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Original Paper

An Atmospheric Scanning Electron Microscope (ASEM)

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Abstract

A new detection configuration for the Scanning Electron Microscope (SEM) has been devised, which allows the imaging of the surfaces of a specimen in the open room, i.e., at atmospheric pressure. Such a device gives rise to a new microscope: the Atmospheric Scanning Electron Microscope (ASEM). In this configuration, a backscattered electron detector is placed between the pressure limiting aperture and the electron column. The electron beam passes through the final aperture, reaches the sample in the open room and the backscattered electrons passing through the same final aperture reach the detector. This principle has been tested and the result reported.

Introduction

In a recent publication (*Danilatos* and *Robinson* 1979), it was reported that specimens could be examined with an SEM having a specimen chamber pressure up to 60 mbar. The detection arrangement is shown in Fig. 1. A principal feature of this arrangement is that the backscattered electron detector (scintillator material) was machined thin enough to allow the placement of the specimen as close to the final aperture as possible (less than 1 mm). This minimizes the amount of electron beam scattering by the presence of gas molecules in the high pressure environment. Another feature is the use of two aligned apertures, one acting as objective aperture, the other as pressure limiting aperture (nominally 50 μm). The maximum pressure (60 mbar), for which useful images were obtained, does not constitute an absolute upper limit of pressure, but only a relative limit for the type of modification effected on the given microscope under the given conditions.

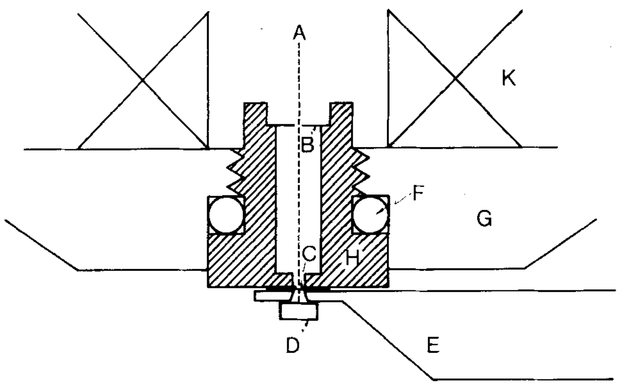

Fig. 1 The configuration for high pressure detection. A = electron beam, B = objective aperture, C = pressure limiting aperture, D = sample, E = backscattered electron detector (scintillator), F = sealing O-ring, G = pole-piece, H = aperture holder, K = scanning coils.

Suggestions have been made as to how the results can be improved, such as by the provision of additional vacuum pumping between the two apertures. Also, in principle at least, if the thickness of the detector is reduced, then even higher pressures could be obtained in the specimen chamber. However, the construction and operation of such a thin detector remains to be tested in practice.

In transmission electron microscopy, numerous attempts have been made by various workers to construct a high pressure environmental cell among which are methods by *Parsons* et al. (1974) as well as by *Swift* and *Brown* (1970)

The detection arrangement shown in Fig. 1 has already opened a new wide area for original research in electron microscopy and, therefore, could be considered the basis of a new instrument to be called Environmental Scanning Electron Microscope (ESEM).

The atmospheric scanning electron microscope (ASEM)

The present paper is concerned with the observation of samples with an SEM under atmospheric conditions. A schematic diagram of the basic components of the new device is shown in Fig. 2. The arrangement in

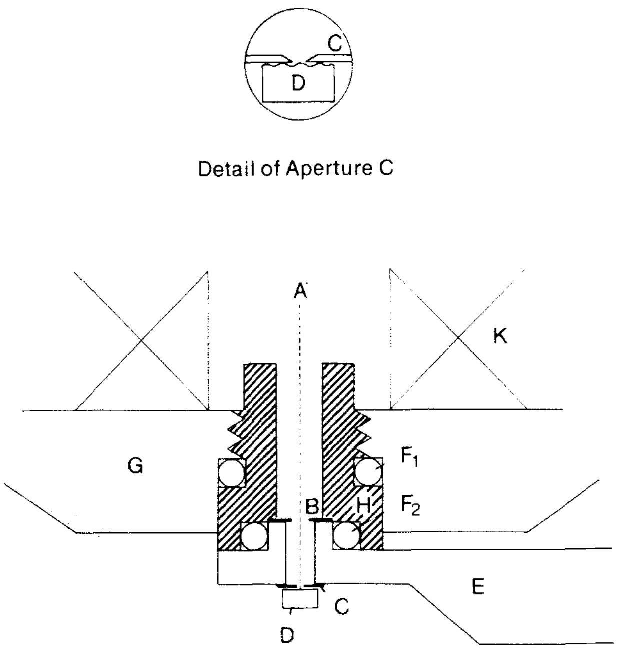

Fig. 2 The ASEM detection configuration. A = electron beam, B = objective aperture, C = pressure limiting aperture, D = sample, E = backscattered electron detector (scintillator), F_1 = aperture holder O-ring, F_2 = detector O-ring, G = pole-piece, K = scanning coils. The final aperture detail is shown in circle.

