

[54] **ELECTRON MULTIPLIER-ION DETECTOR SYSTEM**

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[51] Int. Cl.² **B01D 59/44; G01T 1/20; H01J 39/34**

[58] Field of Search **250/368, 369, 281, 282, 250/283, 298, 299, 300, 213 UT, 489**

[56] **References Cited**

UNITED STATES PATENTS

3,510,647 5/1970 Wood 250/281

3,602,709 8/1971 Hull 250/283
3,774,028 11/1973 Daly 250/299 X

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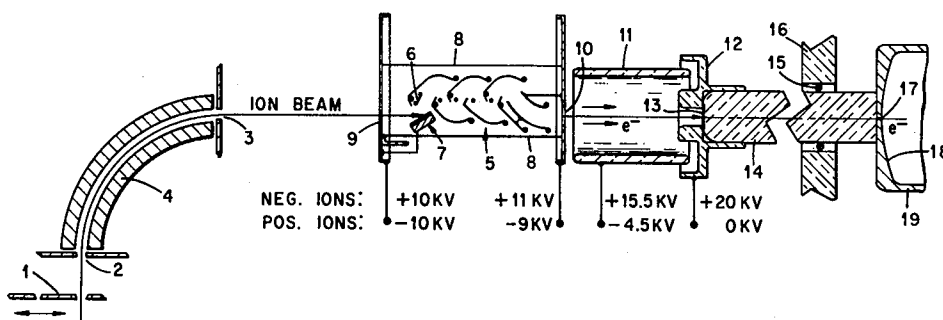
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[57] ABSTRACT

The invention relates to an improved ion detector for use in mass spectrometers for pulse counting single ions which may have a positive or a negative charge. The invention combines a novel electron multiplier with a scintillator type of ion detector. It is a high vacuum, high voltage device intended for use in ion microprobe mass spectrometers.

