MICROSCOPY RESEARCH AND TECHNIQUE 25:354–361 (1993)

Introduction to the ESEM Instrument

G.D. DANILATOS
ESEM Research Laboratory, North Bondi (Sydney), NSW 2026, Australia

KEY WORDS Environmental SEM, Scanning electron microscope, Gaseous detection, Differential pumping

ABSTRACT An outline is presented of the first commercial environmental scanning electron microscope (ESEM). A concise description of this instrument and its operation, from a user's perspective, is given. More specifically, the description includes the electron optics, pressure stages and control, detection modes, resolution, and ancillary equipment.

INTRODUCTION

A review and outline of the general principles and applications of environmental scanning electron microscopy (ESEM) is presented elsewhere (Danilatos, 1991). Broadly speaking, an ESEM allows the examination of surfaces of practically any specimen, wet or dry, insulating or conducting, because it allows the introduction of a gaseous environment in the specimen chamber. The tolerance of gaseous pressures above 609 Pa (4.6 Torr) allows the maintenance of a saturated vapor pressure at temperatures above the freezing point and, hence, the presence of liquid water. The presence of a positive ion supply from the ionized gas ensures the suppression of negative charge buildup on insulating specimens. Furthermore, the gas also serves as a detection medium by way of monitoring the products of signal-gas interactions. Two such interactions are gaseous ionization and gaseous scintillation, both of which have produced excellent imaging capabilities. With a gaseous detection device (GDD) both secondary (SE) and backscattered electron (BSE) images can be produced. Solid scintillating detectors and light pipes can be integrated together with the gaseous detector, with the differential pumping system and the objective lens of the microscope. Therefore, the SE, BSE, and cathodoluminescence (CL) modes are fully represented. The X-ray mode (both EDS and WDS) can also be practiced. An ESEM is not limited to operating only at higher gas pressures: by definition, an ESEM operates in the pressure range from high vacuum to higher than 4.6 Torr pressures (Danilatos, 1978). In future developments, it is envisaged that a universal system of integrated detectors will be incorporated so that the same instrument can operate virtually with all modes of detection within a broad range of all its parameters, such as gas pressure, accelerating voltage, beam current, magnification, scanning rate, and working distance. All types of electron guns can be used, namely, tungsten, lanthanum hexaboride (LaB_6), and field emission. The resolving power of ESEM is ultimately limited by the diameter of the electron probe produced by a given electron optics system. The presence of gas results only in scattering and removing fraction of electrons from the original beam into a broad electron skirt that surrounds the original spot. For a detailed theory and practice of contrast and resolution in ESEM, the reader is referred to the published literature (Danilatos, this issue).

The purpose of this introductory article is to describe the actual instrument used in the papers contained in this issue and elsewhere. The various pressure stages are defined. Detailed diagrams of both SE and BSE detectors are given so that images obtained with them and reported herewith and elsewhere can be better evaluated. A comparison of images with an Everhart-Thornley (E-T) and GDD detector is given. This presentation clearly defines the commercial instrument used, which can thus be distinguished from the general science of ESEM.

ELECTROSCAN'S ESEM

A concise description of the basic aspects of the ElectroScan ESEM (E-S/ESEM) is presented below and, in particular, the one known as model ESEM-20, or having the Type-3 column. For this purpose, only those features are given which are more pertinent to the materials and methods and to the interpretation and understanding of imaging, whereas a tabulation of the manufacturer's specifications can easily be found elsewhere.

Electron Optics, Pressure Stages, and Control

This instrument incorporates a special design of electron optics column that allows a number of pressure levels, or stages, to be maintained along the path of the electron beam as shown in Figure 1. The differential pumping system involves a number of pumps and valves as shown in Figure 2. The ion pump is normally used with a LaB_6 type of gun and adds an extra differential pumping stage (i.e., when a tungsten gun is not in use). Therefore, in total, four or five pressure levels are created inside this particular instrument. In the diagrams, we have called the specimen chamber the 0^{th} stage, where all the thermodynamic parameters are assumed to have a homogeneous equilibrium value, such as stagnation pressure p_0, temperature T_0, etc.; in

Received December 21, 1992; accepted in revised form April 3, 1993.

Address reprint requests to G.D. Danilatos, ESEM Research Laboratory, 98 Brighton Boulevard, North Bondi (Sydney), NSW 2026, Australia.

