PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : G01N 23/00, H01J 37/08 — A1

(11) International Publication Number: **WO 99/15884**

(43) International Publication Date: 1 April 1999 (01.04.99)

(21) International Application Number: PCT/US98/19823

(22) International Filing Date: 22 September 1998 (22.09.98)

(30) Priority Data:
60/059,452 22 September 1997 (22.09.97) US

(71) Applicant: ORYX INSTRUMENTS & MATERIALS CORPORATION [US/US]; 47341 Bayside Parkway, Fremont, CA 94538 (US).

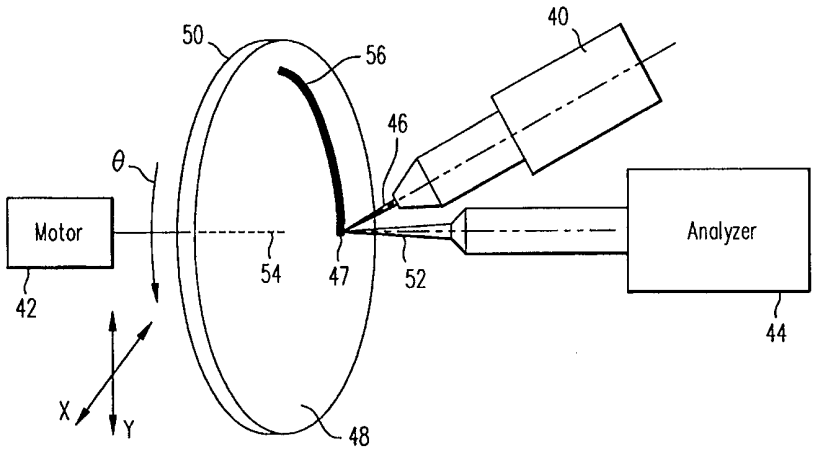
(72) Inventors: HALL, Bernard; 1618 27th Avenue, San Francisco, CA 94122 (US). LINDER, Robert; 2464 Franciscan Court, Santa Clara, CA 95051 (US). EDELSON, Lawrence; 507 Maar Place, Fremont, CA 94536 (US). GREENE, Andrea; 516 Gibraltar, Foster City, CA 94404 (US).

(74) Agents: LIMBACH, Alan, A. et al.; Limbach & Limbach L.L.P., 2001 Ferry Building, San Francisco, CA 94111 (US).

(81) Designated States: JP, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.

(54) Title: MONOLAYER ANALYSIS USING DYNAMIC SECONDARY ION MASS SPECTROMETRY



(57) Abstract

A method and apparatus for studying the composition of a sample (50) using a primary ion beam (46), where an ion source (40) produces a primary ion beam directed onto the surface (48) of a sample for etching material from the sample surface to produce a secondary ion beam (52). The sample is rotated or translated, or the primary ion beam is moved to scan along the sample surface, so that a target spot (47) at which the primary ion beam strikes the sample's surface constantly moves to etch a trench (56) into the sample's surface while an analyser (44) measures either the mass or electrical charge of ions in the secondary ion beam, or the energy ions in the primary beam scattered by the sample, in order to determine the sample's composition. The ion current level of the primary ion beam and the rate of movement between the primary ion beam and the sample are selected so that the trench has a depth coinciding with a predetermined depth of material to be analyzed. Therefore, ions being analyzed originate from or interact with only those atoms or molecules residing in the predetermined depth of interest.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	**ES**	Spain	**LS**	Lesotho	**SI**	Slovenia
AM	Armenia	**FI**	Finland	**LT**	Lithuania	**SK**	Slovakia
AT	Austria	**FR**	France	**LU**	Luxembourg	**SN**	Senegal
AU	Australia	**GA**	Gabon	**LV**	Latvia	**SZ**	Swaziland
AZ	Azerbaijan	**GB**	United Kingdom	**MC**	Monaco	**TD**	Chad
BA	Bosnia and Herzegovina	**GE**	Georgia	**MD**	Republic of Moldova	**TG**	Togo
BB	Barbados	**GH**	Ghana	**MG**	Madagascar	**TJ**	Tajikistan
BE	Belgium	**GN**	Guinea	**MK**	The former Yugoslav Republic of Macedonia	**TM**	Turkmenistan
BF	Burkina Faso	**GR**	Greece			**TR**	Turkey
BG	Bulgaria	**HU**	Hungary	**ML**	Mali	**TT**	Trinidad and Tobago
BJ	Benin	**IE**	Ireland	**MN**	Mongolia	**UA**	Ukraine
BR	Brazil	**IL**	Israel	**MR**	Mauritania	**UG**	Uganda
BY	Belarus	**IS**	Iceland	**MW**	Malawi	**US**	United States of America
CA	Canada	**IT**	Italy	**MX**	Mexico	**UZ**	Uzbekistan
CF	Central African Republic	**JP**	Japan	**NE**	Niger	**VN**	Viet Nam
CG	Congo	**KE**	Kenya	**NL**	Netherlands	**YU**	Yugoslavia
CH	Switzerland	**KG**	Kyrgyzstan	**NO**	Norway	**ZW**	Zimbabwe
CI	Côte d'Ivoire	**KP**	Democratic People's Republic of Korea	**NZ**	New Zealand		
CM	Cameroon			**PL**	Poland		
CN	China	**KR**	Republic of Korea	**PT**	Portugal		
CU	Cuba	**KZ**	Kazakstan	**RO**	Romania		
CZ	Czech Republic	**LC**	Saint Lucia	**RU**	Russian Federation		
DE	Germany	**LI**	Liechtenstein	**SD**	Sudan		
DK	Denmark	**LK**	Sri Lanka	**SE**	Sweden		
EE	Estonia	**LR**	Liberia	**SG**	Singapore		

MONOLAYER ANALYSIS USING DYNAMIC SECONDARY ION MASS SPECTROMETRY

FIELD OF THE INVENTION

The present invention relates to secondary ion mass spectrometry (SIMS), and more particularly to a method and apparatus for collecting static SIMS like data in a dynamic manner.

BACKGROUND OF THE INVENTION

Secondary ion mass spectrometry (SIMS) involves analyzing the secondary ion emissions from a sample to determine surface and bulk properties as well as to detect surface and bulk contamination. In this process, a sample such as a silicon wafer is bombarded with an ion beam (primary ions), whereupon surface atoms and molecules under bombardment are ejected from the sample's top surface. Ionization of the ejected atoms and molecules takes place a few angstroms outside of the sample's surface. These ions (secondary ions) are accelerated into an analyzer device to determine the mass, charge and/or energy of the secondary ions, from which the composition of the sample's surface can be determined. Bulk property determinations are made by continuing the primary ion bombardment, which etches a deeper hole into the sample surface to eject atoms and molecules from deeper within the sample. Thus, analyzing the secondary ions over time reveals the changing composition of different monolayers of the sample.