6 Claims, 2 Drawing Figures



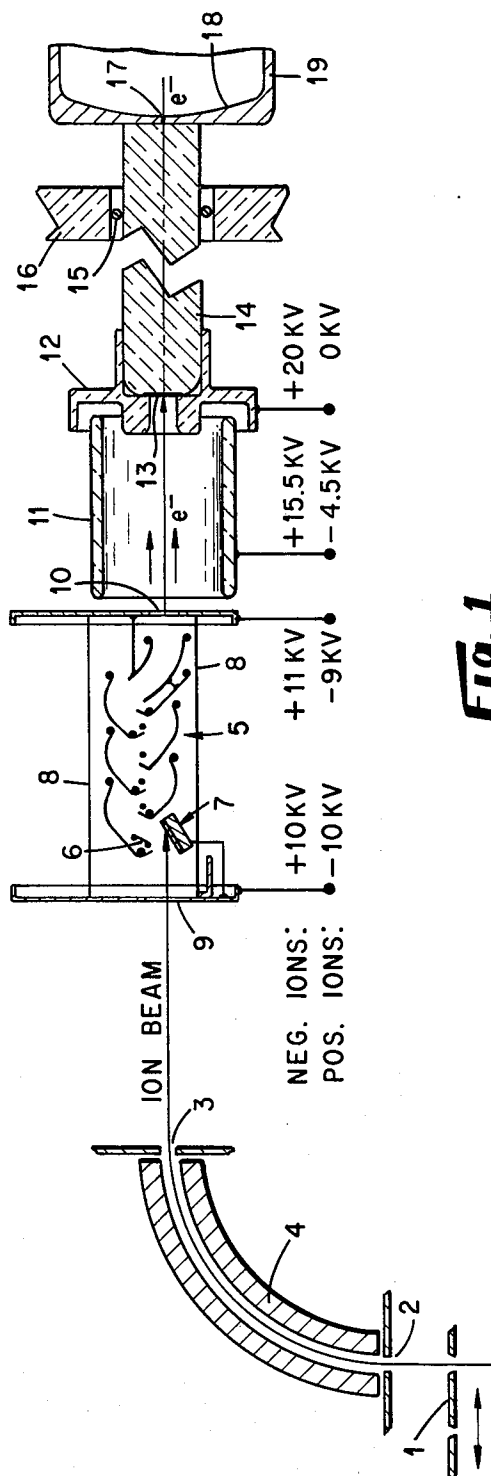


Fig. 1

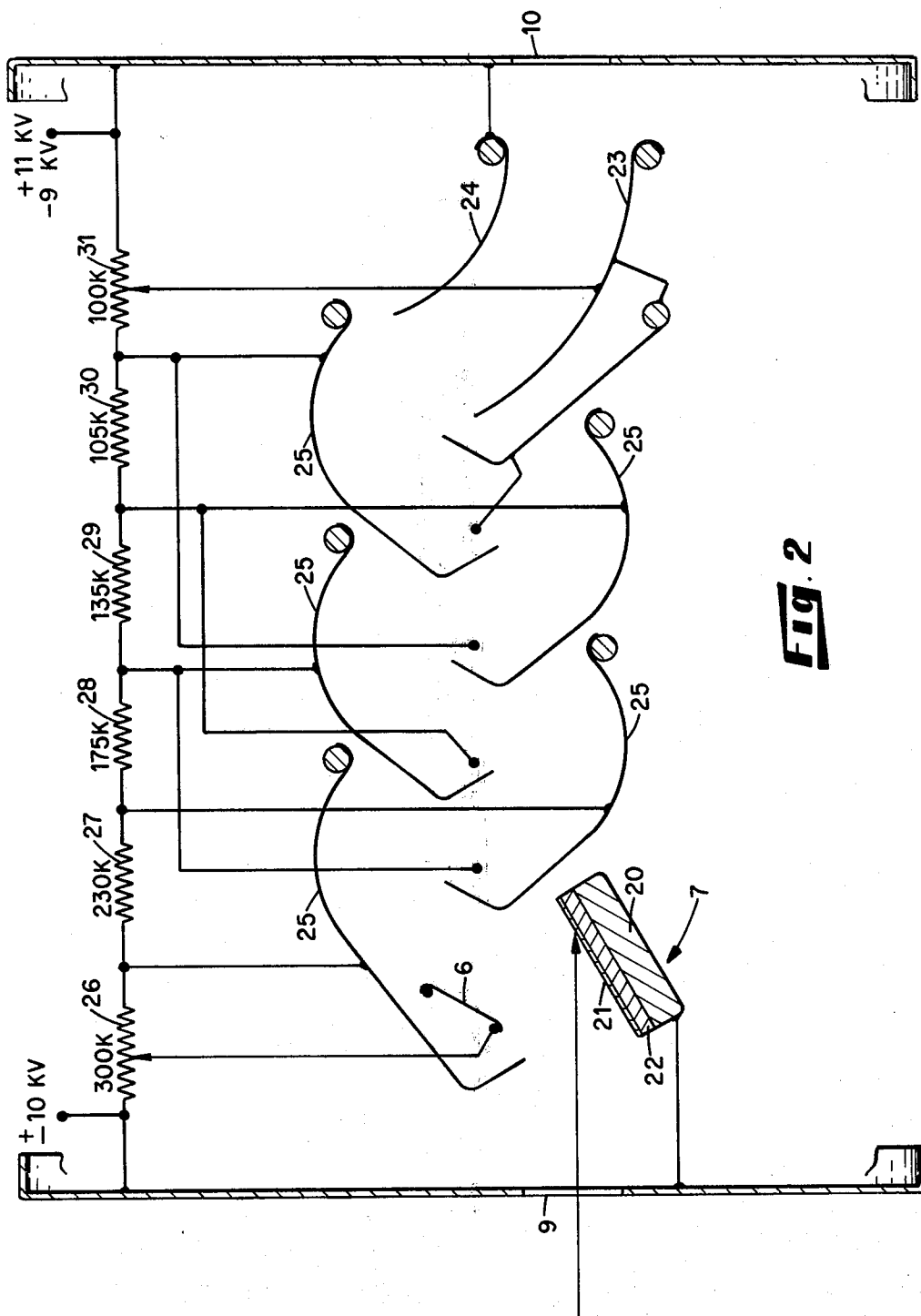


Fig. 2

ELECTRON MULTIPLIER-ION DETECTOR SYSTEM

BACKGROUND OF THE INVENTION

In an ion microprobe mass spectrometer a finely collimated and focused beam of energetic ions strikes a target surface. Secondary ions sputtered from the surface are mass analyzed and pulse counted in a particle detector which detects either positive or negative ions, but not both kinds simultaneously. Prior art devices have had rather high background counting noise of 20-40 counts per second whereas the desired background is 1 count per minute or less. Few mass spectroscopists have background counting rates less than 1 count per second even after many years of experience with the operation. In an effort to improve prior art devices, an electron multiplier was substituted for a scintillator detector in an ion microprobe. The multiplier succeeded well in both the positive and negative ion detection modes insofar as background counts were concerned, i.e., a few counts per minute.

