Development of Field Emission Type High Resolution Scanning Electron Microscope (HFS-2)

Shinjiro Katagiri D. Eng., Assistant Chief Engineer, Naka Works, Hitachi, Ltd.
Tsutomu Komoda D. Eng., Senior Research Staff, Central Research Laboratory, Hitachi, Ltd.
Kazumi Shiraishi Senior Engineer, Naka Works, Hitachi, Ltd.
Shobu Saito Engineer, Naka Works, Hitachi, Ltd.

1. Introduction

As is well know, the scanning electron microscope was developed principally for the purpose of observing the surface structure of specimens. The principle of the scanning electron microscope is that the specimen is scanned by an electron probe, and the signal of secondary electrons emitted from the specimen is amplified and displayed on a cathode ray tube in the form of a two-dimensional image. While the instrument was introduced onto the market less than only ten years ago, its field of application has been markedly expanded, not only to the observation of specimens for metallurgy, materials engineering, biology, medicine and other disciplines, but it is also used in combination with the X-ray Microanalyser for qualitative and quantitative analyses of specific substances.

In the ordinary scanning electron microscope, a thermionic electron gun with a hairpin-like heated tungsten filament is used as the electron source. Since the size and brightness of the source is determined by the diameter and the temperature of the filament, the resolving power of such a scanning electron microscope is limited to 100~70Å.

The field emission electron source has been developed as a source with greater brightness. This source was first applied to the scanning electron microscope by Professor Crewe of Chicago University. The term "field emission (F.E.)" refers to the phenomenon that a sharp-pointed metal tip such as of tungsten, under application of a high negative voltage emits a high density electron beam even without being heated, an observation which was first made many years ago.

Though the field emission electron source has a number of outstanding merits when used for the scanning electron microscope, its practical application requires the maintenance of stable emission and solution of many technical difficulties, such as manufacturing of tips, study of its characteristics, method of tip flashing, design of anode shape with minimum spherical aberration, elimination of FE noise, and specimen handling and exchange within the microscope column under ultra-high vacuum. In the present instrument, Model HFS-2, all these problems have been solved, and a number of units have already been put to practical use. Its outer view is shown in Photo 1, and main characteristics are presented below.

(1)	Resolving power:	
	Secondary electron imaging mode,	30Å.
	Transmitting electron imaging mode,	15Å.
(2)	Accelerating voltage:	1~25kV.
(3)	Magnification:	20~250,000x.
(4)	Vacuum:	
	Electron gun chamber,	5×10^{-10} Torr.
	Specimen chamber,	5×10^{-7} Torr.

The structure and characteristics of the electron gun adopted in this instrument, as well as the construction of the instrument itself, will be described in the following.

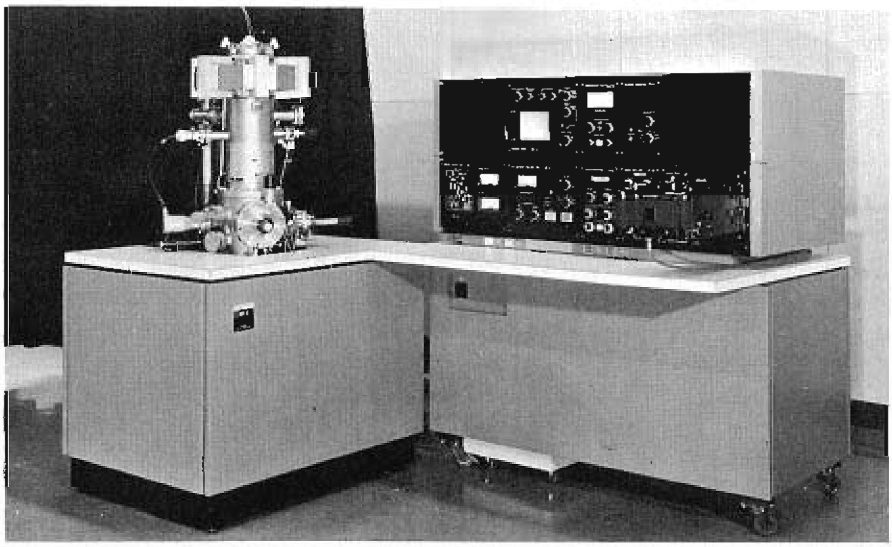

Photo 1. An Outer View of the Model HFS-2 Field Emission High Resolution Scanning Electron Microscope

