Nov. 3, 1964 W. C. NIXON 3,155,827

ELECTRON MICROSCOPE WITH A SECONDARY ELECTRON SOURCE UTILIZED FOR ELECTRON PROBE ANALYSIS

Filed March 24, 1961 3 Sheets-Sheet 1

Fig. 1.

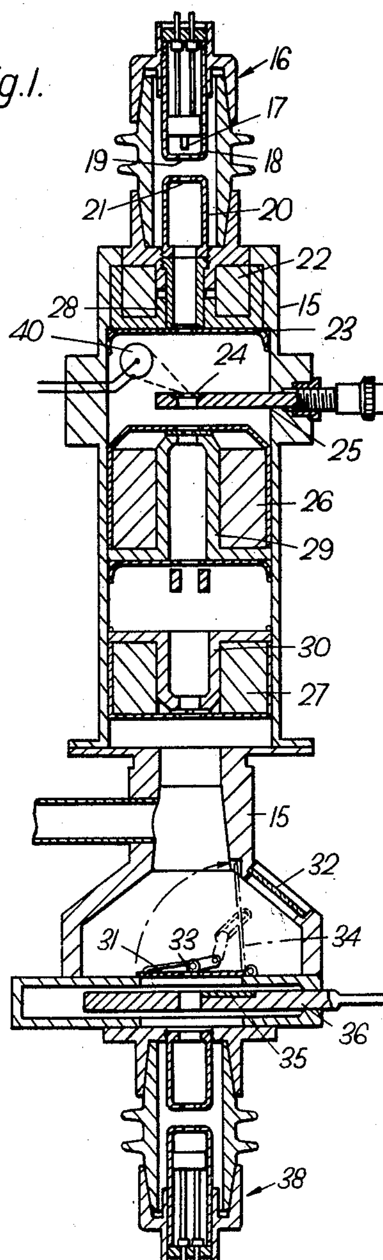

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Fig. 2.

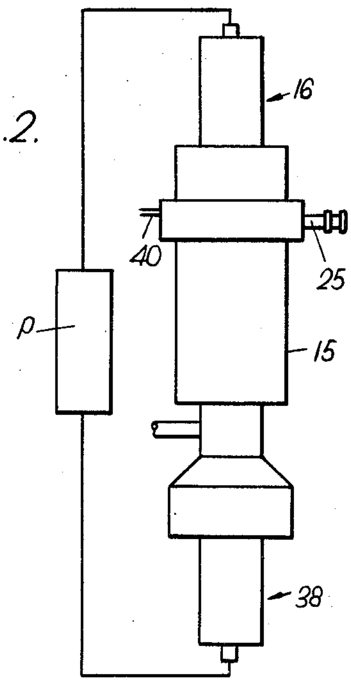

Fig. 3.

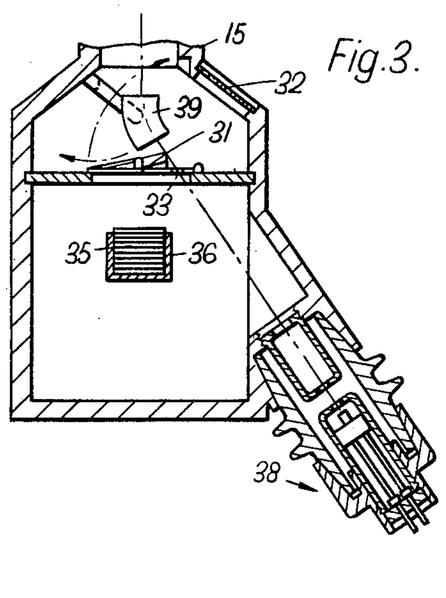

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Fig. 4.

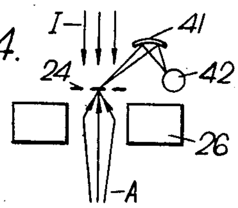


Fig. 5.

