MASTERA Simple Scanning Electron Microscope *

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A Simple Scanning Electron Microscope

A. V. Crewe, M. Isaacson, D. Johnson

ABSTRACT

A simple scanning microscope has been built which uses a field emission electron gun alone, without the aid of auxiliary lenses. The design and operation of the microscope are described and the calculated performance is compared with experiment. Resolution of $100\text{\AA}$ has been obtained and is shown in transmission electron micrographs. The probe current is of the order of 10^{-10} to 10^{-11} amps, a value which is high enough to allow micrographs to be taken with scan times of 10 seconds.

A SIMPLE SCANNING ELECTRON MICROSCOPE

(A. V. Crewe, M. Isaacson, D. Johnson)

1. INTRODUCTION

The ultimate resolution of a scanning electron microscope is determined by the smallest diameter of a focused electron beam that can be achieved at the specimen plane.

In a conventional scanning microscope this electron beam is obtained by demagnifying the image of a hot filament. Several magnetic lenses are normally used in order to obtain a small enough probe diameter.

The minimum probe size that can be used is limited by the requirement that there be an adequate current in the electron beam to provide information at a sufficient rate. Scan times cannot be made arbitrarily long because of the variations which occur in lens currents and accelerating voltages. In addition a long scan time would make focusing a tedious procedure. Scan times are usually no longer than five minutes and this places a lower limit on the beam current of the order of 10^{-12} amperes. Using a hot filament, this in turn places a lower limit on the probe diameter of about 100 Angstroms⁽¹⁾.

The scanning microscope described here uses a field emission source instead of a hot filament. Such a source has a very small effective diameter and a high current density^(2, 3, 4, 5), and therefore the use of this type of source places less stringent restrictions on the probe size which can be achieved.

We have already described⁽⁶⁾ a microscope which uses a field emission source, an electron gun and one auxiliary lens. The microscope has a resolving power of better than $20\text{\AA}$.

The properties of this electron gun⁽⁷⁾, however, are such that when used alone it is capable of producing a focused spot of electrons smaller than $100\text{\AA}$ and with a beam current which is high enough (10^{-10} amps) to form an image in 10 seconds. It is therefore possible to construct a very simple scanning microscope which has a resolution comparable to existing commercial instruments but uses no auxiliary lenses.

II. DESCRIPTION OF MICROSCOPE

The field emission electron gun has been described in detail in Ref. (7) and consists of two anodes and the field emission source. A voltage (V_1) between the field emission tip and first anode controls the emission current from the etched tungsten tip, while a voltage of V_0 between tip and 2nd anode determines the final energy of the electrons. (See Fig. 2.) An aperture in the 2nd anode controls the angular spread of the beam. Between the anodes, the lens action of the electrostatic field focuses the electron beam and forms a real image of the field emission tip at the specimen plane. The particular shape of the anodes was chosen to minimize spherical aberration⁽⁸⁾.

The focused spot is scanned across the specimen in a television type raster by means of an electrostatic deflection system. Eight Inconel plates are mounted on a micalex insulator, in such a way as to completely shield the insulator from the beam. (See Fig. 3.) The deflection voltages are placed on four of the plates, while two quadrupole fields,

one rotated by 45° with respect to the other, are superimposed on the deflection fields using all eight plates. The two quadrupoles are excited through a sine-cosine potentiometer which results in a quadrupole field of arbitrary angle and magnitude for the correction of astigmatism⁽⁹⁾.

Information about the specimen is obtained by detecting transmitted electrons using a scintillator and photomultiplier. The photomultiplier signal is amplified and used to modulate the intensity of a synchronously scanned display tube to form an image of the specimen. Contrast is produced by the fact that some electrons are scattered out of an aperture located between the specimen and the scintillator. A magnetic spectrometer is being constructed for energy analysis of the transmitted beam to utilize the energy loss contrast mechanism⁽⁴⁾, and a secondary electron detector is planned.

The magnification is determined by the size of the display tube raster compared to the beam scan raster. The useful range varies from 400x to 400,000x.

To obtain stable field emission, the microscope chamber is kept at $\sim 10^{-9}$ Torr by a 400 l/sec Varian VacIon pump. The accelerating voltage, the field emission voltage, and a current supply for periodic heating and cleaning the tip are provided by a stable (4ppm/hour) 30 kv supply⁽¹⁰⁾.

