United States Patent

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[33] France
[31] 916,836
Continuation of application Ser. No. 326,566, Nov. 27, 1963.

[54] **MICROANALYZER FOR PRODUCING A CHARACTERISTIC IONIC IMAGE OF A SAMPLE SURFACE**
5 Claims, 4 Drawing Figs.

[52] U.S. Cl. 250/49.5, 250/41.9
[51] Int. Cl. G01n 23/00, H01j 37/00
[50] Field of Search .... 250/41.9 ISB, 41.9 ISE, 41.9 R, 41.93, 49.5

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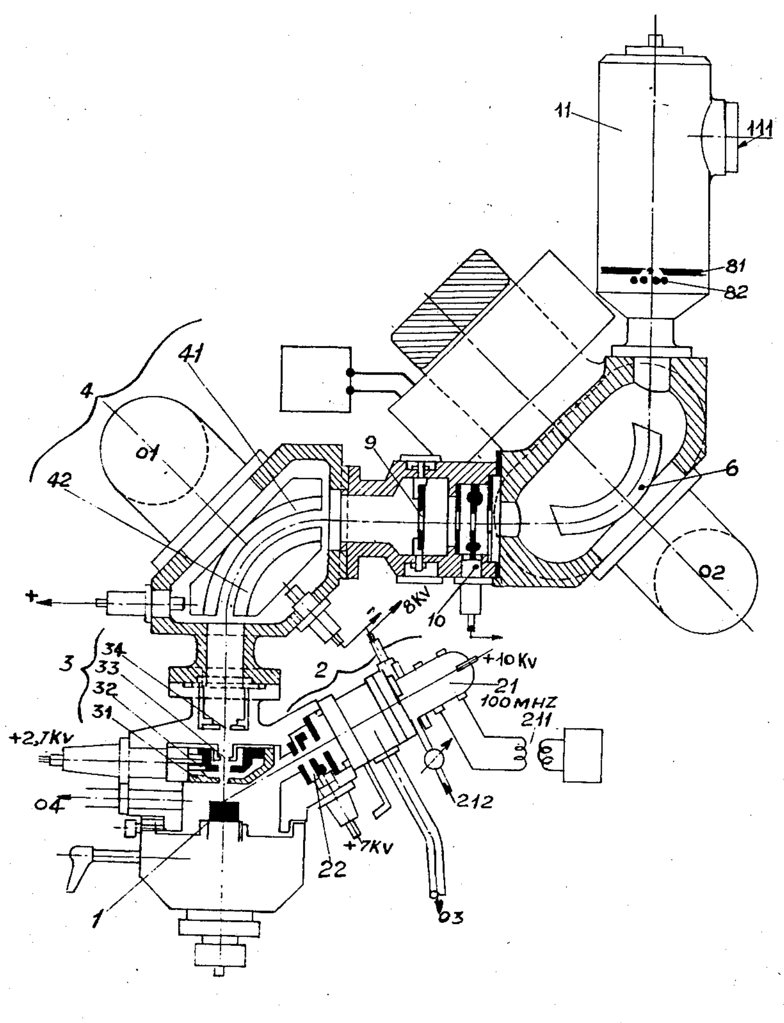
Primary Examiner—William F. Lindquist
Attorney—Cushman, Darby & Cushman

ABSTRACT: The present invention relates to microanalysers and more particularly to microanalysers making use of the secondary ion emission for producing, by means of a corpuscular optical system which combines ion optics and mass spectrography, "characteristic images" of the surface of the sample which indicate the map of distribution of its various elements or isotopes.



