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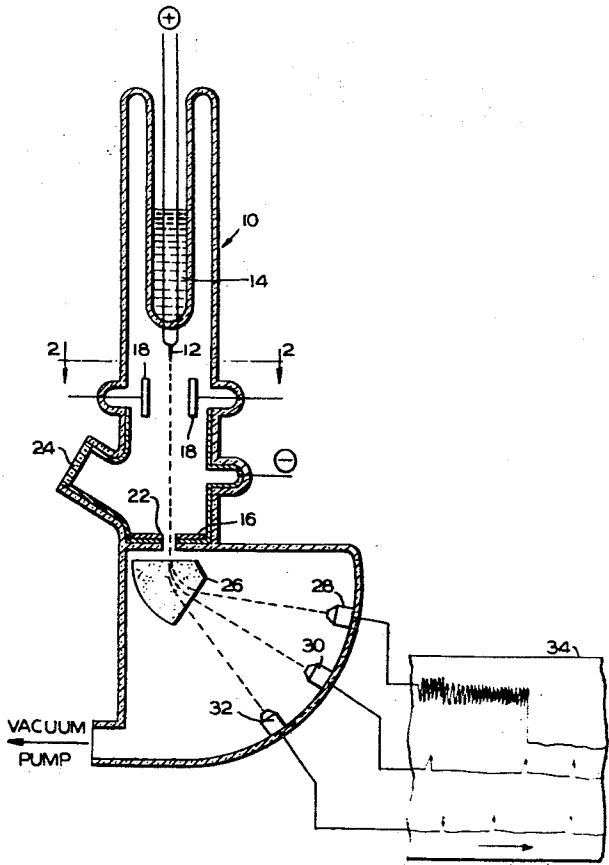
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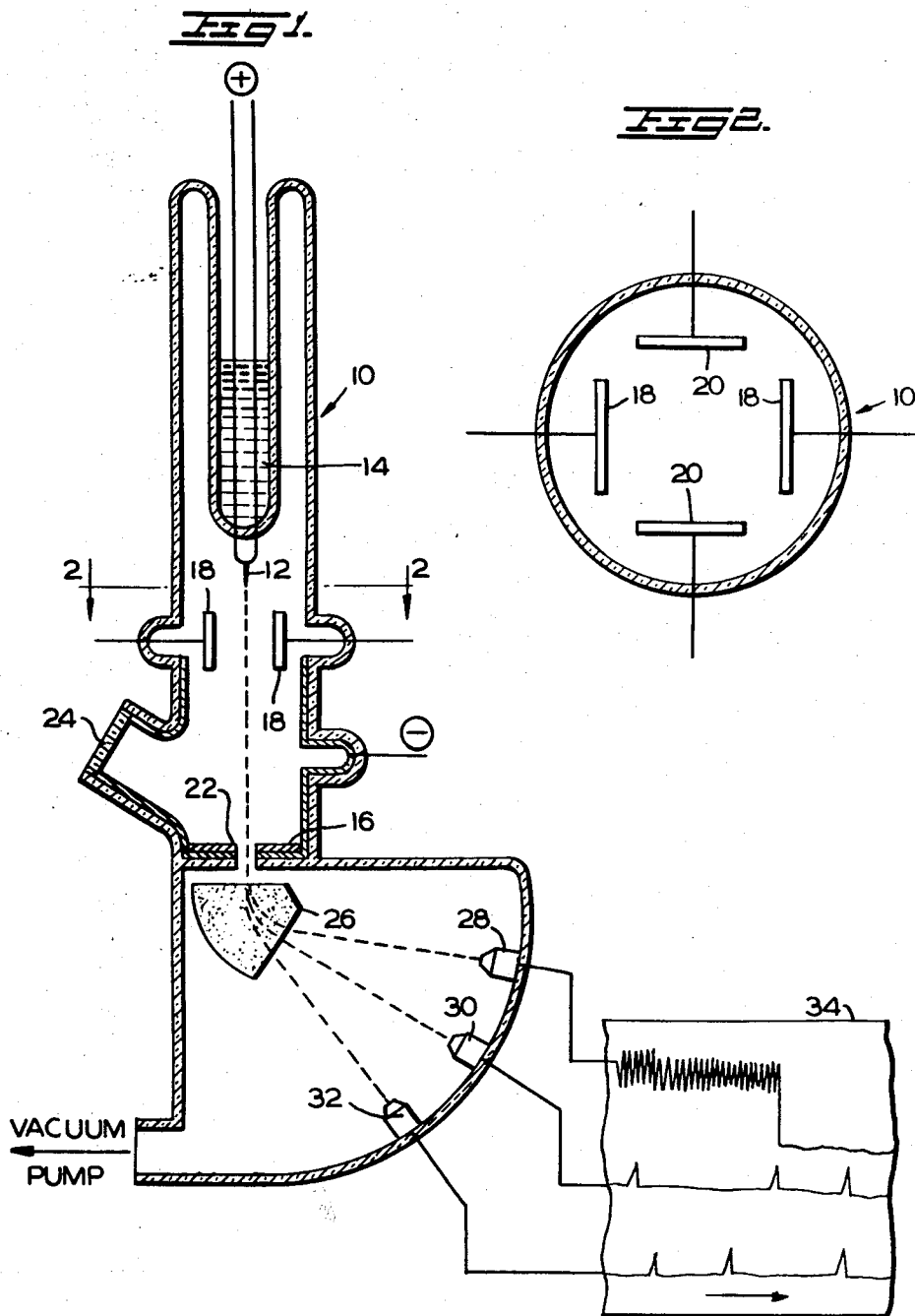
[54] **ATOM PROBE FIELD ION MICROSCOPE HAVING MEANS FOR SEPARATING THE IONS ACCORDING TO MASS**
8 Claims, 5 Drawing Figs.

