

[54] ION MICROANALYZER

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[58] Field of Search ..... 250/309, 307, 306, 310, 250/397

[57] ABSTRACT

An ion microanalyzer which detects and mass analyzes the secondary ions emitted from a specimen surface when it is scanned with an ion beam, comprising a cathode ray tube the electron beam of which is scanned synchronously with the scanning of the above-mentioned ion beam, the intensity of the electron beam being modulated by the directly detected signal of the secondary ions.

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15 Claims, 4 Drawing Figures

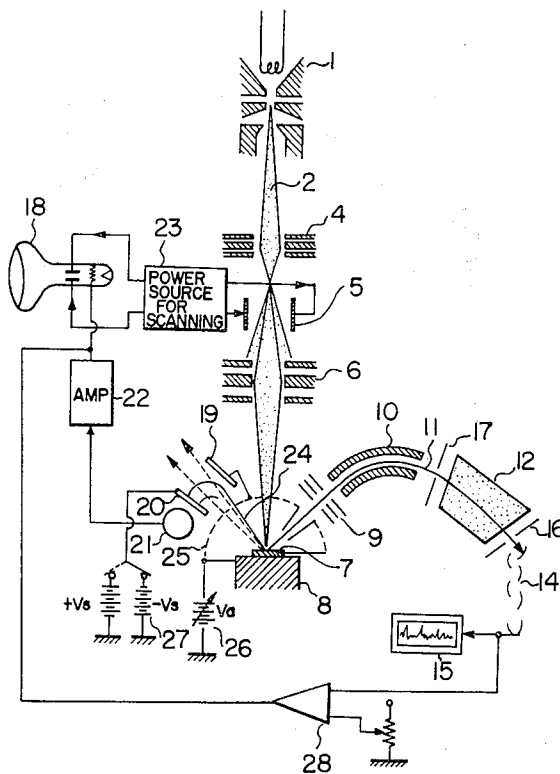


FIG. 1 PRIOR ART

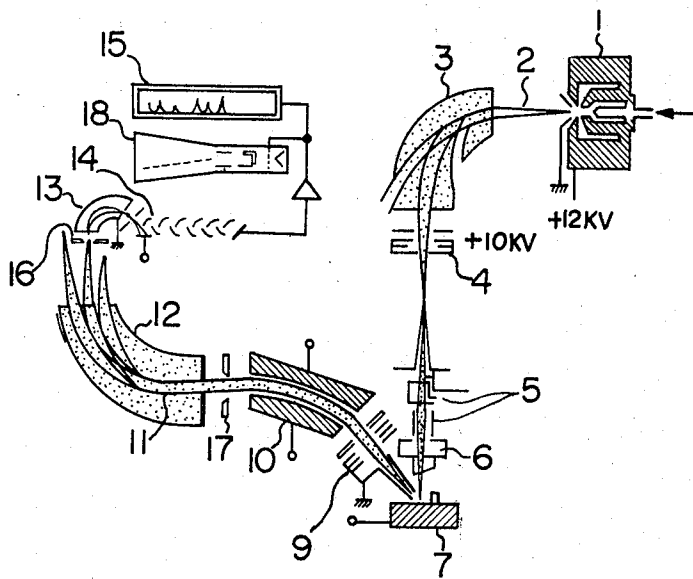


FIG. 2

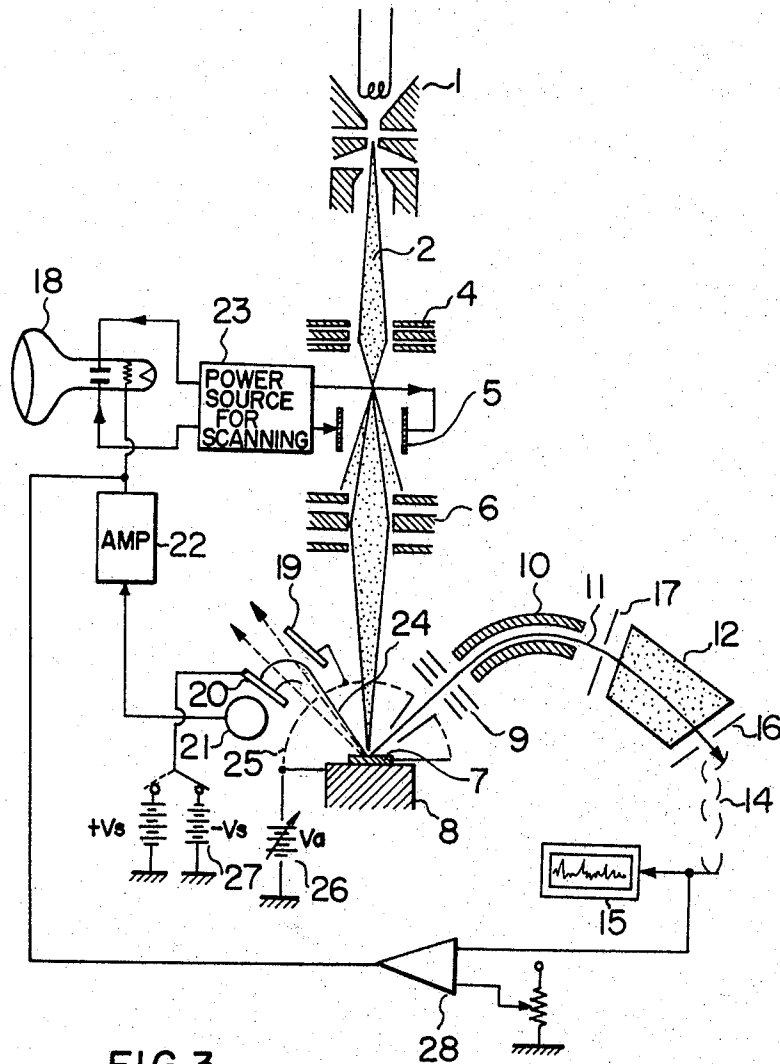


FIG. 3

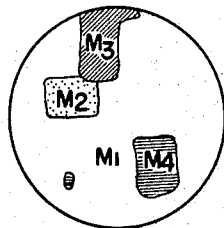
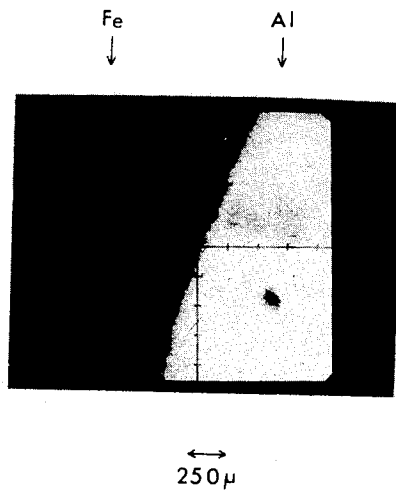


FIG. 4



ATOMIC NUMBER CONTRAST  
( Al — Fe )

## ION MICROANALYZER

The present invention relates to an ion microanalyzer, and more particularly to an ion microanalyzer which can display the image of the secondary ions emitted by a specimen on the face plate of a cathode ray tube.

The ion microanalyzer of the type described is for the purpose of obtaining an image of the surface of a specimen, potential distribution, atomic number contrast (the contrast resulting from the difference in the secondary ion emitting power of elements), or the like on the face plate of a cathode ray tube and/or on the recorder by detecting secondary ions emitted from a specimen by being scanned with an ion beam.

A prior art ion microanalyzer for the preparation of a description of the present invention and a preferred embodiment of the present invention will be described with reference to the accompanying drawings, in which

FIG. 1 is a schematic construction of a prior art ion microanalyzer;

FIG. 2 is a schematic construction of an ion microanalyzer according to the present invention;

FIG. 3 is a secondary ion image on the face plate of a cathode ray tube; and