There are two basic types of SIMS: dynamic and static. A typical dynamic SIMS system uses a primary ion beam with sufficient current (100-1000 nano amps) to erode the sample surface at rates of 10-1000 angstroms per second. This allows for depth profiling of the sample, where the primary ion beam etches through multiple monolayers over time, and atoms and molecules are collected from the different monolayers in the sample and analyzed.

The drawback with dynamic SIMS systems is that they cannot utilize analyzer devices that collect data serially to study secondary ions that are all ejected from the same monolayer in the sample. Serial data collection means

collecting and testing for one species of atoms and molecules at a time. Two such devices are a sector double focusing mass spectrometer and a quadrupole mass spectrometer, which use a magnetic and/or electric field to sweep the ionic species it is analyzing across a detector. This sweeping takes some period of time. Therefore, at any given time, one species is being collected while all others are being discarded. Since the sample is being constantly etched by the primary ion beam, the various analyzed species are ultimately collected from different monolayers in the sample.

Another drawback to dynamic SIMS systems is edge effects that adversely affect the data acquisition. Because the primary ion beam is not a perfect step function, the crater created by the primary ion beam has slightly sloped walls. Therefore, during depth profiling that accesses material deeper within the sample, some secondary ions are produced from the sides of the crater. Edge effects prevent a dynamic SIMS system from analyzing secondary ions produced solely from the bottom of the crater.

While there are sector double focusing instruments (of the Mattauch-Herzog design) which can detect more than one species at any given time, these instruments have significantly lower mass resolution capability (the ability of a mass spectrometer to resolve two different masses).

In a static SIMS system, the ion current is set in the range of 0.01 to 1000 pico amps. The erosion rates with such low primary ion current are as low as 10^{-4} atom layers per second. This allows for analysis of different species all from the top atom layer of the sample, even with spectrometers that collect data serially. However, the number of secondary ions produced by a static SIMS system is quite low, resulting in a low secondary ion signal, low system sensitivity, longer acquisition times and proportionally higher system noise. In addition, a static SIMS system does not allow for analysis of species collected from monolayers deeper within the sample (no depth profiling).

Many dynamic or static SIMS systems include analyzer devices that operate with a continuous secondary ion beam. However, there are time of

flight (TOF) type analyzers that require the secondary ion beam be pulsed, whereby all species leaving the sample surface are recorded for every applied primary ion pulse. However, resolutions greater than 1 part in 15,000 are rare, and are typically much lower. Further, the necessity to pulse the primary or secondary ion beams present numerous technological problems leading to limitations of this type of instrument. In addition, many pulses are required to acquire the necessary data, where the latter pulses produce secondary ions from deeper within the sample. Lastly, if the pulse energy is increased to increase the number of secondary ions produced (to increase the secondary ion signal and decrease the number of pulses), the primary ion beam pulses each may have enough energy to etch through more than one monolayer, thus preventing analysis of secondary ions produced from a single monolayer.

It is known in the art to rotate the sample with a center of rotation coincident about the analysis area to reduce sputter roughening associated with crystalline specimens (see Zaler, Thin Solid Films, 124, 223-230, 1985). In particular, the sample rotation produces an averaging of the incidence angle of the primary ion beam with respect to crystallographic orientation. However, rotating the sample about the analysis area still results in a crater that gets deeper with continuing etching, with secondary ions being produced from different sample depths.

It is also known to provide one chamber for applying a sample to a substrate, and transporting the substrate to a second chamber in which the sample is analyzed in a step-wait-analyze procedure. In U.S. patent 5,001,939, a plurality of data points using an Auger electron spectrometer are taken, where each data point is collected from a new spot on the sample surface. However, this technique has several drawbacks. The sample has to be moved between each data acquisition. This complicates the system because the sample position has to be moved and verified each time data is acquired, and the primary ion beam is shuttered between data acquisitions. Further, the data acquisition process is excessively long due to the added

steps moving the sample between data acquisitions. Lastly, this process does not reduce edge effects.

There is a need for a dynamic SIMS system that can access various depths of the sample, yet be able to extract and analyze species from a single monolayer of the sample using an analyzing device that collects data serially, and reduces edge effects.

SUMMARY OF THE INVENTION

The present invention solves the aforementioned problems by providing a SIMS device that moves the target spot on the sample surface at which the primary ion beam is directed to etch a trench thereon having a predetermined depth.

The apparatus of the present invention includes a particle source for producing a probe particle beam directed onto a surface of a sample to produce a secondary particle beam, an analyzer that measures over a period of time a property of the secondary particle beam, and a device for continuously moving at least one of the probe particle beam and the sample over the period of time so that the probe particle beam is continuously scanned along the surface of the sample over the period of time.

In another aspect of the present invention, the apparatus includes an ion source for producing a primary ion beam directed onto a surface of a sample. The primary ion beam etches material from the sample surface to produce a secondary ion beam. An analyzer measures over a period of time at least one of a mass, an electrical charge and an energy of ions of the secondary ion beam. A device continuously moves at least one of the primary ion beam and the sample over the period of time so that the primary ion beam etches a trench having a predetermined depth into the sample surface.

In yet another aspect of the present invention, a method of analyzing a sample includes the steps of producing a probe particle beam, directing the probe particle beam onto a surface of a sample to produce a secondary

particle beam, measuring over a period of time a property of the secondary particle beam, and moving one of the probe particle beam and the sample in a continuous manner over the period of time so that the probe particle beam is continuously scanned along the surface of the sample over the period of time.

In still yet another aspect of the present invention, a method of analyzing a sample includes the steps of producing a primary ion beam, directing the primary ion beam onto a surface of a sample where the primary ion beam etches material from the sample surface to produce a secondary ion beam, measuring over a period of time at least one of a mass, an electrical charge and an energy of ions in the secondary ion beam, and moving one of the primary ion beam and the sample in a continuous manner over the period of time so that the primary ion beam etches a trench having a predetermined depth into the sample surface.

Other objects and features of the present invention will become apparent by a review of the specification, claims and appended figures.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a partial perspective view of the basic design of a quadrupole spectrometer.

Fig. 2 is a partial side view of the basic design of a time of flight spectrometer.

Fig. 3 is a partial plan view of the basic design of a sector double focusing spectrometer.

Fig. 4 is a perspective view of the secondary ion mass spectrometer of the present invention.

Fig. 5 is a cross-sectional side view of sample and the etching of the top monolayer of the sample by the probe beam.

Fig. 6 is a perspective view of an alternate embodiment of the secondary ion mass spectrometer of the present invention.

Fig. 7 is a perspective view of a second alternate embodiment of the secondary ion mass spectrometer of the present invention.