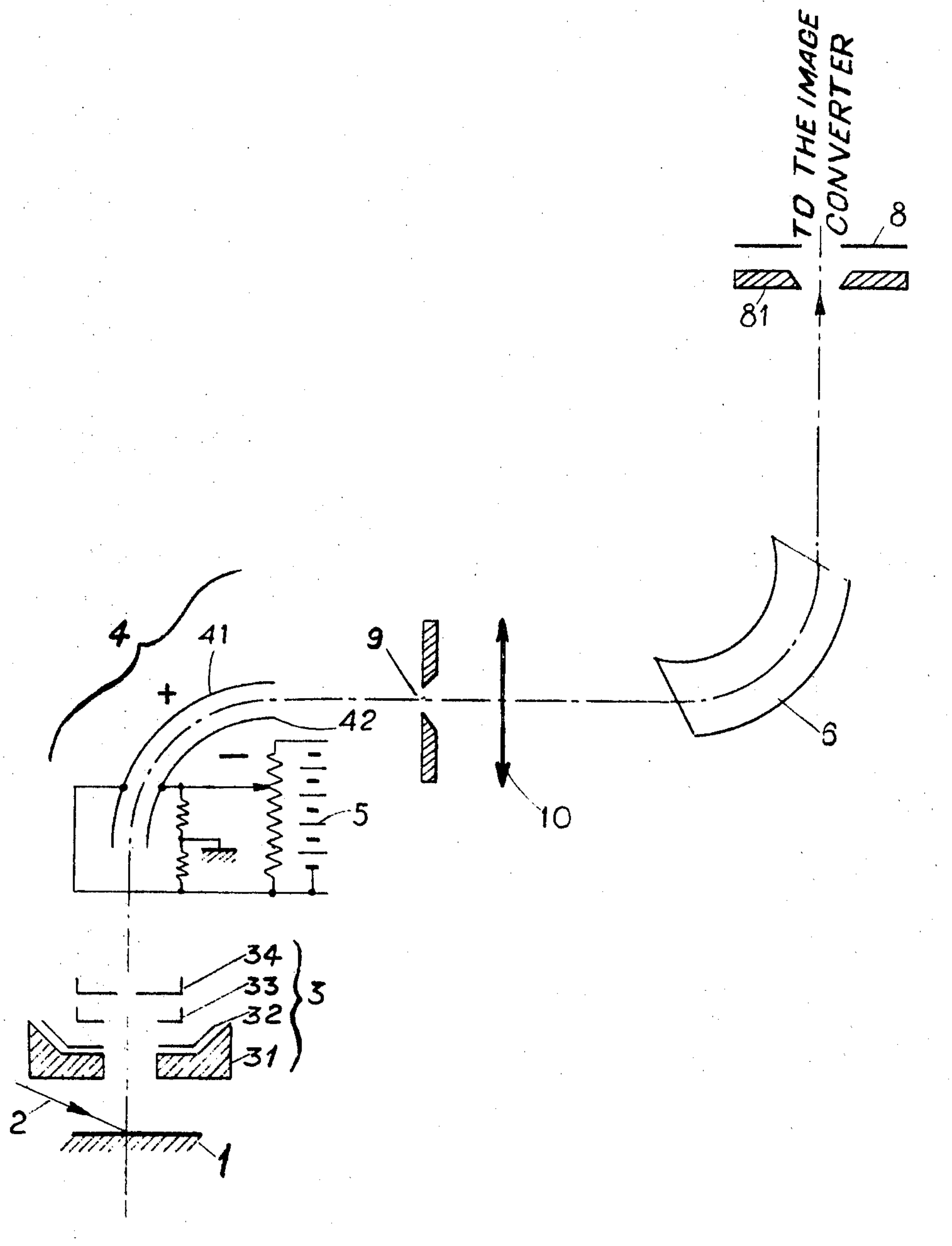