(the microscope column at the left and the display unit with power supply unit at the right)

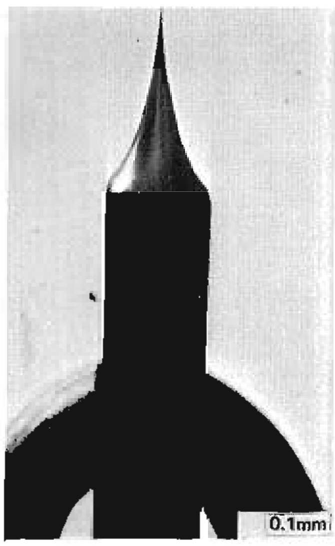

Photo 2. A Photomicrograph of the Field Emission Tip

(tungsten rod electrolytically polished to a radius of curvature of approximately 1,000Å)

2. Field Emission Electron Gun

A tungsten rod is electrolytically polished to form a sharp tip. If the curvature of the tip point is given by R and the voltage between the tip and the anode by V_1, the strength of the electric field E at the tip point is given by

$$E \simeq \frac{V_1}{\delta R}$$

Photo 2 shows a magnified view of an actual tip with R = 1,000Å. This tip gives field emission if V_1 = 2.5kV is applied.

Photo 3 shows a field-emission pattern (FEM pattern) of a tungsten tip. The characteristic electron density pattern depends on the work function of the crystal surface at the tip point. In practice, the crystal surface is selected so that the brightest point of the FEM pattern lies on the optical axis (center of the picture). For this reason, in the case of the tungsten tip, a single crystal with a (310) or (111) plane vector coinciding with the optical axis is employed.

A comparison of the field emission electron source with the conventional thermionic electron source is given below.

(1) Electron Source Size

The field emission source is characterized by a smaller emitting area. Its radius is 10Å or so, much smaller than that of the hair-pin type thermionic source, which is 15~20μ.

(2) Brightness

While the brightness of the thermionic source is limited by the maximum temperature (~3,000°K), that of the FE source increases with the applied voltage, reaching a very high level, approximately 5 x 10^9 A/(cm^2·Sr) (as converted to the brightness at 100kV, the same applying to subsequent similar data), which is 3 orders of magnitude higher than the brightness of the thermionic source, 1 x 10^6 A/(cm^2·Sr).

(3) Energy Distribution

Since the tip temperature is low (the tip is not being heated), an electron beam of uniform energy is obtained. According to measurement, the energy width is about 0.2V, which is smaller than that of the thermionic source, 0.6~2V. This allows the obtaining of a highly coherent electron source.

(4) Life

In principle, the life of an FE source is permanent. However, owing to the flashing operation to be described later, the tip gradually becomes more obtuse, resulting in an increase in the anode voltage for emission. The practical life, as defined to be the time span for the emitting anode voltage to be increased to a prescribed value (5kV, in this case), is 1/2~1 year, which is much longer than the life of a thermionic filament, 50~100 hours.

Fig. 1 shows a schematic diagram of an FE electron gun. The anode voltage (V_1) is applied between the first anode and the tip, and the accelerating voltage (V_0=1~25kV) between the second anode and the tip. The FE electrons emitted from the tip pass through the center bore of the first anode, and are focused as well as accelerated by the second anode, so as to form a fine spot (about 100Å in

