Nov. 23, 1965 G. MÖLLENSTEDT 3,219,817

ELECTRON EMISSION MICROSCOPE WITH MEANS TO EXPOSE THE SPECIMEN TO ION AND ELECTRON BEAMS

Filed Aug. 27, 1962 4 Sheets-Sheet 1

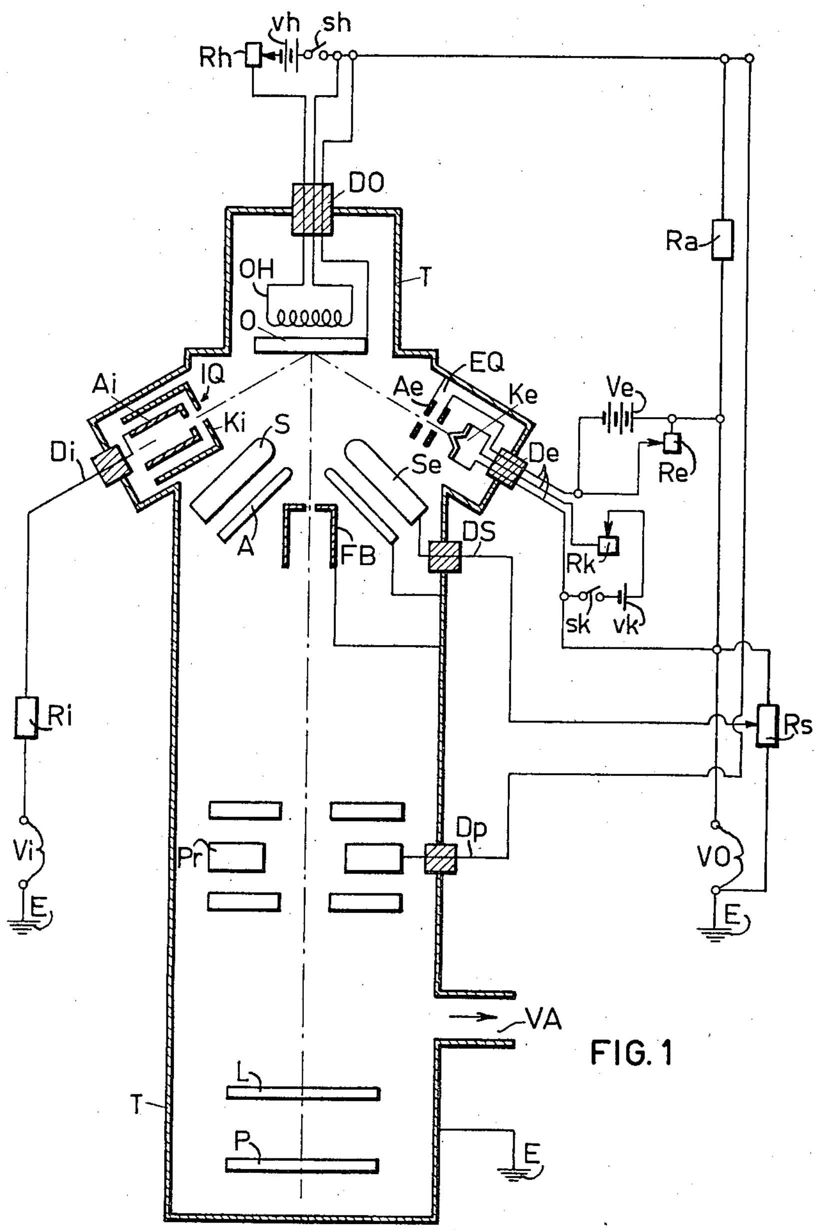

FIG. 1

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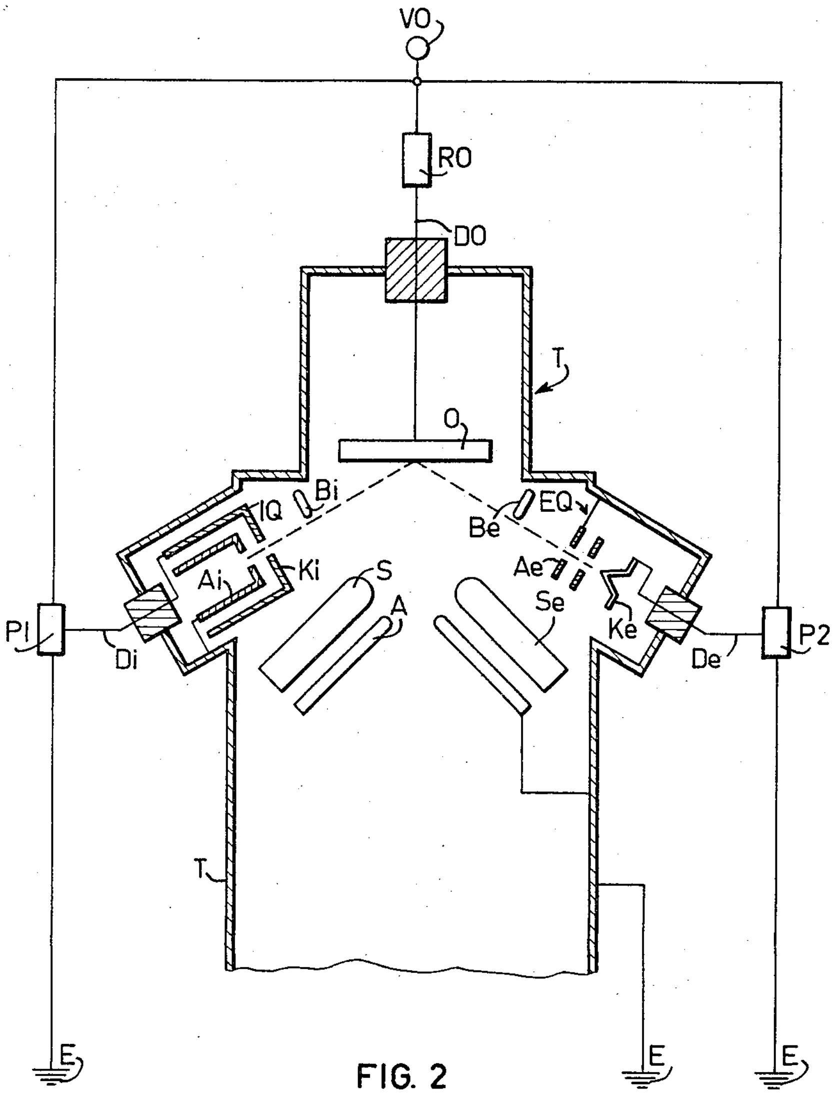
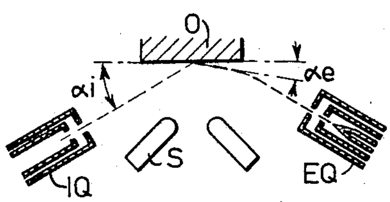