Fig. 8 is a perspective view of a third alternate embodiment of the secondary ion mass spectrometer of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention is a method and apparatus for significantly reducing the total etch rate of the sample by continuous off-axis rotation and/or translation of the sample or secondary beam during SIMS data acquisition. The rotation or translation results in arc scans and profiles or line scans or profiles in which a thin trench is dug into the sample surface. This method and apparatus reduces the etch rate at any given point on the sample to a level comparable to static SIMS, thus allowing a dynamic SIMS system to collect static SIMS like data. Therefore, data can be collected in both a spectral (mass scan) mode and a depth profiling mode, meaning that all the data can be collected from the same depth level in the sample while still employing a high primary ion current level and using an analyzer device that collects data serially. Further, half the edge effects are eliminated.

PRINCIPLES IN SECONDARY ION ANALYSIS

When a probe beam of charged particles (i.e. primary ions) is directed onto a solid surface, atomic and molecular species are dislodged and ejected from the surface. A fraction of these species are ionized, both positively and negatively within a few angstroms of the surface from which they were ejected, and are referred to a secondary ions. The nature of the primary ion beam plays an important role in the surface ionization process. The number of surface ions produced will depend upon the energy, current density and type of ions making up the probe beam. Primary ion beams can have energies below 1 KeV, and are typically between 3 to 10 KeV. As the probe beam energy increases, so does the number of secondary ions produced. As a consequence of this higher energy, the secondary ions are

now ejected from not just the surface but from several atom layers deep. As the ion current of the probe beam increases, from as low as picoamps to as high as microamps, greater erosion of the sample surface takes place. The erosion is very slow (around 10^{-4} atom layers per second) in the picoamp range, but increases above one atom layer per second in the microamp range.

The type of ions in the probe beam used on the sample will have an effect on the resulting secondary ion beam. Typical primary ions are AR^+, O_2^+ and Cs^+, but molecular probe beams as well as neutral probe beams can also be utilized.

Since the presence of oxygen will in general enhance the surface ionization process, oxygen probe beams are useful for enhancing secondary ion yields. Thin surface oxide layers on a sample will also allow for high initial yields which diminish as the surface oxide is etched away.

MASS SPECTROMETERS

Three types of mass analyzer devices have been typically used for SIMS analysis: Quadrupole Mass Spectrometers, Time of Flight (TOF) spectrometers, and Sector Double Focusing spectrometers. Each mass analyzer has its advantages and disadvantages, as described below.

The basic design of a Quadrupole spectrometer is illustrated in Fig. 1, and includes four conducting rods 10, a detector 12, a central axis 14 and a power supply 16. The rods 10 are aligned parallel to the central axis 14 and have curved surfaces facing each other. A voltage $\pm\Phi_o/2$, which includes the combination of rf (V) and dc (U) voltages, is applied to each rod 10 by power supply 16 to produce the quadrupole field along axis 14 between rods 10. This voltage is given by:

$$\Phi_o = U\text{-}V\cos\omega t \qquad (1)$$

The charged secondary ions are directed along axis 14 and through the quadrupole field with a given velocity (typically using an extraction electrode

near the sample), whereby the resulting ion trajectories through this quadrupole field are mass dependent. Thus, in principle, for specific voltage settings for rods 10, an ion having a particular mass will follow a trajectory along axis 14, through the quadrupole field, and will exit and be measured by detector 12. All other ions having other masses will instead hit the rods 10 or instrument walls and not be measured by detector 12.

The relationship between what ion mass will be transmitted to the detector and the applied rf and dc voltages is given by the Mathieu equation, with equations of motion given as:

$$\frac{d^2u}{dt^2} + \frac{e}{Mr_o^2}\bullet(U-V\cos\omega t)u = 0 \qquad (2)$$

where u = x or y, and where:

$$4eU/M\omega^2r_o^2 = a_x = -a_y = a \qquad (3)$$

$$2eV/M\omega^2r_o^2 = q_x = -q_y = q \qquad (4)$$

$$\epsilon = 0.5\omega t \qquad (5)$$

such that the Mathieu equation is:

$$\frac{d^2u}{d\epsilon^2} + (a-2q\cos 2\epsilon)u = 0 \qquad (6)$$

This equation describes the ion trajectories through the quadrupole field. Thus, for particular dc and rf voltages, a given mass will be transported along a stable trajectory and exit the spectrometer to be detected by detector 12 (which is preferably a Faraday cup or electron multiplier). The dc and rf voltages are ramped to scan across and detect the various ion masses in the secondary ion beam.

The basic design of a time of flight (TOF) instrument is illustrated in Fig. 2, and is constructed to allow ions of a known velocity to traverse a known distance. The TOF instrument basically includes a drift tube 20 with ions drawn into one end by an extraction electrode and an ion detector 22 at

the other end. The time (t) for ions of mass (M) with constant energy (Ve) to traverse a drift length (L) is given by:

$$t = L(M/2Ve)^{1/2} \quad (7)$$

and with constant momentum p:

$$t = LM/P \quad (8)$$

In operation, bunches of ions leave an ion source (the sample surface) and the time duration for the arrival of the ions at the detector 22 is measured by comparing the arrival times with the timing of the primary ion pulse. The masses of the arriving ions are calculated using the equations (7) and (8).

More complicated TOF arrangements can be constructed whereby the ions traverse the electrostatic field regions as well as field free regions as they travel from ion source to detector. This allows for corrections due to the fact that not all ions will leave the ion source with precisely the same energy, momentum or angle as is assumed in the equations above.

The basic design of a Sector Double Focusing Mass Spectrometer is illustrated in Fig. 3, and includes an electrostatic analyzer (ESA) 30, a magnet-analyzer 32, and a detector 34 aligned to an optical axis 36. The term "double focusing" refers to the ability of the instrument to direct all ions entering the instrument having a certain mass-to-charge ratio (m/e) to the instrument's detector, independent of the initial angle or velocity of the ions as they enter the spectrometer. This type of spectrometer exhibits both direction and velocity focusing.

Under the general theory for sector double focusing instruments, the ion paths through the consecutive fields and drift regions is mathematically described as a power series in α and β (angular spread and velocity from the ion optical path). When the ion beam enters the magnetic sector of the double focusing combination, the ion beam is dispersed based upon the

charge-to-mass ratio. The magnetic analyzer 32 is of course a momentum analyzer, but since a double focusing arrangement is used, the magnetic sector is considered a mass analyzer. In this sector, the heavier masses traverse the magnetic field with larger radii than do ions with lighter masses. The focal point for lighter ions, after leaving the magnet and traversing some drift space, will be to the inside of the optic axis 36 while the focal point for heavier ions will be to the outside of the optic axis 36. The dispersion of the system can be thought of as the physical distance that the focal points of any two masses are separated. Thus, the greater the dispersion of the instrument, the better the mass resolution.

A sector double focusing instrument is operated first by selecting the appropriate electrostatic analyzer potential between a pair of electrostatic plate 38. For ions with energy (qV) traversing a radial electrostatic field with radius (r_e), the plate voltage (v) between plates 38 is given by:

$$v = \frac{2dV}{r_e} \tag{9}$$

where d is the separation between the electrostatic analyzer plates 38.

