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3868507

THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

Whereas, THERE HAS BEEN PRESENTED TO THE
Commissioner of Patents

A PETITION PRAYING FOR THE GRANT OF **LETTERS PATENT** FOR AN ALLEGED NEW AND USEFUL INVENTION THE TITLE AND DESCRIPTION OF WHICH ARE CONTAINED IN THE SPECIFICATION OF WHICH A COPY IS HEREUNTO ANNEXED AND MADE A PART HEREOF, AND THE VARIOUS REQUIREMENTS OF **LAW** IN SUCH CASES MADE AND PROVIDED HAVE BEEN COMPLIED WITH, AND THE TITLE THERETO IS, FROM THE RECORDS OF THE **PATENT OFFICE** IN THE CLAIMANT(S) INDICATED IN THE SAID COPY, AND WHEREAS, UPON DUE EXAMINATION MADE, THE SAID CLAIMANT(S) IS (ARE) ADJUDGED TO BE ENTITLED TO A **PATENT UNDER THE LAW.**

NOW, THEREFORE, THESE **Letters Patent** ARE TO GRANT UNTO THE SAID CLAIMANT(S) AND THE SUCCESSORS, HEIRS OR ASSIGNS OF THE SAID CLAIMANT(S) FOR THE TERM OF **SEVENTEEN** YEARS FROM THE DATE OF THIS GRANT, SUBJECT TO THE PAYMENT OF **ISSUE FEES** AS PROVIDED BY **LAW**, THE RIGHT TO EXCLUDE OTHERS FROM MAKING, USING OR SELLING THE SAID **INVENTION** THROUGHOUT THE **UNITED STATES.**



In testimony whereof I have hereunto set my hand and caused the seal of the Patent Office to be affixed at the City of Washington this twenty-fifth day of February in the year of our Lord one thousand nine hundred and seventy-five and of the Independence of the United States of America the one hundred and ninety-ninth.

Attest:
McG. M. Gibson, Jr.
Attesting Officer.

C. Marshall Dunn
Commissioner of Patents.

- [54] **FIELD DESORPTION SPECTROMETER**
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- [73] Assignee: **The United States of America as represented by the United States Atomic Energy Commission**, Washington, D.C.
- [22] Filed: **Dec. 5, 1973**
- [21] Appl. No.: **422,048**
- [52] U.S. Cl. **250/287, 250/227, 250/306**
- [51] Int. Cl. **H01j 39/34**
- [58] Field of Search **250/286, 287, 306, 307, 250/309**

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Attorney, Agent, or Firm—John A. Horan; Dudley W. King; Richard E. Constant

[57] **ABSTRACT**

A field desorption spectrometer which is capable of detecting and identifying one or more atoms of a specimen and/or all the atoms in an outer layer of the specimen or throughout the bulk of the specimen may comprise an apertured electrode for applying an electric field to field evaporate or field desorb ions from the specimen through the aperture, an apertured wall for blocking electric fields from the apertured electrode and specimen from a field free drift region and for transmission of the desorbed ions into the drift region, channel electron multiplier array means positioned in the drift region for intercepting of substantially all of said ions and for providing amplification of ion impacts by electron multiplication at locations corresponding with the locations where ions strike the array means, and means for sensing the multiplied electrons corresponding to these locations.