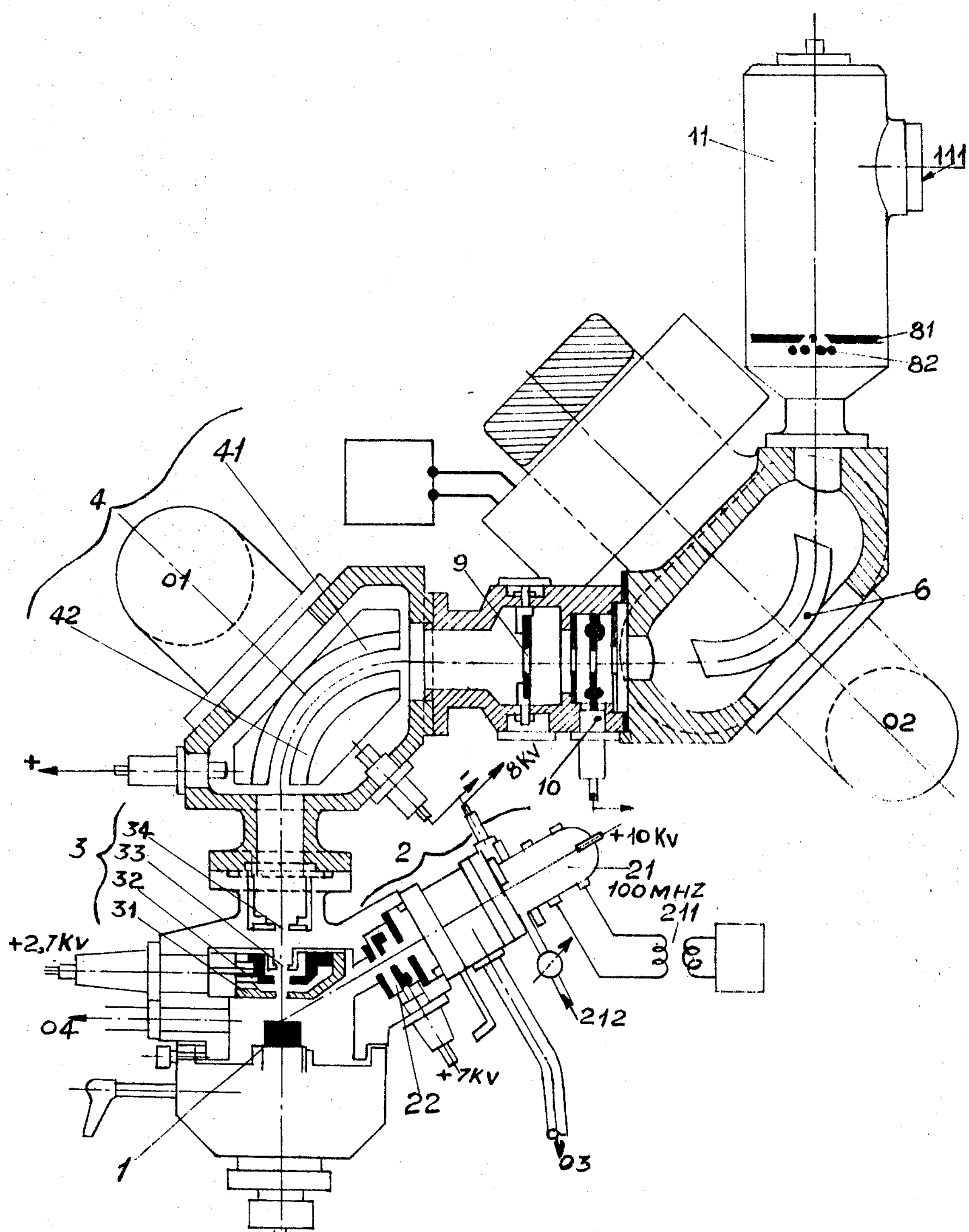


