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SCANNING ION MICROPROBE WITH MAGNETIC SECTOR LENS TO
PURIFY THE PRIMARY ION BEAM
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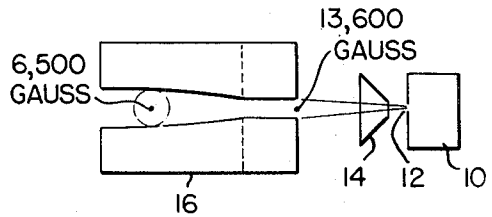


FIG. 2

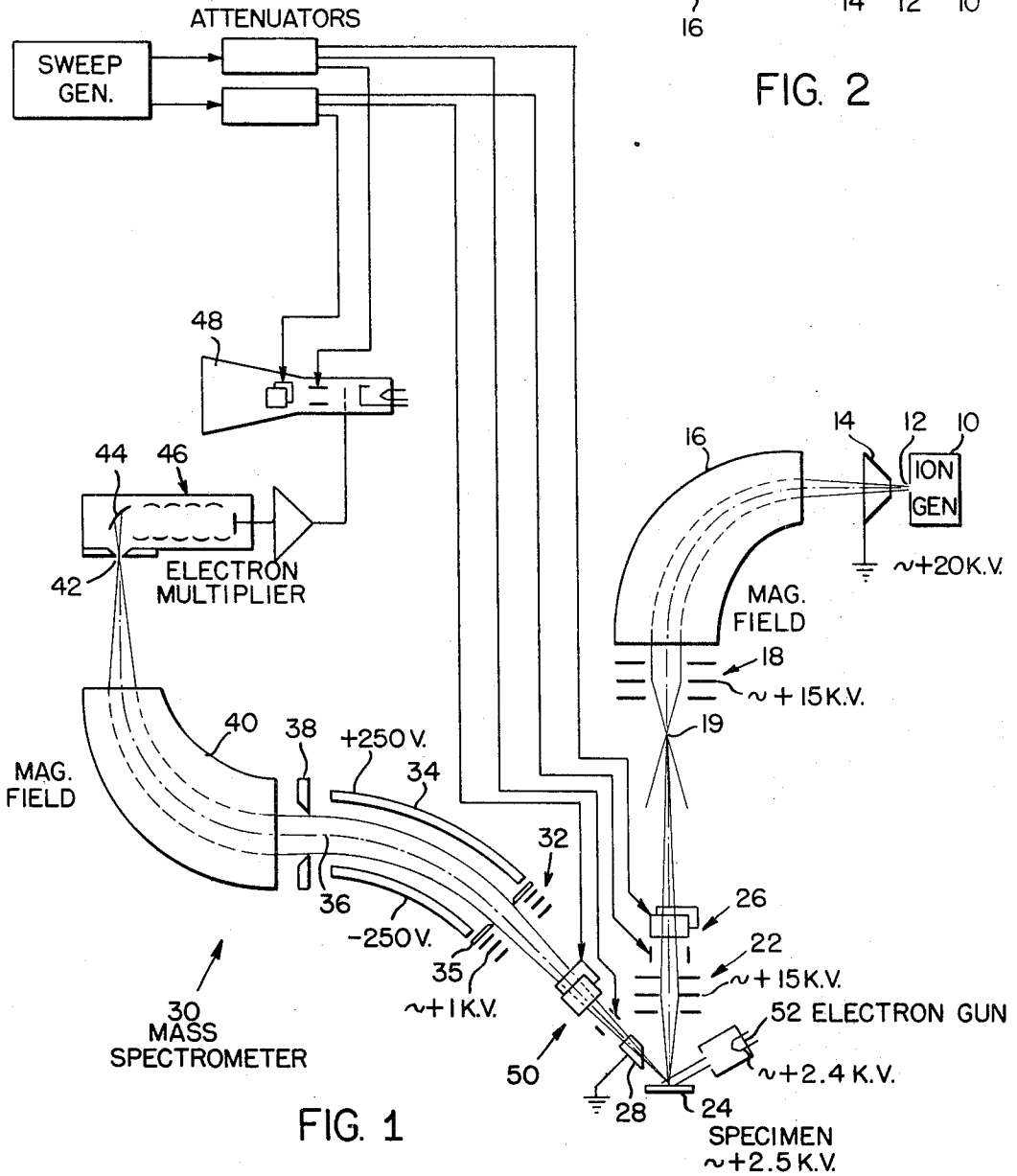


FIG. 1

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SCANNING ION MICROSCOPE WITH MAGNETIC SECTOR LENS TO PURIFY THE PRIMARY ION BEAM