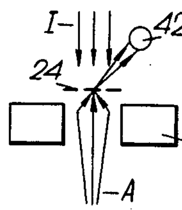


Fig. 6.

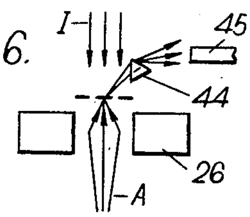


Fig. 7.

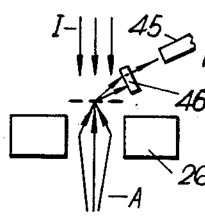


Fig. 8.

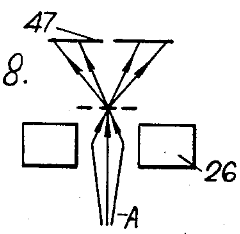


Fig. 9.

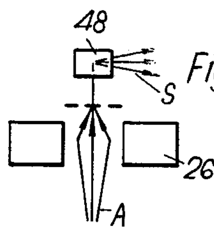


Fig. 10.

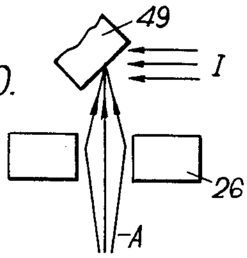


Fig. 11.

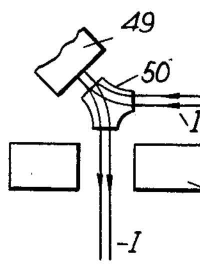


Fig. 12.

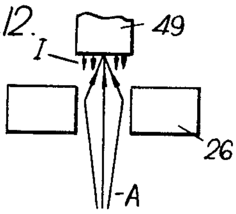
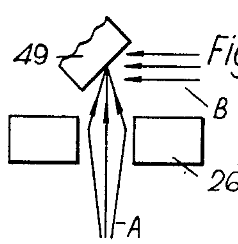


Fig. 13.



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ELECTRON MICROSCOPE WITH A SECONDARY ELECTRON SOURCE UTILIZED FOR ELECTRON PROBE ANALYSIS

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11 Claims. (Cl. 250—49.5)

The present invention relates to electron microscopes, and it has for its object to provide means whereby the versatility of the electron microscope may be substantially improved with a view to selective area analysis by various technical procedures, of part of the area of the specimen which is under examination.

Conventional types of electron microscope provide a source of electrons, commonly termed the electron gun, and a series of beam focusing elements, commonly termed lenses by optical analogy, which serve to produce a magnified image of the specimen, and the electrons of said image may be utilised either to activate a fluorescent screen for direct viewing of the image or for exposing a photographic sensitive material for producing a permanent record of a specimen under examination.

The means by which this result is secured involve the provision between the electron source and the specimen, of means to concentrate the electron beam on the specimen. These means may be regarded as a condenser, and are followed by a plurality of further focusing elements which may include what may be regarded as an objective lens and as a projector lens. These elements produce a greatly magnified image of a predetermined small area of the specimen, the image being then examined on the fluorescent screen or recorded on a photographic sensitive surface. In some cases the specimen is examined by transmission through a thin specimen, in other cases reflection from the surface of a specimen is examined.

The present invention is more particularly concerned with the provision of means for local examination or analysis by various procedures of a specific small portion of the part of the specimen reproduced as a magnified image in the viewing field, for the purpose of examining any particular small section of the area of the specimen which is under examination, by various detection or analysis techniques known in the art, typical examples of which are referred to hereinafter.

In accordance with the present invention, this local analysis of a predetermined area of that section of the specimen which is under examination is obtained by the provision of a further source of electrons which traverse the focusing elements or lenses in the opposite direction to that traversed by the image-forming electron beam and are brought to a focus on the specimen at a particular small section of the portion of the specimen which is under examination. Any suitable detection means, depending upon the nature of the analysis being performed, may be provided responding to the action of the electron beam on the specimen under examination.