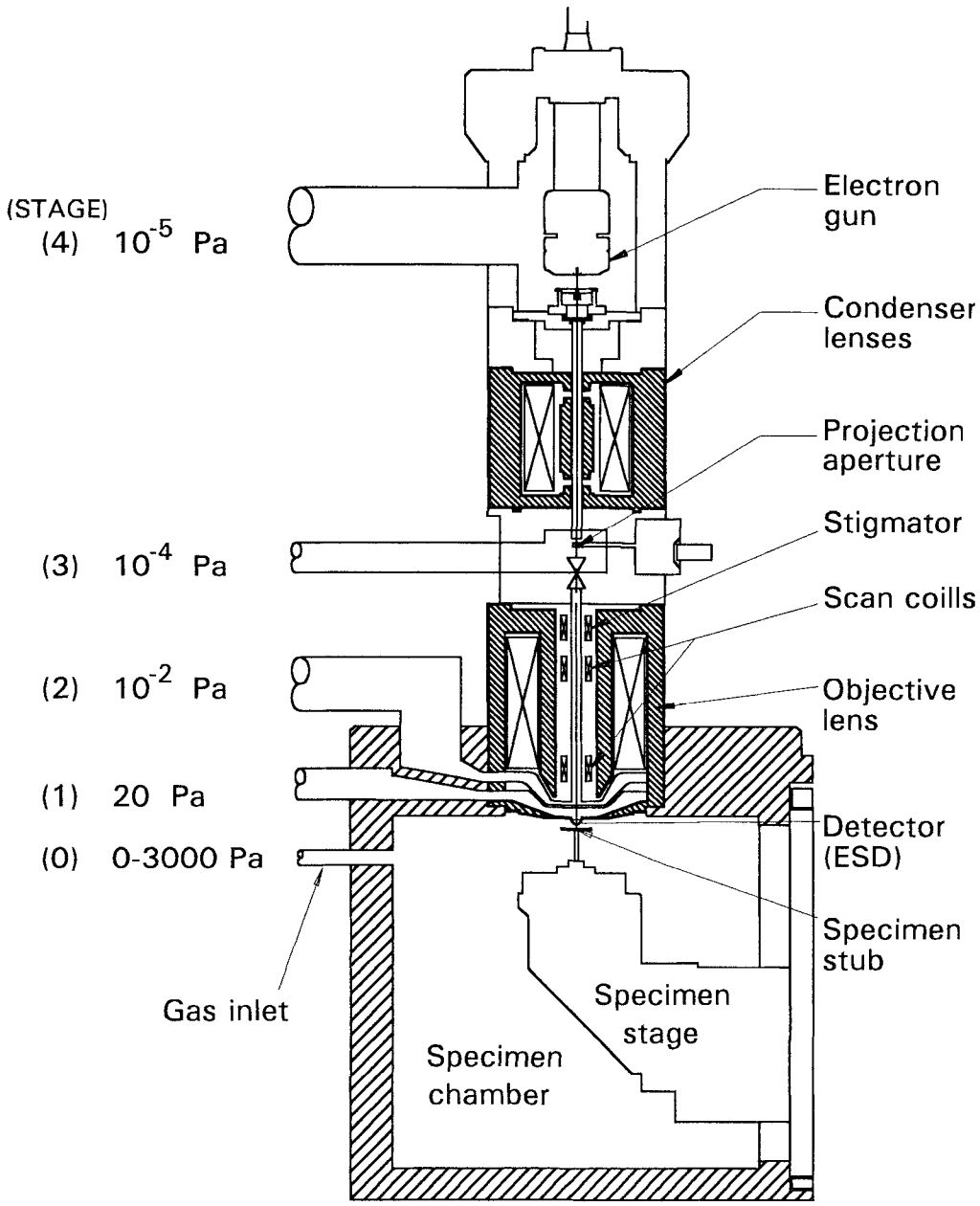


Fig. 1. ElectroScan ESEM model 20 (Type-3 electron optics column).

stage 1, we have p_1, T_1, and so on. The stages are separated by corresponding pressure limiting apertures (PLA). The objective lens, in particular, contains two pressure stages as shown in Figure 3. Two pressure limiting apertures, PLA1 and PLA2, are inserted with a single aperture holder, sometimes known as the *bullet*. This holder has lateral openings for the gas to flow freely, sideways, between the two apertures.

