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An Agency of Industry Canada / Un organisme d'industrie Canada

(11) CA 445638 (13) A
(40) 16.12.1947

(12)

(21) Application number: 445638D
(22) Date of filing: ..

(51) Int. Cl:

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(54) ELECTRON MICROSCOPE SPECIMEN
(54) SPECIMEN DE MICROSCOPE ELECTRONIQUE

(57) Abstract:

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This invention relates generally to electron microscope technique and more particularly to improved methods of and means for providing relief copies of microscopic specimens for use in electron microscope investigations.

Frequently, microscopic specimens of materials are too thick or too dense to permit satisfactory electron microscopic investigations by means of electron transmission through the specimen. Various methods have been employed for providing relief copies of such specimens by molding electron permeable elements in direct contact with the original specimen, and thence placing the molded copy of the specimen in the object chamber of the electron microscope. Such copies are not entirely satisfactory for transmission electron microscopy, since the copies are not characteristic of the internal structure of the specimen. Furthermore, minute air bubbles between the specimen and the copy or imperfect register therebetween sometimes provide an extremely poor copy of the original specimen.

The instant invention contemplates improved methods of and means for juxtaposing a very thin electron pervious element substantially in contact with the microscopic specimen. The electron pervious element may, for example, comprise an extremely thin layer of a metallic bichromated gel, which is supported by an extremely thin membrane of colledion or other electron pervious material. The specimen and the juxtaposed element are irradiated from a point source of short wavelength radiation such as, for example, soft X-rays or high velocity electrons, whereby the metallic bichromated gel is irradiated by X-rays or electrons transmitted by the relatively thick specimen. Tanning of the bichromated gel will be substantially proportional to the intensity of the radiation transmitted by the specimen. The untanned or

partially tanned portions of the bichromated gel may be removed by dissolving these portions in, for example, warm water. The residual tanned material therefore will vary in thickness and density as a function of the thickness and density of the original specimen. The relief copy of the specimen thus provided may be made as thin as desired, thereby providing a satisfactory specimen copy for electron microscope investigation in the usual manner.

Various materials and methods of supporting the original specimen and the relief copy thereof will be described in greater detail hereinafter.

Among the objects of the invention are to provide improved methods of and means for electron microscopically observing microscopic specimens. Another object of the invention is to provide an improved method of and means for securing accurate relief copies of relatively dense microscopic specimens for use in electron microscopy. An additional object of the invention is to provide an improved method of and means for exposing an electron pervious element to X-ray irradiation by transmission through a relatively electron impervious specimen for providing a relief copy of said impervious specimen for electron microscopic observation.

A further object of the invention is to provide an improved method of and means for providing accurate relief copies of relatively dense microspecimens by irradiating an electron pervious element by high velocity electrons transmitted through said dense specimen to provide a microscopic specimen copy characteristic of the electron transmission characteristics of the original specimen. An additional object of the invention is to provide an improved method of and means for subjecting an electron pervious material to radiations of short wavelength by transmission

through a relatively dense microspecimen, and by removing portions of said irradiated element as a function of the transmitted radiation image of the original specimen.

The invention will be further described by reference to the accompanying drawing of which Figure 1 is a partially schematic elevational view of the technique involved. Figure 2 is a fragmentary cross-sectional view of one embodiment of the final product obtained in accordance with the invention and Figure 3 is a fragmentary cross-sectional view of a second embodiment of the final product obtained in accordance with the invention. Similar reference characters are applied to similar elements throughout the drawing.

Referring to Figure 1, a microscopic specimen 1 is secured to an extremely thin supporting membrane 3, such, for example, as a thin collodion membrane formed by dropping diluted collodion on a water surface. The collodion membrane 3 is supported by a metallic ring 5. This method of supporting electron microscopic specimens is well known in the art and requires no further explanation herein. A second supporting ring 7 retains a second supporting membrane 9 in the same manner as described heretofore. The second supporting membrane 9 is covered with an extremely thin electron permeable element 11 which is juxtaposed substantially in contact with the microscopic specimen 1.

The electron permeable element 11 comprises a thin layer of material which is sensitive to radiations of short wavelength such as, for example, soft X-rays or high velocity electrons. Such materials include metallic bichromates which may be combined with gelatin or gum arabic. For example, a satisfactory radiation sensitive element for use with soft X-rays may be obtained by employing a potassium bichromated gel supported by a thin membrane of collodion.

