

[54] ION MICROPROBE ANALYSER

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[52] U.S. Cl. 250/309; 250/288; 250/306

[51] Int. Cl. H01J 37/00

[58] Field of Search 250/307, 309, 310, 288

[56] References Cited

UNITED STATES PATENTS

| | | | |
|-----------|---------|-------------------|---------|
| 3,219,817 | 11/1965 | Möllenstedt | 250/309 |
| 3,517,191 | 6/1970 | Liebl..... | 250/309 |
| 3,567,927 | 3/1971 | Barrington | 250/309 |
| 3,635,561 | 1/1972 | Bordonali | 250/309 |
| 3,665,182 | 5/1972 | Goff et al..... | 250/309 |

OTHER PUBLICATIONS

"Utilization of the Ion Analyzer for the Study of Oxygen Diffusion in Solids and Its Application to Zirconium," Quataert et al., Jour. of Nuclear Mat., 1970, Vol. 36, No. 1, pp. 20 to 28, 250-309.

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ABSTRACT

[57] An ion microprobe analyser in which a specimen to be analysed is irradiated with primary ions of non-reactive gas and the mass-to-charge ratio and intensity of secondary ions emitted from the specimen are measured for the qualitative and quantitative analysis of elements constituting the specimen. In the analyser, means for directing a reactive gas toward a desired spot on the surface of the specimen to be analysed is provided so as to eliminate serious limitations and undesirable side effects due to the use of the reactive gas atmosphere thereby improving the sensitivity and quantitativity of analysis.