The beam of electrons produced by the further source of electrons will hereinafter be referred to as the "analyzing beam" and it will be understood that, due to the focusing action of the lens components, an intense electron beam is brought to a point focus of extremely small dimensions at the point where it strikes the specimen.

In the preferred arrangement according to the present invention an electron microscope is provided wherein, in addition to the usual electron gun adapted to produce a stream of electrons directed towards a specimen, from which a magnified image is produced which may be inspected upon a fluorescent viewing screen, there is provided on the opposite side of the fluorescent screen a second electron gun adapted to project a stream of electrons through the imaging lenses reversely to the image-forming beam so that the second electron beam (the analyzing beam) is finely focussed in the specimen plane. Because of the small angular aperture of the image-forming lenses employed in electron microscopes and because the focusing action is independent of the direction of travel of the electron beams, the analyzing beam has a comparatively large depth of focus at the specimen plane so that the second electron gun need not be in the plane of the fluorescent screen but may be at some small distance on either side of it, but, nevertheless, the finely focussed analyzing beam still remains in point focus at the specimen plane. The fluorescent screen may have a small aperture at the centre to allow the analyzing electron beam to pass from the second gun into the imaging system and towards the specimen plane in the direction opposite to that travelled by the main electron beam.

The features of the present invention will be explained hereinafter and by way of example with reference to the accompanying drawings wherein:

FIG. 1 is a general view in outline form of an electron microscope embodying the features of the present invention, only the essential parts of the equipment being shown.

FIG. 2 is a diagram showing the supply of operating potentials to the tube,

FIG. 3 illustrates a modification of the mounting of the second electron gun for use where the microscope is provided with another type of supply chamber for photographic plates,

FIGS. 4 to 9 are diagrammatic views illustrating various methods of examining transmission specimens by means of the analyzing beam, and

FIGS. 10 to 13 are diagrammatic views illustrating methods which may be adopted for the examination of specimens by reflection.

Referring first to FIG. 1 of the accompanying drawings a typical electron microscope is shown in outline form, said microscope comprising a suitable evacuable outer casing **15** the upper end of which carries an electron source or "gun" indicated generally at **16** and having a heated filament **17** enclosed within a cylinder **18** having a small opening **19** in the axis. Axially aligned therewith is a cylinder **20** having a fine aperture **21** in it through which electrons produced by the filament **17** are projected along the axis of the outer casing **15**.

The casing houses a series of focussing elements which in the example shown consist of electromagnets surrounded by suitable magnetic shields **28** with peripheral gaps in the bores which serve as pole pieces to produce an intense magnetic field. The first element **22** may be regarded as a condenser lens and is followed by an apertured plate **23** which serves as the condenser aperture and allows an intense electron beam to fall upon a specimen mounted on a carrier **24** supported by a suitable specimen stage unit shown in diagrammatic form as a slide member **25**, which permits specimens to be brought into the axis of the equipment without substantial loss of vacuum within the casing **15**. The unit **25** is movable in two directions by well-known methods to permit any desired area of the specimen to be chosen for examination. Any suitable air lock device may be provided to permit insertion of specimens without much loss of vacuum, but it will of course be understood that following normal practice the equipment is operated with a vacuum pump to maintain the necessary low pressure within the equipment. These features enable the specimen to be powerfully illuminated by the electron beam concentrated on it by the action of the condenser **22**.

The equipment also comprises further focussing elements comprising an objective lens **26** and a projector lens **27**, again consisting of electromagnets surrounded by suitable shields 29, 30 designed and proportioned in accordance with well-known techniques so as to produce a magnified image of the area of the specimen mounted on the carrier 24 upon a fluorescent screen 31 which may be examined through a sight window 32 in a suitable housing at the base of the casing 15. The fluorescent screen 31 is provided with a central aperture 33 and is adapted to be rotated to the inoperative position shown in dotted lines at 34 when the equipment is to be used for making photographic records. For this purpose, and as indicated in diagrammatic form, a photographic sensitive element 35 may be mounted upon a suitable slide or other similar carrier 36 operable from outside the equipment.

