

Dec. 5, 1967

I. HOUBART

3,356,844

ROTATABLE SAMPLE CHANGER FOR AN ELECTRON PROBE
MICROANALYZER HAVING MEANS FOR MAINTAINING
A VACUUM IN THE ANALYSIS CHAMBER

Filed June 26, 1964

2 Sheets-Sheet 1

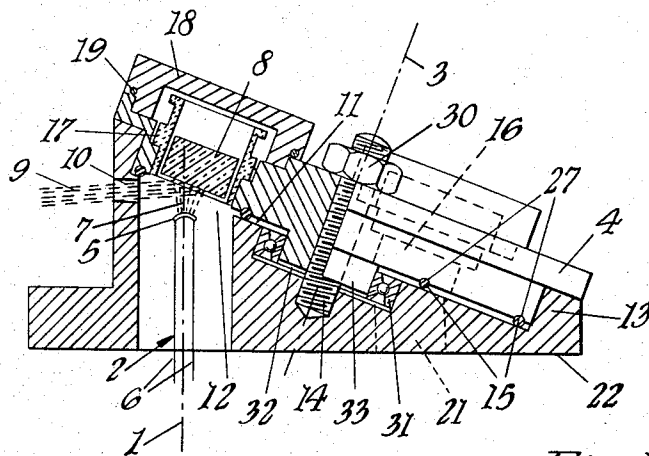


Fig. 1.

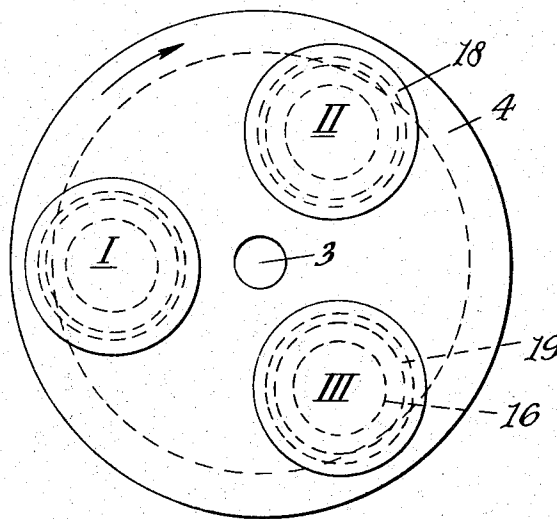


Fig. 2.

Dec. 5, 1967

I. HOUBART

3,356,844

ROTATABLE SAMPLE CHANGER FOR AN ELECTRON PROBE
MICROANALYZER HAVING MEANS FOR MAINTAINING
A VACUUM IN THE ANALYSIS CHAMBER

Filed June 26, 1964

2 Sheets-Sheet 2

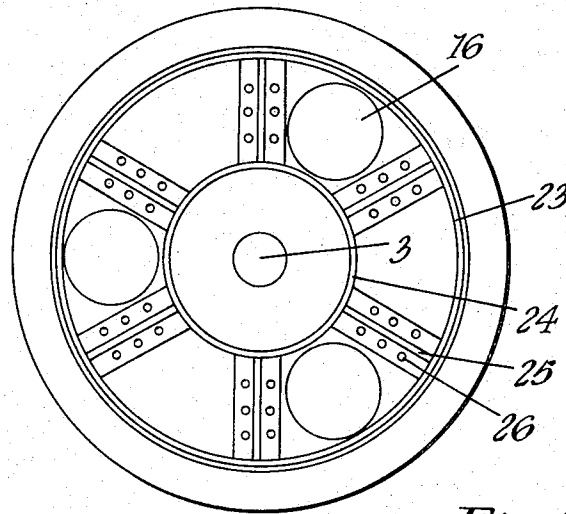


Fig. 3.

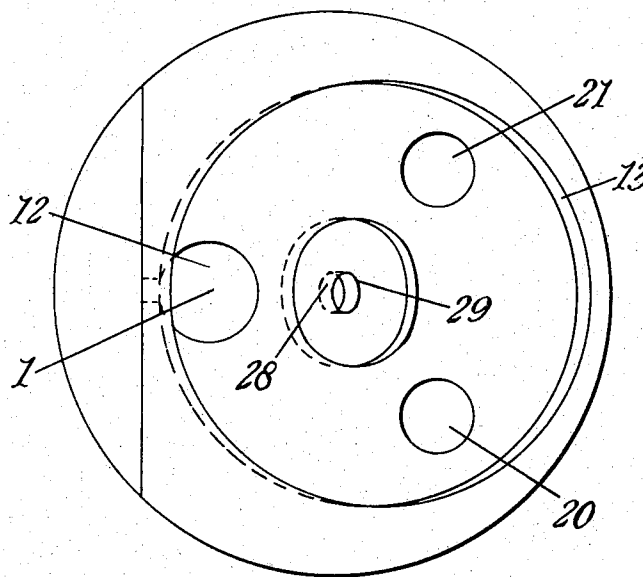


Fig. 4.