III. THEORETICAL CALCULATIONS

The size of the probe at the specimen is determined by the first order image of the field emission tip together with the effect of the aberrations of the gun and diffraction. The effective source size itself

leads to an image with a diameter given approximately by⁽⁵⁾

$$d_s = 2mR \left(\frac{\bar{V}_T}{V_1} \right)^{1/2}$$

where R is the actual radius of the tip, for which we assume

$$R(\text{\AA}) = \frac{V_1(\text{volts})}{2} \quad (11)$$

V_1 is the emission voltage needed to obtain 1 μ amp emission current.

$\bar{V}_T$ is the average transverse energy of the electrons leaving the tip ($\sim 0.2V$)⁽¹²⁾, and m is the magnification of the gun.

This shows the reduction in effective source size by an approximate factor

$$\left(\frac{\bar{V}_T}{V_1} \right)^{1/2},$$

which is characteristic of a field emission source.

The aberrations of the gun contribute two terms to the theoretical spot size:

a) Spherical Aberration $d_s = \frac{mC_s \alpha_1^3}{2}$ where

C_s is the spherical aberration coefficient

α_1 is the entrance half angle of the beam

b) Chromatic Aberration $d_c = mC_c \alpha_1 \Delta V$ where

C_c is the chromatic aberration coefficient

and ΔV is the total energy spread of the electrons.

For ΔV , we consider only the total energy spread of the electrons leaving the field emission tip ($0.2V$)⁽¹³⁾. Variations in V_0 and V_1 can be made negligible.

Finally we must include the effect of diffraction at the defining aperture. Although the actual aperture is located in the 2nd anode, we can calculate its effect by assuming the existence of an equivalent aperture in the first anode. The contribution to the final spot size is then

$$d_d = \frac{m \cdot 0.6\lambda_1}{\alpha_1}$$

where λ_1 is the wavelength of the electrons at the first anode.

To estimate the diameter of the focused spot of electrons we combine the four terms in quadrature⁽¹⁴⁾

$$d_{rms} = \sqrt{d_s^2 + d_a^2 + d_d^2 + d_c^2}$$

A set of theoretical parameters is shown in Fig. 4. The various terms are plotted as a function of V_0/V_1 because the first order properties of the gun are entirely determined by this ratio⁽⁷⁾. Each value of V_0/V_1 corresponds to a different source position when the specimen distance is fixed.

Using these parameters, d_{rms} is calculated, and plotted as a function of V_0/V_1 for different fixed specimen distances in Fig. 5, and for different field emission voltages in Fig. 6. The d_{rms} values are for optimum α_1 , calculated by minimizing d_{rms} with respect to α_1 . Due to the high brightness of the field emission source, the further condition of providing enough beam current need not be imposed in calculating α_1 opt as will be seen later.

In earlier work with this gun⁽⁷⁾, the effect of chromatic aberration was not included leading to somewhat smaller theoretical probe sizes than are now predicted. In fact, the d_{rms} for α_1 opt shown in Figs. 5 and 6

consist in many cases mainly of chromatic aberration and diffraction terms, the spherical aberration and source size terms being small.

In general these curves predict that in order to obtain the smallest d_{rms} , the specimen distance and V_o/V_1 should be made as small as possible in order to increase the demagnification, while at the same time, V_1 should be as large as possible to minimize the effect of chromatic aberration and diffraction. Also, enough distance must be left between the gun and the specimen to allow for the space occupied by the scanning system and a possible secondary electron detector. In the present microscope this corresponds to a specimen distance of ≈ 4 cm with $V_o/V_1 \approx 7$ and $V_1 = 3$ kv.

For these operating conditions, we estimate the four contributions to the probe size to be: $d_s = 20\text{\AA}$, $d_a = 27\text{\AA}$, $d_d = 71\text{\AA}$, $d_c = 45\text{\AA}$, with an $\alpha_1 \text{ opt} = 1.54$ mr giving $d_{rms} = 95\text{\AA}$.

To estimate the current in the probe, we assume the source emits uniformly into one steradian and thus

$$I_{\text{probe}} = I_{\text{tip}} \pi \alpha_1^2 \text{ opt}$$

which for $I_{\text{tip}} = 10\mu\text{a}$

and $\alpha_1 \text{ opt} = 1.54$ mr

gives $I_{\text{probe}} = 7.5 \times 10^{-11}$ amps.