9 Claims, 12 Drawing Figures

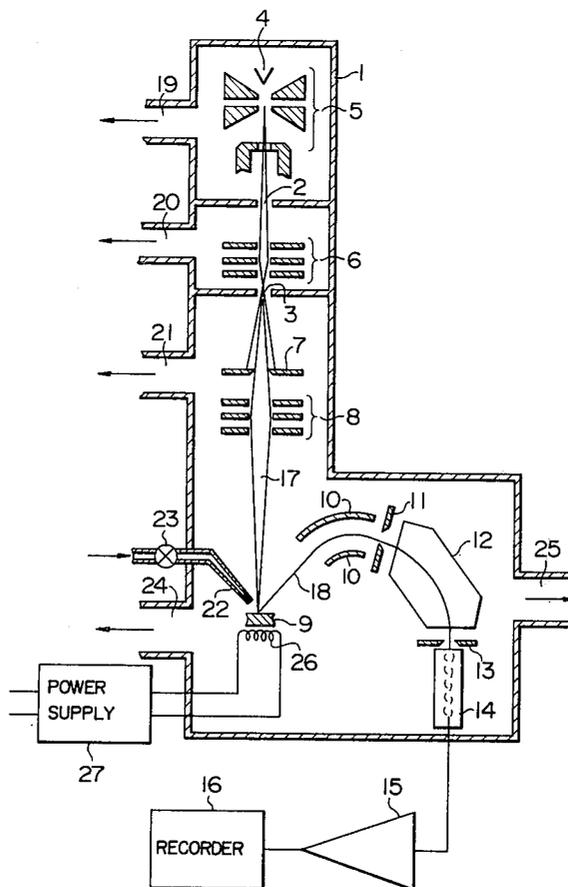


FIG. 2

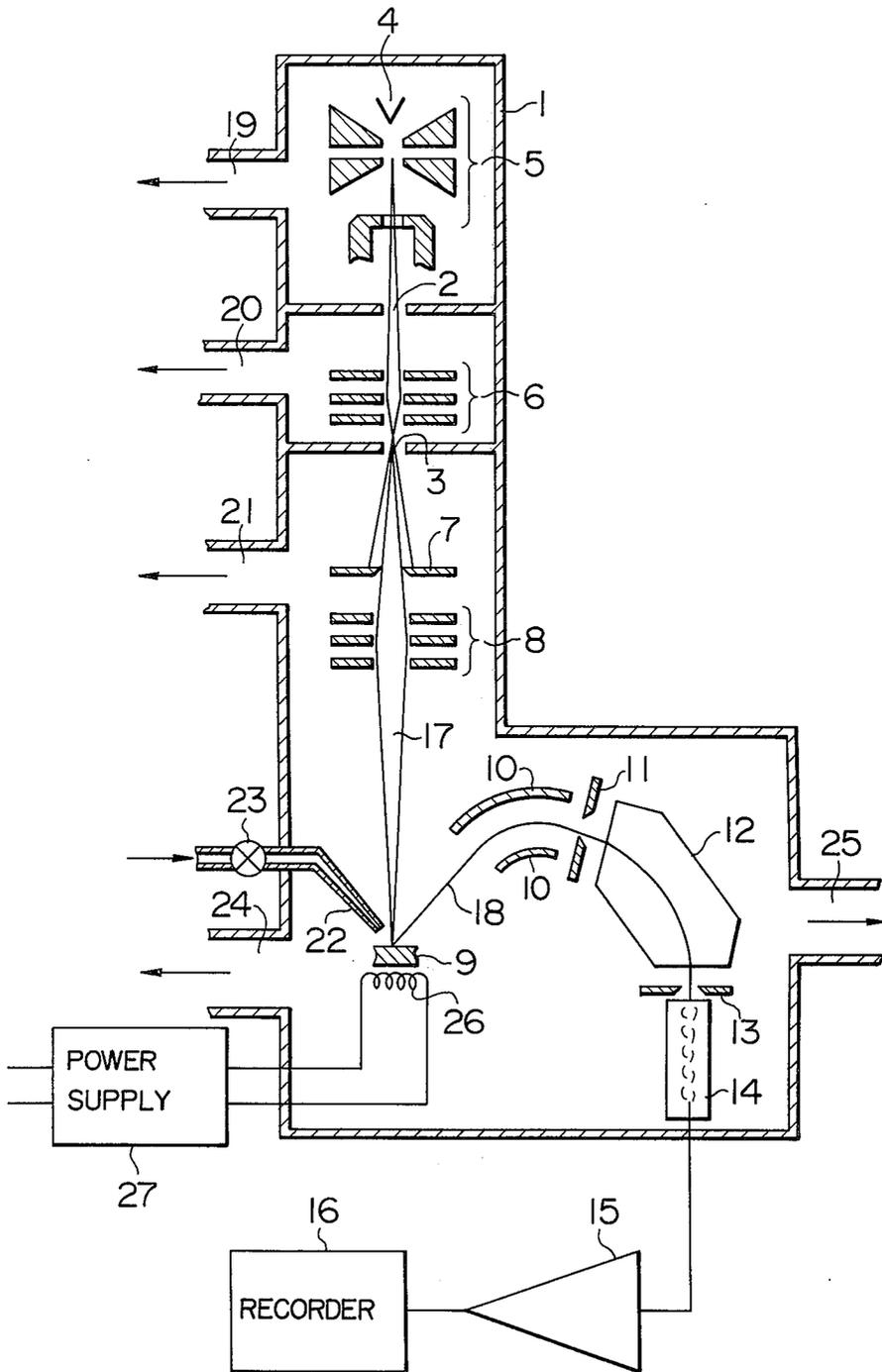


FIG. 3

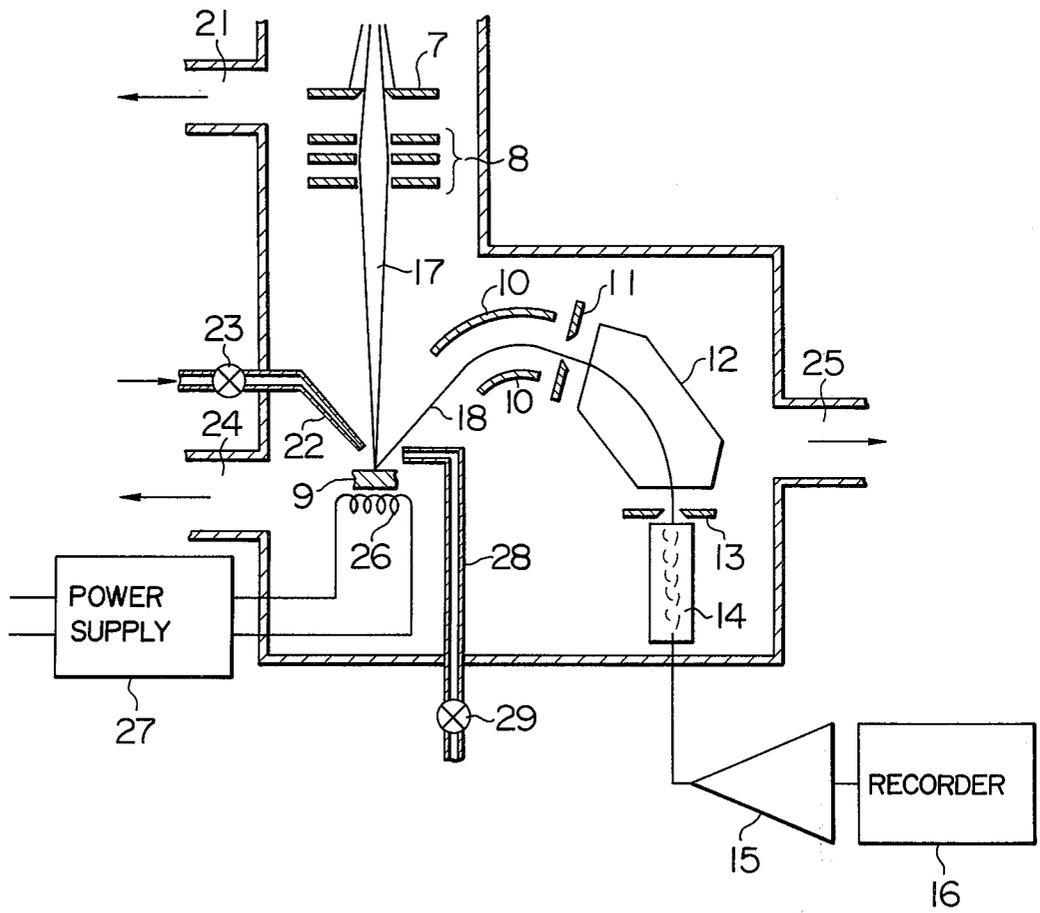


FIG. 5

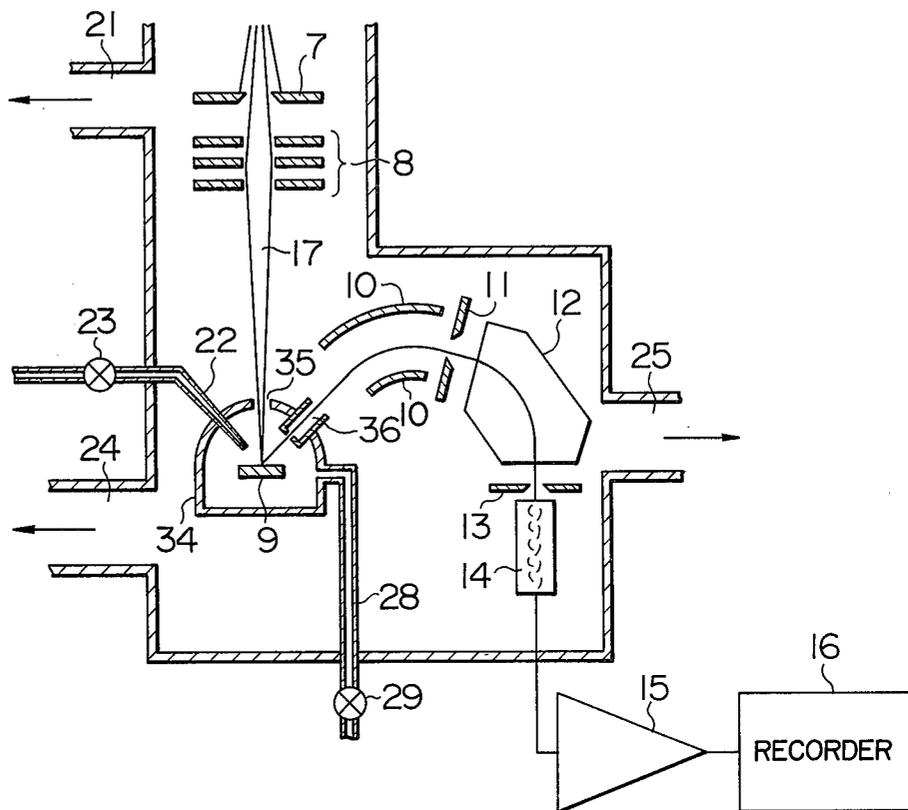


FIG. 6

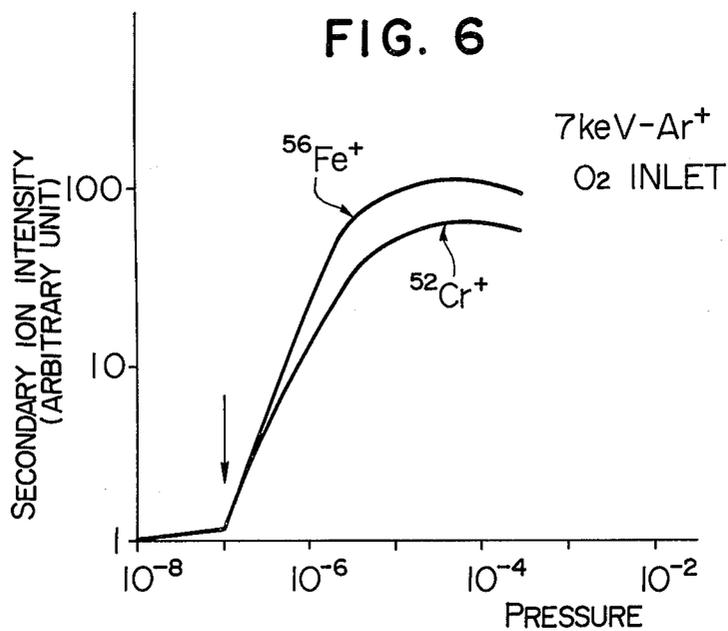


FIG. 7a

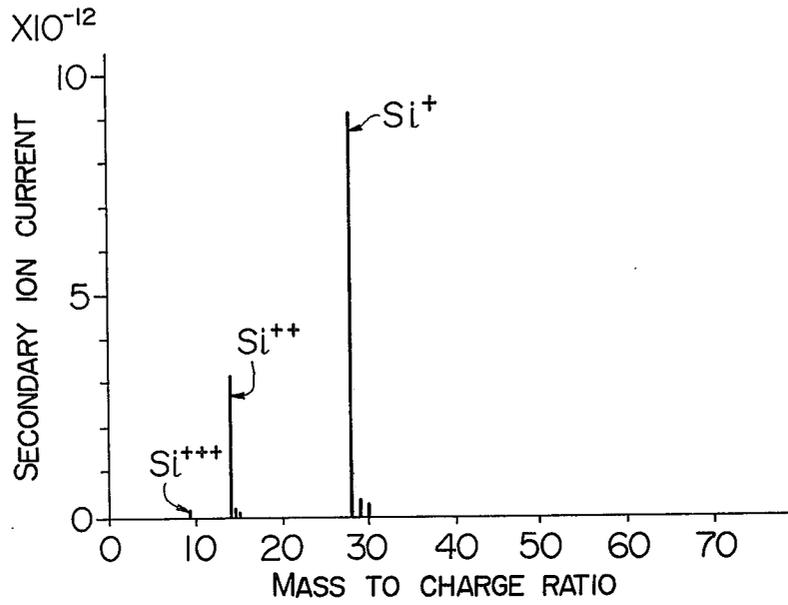


FIG. 7b

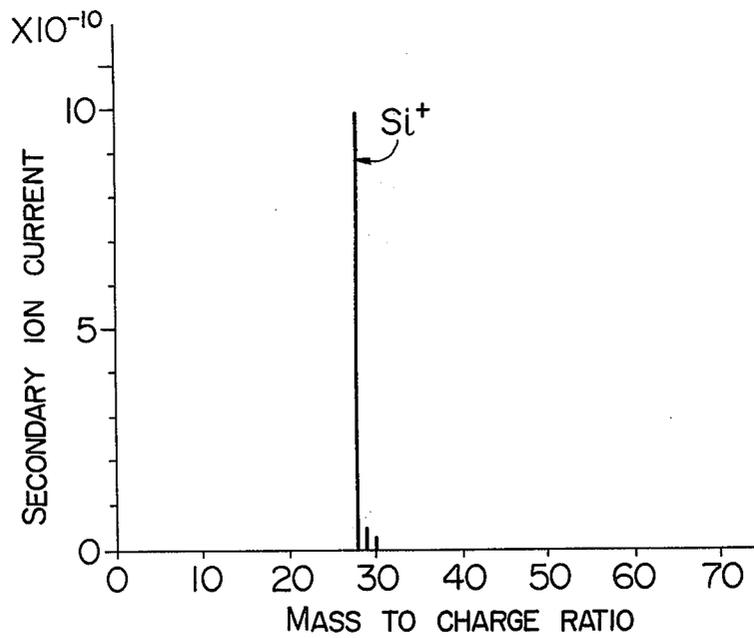


FIG. 8a

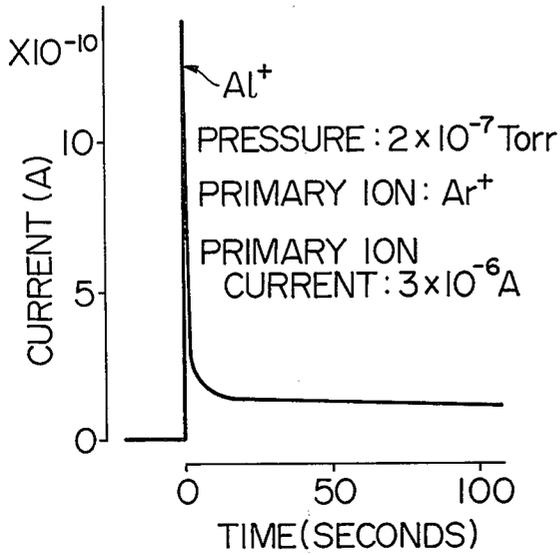


FIG. 8b

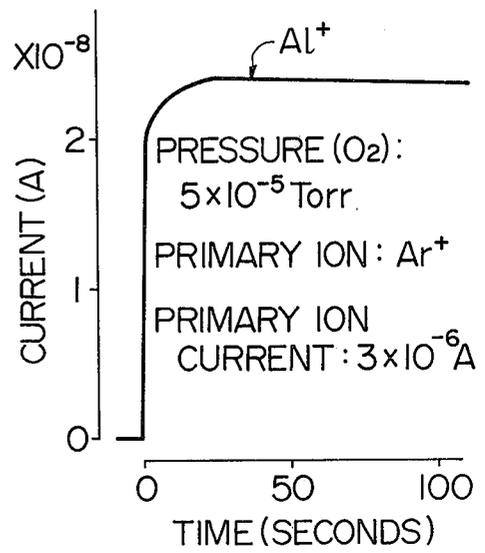


FIG. 9a

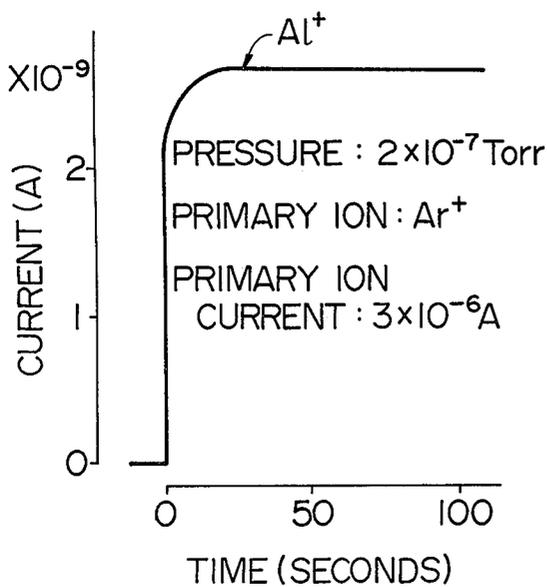
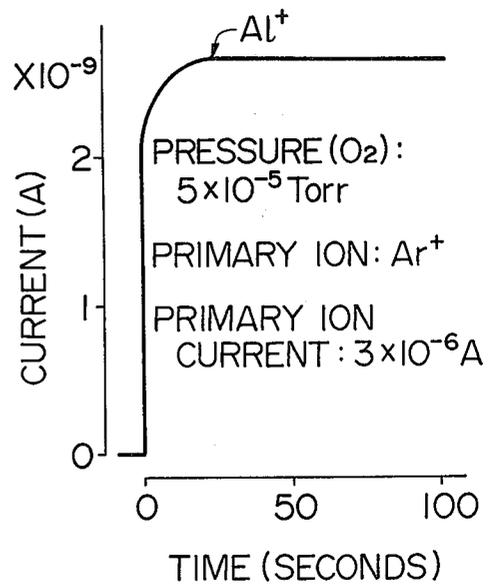


FIG. 9b



ION MICROPROBE ANALYSER

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to ion microprobe analysers and more particularly to an ion microprobe analyser in which desired spot on the surface of a solid specimen to be analysed is irradiated with primary ions of non-reactive gas and secondary ions emitted from the irradiated spot of the specimen are used for the element analysis.

2. Description of the Prior Art