FIG. 1

FIG.2


11
111
81
82
4
41
01
42
9
6
02
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8KV
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32
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+7KV
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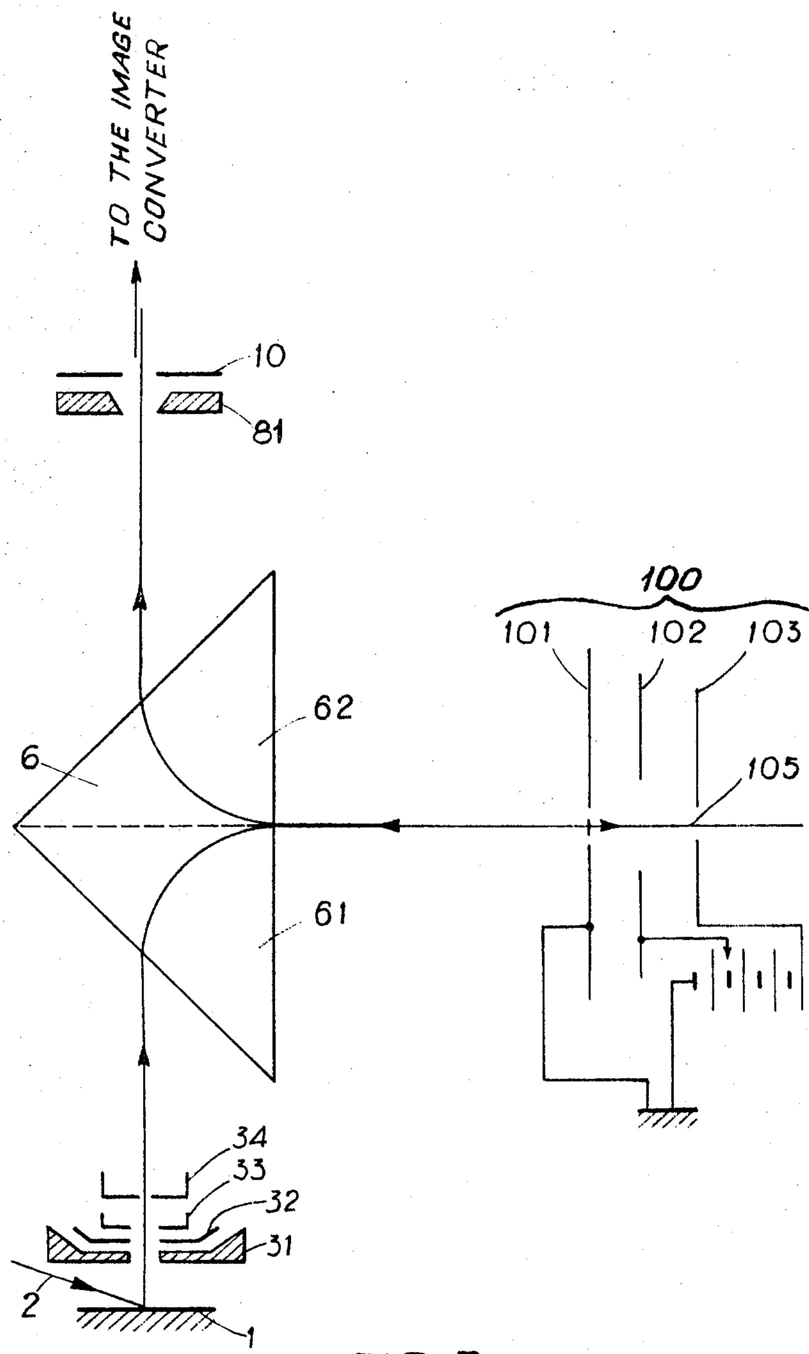
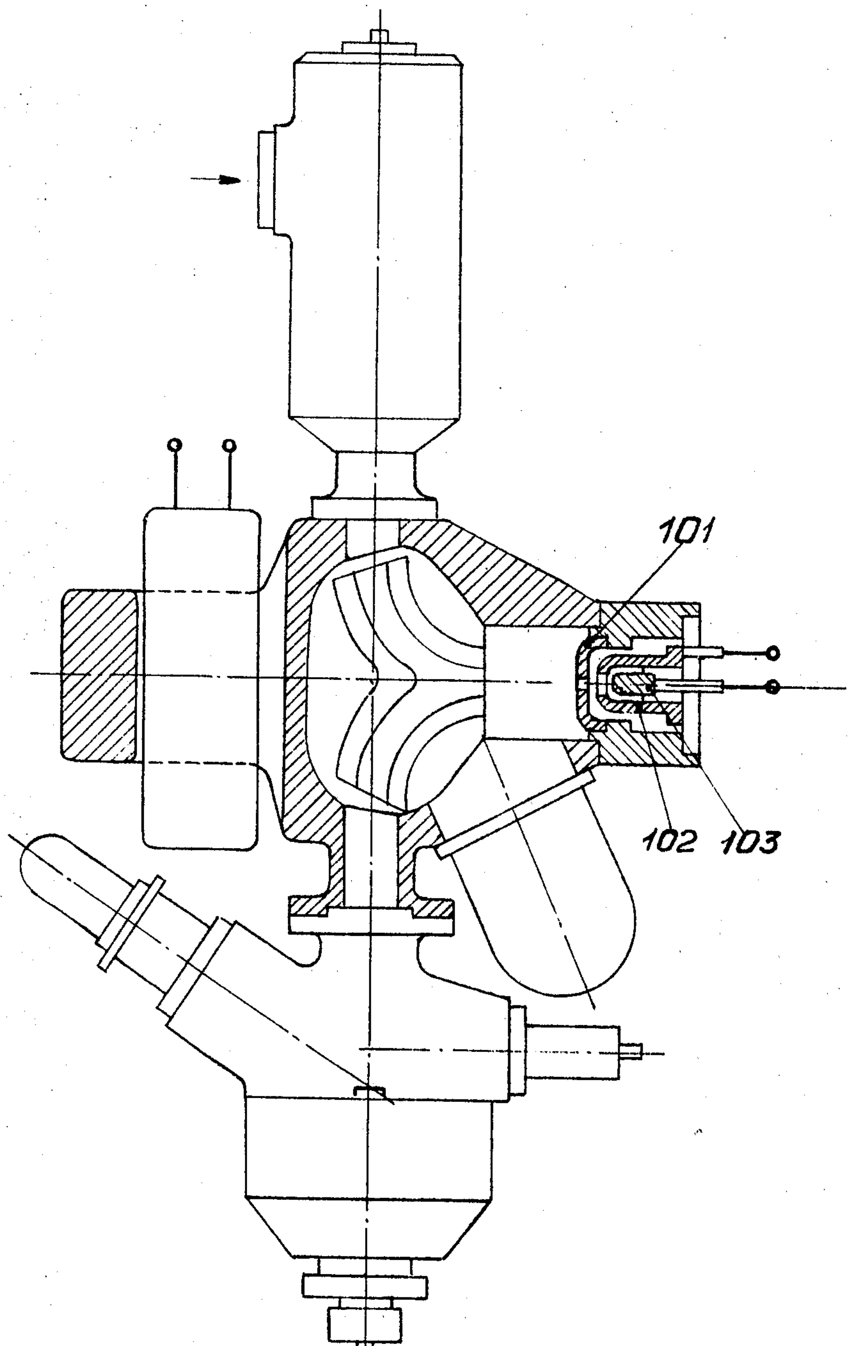