IV. OPERATION OF MICROSCOPE

A. Field Emission Tips.

We generally use (310) and (111) oriented tungsten wire for our field emission tips because they produce intense emission along the wire axis⁽¹⁵⁾. All these tips are checked in an auxiliary tip testing system before they are put into the microscope. Using this auxiliary system we can determine the value of V_1 for a given emission current, and

whether or not the intense emission is centered on the axis of the tip holder⁽¹⁶⁾.

Once in the microscope the tips are first cleaned by sending a brief pulse of current through the filament ("flashing") so that the tip reaches about 1900° K (white hot). After that, the tips are only periodically flashed at about 1000° K (red hot). Because the ambient pressure is only about 10^{-9} Torr, this means that we run the microscope with the surface of the tip covered almost uniformly by a monolayer of adsorbed gases and the emission current gradually rises over a period of time until the current becomes erratic⁽¹⁷⁾. When this happens the V_1 supply is turned off, the tip is "flashed" and the microscope is ready for operation again. The running period between flashes is usually of the order of 30-60 minutes depending upon the local pressure in the vicinity of the tip.

Because there are ions constantly bombarding the tip there are short term fluctuations in the probe current (~ 1 -100 ms duration) due to local changes on the tip surface. These fluctuations are 2-5% of the total probe current and generally do not constitute a serious problem in focusing and taking pictures. Moreover, by slightly differentiating the video signal with a simple RC differentiating filter ($RC \sim 1$ -10 ms) one can eliminate almost all effects of these instabilities (such as streaks in the picture) without changing the information content of the picture.

Using field emission tips in the above fashion we have experimentally measured 10^{-10} amps of beam current in a 100Å spot for 10 μ amps of tip current. Note that this gives $I_{\text{beam}}/I_{\text{tip}} = 10^{-5}$ which is slightly larger than the value we previously calculated assuming the emission

current to be distributed uniformly over one steradian. This is not unreasonable since with (310) and (111) oriented tips the emission current tends to be concentrated in the forward direction.

According to the calculations of Oatley et al.⁽¹⁾, a probe current of 10^{-10} amps should be sufficient to record a 600 line picture in ~ 10 sec. using secondary electrons. Using transmitted electrons and assuming only 10% transmission, we expect that a 600 line picture taken in 10 sec. will provide 3% statistics per resolution point in the image.

Tungsten tips seldom, if ever, expire of their own accord. Their life is always terminated by some other malfunction of the microscope such as an accidental voltage surge. At the present we have succeeded in reducing the frequency of these malfunctions so that the average practical tip lifetime is 100 hours (with some tips lasting over 400 hours of operation).

B. Alignment

Alignment consists only of placing the field emission source on the electron optical axis of the gun, since the gun is pre-aligned⁽⁷⁾. This is done by moving the source so that there is no image movement as V_0 is varied. The calculated and experimental alignment tolerance for a $100\text{\AA}$ probe size is $\sim 25\mu$. Movement of the tip is accomplished through a bellows on the top of the microscope.

C. Vacuum

Attaining a pressure in the microscope of $\sim 10^{-9}$ Torr requires about six hours from atmosphere. This includes a heating of the pump and gate valve for three hours. The microscope itself is completely bakable, but this has only been necessary when a considerable amount of new surface

has been installed (and then only to 150° C). A specimen block capable of holding eight specimens at once reduces the inconvenience of the long pump-down and a specimen changer is being designed to reduce the specimen changing time to a few minutes.

V. EXPERIMENTAL RESULTS

According to the calculations of Section III, this microscope should be capable of 100Å resolution. Resolution of 100-175Å has been consistently obtained and is shown in the following transmission electron micrographs. All micrographs were taken using a 10 second scan time with beam currents of 10^{-11} to 10^{-10} amps. The accelerating voltage was ~20 kv.

In Fig. 7 are micrographs of a thin (~1000Å) Al film evaporated onto a NaCl substrate at a pressure of 10^{-7} Torr. The substrate was then dissolved away in distilled water. Fig. 7a uses the normal transmitted electron signal while in Fig. 7b the signal from the same specimen area is highly differentiated (with RC $\approx$.1ms) to accentuate the changes in the signal.