The radiation sensitive element 11, juxtaposed substantially in contact with the specimen 1, is subjected to soft X-rays or relatively high velocity electrons derived from a remotely situated point source of such radiations 13. An image characteristic of the permeability of the specimen to the radiation source is obtained upon the radiation sensitive element by transmission of the radiations through the specimen to the radiation sensitive element 11. The transmitted X-rays or high velocity electrons change the chemical composition, or tan, the radiation sensitive element 11 as a function of the radiation transmission characteristics of the specimen 1. It should be understood that many specimens which offer poor transmission for electrons are relatively pervious to X-ray. Hence, X-rays may be employed for exposing the radiation sensitive element 11 in instances where electron transmission by the specimen 1 would involve extremely long exposure intervals, or would necessitate extremely high velocity irradiating electrons. Any conventional means may be employed for providing the point source of X-ray or high velocity electron radiation.

After irradiation of the radiation sensitive element 11 for a time interval sufficient to provide a suitable relief image of the specimen, the radiation sensitive element 11, supporting membrane 9 and supporting ring 7 may be immersed in a solution for dissolving or removing the untanned or partially tanned portions of the exposed sensitive element. For example, if the radiation sensitive element comprises a metallic bichromated gelatin, the partially tanned and untanned portions thereof may be removed by immersing the element in warm water. If the metallic bichromate is combined with, for example, gum arabic, the untanned or partially tanned portions may be removed by immersing the element in cold water.

The completed relief copy of the microscopic specimen 1 is shown in the cross-sectional fragmentary views of Figures 2 and 3 wherein the upper surface is characteristic of the relief contour of the specimen 1 after immersion in the solution which dissolves the untanned or partially tanned portions. Figure 2 illustrates a cross-section of a tanned metallic bichromate gel which is self supported on the supporting ring 7. Figure 3 illustrates a similar completed relief copy of the specimen 1 wherein the tanned metallic bichromate gel is supported by a collodion membrane 9 which, in turn, is supported by the supporting ring 7.

It should be understood that various other materials not specifically disclosed herein may be substituted for the specific materials described without departing from the spirit and scope of the methods and products disclosed. For example, egg albumen may be substituted for the gelatin or gum arabic. The type of irradiating source also may be selected to provide the proper transmission contrast for the particular microscopic specimen which is to be copied and the particular radiation sensitive substance may be selected accordingly to provide the desired sensitivity and contrast.

Thus the invention disclosed comprises improved methods of and means for securing accurate relief copies of specimens by irradiation and chemical decomposition of radiation sensitive elements subjected to short wavelength radiations transmitted by said specimens.

Having regard to the foregoing disclosure, the patent of which this specification forms part confers, subject totthe conditioms prescribed in the Patent Act, 1935, the exclusive right, privilege and liberty of making, constructing, using and vending to others to be used, the invention as defined in claims submitted by the patentee as follows:-

1. The method of deriving from a radiation pervious element, having at least a radiation sensitive portion, an electron permeable relief copy of a microscopic specimen comprising juxtaposing said radiation pervious element in close relation with said specimen, irradiating at least said radiation sensitive portion by transmission through said specimen to provide a relief image on said sensitive surface, and modifying the electron permeability of portions of said irradiated element in relation to said image irradiation thereof.

2. The method of deriving from a radiation pervious element, having at least a radiation sensitive portion, an electron permeable relief copy of a microscopic specimen which is too thick for satisfactory continuous electron transmission microscopy comprising juxtaposing substantially in contact with said specimen said radiation sensitive portion of said radiation pervious element, irradiating said specimen by relatively short wavelength radiations, irradiating at least said radiation sensitive portion by transmission through said specimen to provide a relief image on said sensitive surface which is suitable for substantially continuous transmission microscopy, and removing portions of said irradiated element in inverse relation to said image irradiation thereof.

3. The method of deriving from a radiation pervious element, having at least a radiation sensitive portion, an electron permeable relief copy of a microscopic specimen which is too thick for satisfactory continuous electron transmission microscopy comprising juxtaposing substantially in contact with said specimen said radiation sensitive portion of said radiation pervious element, irradiating said specimen by relatively short wavelength radiations, irradiating at least said radiation sensitive portion by transmission through said specimen to provide a relief image on said sensitive surface which is suitable for substantially continuous transmission microscopy, and chemically removing portions of said irradiated element in inverse relation to said image irradiation thereof.