Apart from the provision of the aperture 33 the equipment so far described follows normal practice in the art, the condenser lens 22 serving to illuminate the specimen on the carrier 24 while the lenses 26 and 27 may be regarded as imaging lenses for producing a greatly enlarged image of the illumination of the specimen by the imaging electron beam, and the display field of examination of the specimen is of course visible on the fluorescent screen 31 or can be photographed if required by the equipment 35, 36.

In accordance with the present invention a second electron gun 38 is provided on the opposite side of the fluorescent screen 31, this gun being arranged and operated in the same way as the electron gun assembly 16. This gun 38 produces a beam of electrons (the analyzing beam) which travel in the opposite direction to the imaging beam from the gun 16. The analyzing beam is focused and concentrated by the action of the lenses 26 and 27 so as to fall upon a predetermined small section of the part of the specimen on the carrier 24 which is under examination and can be viewed on the screen 31.

It will of course be understood that the specimen holder 25 is adapted to be moved with a fine adjusting movement in two directions at right angles one to the other which permits a selected area of the specimen to be magnified upon the fluorescent screen 31 in the position shown on the drawings. By further adjusting the specimen any particular portion of the field thereof may be brought over the aperture 33 in the fluorescent screen and the corresponding fractional portion of the specimen will necessarily then receive the finely focused analyzing beam produced by the second gun 38 and the reaction of the specimen to said electron beam may be detected by any suitable detection or measuring equipment.

Various methods of micro-analysis will be described in outline form hereinafter, but it should first be explained that setting up of the equipment for examination of a specimen is carried out by the normal procedures while viewing the fluorescent screen 31. The various corrections, centering, etc., of the lenses are all carried out while viewing the image on the fluorescent screen so as to secure the best possible reproduction of the specimen under examination. Optimum conditions having been established, it follows that these optimum conditions will apply equally well for the analyzing beam from the second electron gun 38. The maximum intensity of the analyzing beam may be ensured by monitoring said beam with a suitable beam current detector 40 placed in the region of the specimen, the beam from the main gun 16 being cut out while this operation is performed.

It is possible in many cases to check that the area being analyzed by the analyzing beam coincides with the required area to be examined by noting the growth of contamination of the specimen at the point where the intense electron beam strikes the specimen.

In some cases both electron beams, namely that from the main gun 16 and that from the second gun 38, can be operative at the same time but if there is any interaction between the beams or if the main beam from the gun 16 produces a background radiation from the rest of the specimen, then the analyzing beam may be used alone or the two beams may be operated in rapid succession on a cyclic basis.

FIG. 2 illustrates the connection of a high voltage power unit P to the electron gun 16 and also to the second gun 38 for producing the analyzing beam so that both units receive a high negative voltage. Apart from the provision of the second electron gun the microscope follows normal practice and it has not been deemed necessary to show or describe the auxiliary features normally provided with such equipment and which are incorporated in the equipment according to the present invention.

FIG. 3 illustrates a modification of the arrangement of the second gun 38 applicable in the case where a number of photographic plates or the like 35 are stacked one over the other in a holder 36 beneath the image field. In such case the second gun 38 may be arranged to one side with its axis running obliquely to the main axis as shown and a deflecting block or "alignment field" 39 is provided. A fluorescent screen 31 is provided in the same position as in FIG. 1 and the block or field member 39 is pivotally mounted above the screen so that it may be swung out of the way of the imaging beam during normal electron microscope inspection of a specimen. The fluorescent screen 31 may be operated by an external operating lever or alternatively an electromagnet may be used, the energising current being switched on for analysis and off for viewing. When the analyzing beam is in use the block 39 is moved into the operative position shown to cause the analyzing beam to be projected reversely along the axis of the microscope. In this case the fluorescent screen may be suitably marked with calibration lines to permit the required selected area to be chosen for examination by the analyzing beam from the gun 38.