The novel electron multiplier-scintillation detection has been developed for conventional magnetic sector mass spectrometers or ion microprobes. It is designed for a beam of ions focused to a small spot size or to a narrow line image at the focal plane of a mass analyzer. However, the basic concept is not limited to focused ion beams of kilovolts energy. With suitable modifications it should work equally well with unfocused ion beams or with low energy ion beams.

SUMMARY OF THE INVENTION

The present invention relates to a novel electron multiplier-scintillator ion detector for pulse counting positively or negatively charged single ions in mass spectrometers, for example, a 30-inch radius Dempster type mass spectrometer. A special feature of the new detector is the concept of an extended first dynode. More specifically, the conversion dynode and the five additional stages of the electron multiplier act as a single unit to convert each ion impact into a pulse of 500 or more electrons, on the average.

The basic objective of this invention is to combine the best features of an electron multiplier detector with the flexibility of a scintillator detector so that either positively or negatively charged ions can be detected with very high efficiency and be pulse counted as single events. Very simply, what is desired is one pulse detected and counted for each ion that strikes the conversion dynode, while at the same time maintaining a low background counting rate in the detector.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. I is a top plan view of the present ion detector system.

FIG. II is a more detailed view of the electron multiplier of FIG. I.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring now to FIG. I, the ion detector system includes a source of an ion beam, 1, which designates a series of mass spectrometer defining slits. Grounded defining slits 2 and 3 terminate the ends of an electrostatic analyzer 4 which functions to provide energy filtering of the mass-analyzed source ion beam. The electrostatic analyzer 4 also serves as a beam deflector to

guide the ion beam into an electron multiplier 5. The electron multiplier 5 consists of a conversion dynode 7, and five stages of secondary electron multiplying dynodes, a focus or deflector electrode 6 and a pair of curved deflector electrodes 23 and 24 (shown more clearly in FIG. 2). A mica insulating slab 8 is positioned at each end of dynodes 20 and 25 and electrodes 6, 23, and 24. The ion beam enters the multiplier section 5 through a rectangular aperture 9 to impinge on the conversion dynode 7. The secondary electrons from the multiplier exit through a circular aperture 10 through a cylindrical accelerating lens 11 and a disc lens with cylindrical symmetry 12 onto a flat plastic scintillator surface 13. The scintillator 13 is covered by a thin film of aluminum, 500-1000 Å thick. The secondary electrons are converted into photons in the scintillator 13 and then the photons are transmitted through a quartz light pipe 14 which is optically coupled to the plastic scintillator 13. The light pipe 14 is supported by an O-ring 15 in a vacuum wall 16 and is in turn optically coupled to a conventional photomultiplier tube 19 which is wired for pulse counting. A photon of light, denoted at 17, is shown striking a photocathode 18 of the photomultiplier tube 19.

A soft iron cylindrical magnetic shield (not shown) would surround the electron multiplier and scintillator assembly. It would extend from outside aperture 9 to beyond the scintillator 13. Vacuum seals, mechanical mountings and high voltage insulation of the light pipe and scintillator would follow conventional practice.

In FIG. II the electron multiplier 5 is shown in greater detail. The conversion dynode 7 consists of a dynode holder 20 made of nonmagnetic stainless steel supporting a glass or other suitable substrate 22 covered by an oxide surface 21. The oxide surface 21 which converts ion impacts into secondary electrons is a thin amorphous film of plasma-anodized aluminum oxide (Al_2O_3) of 100-200 Å thickness, ($1 \text{ Å} = 10^{-8} \text{ cm}$). This film surface 21 gives a higher yield and a more sharply peaked pulse height distribution than that from an aluminum film or solid aluminum metal surface which has been oxidized by exposure to pure oxygen or air.

The focus or deflector electrode 6 may be made of Type 304 stainless steel. The five curved dynodes designated 25 may all be made from CuBe alloy.

Resistors labeled 26, 27, 28, 29, 30, and 31 having the values set forth, namely: 300K, 230K, 175K, 135K, 105K, 100K ohms form a voltage divider connected serially across pairs of the dynodes which supplies a geometric voltage division to the dynodes. Alternatively, a linear voltage division, i.e. equal voltages between dynodes, also works well. The electron multiplier is designed to be floated at a nominal voltage such as $\pm 10 \text{KV}$ from ground potential, to detect negative or positive ions respectively. Approximate electrode voltages are shown in FIG. 1 for both the negative ion detection mode and the positive ion detection mode.