1

3,356,844

ROTATABLE SAMPLE CHANGER FOR AN ELECTRON PROBE MICROANALYZER HAVING MEANS FOR MAINTAINING A VACUUM IN THE ANALYSIS CHAMBER

Ivan Houbart, Chenec, Belgium, assignor to Centre National de Recherches Metallurgiques, Brussels, Belgium

Filed June 26, 1964, Ser. No. 378,182

Claims priority, application Belgium, July 2, 1963, 634,419

1 Claim. (Cl. 250—49.5)

ABSTRACT OF THE DISCLOSURE

This invention relates to an electronic bombardment analysing device comprising a body with an enclosure and orifice and having an outer flat surface around the orifice, evacuating means for the enclosure, an electron source to bombard samples held in the orifice, and a rotatable support mounted in juxtaposition to the flat surface with apertures and having gaskets mounted thereon. These gaskets are a pair of concentric rings with an annular space between and radial strips dividing the annular space into twice as many sectors as there are apertures. The apertures are wholly within a sector, and the gaskets are in sealing engagement with the flat surface to provide an air tight seal when the enclosure is evacuated.

This invention relates to a method of analysis by electromagnetic radiation applicable more particularly to the case of analysis by X-rays.

According to a known method of radiographic analysis, a sample is bombarded under a high vacuum (about 10^{-5} mm. Hg) by means of a beam of electrons of sufficient energy (for example 10 kv.) to cause directly from the sample the appearance of an emitted X radiation which can be analysed by a method known per se. This latter method has the advantage of being particularly sensitive, but also the disadvantage of requiring the vacuum to be broken at every change of sample and of having to wait until the vacuum has been reestablished before carrying out a fresh measurement. This putting of the enclosure under vacuum again requires too long a time (several minutes) to be compatible with the demands for rapidity of industrial control.

An object of the present invention is to provide a sample holder enabling this disadvantage to be avoided.

The device according to the present invention is characterised substantially in that it comprises: an enclosure, a device capable of producing in the said enclosure a very high vacuum (at least 10^{-5} mm. Hg), an electron-emitting device in the enclosure, imparting to the electrons a kinetic energy capable of producing X-rays on a sample struck by the said electrons, an orifice formed in the enclosure for positioning the sample, a support on which are fixed one or more units each composed of a lid, a sample and its sample holder, each lid being fixed in an air-tight manner to the support, and the support being able to slide in an air-tight manner over the outer wall of the enclosure by means of a hermetic seal preferably solid with the said support, which may occupy with respect to the said orifice fixed positions, such that each sample holder may in turn be placed facing the said orifice and the sample subjected in this position to electronic bombardment, and finally a device for putting under a high vacuum at least one of the spaces existing between one lid and the wall of the enclosure.

The accompanying drawings will make it possible to better understand the invention.

These drawings are not given to scale and are merely by way of non-restricting example.

2

FIGURE 1 shows diagrammatically a vertical section through the device according to the invention, the plane of which passes through the vertical axis 1 of the electron gun in an enclosure 2, and the axis 3 of rotation of a support in the form of a plate 4. The electron gun comprises the emitting filament 5 supplied by the wires 6. Under the influence of a considerable difference in potential (for example 10 kv.) not illustrated in the drawings, the electrons emitted by the filament are directed upwards in a beam 7 on to the sample 8, on which they produce a radiation, a beam 9 of which escapes through a window 10 out of the enclosure 2. This enclosure 2 under low pressure (the vacuum generator is not shown in the diagram) comprises towards the top an orifice 12 limited by the incline 11.

Above the incline 11 and outside the enclosure 2, the plate 4 which is in the form of a circular disc rotates about its own axis 3, centred on a pivot 14 and in an annular shoulder 13. The plate 4 rests in an air-tight manner on the face of the incline 11 through a complex seal shown by the dots 15. The plate 4 is provided with three holes 16 arranged symmetrically with respect to the axis 3 and at 120° from one another. These holes each serve as a housing for a sample holder 17 and are covered with a lid 18, the edges of which rest in an air-tight manner on the plate through a seal 19. The references 20 and 21 show conduits formed in the mass of the casing, to which conduits will be connected below the inner face 22 the group of devices for putting under a high vacuum (10^{-3} mm. Hg.).

FIGURE 2 shows, as viewed in a downward direction, the plate 4. One can see the three lids 18 at 120° from one another, the axis 3, the outline of the seals 19 and holes 16 in which the sample holders are housed.

FIGURE 3 shows the same support 4 but viewed in an upward direction. One can see the three holes 16, the axis 3 and the seal 15 composed of the circumference 23 outside the holes 16 and the circumference 24 inside the said holes and six radii 25 connecting the two circumferences 23 and 24 to one another, dividing the ring into six equal parts. The radii 25 are fixed to the support 4 by screws 26. The two seals in the form of a circumference are each housed in a small circular groove 27 formed in the support.