[52] U.S. Cl..... **250/41.9, 250/49.5**
[51] Int. Cl..... **H01j 39/34, H01j 37/26**
[50] Field of Search..... **250/41.9, 41.9I SE, 41.9 R, 49.5**

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ABSTRACT: A field ion microscope capable of isolating and analyzing one or a few atoms of a specimen comprises a field ion microscope section including an emitter tip mounting the specimen to be examined, an apertured screen in the path of the beam of ions emitted by the specimen and means for adjusting the path traversed by the ion beam with respect to the aperture in the screen to cause a selected area of the beam to pass through the aperture and an ion detector positioned in the path of ions passing through the aperture. The path of the ion beam emitted by the specimen by the application of a high voltage may be altered by beam-deflecting means adjacent the beam path or by varying the angular disposition of the emitter tip with respect to the image screen, or the beam may be allowed to drift through a long tube and measured in its time-of-flight in each case using a detector of single particle sensitivity.

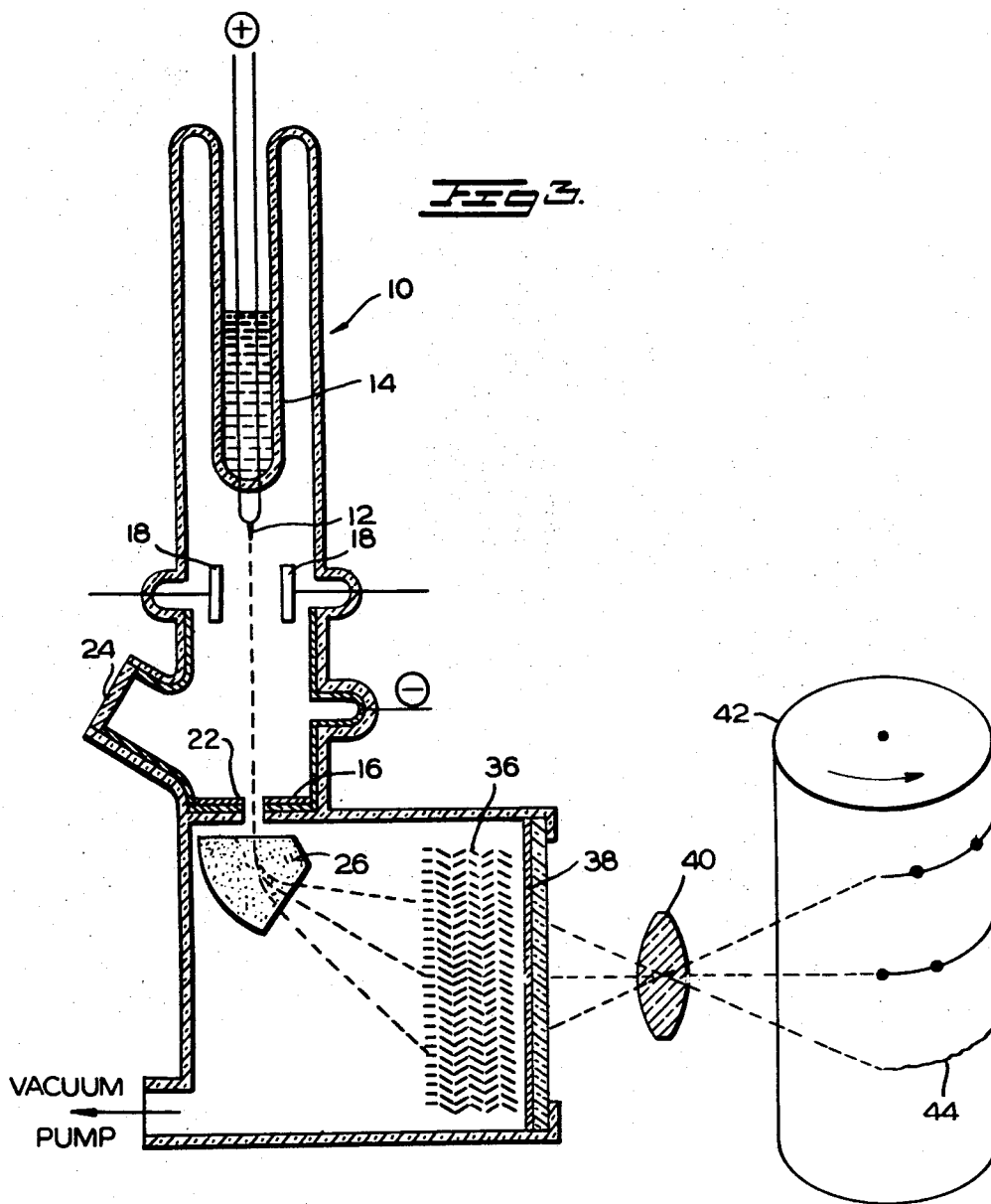




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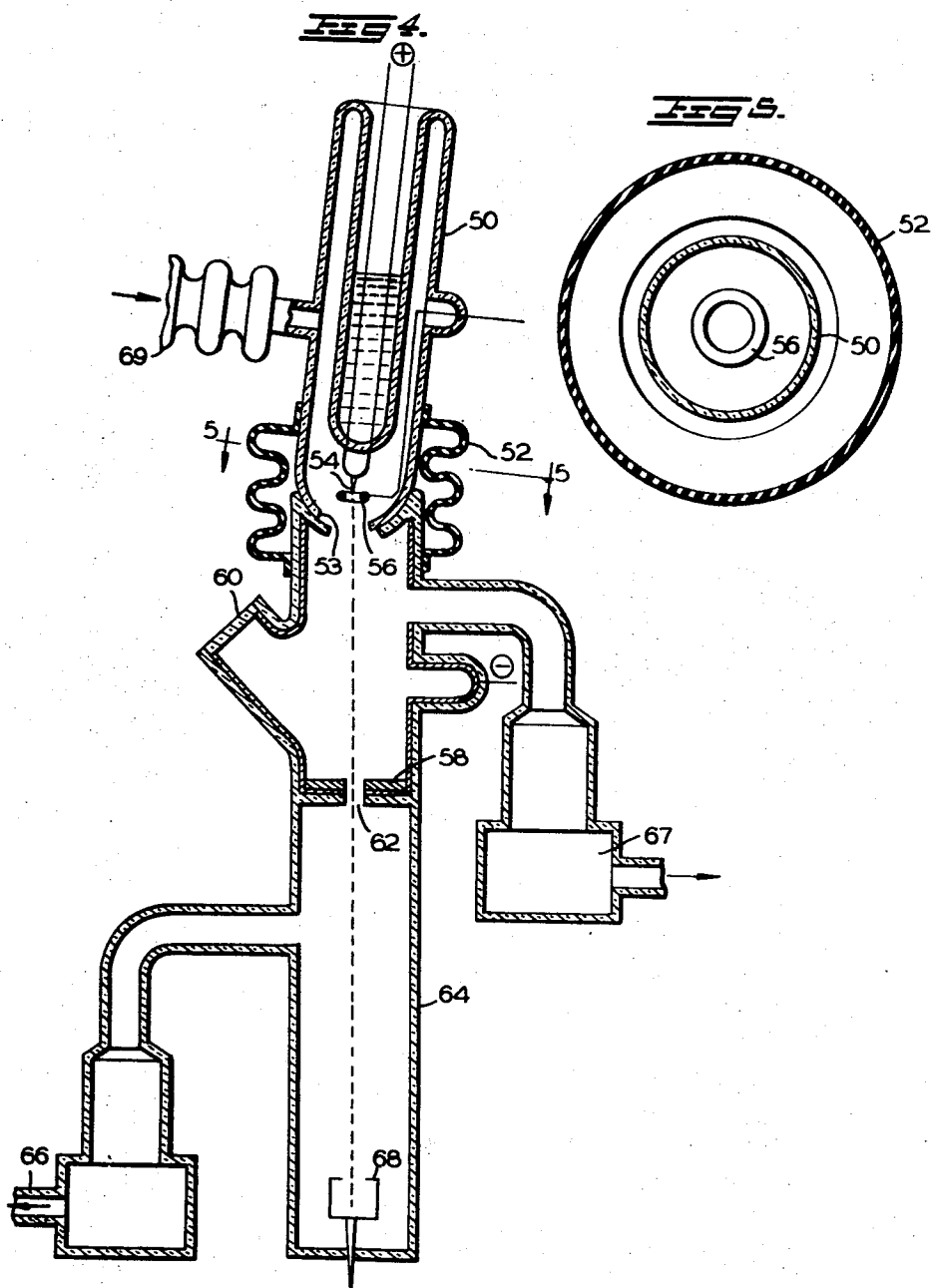