The equipment so far described is in a form suitable for the examination of thin specimens by the transmission of the imaging beam through the specimen, but it will readily be understood by those skilled in the art that the equipment may be readily utilized for other methods of electron microscopy, typical examples of which will be referred to below with reference to FIGS. 10 to 13 of the drawings.

It will further be understood that examination of a selected area of a specimen, whether a transmission specimen or a reflection specimen, may be performed by means of the analyzing beam from the second electron gun 38, various systems being adopted according to the particular method of analysis which is preferred or is appropriate to an investigation being carried out.

FIGS. 4 and 5 are illustrative of the means which may be adopted for responding to the characteristic X-rays emitted by the different elements in the portion of a specimen under examination when bombarded with electrons, the X-rays emitted being analyzed by means well-known in the art to give the exact nature and amount of each element present.

In all of FIGS. 4 to 9 there is shown diagrammatically the objective lens 26 and the path of the imaging beam is illustrated by the arrows I while the path of the analyzing beam is indicated by the arrows A. In each case it will be understood that the two beams need not be in use simultaneously, imaging beam being used primarily to select a predetermined section of a specimen for examination of a selected small portion thereof by the action of the analyzing beam A.

As shown on FIG. 4 X-rays emitted from the specimen 24 are reflected by a curved crystal reflector 41 and are received by an X-ray counter tube 42 connected in well-known manner which will readily be understood by those skilled in this art, to amplifier and counter equipment. FIG. 4 is based on X-ray spectrographic dispersive analysis the specific procedures involved being well recognised in the art.

FIG. 5 illustrates another detection system where the counter 42 responds directly to the X-rays emitted by the specimen under the action of the analyzing beam A this providing for X-ray spectrographic analysis without the dispersive feature. In these arrangements detection of the X-rays responds to the X-ray emission from the specimen and is not affected by either beam of electrons I or A.

FIGS. 6 and 7 illustrate systems based on the visible, ultra-violet or infra-red radiation which may be emitted from some specimens when subjected to the action of the analyzing beam A. In FIG. 6 a prism or other equivalent diffraction member **44** is placed in the path of the emitted radiation and a light guide **45** leads to a photoelectric multiplier whereby the properties of the radiation emitted from the specimen **24** may be determined as to wavelength and as to amount. Similar results may be obtained as shown in FIG. 7 by means of a series of light filters one of which is shown at **46**, which select the light transmitted into the light guide **45** leading to a photomultiplier.

FIG. 8 illustrates the examination of a crystalline specimen by diffraction of the electrons of the analyzing beam, giving a diffraction pattern which is recorded on a suitably mounted photographic plate **47**. The result may be used to determine the amount, type and nature of the material present in the small selected portion of the specimen which receives the analyzing beam.

Electron energy loss in the specimen may also be used for analysis; for this purpose the features shown in FIG. 9 may be used, where **48** is a magnetic energy analyzer device giving an energy loss spectrum (represented at S) characteristic of the small selected area of the specimen which receives the analyzing beam. Other methods of analysis may also be adopted for example based on secondary electron emission and field emission of electrons.

FIGS. 10 to 13 illustrate methods of electron microscopy other than by transmission through a thin specimen. FIG. 10 illustrates reflection microscopy where the surface of a solid specimen is placed obliquely to the axis and receives the imaging beam I transversely to the axis. The analyzing beam A is received on a selected area of the part of the specimen surface displayed on a fluorescent screen and the reaction of the specimen to the analyzing beam is assessed or appraised.

FIG. 11 illustrates to the use of mirror field methods where a deflecting block or "field" **50** receives the imaging beam, throws it on to the surface of the specimen **49** and then projects it along the axis of the microscope. In this case the analyzing beam A is not shown as it cannot be used simultaneously with the imaging beam since the block or mirror field member **50** must be reversed when selected area analysis by the analyzing beam is in use.