The differential pumping system is fully automated by a computer program that controls the proper opening and closing of various valves and pumps during column or specimen chamber venting. The nominal specimen chamber pressure goes up to 25 Torr, but this pressure level can be raised by use of a smaller PLA1. The pressure in the specimen chamber is measured with an absolute pressure gauge in Torr units (1 Torr = 133.33 Pa) and is indicated at the bottom of micrographs with an alphanumeric expression of the form "P = 8.0T" for pressure of 8.0 Torr, as in Figure 8a,b. Different gases, separately or in mixture, can be introduced in the specimen chamber. Because many ESEM operators wish to control the level of moisture in or around their specimen, and because water vapor is a good imaging gas, a water reservoir is connected to the

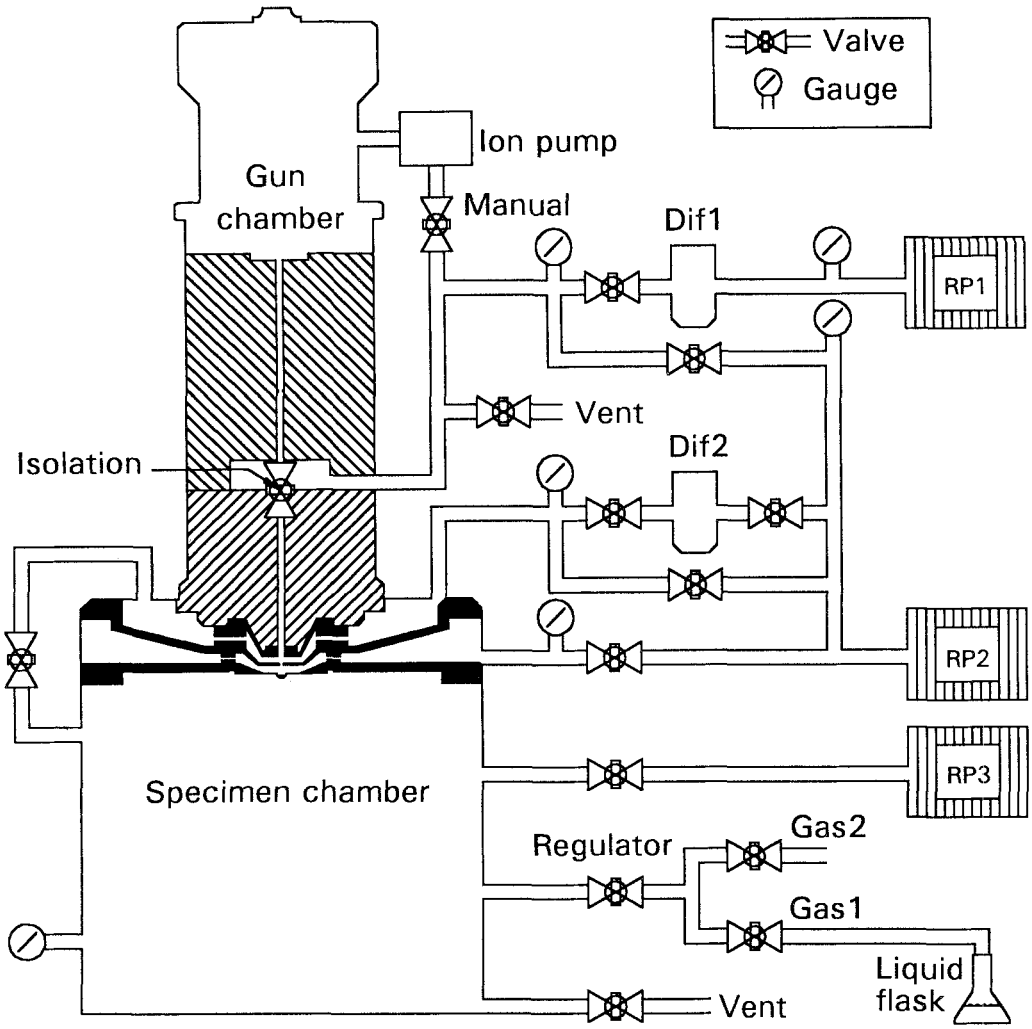


Fig. 2. Differential pumping system of E-S/ESEM. Dif = diffusion pump; RP = rotary pump.

specimen chamber. However, the user has the option of using any other gas that would best suit a particular application.