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ATOM PROBE FIELD ION MICROSCOPE HAVING MEANS FOR SEPARATING THE IONS ACCORDING TO MASS

This application is a continuation-in-part of my application Ser. No. 647,493 filed June 20, 1967 now abandoned.

The invention relates to a field ion microscope capable of isolating and analyzing one or a few atoms of a specimen under examination.

The field ion microscope (FIM), invented by applicant in 1951 (E. W. Müller, Z. Physik 131, 136, 1951), is the most powerful microscopic device known today, and the only one capable of showing the individual atoms as the building blocks of the specimen surface. The microscope is now widely used as a research tool in physical metallurgy of the higher melting point metals. One of the serious limitations of the instrument is its inability to discriminate the nature of the atoms seen on the specimen. As an example, in an image with atomic details of an iron-tungsten alloy one cannot tell which of the atoms is an iron atom, and which is a tungsten atom. Such knowledge is highly desirable for the study of short range and long range order in alloys, or for determining the nature, composition and localization of certain atoms in precipitates, or as solute or interstitial impurities.

On a much coarser scale identification of the constituents of a microscopical specimen is being done in the electron-microprobe analyzer in which the focused beam of an electron microscope causes the spot being investigated to emit X-rays, which are spectroscopically analyzed for the wavelengths characteristic of each chemical element (R. Castaing, "Electron Probe Microanalysis" in Advances Electronics and Electron Physics, Vol. 13, pp. 317-384, Academic Press, New York, 1960). The minimum spot size that can be investigated depends upon the spot size of the electron beam, and to some extent upon the X-ray detection sensitivity. In practice, the spot size is about one micron, and a piece of solid matter of this size contains more than 10 billion atoms. Somewhat greater is the sensitivity of a scanning electron microscope, (A. V. Crewe, SCIENCE 154, 729, 1966), but still the resolution limits the spot size to be analyzed to about 100 Å., or a cluster of 30,000 atoms, and atom species discrimination is crude.

The purpose of the present invention is the provision of methods and apparatus for the analysis of a single atom or a few atoms in the microscopic image that has been selected by the operator. The apparatus is appropriately called an atom-probe field ion microscope (atom-probe FIM).

One form of apparatus of the invention comprises a conventional field ion microscope including an image screen which has a small aperture or probe hole permitting a very small area of the image, usually the size of one atom, to be selected for analysis.

After an atom of interest has been pinpointed on the probe hole by a suitable image shifting device, the electric field strength at the specimen is increased until the atom is torn off from its substrate by field desorption or field evaporation. The atom leaves its site in the form of a singly or multiply charged ion and passes through the probe hole. The atom is analyzed by determining its charge-to-mass ratio, e/m , by the use of a conventional mass spectrometric device that can be made sensitive enough for single particle detection. The invention includes time recording of the detector signal to provide a correlation between the instant of field desorption of the atom to be analyzed and the signal of the detector.

