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# AN ELECTRON LUMINESCENCE MICROSCOPE

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#### INTRODUCTION

mens with electrons frequently shows a mainly monochromatic emission of photons in the visible region of the spectrum. In practice, the corresponding wavelength is a qualitative guide in identifying the composition of the specimen.

A widely known similar effect - although more limited in variety of wavelengths and brightness of emitted light - appears with ultra-violet irradiation. In preliminary experiments it was found that, besides specimens suitable for u.v.-luminescence, many u.v.-insensitive specimens gave excellent electron luminescence effects.

Electron luminescence has been known as a sideline phenomenon since the existence of electron micro-analyzers. As these instruments are not specifically constructed for investigation of electron luminescence, it was decided to design and manufacture a relatively cheap instrument, more suitable for this special application. Besides the requirements of reliability and easy operation, the following feaes were realized.

- 1. Electrons emitted from a directly heated tungsten filament are focused with a variable electromagnetic lens on the surface of the specimen with a focal spot of 30 m or larger.
- 2. The electron luminescence of the specimen is simultaneously observed through a light optical microscope with variable magnification (max. 300x). Moreover, the specimen can be studied with either incident or transmitted polarized light.
- 3. In order to make the instrument suitable for fast inspection of large series of specimens, the entire specimen-changing time consisting of air bleeding, specimen-changing and evacuating down to  $10^{-2} \text{ N/m}^2$  (about  $10^{-4} \text{ torr}$ ) is kept

- within 45 seconds by reducing the volume of the specimen chamber and automating the vacuum system.
- 4. Besides the electron-induced luminescence effects, it is also possible to bring out generated X-rays with wavelengths characteristic of the elements composing the specimen, through two vacuum-sealed beryllium windows in the specimen-chamber wall.

### ELECTRON OPTICS (Figure 1)

To avoid the usual large dimensions of the electron optical column, both the triode electron gun<sup>4</sup>) and the electromagnetic lens<sup>7</sup>) are miniaturized.

The tungsten filament<sup>1)</sup> is standard Philips design and is interchangeable. The Wehnelt cylinder<sup>2)</sup> is centred around the filament tip with three screws<sup>3)</sup>.

The acceleration voltage for the emitted electrons is variable up to 30 kV and beam current control is obtained in a self-biasing electron gun circuit.

The entire electron gun can be mechanically centred<sup>6</sup>) around the fixed electromagnetic lens axis.

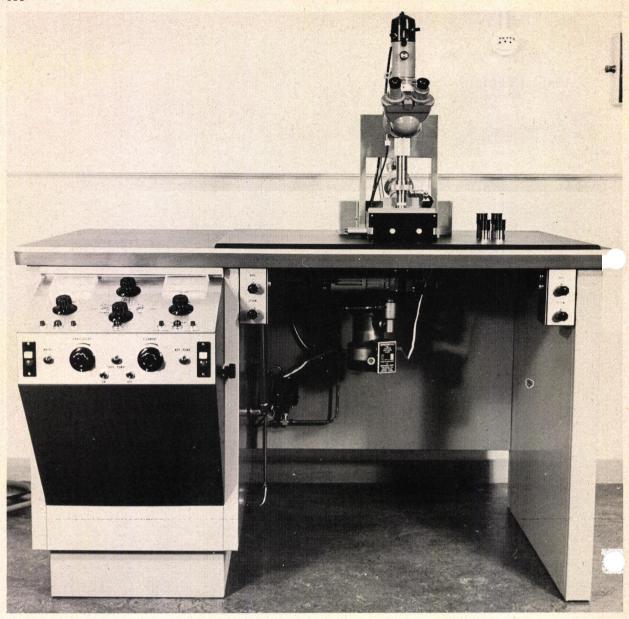
Besides the main focusing coil of the lens, x-y deflectors and stigmators mounted along the circumference provide easy electron beam tilt or astigmatism correction. Proper adjustment of the x-y deflectors effects coincidence of the electron beam spot on the specimen-plane with the viewing centre of the light optics. A stable D.C.-current supply feeds the electromagnetic lens assembly.