Considering the operation of the electron multiplier-scintillating ion detector system further, it will be seen that an ion beam emerging from mass spectrometer slit 1 will be guided through the electrostatic analyzer 4 or other suitable electrostatic lens which acts as a deflector so that the beam impinges onto the conversion dynode 7. The conversion dynode plus the five electron multiplying stages 25, act as a unit or extended first dynode to provide on the average more than 500 electrons per positively or negatively charged ion impacting

the surface of the conversion dynode. The cost in high voltage to accomplish this is small, about 1KV out of the ± 10 KV in the electron multiplier. The electron multiplier plays the role of an electronic preamplifier and provides virtually noiseless amplification of each pulse of secondary electrons from the conversion dynode. A current gain of several hundred is sufficient to establish a favorable pulse-height distribution in the electron multiplier section of the detector. These electrons pass through aperture 10, are accelerated to about 9KV energy and are focused onto the plastic scintillator 13. Conversion of these secondary electrons into photons in the scintillator 13, then back to photoelectrons in the photomultiplier tube 19 has little or no effect on the shape of the pulse-height distribution detected at the anode of the photomultiplier tube. This is true because the statistical multiplication process is determined in the first few stages of the electron multiplier. Any electron or photon amplification after the first few stages increases the DC gain of the system but does not noticeably change the shape of the pulse-height distribution for a given ion species.

The surface film of the conversion dynode has been described as a plasma-anodized metallic film. Thin oxide films of this type can also be formed by RF sputtering of pure oxides. Because the escape depth of ion-induced secondary electrons formed below an oxide surface is only of the order of 10 Å, an oxide film of 30-50 Å thickness produces as high a yield as one several hundred Å thickness. Therefore, the selection of the thickness of the film is a matter of choice. A thin film of pure beryllium oxide may be an alternate choice for the oxide film, since it may provide higher secondary electron yields and more sharply peaked pulse-height distributions than presently is realized from an Al_2O_3 surface film.

Various modifications of the present detector system may be made. For example, for unfocused ion beams or with low energy beams the sensitive area of the conversion dynode may be increased and venetian blind dynodes, transmission dynodes, mesh dynodes, or other secondary multiplication arrangements following the conversion dynode may be added. One obvious application for the new detector principle is in quadrupole, monopole or similar *rf* mass spectrometers. In these spectrometers the energy of the mass-analyzed ions can be as low as a few tens of volts. Post-acceleration of these low energy ions into the kilovolt range is necessary for efficient detection by pulse counting or when

using a secondary electron detector in the DC mode to measure very small ion currents. This detector could also be used to detect energetic neutral atoms.

Tests with a 10-stage 56 AVP photomultiplier tube in the new detector gave an output pulse width of less than 3 nanoseconds full width at half maximum amplitude (FWHM). This suggests a resolution time of 10 nanoseconds or less for the overall detector and counting system.

What is claimed is:

1. A combined electron-multiplier ion-detector system for use in a mass spectrometer comprising:

an ion beam source,
an electrostatic lens which receives, deflects and focuses the ion beam into

an electron multiplier, having an extended dynode unit consisting of a first conversion dynode and five additional electron multiplying dynodes,
an accelerating lens means connecting said electron multiplier to

a scintillator detecting surface,
a light pipe, optically coupled at one end with the scintillator, and

a photomultiplier tube optically coupled to the second end of the light pipe.

2. The combination defined in claim 1 in which the conversion dynode consists of a nonmagnetic dynode holder supporting a substrate covered by a thin amorphous oxide film surface.

3. The combination defined in claim 2 in which the conversion dynode and the five additional dynodes act as a unit to convert a single ion impact into a pulse of 500 or more secondary electrons.

4. The combination defined in claim 2 in which the extended dynode unit is floated at a voltage of ± 10 KV from ground potential to detect negative or positive ions respectively.

5. The combination of claim 3 wherein the accelerating lens means consists of a cylindrical lens and a disc lens which act to accelerate the pulse of secondary electrons and focus the pulse onto the scintillator.

6. The combination of claim 5 in which the scintillator consists of a flat plastic surface covered by a thin film of aluminum which converts the secondary electrons to photons which in turn are converted to photoelectrons in the photomultiplier tube, undergo further charge amplification there, and are pulse counted in associated electronic circuits.

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