FIG. 2

FIG. 3a



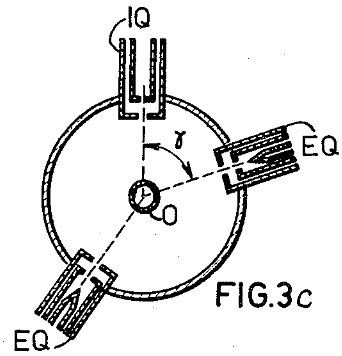


FIG. 3c

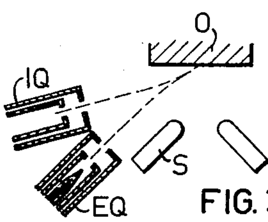


FIG. 3b

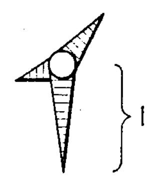


FIG. 3d

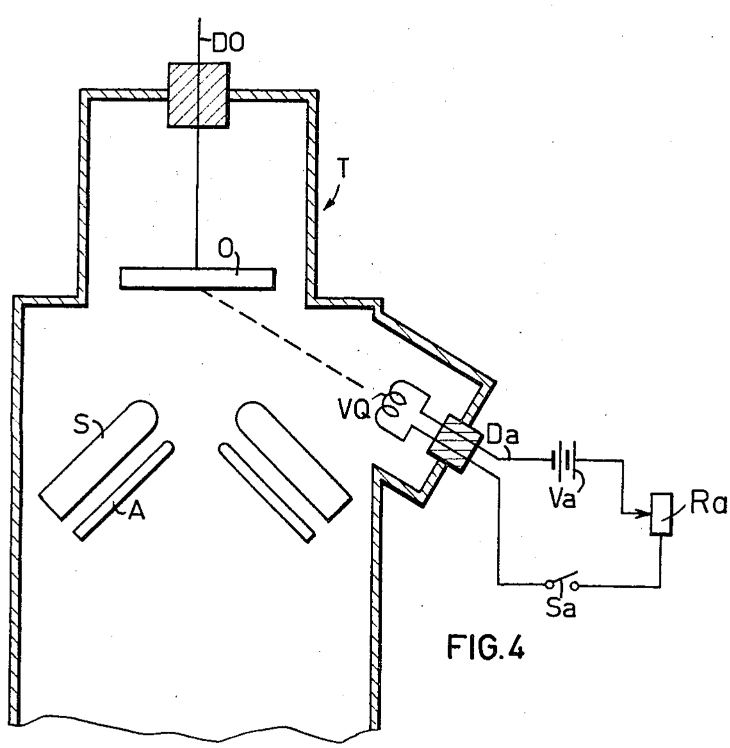


FIG. 4

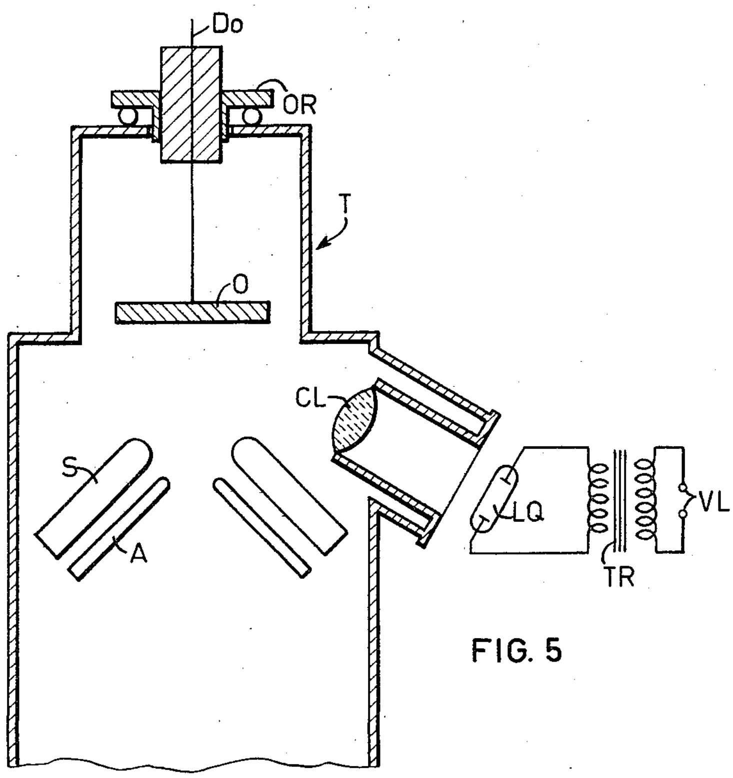


FIG. 5

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ELECTRON EMISSION MICROSCOPE WITH MEANS TO EXPOSE THE SPECIMEN TO ION AND ELECTRON BEAMS

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8 Claims. (Cl. 250—49.5)

This invention relates to electron-emission microscopes for the forming of images of surfaces by electron-optical means and to methods of rendering visible and recording electron-optical images of surfaces by means of an electron-emission microscope.

The emission of electrons in an emission microscope can be effected in three ways which differ in principle: by field emission, thermal emission or secondary emission. The present invention relates mainly to the emission of electrons by secondary emission, although the remaining types of emission are not excluded in an electron microscope equipped according to the invention.

Secondary emission is brought about by irradiating the surface of the specimen with electromagnetic or with corpuscular radiation. There are known secondary emission microscopes, in which the emission of the electrons is effected by ions. In another type of microscope, electrons are used for the emission while in still other types the emission is initiated with ultra-violet light. The most well known secondary emission microscope and the one described in the most detail is the secondary emission microscope in which emission is initiated by means of ions. This instrument is obtainable in the trade under the name "Metioscope."

All the known secondary emission microscopes use a single type of beam for the emission of the electrons.

The method according to the invention is characterized by the feature that the surface, the image of which is to be formed, is bombarded simultaneously or alternatively with electrons and ions.

