SCANNING ELECTRON MICROSCOPY/1972 (Part I)
Proceedings of the Fifth Annual
Scanning Electron Microscope Symposium
IIT Research Institute
Chicago, Illinois 60616, U.S.A.
April, 1972

EXPERIMENTAL RESOLUTION LIMIT IN THE SECONDARY ELECTRON MODE FOR A FIELD EMISSION SOURCE SCANNING ELECTRON MICROSCOPE

T. Komoda
NAKA-Laboratory
Central Research Laboratories
Hitachi Limited
882-Ichige, Katsuta, Ibaraki, Japan

S. Saito
Naka Works
Hitachi Limited
882-Ichige, Katsuta, Ibaraki, Japan

ABSTRACT

A prototype surface scanning electron microscope has been built into which a field emission electron source has been incorporated. Although the field emission electron gun requires ultra high vacuum, such as 10^{-10} Torr, to obtain stable emission current for a reasonable period of time, its greatly increased brightness brings about high resolution in the scanning electron microscope operated in the secondary electron imaging mode.

The gun electrodes have been designed so as to minimize the aberrations and to maintain the brightness of the source through the electron acceleration field. The gun is followed by two magnetic lenses, and the electron beam is focused on the specimen which is 40mm away from the objective lens center. The spot diameter on the specimen can be as small as 30Å.

The electron optical column is evacuated with two ion-pumps differentially down to the pressure of 10^{-10} Torr in the gun chamber and 10^{-8} Torr in the specimen chamber. The clean vacuum system prevents specimen contamination which is one of the most serious problems in high resolution electron microscopy.

Using this microscope, small gold particles in the range of 30-40Å in size have been observed in the secondary electron mode. The probe current has been about 10^{-10}A which is bright enough to obtain a picture to be taken in 20 sec of exposure time. The brightness of the electron beam system has been measured to be as high as 7×10^7A/cm^2 sr at 20kV, which is two orders of magnitude higher than the brightness of the ordinary thermionic electron gun.

EXPERIMENTAL RESOLUTION LIMIT IN THE SECONDARY ELECTRON MODE FOR A FIELD EMISSION SOURCE SCANNING ELECTRON MICROSCOPE

Tsutomu Komoda
Naka-Laboratory,
Central Research Laboratories, Hitachi Ltd.
882-Ichige, Katsuta, Ibaraki
JAPAN

Shyobu Saito
Naka-Works, Hitachi Ltd.
882-Ichige, Katsuta, Ibaraki
JAPAN

Introduction

The resolution of a scanning electron microscope is determined by the diameter of the electron beam at the specimen. This electron beam can be obtained by demagnifying the image of an electron source using several magnetic lenses. The smallest beam size, however, is limited by the requirement to provide sufficient beam current to form an image of the specimen in a reasonable scan time. Therefore, the ultimate resolution of a scanning electron microscope depends closely on the brightness (A/cm^2 sr) of the electron source used in the microscope. When the ordinary hot filament cathode is used as an electron source, the maximum brightness available is of the order of $10^5 A/cm^2$ sr at 20kV, and the resolution is limited to above 100Å.

Various attempts have been made to produce an electron source of higher brightness than a hot filament cathode(1) and the most successful one is a field emission source developed by Crewe(2). Crewe has built a transmission type scanning electron microscope using a field emission electron gun(3) and has succeeded in observing single atom images with his microscope(4). According to his report(5), the field emission source can produce an electron probe smaller than 5Å in size and with a beam current high enough to form an image in 10sec. This indicates that the field emission gun has a brightness a few orders of magnitude higher than of the ordinary hot filament cathode gun.

There is no reason that the field emission gun cannot be used in the surface scanning electron microscope in which the image is formed by secondary electrons emerging from the specimen surface. However, the ultimate resolution in the secondary electron image is limited not only by the size of the electron beam striking the specimen surface but by some diffused distribution of the secondary electrons emerging from the surface. The distribution is caused by the high energy electrons reflected backwards from the inside of the specimen and by the low energy secondaries diffused in the specimen in a range close to the surface. Unfortunately, there has been insufficient information available about the distribution to make an estimate of ultimate secondary electron image resolution.

The authors have built a prototype surface scanning electron microscope incorporating a field emission electron gun. The intent of this paper is to describe the construction of the microscope which we have built, and to show some application pictures. These are presented as experimental data showing attainable resolution in the secondary electron imaging mode.