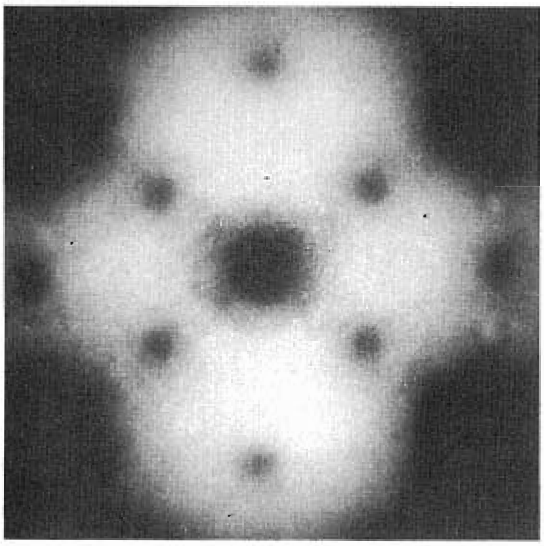

Photo 3. Field-emission Pattern (FEM pattern) Provided by a Tungsten Tip

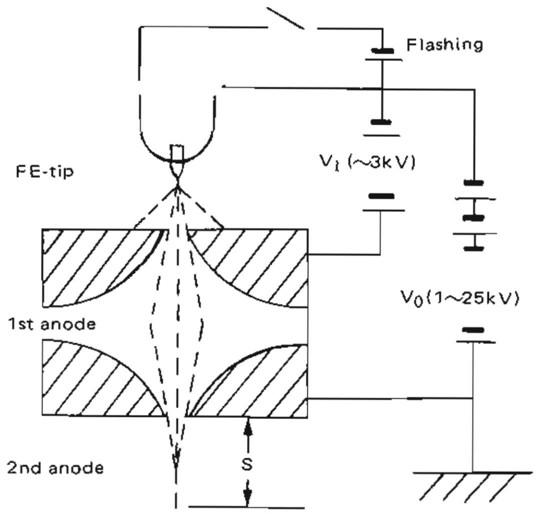

Fig. 1 Schematic Diagram of a Field Emission Electron Gun

(The gun consists of a tip and two anodes, which constitute an electron lens and have a special form so as to minimize spherical aberration.)

diameter) at a distance S below the latter. In order to minimize spherical aberration of the anode lens, an electrode of special form *Butler type) is adopted, as illustrated in Fig. 4.

3. Construction of HFS-2

The HFS-2 consists of the microscope column and the display. Fig. 2 shows an overall construction diagram.

The microscope column with evaluation systems consists of the FE gun chamber, the intermediate chamber, the lens system and specimen chamber, as illustrated. The column is divided into three sections, each of which is separated from the other by a pin-hole diaphragm and independently evacuated by a respective ion pump. The ultimate vacuum of the gun chamber is 10^{-10} Torr, that of the intermediate chamber 10^{-8} Torr and that of the specimen chamber 10^{-7} Torr. Even when the pressure in the specimen chamber rises to 10^{-5} Torr, the high vacuum of the gun chamber is not affected, owing to this three-stage differential evacuation system.

3.1 Electron Gun Chamber

From the point of view of stabilizing the FE current, the ultra high vacuum of 10^{-10} Torr is required, for preventing the tip surface from being contaminated with absorbed residual gas which could cause the apparent work function and field strength of the tip to change and the FE current to fluctuate. Generally, if the FE current is drawn from a tip for a long time, the current starts fluctuating markedly, particularly at a higher current level, If the operation is conducted without flashing, a large current flows, causing a discharge and destroying the tip. In order to prevent this, it is necessary to clean the tip of absorbed gas by heating it with a flashing current. The better the vacuum, the longer the stabilization period between cleanings, and the less the current fluctuates.

In order to obtain a high evacuating efficiency of the ion pump, the gun chamber is directly coupled with the pump. For this reason, evacuation of the gun chamber from atmospheric pressure to 10^{-10} Torr, including the baking treatment of the chamber wall takes only 12 hours. The gun chamber is provided with two tips, which can be exchanged without breaking the vacuum. In consideration of the tip life mentioned above, it is not required to open the gun chamber for 1 year or longer, eliminating the need for troublesome alignment and assuring easy maintenance.