The electron-emission microscope according to the invention for the electron-optical formation of images of surfaces is characterized by the feature that a source of ions and at least one source of electrons are so incorporated in the microscope tube, that the beams of ions and electrons issuing from the said sources impinge upon the surface whose image is to be formed.

The invention will be made clearer by two embodiments of secondary emission microscope according to the invention which are illustrated in the attached drawing.

In the drawing:

FIGURE 1 is a diagrammatic illustration of a first embodiment of microscope according to the invention;

FIGURE 2 is a diagrammatic illustration of a second embodiment;

FIGURES 3*a*–3*d* are diagrammatic illustrations for clarifying certain terminology employed;

FIGURE 4 is a diagrammatic illustration of a portion of a modified electron emission microscope; and

FIGURE 5 is a diagrammatic illustration of a modified portion of the electron emission microscope of FIG. 2 is seen in section at right angles to that of FIG. 2.

The parts in FIGURES 1 and 2 are designated as follows:

T—Electron microscope tube
VA—Vacuum connection
O—Specimen
S—Control electrode
A—Anode
FB—Precision diaphragm

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Pr—Projective lens
L—Luminous screen
P—Photographic plate
OH—Specimen heating means
E—Earth (ground)
V*o*, V*i*—High voltage sources
V*e*, V*k*, VH—Voltage sources
R*o*, R*h*, R*s*, R*i*, R*k*, P1, P2—Resistances or potentiometers
D*i*, D*o*, D*e*, D*s*, D*p*—Vacuum lead-ins
K*e*—Cathode of the electron source
S*e*—Control electrode of the electron source
A*e*—Anode of the electron source
K*i*—Cathode of the ion source
A*i*—Anode of the ion source
S*h*, S*k*—Switches
B*i*, B*e*—Movable beam cut-offs

FIGURES 3*a* and 3*b* serve to define the term angle of impact or incidence; FIGURES 3*c* and 3*d* serve to define the term azimuth angle and also the length of shadow *l*. The reference characters designate the elements as follows:

O—Specimen
IQ—Ion source
EQ—Electron source
S—Control electrodes
αi—Angle of incidence of the ions
αe—Angle of incidence of the electrons
γ—Azimuth angle between ion and electron source
l—Length of shadow on to the specimen

More particularly, FIGURES 3*a* and 3*b* are diagrammatic sectional views parallel to the axis of the image-forming electron beam. FIGURE 3*c* is a diagrammatic sectional view perpendicular to the axis of the image-forming electron beam. FIGURE 3*d* shows the initial image of a conical specimen portion photographed with the arrangement according to FIGURE 3*c*. FIGURE 4 shows a section through the tube of the emission microscope with a vaporizing system. The reference characters for the elements are as follows:

O—Specimen
S, A—Electrodes
T—Tube
VQ—Vaporization source
V*a*—Voltage source
R*a*—Regulating resistance
S*a*—Switch
D*o*, D*a*—Lead-ins

The ion and electron sources in this sectional drawing not located in the same sectional plane as the vaporizing system.

The operation of a secondary emission microscope will now be described, by way of example, with reference to FIGURE 1:

The specimen O cooperating with the control electrode S and the anode A forms an immersion objective, in which the specimen O is the cathode. The anode A is grounded, whereas the specimen O is connected via the blocking resistance R*a* to high voltage V*o* which is negative with respect to ground. The control electrodes receives its negative high voltage located near the specimen voltage via the potentiometer R*s*. An ion source IQ including an anode A*i* and cathode K*i* is fed via the resistance R*i* by a high voltage V*i* which is positive with respect to ground. The ion beam produced by source IQ impinges on the specimen O and accordingly emits secondary electrons. These are accelerated and collected by the immersion objective including specimen O, control electrode S and anode A to produce an emission image of the specimen. This image is projected by a projective lens Pr onto a luminous screen or a photographic plate P. One or more precision diaphragms FB serve to enhance the contrast and improve the emitting capacity. In order to be able to view the object at a high temperature, there is, for example, a specimen heating means OH, shown in FIGURE 1 as a heating wire, which is fed by the voltage source VH via the resistance R*h*. The heating of the specimen may, however, also be carried out, in a known manner, by bombarding the rear of the specimen with electrons.

The electron-optical elements from the specimen O up to the photographic plate P are located in the evacuated tube T including the exhaust connection VA. The vacuum system itself is not shown in FIGURE 1.