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4 Claims

ABSTRACT OF THE DISCLOSURE

An ion probe in which discrimination between successive elemental area portions of a specimen is achieved by focussing the primary ion beam to a very small spot on the specimen. Features include a double-focussing, stigmatic imaging mass spectrometer for analyzing the secondary ions, and a magnetic sector lens for purifying the primary beam.

This invention relates to novel apparatus for microanalysis by secondary ion emission, that is, by bombarding the surface of a material to be analyzed with ions to cause the material to emit ions characteristic of its composition, and then analyzing the ions so emitted to determine the composition of the material or the concentration of a particular constituent thereof.

Electron microprobes of the type, for example, shown by Wittry in U.S. Pat. No. 3,107,297 are in wide use for microanalysis. In such instruments, relatively small surface areas of specimens to be analyzed are irradiated by electron beams to cause them to emit X-ray radiation characteristic of their composition. The emitted X-rays usually analyzed by X-ray spectrometers. Electron microprobes are capable of analyzing the spatial distribution of many elements on the surface of a specimen with a resolution of the order of one micron. Electron microprobes, however, are subject to certain basic limitations. The sensitivity of X-ray spectrometers is poor for elements of low atomic number, and the first few elements of the periodic table cannot be analyzed at all. Also, the sensitivity of electron microprobes, that is, their ability to detect elements present in relatively small concentrations, is limited by the presence of a background continuum.

These limitations are largely avoided by methods of analysis by secondary ion emission, in which a specimen to be analyzed is bombarded by energetic ions causing the specimen to emit secondary ions characteristic of its composition. The secondary ions may then be analyzed by a mass spectrometer.

One analytical system based upon ion bombardment and analysis is described by George S. Slodzian in his doctoral thesis starting at p. 591 of *Annals de Physique*, vol. 9, 1964. Slodzian teaches bombarding a relatively large surface element with an ion beam, the then focussing ions emitted in response to the bombardment to form an enlarged ion image of the entire surface element. This method suffers from poor sensitivity due to the fact that the apertures in the ion optical imaging system must be very small, with the result that only a relatively small proportion of the ions emitted by the specimen can be utilized to form the image.

Accordingly, the principal object of the present invention is to improve the technique of chemical analysis by secondary ion emission.

A further object is to provide apparatus for rapid chemical analysis which overcomes to a large extent the basic limitations of the electron microprobe and of the methods of ion analysis heretofore known, and which

permits the rapid analysis of materials without limitation as to the light elements and with a very high degree of sensitivity.

The invention will now be described in detail in connection with the accompanying drawing, wherein;

FIG. 1 is a schematic diagram of an ion microprobe according to the presently preferred embodiment of the invention; and,

FIG. 2 is a fragmentary, elevational view, in schematic form of a portion of the microprobe shown in FIG. 1, looking generally in the direction of the arrow 2 in FIG. 1.

Briefly, it has now been found that improved results, especially with regard to sensitivity, may be achieved in an ion analyzer by focussing the bombarding beam upon a small surface area of the specimen, an area smaller than the ion outlet orifice of the ion source. The ions emitted by the specimen responsively to the bombardment are analyzed by a mass spectrometer having a large aperture and a high transmission factor. In this way, a relatively large proportion of the emitted ions are gathered into the mass spectrometer and used in the analysis, and a high sensitivity and signal to noise ratio are achieved.

The size of the bombarding beam at its point of impact on the specimen determines the resolution of the analyzer, and this may be made comparable to the resolution achieved by Slodzian and by the electron microprobe. The ions passed through the spectrometer are simply detected, and are not used to produce an enlarged ion image of the excited area of the specimen. Relatively large aberrations may, therefore, be tolerated in the spectrometer without significant loss of resolution, so that it is possible to use a spectrometer having a large aperture, thus enabling a relatively large proportion of the secondary ions to be directed into and analyzed by the spectrometer.