An ion microprobe analyser has the features of being capable of analysis of the thin surface layer of a specimen and capable of measurement of a concentration distribution of a specific ion, that is, an element of the specimen in the depth direction thereof. The analysis of the thin surface layer is a merit not possessed by the other like devices and is profitably put into practical use at present. Such an ion microprobe analyser is used to analyse iron and steel materials, semiconductor materials, surface treating materials, insulator materials, surface pollution, organic materials, and so forth.

FIG. 1 shows schematically the structure of a prior art ion microprobe analyser. This prior art analyser is broadly divided into a primary ion beam focusing system and a mass spectrometric system. The primary ion beam focusing system comprises a filament 4 for emitting an electron beam, an ion gun means 5 for emitting a primary ion beam 17, a condenser lens 6, an objective lens 8 and an objective aperture 7 for focusing the primary ion beam 17 on a solid specimen 9, and a specimen chamber having means for replacing and holding the specimen 9 in position although the details of the specimen replacing and holding means are not shown. The mass spectrometric system comprises an electric sector field 10, a β -slit 11, a magnetic sector field 12, an ion collector slit 13, an ion detector 14, an ion beam amplifier 15 and a recorder 16. The parts except the amplifier 15 and recorder 16 are housed within a vacuum vessel 1, the interior of which is partitioned into a plurality of spaces by partitions provided with respective apertures 2 and 3 having a small evacuating conductance, and these spaces are connected through apertures 19, 20 and 21 to a differential pumping system. The electric sector field formed between a pair of electrodes 10 serves to select the energy of a secondary ion beam 18 emitted from the specimen 9, while the magnetic sector field formed between a pair of pole pieces 12 serves to separate the secondary ion beam 18 according to the mass-to-charge ratio for the element analysis of the specimen 9.

