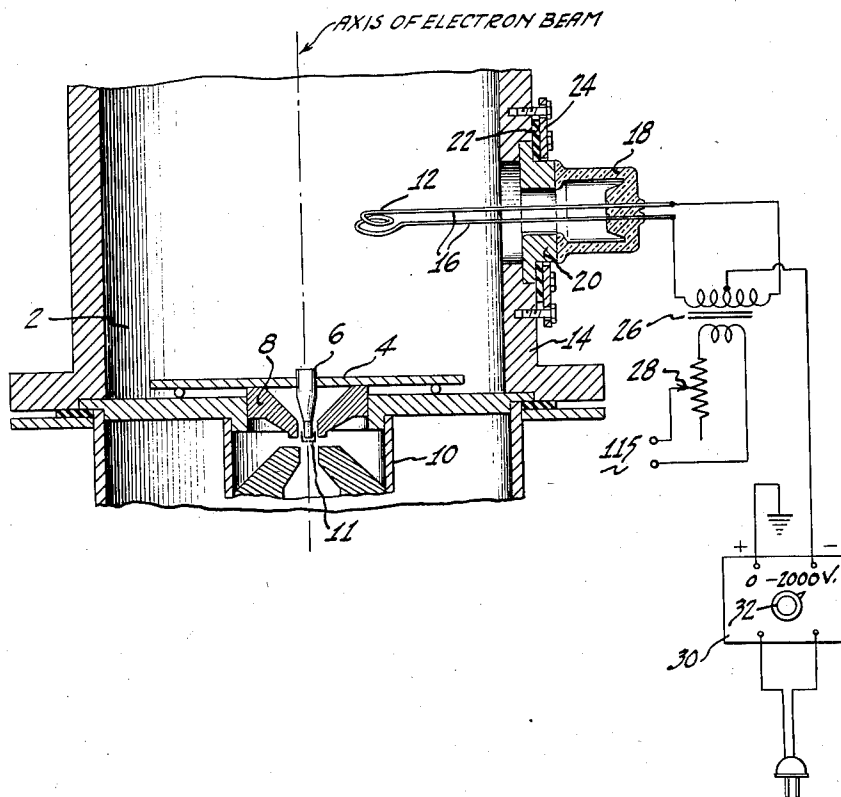


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REDUCING SPECIMEN CONTAMINATION
IN ELECTRON OPTICAL INSTRUMENTS
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REDUCING SPECIMEN CONTAMINATION IN
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The present invention relates generally to electron optical apparatus and, more particularly, to an improved method of reducing specimen contamination in an electron optical instrument utilizing a concentrated beam of electrons to bombard a specimen.

The method applies generally to electron microscopes, microanalyzers, diffraction cameras, and the like but is most effective in the probe type of instrument exemplified by the microanalyzer and the diffraction camera. These instruments utilize a more concentrated beam of electrons than does the microscope, for example, and contamination seems to be greatest in those types of apparatus in which electron bombardment is most intense.

In any demountable type of electron optical equipment in which a specimen is bombarded with a stream of electrons, difficulty is experienced in the continued observation of a single area of a specimen due to the deposition of a contaminating substance produced by the bombardment of the surface of the specimen by the electron beam in the presence of organic vapors.

In the electron probe type instrument where the localized bombardment is extremely intense, the rate of deposition is sufficient to render the specimen opaque to electrons within a few seconds of the start of bombardment.

The nature of the contaminating deposit has been the subject of much careful study by a number of workers. Carbon only was found by J. Hillier and R. F. Baker ("Further Developments of the Microanalyzer," J. Appl. Phys., vol. 16, p. 266, 1945). The instrument used by these workers was unable to detect hydrogen, however, so the contaminating substance could also contain hydrogen. Other workers have obtained results which seem to indicate that the chief chemical process involved in the contamination is the polymerization of a hydrocarbon with methane being the initial gas responsible.

Whatever the precise nature of the contaminating deposit may be, it seems to be clear that the source of the contamination is in the organic vapors which diffuse slowly out of the metal walls of the specimen chamber, and out of the synthetic rubber gaskets and lubricating greases of the instrument. There are indications that it does not come from the oil diffusion pumps. The contaminant is formed rapidly under electron bombardment and is immediately redeposited in solid form on areas bombarded by electrons.

The present invention relates to a method of preventing deposition of the contaminating sub-

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stance on the specimen and to simple apparatus for carrying out the method.

One object of the invention is to provide a method of preventing the deposition of carbonaceous deposits on a specimen under observation in an electron optical instrument.

Another object of the invention is to provide apparatus which may be incorporated in any electron optical instrument of the type which employs a beam of electrons to bombard a specimen and which may be utilized for preventing contamination of a specimen under observation in the instrument.

Another object of the invention is to provide a method of improving the operation of an electron optical instrument of the type which employs a concentrated beam of electrons to bombard a specimen.

Another object of the invention is to provide an improved method of subjecting a specimen to observation in an electron optical instrument.

Another object of the invention is to provide an improved method of operating an electron optical instrument of the probe type.

These and other objects will be better understood from a consideration of the following specification and illustrative drawings of which the single figure illustrates a partial view in longitudinal cross section of an object chamber of an electron microscope having an auxiliary electron source within the chamber, together with a schematic diagram of a control circuit for operating the auxiliary electron source.

The objects of the invention are achieved by spraying the walls of the object chamber with a stream of electrons for a period of time which depends upon the intensity at which the electron source is operated. This not only cleans the object chamber of vapors but also appears to release that gas which is present in the surface layers of metal of the object chamber walls and to re-deposit the gaseous material in solid form.