4. The method of deriving from a radiation pervious element, having at least an X-ray radiation sensitive portion, an electron permeable relief copy of a microscopic specimen which is too thick for satisfactory continuous electron transmission microscopy comprising juxtaposing substantially in contact with said specimen said radiation sensitive portion of said radiation pervious element, irradiating said specimen by X-ray radiations, irradiating at least said radiation sensitive portion by X-ray transmission through said specimen to provide a relief image on said sensitive surface which is suitable for substantially continuous transmission microscopy, and removing portions of said irradiated element in inverse relation to said image irradiation thereof.

5. The method of deriving from an electron pervious element, having at least an X-ray radiation sensitive portion, an electron permeable relief copy of a microscopic specimen which is too thick for satisfactory continuous electron transmission microscopy comprising juxtaposing substantially in contact with said specimen said X-ray radiation sensitive portion of said electron pervious element, irradiating said specimen by relatively short wavelength X-ray radiations, irradiating at least said radiation sensitive portion by X-ray transmission through said specimen to provide a relief image on said sensitive surface which is suitable for substantially continuous electron transmission microscopy, and removing portions of said irradiated element in inverse relation to said X-ray image irradiation thereof.

6. The method of deriving from an electron pervious element, having at least an X-ray radiation sensitive portion, an electron permeable relief copy of a microscopic specimen which is too thick for satisfactory continuous electron transmission microscopy comprising juxtaposing substantially in contact with said specimen said X-ray radiation sensitive portion of said electron pervious element, irradiating said specimen by relatively short wavelength X-ray radiations, irradiating at least said radiation sensitive portion by X-ray transmission through said specimen to provide a relief image on said sensitive surface which is suitable for substantially continuous electron transmission microscopy, and chemically dissolving and removing portions of said irradiated element in inverse relation to said X-ray image irradiation thereof.

7. The method of deriving from an electron pervious element, having at least an electron sensitive surface portion, an electron permeable relief copy of a microscopic specimen which is too thick for satisfactory continuous electron transmission microscopy comprising juxtaposing substantially in contact with said specimen said electron sensitive portion of said electron pervious element, electron irradiating said specimen, electron irradiating at least said electron sensitive surface portion by electron transmission through said specimen to provide a relief image on said sensitive surface which is suitable for substantially continuous electron transmission microscopy, and removing portions of said irradiated element in inverse relation to said electron image irradiation thereof.

8. The method of deriving from an electron pervious element, having at least an electron sensitive surface portion, an electron permeable relief copy of a microscopic specimen which is too thick for satisfactory continuous electron transmission microscopy comprising juxtaposing substantially in contact with said specimen said electron sensitive portion of said electron pervious element, irradiating said specimen, electron irradiating at least said sensitive surface portion by electron transmission through said specimen to provide a relief image on said sensitive surface which is suitable for substantially continuous electron transmission microscopy, and chemically dissolving and removing portions of said irradiated element in inverse relation to said electron image irradiation thereof.

9. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element which is produced by juxtaposition with said specimen, by irradiation by transmission through said specimen to provide a radiation relief image, and wherein the electron permeability of portions of said element are modified in relation to said image irradiation thereof.

10. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element which is produced by juxtaposition with said specimen, by irradiation by transmission through said specimen to provide a radiation relief image, and wherein portions of said element are chemically removed in inverse relation to said image irradiation thereof.

11. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element which is produced by juxtaposition with said specimen, by irradiation by transmission through said specimen to provide a radiation relief image, and wherein portions of said element are chemically dissolved and removed in inverse relation to said image irradiation thereof.

12. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element including bichromated colloids which is produced by juxtaposition with said specimen, by irradiation by transmission through said specimen to provide a radiation relief image, and wherein portions of said element are chemically removed in inverse relation to said image irradiation thereof.

13. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element including a metallic bichromate gel which is produced by juxtaposition with said specimen by irradiation by transmission through said specimen to provide a radiation relief image, and wherein portions of said element are chemically removed in inverse relation to said image irradiation thereof.

14. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element including a metallic bichromate and gum arabic which is produced by juxtaposition with said specimen, by irradiation by transmission through said specimen to provide a radiation relief image, and wherein portions of said element are chemically removed in inverse relation to said image irradiation thereof.

15. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element including metallic bichromated egg albumen which is produced by juxtaposition with said specimen, by irradiation by transmission through said specimen to provide a radiation relief image, and wherein portions of said element are chemically removed in inverse relation to said image irradiation thereof.

16. An electron permeable relief copy of a microscopic specimen said copy comprising a radiation sensitive electron pervious element including potassium bichromate and gelatin which is produced by juxtaposition with said specimen, by irradiation by transmission through said specimen to provide a radiation relief image, and wherein portions of said element are chemically dissolved and removed in inverse relation to said image irradiation thereof.