For any given magnetic field B in magnetic analyzer 32, an ion with a charge to mass ratio q/m will be transported through the sector field along a radius r_m according to:

$$\frac{q}{m} = \frac{2V}{B^2 r_m^2} \tag{10}$$

where qV is the kinetic energy of the ion.

In most cases, this type of analyzer is operated by scanning the magnetic field while at the same time monitoring the ion current exiting the spectrometer using detector 34 (preferably an electron multiplier, a faraday cup or a phosphor screen coupled with photomultipliers). In this manner, a plot of the magnetic field versus ion intensity is produced. A mass spectrum is produced by using the simple relationship to convert magnetic field to mass as follows:

$$B \propto (m)^{1/2} \quad (11)$$

ENERGY SPECTROMETERS

Ion Scattering Spectrometers utilize a ion probe beam in a similar manner as described above, but they measure energy instead of mass. In this technique, the primary ions directed onto the sample surface are also scattered, in a binary elastic collision, by the sample surface. In a binary elastic collision, a particle (in this case a primary ion) collides with an atom or molecule on or near the sample surface. Kinetic energy is conserved in the system because the collision is elastic, and the collision is binary because only two particles are involved. Thus, the energy of the scattered ions will be dependent upon the mass of the atoms on the surface of the sample which were responsible for the scattering. The scattered primary ions are energy analyzed using, for example, an electrostatic analyzer. The ratio of the energy of the scattered primary ion to the energy of the primary ion before scattering is related to the mass of the atom on the sample surface responsible for the scattering.

SECONDARY ION MASS SPECTROMETER

The secondary ion mass spectrometer of the present invention is illustrated in Figs. 4 and 5, and includes a primary ion source 40, a motor 42 and an analyzer 44.

The primary ion source 40 produces a primary ion beam (probe beam) 46. Examples of primary ion sources 40 include liquid metal sources, duoplasmatrons, and surface ionization sources.

The probe beam 46 is directed to a target spot 47 on a top surface 48 of a sample 50, whereupon a secondary ion beam (secondary beam) 52 is emitted therefrom and directed into the analyzer 44 (typically using an extraction electrode). The motor 42 is connected to the sample 50 to rotate it about a central axis of rotation 54 that is not coincident with the target spot 47 being irradiated by the probe beam 46. The analyzer 44 can be any

analyzer device that measures aspects of the ions in the secondary beam (such as mass, charge and/or energy), including those mass/energy spectrometers described above.

The essential feature of the present invention is the continuous movement of target spot 47 on sample surface 48 during data acquisition. This is accomplished in the embodiment of Fig. 4 by off-axis sample rotation, which is rotation of the sample 50 about a center axis of rotation 54 (through angle θ) which is not coincident with the target spot 47. Thus, when the motor 42 rotates the sample 50, the probe beam 46 etches a trench 56 into the sample surface 48 instead of a single hole.

The rotation speed of the sample 50, spot size of target spot 47, and the ion current of the probe beam 46 can selected so that trench 56 has any desired depth (such as a single monolayer) and width. For example, as shown in Fig. 5, the sample 50 has a plurality of monolayers 58. The sample rotation speed, spot size of target spot 47 and probe beam ion current can be set to etch only the first (top) monolayer. Thus, secondary ions are produced only from the first monolayer throughout one complete revolution of the sample, which is ideal for analyzing multiple species from a single monolayer using particle analyzers that collect data serially. If one revolution of the sample does not provide a long enough stream of secondary ions, additional trenches 56 with different diameters, or a spiral trench 56, can be etched during additional revolutions of the sample. Once the analysis of the first monolayer is complete, deeper monolayers can be studied by etching material from existing trenches 56.

Moving the sample in the manner described above allows dynamic SIMS, with high ion current probe beams and therefore higher secondary beam signals, to serially collect data from single layers of the sample. Even for TOF systems, higher power pulses can be used, where the pulses etch a trench pattern to produce more secondary ions all from a single monolayer. Thus, higher ion currents can be used with dynamic SIMS to study single monolayers. If depth profiling is desired, motor 52 can be deactivated so

that the probe beam 46 etches sample material from a single spot, or the probe beam 46 can retrace the same pattern of trenches to etch deeper into the sample.

As stated above, the key to serial collection of data from constant sample depths (or even single monolayers) using a dynamic SIMS system is the constant movement of the target spot 47 along the sample surface during data acquisition to form trench 56. In Fig. 4, constant movement of the target spot 47 is performed by non-axis rotation of the sample. However, there are several ways of scanning the probe beam along the sample surface for constantly repositioning the target spot 47 and etching a predetermined trench pattern of specified depth onto the top surface of the sample during data acquisition. For example, the sample can be placed onto a translation stage 58 to move the sample relative to the probe beam 46, as illustrated in Fig. 6. Alternately, the probe beam 46 can be redirected by beam deflection devices, movable mirrors 60 (Fig. 7), or by moving or tilting the primary ion source 40 itself using an actuator, translation stage or motor 62 (Fig. 8). Lastly, any combination of ways for moving probe beam 46 relative to the sample listed above can be simultaneously or serially used to scan probe beam 46 along the surface of the sample.

Adverse edge effects are reduced by gathering data through trench edging under the present invention. Adverse edge effects result from etching deep into the sample where the edges of the hole or trench have a range of depth that contribute to the secondary beam signal. As data is acquired through etching of existing trenches, only the side walls contribute to adverse edge effects (side wall material ejecting secondary ions from layer depths not being currently analyzed). In contrast, when etching a crater with a stationary probe beam, the entire circumference of the crater wall contributes to edge effects.

Edge effects can be further reduced by rasterizing the trenches 56. Rasterizing includes moving the probe beam back and forth in a direction perpendicular to the trench length as the trench is being etched. The data

acquisition is then gated, meaning that data acquired when the beam is etching material away from the center of the trench is ignored, thus maximizing the amount of secondary ions produced from the bottom of the trench (which is presumably at a constant depth).

It is important to note that the present invention is not limited to examination of separate monolayers, but rather is ideal for analyzing material of a sample found at any desired depth within the sample. The formation of trenches under the present invention effectively limits data acquisition for materials existing at any predetermined depth in the sample because the trenches formed by continuously moving the sample and/or the probe beam have very stable fixed depths. Thus, re-etching of existing trenches provides a constant stream of ions from any fixed depth for depth profiling.

The present invention is not limited to secondary ion mass spectrometry, but includes any material analysis that uses a probe beam that etches or degrades the sample material over time while producing a secondary beam for analysis. Specifically, laser mass spectrometry uses a laser probe beam to etch material and produce secondary ions. Auger electron spectrometry and X-ray photo electron spectrometry use an electron probe beam to produce a secondary beam of electrons and/or protons that can be analyzed to determine sample material composition. The electrons in the probe beam embrittle and degrade the sample material, so it is beneficial to continuously move the probe beam over the sample to generate a constant stream of electrons and/or protons that properly reflect the properties of the sample.