Secondary Electron Detection

The gaseous detection device (GDD) is the main mode of detection upon which this instrument is based (Danilatos, 1990). In particular, the ionization GDD, which utilizes the ionization of the gas for the detection of secondary electrons from the specimen surface, is the prime feature of the instrument. To stress the possibility of SE detection in a gaseous environment, Electro-Scan introduced the acronym ESD (for environmental secondary detector). An expanded view of this detector is shown in Figure 4, with actual dimensions and proportions. The critical component of the detector consists of an electrode that is integral with PLA1. The electrode has the shape of a truncated cone, of which the small flat base has a diameter of 1.4 mm. The PLA1 diameter is 0.5 mm and the edge (or rim) thickness is 0.3 mm. The specimen can be physically placed at any distance below the electrode, as allowed by the specimen stage movement. On the micrographs recorded and presented, we usually see an alphanumeric expression stating the distance of the specimen, but it must be clarified that this distance (D) is measured from the bottom of the pole-piece as is shown in the diagram. This distance contains a fixed component (usually 5 mm), which is the length of the standard bullet protruding below the pole-piece. Therefore, the distance that mostly matters to the user is the difference $x = D - 5$ (in mm), which we may call *clearance*. In reporting a (working) distance with micrographs, sufficient information to deduce the clearance must also be provided if non-standard bullets are used. The detector electrode is biased with a few hundred volts (positive), via a floating wire (connector) that is connected to the input of a preamplifier and associated electronics located at the specimen chamber wall.

The E-S/ESEM fulfils its basic aim of providing ex-

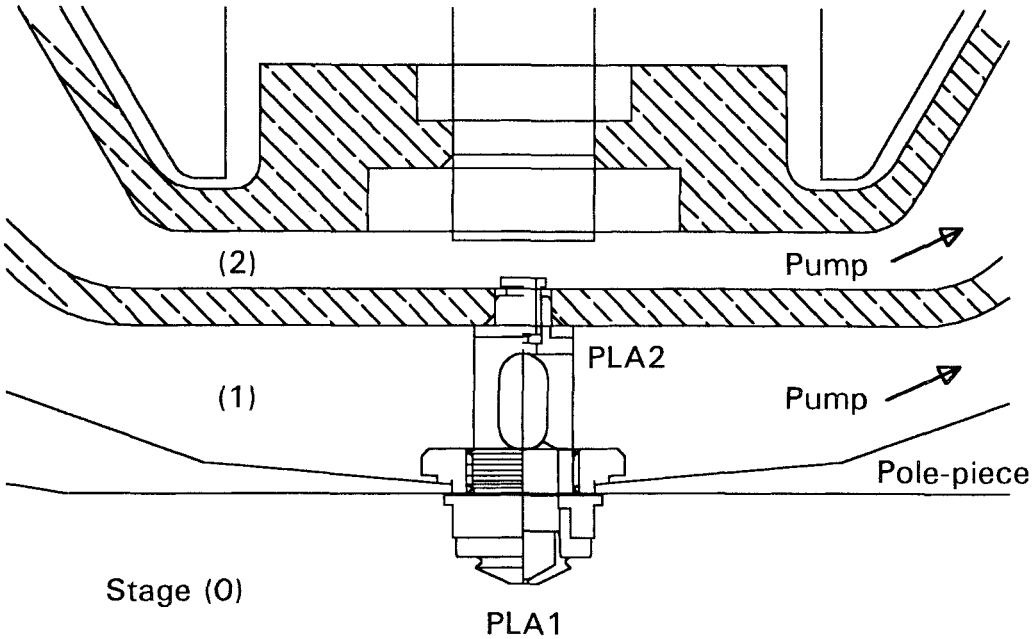


Fig. 3. Differential pumping integrated with objective lens. The final pressure limiting aperture (PLA1) separates the specimen chamber from the rough vacuum of the next stage (1), which is separated with aperture PLA2 from the following stage (2). Both apertures are inserted with a common holder, the bullet.

cellent and faithful SE imaging within a particular range of operating conditions. The principle of ESD is explained in Figure 5. I et us consider a low energy electron emitted from the surface of an electrode in a gaseous environment within an electric field. This field is produced by applying a potential to another electrode above the first. This electron is accelerated between consecutive collisions with the gas molecules. There is a significant probability with each collision that an additional electron will be liberated so that an avalanche process ensues. The necessary conditions are that the applied external field imparts sufficient energy to the electrons between consecutive collisions to ionize the gas and that the mean ionization free path is smaller than the total travel distance. The precise interplay among various parameters for the avalanche process can be found elsewhere (Danilatos, this issue). The important point is that the ESD acts as a proportional amplifier and, therefore, the output from this amplification process faithfully represents the input signal. With the present geometry of ESD, when the specimen is placed within 1 or 2 mm below the PLA1 and at pressures below about 10 Torr, the dominant signal is that of the secondary electrons from the specimen surface. However, as we raise the pressure or distance, the backscattered electrons start producing a significant proportion of primary ionization in the gas. This component also gets amplified in the gas and contributes to the formation of image. Subsequent electronics circuitry is used to further amplify, integrate, and average the resulting signal. Therefore, we can be confident about the physical meaning of the produced images.