The microscope of FIGURE 1 further includes an electron source EQ whose electron beam is directed to the same point of the specimen O as the ion beam from the ion source IQ. The electron source includes, for example, an anode A*e*, a control electrode S*e* and a cathode K*e*. The anode A*e* is grounded. The control electrode S*e* and the cathode K*e* are connected to the negative high voltage V*o*. The cathode K*e* is heated by the voltage source V*k* via the resistance R*k* and the control electrode S*e* receives an initial potential from the voltage source V*e* via the resistance R*e*. The electron source EQ may also include a known cold cathode. In order that the electrons can reach the specimen, the cathode K*e* must be more strongly negative than the specimen O. This is effected by the voltage drop at the resistance R*a*. Should the electrons reach the specimen at a relatively high velocity a separate high voltage source may also be used for feeding the electron source EQ.

The arrangement described has manifold advantages over a conventional emission microscope, which is only provided with either an ion source or an electron source.

Emission by ions presents certain difficulties, when the specimen is an electrically insulating surface, since such surfaces become charged with a part of the electrons emitted. The formation of the image is distorted by this charging. Simultaneous irradiation with slow electrons has the effect of forming in the residual gas in front of the specimen a sufficient number of positive ions to discharge the specimen continuously, so that a clear image formation is possible.

In general, when irradiating with ions alone or with electrons alone, a space charge is formed in the residual gas in front of the specimen. The quality of the image formation in an emission microscope is considerably influenced by the homogeneity of the electric field between specimen O and control electrode S. A space charge in front of the specimen disturbs this homogeneity and hence impairs the image formation. Simultaneous irradiation with ions *and* electrons reduces the space charge and therefore improves the quality of image formation and the emitting capacity.

Whereas in the case of irradiation with ions, contamination of the specimen is, as is known, minimized by the fact that the ions have an etching action on the surface of the specimen, this is not the case with electron radiation. The electrons have too small an etching effect, so that the specimen is contaminated after a short period of irradiation and the differentiation of substance is lost through the varying emission coefficients. This disadvantage of electron emission can be removed if an ion source is present in the instrument simultaneously. The specimen can then be decontaminated by means of a brief ion etching and the image emitted by electrons can be viewed immediately afterwards. In this way by irradiating alternately with ions and electrons a clear electron-emitted secondary emission image is obtained. It is clear that this object cannot be achieved, if the microscope must first be converted from ion to electron emission.

The possibility thus created of obtaining ion-emitted and electron-emitted images one after the other, widens

the possibilities of interpretation of the images. Substance differentiation is generally different for ion and electron emission. Two substances or phases, which cannot be distinguished with one type of emission, can therefore be distinguished with the other type. Moreover, the certainty of determining the phases or substances from the brightness of the image increases.

The same applies to the image contrast. This is greater, the stronger the difference between the emission coefficients of the phases obtained in the surface. Image contrast is, however, also reduced by growing layers of contamination. The possibility of using ions and electrons simultaneously or alternately for the emission is therefore a means of heightening the contrast.

The advantages of the method according to the invention may therefore be summarized as follows:

Discharge of insulating specimens with ion emission;

Reduction of the space charge in the immersion objective;

Improvement of the electron-optical properties of the image formation field;

Improvement of the emission;

Prevention of the formation of layers of contamination with electron emission;

Increase in the image contrast; and

Improvement in the phase and substance differentiation.

Some of the effects described above become particularly strongly marked, if slow electrons are used, i.e. if the voltage drop at the resistance R*a* is small. If, on the other hand, the emission is to be effected by the electrons, then high velocity electrons are necessary.

The method is therefore carried out in one case with slow electrons and in the other case with high velocity electrons.

One embodiment of the invention has been illustrated in FIGURE 1 and is described above.

A second embodiment contains, in addition to the sources IQ and EQ shown in FIGURE 1, one or more further electron sources, the axis of which is no longer located in the image plane, but the electron beams of which are likewise aimed at the specimen O. One of these electron sources is operated, for example by a volttage which is strongly negative with respect to the specimen voltage and serves to emit the image-forming electrons, while all the remaining electron sources emit slow electrons, which discharge the insulating specimen surface. The ion source serves in this case to decontaminate or to etch the specimen. An embodiment with one ion source and two electron sources is shown, by way of example, in FIGURE 3*c*. This embodiment is characterized by the feature that several electron sources are provided, one of these electron sources emitting high velocity electrons and the remainder emitting slow electrons.

