Development of a compact FE-SEM and X-ray microscope with a carbon nanotube electron source

M Irita\textsuperscript{1,2}, S Yamazaki\textsuperscript{1}, H Nakahara\textsuperscript{1} and Y Saito\textsuperscript{1}

\textsuperscript{1} Nagoya University, Department of Quantum Engineering, 464-8603 Nagoya, Japan
\textsuperscript{2} Nagoya University, Venture Business Laboratory, 464-8603 Nagoya, Japan

E-mail: masaru.irita@surf.nuqe.nagoya-u.ac.jp; ysaito@nagoya-u.jp

Abstract. A carbon nanotube (CNT) possesses various benefits as a field electron emitter. Multi-walled CNT (MWNT) have exhibited high brightness and the field emission (FE) current is stable even under poor high-vacuum condition of $10^{-6} - 10^{-7}$ Pa without ion pumping. In this study, we have experimentally manufactured a compact composite field emission scanning electron microscope (FE-SEM) and X-ray microscope (XRM) with a single isolated MWNT electron source. An electron beam from a MWNT was focussed and accelerated by a Butler electrostatic lens. Stable SEM images were observed at a Butler voltage $V_{\text{but}} = 1.5$ kV and accelerating voltage $V_{\text{acc}} \approx 15$ kV with a focussed probe current $I_{\text{foc}} = 61$ pA. The spatial resolution of the SEM, which was estimated from 80 - 20 % edge profile of polystyrene latex spheres, was about 9 nm. XRM images were acquired by 30-min exposure at $V_{\text{but}} = 1.5$ kV and $V_{\text{acc}} \approx 17$ kV. A spatial resolution of about 200 nm was obtained for XRM. The present results prove the high performance of the compact FE-SEM and XRM using a single isolated MWNT electron source at $10^{-7}$ Pa.

1. Introduction

Carbon nanotube (CNT) possesses various benefits as a field electron emitter, e.g., needle shape with a sharp tip, high chemical stability, mechanical strength and electrical conductivity. The sharp tip enables not only low voltage operation but also realisation of high brightness and restriction of emission area, which are advantageous for forming a finely focussed electron beam with a simple lens system. Chemical inertness and low atomic diffusion of a CNT surface make an electron emission stable even under moderate ultrahigh vacuum, which alleviates the need for a massive vacuum pumping system. By employing CNT as an electron source, therefore, a field emission scanning electron microscope (FE-SEM) can be miniaturised whilst keeping high performance.

FE from CNT have been studied for over 25 years [1]. Multi-walled CNT (MWNT) have exhibited high brightness of $10^9 - 10^{10}$ A/(cm$^2$•sr) as an electron source for FE-SEM [2]. Furthermore, the FE current is stable even under poor high-vacuum condition of $10^{-6} - 10^{-7}$ Pa without ion pumping [3]. In previously demonstrated CNT-based FE-SEM [4, 5], the residual gas pressure in their electron gun chambers was at a level of $10^{-3}$ Pa, being inadequate to maintain stable FE even for CNT. Thus, a ballast resistance of 10 - 20 M$\Omega$ had to be added in series to the emitter in order to stabilise the emission current. [4, 5]. In the scene of an archaeological site excavation and planetary exploration, for example, mobile microscopes are necessary to investigate samples on site.
In the present study, we have experimentally manufactured a compact dual-mode microscope of FE-SEM and X-ray microscope (XRM) with a single isolated MWNT electron source. The electron source and electrostatic lenses for extraction and focussing (Butler lens, indicated in figure 1b) are installed in an ultra-high vacuum chamber. A stable electron emission without any ballast resistance is demonstrated, and the performance of the SEM and XRM is reported.

![Figure 1](image-url)

**Figure 1.** (a) Structure of the CNT-FE-SEM/XRM. (b) Electrostatic lens system. (c) Top view of sample chamber design.
2. Experimental
A drawing of the dual microscope equipped with a CNT FE emitter is shown in figure 1a. The CNT electron source and a set of electrostatic lenses for extracting and condensing an electron beam are installed in an ultra-high vacuum gun chamber, which can be baked and is evacuated by a turbo-molecular pump system. The gun/condenser lens chamber is connected, via an ICF 114 flange, with a column of a commercially-available compact SEM (Tiny-SEM, Technex Lab Co) in which scanning coils and an objective lens made of permanent magnets are installed. Figure 1c shows a schematic of the sample chamber. The Tiny-SEM sample-chamber is equipped with a secondary electron detector and is also employed. The electron source chamber and the sample chamber were differentially pumped with two turbo-molecular pumps. The ultimate pressure of the electron source chamber was 10⁻⁷ Pa. For measuring X-ray micrographs, a target made of Au is placed at the position where the electron beam is focussed and a sample is set between the X-ray source and the X-ray detector (XRI-UNO, XRAY IMATEK Co) as shown in figure 1c. The X-ray detector is a two-dimensional imaging device of 14.1 × 14.1 mm² with 256 × 256 pixels. The detector is based on a silicon sensor being effective for X-ray photons with energy in a range between 4 and 25 keV. A projection-type XRM based on the CNT-FE-SEM was constructed with the Au target for the X-ray source. The total length of the column from the top of the gun to the bottom of the sample chamber is 317 mm.