Referring to the figure, there is illustrated a cross section partial view of the objective chamber 2 of an electron microscope. The drawing has been simplified by omitting many of the parts unnecessary to an explanation and understanding of the invention. Within the chamber 2 may be a specimen stage 4 carrying a specimen cartridge 6. The specimen cartridge may be suspended within the entrance pole piece 8 of an objective lens having an exit pole piece 10. The specimen cartridge may carry a specimen holder 11, comprising a fine mesh screen with

clip, over its lower end. The specimen rests on the screen.

An auxiliary electron source 12 is inserted through the wall 14 of the object chamber 2 by means of a vacuum sealed unit. The electron source may consist of a simple filament of tungsten wire having leads 16 extending through the base of a glass insulator 18. The glass insulator may be sealed to a flanged sealing ring 20, which ring is firmly secured to a recessed portion of the wall 14 of the vacuum chamber. A gasket 22 may be provided to assure a vacuum-tight seal and the sealing ring may be secured to the chamber wall by means of a clamping ring 24 bolted to the chamber wall.

The filament 12 may be supplied with heating current through a filament transformer 26 connected to a source of 115 v. alternating current and provided with a regulating control 28. The secondary of the filament transformer may also be connected to a high voltage supply 30 having a control 32 for regulating the voltage. This high voltage source may be of a type which can supply 0-2000 v. at 50 ma. but sources supplying almost any other higher voltage up to at least 50,000 volts could also be used. The limitations in this respect are only those introduced by practical considerations. A voltage should not be used which introduces the danger of arc-over to the grounded equipment.

The preferred method of operation is as follows. The specimen to be observed in the usual way in the instrument is introduced into the usual specimen holder retained on the specimen cartridge and placed in the object chamber. The chamber is then sealed, the equipment is evacuated and all other steps preliminary to turning on the high voltage of the primary source of electrons are carried out. But before the high voltage source is turned on, thus activating the electron gun and bombarding the specimen with a beam of electrons, the auxiliary source of electrons in the object chamber is turned on for about 1 minute, using a current which may be at 50 ma. and at an accelerating potential of 1000 v. Electrons are emitted from the surfaces of the filament and travel in all directions toward the chamber walls. This usually cleans the system for at least a full day's operation. Lower or higher currents may also be used. Currents of only a few ma. are usable and a current of 15 ma. at 1000 v. provided a clean-up which was effective for several hours of operation. Since each type of instrument is different and operating conditions may differ from time to time, it is only possible to say that, in general, the chamber should be sprayed with electrons at an intensity and for a time sufficient to deposit in solid form any contaminating gaseous material present within the chamber, this being meant to include, also, any gaseous material within the wall structure close to the surface.

The manner in which the clean-up of the source of contamination is accomplished is not fully understood. It is believed, however, that, when the walls of the object chamber are sprayed with electrons, they become activated in some manner. This activation causes the walls either to adsorb or react chemically with the molecules of vapor.

Another source of contamination is the fine mesh metal screen utilized to hold the specimen being observed. These screens are usually made of copper. In the course of bombarding the specimen with electrons, the screen also is sub-

ject to bombardment. Where the cross-sectional area of the electron beam striking the specimen is relatively large, bombardment of the screen, itself, causes rapid release of vapors and so speeds up the reaction which causes contamination of the specimen that the effect cannot be completely eliminated by the electron spray method described. The effect of the screen can be lessened, however, by using a material such as stainless steel, platinum or tantalum instead of copper and strongly heating the screen to drive out the gases which the metal would normally contain.

In the electron probe type of instrument, such as the microanalyzer or diffraction camera, the beam is concentrated such that the screen is not bombarded to any extent. In this type of instrument, spraying, alone, by the method of the present invention is enough to reduce contamination to a negligible amount.

The reaction which causes deposition of the contaminating deposit on the specimen is believed to be catalyzed by the surface which is under electron bombardment. Certain metals appear to have a particularly great catalyzing action.

The method of the present invention differs from methods which spray the specimen, itself, with a low intensity stream of electrons for the purpose of removing an electrical charge. In the present invention, the specimen is not sprayed. The apparatus for carrying out the method is preferably designed such that electrons from the auxiliary source will not reach the specimen.

Although but a single embodiment of simple apparatus has been described for carrying out the invention, it will be obvious to those skilled in this art that many different designs of auxiliary electron sources could be used within the spirit of the invention and for carrying out the general method.

I claim as my invention:

1. A method of reducing contamination of a specimen in an electron optical apparatus comprising cleaning the object chamber of the apparatus prior to bombarding the specimen with an electron beam by spraying the walls of said chamber with electrons.

2. A method of reducing contamination of a specimen in an electron optical apparatus from carbonaceous deposits produced by decomposition of organic vapors which may be present in the object chamber of the apparatus, comprising spraying the walls of the object chamber with electrons prior to subjecting the specimens to electron bombardment.

3. A method of reducing contamination of a specimen in an electron optical apparatus of the type adapted to subject a specimen to a beam of electrons and having an object chamber for introducing the specimen into the apparatus, said method comprising spraying the walls of the object chamber with electrons prior to subjecting the specimen to electron bombardment.

4. The method of claim 3 in which the spraying operation is accomplished by projecting electrons from an auxiliary source of electrons within the object chamber.

5. In an electron optical apparatus which includes a primary source of electrons and a vacuum system including an object chamber, an auxiliary source of electrons within said chamber and means for projecting electrons from said auxiliary source to the walls of said chamber.

6. In an electron optical apparatus which in-

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cludes a primary source of electrons, an evacuable column including an object chamber and means within said chamber for holding a specimen in the path of a beam of electrons from said source, an auxiliary source of electrons within said chamber, said auxiliary source being positioned such that electrons projected therefrom

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will not strike the specimen on said holder, and means for projecting electrons from said auxiliary source to the walls of said chamber.

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No references cited.