It is desirable for the different ion and electron sources to be capable of being switched off as desired. The switching off is accomplished either by switching off the voltage by means of the switches such as S*k* (FIG. 1) or by means of conventional beam cut-offs B*i* and B*e* as shown in FIGURE 2.

A further embodiment copes with the known shadow effect of the ion or electron bombardment on the specimen. A single ion source, as for example in the "Metioscope," is already, as known, so incorporated that the angle of incidence αi (FIGURE 3*a*) of the ions on the specimen is adjustable. The same adjustability is desirable for electron sources as well as in the case of the ion beams.

The etching action depends on the angle of incidence; in the case of electron beams angles of 5° to 20° are desirable for the emission of secondary electrons while for the discharge angles of incidence of up to 0° are sometimes required. The various ion and electron sources are in this embodiment so arranged that the angle of incidence of the ion and electron beams on the specimen plane is adjustable.

The ion and electron beams do not travel in a straight line towards the specimen, but are deflected through the electric field between specimen, control electrode and anode. The ion beams undergo a curvature towards the specimen, while the electron beams undergo a curvature away from the specimen. This curvature is indicated for the electron beam in FIGURE 3*a*. As the curvature varies with the acceleration voltage of the electrons, the angle of incidence αe can be varied with the same position of the electron source EQ, if the voltage at the electron source is varied, for example by means of the potentiometer P2 in FIGURE 2. It is therefore advantageous for one or more of the ion and electron sources to be operated with a variable voltage.

If one ion source and one or more electron sources are used simultaneously for the electron emission (FIGURE 3*c*), then images with two or more shadow directions (FIGURE 3*d*) are generally formed. A single geometric figure projecting from the surface of the specimen will therefore have several shadows, which point in several different directions. This is often of advantage with fine-grained specimens for distinguishability between the grains.

One embodiment is therefore characterized by the feature that one ion source and one or more electron sources are so arranged, that the shadows produced in the picture by the oblique incidence of the ion and electron beams on the specimen point in different directions.

On the other hand this multi-shadow formation may be distorting, so that an image with a clear shadow is desired. This can be achieved in the case of simultaneous emission by an ion and an electron beam by the feature that the ion source and the electron source are arranged one above the other (FIGURE 3*b*), i.e. that the azimuth angle γ (FIGURE 3*c*) is equal to zero. The ions and the electrons then impinge on the specimen from the same direction and the shadows are projected in the same direction. The shadows may still be of different length l. If however the angle of incidence of the ions is made equal to that of the electrons, which is possible due to the curvature of the beams, then the shadow lengths are also equal.

In this embodiment an ion and an electron source are so arranged that the shadows produced in the image point in the same direction and that the shadows coincide with respect to length and direction.

For reasons already mentioned it is an advantage if the specimen can be heated.

A further advantage arises if the specimen is not only movable, as is usual, in three directions, but is also rotatable additionally about an axis parallel with the axis of the microscope. Then, for example, the surface of the specimen can be etched by the ion etching, the etching failing to take place in the shadows. After rotation of the specimen the unetched places can be compared with the etched ones. In FIG. 5 there is shown a microscope in which the specimen O can be rotated. For this purpose the specimen is supported in a bearing OR in which is accommodated the vacuum lead-in DO.

A further embodiment facilitates the observance and photographing of insulating specimen surfaces. For this purpose it is an advantage, if the non-observed parts of the specimen are coated with an electrically conducting layer. This can, for example, be done by vaporizing a metal layer onto the surface of the specimen, which is then removed by ion etching at the place of observation or etched sufficiently thinly for observation of the specimen structure.

This embodiment is characterized by the feature that the microscope tube contains a vaporizing device, by means of which the surface of the specimen can be vaporized. Such a device is shown e.g. in FIG. 4. The vaporization source consists of a heated wire VQ from which the evaporated material comes to the object O. The source may be e.g. heated by a voltage source V*a* through a switch S*a* and a regulating resistance R*a*. The vacuum conduit D*a* consists of heat resistant material or is cooled.

For the photographing of processes presenting, for example, as a function of the temperature of the specimen or the duration of the ion etching, rapidly changing image formations, it is an advantage if the camera incorporated in the microscope is a film camera, as is also known in transmission electron microscopes.