Instrumentation

Design of a field emission gun

The field emission gun consists of a field emission source (tip) and two anode electrodes. The voltage (V_1) applied between the tip and the first anode defines the emission current from the tip and the voltage (V_0) defines the electron energy. While a field emission source brightness as high as $1\times10^9 A/cm^2$ sr for 20kV electrons has been measured(6), it is necessary in the gun used here to allow for aberrations in the space between the first and second anodes. In order to minimize these aberrations it is essential to use a Butler type anode system(7).

We have computed the shape of the anode electrodes for minimum aberrations using a modification of Butler's method. We have assumed an axial potential distribution in the anode field containing one parameter "a" as follows,

$$\phi(z)=(\tfrac{3}{2}+a)z-(2a+\tfrac{1}{2})z^3+az^5 \qquad (1)$$

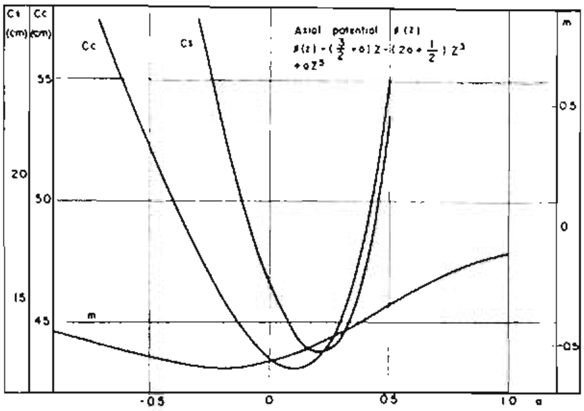


Fig.1 Electron optical properties of the electro-static field ϕ (z).

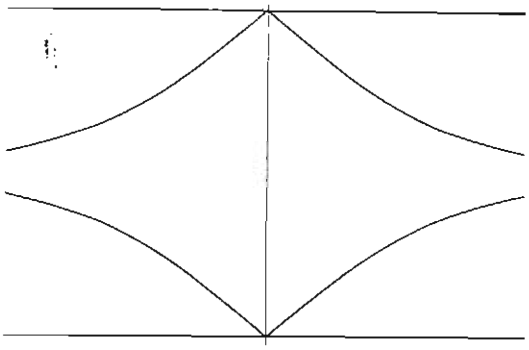


Fig.2 An anode shape designed so as to minimize the spherical aberration.

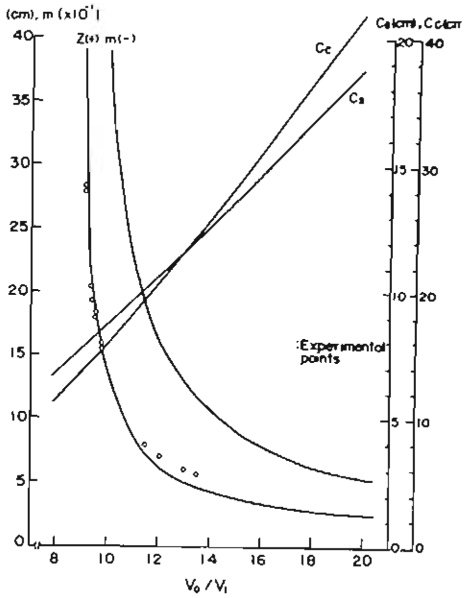


Fig.3 Electron optical properties of the field emission gun designed as shown in Fig.2.

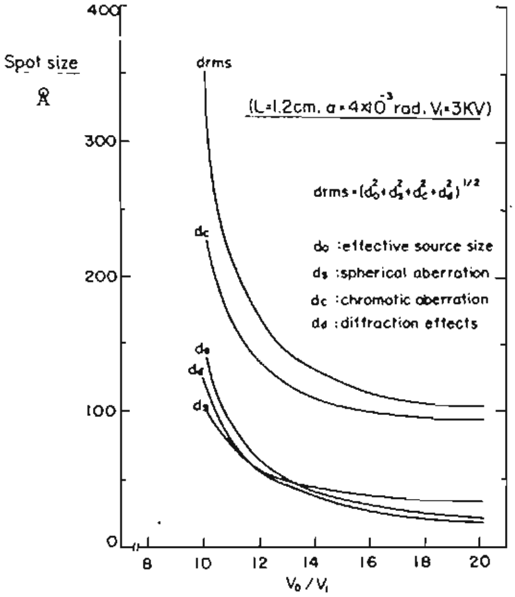


Fig.4 Spot diameter of the electron beam focused by the field emission gun.