The primary ion beam 17 can be emitted from the ion gun means 5 by initially evacuating the space surrounding the ion gun means 5 to a pressure of 10^{-6} to 10^{-7} Torr, introducing a gas corresponding to a desired ion into the space surrounding the ion gun means 5, and while maintaining the pressure in the space surrounding the ion gun means 5 solely at 10^{-1} to 10^{-2} Torr, causing discharge across the cathode and anode of the ion gun 5 to produce a plasma state. The primary ion beam 17 is extracted by applying an electric field to this plasma. The primary ion beam 17 which is either positive or negative can be freely extracted by applying a positive or negative primary ion extracting voltage. Due to the fact that the space surrounding the ion gun means 5 is connected to the ion accelerating space by the aperture 2 having a small evacuating conductance,

the ion accelerating space can be maintained at a high vacuum so that discharge may not occur even when the gas is introduced into the space surrounding the ion gun means 5.

The primary ion beam 17 obtained in this manner is focused on the specimen 9 by the condenser lens 6 and objective lens 8. Electrostatic lenses of three electrode structure are utilized for the focusing of the primary ion beam 17. In response to the irradiation of a desired spot on the surface of the specimen 9 with the accelerated and focused primary ion beam 17, a secondary ion beam 18 of the specimen atoms is emitted from the specimen 9. The secondary ion beam 18 is directed into the mass spectrometric system where the secondary ion beam 18 is separated according to the mass-to-charge ratio for the element analysis of the specimen 9.

In the prior art ion microprobe analyser having such a structure, ions of non-reactive gas such as argon have been used as the primary ions in order to minimize undesirable contaminating influences in various portions of the analyser.

However, difficulties of major concern have been encountered having to do primarily with sensitivity and stability. It was found that only a small fraction of the sputtered material was in the form of ions available for analysis so that the desired degree of sensitivity could not be achieved. Also, as the bombardment continued the fraction of the sputtered particles that carried electric charges became progressively smaller and the output signal continuously declined, so that in many cases it was extremely difficult, though not impossible, to calibrate the output signals of ion bombardment instruments. They were not stable with respect to time. Further, it was difficult to use such analyser for the quantitative analysis in that the secondary ion yield (the number of secondary ions emitted from the specimen per primary ion) differed depending on the element.

U.S. Pat. No. 3,508,045 discloses a method in which an ion gun of special structure is employed and chemically reactive gas ions such as oxygen ions or halogen ions are used to provide a primary ion beam. According to this method, the secondary ion production efficiency can be improved. However, when such reactive gas ions are used as the primary ions, the cathode of the ion gun is heated to a high temperature resulting in damage to the electrode materials forming the cathode and in an extremely short service life of the ion gun. Thus, this method is defective in that the ion gun must have a special structure. A further serious defect of this method resides in the fact that the secondary ion yield varies within the wide range up to the order of 10^3 depending on elements. Thus, it has been difficult to improve the spectroscopic accuracy of quantitative analysis due to the fact that complex calibration is required for the quantitative analysis and analysis of an element having a low sensitivity is difficult to achieve.

Another proposal has been made according to which a reactive gas to contact and react with the specimen is introduced into a chamber wherein the irradiation is carried out. However, this method is defective in that secondary ions flying within the mass spectrometric system collide with reactive gas molecules and are thereby scattered to lower the resolution and sensitivity, and at the same time, the primary ion beam is also scattered resulting in a reduction of the degree of focusing the ion beam. Therefore, the proposed analyser has not still been put into practical use due to the fact that the primary ion current is severely restricted and

the total number of secondary ions is small.

SUMMARY OF THE INVENTION

It is a primary object of the present invention to provide an improved ion microprobe analyser suitable for the qualitative and quantitative analysis of secondary ions emitted from a solid specimen in response to irradiation with primary ions.

Another object of the present invention is to realize the practical use of an ion microprobe analyser of the type in which a non-reactive gas is used to provide a primary ion beam and a specimen is held in an atmosphere of a chemically reactive gas.

Still another object of the present invention is to provide an ion microprobe analyser which can provide improved sensitivity of analysis.

Yet another object of the present invention is to provide an ion microprobe analyser which can provide improved quantitativity.

A further object of the present invention is to further improve the sensitivity of analysis by providing means for heating the specimen.

The principal feature of the present invention which attains the objects above described resides in the fact that, in an ion microprobe analyser of the kind in which a desired spot on the surface of a solid specimen to be analysed is irradiated with primary ions of non-reactive gas and the mass-to-charge ratio and intensity of secondary ions emitted from the specimen are measured for the qualitative and quantitative analysis of elements constituting the specimen, means are provided for introducing a chemically reactive gas toward the desired analysed spot on the surface of the specimen. Another feature of the present invention resides in the fact that partitions, each of which is provided with an aperture having a small evacuating conductance, are disposed between the space surrounding the specimen and the ion lens system and between the space and the mass spectrometric means so that the space surrounding the specimen, the ion lens system, and the mass spectrometric means can be subjected to differential evacuation independently of each other. Still another feature of the present invention resides in the fact that a casing for confining the reactive gas therein is disposed in the vicinity of the specimen in such a manner that it surrounds the specimen. Yet another feature of the present invention resides in the fact that means for discharging the reactive gas is further provided.

