Abstract

Field Emission Scanning Electron Microscope (FE-SEM) provides ultra-high resolution imaging at low accelerating voltages and small working distances. The GeminisSEM 500, a new FESEM imaging facility will be installed soon in MTEC, BTI. It provides resolution of the images as low as 0.6 nm at 15 kV and 1.2 nm at 1 kV, allowing examination of the top surface of nanopowders, nanofilm and nanofiber in the wide range of applications such as mineralogy, ceramics, polymer, metallurgy, electronic devices, chemistry, physics and life sciences. This system is equipped with several detectors to detect various signals such as secondary electrons (SE) detector for topographic information and back-scattered electrons (BSE) detector for materials composition contrast. Energy dispersive x-ray spectroscopy (EDS) with detector energy resolution of < 129 eV and detection limit in the range of 1000-3000 ppm coupled with FE-SEM is used to determine the chemical composition of micro-features including boron (B) to uranium (U). Wavelength dispersive x-ray spectroscopy (WDS) which has detector resolution of 2-20 eV and detection limit of 30-300 ppm coupled with FE-SEM is used to detect elements that cannot be resolved with EDS. The ultra-high resolution imaging combined with the high sensitivity WDS helps to resolve the thorium and rare earth elemental analysis.

Key words : field emission scanning electron microscope, low kV imaging, energy dispersive spectrometer, wavelength dispersive spectrometer

INTRODUCTION

The first true scanning electron microscope (SEM) was described and developed in 1942 by Zworykin, who showed that secondary electrons (SE) provided topographic contrast by biasing the collector positively relative to the specimen. He reached the resolution of 50 nm when using an electron multiplier tube as a pre-amplifier of the SE emission current [1]. Since then, many improvements had been made until the first commercial SEM was made in 1965 by Cambridge Scientific Instruments Mark I called ‘Stereoscan’ [2]. This instrument showed great SE detection using Everhart-Thornley detector (ETD) which was found by Everhart and Thronley in 1960. ETD is a detector to collect electrons with a positively biased grid comprising a scintillator to convert the electrons and a light-pipe to transfer the light directly to a photomultiplier tube (PMT) [1]. The SEMs that we are using today are not very different from this one.

The early SEM used heated tungsten hairpin or filament cathode as the electron source, which known as thermionic emitter (Fig. 1a). The development of lanthanum hexaboride (LaB$_6$) cathodes in 1975 which replacing tungsten became the major improvement in instrument performance [3] and still can be found on many instruments today. Thermionic emitter emits high current with beam size of 4-8 nm. It is although inexpensive and reliable, the beam currents produced are definitely low brightness and resulting evaporation of filament so-called thermal drift, which limits the optical performance especially at high-resolution. This classic SEM also accustomed users to operating at high beam voltage i.e 15-30 kV either necessary or not. This has led to many assumptions that SEM is incapable of producing high-resolution images for many heat intolerant samples such as biological and polymer materials.
The only electron source designed for high-resolution imaging and suitable for various kinds of materials is field emission, which uses field emitter gun (FEG) to emit electrons. The SEM that uses FEG as the emitter type is called field emission scanning electron microscope (FESEM). The SEM that uses FEG as the emitter type is called field emission scanning electron microscope (FESEM). Field emission, which uses field emitter gun (FEG) to emit electrons. The SEM that uses FEG as the emitter type is called field emission scanning electron microscope (FESEM), whereby the emitter type is used as a part of its name to distinguish it from the classic SEM. FEG is made up of tungsten wire that has diameter about 100 nm (Fig. 1b), preferably a single crystal type, designed in such that the 310 plane is perpendicular to the electron optical axis. The gun tip is placed near an anode which held at +2-6 kV, the sharpness of the point produces fields of $\sim 10^{10}$ V/m near its surface (Fig. 1c). This will result electrons to tunnel through the barrier into the vacuum at a much focused beam ($\sim 2$ nm). The current emitted is seldom reaching more than 5-10 μA, however it has brightness far higher than thermionic emitter [4]. The first reliable FESEM was developed in 1968 by Prof. Crewe at Argonne National Laboratory [5].