The principles of the invention will be further described with reference to the accompanying drawings, in which:

FIG. 1 is a diagrammatic representation of an atom-probe field ion microscope including a magnetic mass analyzer;

FIG. 2 is a transverse section on line 2-2 of FIG. 1;

FIG. 3 is a diagrammatic representation of a modification in which an electron multiplier is positioned in the ion beam emerging from a magnetic mass analyzer;

FIG. 4 is a diagrammatic representation of a form of the invention in which selection of an ion image from the ion beam of a field ion microscope is effected by varying the angular disposition of the ion beam with respect to the image screen; and

FIG. 5 is a transverse section on line 5-5 of FIG. 4.

In FIGS. 1 and 2, the evacuated microscope section 10 contains the emitter tip specimen 12, which may be cooled by a cryogenic liquid 14, and kept at a high positive potential with respect to the screen 16. Between the tip and the screen are placed two pairs of deflection plates 18 and 20, similar to the x-y beam deflection plates in a conventional cathode ray tube. Upon the application of a suitable voltage plates 18 will electrostatically deflect the ion beam in the direction parallel to the plane of the drawing. The other set of plates, 20, operate in a direction normal to the plane of the drawing. Thus the ion image beams can be deflected at will to place the desired atom spot of the ion beam image on the probe hole 22. Additional deflection plates may be provided to assure a normal incidence of the beam on the hole. Observation or photography of the overall ion image is effected through window 24. The microscope section of the device is filled with a suitable imaging gas, preferably helium, of a few microns or less pressure.

The magnetic analyzer beyond the probe hole consists of a magnetic deflection field 26 normal to the plane of the drawing, and multichannel ion detectors 28, 30 and 32. The number of channels required depends on the desired discrimination. For the analysis of an alloy of known composition, say iron-tungsten, three channels in adjustable positions will suffice. One channel 28 will be set where mass 4 of the imaging gas, helium, will appear. Channel 30 will be set on mass 56 where singly charged Fe^+ ions are to be expected, and channel 32 on mass 184 for singly charged W^+ ions. The amplified outputs of the three detectors will be recorded on 34 as a function of time, while the electrical field applied to the tip is slowly increased. The flux of He^+ from the selected atom spot will suddenly cease when the atom in question is field desorbed. The ion of the latter will appear as a single impact on the proper detector. Noise discrimination is very good as only the detector signal simultaneous with the cessation of the helium ion flux is significant. A typical noise level even of an unsophisticated electron multiplier detector is one per second, while the helium ion flux is typically of the order of $10^4/\text{sec.}$, as indicated by the dense sequence of spikes in the recorder diagram. The sweep time of the recorder is of the order of one second. Instead of using the step in the time recording of the image gas ion beam it is also possible to record the beam of fast neutral atoms of the imaging gas which has its origin in charge exchange between the ion beam and the gas in the FIM vessel. This beam will not be deflected in the magnetic analyzer, thereby simplifying the design. For increasing the correlation probability, both the image gas ion and neutral beam may be time-recorded to mark the event of field desorption of the specimen atom in question.

In a modified embodiment of the invention a multichannel detector can be used in the image plane of the mass separation magnet. The detector section of an atom-probe FIM with this feature is shown in FIG. 3. Here the image plane of the analyzer magnet 26 is occupied by a Venetian blind-type electron multiplier 36 which sends its signals to an output screen 38 by means of lens 40. This screen can be viewed or can be photographically recorded on a drum-type film camera 42 to record an unknown ion impact at the instant of cessation of the image gas ion flux 44.

A different mode of ion discrimination is shown in FIGS. 4 and 5. The vacuum vessel 50 of the FIM section includes a bellows device 52 which may be used to tilt the specimen tip 54; in any desired direction. Preferentially the tilting motion of the tip is constrained by a spherical joint 54 so that the imaged tip and associated auxiliary ring electrode 56 retains its position in space. With this feature any part of the ion image as it appears on the phosphor screen 58 and is viewed through the

window 60 can be brought onto the probe hole 62. Normal incidence of the probe hole beam is thus assured at all times. The mass discrimination of the ions after passing the hole is effected by the conventional method of time-of-flight mass spectrometry. The analyzer section beyond the probe hole consists essentially of a drift tube 64 typically a few to about 50 inches in length and differentially pumped through the vacuum system 66. An ion detector 68 at the end, having single particle sensitivity, will pick up the arriving ions and the output of the detector is recorded on a fast oscilloscope as a function of time. The sweep time of the oscilloscope is typically of the order of 1 to 100 microseconds. The scope sweep is triggered by a nanoseconds risetime pulse given onto the auxiliary electrode 56 or is superimposed on the DC tip voltage. This pulse also causes the atom under study to be torn off from the tip specimen. A few microseconds later, the particle will arrive at the detector and will be recorded. The time-of-flight is a measure of the e/m ration which gives the desired atom identification. The arrival at the detector of the imaging gas ions is not disturbing, as such events, occurring in an average time spacing of 100 microseconds or more, will only rarely be recorded during the short sweep period.