Backscattered Electron Detection

Optionally, a plastic scintillating BSE detector is fitted as shown in Figures 6 and 7. This detector is made from clear acrylic material coated with a film of plastic scintillator. The scintillating coating is made by painting a toluene solution of Nuclear Enterprises scintillator. The light pipe has a double bend before it finishes at the window of a "head-on" photomultiplier (PMT) (some early users may still have a "side-on" window PMT). The light pipe starts with a flat shape at the detecting end with about 4 mm height and 22 mm width, which gradually converges to a cylincrical shape of 12 mm diameter towards the PMT. At the flat end, there is a conical hole so that the detector fits around the bullet as shown in Figure 7. Laterally, in the light pipe, there is also a groove, through which the connector electrode from the ESD passes. The effective angle subtended by the detector at the specimen is delineated by the dotted lines shown. This angle is a function of the specimen positioning, and a typical situation is shown in Figure 7.

The geometry of this particular detector produces better images at long working distance, at low pressure, and at high keV, conditions which favor a better electron collection angle. For a wet environment, the specimen is usually placed around 1 mm from the PLA1 and the detector accepts only the low take-off angle electrons with a preferential detection of those BSE striking the side of the detector in the direction of the PMT. Those BSE emitted at high take-off angle (within angle Φ in Fig. 7) lose most of their energy in collisions between the walls of ESD, pole-piece, and specimen, and