FESEM is developed based on a technology for high-resolution imaging and different contrasting methods aiming for a comprehensive characterization of specimens. FESEM can be used in wide range of applications including imaging surface sensitive and non-conductive samples without the need for pre-treatment. FESEM also comes with various attachments for elemental analysis. This paper describes the technical specifications and possible applications of new FESEM facility in MTEG, BTI.

**TECHNICAL SPECIFICATIONS**

These technical specifications describe FESEM with model GeminiSEM 500 developed using Gemini technology by Carl Zeiss (Fig.2). This SEM is designed based on more than 20 years of experience in imaging technology. The GeminiSEM 500 is a system comprising a high-resolution field emission microscope, airlock chamber for specimen changing, plasma cleaner for sample and chamber cleaning and an in situ cleaning apparatus. There are three main advantages offered by this model; efficient detection, excellent resolution and unsurpassed ease-of-use.

**Electron optical system**

One of the main components in FESEM is electron beam column that describe the electron optic design. The column accommodates the FEG and lenses. The design is to enable smallest probe size possible for high resolution imaging. The novel optical design of the GeminiSEM is mainly about the innovation to the FEG mode, novel optics and beam booster technology (Fig.3) [6]. In Gemini technology, a special gun mode was
developed to reduce the energy spread of the primary beam to reduce the effect of chromatic aberration. Most FEG is designed to have energy spread as low as 0.35 eV as compared to 1.5 eV for thermionic emitter [7]. The probe current is in the range of 3 pA to 100 nA. The probe size is another important criterion for high resolution imaging. Electron optic is used to demagnify the size of electron source to form smallest possible probe for high resolution. The demagnification is achieved using a series of lens known as ‘probe-firming’ lens comprising condenser and objective lens [7].

![Figure 3: The electron optic column design for GeminiSEM 500 [6]](image)

Aberrations are lens imperfections due to spherical or chromatic effects which limit the ability of focusing the electron beam on the surface and thus blur the image. In spherical aberration, rays travelling from the optical axis are focused more strongly than those close to the axis whereas in chromatic aberration electrons with slightly different wavelengths are focused more or less strongly. To reduce spherical aberrations, objective lens aperture is used to limit the angle if the outer rays through the lens, while electron beam with low energy distribution is used to limit the chromatic aberrations. The Gemini lens design is Nano Twin Geminin lenses comprising compound magnetic-electrostatic objective lens (Fig.3)[6]. These lens have been further optimized in terms of geometry and electrostatic as well as the magnetic fields distributions. In this way these lens may provide resolution of 1.2 nm at 500 V, 1.1 nm at 1 kV and 0.6 nm at 15 kV.

Moreover, the Gemini beam booster technology can guarantee the imaging is done using small probe sizes and high signal-to-noise ratios down to ultra-low accelerating voltages (Fig.3)[6]. This technology ensures that small probe size imaging can be done even at voltages lower than 1 kV. Therefore the chromatic and spherical imaging aberrations (Cc and Cs) are decrease significantly with decreasing beam energy [8]. Furthermore, the sensitivity to external stray fields is minimized by keeping the beam at high voltage throughout the column until its final deceleration [6].

**Low voltage imaging**

The high resolution imaging at low voltage is needed to avoid beam damage and to balance the secondary electron (SE) yield and beam current for charge neutrality. The advantage of low voltage imaging is to improve image contrast, for example by reducing operating voltage from 10-20 kV to perhaps 1.5 kV can solve problem of low topographic image contrast on bulk specimen. As the electron range is proportional to E^{5/3}, dropping beam voltage by this amount decreases the approximately spherical interaction volume by about 10^6 [4]. Previous studies showed that, by reducing the beam energy from 20 kV to 1.5 kV the smallest features visible in the final image was the size of the probe [9]. The nanoscale details can be resolved with high contrast images at low beam voltages. The advantage of Gemini system is to achieve high resolution at low voltages. The acceleration voltage for GeminiSEM 500 is from 0.02 to 30 kV. By combining the low probe current with smallest possible probe size and at low acceleration voltage GeminiSEM 500 provides magnification from 20 to 2,000,000 times.