Fig. 8 contains two micrographs of the same area of a thin gold film, taken several minutes apart. The gold, approximately 170Å thick, was evaporated onto a carbon film. The markers on the micrographs denote 100Å.

Fig. 9 is a micrograph of a thin section specimen, Helix Aspersa (snail sperm tails), embedded in Epon and sectioned to about 500Å thickness.

Fig. 10 is a micrograph of tobacco mosaic virus on a thin carbon film. The virus was positively stained with uranyl acetate and was sprayed onto the carbon using a 2.5 mg/ml solution.

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FIGURE CAPTIONS

- Fig. 1 The gun microscope. The field emission tip sits approximately at the level of the top row of ports and the specimen sits slightly above the level of the bottom row of ports. Two micrometer motions on the lower ports are used to move the specimen. The photomultiplier-scintillator combination used to detect the transmitted electrons is below the bottom flange (not visible in this picture). It is similar to the device used for detecting secondaries which is seen coming out of a side port in the lower left of the picture.
- Fig. 2 A schematic diagram of the microscope. The voltage (V_1) between the tip and first anode controls the emission current from the etched tungsten tip, while the voltage V_0 between tip and second anode determines the final energy of the electrons. Between the anodes, the lens action of the electrostatic field focuses the electron beam and forms a real image of the field emission tip at the specimen plane.
- Fig. 3 Diagram of the electrostatic stigmator and deflection system. The inconel plates are mounted on a micalex insulator so that the insulator is completely shielded from the beam. The deflection voltages are placed on

four of the plates, while two quadrupole fields, one rotated by 45° with respect to the other, are superimposed on the deflection fields using all eight plates. The two quadrupoles are excited through a sine-cosine potentiometer which results in a quadrupole field of arbitrary magnitude and angle for the correction of astigmatism.

- Fig. 4 Theoretical optical properties of the electron gun. The four curves shown are for the geometrical magnification (m), the source distance (S_1), the spherical aberration constant (C_s) and the chromatic aberration constant times the field emission voltage ($C_c \cdot V_1$). (C_s and C_c are referred back to the source.)
- Fig. 5 Graph of the dependence of the optimum probe diameter (d_{rms}) upon the ratio V_o/V_1 for various fixed specimen distances.
- Fig. 6 Graph of the dependence of the optimum probe diameter (d_{rms}) upon the ratio V_o/V_1 for various values of the field emission voltage V_1 . Each value of V_1 is that voltage required to obtain 1 μ a emission current. The specimen distance is fixed so that each value of V_o/V_1 corresponds to a different source position.

- Fig. 7 Micrographs of a thin ($\sim 1000\text{\AA}$) evaporated AL specimen. Fig. 7a is taken using the normal transmitted signal. The same specimen area is shown in Fig. 7b, but using a highly differentiated signal.
- Fig. 8 Micrographs of Au ($\sim 170\text{\AA}$ thick) evaporated onto a thin carbon film. Figs. 8a and 8b show the same area on the specimen taken several minutes apart. The markers denote $100\text{\AA}$ spacings.
- Fig. 9 Typical micrograph of sectioned Helix Aspersa (snail sperm tails).
- Fig. 10 Micrograph of tobacco mosaic virus on a thin carbon substrate.

Top

A.V. CREWF.
FIG. 1

REORDER PRINTS
BY NUMBER
NUMBER 68-905, X

GUN MICROSCOPE

A.V. CREWE
FIG. 2

68-481,3

A.V. CREWE
FIG. 3

68-905, 6

Top

CREWE
FIG 4

68-905, 2

d_{RMS} vs V_0/V_1 ($V_1 = 3\text{KV}$)

TOP

CREWE
FIG 5

68-905,4

d_{RMS} vs V_0/V_1

TOP

CREWE
FIG 6

68-905,1

TOP

CREWE
FIGURE 7A

68-926,4

TOP
↑

CREWE
FIGURE 7B

68-926,3

Top

CREWE
FIG 8a

68-926,7

Top

CREW
FIG 8b

68-926, 8

TOP
↑

CREWE

FIGURE 9

68-926,2

TOP
↑

CREWE
FIGURE TO

68-926,1