12 Claims, 8 Drawing Figures

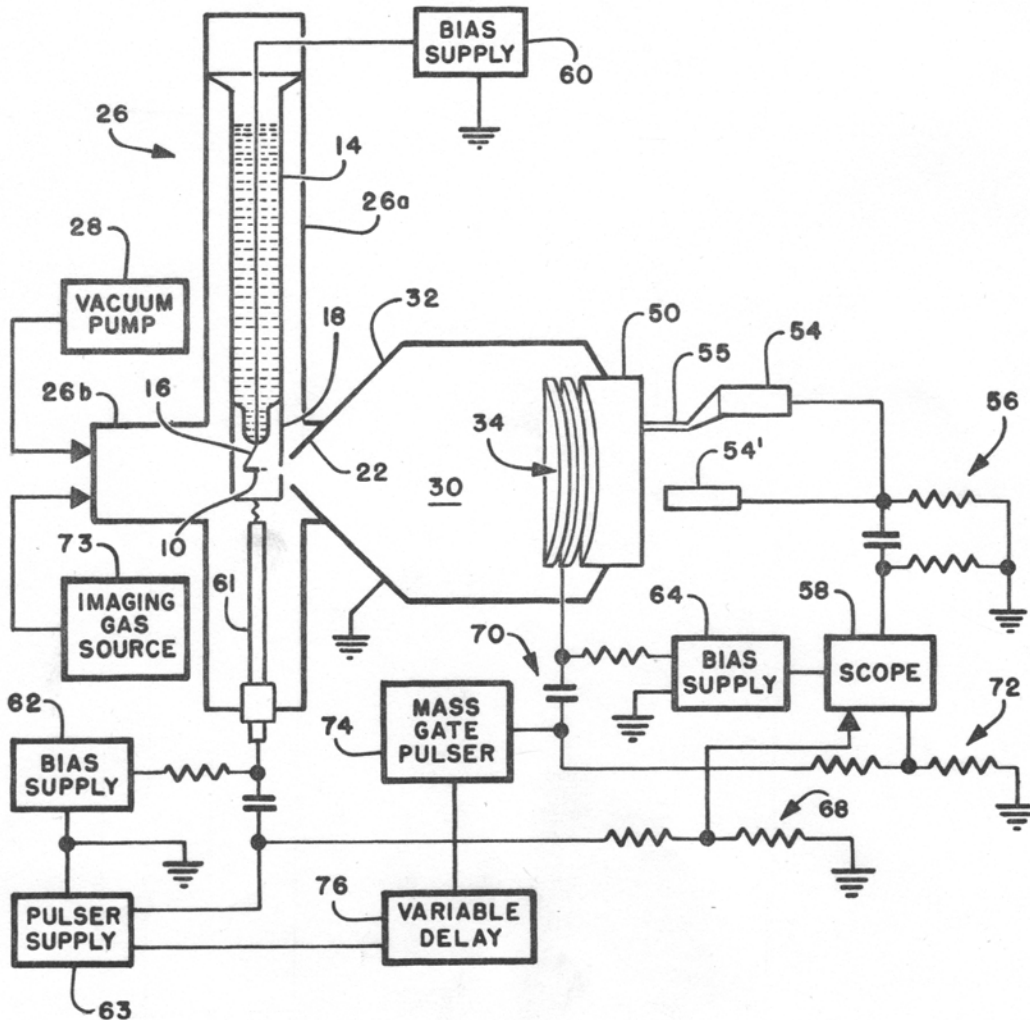


FIG. 1

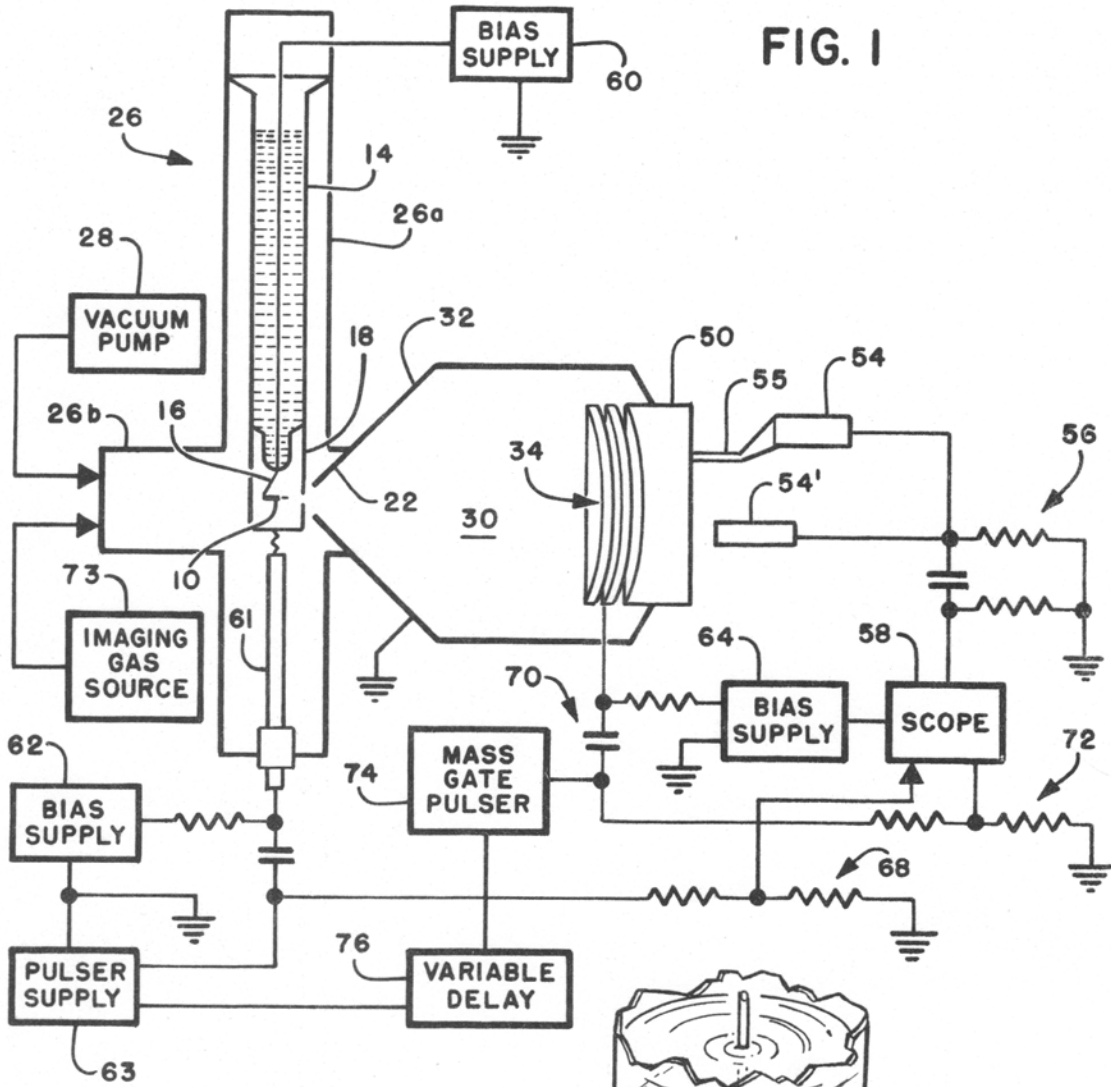


FIG. 2b

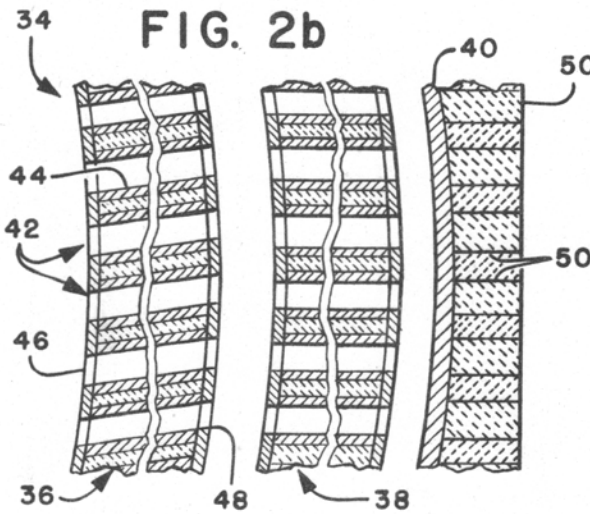
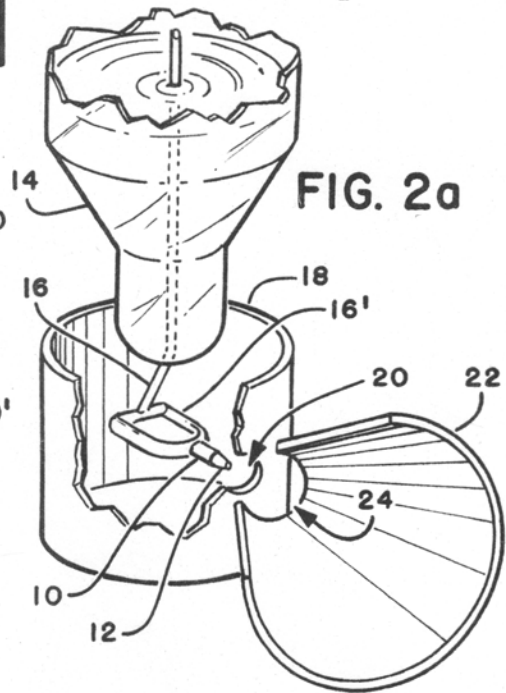
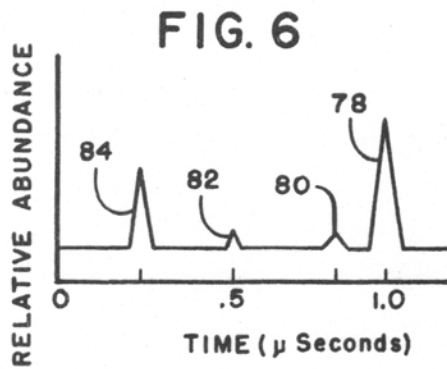
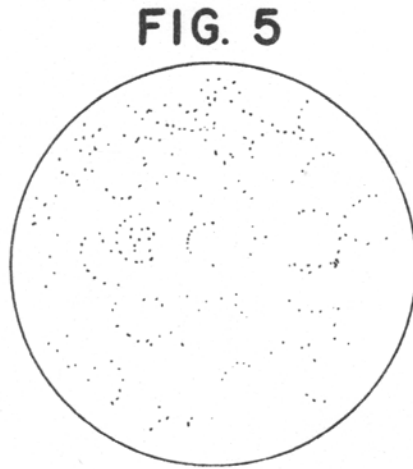
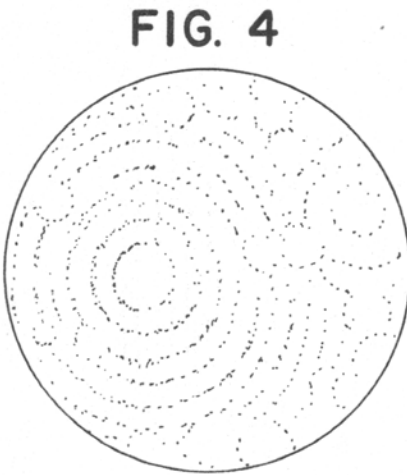
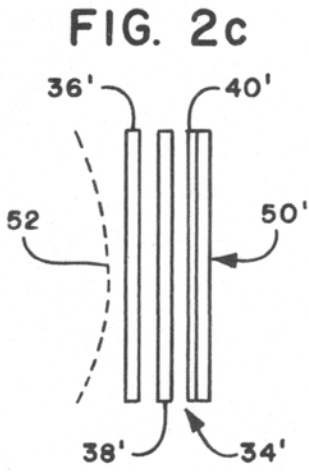


FIG. 2a





FIELD DESORPTION SPECTROMETER

BACKGROUND OF INVENTION

Attempts have been made to provide atom-by-atom identification of specimen surfaces using an apparatus sometimes referred to as an atom-probe field ion microscope. With this microscope, a "map" of the surface atoms is generally made using an ionized imaging gas to identify the position of atoms or an atom of interest. The specimen is then positioned so that the image of the surface atom of interest is aligned with an aperture or probe hole spaced some distance from the specimen, generally about 10 centimeters, and located on a phosphorescent screen, the aperture being equal in size to only one or at most a few atom images. The outer layer of atoms from the specimen would then be field evaporated or field desorbed from the specimen and the ion or ions resulting from the desired atom or atoms passed through the aperture or probe hole into either a drift-type mass spectrometer or a magnetic sector spectrometer which would then identify the atom species. The phosphorescent screen would prevent all other atoms from reaching the detector.

The passage of a preselected ion from the specimen through the "probe hole" may be difficult to achieve due to a difference in trajectory between the imaging gas atoms and the corresponding atom field desorbed from the surface. In addition, because of the small size of the specimen and the hole in the screen and the distance between them and between the screen and spectrometer detector, alignment between specimen and detector will be difficult. In addition, the specimen must be moved within a vacuum chamber to shift the image of its surface with respect to the probe hole so as to position the selected surface atom image over the probe hole, and to accomplish this while providing specimen cooling to cryogenic temperatures and high voltage and pulse voltage connection to the specimen.

These microscopes were generally of fairly large volume which required differential pumping of the specimen chamber or portion of the microscope and the spectrometer section together with the associated gauging and pumping apparatus.