**Detection system**

Various signals are generated as a result of the impact of incident electrons to the specimen. There are mainly low energy secondary electrons (SE) (energies of < 50 eV), high energy backscattered electrons (BSE) (energies...
of > 50 eV) and characteristic x-rays (Fig. 4). These signals are collected using detectors to form an image or to analyze the samples’ surface. The right detection of these electrons will give detailed information about the samples. To get high resolution images, the sufficient signals from surfaces are vital. These signals are converted from the electrons that are coming from the surfaces. The GeminiSEM system comes with significantly improved detection efficiency. The detection concept ensures efficient signal detection by detecting SE and BSE electrons in parallel. These so-called ‘In-lens’ detectors are arranged on the optical axis, which reduces the need for realignment and thus minimizes time-to-image. The higher signals give advantage by getting images at reduced time and this is very useful especially when dealing with low current imaging to avoid sample damage. The types of detectors offered are standard In-Lens SE detector, high efficient Everhart Thornley (ETD) SE detector and angular selective BSE detector.

In-lens SE detector is used to detect SE signals directly from sample’s surface (Fig. 5a). It is mounted on the axis with electron beam path in the objective lens (annular type) for ultra-high SE detection. SEs are attracted to the detector by the electrical field in the column and are deflected to the plane of SE detector by the objective lens. The high efficiency Everhart-Thornley SE detector (ETD) is used for higher bandwidth electrons (wide range of angles relative to the primary beam), high quantum efficiency and without the addition of substantial noise. Moreover the high efficiency angular BSE detector is used to detect signals from high angle back-scattered electrons (HABE) (Fig. 5b). BSE are electrons scattered backward from specimens comprising higher energy than SE (> 50 eV). They are deflected to different angle from SE (15° relative to the primary beam) and have single and multiple scattering depending on deflection angle. In Gemini design, the detector used to detect BSE is called In-Lens EsB (energy selective backscatter) detector, mounted on the optical axis in the column. This detector comes with an energy filtering grid to select or filter energy from 0 to 1500 eV so that a separation of SE and BSE is enabled. Energy filtering is important to get pure HABE that is useful for topographic contrast and compositional imaging (Fig. 5b). EsB detector works well even at low voltages and show excellent sensitivity. This system also comes with another detector for low angle BSE (LABE) with lower energy. LABE does not enter column but land on objective lens, thus they are detected via low angle BSE detector called AsB4 (angular BSE detector). LABE signals give information about compositional and crystallography contrast of materials which can be used in 3D surface modelling.
All detectors usually optically coupled with a photomultiplier and collector which allow faster scan speed image acquisition compared to standard ETD with the same signal to noise ratio. The Gemini objective lens with a novel design optimizes in-lens SE detection as it not only acts as an imaging lens, but also enhances detection for SE and BSE emitted by the sample: the electron trajectories for the detection path are further improved by the novel design of the objective lens. Here, the in-lens SE signal is up to 20 times higher compared to classic SEM designs. This enables imaging at very low voltages and usage of fast scan speeds for high speed sample investigation. At the same time the in-lens detector signal is boosted by up to 20 times under low voltage imaging conditions.

**Elemental analysis**

Besides surface imaging, FESEM can be used for compositional analysis for determining elements present in a sample. For this, two different types of spectrometers are attached to FESEM to perform this function; an energy dispersive spectrometer (EDS) and wavelength dispersive spectrometer (WDS) (Fig. 6). The elements are identified through detecting the characteristic X-rays that emitted from specimens when bombarded with electrons. Each specimen produces a unique characteristic X-rays with a specific energy and wavelength, representing each element in the sample. EDS identify the X-rays based on their energy; while WDS separate the X-rays based on their wavelengths. In EDS system, the central component is a semiconductor solid-state detector whereas in WDS the main components are analyzing crystals and a detector. Based on these two different components, EDS and WDS have distinct operating principles.

