The Latest Analytical Method and Future Potential with In-lens Thermal FEG

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Introduction

We developed a new field emission scanning electron microscope (FE SEM), Model JSM-6500F, which has an in-lens thermal FEG (II) and is based on the concepts of multipurpose and high-resolution observation and analysis.

It has undergone improvement mainly in respect of spatial resolution. At present, a spatial resolution of 1.0 nm can be realized at an accelerating voltage of 15 kV, and a spatial resolution of 2.2 nm can be realized even at an accelerating voltage of 1 kV, by using a cold field emission gun (C-FEG) that has a small energy spread and also a semi in-lens type objective lens.[1]

The method of performing analysis using an FE SEM on which a C-FEG is mounted is restricted to quantitative and/or qualitative analysis by means of energy dispersive X-ray spectrometry (EDS). This is because it is in practice impossible to make use of the C-FEG for analysis using wavelength dispersive X-ray spectrometry (WDS) or cathode luminescence (CLD) because the maximum probe current of the C-FEG that can be obtained is no more than several nA, it is essential to perform flashing once every 10 hours or so, and also a stable probe current cannot be maintained over a long period.

Also, analysis using an EDS is limited to a high accelerating voltage, hence the excitation area of the X-rays widens, making it difficult to perform microscopic area analysis that effectively utilizes the characteristics of the FE SEM.

Recently, however, in various fields including the semiconductor field, it is becoming increasingly necessary to perform not only observation but also analysis of more microscopic areas, and the appearance of an FE SEM that is intended mainly for analysis is eagerly awaited.

In the case of an FE SEM that is intended for analysis, it is absolutely essential that a large probe current be obtained, and also that the probe current be stable. In order to ensure analysis over a more microscopic area, a finely focused electron beam is required even when the probe current is large.

Development of the In-lens Thermal FEG

An in-lens thermal FEG effectively combines a Schottky field emission electron gun and a first stage condenser lens (CL1) in order to efficiently condense the electron beam emitted from the emitter and provide a probe current of 200 to 400 nA. Figure 1 shows the difference between an in-lens thermal FEG and a conventional FEG. In the case of an in-lens thermal FEG, CL1 is directly beneath the FEG emitter, enabling a large probe current to be obtained efficiently. On the other hand, in the case of a conventional FEG, the electron gun emitter and CL1 are separated from each other; hence it is not possible to adequately condense the electron beam emitted from the emitter.

As a result of the adoption of an in-lens thermal FEG, the JSM-6500F can provide a probe current of 200 nA, which is 10 times that of a conventional SEM that uses a thermal FEG.

Also, if the aperture angle (II) of the objective lens is optimum (optimum II), the spatial resolution when a large probe current flows in the FE SEM will be affected by the spherical...
aberration of CL1[2]. For this reason, we designed CL1, which is used in an in-lens thermal FEG, in such a way that it had a sufficiently low spherical aberration coefficient.

Figure 2 shows a secondary electron image obtained under analytical conditions. We confirmed that the spatial resolution was 3 nm under conditions that are often used for analysis (accelerating voltage 15 kV, probe current 5 nA, and WD 10 mm). Figure 3 shows the observed image of evaporated gold particles that were deposited using an accelerating voltage of 15 kV, a probe current of 100 nA and a WD of 10 mm.

The Schottky field emitter used as the electron source features high brightness, small energy spread, and good stability[3]. The results of evaluating the stability are shown in Fig. 4. We confirmed that the stability after 13 hours under conditions frequently used during analysis (accelerating voltage 15 kV, probe current 5 nA) was 1% or better.

Application Example

The following are examples of data obtained for applications that require a large probe current.

Electron backscattered diffraction (EBSD)

An EBSD is an instrument that has been attracting much attention recently in various fields. It assigns an index to the crystal orientation of a specimen. The general observation conditions used in an EBSD are an accelerating voltage of 15 kV to 25 kV and a probe current of 0.5 nA to 10 nA.

Conventionally, when performing EBSD analysis, a multi-purpose SEM (W-SEM) that has a tungsten hairpin filament is used because a large probe current and long acquisition time are required to perform measurement. However, in a W-SEM, the beam diameter under the above observation conditions increases to above 50 nm; hence it is not possible to obtain a pattern in the case of a specimen that has microscopic crystal grains such as subgrains.

Figure 5 shows an example of the results of EBSD analysis, which can only be obtained with a FE SEM. The results shown here are for a cross-section of cold-rolled steel. Steel sheet used in various fields such as the automobile industry is required to have excellent forming ability; hence the crystal orientation at the cold-rolling and heat treatment processes is controlled.

Figure 6 shows an example of analysis of 0.3 µm Cu wire in a semiconductor. The distribution of the crystal orientation in this microscopic area can be seen clearly.

In this way, EBSD is used as an important evaluation tool that determines the manufacturing process in various fields.

Example of application to wavelength dispersive X-ray spectrometry

In WDS, the probe current must be 100 nA or more and the beam must have a stability of 1% or lower per hour. In an FE-SEM installed with a C-FEG, it has been difficult to satisfy these conditions.

The JSM-6500F can meet these conditions. Figure 7 shows a measurement example obtained using the JSM-6500F.

This specimen is black ore (Fig. 7(a)) which includes a layer that contains Pb and S. In EDS, the Pb and S peaks overlap each other, as shown in Fig. 7(b). In WDS, the peaks are separated, as shown in Fig. 7(c).

In this way, WDS, which has high energy resolution, permits quantitative and qualitative analysis of elements, including those that cannot be separated by EDS.

Example of application to energy dispersive X-ray spectrometry

EDS is the most commonly used method of analysis in an FE-SEM that has a C-FEG. The characteristic X-rays emitted from the specimen are generated from a range that depends upon the specimen and the accelerating voltage. Consequently, the lower the accelerating voltage, the smaller is the area over which analysis can be performed.

However, with the C-FEG, the probe current obtained at a low accelerating voltage becomes
small, preventing adequate sensitivity from being obtained, during EDS analysis.

This instrument permits adequate probe current to be obtained, even when the accelerating voltage is reduced to the lower limit at which X-rays are generated. As a result, superficial parts of the specimen, that is, areas where the X-rays are not dispersed, can be mapped. **Figure 8** is a two-dimensional image of a lava from Miyake Island in Tokyo, Japan. **Figures 9(a) to (c)** are the results of EDS mapping of O, Mg and Al measured at an accelerating voltage of 15 kV and a probe current of 1.8 nA. **Figures 10(a) to (c)** are the results of EDS mapping of O, Mg and Al measured at an accelerating voltage of 5 kV and a probe current of 2.8 nA.

Thus, a large probe current can be obtained at a low accelerating voltage, enabling a sharp map to be obtained in a short period, even when mapping on the light element side where the image becomes blurred at a high accelerating voltage.

**Other conceivable applications**
The finely focused large probe-current electron beam available with this instrument can conceivably be used in many applications such as electron-beam pattern writing and CLD.

**Conclusion**
By using this in-lens thermal FEG, we were able to obtain a maximum probe current of 20 times that of a conventional thermal FEG. Because we were also able to obtain a large probe current with an FE SEM, it is now possible to perform analysis using WDS, EBSD, and CLD, in addition to EDS.

By using the above-mentioned instrument that has a high spatial resolution and is capable of obtaining a more stable and large probe current, it is now possible to analyze the structure of a microscopic area of a specimen that could only be morphologically observed, but not analyzed, using a conventional instrument.

**References**