It is to be understood that the present invention is not limited to the embodiments described above and illustrated herein, but encompasses any and all variations falling within the scope of the appended claims. For example, when translating the sample or moving the probe beam, the trench can be formed in any desired pattern, not just circular or linear. Further, the sample could also be a liquid.

What is claimed is:

1. An apparatus for analyzing a sample, comprising:

a particle source for producing a probe particle beam directed onto a surface of a sample to produce a secondary particle beam;

an analyzer that measures over a period of time a property of the secondary particle beam; and

a device for continuously moving at least one of the probe particle beam and the sample over the period of time so that the probe particle beam is continuously scanned along the surface of the sample over the period of time.

2. The apparatus of claim 1, wherein the probe particle beam etches a trench having a predetermined depth into the sample surface.

3. The apparatus of claim 1, wherein the property is at least one of a mass, an electrical charge and an energy of particles of the secondary particle beam.

4. The apparatus of claim 1, wherein the secondary particle beam comprises at least one of particles from the probe particle beam and particles from a material of the sample.

5. The apparatus of claim 2, wherein a current level of the probe particle beam and a rate of continuous movement between the probe particle beam and the sample are selected so that the predetermined depth corresponds to a depth of sample material to be analyzed.

6. The apparatus of claim 1, wherein the device is a motor that continuously rotates the sample over the period of time about an axis of rotation that does not correspond to a spot on the sample surface at which the probe particle beam is directed.

7. The apparatus of claim 1, wherein the device is a translation stage that continuously translates the sample over the period of time.

8. The apparatus of claim 1, wherein the device deflects the probe particle beam to continuously scan the probe particle beam across the surface of the sample over the period of time.

9. The apparatus of claim 1, wherein the device includes at least one of an actuator that tilts the particle source and a translator that translates the particle source to continuously scan the probe particle beam across the surface of the sample over the period of time.

10. An apparatus for analyzing a sample, comprising:

an ion source for producing a primary ion beam directed onto a surface of a sample, wherein the primary ion beam etches material from the sample surface to produce a secondary ion beam;

an analyzer that measures over a period of time at least one of a mass, an electrical charge and an energy of ions of the secondary ion beam; and

a device for continuously moving at least one of the primary ion beam and the sample over the period of time so that the primary ion beam etches a trench having a predetermined depth into the sample surface.

11. The apparatus of claim 10, wherein the secondary ion beam is at least one of ions from the primary ion beam that are scattered by the sample and ions from the etched material of the sample.

12. The apparatus of claim 10, wherein an ion current level of the primary ion beam and a rate of continuous movement between the primary ion beam and the sample are selected so that the predetermined depth corresponds to a depth of sample material to be analyzed.

13. The apparatus of claim 12, wherein the device is a motor that continuously rotates the sample over the period of time about an axis of rotation that does not correspond to a spot on the sample surface at which the primary ion beam is directed.

14. The apparatus of claim 12, wherein the device is a translation stage that continuously translates the sample over the period of time.

15. The apparatus of claim 12, wherein the device deflects the primary ion beam to continuously scan the primary ion beam across the surface of the sample over the period of time.

16. The apparatus of claim 12, wherein the device includes at least one of an actuator that tilts the ion source and a translator that translates the ion source to continuously scan the primary ion beam across the surface of the sample over the period of time.

17. The apparatus of claim 12, wherein the analyzer is a sector double focusing spectrometer that includes:

an electrostatic analyzer having a pair of plates chargeable to create an electrostatic field;

a magnet analyzer having a magnet for creating a magnetic field; and

a detector;

wherein the secondary ion beam is directed through the electrostatic analyzer and the magnet analyzer, and wherein the amplitudes of the electrostatic field and magnetic field are selected so that ions of the secondary ion beam having

a particular mass-to-charge ratio are directed to the detector while the other ions of the secondary ion beam are not directed to the detector.

18. The apparatus of claim 12, wherein the analyzer is a time of flight (TOF) spectrometer that includes:

a drift tube with one end that is open, and

a detector aligned with the other end,

wherein the secondary ion beam is pulsed and directing into the open end, and wherein the detector measures the time it takes the ions of the secondary ion beam to traverse the length of the drift tube.

19. The apparatus of claim 12, wherein the analyzer is a quadrupole mass spectrometer that includes:

a plurality of conductive rods extending parallel to and surrounding a central axis,

a detector aligned disposed along the axis, and

a power supply connected to the plurality of rods for applying voltages to the rods to create a quadrupole field along the axis,

wherein the secondary ion beam is directed along the central axis and through the quadrupole field such that ions of the secondary ion beam having a particular mass will pass through the quadrupole field and strike the detector, while the other ions of the secondary ion beam not having the particular mass will be drawn away from the central axis and not strike the detector.

20. The apparatus of claim 12, wherein the moving device oscillates the primary ion beam across a width of the trench as the trench is etched into the sample surface, and an output of the analyzer include only a portion of data collected from the analyzer measurements that corresponds to ions of the second ion beam that are produced from a center portion of the trench width.

21. A method of analyzing a sample, comprising the steps of:

producing a probe particle beam;

directing the probe particle beam onto a surface of a sample to produce a secondary particle beam;

measuring over a period of time a property of the secondary particle beam; and

moving one of the probe particle beam and the sample in a continuous manner over the period of time so that the probe particle beam is continuously scanned along the surface of the sample over the period of time.

22. The method of claim 21, wherein the probe particle beam etches a trench having a predetermined depth into the sample surface.

23. The method of claim 21, wherein the property is at least one of a mass, an electrical charge and an energy of particles of the secondary particle beam.

24. The method of claim 21, wherein the secondary particle beam comprises at least one of particles from the probe particle beam and particles from a material of the sample.

25. The method of claim 22 further comprising the step of:

selecting a current level of the probe particle beam and a rate of continuous movement between the probe particle beam and the sample so that the predetermined depth corresponds to a depth of sample material to be analyzed.

26. A method of analyzing a sample, comprising the steps of:

producing a primary ion beam;

directing the primary ion beam onto a surface of a sample, wherein the primary ion beam etches material from the sample surface to produce a secondary ion beam;

measuring over a period of time at least one of a mass, an electrical charge and an energy of ions in the secondary ion beam; and

moving one of the primary ion beam and the sample in a continuous manner over the period of time so that the primary ion beam etches a trench having a predetermined depth into the sample surface.

27. The method of claim 26, further comprising the step of:

selecting an ion current level of the primary ion beam and a rate of movement between the primary ion beam and the sample so that the predetermined depth corresponds to a depth of sample material to be analyzed.

28. The method of claim 27, wherein the moving step includes continuously rotating the sample over the period of time about an axis of rotation that does not correspond to a spot on the sample surface at which the primary ion beam is directed.

29. The method of claim 27, wherein the moving step includes continuously translating over the period of time at least one of the primary ion beam and the sample.