FIGURE 4 shows a view in a downward direction of the casing surrounding the enclosure 2 after the support 4 has been raised. There can be seen the axis 1, the path 28 of the axis 3, the housing 29 of the pivot 14, the circular shoulder 13, the orifice 12 formed in the incline 11, the orifices of the conduits 20 and 21 in the mass of the casing.

The whole device functions as follows. During a measuring operation, one of the three sample holders is in position I (FIGURE 2) where it is subjected to electronic bombardment, the second sample holder is in position II above the conduit 21 where it is removed and replaced with another and then subjected to a high vacuum. These three sample holders are insulated from one another from the pressure point of view by means of the seals which surround them completely. Once the measurement is terminated, the support 4 is subjected to a rotation of 120° about its own axis 3 in the direction of the arrow without the three samples ceasing to be insulated from the pressure point of view. The sample holders which occupied the positions I, II, III, occupy respectively at this moment the positions II, III, I. A new measurement may take place immediately because the loss of vacuum in the enclosure is minimum having regard to its connection to the sample holder coming from the position III; it is sufficient to keep in operation the pump serving to create the vacuum of 10^{-5} mm. Hg in the enclosure. During this time the sample coming from I is retained at II

where temporary return to atmospheric pressure makes it possible to remove it and replace it with another. During this time also, the sample coming from II (cavity under primary vacuum) is located at III where it is subjected to a vacuum of 10^{-5} mm. Hg preparatory to its introduction into the enclosure 2. The cycle of operations continues by rotation.

During the rotating of the support 4, the circular seals 23 and 24 move with the said support without being deformed by means of the grooves 27; the radial seals 25 likewise move without deformation because they are held solid with the support by means of slides (not shown) which are screwed (screws 26). Owing to the nut 30 (FIGURE 1) which engages over a threaded bearing on the pivot 14, the clamping of all the seals between the support 4 and the face 11 is regulated at an intensity which ensures complete air-tightness between the portions of the ring bounded by the different branches of the seal, without crushing the seal and without deforming it whilst it slides on the surface 11. In case of wear after a high number of measuring operations, tightening up can be carried out without replacing the seal, or one can replace the seal, which operation only takes a few minutes. The movement of rotation of the support 4 is facilitated by a ball bearing 31 (FIGURE 1) fitted in a cavity 32 formed in the mass of the casing and enclosing a bearing seat 33 solid with the support 4.

Amongst the advantages of the device according to the invention, one may mention the possibility of replacing a sample without breaking the vacuum, the form of the plate and the dimensions of the holes formed in the latter, which dimensions permit the use of samples of very different shapes, the sensitivity of X-ray emission, which is notably greater than that obtained with an X-ray bombardment. It should be expressly understood that the scope of the present invention extends to the use of electronic beams capable of producing on the samples radiations of wave lengths different from those of X-rays, that is to say ultra-violet and visible radiations.

I claim:

A device for analysing samples by electronic bombardment comprising a body defining an enclosure provided with an orifice, said body having an outer flat surface surrounding said orifice, means for evacuating the enclosure to a vacuum of at least 10^{-5} mm. Hg, a source of bombarding electrons to excite characteristic radiations from a sample held in the orifice, a support mounted on

said flat surface and completely covering said orifice, the support having at least three apertures and being rotatable about an axis normal to the flat surface, gasket means individually surrounding each aperture in said support and in sealing engagement with said flat surface to prevent ingress of air between the support and the flat surface to the orifice in all positions of the support to provide air tight sealing of the apertures, said gasket means being formed by a pair of concentric rings of sealing material having an annular space therebetween and connected by radial strips of sealing material dividing the annular space into mutually sealed substantially equal sectors in number equal to twice the number of apertures in said support, said gasket means being so arranged that each aperture opens wholly into a separate sector and the sectors are alternately with and without apertures opening thereinto, at least two conduits provided in said body and having open ends equally spaced with said orifice from and about the axis of rotation, the apertures in the support being also equally spaced from and about the axis of rotation so as to be brought successively into alignment with the orifice and the open ends of the conduits as the support is rotated about said axis of rotation, means for producing a vacuum of at least 10^{-5} mm. Hg in at least one of said conduits having its open end nearest said orifice, and means for selectively putting another one of said conduits into communication with the surrounding atmosphere, the support further comprising removable covers for tightly sealing each of the apertures, each of said covers being provided with a sample holder whereby a sample may be placed in position by fitting the cover to one of the apertures and rotating the support to a position wherein said one aperture is in communication with said one conduit and further rotating the support to align said one aperture with said orifice in the enclosure.

References Cited

UNITED STATES PATENTS

2,264,210	11/1941	Krause	250—49.5
2,849,619	8/1958	Eisfeldt	250—49.5
3,073,951	1/1963	Burdg	250—41.9
3,117,223	1/1964	Brunnee	250—49.5
3,177,360	4/1965	Hague et al.	250—49.5

RALPH G. NILSON, *Primary Examiner*.

A. L. BIRCH, *Assistant Examiner*.