In some cases the surface of a specimen **49** may be examined by thermionic electron emission from the surface when heated as shown in FIG. 12. Such emission forms the imaging beam I and selected area analysis may be performed by the analyzing beam A.

In other cases the emission of electrons from the surface of the specimen may be induced by other procedures. FIG. 13 illustrates ion-induced emission where a beam of ions B is directed towards the specimen **50**, the surface of which emits electrons which provide the imaging beam, a selected portion of the image being analysed by the analysing beam A. It will be observed that in FIG. 12 the thermionic emission surface may be placed normal to the microscope axis while in FIGS. 10, 11 and 13 the surface is obliquely positioned. The reaction of the specimen of any one of FIGS. 10 to 13 may be assessed by any one or more of the methods described with reference to FIGS. 4 to 9.

What I claim is:

1. An electron microscope comprising means to form a stream of electrons traveling away from a specimen to be examined, magnetic focussing means adapted to act upon said electrons so as to form a magnified image of a predetermined area of said specimen capable of being viewed on a fluorescent screen, means to project a further stream of electrons to traverse said magnetic focussing means in the opposite direction to the first named stream of electrons, said focussing means acting upon said further stream to produce a point-focussed beam of electrons on a selected portion of the said predetermined area of said specimen for local area analysis thereof and means responsive to the action of said further electron stream focussed on said selected portion of said specimen for technical examination and analysis of said selected area.

2. An electron microscope as claimed in claim 1 wherein the first stream of electrons is produced by an electron gun at one end of the microscope and the further stream of electrons is produced by a second electron gun at the other end of the microscope.

3. An electron microscope as claimed in claim 2 wherein the second electron gun is located in the axis of the microscope.

4. An electron miscroscope as claimed in claim 2 wherein the second electron gun is positioned to one side of the axis and the stream of electrons produced thereby is deflected to travel along the axis by means of a deflecting means.

5. An electron microscope according to claim 1 comprising a fluorescent screen upon which a magnified image of the predetermined area of said specimen is made visible for inspection, said screen being arranged to permit a selected portion of said predetermined area to be chosen for examination by said further electron beam.

6. An electron microscope according to claim 5 wherein said fluorescent screen is provided with a central aperture through which pass the electrons emitted by the second electron gun.

7. An electron microscope according to claim 5 wherein said fluorescent screen is calibrated at a point corresponding to the axis of the microscope along which the further electron beam is projected by the action of the deflecting means to permit the required portion of the predetermined area of the specimen to be subjected to analysis.

8. An electron microscope according to claim 1 wherein X-ray emission from the specimen by the action of the second electron stream is appraised by X-ray spectrographic detection.

9. An electron microscope according to claim 1 wherein light emission is emitted by the action of the further electron beam on the specimen and means for determining the wavelength and amount of light emitted comprising a diffraction member and a photomultiplier device.

10. An electron microscope according to claim 1 wherein a crystalline specimen is arranged so that diffracted electrons resulting from the reaction of the specimen to the second electron beam are used to form a diffraction pattern, the latter being then employed to determine the amount, type and nature of the material present in the specimen.

11. An electron microscope according to claim 1 comprising means to analyze the reaction of the specimen by analyzing the electron energy loss to give a spectrum of electron energy loss representative of the materials present in the specimen.

References Cited in the file of this patent

UNITED STATES PATENTS

2,356,633	Von Ardenne	Aug. 22, 1944
2,890,342	Columbe	June 9, 1959
2,928,943	Bartz et al.	Mar. 15, 1960
3,049,618	Thome	Aug. 14, 1962
3,086,114	Herrmann	Apr. 16, 1963

FOREIGN PATENTS

763,522	Great Britain	Dec. 12, 1956