By virtue of such a structure, the reactive gas can be effectively supplied to the desired spot on the surface of the specimen irradiated with the primary ions of non-reactive gas. Therefore, it is unnecessary to supply any excessive amount of reactive gas into the analyser, and as a result, the possibility of undesirable diffusion of the reactive gas toward various other portions of the analyser can be reduced. This is advantageous in that an undesirable reduction of the resolution and sensitivity due to collision of the secondary ions flying within the mass spectrometric system with the reactive gas molecules resulting in scattering of the secondary ions can be avoided, and at the same time, an undesirable reduction of the degree of ion beam focusing due to scattering of the primary ion beam can also be avoided. Further, the severe restriction of primary ion current which has been encountered with the prior art analyser using the reactive gas can also be eliminated with the result that the reactive gas supplied to the specimen can be effectively adsorbed into the surface of the solid

specimen and the secondary ion production efficiency can be remarkably improved.

Therefore, the sensitivity of analysis can be improved by virtue of the improvement in the secondary ion yield which is the merit of causing emission of secondary ions in an atmosphere of reactive gas, and the quantitativity can be improved by virtue of the fact that the difference between the secondary ion yields of different elements can be reduced to within the range below the order of ten. Thus, the ion microprobe analyser provided by the present invention is suitable for the qualitative and quantitative analysis and is especially suitable for the quantitative analysis.

Furthermore, due to the fact that a non-reactive gas is used to provide primary ions, the ion gun need not be of any especial structure. Thus, the ion microprobe analyser according to the present invention has a simple construction, maintenance thereof can be easily accomplished, and the analyser can be easily manufactured at low costs.

Means for heating the specimen may be additionally provided for promoting the reaction between the elements constituting the specimen and the reactive gas so that the sensitivity of analysis can be further improved.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic sectional view showing the structure of a prior art ion microprobe analyser.

FIG. 2 is a schematic sectional view showing the structure of an embodiment of the ion microprobe analyser of the present invention which is provided with means for directing a reactive gas toward a desired spot on the surface of a specimen.

FIG. 3 is a schematic sectional view of parts of another embodiment of the ion microprobe analyser of the present invention in which means for discharging the reactive gas is additionally provided in the device shown in FIG. 2.

FIG. 4 is a schematic sectional view of still another embodiment of the ion microprobe analyser of the present invention in which additional partitions are provided between the space surrounding a specimen and an ion lens system and between the space surrounding the specimen and mass spectrometric means so that the space surrounding the specimen and the mass spectrometric system can be subjected to differential evacuation.

FIG. 5 is a schematic sectional view of parts of yet another embodiment of the ion microprobe analyser of the present invention in which a casing for confining reaction gas therein and having means for directing the reactive gas toward a desired spot on the surface of a specimen is disposed in the vicinity of the specimen.

FIG. 6 shows the technical effect of the present invention and is a graphic representation of the relation between the pressure and the secondary ion intensity.

FIGS. 7a and 7b show also the technical effect of the present invention and are graphic representations of the relation between the mass-to-charge ratio and the secondary ion current.

FIGS. 8a, 8b and FIGS. 9a, 9b show also the technical effect of the present invention and are graphic representations of the relation between the duration of irradiation and the secondary ion current.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

FIG. 2 is a schematic sectional view showing the structure of an embodiment of the ion microprobe analyser according to the present invention and like reference numerals are used therein to denote like parts appearing in FIG. 1. Referring to FIG. 2, the ion microprobe analyser comprises a primary ion beam focusing system, a mass spectrometric system, an ion beam amplifier 15 and a recorder 16. The primary ion beam focusing system includes a filament 4 for emitting an electron beam, an ion gun means 5, a condenser lens 6, an objective lens 8, an objective aperture 7, a nozzle 22 for supplying a jet of reactive gas, and a valve 23 in the reactive gas supply conduit. The mass spectrometric system includes a pair of electrodes 10 for forming an electric sector field, a β -slit 11, a pair of pole pieces 12 for forming a magnetic sector field, an ion collector slit 13, and an ion detector 14. The parts except the ion current amplifier 15 and the recorder 16 are housed within a vacuum vessel 1, the interior of which is partitioned into a plurality of spaces by partitions provided with respective apertures 2 and 3 having a small evacuating conductance, and these spaces are connected to a differential pumping system through apertures 19, 20, 21, 24 and 25 for differential evacuation. A heater 26 for heating a solid specimen 9 and a power supply 27 for the heater 26 are additionally provided, but these means are not essentially required.

In the ion microprobe analyser having such a structure, the reactive gas supply nozzle 22 is disposed in such a manner that the nozzle tip thereof is directed toward the spot on the surface of the specimen 9 irradiated with the primary ion beam. In operation, the valve 23 in the reactive gas supply conduit is opened to supply a jet of reactive gas from the reactive gas supply nozzle 22 onto the spot on the surface of the specimen 9 irradiated with the primary ion beam 17 focused by the primary ion beam focusing system. A secondary ion beam 18 emitted from the specimen 9 is analysed by the mass spectrometric system and the result of analysis is displayed on the recorder 16. The reactive gas jetted onto the surface of the specimen 9 from the reactive gas supply nozzle 22 diffuses within the vessel 1 to raise the pressure within the vessel 1. The apertures 24 and 25 are provided so that this undesirable build-up of pressure and contamination of the parts within the vessel 1 can be prevented.

FIG. 3 shows a modification of the embodiment shown in FIG. 2. Referring to FIG. 3, a reactive gas discharge conduit 28 having its end opening disposed adjacent to the irradiated spot on the surface of the specimen 9 is additionally provided so that diffusion of the reactive gas toward the other parts within the vessel 1 can be more effectively prevented. This reactive gas discharge conduit 28 is connected through a valve 29 to an external evacuating system.

FIG. 4 shows another embodiment of the ion microprobe analyser according to the present invention and like reference numerals are used to denote like parts appearing in FIG. 2. Referring to FIG. 4, an additional partition 31 provided with an aperture 30 having a small evacuating conductance is disposed between an objective lens 8 and the space surrounding a specimen 9 within a vacuum vessel 1, and another additional partition 33 provided with an aperture 32 having a small evacuating conductance is disposed between a

mass spectrometric system and the space surrounding the specimen 9. By virtue of the provision of these partitions 31 and 33, a reactive gas supplied from a nozzle 22 can be prevented from diffusing toward the objective lens 8 and mass spectrometric system, and secondary ions that may be emitted in very small amounts can be prevented from being scattered.