Further features of the invention relate to electron bombardment of the secondary emission to increase the number of ions available for analysis, and to the provision of a deflecting system for both the bombarding and the secondary ion beams to enable rapid and convenient point by point micro-analysis of a selected region of the specimen surface.

Referring now to the drawing, an analyzer according to the presently preferred embodiment of the invention includes an ion source 10 which may be of any desired type. It may, for example, consist of a vessel containing an atmosphere of a noble gas such as argon at a few microns of mercury pressure. Electrodes (not shown) within the vessel are energized to produce a low voltage arc discharge through the gas to produce ions, positive ones of which emerge through orifice 12 into the relatively highly evacuated part of the analyzer. The ion source 10 is conveniently kept at a relatively high positive potential, typically 20 kv. relative to ground, and the ions are accelerated away from the orifice 12 through a grounded accelerating electrode 14 toward a filter device 16.

The ions emitted from the orifice 12 include a major proportion of ions of the noble gas, but also a minor proportion of impurity ions stemming from gasses desorbed from the walls of the vessel enclosing the gas and from particles sputtered from the electrodes that produce the arc. The purpose of the filter device 16 is to divert the impurity ions away from the ions of the noble gas, and to concentrate the noble gas ions in substantially pure form in a direction toward the specimen 24 to be analyzed. As shown, the filter 16 consists of a wedge-shaped magnetic sector field having a plane of symmetry in the plane of the drawing and tapering toward the ion source 10. Such a field focusses both in the plane of symmetry and in the direction normal to it so that the desired ions of a selected momentum emerge from it along parallel paths. The field

is described in greater detail in my copending application for patent Ser. No. 494,378, filed concurrently herewith, and entitled, "Double Focussing Mass Spectrometer Including a Wedge-Shaped Magnetic Field" now U.S. Pat. 3,445,650.

After passing through the filter 16, the noble gas ions pass through an electric unipotential lens 18, which forms an ion image 19 of the orifice 12 demagnified by about ten diameters. The lens 18 may be referred to as the condenser lens. A portion of the ions emerging from the condenser lens 18 is received by an objective lens 22, which, as shown, is also a unipotential lens, and which produces a further demagnification of the ion image at the surface of the specimen 24 to be analyzed. The ion beam is thus purified and concentrated to a spot on the surface of the specimen about $\frac{1}{100}$ the diameter of the ion exit orifice 12, so that if, for example, the orifice 12 is about 0.1 mm. in diameter, an area of only about one micron diameter will be bombarded by the ion beam.

A set of deflection plates 26 is positioned just above the objective lens 22 for deflecting the ion beam in each of two coordinate directions so that it can be caused to scan a selected area of the specimen surface.

Wherever the ion beam impinges upon the specimen, particles of the specimen material sputter off the surface. These particles are predominantly neutral atoms representative of the composition of the specimen. A small fraction of the sputtered particles, however, are positive ions. The specimen is kept at a positive potential, typically 2.5 kv. relative to ground, so that the positive ions emitted are accelerated through a grounded electrode 28 toward the mass spectrometer, generally designated 30. Due to the small size of the ion emitting spot on the specimen surface, an entrance slit is not necessary for the mass spectrometer.

The mass spectrometer shown is described in greater detail and claimed in my hereinabove identified copending application. A brief description will suffice for the purpose of this application. The spectrometer 30 is double focussing in the sense generally intended in the art of mass spectrometry, meaning that it produces simultaneous angular and energy focussing. It is stigmatic imaging, meaning that the first order, or Gaussian image point in the radial plane (the plane of curvature of the median particle path 36, which is also the plane of the drawing) coincides with the first order image point in the axial plane (the plane through the median particle path at the image point and normal to the radial plane). It also has a relatively large acceptance angle. These features contribute to a relatively high ion transmission factor, enabling the analysis of a relatively large fraction of the ions emitted by the specimen 24, and thus providing a high sensitivity.