It may be advantageous to interrupt the supply of imaging gas, usually helium, just before the desorption process is initiated. This can be done by closing a valve in the supply line 69. During adjustment of the image, the required helium gas pressure is maintained by either valving off the pumping lines, or by operating in a dynamic gas supply mode. (B. Wacławski and E. W. Müller, J. Appl. phys. 32, 1472, 1961). In the latter case, the microscope has the gap between the upper part 50 and the lower part of the microscope section much narrower than the cross section of the upper pumping system 67. This can be effected by providing the ring electrode 56 with a small orifice, near the center of which is the specimen tip 54. As a result, the helium gas pressure in section 50 and at the vicinity of the tip will be higher than in the rest of the instrument.

The probe hole in the screen of the field ion microscope section will usually be of a size equivalent to the to the apparent diameter of the atom selected for analysis. This size varies with the tip radius and the magnification of the microscope. The microscope therefore preferably includes means for adjusting the size of the probe hole during observation. This may be done by selecting a desirable size from an array or a revolver plate of various size holes, or using an iris-type variable aperture. For certain applications, such as the study of ordered alloys, it may be desirable to make the probe hole large enough to cover an area of several adjacent atoms that will be field desorbed simultaneously. The probe hole may also have an oblong shape of variable size and means to change the direction of the probe slot with respect to the ion image symmetry. This allows one to place the probe slot along a chain of atoms which are to be analyzed simultaneously.

It is not essential that the area of the selected ion beam be determined by the probe hole 62 of the apparatus of FIG. 4 as

the hole 62 may be larger and a field of view limiting aperture placed further along the beam path between phosphor screen 58 and the detector 68. If such a field of view limiter is used, the image screen may be unapertured and be tilted out of the path of the ion beam when the selected portion of the beam is to be analyzed.

It will be understood that any combination of the features of the systems described above may be applied in the utilization of the invention. For example, the magnetic analyzer type atom-probe FIM may be equipped with the bellows type of image point selection as well as with the differential pumping scheme in the analyzer section, and vice versa.

I claim:

1. An atom-probe field ion microscope comprising a microscope section including an emitter tip mounting a specimen to be examined, means for introducing an ionizable gas into the section, an image screen in the path of the beam of ions emitted by the specimen apertured to pass the image of a single atom of the emitter tip, means for adjusting the path traversed by the beam with respect to the aperture in the screen to cause a selected area of the beam to pass through the aperture and means for increasing the electrical field strength at the emitter tip to effect emission of ionized atoms of the emitter tip material and an ion detector positioned in the path of ions passing through the aperture.

2. An atom-probe field ion microscope as defined in claim 1 including paired deflector electrodes adjacent the path of the ion beam emitted by the specimen for varying the direction of travel of the ion beam.

3. An atom-probe field ion microscope as defined in claim 1 wherein the mounting means for the emitter tip includes a flexible bellows for varying the angular position of the emitter tip with respect to the apertured screen.

4. An atom-probe field ion microscope as defined in claim 1 including an ion detector channel detecting ions of gas atoms ionized at the emitter tip and at least one further channel detecting ions of desorbed specimen atoms passing through the aperture.

5. An atom-probe field ion microscope as defined in claim 1 including means establishing a magnetic field transversely of the path of ions passing through the aperture and at least one ion detector channel in the path of ions deflected by the magnetic field.

6. An atom-probe field ion microscope as defined in claim 1 including means for pulsing the field on the emitter tip to effect the desorption of atoms from the specimen.

7. An atom-probe field ion microscope as defined in claim 6 including means for relating the time of a pulse and the time of detection of an atom desorbed by such pulse to determine the e/m ratio of such atom.

8. An atom-probe field ion microscope as defined in claim 1 including means for altering the size, shape or location of the effective beam limiting aperture.