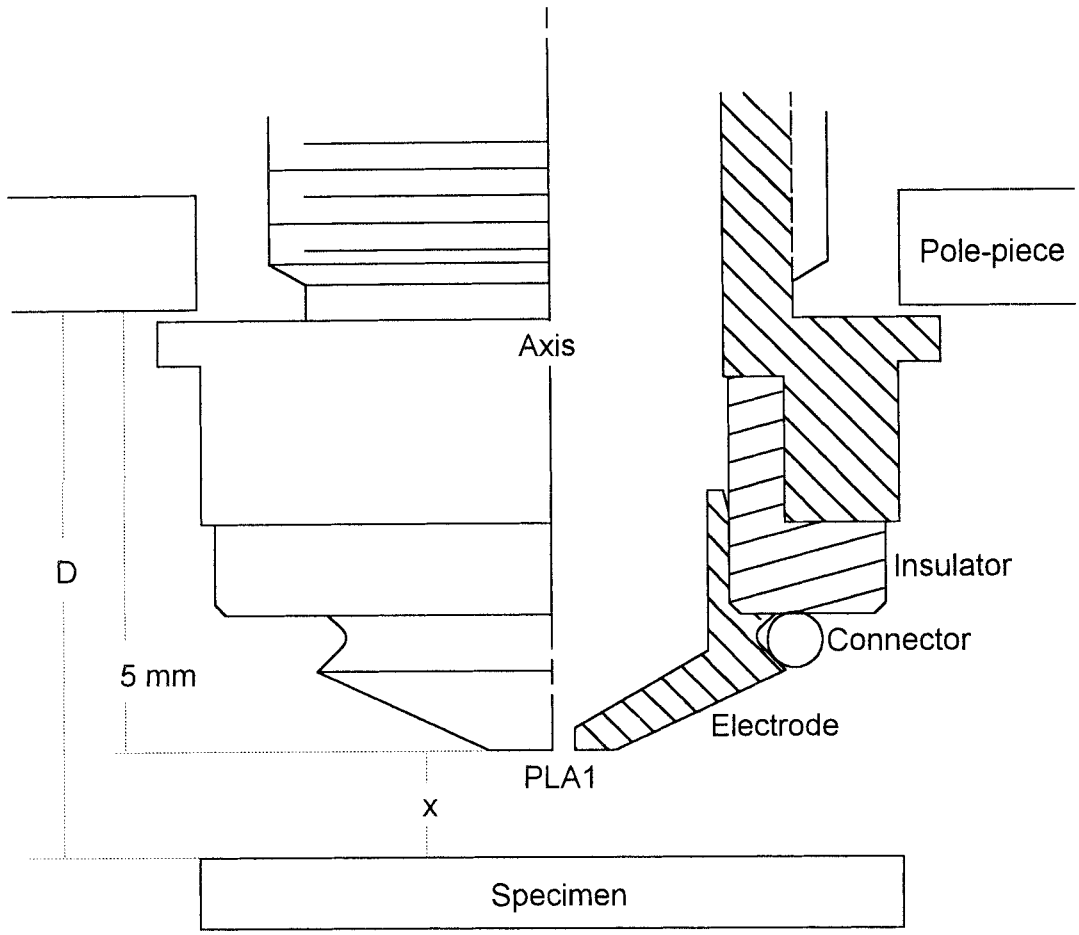


Fig. 4. Enlarged view with actual proportions of the environmental secondary detector (ESD). The detector electrode is integrated with PLA1 and the connector wire feeds the signal to accompanying electronics.

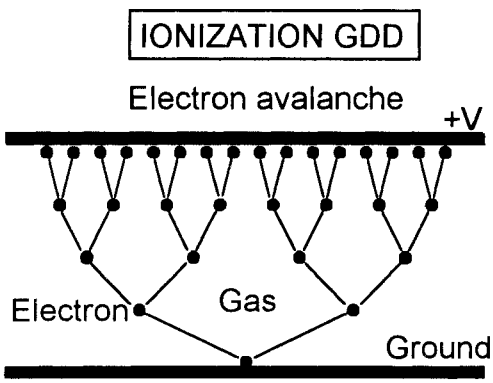


Fig. 5. Electron avalanche multiplication generated in the gas from a single secondary electron by the applied potential difference (+V) between electrodes.

only a fraction of energy is dissipated on the BSE detector after multiple-backscattering among the walls. Generally, this detector is characterized by a directionality (shadowing) from the low collection angle and from the photon losses around the hole and light pipe.

Resolution

It has been shown that the resolving power of the instrument is ultimately limited only by the diameter of the probe formed by the unscattered fraction of electrons. Sufficient current in the probe to create satisfactory contrast for a particular feature is also required. The diameter of the useful probe is the same as the original diameter formed in vacuum. This can be demonstrated by comparing a pair of micrographs of a test resolution specimen, such a gold on carbon. In Figure 8a, the image was obtained with a conventional E-T detector in vacuum. An image with the ESD at 8 Torr is shown in Figure 8b. We see no effects or loss of resolution due to a hypothetical spot-spreading, which has been shown theoretically not to exist. Therefore, the same resolution is obtained in both cases.

Other Options and Ancillaries

The E-S/ESEM is supplied with the ESD as the standard imaging mode. However, the other imaging modes

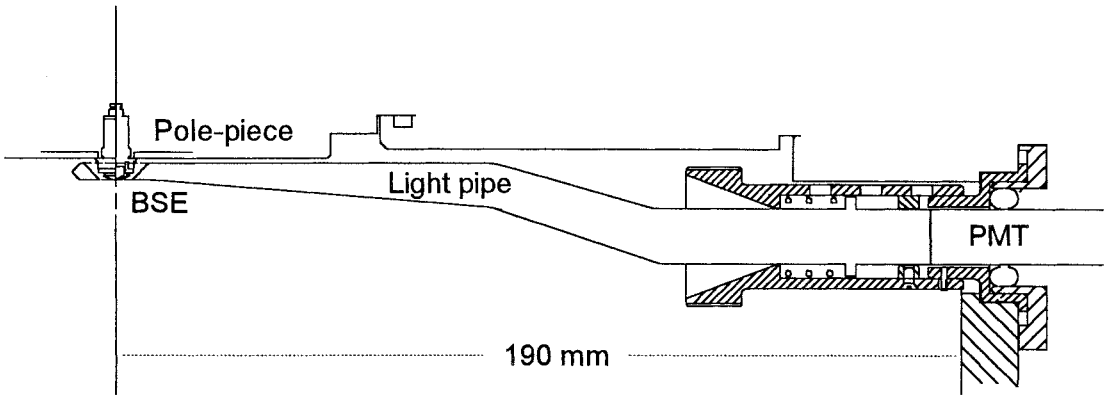


Fig. 6. Plastic scintillating BSE detector with actual proportions and positioning.