#### LIGHT OPTICS (Figure 2)

Both illumination systems for reflected and transmitted light consist of a light source<sup>1)</sup> with variable aperture<sup>2)</sup>, a condenser lens<sup>3)</sup> and a polarizer<sup>5)</sup> mounted in a bearing<sup>4)</sup>.

The specimen is imaged through an objective lens<sup>9)</sup>, (N.A. = 0.25), an 80% reflecting prism<sup>6)</sup>

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and a binocular viewing head<sup>7</sup>) with interchangeable eyepieces. An overall magnification range of 90 x, 150 x and 300 x is obtained with corresponding 6 x, 10 x and 20 x eyepieces.

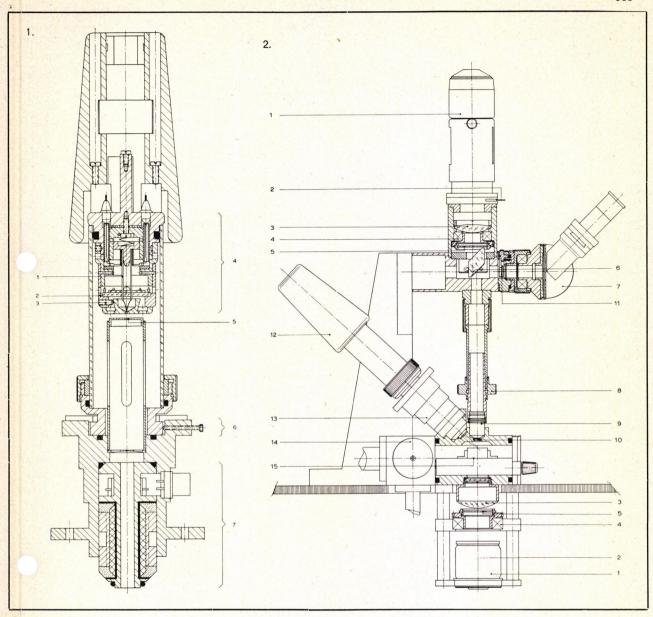
The large clear object distance of 11 mm is chosen in order to keep the light optics completely outside the evacuated specimen chamber and separated from it by a plane parallel glass window<sup>10</sup>). In case of contamination, the window can be easily interchanged.

The objective lens is focused with a rotating dial<sup>8</sup>). The polarizers<sup>5</sup>) are rotated simultaneously with the analyzer<sup>11</sup>) mounted in front of the binocular viewing head. Simultaneous rotation of all polari-

zers - unlike conventional polarizing microscopy - is required because of difficulties in manufacturing a specimen stage with accurate rotation around the fixed optical axis, independent of the orthogonal specimen translations and mounted inside the evacuated specimen chamber.

#### THE SPECIMEN STAGE AND CHAMBER

The specimen stage is designed for cylindrical specimens with diameters up to  $25.4~\mathrm{mm}$ , or glass substrates (76 x 26 mm are incorporated. The entire specimen stage can easily be taken out of the spe-



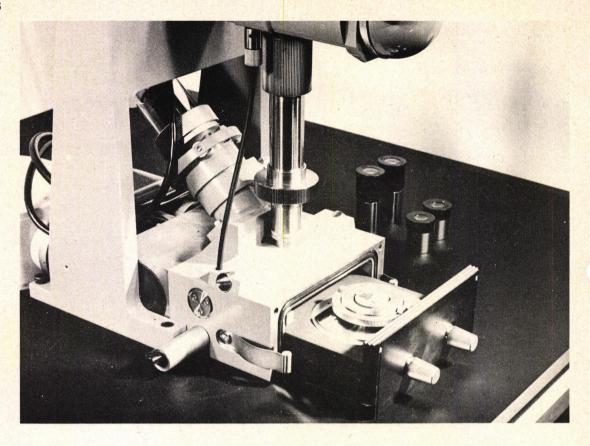
cimen chamber.

Two X-ray windows mounted in opposition, sealed off with beryllium foil, provide an X-ray take-off angle of 15°. If ultra-violet irradiation of the specimen is desired, both X-ray windows are replaced by quartz windows. A yoke is mounted on top of the specimen chamber to support the light column.