TO THE IMAGE CONVERTER
10
81
100
101
102
103
62
6
105
61
34
33
32
31
2
1

FIG. 3

FIG. 4



MICROANALYZER FOR PRODUCING A CHARACTERISTIC IONIC IMAGE OF A SAMPLE SURFACE

This application is a continuation application continuing the application Ser. No. 326,566, filed Nov. 27, 1963 for "Improvements in microanalysers by secondary emission."

Microanalysers wherein the analysis of a sample takes place by collecting and focusing into an image of the sample the secondary ions emitted by the sample under the impact of a beam of primary positive ions, are known in the art. A suitable optical system was described in the French Pat. No. 1,240,658 and in a scientific paper by the applicants R. CASTAING and G. SLODZIAN "PREMIERS ESSAIS DE MICROSANALYSE PAR EMISSION IONIQUE SECONDAIRE" - Comptes Rendus Academie des Sciences de Paris 255, 1893—1895 (15th Oct. 1962). Its operation is generally satisfactory. It has, however, the drawback of not allowing an easy separation of the secondary ions of high and nearly equal mass on account of the fact that all the secondary ions do not leave the sample at the same initial velocity.

It is an object of the invention to provide an arrangement which avoids this drawback.

The arrangement for microanalysis by bombardment with primary positive ions or neutral atoms according to the invention comprises an optical system for focusing secondary ions, including means for eliminating by filtering those secondary ions whose initial energy is higher than a predetermined value.

According to a first embodiment of the invention, the said electron optical system includes a spherical capacitor which, by means of a slit, ensures the elimination of ions whose initial energy is higher than or different from a predetermined value.

According to another embodiment of the invention, the electron optical system includes means for deflecting the secondary ion beam onto an electrostatic mirror in order to eliminate the excessively fast ions while reflecting the others.

The invention will be better understood from the following description and appended drawings, in which:

FIG. 1 shows very schematically a first embodiment of the invention;

FIG. 2 shows in more detail a possible arrangement of the different parts according to this first embodiment;

FIG. 3 shows another embodiment of the invention; and

FIG. 4 shows in more detail a possible arrangement of the different parts according to this second embodiment.

In the arrangement shown by way of example in FIG. 1, a sample 1 emits, under the impact of a beam of positive primary ions or fast neutral atoms 2, characteristic secondary ions which are taken up by the acceleration optical system 3, comprising electrodes 31 to 33 and an aperture 34. A spherical capacitor 4 is placed in the path of the beam of secondary ions. It comprises two plates 41 and 42, held at different potentials by means of a source 5; it gives of the crossover of the acceleration optical system 3, which is located in the aperture 34, a real image crossover located in a symmetrical position, at a distance of the outlet of the spherical capacitor, equal to the radius of curvature of the mean ion trajectory. At the level of this image of the crossover, a wall, provided with a slit g, stops some of the ions which have gone through the capacitor 4, and the remaining ions are focused by a lens 10. The optical system includes, in addition, the magnetic sector 6, which deflects ions which propagate through it and is adjustable by a suitable control device, and lastly an ion optical system 8 which comprises a selection slit 81, a stigmator and other electrodes (not shown) which are parts of an image converter, as disclosed in the above-mentioned French Patent.