Lens properties such as the spherical aberration, C_s, chromatic aberration, C_c, and magnification, m, are computed as a function of "a" by using a hybrid computer. As shown in Fig.1, the spherical aberration coefficient becomes minimum at the optimum value of "a" where the aberration is 30% smaller than Butlers. An anode has been designed based on the optimum axial potential distribution. The shape is shown in Fig.2, which is somewhat different from Butler's design. The degradation of brightness in the anode space is minimized and is 1×10^8A/cm^2 sr at 20kV.

Optical properties of the field emission gun which we designed have been calculated as a function of the voltage ratio V_0/V_1. An example is shown in Fig.3 in which the distance between the tip and the first anode is 12mm. Z is the distance between the focal point and the top surface of the first anode. The experimentally measured values of Z agree well with calculated ones. The diameter of the focused beam is also calculated as shown in Fig.4, where the effective source size, spherical aberration, chromatic aberration, and diffraction effects are considered. A spot diameter around 100Å can be expected without the use of a demagnifying lens.

Construction of a SEM system

In Fig.5 is shown a schematic diagram

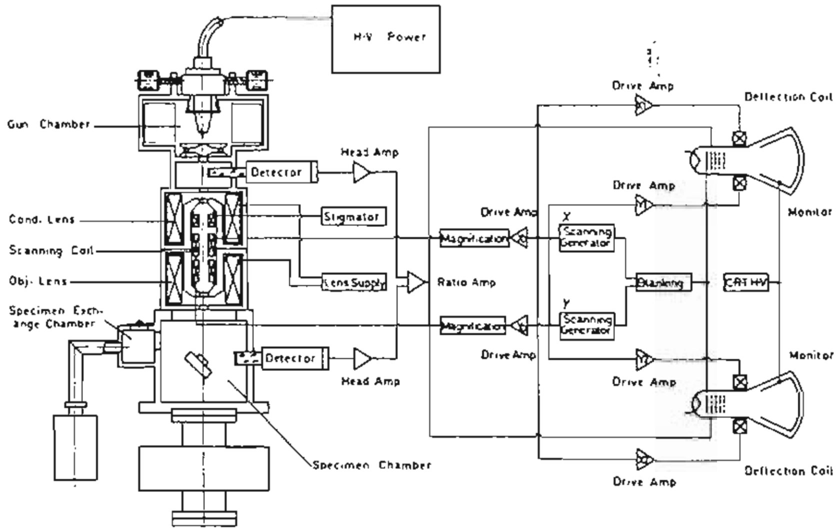


Fig.5

Schematic diagram of the scanning electron microscope operating in secondary electron imaging mode.

of the prototype scanning electron microscope which we have built.

Gun. Emission current fluctuation is one of the most serious problems in field emission guns. It depends on the vacuum condition around the tip, and on the total emission current emerging from the tip. In order to provide a good vacuum environment, the gun assembly is located in the center of an ion-pump system which is symmetrically laid out relative to the electron optical axis. Tips are mounted on a turret holder and they are exchangeable quickly from the outside of the vacuum system without disturbing the vacuum in the gun chamber. A stable emission current in the order of 10μA is obtainable at the normal operating vacuum, which is better than $5x10^{-10}$Torr.

Lens column. The field emission gun is followed by two magnetic lenses. Only one demagnifying lens is necessary to form an electron probe smaller than 10Å in diameter. A second magnetic lens is provided between the electron gun and the objective lens in order to provide desirable flexibility. The spot size as well as the intensity of the electron probe can be controlled easily by means of the additional lens. The focal length of the objective lens is about 40mm, allowing a large working distance for convenient specimen handling. Specimens are exchangeable from outside the instrument in less than ten minutes.

As shown in Fig.5, the upper detector is used as a beam fluctuation monitor. The image forming signal is picked up by the lower detector and divided by the beam monitor signal in order to smooth out the noise in the picture caused by fluctuations of the field emission current. At present, however, the ratio-amplification system is unnecessary because the field emission is sufficiently stable. The pictures here were all taken without the tip noise compensation system.