3.2 Intermediate Chamber

The intermediate chamber is located between the gun chamber and the lens system, functioning as a means for differential evacuation. It is provided with an independent ion pump, which is not shown in Fig. 2. In addition, the intermediate chamber includes an air lock device for the gun chamber, a beam current monitoring device for detecting fluctuations in FE current and an aperture plate with ex-

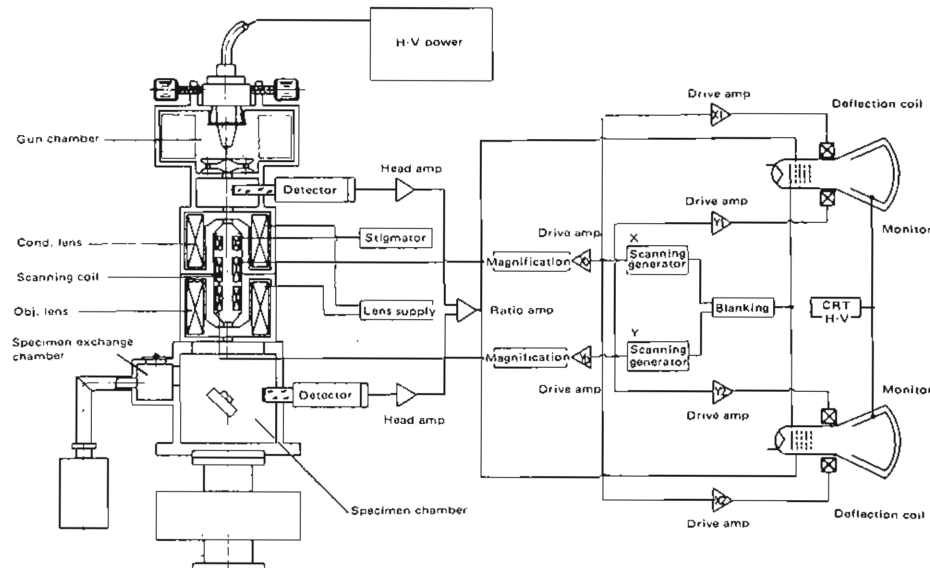

Fig. 2. Overall Construction Diagram of HFS-2 System
(The construction of the column and power supply is shown. The electrons emitted from the gun are fine-focused by the electromagnetic lens and are deflected by the deflecting coil so as to scan over the specimen.)

changeable apertures. Owing to the clean vacuum environment, the aperture is not contaminated with hydrocarbons after use over 1 year or longer without cleaning.

3.3. Lens System

The lens system is a 2-stage demagnifying lens system. Since the FE electron gun alone provides an electron probe with a diameter as small as 100Å, adding a demagnifying lens to it easily assures high resolution. Moreover, as the brightness is sufficiently high, a large beam current is obtained with a reduced probe diameter. Denoting the brightness of the electron source as R, the probe radius as γ and the illuminating angle as β, the beam current I_S is given by

$$I_S = \pi^2 \gamma^2 \beta^2 R$$

If $R = 1 \times 10^8$ A/(cm^2·Sr), $\gamma = 2$Å and $\beta = 1 \times 10^{-2}$ rad, the resultant beam current is $I_S = 4 \times 10^{-11}$ A, which is large enough for the probe current of the scanning electron microscope. That is, if an FE electron gun and a demagnifying electromagnetic lens are provided, it is essentially possible to obtain a scanning electron microscope with a resolving power of a few Å. However, in order to realize such a performance, it is necessary to operate the instrument with the curvature of the tip point always kept constant, that is, holding the anode voltage (V_1) at a prescribed constant level. This is rather difficult in practice.

In the HFS-2 system, therefore, a condenser lens is inserted between the electron gun and the objective lens, so as to make the probe current widely variable. With this provision, it becomes easy to realize either a 20~30Å probe diameter and a $1 \sim 5 \times 10^{-11}$ A probe current for observation with high resolution, or a 100~200Å probe diameter and a $1 \sim 3 \times 10^{-9}$ A probe current for TV image observation and X-ray analysis with a large current, through adjustment of the condenser lens, thus greatly improving the operational convenience.

3.4 Specimen Chamber

The specimen chamber includes a specimen stage and a specimen exchanging device. The specimen stage is provided with a mechanism of fine control required for observation at high resolution and a tilting mechanism for stereoscopic observation. These mechanisms are incorporated with means of absorbing vibrations and of preventing specimen contamination by electron bombardment, with adequate consideration of the materials. The applicable specimen size is as large as 20mm in diameter and 8mm in thickness. The stage can be moved ±7mm in both the X and Y directions, tilted over 0~45° and rotated through 360°. The specimen exchanging device consists of a pre-evacuation chamber, air-lock device and exchanging mechanism. The specimen is first evacuated in the pre-evacuation chamber, and then transferred to the specimen chamber by use of the air-lock device and exchanging mechanism. The latter functions by pushing and pulling the specimen in a single action. The whole process of exchanging the specimen takes about 3 minutes.