The arrangement operates as follows:

The ions accelerated by electrodes 31 to 33 are deflected by capacitor 4 towards magnetic sector 6. The angle of deflection of each ion trajectory depends on the energy of the corresponding ion. For the purpose of simplification, it is assumed that the ions considered are positive and of the single charge type.

Ions whose initial energy is too high are stopped at the upper edge of slit 9. In this way, ions whose initial energy is too high are eliminated from the beam entering the magnetic sector 6.

In the same way, ions whose initial energy is too low may be stopped by the lower edge of slit 9 and eliminated from the beam entering the magnetic sector 6 if that is considered necessary for the analytical procedure.

The electrostatic lens 10 is positioned and excited in such a way that the ion beam, after passing it, enters the magnetic sector under suitable conditions, as is known in the art and described in the French Patent No. 1,240,658, for ensuring focusing in both transverse directions and production of an exit crossover at the level of slit 81.

However, as is well known, the radius of curvature of the trajectories in the magnetic sector depends only on the momentum of the emitted particle, for a given induction, that is to say on the value EM, where E is the energy of the particle and M is its mass number.

In the case of a mere magnetic filtering of the ions without any prior filtering of the ions according to their respective energies, there would be added to ions of mass number M, emitted with zero initial energy, ions of mass numbers M—I ...... M—n, if any, emitted with some initial energy ΔE. This cannot occur after prior elimination of ions emitted with an initial energy higher than desired. A simple calculation shows that the additional amount of energy ΔE required to ensure that an ion of mass number M—n is deflected in the magnetic sector with the same curvature as an ion of mass number M and energy E, if $\Delta E = nE/M$.

As a result, an arrangement based only on magnetic deflection is unable to secure proper separation of ions of nearly equal mass. Diaphragm 34 does actually ensure some elimination of ions emitted at a certain initial velocity, but such filtering is insufficient since it acts on the component of the initial velocity of the secondary ions perpendicular to the axis of the system only. The spherical capacitor which deflects the ions as a function of their energy and not of their momentum, ensures a perfect filtering.

It can be shown that in order to separate ions of nearly equal mass, for example mass numbers 250 and 251, the filtering has to be effected to within 8 volts, for an accelerating voltage equal to 2,000 volts. A rather large capacitor is necessary, or at least a great distance has to be allowed between the first beam crossover and the image of it which is produced by the spherical capacitor.

The optical system which comprises the first emission lens 3, the spherical capacitor 4, the intermediate lens 10 and the magnetic sector 6 gives, when properly adjusted, a characteristic image of the sample surface produced by ions of any desired mass, which is achromatic, that is to say insensitive to the slight heterogeneity of the ion energies which is allowed by the width of slit 9.

The correction of the astigmatism of this image and its convenient observation and recording occur in the image converter, known in the art and described in French Patent No. 1,240,658 and in scientific publications: R. Castaing, B. Jouffroy and G. Slodzian, "Sur les possibilites d'analyse locale d'un echantillon par utilisation de son emission ionique secondaire," C.R. Acad. Sc. Paris 251, 1010—1012 (22 Aug. 1960).

FIG. 2 shows in more detail a possible arrangement of the different parts.

The sample 1 is bombarded by the ion beam produced by the focusing gun 2 which comprises an ion source 21, excited by high frequency generator 211 and fed by gas inlet 212, and a condenser lens 22. Means for neutralizing the primary ions have not been represented here. The secondary ions are accelerated and focused into an image of the sample surface by the emission lens 3 which comprises accelerating electrode 31, focusing electrodes 32 and 33 and aperture 34. The crossover at the aperture 34 acts as a source for the energy selecting spherical capacitor 4 comprising two plates 41 and 42. The distance between aperture **34** and the capacitor entrance is equal to the radius of curvature of the mean trajectory in the capacitor. At an equal distance of the capacitor outlet is produced an exit crossover and slit **9** is located there. The three electrode lens **10** gives of the crossover **9** an image in a position suitable for a convenient focusing by the magnetic sector **6** and filtering according to moment by slit **81**. The stigmator **82** corrects for astigmatism the characteristic image of the sample surface which is produced by the whole optical system. This image is then taken up by the image converter **11**, known in the art, for convenient observation and recording through a window **111**. The system is evacuated through openings **01**, **02**, **03** and **04**.