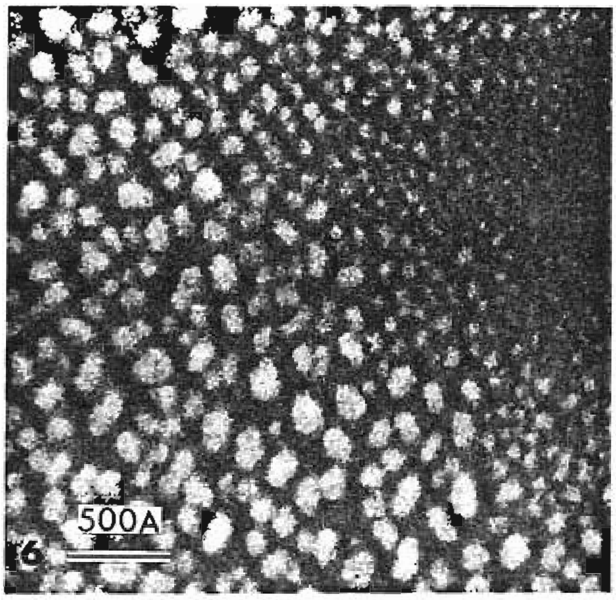
Evacuation system. The microscope column is divided into two sections which are differentially pumped with two separate ion-pumps. The pressure in the lens section and the specimen chamber is in the order of 10^{-8}Torr, while the pressure in the gun chamber is in the low 10^{-10}Torr range. Specimen contamination is one of the most serious problems in conventional high resolution scanning electron microscopes. The oil-free evacuation system used in the microscope described here, however, completely removes this problem. It is possible to observe the same area of a specimen for several hours at a magnification as high as 100,000 times.

Experimental

Resolution

The ultimate resolution of the prototype surface scanning electron microscope which we have built is mainly limited by the spherical aberration of the objective lens and diffraction, and is given by the well known formula

$$d = 0.6 \cdot C_s^{1/4} \cdot \lambda^{3/4} \qquad (2)$$



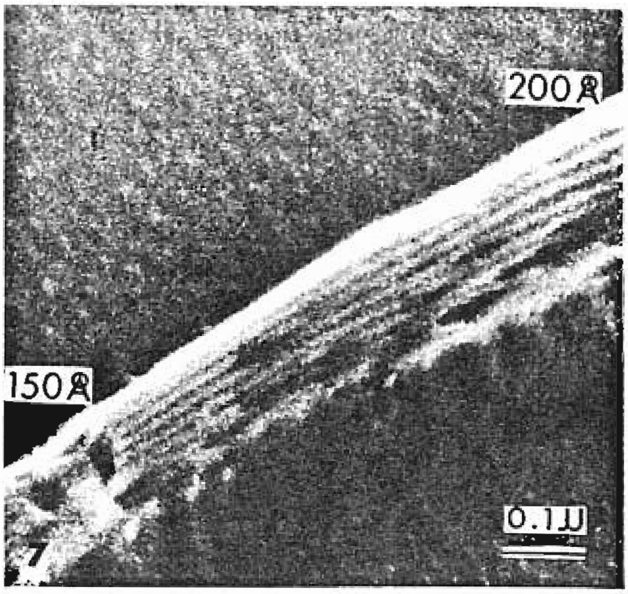


Fig.6 Secondary electron image of gold particles deposited on a cleaved mica surface which is coated by a thin carbon layer beforehand. Accelerating voltage(V_0) 20kV, total emission current(I_e) 5μA, probe current(I_s) 2.5×10^{-10}A, exposure time(t) 20sec.

Fig.7 150Å spacing GaAs-GaAsP "superlattice". V_0=20kV, $I_s=1\times10^{-10}$A, t=20sec.

Substituting C_s=550mm (focal length, 40mm), λ=0.087Å (20kV), the resolution is given as d=26Å. Considering the size of the gaussian image of the source and the effect of chromatic aberration, the spot diameter in the range of 30-40Å can be expected in practice.

In Fig.6 is shown an example of a high resolution picture taken by the secondary electron mode with the microscope. The specimen in the picture was prepared by means of a shadowing technique which provides a range of small gold particles suitable for resolution evaluation. The substrate used is a cleaved mica surface which is covered by a thin carbon layer in order to reduce charging effects. Deposited gold particles smaller than 40Å in size can be seen in the picture. This confirms that the beam diameter is really in the range that theory predicts. The picture also shows that for this specimen the resolution does not deteriorate significantly as a result of secondary electron diffusion.