In these previous instruments or microscopes, the ion kinetic energy was generally determined by the sum of a DC bias voltage and the amplitude and shape of a high voltage pulse. Since it is difficult or impossible to measure or terminate the pulse transmission line with its characteristic impedance at the specimen, the pulse shape and amplitude may be indeterminate and consequently the ion kinetic energy was uncertain. In order to provide reasonably reproducible results, a complicated calibration procedure was required. As the specimen shape is altered by the evaporation or desorption of atom layers, the DC bias or evaporation pulse or both had to be increased to maintain a constant evaporation field. This meant that the ion kinetic energy would change and a new calculation, and calibration, for each unknown species would have to be performed at each new value of applied voltage.

SUMMARY OF INVENTION

In view of the problems associated with the various atom-probe field ion microscopes and their inability to provide certain information, it is an object of this invention to provide a field desorption spectrometer which is capable of identifying one or more atoms at

one or more locations on the surface of the specimen and simultaneously providing a measurement of the number of atoms of each species over the entire surface or a portion of the specimen.

It is a still further object of this invention to provide a field desorption spectrometer which is also capable of providing a "map" of the location of every atom of a single species in each layer or layer edges of the specimen.

It is another object of this invention to provide a field desorption spectrometer which is capable of providing these measurements without movement of the specimen or alignment thereof with a particular location of the spectrometer detector.

It is a still further object of this invention to provide a field desorption spectrometer which provides ions of substantially constant kinetic energy at the detector portion of the spectrometer and thereby to eliminate the complicated and containing calibration procedures of previous atom probes.

It is a still further object of this invention to provide a field desorption spectrometer which is capable of providing these measurements in an instrument having a relatively small volume.

Various other objects and advantages will appear from the following description of the invention, and the most novel features will be particularly pointed out hereinafter in connection with the appended claims. It will be understood that various changes in the details, materials and arrangements of the parts, which are herein described and illustrated in order to explain the nature of the invention, may be made by those skilled in the art.

The invention comprises an ion source utilizing a triode-type electrode configuration which includes the specimen biased at a constant positive voltage, an electrode having an aperture positioned adjacent the specimen for passage of ions desorbed from the specimen by a negative voltage applied to the electrode, and a grounded wall having an aperture aligned with the specimen and electrode aperture for passage of the ions into a field free drift region beginning at said aperture and continuing to a channel electron multiplier array which is disposed in direct line of flight with ions from said specimen through said apertures.

DESCRIPTION OF DRAWING

The invention is illustrated in the accompanying drawings wherein:

FIG. 1 is a diagrammatic view of the field desorption spectrometer of this invention showing the relative locations and sizes of the various portions of the apparatus and the associated biasing and control circuits to provide the various modes of operation;

FIG. 2a is an enlarged partially cutaway, perspective view of the specimen and its associated electrode configuration;

FIG. 2b is an enlarged and somewhat diagrammatic, not to scale, cross section of a portion of the channel electron multiplier array and fiber optic bundle of FIG. 1;

FIG. 2c is a diagrammatic view of an alternate channel electron multiplier array which may be utilized in the spectrometer of FIG. 1;

FIG. 3 is a representation of a typical helium gas image of a tungsten specimen produced with the spectrometer of FIG. 1;

FIG. 4 is a representation of a typical image of the tungsten⁺³ ion species from the same tungsten specimen utilized in FIG. 3;

FIG. 5 is a representation of the hydrogen desorbed from the tungsten specimen of FIG. 3; and

FIG. 6 is a graph of the ion species and their quantities detected by the spectrometer of FIG. 1 during a typical evaporation event from a tungsten specimen.

DETAILED DESCRIPTION

The field desorption spectrometer of this invention is illustrated in FIG. 1 in somewhat diagrammatic and simplified form bringing out the relative positions, proportions and sizes of the respective elements of the spectrometer and typical electrical control and biasing apparatus to provide the desired modes of operation. The size, shape, and material of the specimen 10 (shown in greater detail in FIG. 2a) and the various power supplies are selected to provide an electric field of the order of magnitude of a few hundred million volts per centimeter at the surface of tip 12 of the specimen 10 resulting in field evaporation or field desorption of atoms from the surface of tip 12 and also to provide electric fields of the order of magnitude of about several hundred million volts per centimeter resulting in gas imaging of the surface atoms of tip 12. Generally, the specimen 10 is formed in the shape of a rod of about 0.1 millimeter in diameter comprised of a conical needle ending in an extremely sharp point and is made of a material to be studied and analyzed. The tip 12 may be formed with a generally hemispherical shape which is produced and otherwise finished by mechanical or electrochemical etching and polishing to dimensions well beyond the range of an optical microscope and the tip finally finished by field evaporation to an atomically smooth surface. In this condition, tip 12 provides atoms from its surface which are desorbed and magnified and whose relative positions are magnified to several million diameters by the spectrometer. From a so-formed specimen, the primary atoms of interest will be emitted in a generally conically diverging beam centered on the longitudinal axis of the specimen 10 and having a conical half angle of from about 40° to 45°. It will be appreciated that as the radius of curvature of the tip 12 increases, the electric field density will decrease and may reduce the field strength below the level at which desorption may take place. The ions leaving the tip 12 may follow very closely along lines of radius from the tip 12, if maintained in a field free environment, and may provide a resolution of about 1 to 4 Angstroms and several million magnification.