An electrostatic lens system, consisting of a control electrode, an extraction electrode and Butler lens, was specially designed for the CNT emitter as shown in figure 1b. The control electrode, covering the CNT emitter, was set at the same electric potential as the CNT emitter. The geometrical sizes and potentials of the electrostatic electrodes were optimised by paraxial approximation and by electron trajectories simulations (ELFIN). At an acceleration voltage of between 15 and 17 kV, an electron beam from a CNT emitter was focussed at an object plane of the magnetic objective lens, and then the focussed beam is projected on a sample plane. The CNT used was a MWNT produced by an arc discharge method. A single isolated MWNT, which was attached to the tip of a tungsten (W) needle using micromanipulators under an ordinary SEM observation [3], was used as the FE electron source. Figure 2 shows the MWNT electron sources used for SEM and XRM operation, respectively. The length of MWNTs was 150 and 30 nm extending from the W needle apex as shown in figures 2a and 2b, respectively.

![Figure 2. SEM images of CNT emitters used for a) SEM, and b) XRM operation, respectively.](image-url)
3. Results and discussion

The MWNT electron source and Butler electrostatic lens were installed in the gun chamber, which was evacuated with a turbo-molecular pump down to $10^{-7}$ Pa. The CNT emitter operated stably without any ballast resistance. Insertion of a ballast resistance is not desirable, because the electric potential at the emitter tip fluctuates due to a voltage drop in the ballast resistance. Fluctuation of the emitter potential broadens the energy distribution of emitted electrons, which deteriorates the resolution of SEM images. In the present FE-SEM, such fluctuation of the emitter potential due to a ballast resistance is avoided. FE from a single isolated MWNT (figure 2a) was focussed and accelerated by a Butler electrostatic lens. FE current $I_{\text{emi}}$ and focussed beam current $I_{\text{foc}}$ (at Butler voltage $V_{\text{but}} = 1.5 \text{ kV}$ and accelerating voltage $V_{\text{acc}} = 15 \text{ kV}$) as a function of the extraction voltage $V_{\text{ext}}$ are presented in figures 3a and 3b, respectively. The focussed beam current was measured by a Faraday cup. It has been experimentally observed that $I_{\text{foc}} / I_{\text{emi}}$ was less than 0.01 %. High current electron beam is not necessary for achieving nano-resolution SEM images. The beam diameter becomes large when a high beam current is used. Thus, the resolution of SEM images depended on the current of the focussed beam. The most stable SEM images were observed at $I_{\text{foc}} = 61 \text{ pA}$ in our microscope.

![Figure 3. a) FE current, and b) beam current versus the extraction voltage characteristics.](image)

An example of an SEM image acquired using $I_{\text{foc}} = 61 \text{ pA}$ at $V_{\text{but}} = 1.5 \text{ kV}$ and $V_{\text{acc}} = 15 \text{ kV}$ is shown in figure 4a. The sample is Au-Pd coated polystyrene latex spheres (PLSs), which are 200 or 500 nm in diameter. Figure 4c shows the line profile across an edge of a PLS. The spatial resolution, which was estimated from the width at 80 - 20 % height of a fitting profile across the SEM image, was 9 nm, suggesting that the beam diameter was approximately 9 nm.

It should be noted that a jaggy-like noise is contained in the SEM image as shown in figure 4a. The period of noise was 60 Hz, indicating the noise is due to a power source. If the noise is removed, the resolution would be improved.

Next, we observed XRM images using the MWNT electron source shown in figure 2b. A typical XRM image is shown in figure 4b, which was acquired by 30-min exposure at $V_{\text{but}} = 1.5 \text{ kV}$ and $V_{\text{acc}} = 17 \text{ kV}$. The sample is 400-mesh Au grid. In contrast to the SEM image, the XRM image shows that the regions of high transmission appear bright. In the XRM image, the mesh hole appears bright and the bar appears dark. Figure 4d shows a line profile across the bar. The spatial resolution is estimated to be about 200 nm from the width at 80 - 20 % of an intensity change at the edge.
In our microscope, the electron beam spot size $\delta_e$ is considered to be approximately equal to the resolution of SEM, i.e., $\sim 9$ nm. The X-ray source size $\delta_X$ is estimated to be $220$ nm for the Au target at 15 keV by using Castaing's equation [6]. Smearing due to Fresnel diffraction is expressed as $\delta_P \approx \sqrt{a\lambda}$, where $a$ is the distance between source and sample, $a = 0.48$ nm and $\lambda$ is wavelength of X-ray. Using $\lambda = 0.13$ nm, which is the characteristic X-ray of Au-Lα1, $\delta_P \approx \sqrt{a\lambda} = 200$ nm. Therefore, the theoretical spatial resolution is $\delta = \sqrt{\delta_e^2 + \delta_X^2 + \delta_P^2} \sim 300$ nm. The theoretical resolution is a little bit worse (1.5 times) than the experimental one, which may be due to the rough estimation of $\delta_X$.

4. Conclusion
The present study has demonstrated the high performance of FE-SEM and XRM observation equipped with a single MWNT emitter operated at $10^{-7}$ Pa. The microscope has been miniaturised to a compact size that it can be placed on a desk. The present study shows a potential that the CNT-based SEM and XRM are applied to the scene of an archaeological site excavation and future planet search.
Acknowledgments
The authors also thank H. Murata, Meijo University for paraxial trajectory calculations and T. Ohno, Technex Lab Co. for valuable advice on modification of the Tiny SEM. The authors acknowledge the financial support from the Ministry of Education, Culture, Sports, Science and Technology of Japan (Grant-in-Aid for Scientific Research (B), No. 25286024).

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