FIG. 5 shows a still another embodiment of the ion microprobe analyser according to the present invention and like reference numerals are used to denote like parts appearing in FIG. 2. Referring to FIG. 5, a substantially hemispherical casing 34 surrounds a specimen 9 within a vacuum vessel 1. A nozzle 22 extends into the casing 34 for supplying a jet of reactive gas toward a desired spot on the surface of the specimen 9, and a primary ion beam inlet aperture 35 and a secondary ion beam outlet aperture 36 are bored in the wall of the casing 34. The interior of the casing 34 is connected by a reactive gas discharge conduit 28 and a valve 29 to an external evacuating system so that diffusion of the reactive gas toward the other parts within the vessel 1 can be effectively prevented. However, this reactive gas discharge means 28 is not an essential part and the desired objects can be attained without the provision of such discharge means 28. The structure shown in FIG. 5 is advantageous in that the pressure of reactive gas within the casing 34 can be remarkably increased compared with that in the preceding embodiments, and the gas pressure may be about 100 times that in the preceding structures. Further, due to the fact that the casing 34 may have a small internal volume, the required amount of reactive gas supplied into the vessel 1 can be considerably reduced to lessen the adverse effects due to diffusion of the reactive gas. Further, partitions each provided with an aperture having a small evacuating conductance may be disposed between an objective lens 8 and the space surrounding the specimen 9 and between a mass spectrometric system and the space surrounding the specimen 9 respectively in the ion microprobe analyser shown in FIG. 5 so as to further improve the effect of preventing undesirable diffusion of the reactive gas.

In FIGS. 2, 3 and 4, the heater 26 and the heater power supply 27 are provided to heat the specimen 9 in the atmosphere of reactive gas so that a substantially complete chemical compound can be formed on the specimen surface by the reaction between the reactive gas and the specimen. The provision of such specimen heating means is advantageous in that the secondary ion yield can be increased compared with the case in which the specimen is placed in an atmosphere of room temperature and the sensitivity of analysis and spectroscopic accuracy can be improved. Although an indirect heating means (resistance heating means) using a heater is shown in FIGS. 2, 3 and 4, any other suitable heating means such as an electron impact heating means (direct heating means) may be employed in lieu of the indirect heating means.

Further, although a nozzle is used to supply a reactive gas in the foregoing embodiments, other suitable reactive gas supplying means such as a tubular pipe may also be employed.

The technical effects of the present invention will now be described.

The conditions of primary ions used for irradiating a specimen of iron alloy are as follows:

| | |
|---------------------------|------------------------|
| Primary ion | Ar ⁺ |
| Primary ion energy | 10 KeV |
| Primary ion beam diameter | 100μm |
| Primary ion current | 1 × 10 ⁻⁶ A |

The secondary ion yield is measured for the comparison's sake in a conventional argon atmosphere in which, although argon is not especially introduced, argon leaks little by little from the ion gun and the partial pressure of argon occupies substantially the space surrounding the specimen. Then, the secondary ion yield is measured according to the present invention by initially evacuating the specimen chamber or the space surrounding the specimen to a pressure of 10⁻⁷ Torr and subsequently regulating the amount of oxygen supplied from the nozzle until the reading of an ionization gage provided at the outlet of the specimen chamber gives a pressure of 10⁻⁵ Torr. In this latter case, the pressure of oxygen in the vicinity of the nozzle tip, hence in the vicinity of the specimen surface, is presumed to be of the order of 10² Torr. Table 1 shows the results of measurement of the secondary ion yield of various elements contained in the iron alloy under the conditions above specified. Table 1 shows the relative secondary ion yield of the elements other than iron when the secondary ion yield of iron is taken as unity.

Table 1

| Element | Relative sensitivity | | |
|---------|---|--|--|
| | Ar atmosphere (conventional) 2 × 10 ⁻⁷ Torr Secondary ion yield of Fe taken as unity | O ₂ atmosphere (present invention) 8 × 10 ⁻⁵ Torr Secondary ion yield of Fe taken as unity | Conventional secondary ion yield of Fe taken as unity |
| Fe | 1.0 | 1.0 | 76 |
| Si | 0.6 | 0.5 | 38 |
| V | 32 | 3.5 | 270 |
| Cr | 14 | 3.1 | 240 |
| Mn | 11 | 1.7 | 130 |
| Ni | 0.25 | 0.41 | 31 |
| Cu | 0.12 | 0.15 | 11 |
| Mo | 3.1 | 0.95 | 72 |

The absolute value of the secondary ion yield in the conventional argon atmosphere is about 1/100 of that in the oxygen atmosphere employed in the present invention. This means that the sensitivity of analysis can be improved to a value which is about 100 times that in the conventional atmosphere by virtue of the introduction of oxygen, and the analyser according to the present invention is remarkably improved over the prior art analyser.

Further, when reactive gas ions are utilized as primary ions as in conventional practice or when the specimen is irradiated with non-reactive gas ions in a conventional argon atmosphere as shown in Table 1, the secondary ion yield differs over a wide range depending on elements resulting in degradation of the spectroscopic accuracy of analysis of the elements whose secondary ion yield is relatively low. According to the method of the present invention, however, it is apparent from Table 1 that the difference in the secondary ion yield can be remarkably reduced compared with the prior art method and is less than the prior art difference by the order of 10².

In an experiment conducted with the ion microprobe analyser shown in FIG. 3, Ar⁺ ions having ion energy of 7 KeV were used to provide a primary ion beam and O₂ was supplied as a reactive gas to the surface of a specimen, which was an alloy of iron and chromium, for the analysis of this alloy. FIG. 6 shows the relation between the secondary ion intensity of the noted ions Cr⁺ and Fe⁺ and the pressure in Torr of O₂. In this experiment, the reactive gas supplied from a gas source was fed through a pressure reducing valve and a needle valve to be jetted onto the specimen surface from the nozzle which had an inner diameter of 0.2 mm and a length of 200 mm.