In Fig.7 is shown a portion of "superlattice" structure in a gallium arsenide crystal (8) which was supplied from IBM. Spacing of the layers is stated as 150Å, but the spacing is somewhat different from place to place as shown here. Such images were seen even on a T.V. display. A smaller spacing than 100Å of "superlattice" is required for the resolution test of this microscope.

Brightness

The brightness of an electron beam system can be given roughly by the following formula,

$$B = \frac{4\ I_s}{\pi^2 \cdot d_s^2 \cdot \alpha^2} \qquad (3)$$

where I_s is the electron probe current striking the specimen, d_s the probe diameter and α the angular aperture of the objective lens. Substituting the operating conditions in which the picture shown in Fig.6 was taken, that is $I_s=2.5\times10^{-10}$A(total emission current, 5μA), d_s=40Å (worst case) and $\alpha=3\times10^{-3}$ rad, the brightness is given as $B=7\times10^7$A/cm^2/sr at 20kV. Even the most pessimistic estimates of field emission gun brightness indicate an order of magnitude higher brightness than that of the LaB_6 rod Shottky emission gun (1) and two or more orders of magnitude higher than that of the ordinary thermionic electron gun. There is a theoretical limit to the brightness of the thermionic electron gun dependent upon the cathode temperature but no such limitation is imposed in the field emission gun. The brightness increases almost in proportion to the total emission current emerging from the tip cathode. Therefore, much higher brightness can be expected in the field emission gun. The gun can provide a 100Å electron probe with a beam current of the order of magnitude of 10^{-9}A, which is adequate for the fast scanning display such as the T.V. scan.

Other applications

In Fig.8 and 9 are shown some examples of secondary electron micrographs taken with the field emission source scanning electron microscope. Both biological and non-biological specimens are shown. The beam intensity is so high that specimen charging is a problem. The use of a thicker coating on the specimen surface will smooth out fine details. Special specimen preparation techniques must be investigated in order to optimise image detail and prevent image charge up.

Conclusion

The high brightness of the field emission electron gun brings about high resolution in the scanning electron microscope operated in the secondary electron mode. The deterioration of resolution caused by the diffusion of secondary electrons in the specimen material is not significant for a specimen such as shown in Fig.6. It is quite obvious that the high brightness provides improved signal to noise ratio in the microscope image and shorter exposure time to form an image. Special specimen preparation techniques must be studied for optimising high resolution scanning electron microscopy.

References

(1) A.N.Broers, Scanning Electron Microscopy/1970, IITRI, Chicago (1970) 3

(2) A.V.Crewe, J.Wall, L.M.Welter, Rev. Sci. Inst., 39 (1968) 576

(3) A.V.Crewe, J.Wall, L.M.Welter, J.Appl. Phys., 39 (1968) 5861

(4) A.V.Crewe, J.Wall, J.Langmore, Microscopie Electronique/1970, Grenoble, 1 (1970) 467

(5) A.V.Crewe, J.Wall, Proc. 27th Ann. Meeting EMSA/1969, Minnesota (1969) 48

(6) A.Tonomura, T.Komoda, Microscopie Electronique/1970, Grenoble, 1 (1970) 467

(7) J.W.Butler, Proc. 6th Int. Cong. Electron Microscopy/1966, Kyoto, 1 (1966) 191

(8) A.N.Broers, Microscopie Electronique/1970, Grenoble, 1 (1970) 239.

DISCUSSION WITH REVIEWERS

Reviewer I: How did you obtain equation (1)?

Authors: We assume an axial potential distribution as follows:

$$\phi(Z) = \sum_{n=o}^{5} a_n z^n$$

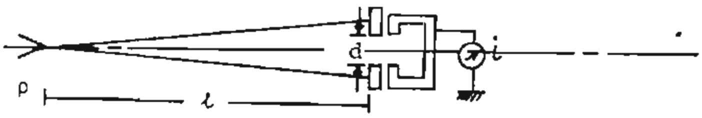
By substituting the boundary conditions (ϕ (±1) = ±1, ϕ(0) = 0, ϕ'(±1) = 0), equation (1) is obtained. z = ±1 corresponds to the 1st and 2nd anode positions.

Reviewer I: What do the signs for Z(+) and m(-) in Fig. 3 signify?