FIG. 4 is a photograph of a secondary ion image.

As a preparatory description for the present invention a prior art ion microanalyzer will first be described with reference to FIG. 1. The prior art ion microanalyzer shown in FIG. 1 includes a primary ion irradiation system, a double focussing type mass-spectrometer, a secondary ion detecting system, and a cathode ray tube. The primary ion irradiation system consists of an ion source 1, a primary ion separator 3 for ions 2 emitted by the ion source 1, a condenser lens 4, an objective lens 6 and deflection electrodes 5. An ion beam emitted by the ion source 1 is converged by the condenser lens 4 onto a specimen 7 to a diameter of several microns or less, and scanned by the deflection electrodes 5 synchronously with a cathode ray tube 18 for observation. A part of the secondary ions emitted by the specimen 7 passes through an electrostatic lens 9 and enters the mass-spectrometer which consists of an electric field device 10, a magnetic field device 12 and slits 16 and 17. The mass-analyzed ions pass through a deflector 13, are detected and amplified by a detector such as a secondary electron multiplier 14, and supplied as a video signal to the cathode ray tube 18 and a recorder 15 to provide an image for the concentration distribution of a particular element in the surface of the specimen 7.

This ion microanalyzer can display an image of a particular element as described above, but the image is not clear and distinct because the intensity of the video signal for forming the image is low due to the fact that the video signal is obtained by detecting the ions analyzed by the mass-spectrometer. That is, this image forming method has the following disadvantages:

1. Though this method is relatively effective for an element having a high secondary ion emissivity (the ratios of emitted ions to atoms) and having a concentration of several percent to 100 percent in a specimen such as Al, Fe, Cr, Na or K, other elements are only with difficulty observed as images.