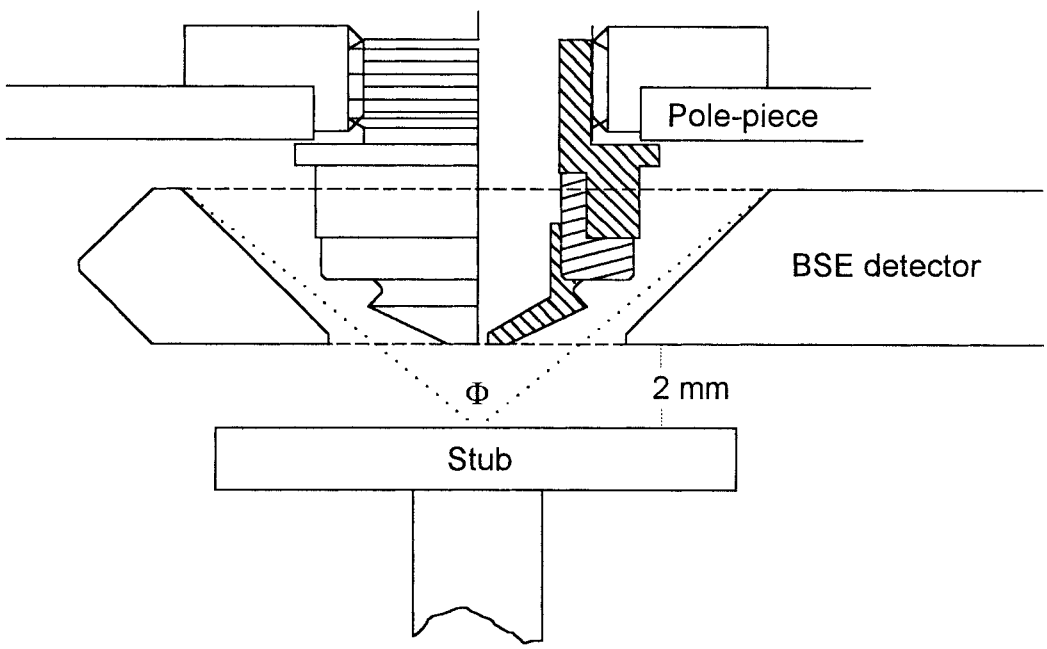
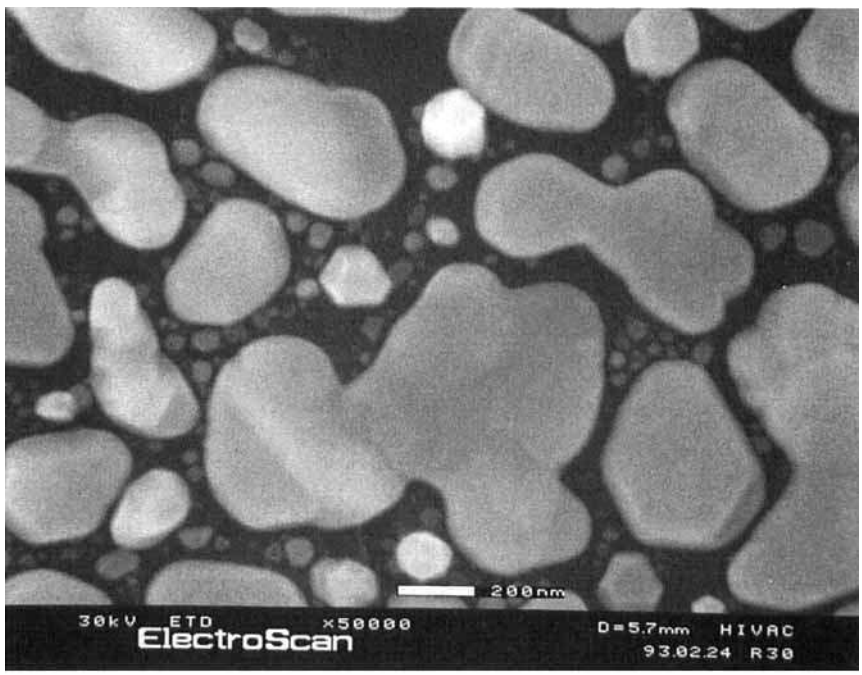


Fig. 7. Enlarged view showing the angle subtended by plastic BSE detector, together with ESD detector and specimen positioning.