Fig. 2 is similar to that of Fig. 1 except that the backscattered electron detector and the pressure limiting aperture have interchanged their positions, i.e., the detector is placed between the final aperture and the electron column. The detector fits at the bottom of the column so that air leakage into the column can only take place through the aperture. The aperture can be chosen sufficiently small so that the vacuum in the column is high enough for normal operation of the microscope while the specimen is being examined in open air. The backscattered electrons passing through the final aperture reach the detector and produce the required signal if two conditions are fulfilled:

a) The specimen is placed at a distance from the final aperture, which is less than the diameter of the aperture.
b) The thickness of the aperture grid must be sufficiently smaller than the diameter of the aperture or alternatively the aperture must be conically shaped as is shown in circle in Fig. 2.

The above two conditions are necessary in order to achieve a sufficiently wide collection angle for the backscattered electrons.

The backscattered electron detector was constructed from scintillator material, but any other type of backscattered electron detector which is not affected by the presence of high pressure gas, could be used.

Results

The principle of the ASEM has been tested and proven by using a JEOL JSM-2 SEM. The pumping system of the microscope was modified as reported previously (*Danilatos* and *Robinson* 1979) and the configuration for detection of Fig. 2 was employed. However, the aperture grid used in the present work was neither sufficiently thin nor did it have a conically shaped aperture, as pointed out above. To demonstrate the principle of the ASEM it was sufficient to use an ordinary commercially available copper grid with an opening of 22 μm. The detector was constructed from scintillator material. The primary electron beam was passed through a hole in the detector having 1 mm diameter.

Preliminary observation of samples in an open room was possible with very encouraging results. Wool fibres and a dust particle were observed and recorded. In Fig. 3, two micrographs of the dust particle are shown at two different magnifications.

Discussion

The micrographs presented in this report are not designed to demonstrate quality of imaging, but rather to demonstrate the feasibility of the principle of the ASEM. The results have been obtained with a method departing considerably from the optimum conditions for best electron collection angle and vacuum pumping; these results are better than initially anticipated.

By using a 58 μm final aperture based on previous experience, a useful image of uncoated fibres could be obtained when the fibres were placed at 0.7 mm distance from the final aperture at a specimen chamber pressure of about 50 mbar. By extrapolation, the same image should be obtained if the sample was placed at 34 μm distance from a 13 μm diameter aperture at a specimen chamber pressure of 1013 mbar (i.e. one atmosphere).

Using the ASEM detection configuration, the detector can have a sizable thickness for all practical purposes, rather than a thickness of 34 μm required by the arrangement of Fig. 1. The beam scattering should be less if the sample is placed at a distance less than the magnitude of the final aperture (13 μm). It follows that problems should not occur as a result of scattering of electrons by the air layer above the sample.

When these calculations and assumptions were put to the test, encouraging results were obtained. The vacuum of the column could be maintained satisfactorily for the operation of the SEM even if a 22 μm fi-