Because of the geometry of specimen 10 comprising tip 12, the practical limit of the ion beam dimensions is near or below the recited conical half angle above. In order to maintain the evaporation voltage at as low a level as practical and to eliminate electrical breakdown and other considerations, the specimen radius of curvature at the areas where evaporation is to occur should be maintained at below about 1,000 Angstroms. It has also been found that it is desirable that the specimen 10 be maintained at cryogenic temperatures, such as below about 78° to 21° Kelvin, by use of liquid gases like nitrogen or neon, to enhance the image formed with the desorbed and ionized atoms and from gas imaging ions. In order to prevent interference from other materials and gases, it is preferred that the field desorp-

tion and ion magnification be carried out in an evacuated atmosphere of from about 10⁻⁶ to 10⁻¹⁰ Torr.

The specimen 10 to be examined may be mounted at the base of a glass or ceramic "cold finger" 14 at the end of an electrically conductive support element 16. The support element 16 may include one or more conductors carried by and within the cold finger 14 and sealed therein in an appropriate manner which may terminate with a generally U-shaped support 16' and the specimen 10. The cold finger 14 may be partially filled with a liquid gas to maintain the specimen 10 through the support member 16 at cryogenic temperatures. A first electrode 18 having an aperture or passageway 20 aligned with the specimen 10 and the ion beam evolved therefrom is positioned at a location adjacent specimen 10 and tip 12 which will provide the desired field desorption electric field to tip 12. The aperture 20 dimension is selected to be sufficiently large so as not to impede or block the diverging ion beam from tip 12. The spacing between tip 12 and electrode 18 may be typically about 1 millimeter while the aperture 20 may be about 2 millimeters in diameter. The electrode 18 may be in the hollow tubular shape shown or in a planar or spherical shape depending upon the desired means of imaging and positioning of the electrode. An additional electrode or wall 22 may then be positioned adjacent electrode 18 with electrode 18 intermediate tip 12 and electrode 22. The electrode 22 is provided with an aperture or opening 24 which is aligned and symmetrical with the longitudinal axis of specimen 10 and of sufficient diameter so as to minimize blocking or impeding of the ion beam diverging from tip 12. Even though the aperture 24 should generally be made of sufficient diameter to be outside the desired diverging ion beam, its diameter should be small enough to act to contain the electric field produced by electrode 18 and should thus preferably be just slightly greater than the diameter of the desired ion beam. Additional shielding may be achieved by placing a grid across aperture 24, however, with some consequent degradation of the ion beam. The apertures 20 and 24 may form a diverging conical path from specimen 10 with a conical half angle of from about 15° to about 45°.

The specimen 10, and electrodes 18 and 22 form a triode-type ion source section of the field desorption spectrometer and may be supported within an appropriate vacuum chamber 26 which may be sealed and evacuated to a desired low pressure. Vacuum chamber 26, in the area adjacent to and surrounding the ion source section, should be formed from or include electrically conductive wall portions which may be electrically grounded to act as a shield for the ion source section. The various elements and conductive feed-throughs to the ion source section may be suitably mounted and sealed to maintain the desired vacuum levels through a suitable vacuum pump 28. By way of example, the vacuum chamber 26 may be formed from intersecting metal tubular sections 26a and 26b, as shown, with the cold finger 14 and ion source section supported in tubular section 26a and the vacuum pump 28 and ion detector section, to be described, supported in tubular section 26b.

The ion detector section of the field desorption spectrometer may include an ion drift region 30 which is maintained in a substantially electric and magnetic field free condition by the non-magnetic conductive wall 32, a part of which forms electrode 22. The wall

32 may expand conically or in any other appropriate manner from the aperture 24 of electrode 22 to a diameter larger than the desired magnified diameter of the ion beam. A channel electron multiplier array 34, as shown for purpose of illustration in greatly expanded cross section in FIG. 2b, is positioned at the end of the drift region 30 within walls 32 at a location where the ion beam reaches the desired magnification level. The diameter of the channel electron multiplier array may typically be about 8 centimeters and be spaced about 10 centimeters from tip 12 of specimen 10. The channel electron multiplier array 34 may be provided with a curvature having its center of radius at tip 12 to provide equal or substantially equal travel distances for ions evolved from tip 12 to the array 34.

The channel electron multiplier array 34 may be made up of one or more channel plates, for example channel plates 36 and 38 in FIG. 2b, and a luminescent or phosphorescent screen, such as phosphor screen 40, disposed adjacent the channel plates on the side opposite to the ion source section of the spectrometer. The channel plates are formed of a multiplicity of parallel circular passageways arranged in a honeycomb-like array and are often formed from bundles of fused optical fibers which have been partially etched away. Each of the passageways, for example passageways 42 in channel plate 36 of FIG. 2b, are formed with a length equal to about 40 times their diameters and may typically be about 37 micrometers in diameter with a distance between passageway centers of about 50 micrometers. The inside surface of each passageway is coated with a semi-insulating layer having a resistance typically of about 10^8 ohms, such as layer 44, while the outer surfaces of the plates are coated with a conductive layer, such as layers 46 and 48 for channel plates 36, to effectively connect all the passageways in a channel plate in electrical parallel, each passageway functioning as a distributive dynode multiplier when an appropriate bias is applied between the conductive layers 46 and 48. The passageways are preferably positioned at an angle, such as about 15° , with respect to a particle or ion which is traveling from tip 12 of specimen 10 so as to make the channel plates effectively opaque to ions striking the plates at normal incidence. The second channel plate 38 has its passageways positioned at about normal incidence or an appropriate angle so that it is effectively opaque to the electrons produced in the first plate 36. When an ion enters a passageway of the first channel plate 36, secondary electrons are emitted from the semi-insulating layer 44 surface which in turn strike the inner wall producing further secondary electrons. This process may be repeated many times along the passageway with many electrons emerging from the far end of the passageway. These electrons may be accelerated to the next channel plate by the bias applied to its surface layer and enter an adjacent passageway thereof and repeat this secondary electron multiplication. With appropriate potentials applied to the respective outer surface layers of the channel plates beginning at ground potential at layer 46, gains of 10^6 and more may be achieved. The electrons exiting from the passageway of the second channel plate 38 may be accelerated against the phosphor screen 40 by a bias applied thereto produce light images at these locations which thus correspond with the magnified ion image evolved from tip 12 of specimen 10.