The first element of the mass spectrometer 30 is an electric unipotential lens 32, which directs ions from the accelerator electrode 28 into a spherically curved, toroidal condenser 34 wherein the median particle path 36 is deflected through an angle of about 45°. The entrance aperture of the condenser 34 is defined by an apertured diaphragm 35, which is preferably adjustable to permit adjustment of the resolution and sensitivity of the spectrometer. The toroidal condenser acts as an energy filter to spread the ions in accordance with their respective energies. Ions having energies within a selected range then pass through an apertured diaphragm 38, the energy selector, which is also preferably adjustable and which permits only ions within the selected range of energies to pass into a wedge-shaped magnetic sector field 40. The magnetic sector field 40 disperses the ions in accordance with their respective momenta, or masses, and focusses ions of a selected mass upon the exit aperture 42, which is also preferably adjustable in width. The width of the exit aperture 42 determines the mass resolving power of the spectrometer, which is also affected by the sizes of the entrance aperture in the diaphragm 35 and of the selector aperture in the diaphragm 38. In operation all of these apertures

are ordinarily adjusted together to achieve the maximum resolution consistent with a desired sensitivity.

Ions passing through the exit aperture 42 impinge upon the receptor 44 of an electron multiplier 46, which produces an electrical signal responsively to the intensity of the ion current arriving at the receptor 44. The signal may be displayed upon a meter, or, as shown, is preferably amplified and used to modulate the intensity of the electron beam of an oscilloscope 48.

In the embodiment shown, the beam deflection of the oscilloscope 48 is synchronized with the deflection of the primary ion beam in both coordinate directions, so that, when a selected area of the specimen is scanned by the bombarding ion beam, the screen of the oscilloscope will display an enlarged picture depicting the distribution of a selected element, or isotope on the specimen surface, differences in concentration from point to point being shown by differences in brilliance. The particular element, or isotope is selected by appropriate tuning of the mass spectrometer in accordance with known principles.

An auxiliary set of deflection plates 50 is preferably positioned between the secondary accelerating electrode 28 and the entrance to the mass spectrometer 30 to maximize the resolving power of the microprobe by compensating for the effect of scanning the bombarding beam across the surface of the specimen. In the absence of the auxiliary deflecting plates 50, the crossover of the secondary ion beam at the exit aperture 42 of the mass spectrometer will move as the primary, or bombarding beam is scanned across the specimen surface, and it would be necessary to enlarge the exit aperture 42 sufficiently to allow for this movement. Enlarging the exit aperture 42 would reduce the mass resolving power of the spectrometer. Provision of the auxiliary deflecting plates 50 obviates the need to enlarge the exit aperture 42, because by energizing the auxiliary plates 50 synchronously with the primary deflecting plates 26, the crossover at the exit aperture 42 can be kept steady.

An electron gun 52 is provided according to a further feature of the invention for increasing the ion emission of the specimen 24. Only a small fraction of the particles sputtered from the specimen responsively to the ionic bombardment leave the specimen 24 in an ionized state. Most of the sputtered particles are in the form of neutral atoms and molecules. According to the invention, the electron gun 52 is laterally offset from the ion beam and positioned to direct an electron beam toward the area of the specimen 24 under investigation. By appropriate choice of the magnitude of the electron beam current and energy, which is most conveniently determined by trial and error separately for each constituent subject to analysis, a substantial portion of the sputtered neutral particles may be ionized by the electron impact, thereby further increasing the ions available for analysis.

Typical operating voltages (all relative to ground) of the various elements of the microprobe are indicated in the drawing. These values are not limiting factors in the practice of the invention but have been determined to be within optimum ranges for an ion microprobe in which the various elements have the following respective approximate dimensions and spacings.

Distance from ion outlet orifice 12 to the filter 16—5 inches.

Average radius of curvature of the median ion path through the filter 16—5.5 inches.

Spacing between the respective center electrodes of the unipotential lenses 18 and 26—10 inches.

Spacing between the center electrode of the second unipotential lens 26 and the specimen 24—1 inch.

Median radius of curvature of the spherical condenser 34—10 inches.