2. It is difficult to distinguish between the image of the surface of a specimen and the atomic number contrast due to the concentration of the element because

the contrast due to the unevenness of the surface of the specimen is observed superposed on a particular element.

3. It is not practical to utilize the secondary ion image for the location of the analyzing position because the intensity of signal is low for that purpose.

An object of the present invention is to provide an ion microanalyzer which overcomes the above-described disadvantages of the prior art ion microanalyzer.

According to the present invention the above-described disadvantages are overcome by directly detecting secondary ions emitted by a specimen to supply a video signal.

To achieve this purpose, the present invention provides an ion microanalyzer comprising means for producing an ion beam, means for scanning the surface of a specimen with the ion beam, means for detecting secondary ions emitted by the specimen, a cathode ray tube having means for scanning the electron beam thereof synchronously with the scanning of the ion beam, means for mass-analyzing the secondary ions, and means for modulating the intensity of the electron beam of the cathode ray tube by the detected signal from the secondary ion detecting means.

There is another prior art ion microanalyzer which displays a secondary electron image instead of a secondary ion image. However, the secondary ion emissivity of an element is far higher than the secondary electron emissivity. Consequently, according to the present invention which employs secondary ions a very clear and distinct atomic number contrast can be observed. Also, since a secondary ion image is far less affected by scattered electrons than a secondary electron image, the present invention can provide an essentially low noise image.

An embodiment of the present invention will now be described with reference to FIG. 2. A primary ion irradiation system consists of, similarly to the prior art one described with reference to FIG. 1, an ion source or gun 1, a condenser lens 4, an objective lens 6, and deflection electrodes 5 for scanning. A cathode ray tube 18 for the observation of a specimen image and a mass-spectrometer are the same as those of the prior art.

To directly detect secondary ions 24 emitted from a specimen 7 placed on a support 8, a secondary ion detector consists of a photosensitive surface 20 and an electrode 19 for secondary ion deflection maintained at the same potential as a shielding electrode (mesh or perforated metal electrode) 25 arranged around the specimen 7. Though the shielding electrode 25 is not always necessary, it has the function of maintaining the frontal space of the specimen 7 at zero electric field to prevent an adverse effect by the deflection of secondary ions. In some cases, to perform energy analysis of the secondary ions to be detected a variable electric source 26 is connected to the shielding electrode 25 so that the potential  $V_a$  of the shielding electrode 25 is made variable over a range of from several volts to several hundreds of volts relative to the specimen 7. The photosensitive surface 20 consists of a scintillator coated with an electrically conductive film. The output light of the photosensitive surface 20 is received by a photomultiplier 21 to be subjected to photoelectric conversion. The electric signal produced by the conversion is amplified by an amplifier 22 and modulates the intensity of the electron beam of the cathode ray

tube 18. The photosensitive surface 20 is arranged such that it cannot directly be viewed from the beam irradiated point on the specimen 7 so that the reduction of the sensitivity of the photosensitive surface 24 due to the deposition thereon of particles sputtered by the specimen 7 is prevented. For this purpose an electric source 27 is connected to the electrically conductive film on the photosensitive surface 20 so that a negative potential  $-V_s$  is applied thereto relative to the opposing electrode 19 when positive secondary ions are to be detected, and a positive potential  $+V_s$  is applied thereto when negative secondary ions are to be detected to deflect the ions so that they impinge upon the photosensitive surface 20.

Instead of the scintillator and photomultiplier 21 a secondary electron multiplier may be used.

By the above-described structure according to the present invention an amount of secondary ions about  $10^3$  times as high as that provided by a prior art apparatus could be detected for primary argon ions  $Ar^+$  having an energy of 10 Kev and an ion current of  $10^{-8}$  A. As a result, a clear and distinct secondary ion image of a resolution of 0.5 micron or less could be observed on the cathode ray tube.

The thus obtained secondary ion image represents the atomic number contrast between different kinds of elements present in the surface of a specimen, which could not be observed by the prior art apparatus.

In the apparatus according to the present invention the output of the secondary electron multiplier 14 may be superimposed on the output of the amplifier 22 through a comparator 28. By doing so an element having a particular mass, for example  $M_3$ , can be specially brightly displayed on a cathode ray tube as shown in FIG. 3. Consequently, an image of the entire surface of a specimen can be displayed brightly irrespective of the composition of the specimen surface, and yet it is possible by scanning the mass-spectrometer to know which regions of the image corresponds to what mass. In FIG. 2, reference numeral 23 designates a power source for scanning.

FIG. 4 is a photograph of a secondary ion image of a specimen consisting of an iron plate and an aluminum film evaporated thereon to a thickness of about 200 Angstroms. The atomic number contrast between aluminum and iron is clearly observed in FIG. 4.