The respective conductive layers on the outer surfaces of channel plates 36 and 38 may be formed from appropriate deposition of such as gold while the resistance layers in the passageways may be formed of lead or the like by heat treatment of the channel plates.

A fiber optic bundle 50, formed from a multiplicity of optical fibers (shown as fibers 50') fused together to form an array having outer dimensions similar to that of the channel electron multiplier array 34, or a glass plate or other transparent window, may be positioned adjacent to the array 34 through which phosphor screen 40, which may be deposited or settled on its surface facing array 34, may be viewed. The fiber optic bundle 50 may form the outer wall of the vacuum chamber 26 at the terminal end of the drift region 30. The optical fibers may be individually stacked to form the bundle 50 with the interstices filled with appropriate bonding material and the entire bundle then heated to a temperature to fuse this bonding material to the fibers to form a solid, gas impervious structure which leaves the integrity of the individual fibers intact. The surface of the fiber bundle 50 adjacent to the channel electron multiplier array 34 may be spherically curved to correspond with the spherical curvature of array 34 to minimize distortion of the image on the phosphor screen 40 from the channel plates. It should be noted that in most applications, the fibers may typically have a cross section of from about 20 to 40 micrometers so that more than one fiber of the fiber bundles 50 may view the light emissions caused or produced through one or several of the passageways of the channel plate and may provide collimation and relatively distortion free viewing of the image on phosphor screen 40.

As mentioned previously, the channel electron multiplier array 34 may be shaped with a spherical curvature having a radius centered at tip 12 of specimen 10 to insure equal or approximately equal travel distances for all ions evolved from tip 12. The channel plates may also be formed with a planar configuration, such as shown by channel plates 36' and 38' and phosphor screen 40' settled on glass or fiber plate 50' of array 34' in FIG. 2c, and a high transmission, mesh or grid 52 having a spherical radius of curvature centered at tip 12 placed in front of the array 34'. With appropriate bias applied to the grid 52 and the array 34', such as by grounding the grid 52 and applying a negative voltage to the first conductive layer of channel plates 36', the difference in travel time of an ion located towards the center of the array 34' compared to that of an identical ion towards the periphery of the array 34' may be minimized.

The phosphor screen 40 or 40' may be formed in a conventional manner with one or more types of phosphorescent or fluorescent material deposited or settled on a conductive layer or forming a part thereof. For example, a high speed phosphorescent material having a low or short residual luminescence may be utilized where differentiation between species having very similar or close travel times is desired provided the apparatus utilized to sense the phosphorescence light image of screen 40 has sufficient speed to measure or detect this image. A slower, longer residual luminescent material may be utilized where the detecting or sensing apparatus requires longer periods of time to record the image. In some applications, a mixture of two or more luminescent materials may provide a compromise for combining of these functions.

The light image on screen 40 or 40' may be sensed through the fiber optic bundle 50 by use of a light sensing or detecting apparatus such as a camera to obtain an image of the entire phosphor screen 40 or 40' simultaneously on a photographic plate, by use of a photosensitive device such as a photomultiplier like photomultiplier 54 having a sensitivity at the particular luminescence wavelength of phosphor screen 40 or 40' or by use of a photomultiplier having similar spectral response. The photomultiplier 54 may be provided with a fiber optic 55 or similar light collimating structure for isolating those fibers of bundle 50 producing a single luminescence spot at a particular location of screen 40. It will be apparent that with the latter arrangement two or more photomultipliers with or without light collimators, such as photomultiplier 54', may be utilized to measure the luminescence simultaneously at more than one location of screen 40. Apparatus may be provided to move either the photomultiplier or any fiber optics associated therewith from location to location about the outer surface of the fiber optic bundle 50 for these measurements. The signal generated by photomultiplier 54 and 54' may be coupled through an appropriate differentiating circuit 56 to an oscilloscope 58 or other recording means. It is also apparent that by use of appropriate optical mechanisms, such as beam splitters and the like and electrically controllable shutters, that more than one optical sensing means may be utilized simultaneously or in sequence to sense the information and images produced by the channel electron multiplier array 34, including image storage tubes or image dissecting tubes, vidicon tubes and photodiode or the like arrays or combinations of one or more cameras with one or more photomultiplier devices viewing the fiber optic bundle 50 simultaneously or in controlled sequences.

As mentioned previously, the specimen, through support element 16, may be biased to a constant positive voltage to give the ions evolved from specimen 10 a predetermined kinetic energy. The voltage bias should be sufficiently large to assure that external magnetic fields do not affect the trajectories of the ions. For the size drift region of the present spectrometer, this may be at voltages somewhat greater than 50 volts. The voltage bias should be at a level which insures detectable and reliable travel times for each ion species, and may be generally from about 2 to 4 kilovolts and preferably less than one kilovolt. This bias voltage may be applied via element 16 by the bias supply 60. The travel time of a given ion species may be determined with sufficient accuracy by the equation:

$$T = d[(m/n)/(0.193 V_A)]^{1/2},$$

where T is in microseconds, d is the distance between tip 12 and the channel electron multiplier array 34 in meters, m/n is in amu and is the charge to mass ratio of the ion species, and V_A is in kilovolts. The electrode 18 may be held at a negative potential sufficient to establish the required imaging field by means of a suitable feed-through conductor 61 and bias supply 62. The electrode 18 voltage bias may typically be from about 0 to -20 kilovolts. If a high voltage pulse of sufficient amplitude to field desorb an atomic layer from tip 12 of specimen, such as from about 0 to -20 kilovolts, is applied to electrode 18 from pulser supply 63 through an appropriate pulse forming network, an atomic layer may be desorbed from the tip 12 surface and ionized.