FIG. **3** shown another embodiment of a system according to the invention. shows for simplification, it is assumed that the secondary ions are positive, but negative secondary ions could be used as well in the embodiment of FIGS. **1** and **2** as in the embodiment of FIGS. **3** and **4**.

In FIG. **3** the same reference numerals designate the same parts as in FIG. **1**. The shape of the magnetic sector **6** is as shown.

Its lower part **61** deflects the ions towards an ion mirror **100** comprising three electrodes **101**, **102** and **103**; electrode **103** is at a slightly higher positive potential than target **1**. This electrode may be provided with an aperture **105**; in this case excessively fast ions pass through this aperture and do not return, while slower ions are decelerated and returned by electrode **103** towards the upper part **62** of sector **6** which deflects them towards selection slit **81**, stigmator **82** and the image converter.

In another embodiment electrode **103** may be solid (devoid of the aperture **105**) to capture excessively fast ions, while reflecting slower ions.

The use of such an arrangement comprising deflection, reflection, and deflection for obtaining images filtered according to the energy is known in the art in the case of electron images and has been described by R. Castaing and C. Henry, "Filtrage magnetique des vitesses en Microscopie Electronique," C.R. Acad. Sc. Paris 255, 76—78 (2 July 1962).

When applied to ions of various masses and energies it provides a double filtering: according to momentum through the magnetic deflection and according to energy by mirror electrode **103** which stops or eliminates the ions whose energy is higher than a given value.

The arrangement of FIG. **3** provides, over that shown in FIG. **1**, the additional advantage of making it possible to place the sample coaxially with the display screen (not shown).

On the other hand, the arrangement of FIG. **1** provides the advantage of making it possible to pick up for obtaining the image the secondary ions which are emitted by the sample with a given initial energy and rejecting those of the secondary ions which are emitted with a lower or higher initial energy.

FIG. **4** shows in more detail the arrangement of the various parts of the system; the mirror electrode **103** is in this figure devoid of the aperture **105** and it stops the ions whose energy is too high.

Of course, the invention is not limited to the embodiments shown which were given solely by way of example.

We claim:

1. A microanalyser arrangement for providing a characteristic ionic image of the surface of a sample by bombarding said sample with particles causing the emission of secondary characteristic ions, said arrangement comprising:

a primary ion source for bombarding said sample;

lens means for concentrating said secondary ions to form a beam providing an ionic image of the sample surface;

means positioned in the path of said beam for eliminating from said beam, ions having an energy higher than a predetermined value, for obtaining an energy-filtered beam;

magnetic field means positioned in the path of said filtered beam for rejecting therefrom the ions having a momentum different from a predetermined value, said magnetic means having an outlet; and

means located at said outlet for displaying said ionic image,

wherein said magnetic field means comprise a magnetic sector and means for controlling the magnetic field in said sector,

wherein said eliminating means comprise a spherical capacitor, having two plates facing each other and an outlet, means for creating between said plates a direct current electric field, and a wall having a slit, being positioned near said outlet.

2. A microanalyser arrangement for providing a characteristic ionic image of the surface of a sample by bombarding said sample with particles causing the emission of secondary characteristic ions, said arrangement comprising:

a primary ion source for bombarding said sample;

lens means for concentrating said secondary ions to form a beam providing an ionic image of the sample surface;

means positioned in the path of said beam for eliminating, from said beam, ions having an energy higher than a predetermined value, for obtaining an energy filtered beam;

magnetic field means positioned in the path of said filtered beam for rejecting therefrom the ions having a momentum different from a predetermined value, said magnetic means having an outlet; and

means located at said outlet for displaying said ionic image,

wherein said magnetic filed means comprise a magnetic sector and means for controlling the magnetic field in said sector,

wherein said eliminating means comprise an ion mirror, said ion mirror comprising means for reflecting ions whose energy is lower than a predetermined level and for eliminating the other ions.

3. An arrangement as claimed in claim **2**, comprising a further magnetic sector positioned for deflecting said beam towards said ion mirror.

4. An arrangement as claimed in claim **3**, wherein said mirror comprises an electrode which collects the ions having an energy higher than said predetermined level.

5. An arrangement as claimed in claim **3**, wherein said mirror comprises an apertured electrode, having a center hole for allowing the ions, whose energy is higher than said predetermined level, to propagate beyond said mirror.