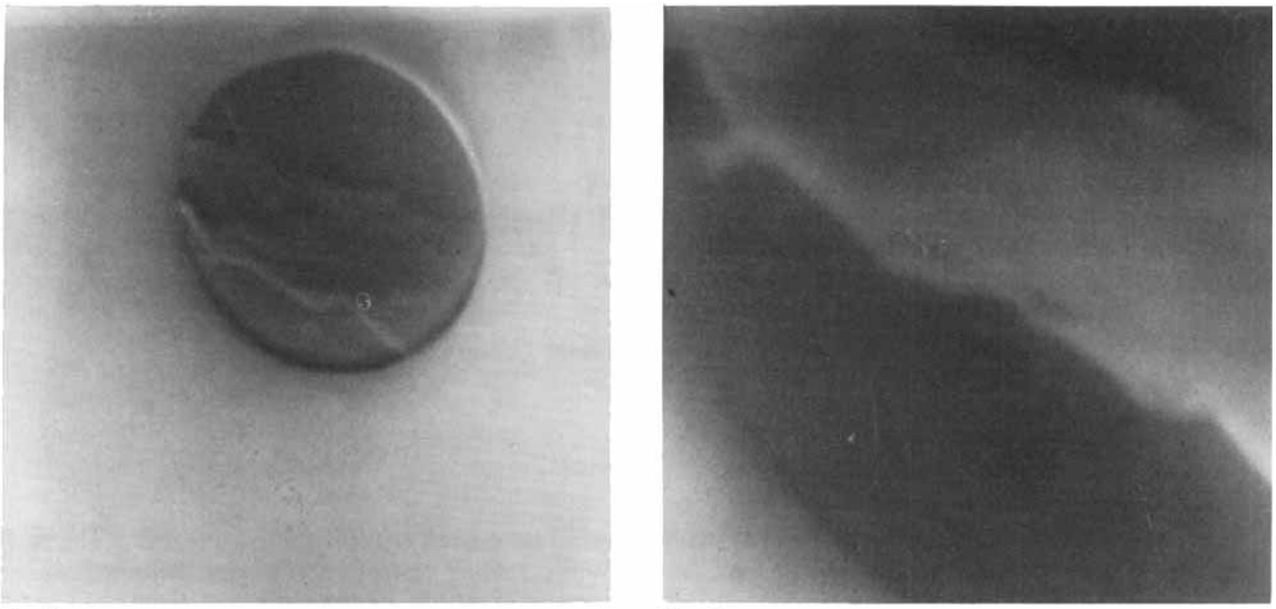

Fig. 3a Fig. 3b

Fig. 3 Two micrographs of a dust particle in open room at two different magnifications; a) horizontal field width = 45 μm, b) horizontal field width = 17 μm. The accelerating voltage used was 20 kV.

nal aperture was used. This result is very important as it means that lower magnifications could be achieved with the ASEM having a larger final aperture. The low magnification range obviously suffers from the restricted field of view due to the final aperture.

At this stage, it can be said that the ASEM is a reality and further developmental work continues. Conically shaped aperture grids can be manufactured and additional pumping between the two apertures, as suggested previously, will be introduced. The electron detection and imaging should be further improved by choosing appropriate materials for the final aperture grid, e.g., by choosing a stiffer material to prevent early deformation of the geometry around the hole, by coating the inside surface of the grid with a material of low atomic number (e.g. carbon), and also by coating the outside surface of the grid with a thin layer of scintillating material which, presumably, could contribute to the detection signal.

The disadvantages and limitations of the ASEM, e.g., the difficulty or impossibility of viewing liquid surfaces, fine powders, very rugged surfaces, etc. cannot be conclusively established at the present stage. The same applies to the advantages and the new vistas of research created by the ASEM.

However, it can be concluded that materials may be viewed in an open room, e.g., studies of fatigue of metals in situ can be realized. Examination of materials, not only in their natural state but also in their natural position, can now be realized. This is achieved by constructing a portable electron column. This feature of the ASEM could prove to be extremely useful in areas where sampling is impossible, expensive or critical for the test subject (skin, live tissue, archaeological findings, machine components, aircraft wings, etc.).

It should also be understood that, when the sample is examined in an open room, the examined surface experiences a pressure less than atmospheric due to the pressure gradient existing around the final aperture as outlined by *Parsons* et al. (1974).

An alternative mode of operation of the ASEM can be at intermediate pressures in the specimen chamber, e.g., at 400 mbar. In this case, a larger final aperture can be used, allowing a bigger field of view and a general improvement in imaging at the expense of pressure.

In conclusion, the principles for the manufacture of a new instrument, the ASEM, have been laid down and shown to work. Further work is in progress, the results of which will be reported in a later paper.

References

Danilatos G D, Robinson V N E: Principles of scanning electron microscopy at high specimen chamber pressures. Scanning 2, 72–82 (1979)

Parsons D F, Matricardi V R, Moretz R C, Turner J N: Electron microscopy and diffraction of wet unstained and unfixed biological objects. Advances in Biological and Medical Physics, 161–271 (1974)

Swift J A, Brown A C: An environmental cell for the examination of wet biological specimens at atmospheric pressure by transmission scanning electron microscopy. J Phys E 3, 924–926 (1970)