any ion (argon, oxygen, nitrogen, and cesium are common) has an energy of a few thousand electron volts. Collisions with atoms near the surface soon bring the ions to a halt, dislodging in the process a conglomeration of neutral atoms and negatively and positively charged ions that fly away from the surface. As many of the low-energy secondary ions as possible

are collected and transported to a mass spectrometer (almost always a quadrupole analyzer).

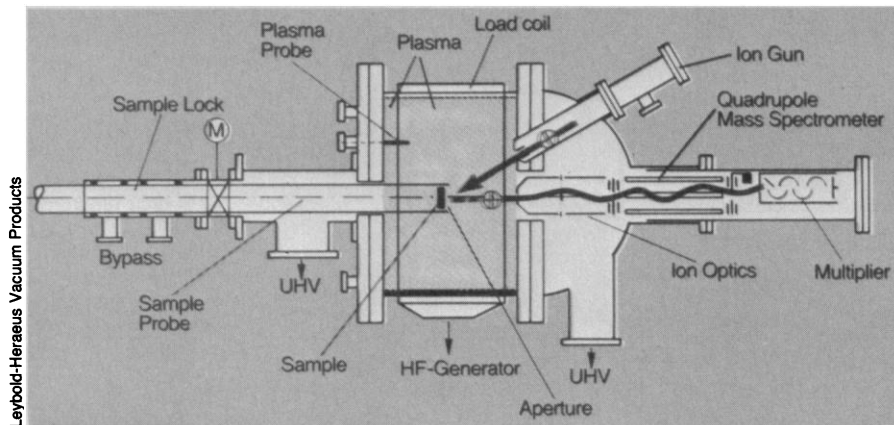
Advantages of SIMS include a high sensitivity (in the parts-per-billion range for some elements), a fairly high spatial resolution (down to 1 micrometer or so), and an ability to do depth profiling; that is, obtain concentrations as a function of depth below the initial surface, when the surface layers are one after another sputtered away by the primary ion beam. The main disadvantage is a tremendous variation (a factor of 10,000 or more) in the efficiency of producing secondary ions, according to the identity of the ion, its neighbors in the solid sample prior to being sputtered, and the primary ion species. This variation makes SIMS difficult, although not impossible, to quantify.

Rutherford backscattering (RBS) is, to physicists at least, an ancient technique that has now been neatly packaged by the General Ionex Corporation (Newburyport, Massachusetts) and presented for the first time to Pittsburgh Confer-

ence attendees in the form of the model 4175 Rutherford Backscattering Surface Analyzer.

RBS by no means replaces SIMS but instead is complementary. The sensitivity is about 10,000 times poorer than that of SIMS, and there are for the moment no focused-beam, scanning systems to provide a high spatial resolution. However, the technique is absolutely quantitative and there is no need whatsoever for standards. Moreover, RBS is nondestructive. It permits quantitative depth profiling of all elements from lithium through uranium without the need to sputter away the surface.

The quantitativeness comes from the fact that the physical principle of RBS is not much more complicated than that in the freshman physics laboratory experiments studying elastic collisions between steel balls. A beam of doubly ionized helium ions of energy 2 million electron volts (MeV) enters the surface. When an ion strikes an atom in the sample head on, the ion reverses its path and comes back out. The energy of the



### Depth profiler

Schematic diagram shows the Leybold-Heraeus INA 3 operating in the SNMS-separate bombardment mode.

backscattered ion gives the mass and hence the identity of the atom it collided with. Concentrations of each species come from the number of backscattered ions at the appropriate energies. Inelastic collisions on the way in and out give an additional energy loss that can be converted into the depth below the surface of the scattering atom.

At the center of the General Ionex system is a 2-MeV electrostatic accelerator that provides a beam with cross section 2 millimeters by 2 millimeters. Cost of a complete system, including control computer and software, is \$250,000.

Leybold-Heraeus Vacuum Products, Inc. (Export, Pennsylvania), has managed to preserve (and enhance in some cases) the sensitivity of SIMS while considerably improving its quantitiveness with the introduction of its INA 3 Quantitative Depth Profiler. The secret of the instrument is the separation of the ionization from the sputtering processes, which has two effects. Because the ionization takes place away from the surface, the factors that cause the tremendous variations in production of ions in SIMS are largely eliminated. And be-

cause the more numerous neutral atoms that go undetected in the normal SIMS process are ionized, more ions are collected for mass analysis, which enhances the sensitivity.