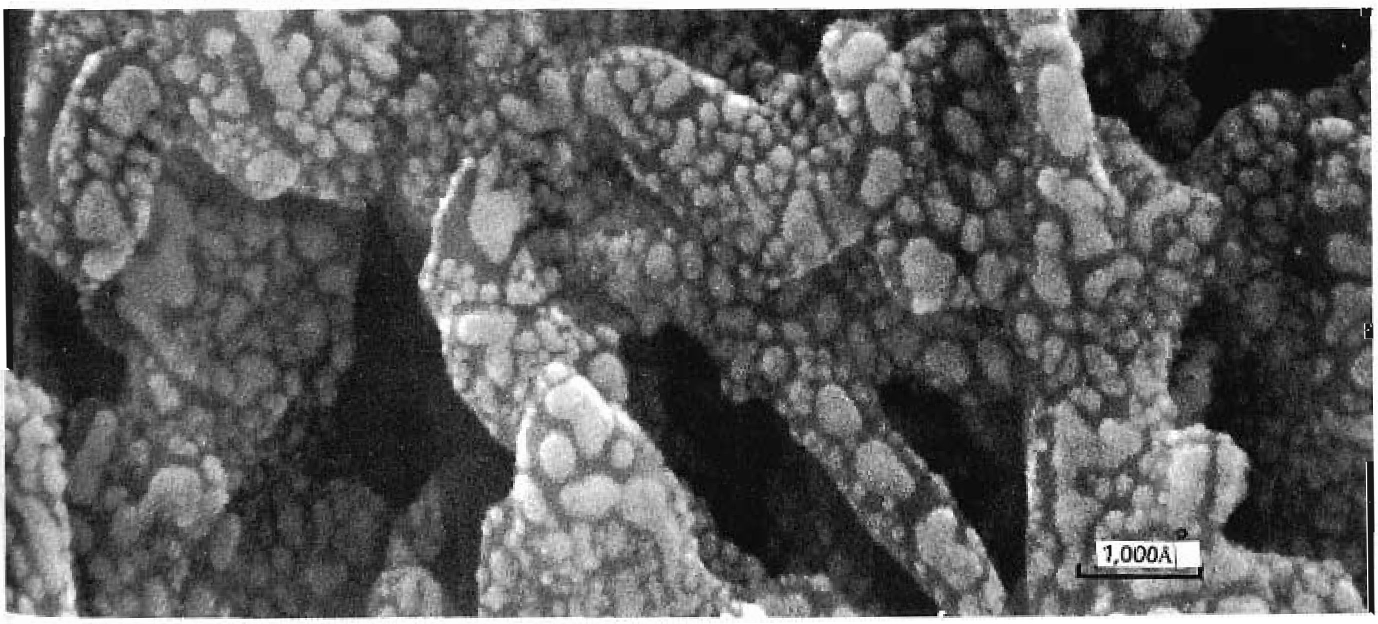

Photo 4. Surface of Magnetic Tape after Gold Vapor Deposition

3.5 Evacuation System

As mentioned in the above, the evacuation system involves three units of ion pumps. In particular, the gun chamber itself constitutes an ion pump chamber, and assures high evacuating efficiency, easily attaining the ultra-high vacuum of 10^{-10} Torr. For the pre-evacuation, a sorption pump and an oil rotary pump with a trap are used in order to make it a clean evacuating system. The trap includes a molecular sieve as the absorbing material to prevent oil vapor from back-streaming out of the oil rotary pump.

3.6 Display System

The electronics are all solid-state, and the circuits are modularized for each function. Controls for ON-OFF and adjustment of high voltage, focusing, astigmatism correction and magnification are provided. Like the conventional scanning electron microscope, modes of secondary electron imaging, and transmitting electron imaging are available on the CRTs for observation and photographing. The characteristic function of the HFS-2 system is that of an imaging amplifier which processes signals from the beam monitor in the intermediate chamber and those from the specimen so as to eliminate the FE noise. This allows the obtaining of a high quality image free from noise.