FIGS. 7a and 7b show mass spectra of a specimen of silicon. More precisely, FIG. 7a shows the secondary ion intensity of silicon placed in a conventional atmosphere having a pressure of 2 × 10⁻⁷ Torr, and FIG. 7b shows the secondary ion intensity of silicon placed in an oxygen atmosphere having a pressure of 5 × 10⁻⁵ Torr. In FIGS. 7a and 7b, the horizontal axis represents the mass-to-charge ratio and the vertical axis represents the secondary ion current in amperes. It will be apparent from comparison between these spectra that supply of oxygen (O₂) to the surface of the specimen is advantageous in that (1) the Si⁺ ion intensity can be increased by about 100 times and the minimum sensitivity of detection can be correspondingly increased, and (2) the multivalent ions of silicon such as Si⁺⁺ (divalent ions of silicon) and Si⁺⁺⁺ (trivalent ions of silicon) disappear substantially from the spectrum. Therefore, the present invention is advantageous among others in that the mass spectra is simplified to simplify the analysis of the spectrum. Similar tendency is also seen in many elements such as aluminum, boron, iron, nickel, chromium, copper, molybdenum and manganese beside silicon. For example, the lowest detectable limit of boron contained in silicon can be improved to 2 × 10¹³ atoms/cm³ compared with the prior art value of 1 × 10¹⁵ atoms/cm³ and thus the detection sensitivity can be remarkably improved.

FIGS. 8a, 8b and FIGS. 9a, 9b are graphs showing other technical effects of the present invention. In these figures, the horizontal axis represents the duration of irradiation with primary ions in seconds and the vertical axis represents the ion current in amperes. FIG. 8a shows the variation of ion current relative to time of Al⁺ ions produced when a specimen of aluminum placed in a high vacuum of 2 × 10⁻⁷ Torr was irradiated with a primary ion beam of Ar⁺ ions. It will be seen that in FIG. 8a that, although the yield of Al⁺ ions is high in the period of time of about 10 seconds after irradiation with the primary ion beam due to the influence of the gas absorption layer in the surface of the specimen, the yield is subsequently settled at a constant value due to the fact that the gas absorption layer is subjected to ion etching. FIG. 8b shows the variation of ion current relative to time of Al⁺ ions produced when a specimen of aluminum was placed in a high vacuum of 2 × 10⁻⁷ Torr and then, while supplying a reactive gas, oxygen, to the surface of the specimen to establish a pressure of 5 × 10⁻⁵ Torr around the specimen, the specimen was irradiated with a primary ion beam of Ar⁺ ions. It will be seen in FIG. 8b that the ion current value of Al⁺ ions is about 100 times that in FIG. 8a and is gradually increased with the lapse of time without being decreased until finally it is settled at a constant value. Thus, the sensitivity of analysis can be remarkably improved. FIGS. 9a and 9b show the variation of ion

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current relative to time of Al⁺ ions when specimens of Al₂O₃ placed in a high vacuum and in any oxygen atmosphere respectively were irradiated with a primary ion beam of Ar⁺ ions. It will be seen in FIGS. 9a and 9b that the variation of the Al⁺ ion current relative to time shown in FIG. 9a is substantially similar to that shown in FIG. 9b. Thus, it can be seen that there is not any substantial scattering of the primary and secondary ions due to the supply of oxygen.

While the invention has been described by reference to particular embodiments thereof, it will be understood that numerous and further modifications may be made therein by those skilled in the art without actually departing from the spirit of the invention. We, therefore, aim in the appended claims to cover all such equivalent variations as come within the true spirit and scope of the foregoing disclosure.

We claim:

1. An ion microprobe analyser comprising an ion gun for emitting a primary ion beam of non-reactive gas, at least one ion lens for focusing said primary ion beam to irradiate a desired analysed spot on the surface of a specimen with said primary ion beam, mass spectrometric means for analysing the mass-to-charge ratio of a secondary ion beam emitted from the specimen due to irradiation with said primary ion beam, means for detecting the result of analysis by said mass spectrometric means, and means for supplying a chemically reactive gas toward the desired analysed spot on the surface of said specimen.

2. An ion microprobe analyser as claimed in claim 1, wherein means is provided for heating said specimen.

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3. An ion microprobe analyser as claimed in claim 1, wherein means for discharging said reactive gas is disposed in the vicinity of said desired analysed spot on the surface of said specimen.

4. An ion microprobe analyser as claimed in claim 1, wherein partitions each provided with an aperture having a small evacuating conductance are disposed between the space surrounding said specimen and said ion lens and between the space surrounding said specimen and said mass spectrometric means respectively, and differential pumping means are provided for the differential evacuation of said space surrounding said specimen and said mass spectrometric means.

5. An ion microprobe analyser as claimed in claim 1, wherein a casing is disposed in the vicinity of said specimen in such a manner that it surrounds said specimen and confines the reactive gas therein, said reactive gas supplying means being connected to said casing to supply the reactive gas into said casing, and a primary ion beam inlet opening and a secondary ion beam outlet opening are bored in the wall of said casing.

6. An ion microprobe analyser as claimed in claim 5, wherein said casing for confining said reactive gas therein is provided with gas discharging means.

7. An ion microprobe analyser as claimed in claim 1, wherein said reactive gas supplying means is a nozzle connected to a reactive gas source.

8. An ion microprobe analyser as claimed in claim 2, wherein said reactive gas supply means is a nozzle connected to a reactive gas source.

9. An ion microprobe analyser as claimed in claim 4, wherein said reactive gas supplying means is a nozzle connected to a reactive gas source.

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