The INA 3 can operate in three modes. One is the normal SIMS mode in which a 125-micrometer-diameter beam sputters ions from the surface, which are collected and analyzed by a quadrupole mass spectrometer. The second is the secondary neutrals mass spectrometry (SNMS)—separate bombardment mode, in which neutral species sputtered from the surface by the ion beam are ionized by electrons in a hydrogen fluoride plasma that is generated around the sample. The third is the SNMS—direct bombardment mode, in which the ions in the plasma do the sputtering, while the electrons do the ionizing. All three modes are capable of depth profiling. However, the spatial resolution of SIMS is not available. Cost is \$200,000 or more.

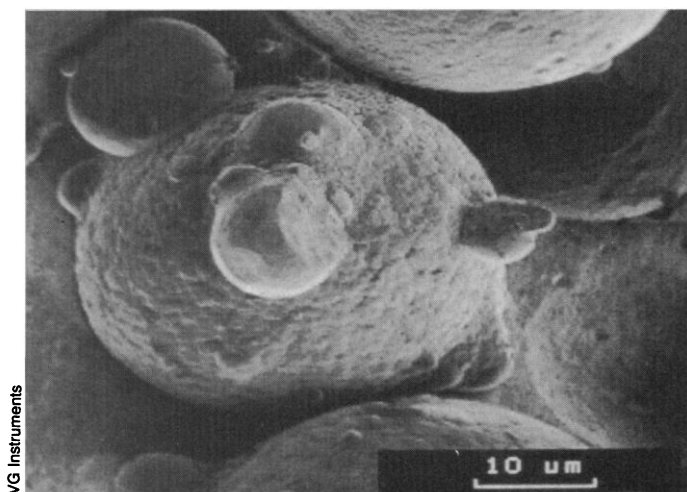
VG Instruments, Inc. (Stamford, Connecticut), exhibited a new instrument that makes use of the same principle as the SNMS—direct bombardment mode; namely, the VG 9000 Glow Discharge

Mass Spectrometer. The VG 9000 is not intended, however, for surface analysis or depth profiling; it is a high-sensitivity (10 parts per billion atomic is typical) mass spectrometer for high-speed analysis of solids. Positive ions in an argon (or other gas) plasma with energies of a few hundred electron volts sputter mostly neutral species from the surface. These diffuse through the plasma and are ionized by collisions. Positive ions are collected and accelerated into a double-focusing (magnetic sector for mass and electrostatic analyzer for energy) mass spectrometer of resolution 4000. A special feature is a dual detector system that gives the instrument a dynamic range of about  $10^8$ . The control computer automatically switches in the detector appropriate for low and high concentrations. Cost of the VG 9000 is approximately \$350,000.

Because SIMS is by now a venerable technique, instrument manufacturers to provide equipment are numerous. Among those at the Pittsburgh Conference this year were Atomika, Inc. (Inglewood, California), Cameca Instruments, Inc. (Stamford, Connecticut), Dubilier Scientific (represented in the United States by Ion Beam Technologies, Beverly, Massachusetts), the Kratos Analytical division of Spectros, Inc. (Ramsey, New Jersey), the Physical Electronics division of Perkin-Elmer (Eden Prairie, Minnesota), Advanced R&D, Inc. (St. Paul, Minnesota), the Ribier division of ISA, Inc. (Metuchen, New Jersey), and VG Instruments.

Mainline SIMS instruments run from about \$150,000 at the low end to about \$600,000 for Cameca's unique imaging SIMS machine. However, Advanced R&D introduced what it called a mini-SIMS system for \$59,900 (excluding a computer, which adds from \$5,900 to \$29,000) that is intended for high-sensitivity but low-resolution routine analysis. The hope, if not the expectation, is that the system will do for SIMS what an analogous low-cost scanning electron microscope did for scanning electron microscopy several years ago.

In the opposite direction, Dubilier Scientific and VG Instruments offered ultra-high spatial resolution (500 angstroms was advertised) SIMS systems featuring liquid metal ion sources (*Science*, 14 September 1984, p. 1137). Scientists are just learning how to use this kind of spatial resolution. One negative feature is that the analyzed area contains too few atoms to have simultaneously a high spatial resolution and a high sensitivity for trace elements. Prices start at \$250,000.—ARTHUR L. ROBINSON



### SIMS Image

In addition to depth profiles, SIMS is capable of imaging with ions in a manner analogous to scanning electron microscopy, as in this micrograph of 30-micrometer-diameter steel balls on an alumina support made by the VG Instruments SIMSLAB.