The ions may travel from tip 12 through apertures 20 and 24 into the drift region 30 and strike channel electron multiplier array 34. Between the tip 12 and electrode 18, the ions are accelerated and between electrode 18 and wall 22 they are decelerated so that the ion kinetic energy within the field-free drift region 30 is determined solely by the magnitude of the positive bias voltage from bias supply 60. With the aperture 24 at the size indicated, the ions will reach this kinetic energy level determined by the bias supply 60 directly adjacent to the aperture 24 regardless of the amplitude of the bias or pulse at electrode 18 so that identical ions will travel for the same time at each evaporation pulse. If it is desired, temperature dependence or other effects may be measured by supplementally heating specimen 10 during the desorption pulse by use of a laser, electrical resistance heating by additional leads coupled to specimen 10, by heated fluid inserted in cold finger 14, or otherwise.

The oscilloscope 58 sweep may be initiated by sampling the pulser supply 63 via the voltage divider 68 or other suitable means. The phosphor screen 40 and the channel electron multiplier array 34 may be suitably biased through bias supply 64. Screen 40 may be coupled through network 70 and voltage divider 72 to the oscilloscope 58 so that the travel times of all ions striking the channel electron multiplier array 30 may be measured from the time of the desorption pulse from pulser 63. All of the ions produced by the desorption event may be monitored at one time in this manner to provide a rapid evaluation of species concentration and identity over the entire surface as well as the identification of these species at various separate locations and their quantity. It will be apparent that these measurements can be made at the same time using separate oscilloscopes or multiple traces on a single oscilloscope.

If it is desired to make an atom-by-atom analysis of one or more locations on tip 12 using the photomultiplier 54 and a fiber optic coupler 55 between the fiber optic bundle 50 and photomultiplier 54, a suitable imaging gas may be introduced through imaging gas source 73 into the vacuum chamber 26 to a pressure of about 10⁻⁶ Torr to produce a conventional ion image of the specimen surface on phosphor screen 40 of the channel electron multiplier array 34. The suitably apertured photomultiplier 54 may then be placed over a selected image spot through the fiber optic bundle 50. The imaging gas may then be pumped from the chamber 26 and an atomic layer desorbed from specimen tip 12 by the pulse from pulser 63. By differentiating the output of the photomultiplier 54 and feeding its signal to the oscilloscope 58, which was triggered by the desorption pulse from pulser 63, the travel time of the ion desorbed from the tip 12 at this location may produce luminescence on the phosphor screen 40 at a time after desorption determined by the ion species and opposite the apertured photomultiplier so that the atom at that single image spot may be analyzed and identified. As noted above, this analysis may be modified by the use of several apertured photomultipliers to provide atom-by-atom analysis at several different locations simultaneously.

The pulser supply 63 may be a pulsed generator capable of providing a pulse amplitude of from about 0 to -20 kilovolts with a pulse width of about 20 nanoseconds or less and a rise time of less than about 1 nanosecond. Pulser supply 63 may be manually or au-

tomatically triggered to supply single pulses, as required. Since changes in pulse amplitude or duration only affect the evaporation field and not the ion energy in the drift region 30, carefully terminated pulse lines may no longer be required for successful operation. The bias supply 60 may be an ultra-stable power supply with about 0.001 percent regulation and a ripple of less than about 200 milivolts. As the desorption from the tip 12 of specimen 10 occurs, the radius of curvature of the tip may change from evaporation pulse to evaporation pulse and require some change in the amplitude of the evaporation pulse from pulser supply 63 or bias supply 62 or a combination thereof, however with no effect on the ion kinetic energy of the ions entering the drift region 30. Also, as the radius of curvature of tip 12 changes from evaporation pulse to evaporation pulse, any shifting of the image at the channel electron multiplier array 34 may be compensated for merely by movement of the photomultiplier 54 and its fiber optics, or other light detector, external to the vacuum system.

The respective elements of the channel electron multiplier array 34 may be biased by suitable bias supplies similar to bias supply 64 which is shown in simplified form for purpose of illustration without illustrating the different connections to each element of array 34 through appropriate voltage dividers or separate bias supplies to achieve the desired electron multiplication and ion detection. For example, the surface layer 46 of channel plate 36 will normally be grounded while the back layer 48 is biased at about 1 kilovolt with the front layer of channel plate 38 biased at about 1 kilovolt to 1.4 kilovolts and the back layer at about 2 kilovolts with screen 40 at about 7 kilovolts.

If it is desired to record all the ions of a single species, e.g., provide an image at phosphor screen 40 of this ion species only, the back layer of channel plate 38 may be biased at a direct current potential of about 1,200 volts by bias supply 64 and the layer pulsed by a positive pulse of about 800 volts from mass gate pulser 74 at the desired time period after the evaporation pulse is generated by pulser supply 63, as determined by a variable delay circuit 76. Thus, the channel electron multiplier array 34 may be gated on only during the arrival time of the desired ion species and the resulting electrons produced therefrom by the array 34 reach the back surface layer of channel plates 38 to produce an image on phosphor screen 40 of only these ions. If the mass gate pulser 74 voltage pulse is terminated before any other ion species may arrive, the image will be of only the desired ion species. In order to optimize viewing and photographing of the gated image, a long decay time phosphor may be desirable, but since the arrival of the ions at the channel electron multiplier array 34 must be accurately determined, a short rise time is also required. These two diverse conditions may be satisfied by utilizing a mixture of phosphors on phosphor screen 40.