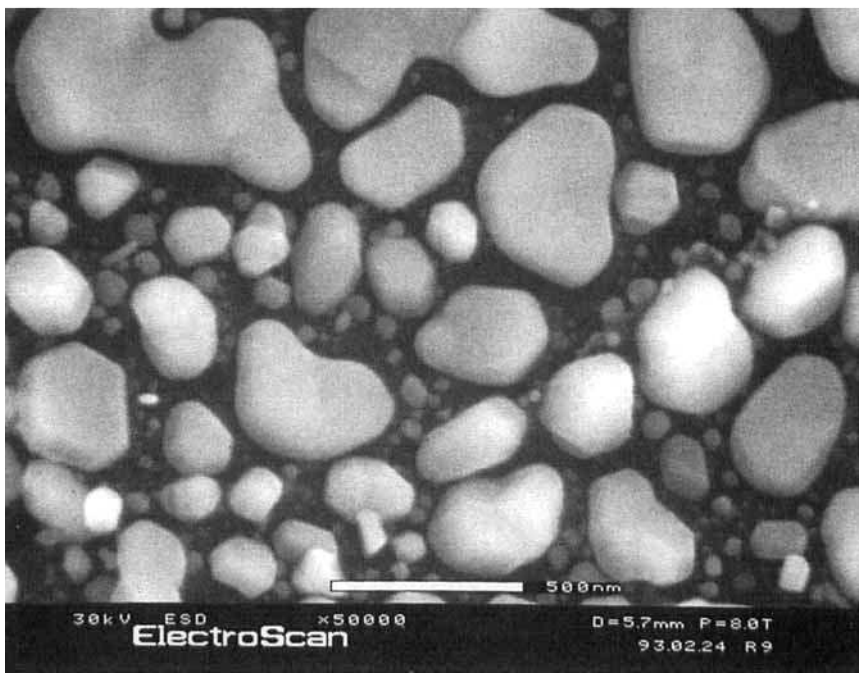
are also of great significance. These other modes offered as "options" should be distinguished from other "options" which relate to ancillary equipment for individual applications. The first set of options (i.e., alternative imaging modes) are integral and an inalienable part of ESEM technology. In general, we may refer to them as *ESEM accessories* for the E-S/ESEM. The second set of options are or may be made to aid ESEM technology, and we may refer to them as *ESEM ancillaries*.

In the ESEM accessories category of options there are BSE detectors, windowed X-ray detectors, CL detectors, and a conventional E-T detector (for high vacuum imaging). These options can be accompanied with the usual hardware for signal and image manipulation.

Examples of ESEM ancillaries, some already available and some potentially attachable to the instrument, are (1) a cooling stage based on the Peltier effect (this helps control the temperature of the specimen, especially when we wish to finely control the relative humidity, which is a strong function of temperature); (2) a hot stage, which helps control and raise the temperature by several hundred or even by more than 1,000°C; (3) a microinjector to supply a flow of various liquids, or gases, which helps study in situ surface reactions with various agents; (4) micromanipulators for mechanical deformations such as tensile, twist, and fatigue studies can be incorporated; and (5) other major techniques and equipment may be interfaced, such as mass spectrometer, atomic force microscope, scanning tunnelling mi-



a



b

Fig. 8. **a:** SE image obtained with E-T detector at high vacuum. **b:** SE image obtained with ESD at 8 Torr pressure (gold on carbon).

croscope, ion beam milling, thermal analysis routines, thin film deposition, lasers, UV sources, etc.

CONCLUSIONS

The above description provides a reference point for E-S/ESEM users. This machine represents a radical step forward in the instrumentation of electron microscopy and, for the first time, it allows the general user access to areas of research not previously possible. In particular, the formation of SE images of uncoated and untreated wet or insulating specimens has ushered a new era in many areas of research. X-ray microanaly-

sis of the same specimens has started yielding some very useful information. It is envisaged that the advent of this particular instrument is only the precursor of a new series of equipment to cover and serve the wider possibilities and needs of ESEM science.

REFERENCES

Danilatos, G.D. (1978) Foundations of environmental scanning electron microscopy. Advances in Electronics and Electron Physics, 71: 102–250.

Danilatos, G.D. (1990) Theory of the gaseous detector device in the environmental scanning electron microscope. Advances in Electronics and Electron Physics, 78:1–102.

Danilatos, G.D. (1991) Review and outline of environmental SEM at present. J. Microsc., 162:391–402.