17. An electron permeable copy of a microscopic specimen comprising a radiation sensitive electron pervious element having the electron permeability of different portions thereof modified as a function of radiation transmitted through said specimen to said copy.

18. An electron permeable copy of a microscopic specimen comprising a radiation sensitive electron pervious element having the electron permeability of different portions thereof modified as a function of the radiation transmission characteristics of said specimen.

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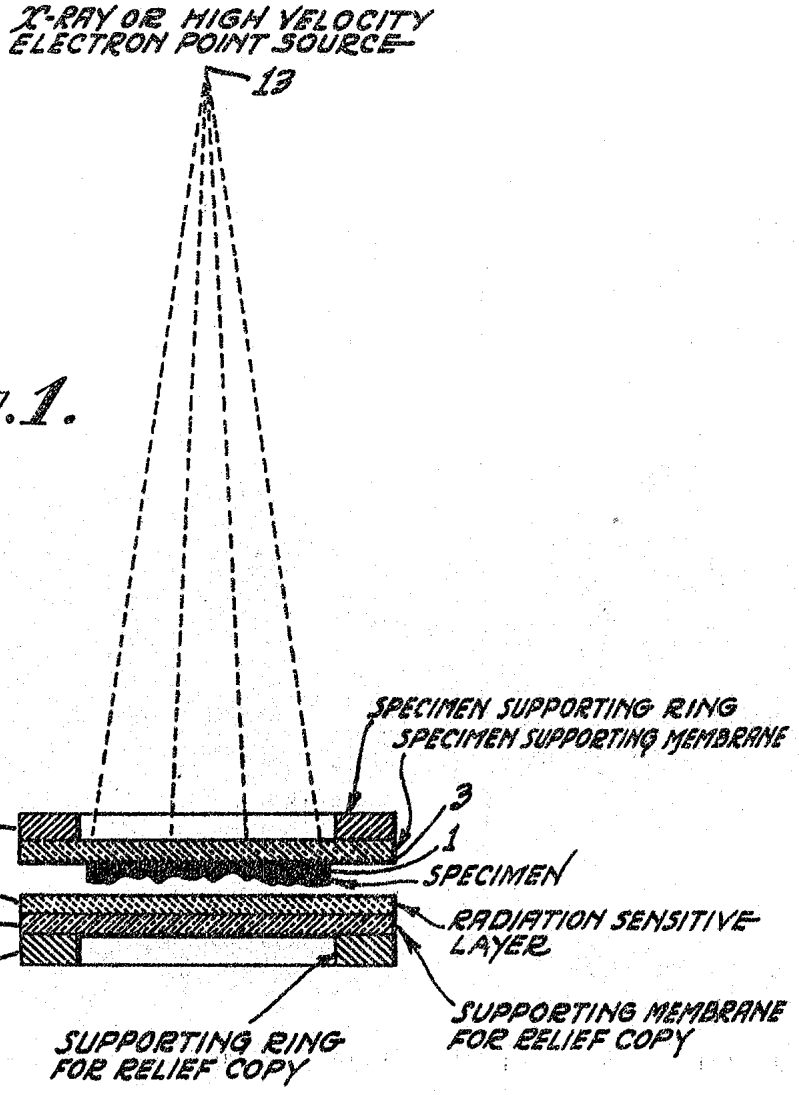

Fig. 1.

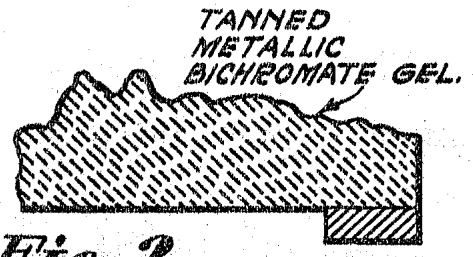

Fig. 2.

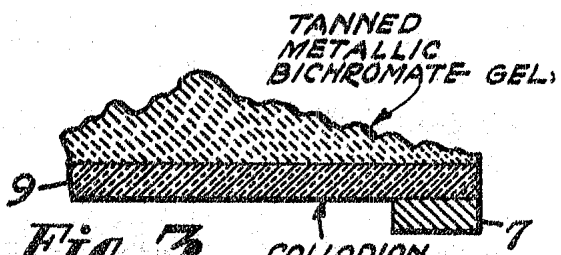

Fig. 3.

INVENTOR
James Hillier

Certified to be the drawings referred to in the specification hereunto annexed.

Montreal, Que., Oct. 31st 1944.

W. H. Haidy
ATTORNEY