FIG. 3 illustrates a typical helium ion image of a tungsten specimen which may be utilized to obtain the crystallographic orientation of the specimen 10 tip 12. FIG. 4 illustrates the image of the tungsten⁺³ ion species from a tungsten specimen using the mass gate pulser 74 and time delay circuit 76. FIG. 5 illustrates an image formed from hydrogen ion species using mass gate pulser 74 and a different time delay by delay circuit 76. FIG. 6 illustrates a typical spectrometer record of the different ion species evolved from a tungsten specimen

illustrating by peak 78 the quantity of tungsten⁺³ ion species, by peak 80 the quantity of tungsten⁺⁴ ion species, by peak 82 the quantity of oxygen ion species and by peak 84 the quantity of helium ion species in a single evaporation pulse as detected from the signals produced on phosphor screen 40 and coupled to scope 58 by network 70 and voltage divider 72. For example, the peak 78 and the peak 80 indicates that there is about 10 times as many tungsten⁺³ ion species as tungsten⁺⁴ ion species produced during the evaporation pulse at 78°K. It will be apparent that the spectrometer record illustrated in FIG. 6 may be provided by any electron multiplier arrangement other than and including the channel electron multiplier array described.

Since the ion energy is determined solely by the direct current bias supply to specimen 10 and is not affected by the amplitude or duration of the desorption pulse, the field desorption spectrometer described does not require extremely precise pulse characteristics. With this field desorption spectrometer, various measurements and determinations may be made of the specimen and its makeup as described previously without affecting or entering the vacuum chamber 26. The combined volume of both the ion source section and detector section of this spectrometer may be about 1 liter.

What is claimed is:

1. A spectrometer for detecting and identifying ions in a diverging generally conical ion beam field desorbed from a specimen surface, comprising a vacuum chamber having electrically conductive walls surrounding an ion source section and a detector section; said ion source section including means for supporting said specimen, means for coupling a constant voltage to said specimen through said supporting means, an electrode adjacent said specimen having an aperture aligned therewith for emission and conical definition of ions from said specimen, and means for coupling an ion desorption field to said specimen through said electrode opposite in polarity from said constant voltage for thus providing a beam of ions from said specimen surface diverging in a cone-shape through said aperture; an electrically conductive wall portion intermediate said ion source section and said detector section and spaced from said electrode, said conductive wall portion having an aperture aligned with said specimen and said first mentioned aperture of size no less than that of said first mentioned aperture and no greater than outer margins of said ion beam as defined by said first mentioned aperture; said detector section including a channel electron multiplier array means disposed in registry with each of said apertures and said ion beam as defined thereby and impervious to direct passage of ions in said ion beam for producing and multiplying electrons in response to said ions at locations where said ions strike said array, said array means being spaced equidistant from said specimen for all paths of travel of ions in said ion beam, phosphor means for sensing means multiplied electrons adjacent said locations, said array means having sufficient gain to detect ions from said specimen surface, and means responsive to said sensing means for identifying ion species in said ion beam; means for coupling said chamber to a vacuum pump; and means for connecting said chamber walls and said electrically conductive wall portion to ground potential to provide shielding for said ions and an ion drift region between said electrically conductive wall

portion and said channel electron multiplier array means.

2. The spectrometer of claim 1 wherein said identifying means includes variable delay means for gating said channel electron multiplier array means on at a time commensurate with the arrival of a particular ion species from said specimen surface for sensing of said particular ion species by said sensing means.

3. The spectrometer of claim 1 wherein said identifying means includes an oscilloscope and means for initiating the sweep of said oscilloscope by said ion desorption field.

4. The spectrometer of claim 1 wherein said identifying means includes a photosensitive device external to said vacuum chamber.

5. The spectrometer of claim 1 wherein said channel electron multiplier array means includes a plurality of adjacent, generally coextensive channel plates facing and aligned with said apertures and said ion beam, each having a generally spherical radius of curvature centered at said specimen for providing substantially equal travel distances for each ion from said specimen surface to said plates.

6. The spectrometer of claim 1 wherein said channel electron multiplier array means includes a plurality of adjacent, generally coextensive planar channel plates facing and aligned with said apertures and said ion beam, and a generally spherical grid electrode disposed between said channel plates and said specimen with a

radius of curvature centered at said specimen for providing substantially equal travel distances for each ion from said specimen surface to said array means.

7. The spectrometer of claim 1 wherein said sensing means includes an array of optical fibers on which is settled a phosphorescent screen to provide optical coupling between said channel electron multiplier array means and the exterior of said vacuum chamber.

8. The spectrometer of claim 7 wherein said optical fiber array includes a generally spherical surface adjacent said channel electron multiplier array with a radius of curvature centered at said specimen.

9. The spectrometer of claim 7 including means for sensing the locations of ions at said channel electron multiplier array means through said fiber optic array.

10. The spectrometer of claim 7 including means for detecting the location of an ion at said channel electron multiplier array means through said fiber optic bundle.

11. The spectrometer of claim 1 wherein said apertures are circular and form a diverging conical path from said specimen with a conical half angle of from about 15° to 45°.

12. The spectrometer of claim 11 wherein said electrode is spaced from said specimen a distance of about 1 millimeter and from said apertured wall a distance greater than the voltage breakdown distance therebetween and less than about 2 millimeters.

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