30. The method of claim 27, wherein the moving step includes continuously deflecting over the period of time the primary ion beam to scan the primary ion beam across the surface of the sample.

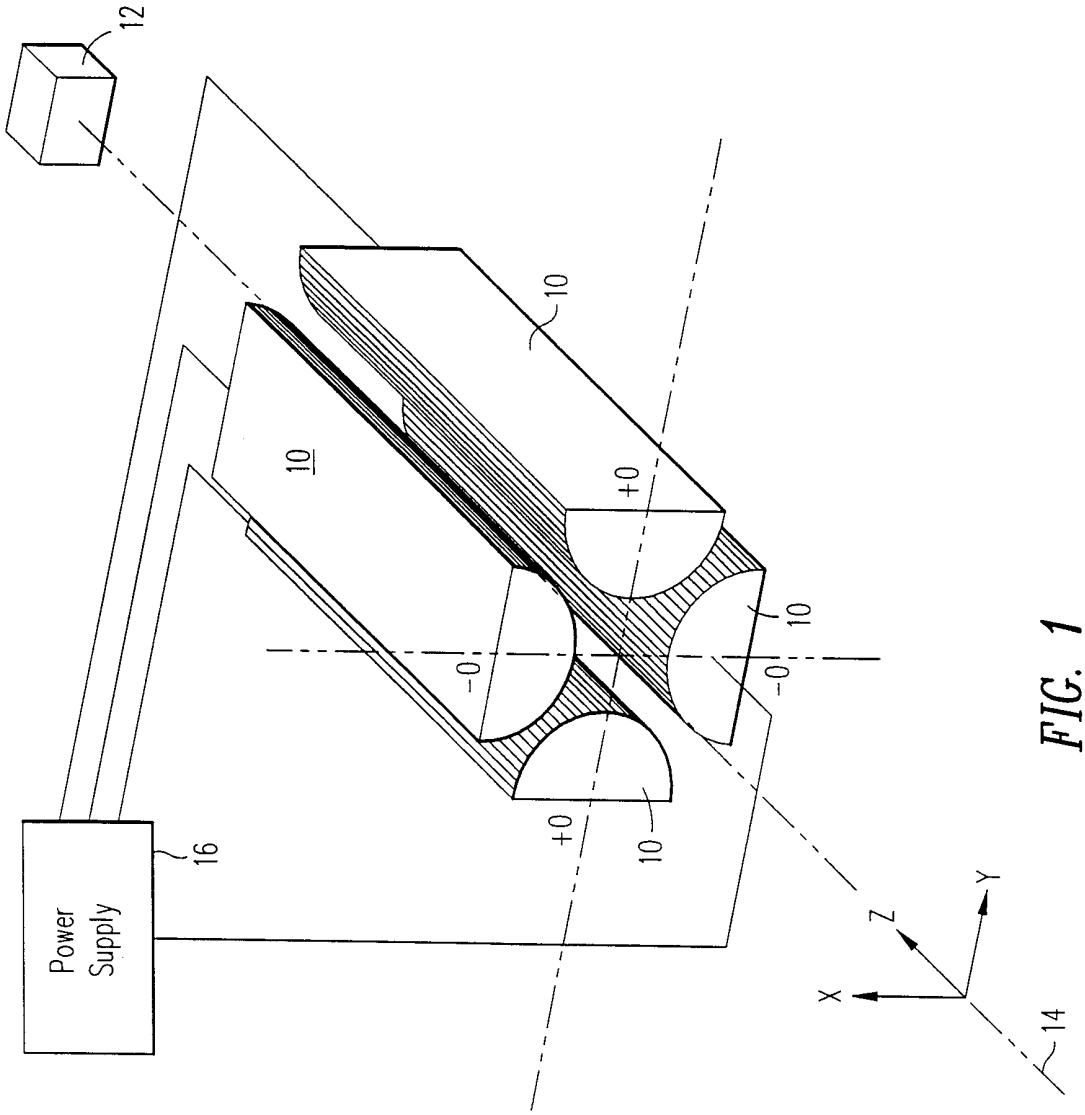


FIG. 1

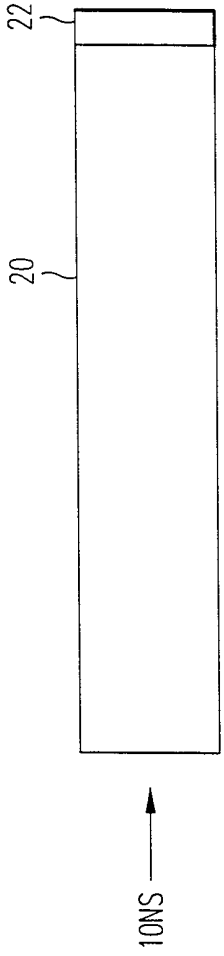


FIG. 2

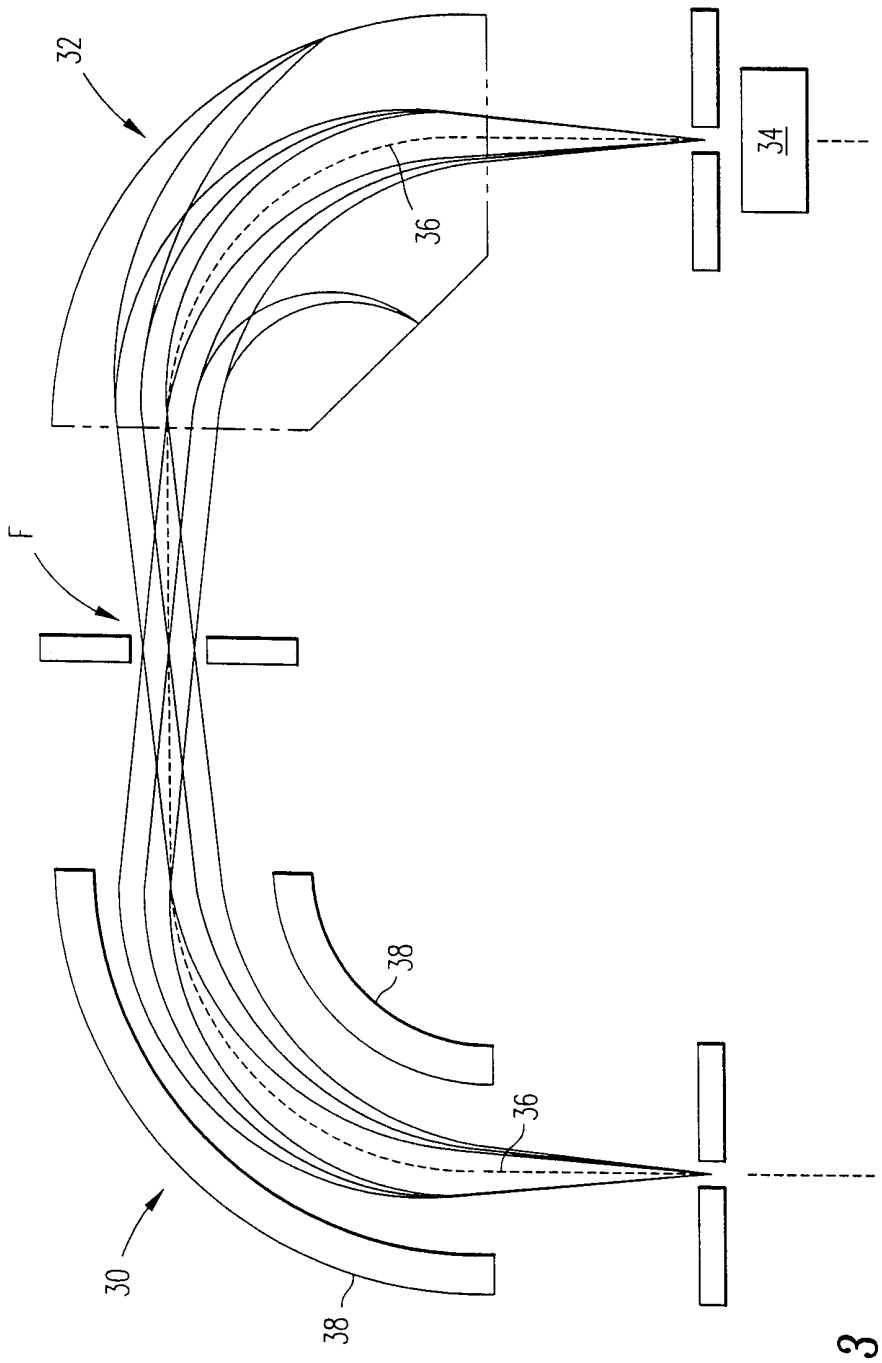


FIG. 3

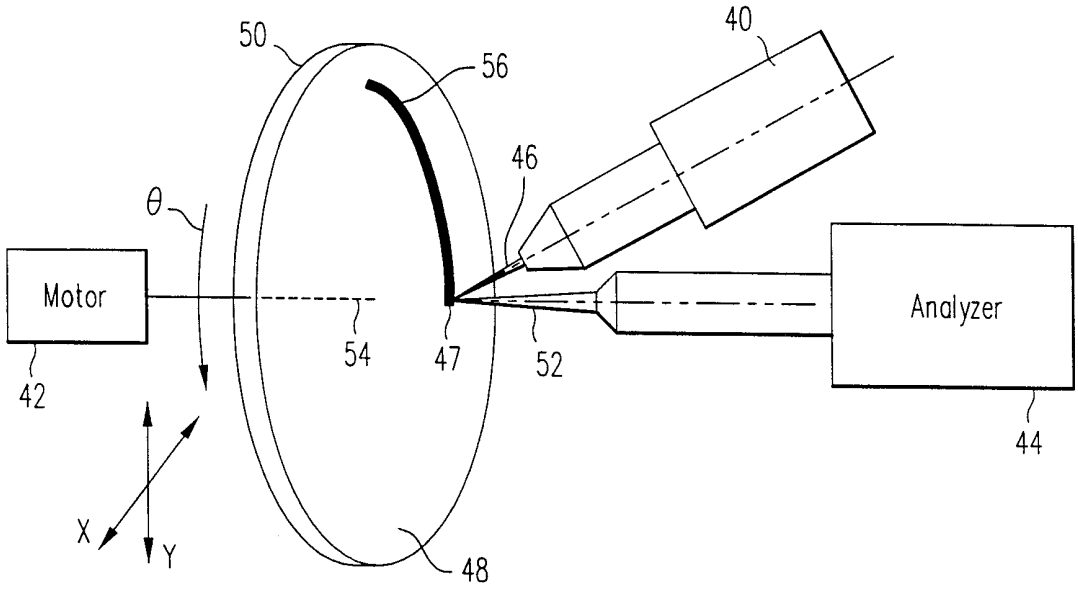

50
56
40
46
θ
Motor
Analyzer
42
54
47
52
44
X
Y
48

FIG. 4

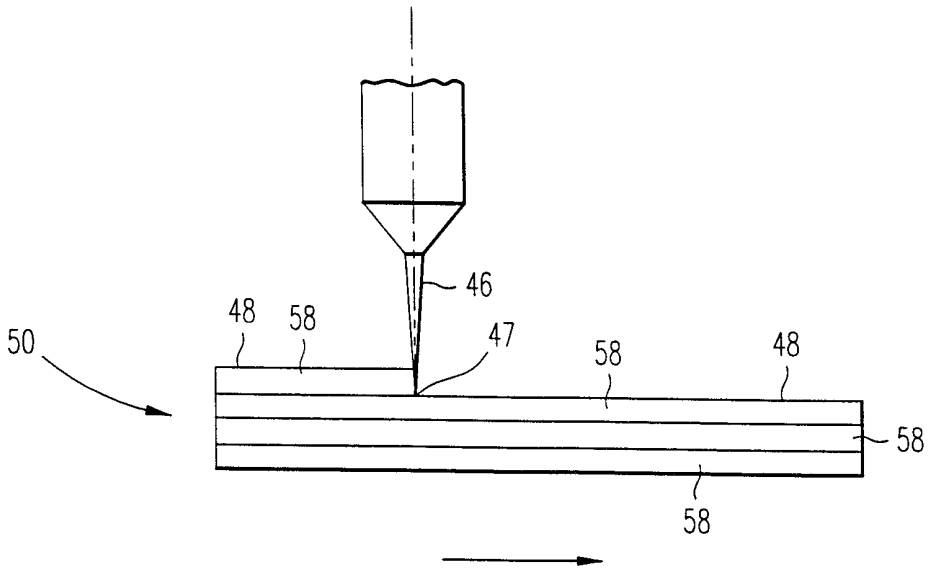

46
48
58
50
47
58
48
58
58

FIG. 5

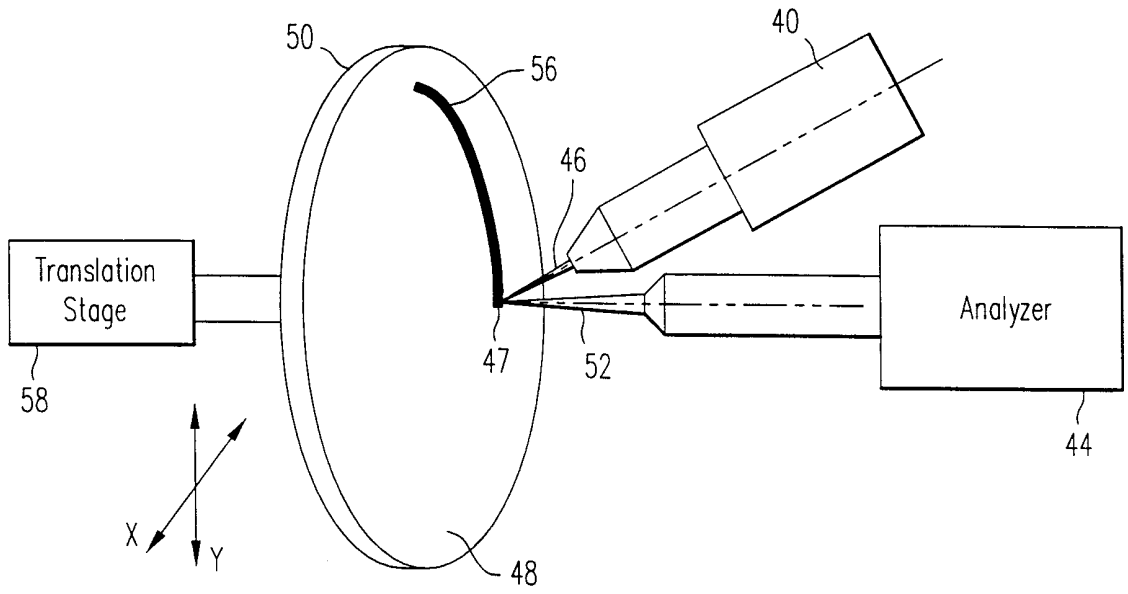

50
56
40
46
Translation Stage