What we claim is:

1. An ion microanalyzer comprising:  
 means for producing an ion beam;  
 means for scanning the surface of a specimen with said ion beam;  
 means for directly detecting a first portion of the secondary ions emitted from said specimen;  
 a cathode ray tube having means for scanning the electron beam thereof synchronously with the scanning of said ion beam;  
 a mass spectrometer for mass-analyzing a second portion of said secondary ions and for providing a signal representative of the mass-analysis;  
 means for modulating the intensity of the electron beam of said cathode ray tube by the detected signal from said means for directly detecting a first portion of the secondary ions emitted from said specimen; and  
 means for superimposing the signal obtained from said mass spectrometer on the signal obtained from

said means for directly detecting a first portion of the secondary ions emitted from said specimen.

2. An ion microanalyzer according to claim 1, further, comprising shielding means surrounding the specimen.

3. An ion microanalyzer according to claim 2, in which the secondary ion detecting means is provided outside the shielding means.

4. An ion microanalyzer according to claim 1, wherein said means for directly detecting a first portion of the secondary ions emitted from said specimen is a secondary electron multiplier.

5. An ion microanalyzer according to claim 1, further comprising deflection means for deflecting the secondary ions to direct them to said means for directly detecting a first portion of the secondary ions emitted from said specimen.

6. An ion microanalyzer according to claim 5, wherein said deflection means for deflecting the secondary ions consists of a pair of electrodes one of which is made of a conductive film deposited on a scintillator, and said means for directly detecting a first portion of the secondary ions emitted from said specimen is constituted by said deflection means and a photomultiplier opposed to said scintillator.

7. An ion microanalyzer according to claim 6, wherein said ion microanalyzer includes means for maintaining one of said electrodes at the same electric potential as said specimen and shielding means surrounding said specimen, and further includes means for changing over the polarity of an electric potential applied to the other of said electrodes.

8. In an ion microanalyzer including:

first means for producing an ion beam;

second means for scanning the surface of a specimen with said ion beam;

a cathode ray tube having means for scanning the electron beam thereof in synchronism with the scanning of said ion beam; and

third means receiving a principal ion beam which includes a portion of the secondary ions emitted from said specimen for mass-analyzing said principal ion beam and for providing a first signal representative of the mass-analysis thereof;

the improvement comprising

fourth means, disposed separately from said third means, for directly detecting secondary ions emitted from said specimen separate from the portion of the secondary ions included in said principal ion beam and for providing a second signal representative of the directly detected secondary ions; and

fifth means, coupled to said fourth means and said cathode ray tube for modulating the intensity of said electron beam in accordance with said second signal provided by said fourth means, and including means for superimposing said first signal on said second signal for modulating the intensity of said electron beam in accordance with said superimposed signals.

9. The improvement according to claim 8, wherein said fourth means comprises a secondary electron multiplier.

10. The improvement according to claim 8, wherein said third means comprises a mass analyzer and a comparator having first and second inputs and an output, said first input being connected to said mass analyzer,

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said second input being connected to a reference voltage and said output being coupled to the output of said fourth means.

11. The improvement according to claim 8, wherein said fourth means comprises deflection means for deflecting the secondary ions and directing the secondary ions into an ion detector.

12. The improvement according to claim 11, wherein said deflection means comprises a pair of electrodes, one of which is made of a conductive film deposited on a scintillator, and said ion detector comprises a photomultiplier disposed opposite said scintillator.

13. The improvement according to claim 12, further comprising shielding means surrounding said specimen with said fourth means located outside said shielding means.

14. The improvement according to claim 13, further comprising means for maintaining one of said electrodes at the same electric potential as said specimen and means for reversing the polarity of the electric potential applied to the other of said electrodes.

15. A method of analyzing a specimen surface by an

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ion microanalyzer comprising the steps of:

- a. generating a primary ion beam;
- b. continuously scanning a specimen surface with said primary ion beam;
- c. directly detecting a first portion of the secondary ions emitted from said specimen;
- d. scanning the electron beam of a cathode ray tube synchronously with the scanning of said primary ion beam;
- e. mass-analyzing a second portion of the secondary ion emitted from said specimen and providing a signal representative thereof;
- f. modulating the intensity of the electron beam of said cathode ray tube by the detected signal of said mass-analyzed secondary ions; and
- g. superimposing the signal obtained from said mass-analyzing of said second portion of the secondary ions on the signal obtained by directly detecting the first portion of the secondary ions emitted from the specimen.

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UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

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Inventor(s) Hifumi TAMURA, Toshio KONDO

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Title page as it reads now:

[30] Foreign Application Priority Data

March 17, 1972 Switzerland.....26561/72

Title page as it should read:

[30] Foreign Application Priority Data

March 17, 1972 Japan.....26561/72

**Signed and Sealed this**

*eighteenth Day of November 1975*

[SEAL]

*Attest:*

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