Authors: The signs in brackets indicate whether the source image is real or virtual. Z(+) shows focal point distance of a real image and m(-) shows the magnification.

Reviewer II: Can you give the experimental details for measuring brightness of the field-emission source?

Authors: Using the following experimental arrangement



beam current (i) which goes through the aperture (d) was measured. The brightness was estimated by

$$B = \frac{i \cdot \ell^2}{(\pi/4)^2 - d^2 \cdot \rho^2}$$

ρ is a virtual source size which was estimated by determining the tip radius with an electron microscope. The brightness of the source ranged from 8 x 10^7 to 2 x 10^{10} A/cm^2.sr at 20 KV, and the average brightness at a tip current of 5 μA was 1 x 10^9 A/cm^2.sr.

Discussion continued - p. 136

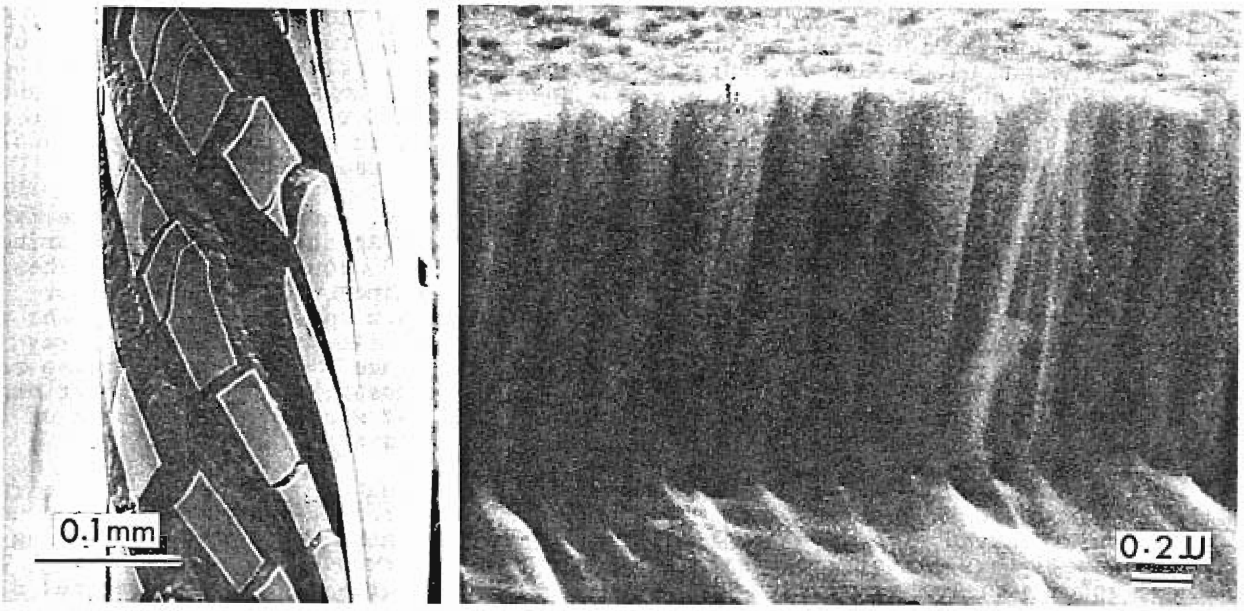


Fig.8 Cross section of oxalic acid alumite, showing a tubular structure in an oxide layer. Folded alumite sheet (left).
V_0=20kV, I_s=1x10^{-10}A, t=20 sec.