58
47
52
Analyzer
44
X
Y
48

FIG. 6

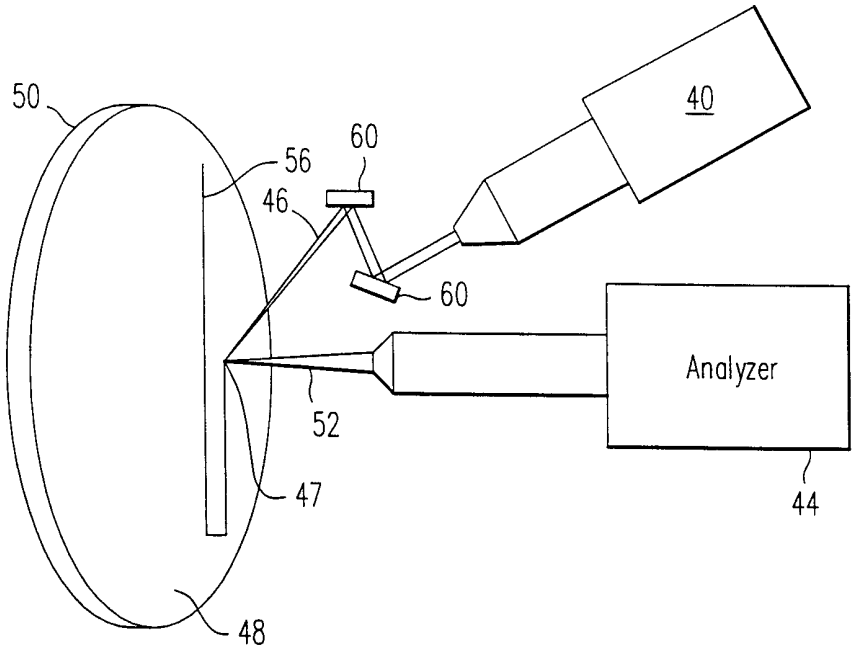

50
40
60
56
46
60
Analyzer
52
47
44
48

FIG. 7

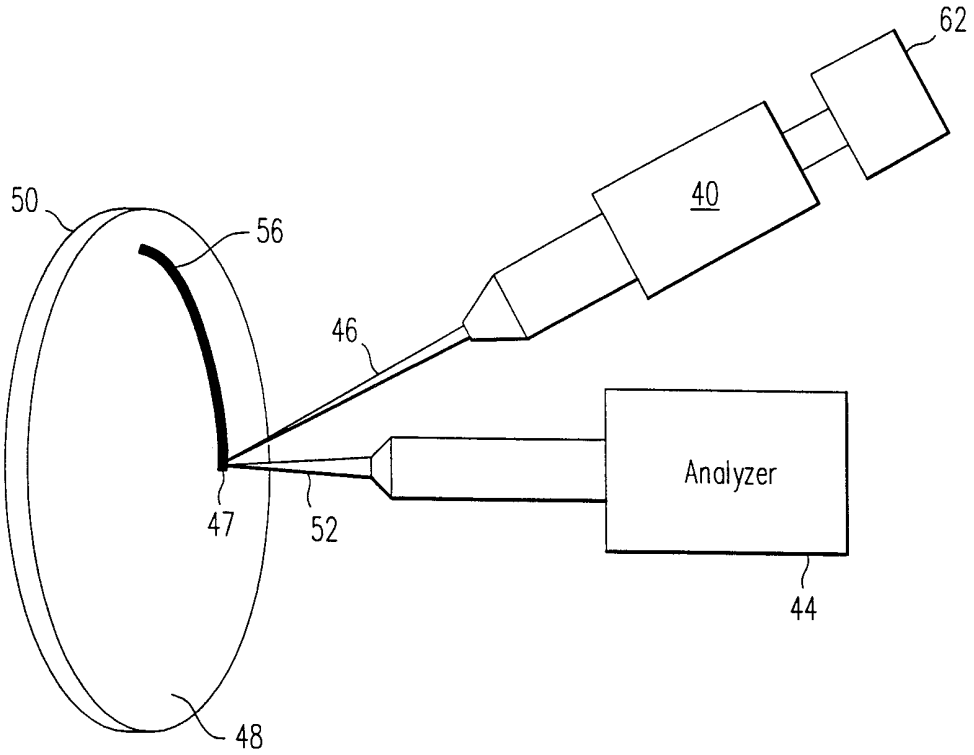


FIG. 8

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/19823

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :G01N 23/00; H01J 37/08
US CL : 250/309,307,397

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 250/309, 307, 397, 492.21

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,578,821 A (MEISBERGER ET. AL.) 26 NOVEMBER 1996 (26.11.96). SEE CLAIMS.	1-30
Y	US 4,874,946 A (KAZERSKI) 17 OCTOBER 1989 (17.10.89). SEE CLAIMS.	1-30
Y	US 4,983,831 A (MIGEON ET. AL.) 08 JANUARY 1991 (08.01.91). SEE FIGURES.	1-30
Y	US 4,510,387 A (IZUMI ET. AL.) 09 APRIL 1985 (09.04.85). SEE FIGURE 2.	1-30
A	US 4,912,325 A (VANDERVORST ET. AL.) 27 MARCH 1990 (27.03.90).	
A	US 4,939,364 A (ISHITANI ET. AL.) 03 JULY 1990 (03.07.90).	

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search 28 OCTOBER 1998	Date of mailing of the international search report 12 NOV 1998
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer BRUCE ANDERSON Telephone No. (703) 308-4851

Form PCT/ISA/210 (second sheet)(July 1992)★