While FIG. 1 shows independent high voltage sources for the various ion and electron sources, it is, however, also possible to energize all the ion and electron sources by only one high voltage source. Such an embodiment is shown in FIGURE 2. The negative high voltage V*o* feeds the cathode O of the immersion objective via a blocking resistance R*o*. At the same time, it feeds two or more potentiometers P1 and P2, to which the electron sources EQ and the ion source IQ are connected with such polarity that the ions and electrons are accelerated towards the cathode O. In this embodiment, therefore, at least some of the ion and electron sources are connected to the high voltage source of the emission microscope.

The embodiment indicated in FIGURE 1 with a separate high voltage supply for the ion source is therefore particularly interesting, because the positive high voltage supply V*i* with respect to ground is added to the voltage difference between ground E and cathode O. In this way, for example, ions with double the acceleration voltage can be used, without any part of the structure requiring insulation for this doubled voltage. The embodiment is characterized by the feature that at least some of the ion and electron sources are operated with separate voltage sources.

In order to widen the possibilities of the electron-emission microscope according to the invention still further, in another embodiment a source of ultra-violet light is incorporated in addition to the ion and electron sources, which likewise irradiates the surface of the specimen. This ultra-violet source is used as an emission source, while with simultaneous operation, the ion source is used for etching and preventing contamination and the electron source for discharging the specimen or for improving the optical properties of the immersion objective. Thus as seen in FIG. 5 there is illustrated a device for producing an ultra-violet beam such as mercury-quartz lamp LQ connected to a transformer TR and a current supply VL. To focus the ultra-violet-light on the object surface there is provided a quartz lens CL. As in the case of electron and ion sources, the ultra-violet source embodiment also operates with one or more of the remaining sources simultaneously or alternately. The ultra-violet source thus in effect replaces one of the electron sources EQ in FIGURE 3*c*. The embodiment is characterized by the feature that in addition to the ion and electron sources a source of ultra-violet light is incorporated, the light beam of which is directed towards the surface of the specimen of which the image is to be formed.

As the intensity of the irradiation of the specimen is a deciding factor in the intensity of the emission of secondary electrons, strong ultra-violet sources are used. The use of the intensive light sources known under the name Laser is particularly interesting.

What we claim is:

1. An electron emission microscope for examining a surface portion of a specimen, said microscope comprising an evacuated tube having a longitudinal axis, means for supporting a specimen within the tube for rotation about an axis parallel to that of the tube, means applying a negative voltage to said specimen, a source of ions, means supported in said tube for directing ions from said source in a relatively fast moving beam obliquely onto a surface portion of the specimen to cause emission of secondary electrons from said specimen substantially along the longitudinal axis of the tube, a source of electrons,

means in said tube for directing electrons from said source of electrons in a relatively slow moving beam obliquely onto the surface portion of the specimen to discharge electric charges at the surface portion of the specimen, the entire specimen being supported in the tube and completely open and exposed to the ion beam and electron beam, transducing means in said tube for receiving and converting secondary electrons to visual indicia, and means in said tube for focusing the emitted secondary electrons onto the transducing means.

2. The electron-emission microscope of claim **1** including means for controlling the angle of incidence of at least one of said beams with respect to said surface portion of said specimen.

3. An electron-emission microscope of claim **1** wherein said sources and directing means are so oriented that the shadows produced in the image by the oblique incidence of said ion and electron beams on the surface portion of said specimen point in different directions.

4. An electron-emission microscope of claim **1** wherein said sources and directing means are so oriented that the shadows produced in the image by the incidence of said ion and electron beams on said surface portion of said specimen point in the same direction.

5. An electron-emission microscope as claimed in claim **1**, wherein said sources and directing means are so oriented that the shadows produced by the incidence of said ion and electrons beams on said surface portion of said specimen coincide with respect to length and direction.

6. An electron-emission microscope as claimed in claim **1** including a common source of high voltage for energizing said ion and electron sources and said focusing means.

7. An electron-emission microscope as claimed in claim **1** including separate high voltage sources for energizing said ion and electron sources.

8. An electron-emission microscope as claimed in claim **1** further including a source of ultraviolet light whose light beam is directed towards the surface portion of said specimen on which an image is to be formed.

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