4. Application Examples

Photo 4 shows an example of a high resolution electron micrograph, that is, a high magnification image of the surface of a magnetic tape after gold vapor deposition. The resolving power is 30Å.

Photo 5 shows cilia on the tracheal eqithelium of the rat.

Photo 6 shows the surface structure of an anodized film of aluminum.

5. Conclusion

The essential details of the principle, construction and features of the field emission source scanning electron microscope have been presented above.

The authors intend to develop a more stable high resolution electron microscope through the improvement of ultra-high vacuum techniques and tip construction and thus contribute to the further development of this instrument.

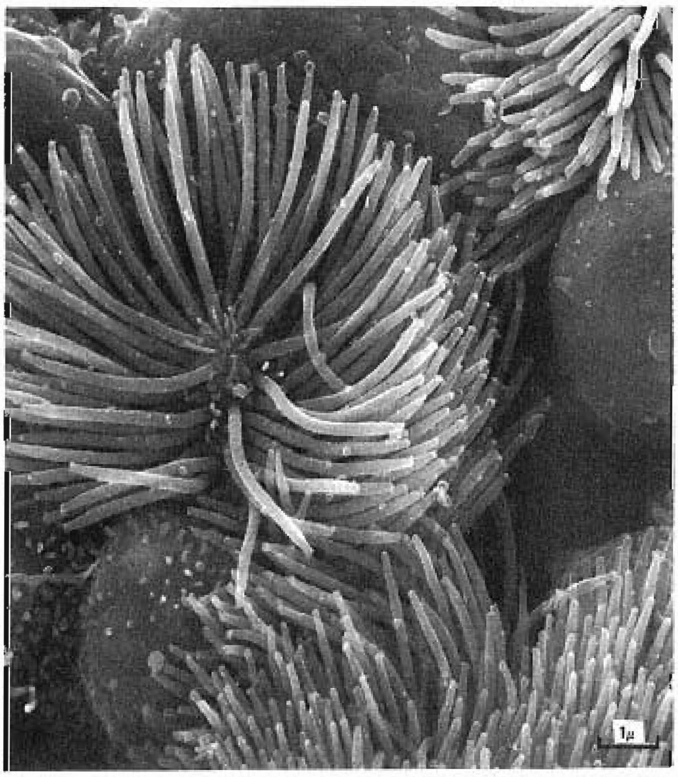

Photo 5. Silia on the Tracheal Epitherium of the Rat

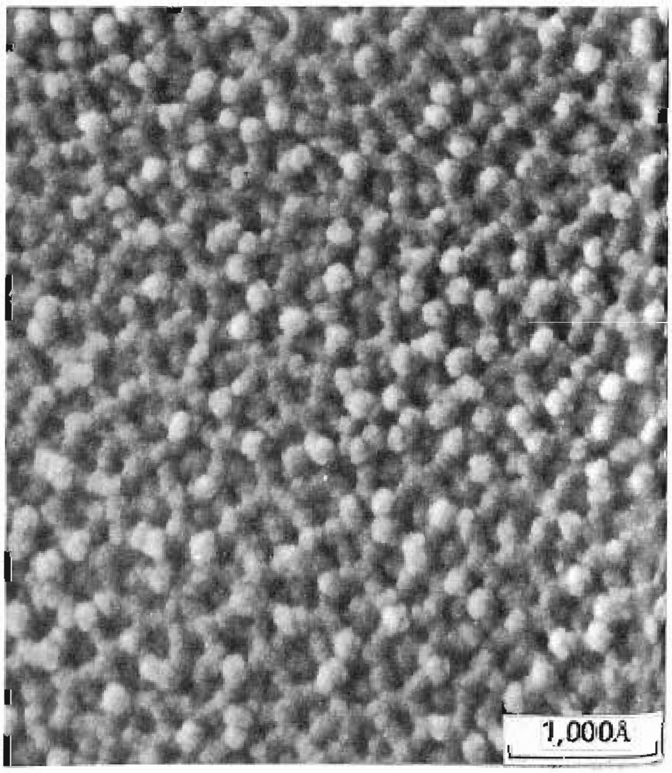

Photo 6. Surface of Anodized Film of Alminum