The entrance aperture 35 and the selector aperture 38—adjustable from about 1 mm. to about 10 mm. dia.

Average radius of the median ion path through the magnetic sector field 40—5.5 inches.

Width of the exit aperture 42—adjustable between 1.2 mm. and 10 microns.

The invention is not intended to be limited to the particular arrangements illustrated for focusing the bombarding ion beam upon the specimen 24, nor to the particular spectrometer shown. It is directed primarily to the concept in microanalysis by secondary ion emission of achieving fine resolution by exciting only a very small surface area of the specimen under analysis, and using a mass spectrometer of large aperture to analyze ions sputtered from the excited area. The resolution attainable in the practice of the invention is comparable to the best heretofore achieved in microanalysis, while the sensitivity, i.e., the ability to detect constituents present in only small concentrations, is improved by a substantial factor relative to previous work in microanalysis by secondary ion emission that I am aware of.

In the embodiment illustrated herein, the mass spectrometer covers the mass range up to mass 1000. Its mass resolving power may be controllably varied from about 100 up to about 10,000. The concentrational sensitivity is in the range of a few parts per million for most elements, and a few parts per billion for most elements, and a few parts per billion for those elements that are relatively easily ionized such as, for example, the alkali metals.

What is claimed is:

1. An ion microprobe comprising means for producing a primary ion beam and directing it toward a specimen to be analyzed, focusing means to focus the ion beam upon the specimen, deflection means for deflecting the beam across the specimen so that it strikes successive incremental area portions of the surface of the specimen, a mass spectrometer for analyzing secondary ions sputtered from the specimen responsively to the impact of the primary ion beam, a transducer for producing electrical signals responsively to secondary ions analyzed by said mass spectrometer, an oscilloscope, means for modulating the intensity of the electron beam of said oscilloscope responsively to signals produced by said transducer, and deflection means for deflecting the electron beam of said oscilloscope synchronously with the deflection of the primary ion beam across the specimen, and deflection means along the secondary ion path between the specimen and said transducers for deflecting the ions analyzed by said mass spectrometer synchronously with the deflection of the primary ion beam to keep the selected output of said mass spectrometer at one location thereby to compensate for the deflection of the primary ion beam and permit use of an exit slit of minimum width in said mass spectrometer.

2. Ion probe apparatus for chemical analysis of surface portions of a specimen by bombarding the specimen with primary ions and mass-spectrometrically analyzing secondary ions emitted by the specimen responsively to the primary ions comprising:

- (a) an ion source,
- (b) focusing means arranged between said source and a predetermined specimen position for focusing ions emitted by the source upon a specimen, and
- (c) a magnetic sector lens between said source and

said focusing means for directing only ions of a preselected kind to said focusing means and for diverting all other ions away therefrom.

3. Apparatus for microanalysis including means for directing a beam of primary ions upon a selected small portion of the surface of a specimen material, and means for analyzing secondary ions emitted by the surface responsively to the primary ions directed thereupon, said directing means comprising an ion generating device having an outlet orifice of predetermined size, means for accelerating ions away from said orifice toward a specimen, ion focusing means for forming ions so accelerated into a narrow beam having a cross-sectional area at the specimen surface smaller than said outlet orifice, and a magnetic sector lens along the path of the primary ions for purifying the beam by directing only selected ions toward the specimen and diverting all other ions away from the specimen.

4. Apparatus for microanalysis including means for directing a beam of primary ions upon a selected small portion of the surface of a specimen material, and means for analyzing secondary ions emitted by the surface responsively to the primary ions directed thereupon, said directing means comprising an ion generating device having an outlet orifice of predetermined size, means for accelerating ions away from said orifice toward a specimen, ion focusing means including two electric unipotential lenses arranged in series along the path of the ions for forming ions so accelerated into a narrow beam having a cross-sectional area at the specimen surface smaller than said outlet orifice by a demagnification of at least about 100, and a magnetic sector lens along the path of the primary ions for purifying the beam by directing only selected ions toward the specimen and diverting all other ions away from the specimen.

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WILLIAM F. LINDQUIST, Primary Examiner

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250—41.9