#### THE VACUUM SYSTEM (Figure 3)

Combination of the low- and high-vacuum valves into one integrated stainless steel triple gate valve<sup>1)</sup> provides a reliable automated vacuum system. A motordriven piston<sup>2)</sup> moves the valve gates sealed

with O-rings past the various vacuum openings. This mechanically programmed vacuum system reduces the pressure inside the specimen chamber from atmospheric (position 1) to  $10^{-2}$  N/m<sup>2</sup> (about  $10^{-4}$  torr) (position 2) in 35 seconds. The time required to bleed the specimen chamber is 10 seconds. Operation of this vacuum system is safeguarded against sudden vacuum leakages and electric power or coolant flow failure. Two push buttons with pilotlamps control the vacuum system. Most of the vacuum components are standard Edwards products including the baffled oil diffusion pump EO 2 and the mechanical roughing pump ES 35.

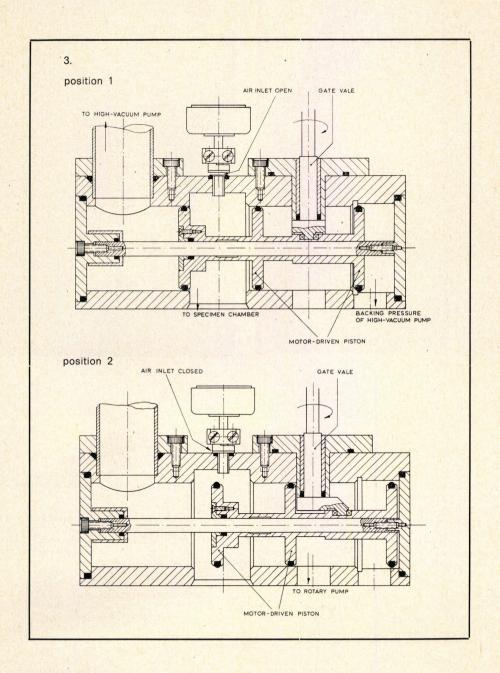


## MISCELLANEOUS

The specimen chamber with both electron and light optics is mounted on a standard desk, while power supplies and accessory electronics are inside the cabinet. The screened oil diffusion pump

and light source for transmission mode are fitted under the desk top.

Figures 1 to 6 illustrate some results obtained with this instrument.







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Fig. 1 - magnification 300x - reflected light - The brown mineral is coarse crystalline cassiterite (SnO2, reflectivity approximately 12%) in a polished section of ore from Ivigtut, Greenland. The black spots are pits. Between crossed polarizers, the internal reflection is noticeable, with its light brown colour.

The anisotropism is clearly observable and not masked by internal reflection. No crystalline texture can be observed.

Fig. 2 - magnification 300x - electron luminescence - This is the same specimen spot as in Fig. 1, under irradiation with electrons. The cassiterite fluoresces green and the crystalline texture is very clearly visible. Under u.v. irradiation no fluorescence was observed.

Fig. 3 - magnification 300x - reflected light - The brown idiomorphic crystals are cassiterite in a polished section of a Bolivian ore. The pale green parts are mineralinclusions. Between crossed polarizers, the internal reflection is

very strong, with a light brown colour. No crystalline texture was observed.

Fig. 4 - magnification 300x - electron luminescence - This is the same specimen spot as in Fig. 3, under irradiation with electrons. The cassiterite fluoresces browngreen and crystalline textures are clearly shown.

Fig. 5 - magnification 300x - reflected light - These are some grains in a polished section of a grain concentrate of scheelite (CaWO<sub>4</sub>). The larger light brown grains are the scheelite and the

smaller slightly blue grains (Fe<sub>3</sub>O<sub>4</sub>).

The grains are mounted in a plastic matrix.

Fig. 6 - magnification 300x - electron luminescence - The same specimen spot as in Fig. 5, under irradiation with electrons. The scheelite grains show the very bright blue-white fluorescing colour much stronger than with u.v. irradiation. The magnetite grains remain dark.





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