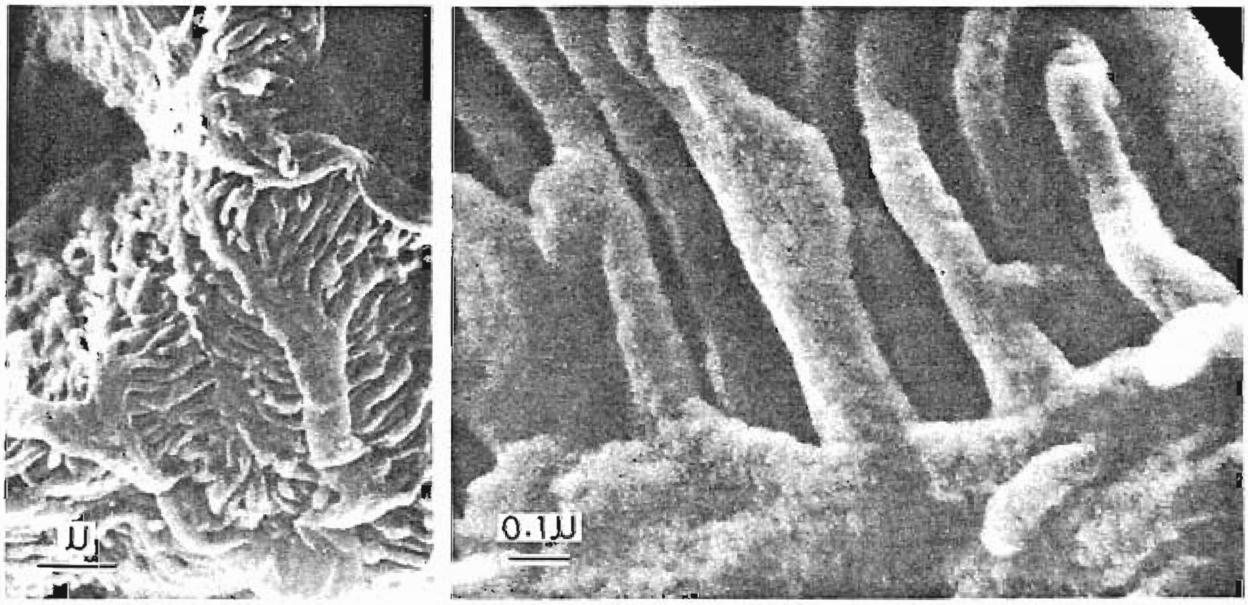


Fig.9 A portion of cat kidney. By the courtesy of Prof. T.Fujita, Niigata University.
V_0=20kV, I_s=1x10^{-10}A, t=20sec.

DISCUSSION WITH REVIEWERS - continued

Reviewer II: What is the electrode spacing at the insulator isolating the two halves of the gun electrode structure shown in Fig. 2, and have any difficulties with breakdown been experienced?

Authors: Electrode spacing at the insulator was 18mm. 30KV was applied on these electrodes without any difficulty of insulation breakdown. The breakdown problems are apparently eliminated by the ultra high vacuum.

Reviewer III: You state that in Fig. 6 particles as small as 40 Å can be seen. This does not give an accurate estimate of the actual resolution of the instrument. What is the smallest point-to-point resolution you have measured?

Authors: Two gold particles spaced at 50-60 Å were resolved. The gold particle size is usually small inside the shadow but the spacing of the particles usable for point-to-point resolution test is not smaller than 50-60 Å. For the measurement of ultimate resolution, it is necessary to prepare a specimen in which the particle separation will be smaller than 50 Å (such as Pt - Pd evaporated particles).

Reviewer IV: The vacuum system of your instrument appears to be quite ingenious. What parts of the system, if any, are baked and what materials are used for the scintillator and light-pipe for the detectors?

Authors: Only the outer shell of the ion pump of the gun chamber is baked out. Other parts of the electron optical column do not require baking. This permits the use of an ordinary plastic scintillator and glass light-pipe.

Reviewer IV: The small beam size attained in this instrument is of obvious advantage for studying specimens of the special type shown in Fig. 6. Do you anticipate that it will have any important advantage for the study of the more usual type of specimen?

Authors: Yes, because firstly, the fine structure of specimen surface measuring less than 100 Å could be observed. The long working distance is advantageous for permitting studies on large specimens with freedoms of tilt, rotation, and X-ray analysis. Since high signal-to-noise ratio images are possible with very short exposure time, TV scanning with probes of less than 100 Å is a real possibility.

Reviewers I and IV: Welter and McKee (p. 161, these proceedings) describe the use of fast scanning and lower accelerating voltages to reduce specimen charging. What are your experiences in this regard?

Authors: We do not have enough experimental data on fast scanning. Following are the general advantages at low voltage operation in our SEM, but we have no results on effect of low accelerating voltage on specimen charging at present.

a. The field emission voltage (V_1) is kept constant while the accelerating voltage changes so that the brightness does not deteriorate significantly at low voltage operation. It is possible to reduce accelerating voltage down to a few hundred volts.

b. Apertures and other parts of the column are free from contamination due to the ultra-high vacuum system and they have less charging effects such as anomalous astigmatism.