![Figure 6: The arrangement of elemental analysis attachments; EDS and WDS on GeminiSEM 500](image)

In EDS, when each X-ray photon hits the detector, a very small current is produced by knocking out electrons from the semi-conductor. Each electron ejected from a silicon electron shell consumes a specific energy of the element. By measuring the amount of current produced by each X-ray photon, the original energy of the X-ray can be calculated, thus the element is identified. On the other hand in WDS, those characteristic X-rays that hit the crystal will diffract and enter the detector. Whether an X-ray photon will diffract depends on its wavelength, the orientation of the crystal, and the crystal's lattice spacing. Only X-rays of a given wavelength will enter the detector at any one time. WDS spectrometer typically has between two to five analyzing crystals, each with a different lattice spacing, because each type of crystal can diffract only a given range of wavelengths. To measure X-rays of another wavelength, the crystal and detector are moved to a new position.

The most significant difference between WDS and EDS systems is their energy resolution. A Mn Ka X-ray line on an EDS system have typically between 135-150 eV wide; whereas on a WDS system, this same X-ray line will only be about 10 eV wide. This means that WDS has 10 times better energy resolution than EDS, hence the amount of overlap between peaks of similar energies is much smaller. However, since a specific WD spectrometer can measure only one X-ray wavelength at a time, it requires longer analysis time than EDS. Another disadvantage of EDS is the detector that using Berillium as window; produce detection limit in the range of 1000-3000 ppm that is not useful for lightest elements determination (below atomic number of Na). Meanwhile WDS has detection limit in the range of 30-300 ppm hence show a much better performance for light elements analysis than EDS.

Both EDS and WDS spectra are presented as histogram of the number of X-rays measured at each energy. In a normal surface imaging that requires quick phase identifications of major elements, typically EDS is used. The spectrum can be collected in a very short period of time. For challenging samples such as mineral containing rare earth elements, WDS is the best tool because the overlapping X-ray lines can be to identify minor and trace elements throughout the entire periodic table. EDS and WDS scanning can be used simultaneously during
imaging to provide more accurate results especially for rare earth elements. Furthermore, EDS and WDS are both can do elemental mapping to show distribution of X-ray counts for all identified elements in the acquisition. Using mapping the positions of specific elements emitting characteristic x-rays can be indicated by unique color. The benefits of mapping are; the visual assessment of the spatial distribution of the elements in the sample; and the RGB color overlays to assess regions of interest.

APPLICATIONS

The GeminiSEM imaging facilities are the ideal choice for maximum sample flexibility for high performance, high resolution imaging and excellent compositional materials analysis. This advance facility can be used in a wide range of options, application-specific modules and workflows and give satisfaction for various applications for top surface imaging and elemental analysis of nanopowders, nanofilm and nanofiber. These cover various fields such as mineralogy, ceramics, polymer, metallurgy, electronic devices, chemistry, physics and life sciences. In nanoscience researches, the GeminiSEM 500 Nano-twin lens can be used to get image for materials that beam-sensitive and detailed nanoscale structures at low beam energy. The efficient detection allowing operating at low currents for minimum beam damage and excellent materials contrasts can be obtained. This equipment has successfully used to characterize the carbon nanostructures, engineered and self organized nanosystems, and nanocomposite materials [6]. In metallurgy studies, the Gemini complete detection system can be used to characterize inclusions at ultra-high resolution and discriminate between different phases with unparalleled contrast. Whereas in electronic devices and semiconductors, GeminiSEM 500 enables rapid, reliable and damage-free characterization of nanoscale defects and sensitive resist structures at low beam energies. For life sciences applications, GeminiSEM 500 can give images of subcellular structure and tissue mapping. In polymeric materials, the image of nano fiber such as kenaf and high density polyethylene can be obtained at high resolution. In MTEC, BTI we tried to use FESEM to characterize the minerals; monazite and xenotime to support the thorium flagship research. We used EDS elemental mapping to get the distribution of elements present. Furthermore we use WDS mapping to get the distribution of low concentration elements in the materials and quantitative analysis to accurately measure the content of the elements that cannot be detected by EDS.

CONCLUSION

The GeminiSEM 500 offer complete and efficient detection, excellent resolution and the ease of use based on the 20 years of experience in imaging technology. With the latest Gemini lens design, the imaging can be done at highest resolution at wide range beam voltages covering from 30 kV to as low as 10 V. This equipment offers high flexibility in imaging and elemental analysis with the help of various detectors such as In-Lens SE, In-Lens EsB, EDS and WDS respectively.

REFERENCE