Application of Low Energy STEM with the In-lens Cold FE-SEM

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Abstract. We have developed a high resolution STEM imaging method using accelerating beam voltages of 30 kV and less. In this study, we examined and determined the low accelerating voltage parameters necessary for obtaining lattice resolution in STEM images, using the cold field emission scanning electron microscope (CFE-SEM), Hitachi SU9000. We also investigated and optimized the objective lens conditions for optimum lattice fringe imaging. The STEM images and associated Fourier transform image obtained at 30 kV show the Si (222) lattice fringes and reflection spots, corresponding to the d-lattice spacing of 0.157 nm. The STEM images and associated Fourier transform image at 15 kV show the Si (111) lattice fringes and reflection spot, corresponding to the d-lattice spacing of 0.314 nm.

Introduction

In the past, the transmission electron microscope operating at an accelerating voltage of 200 kV or higher has been used extensively for structural characterization of semiconductor, metallic and ceramic materials. As the dimensions of IC semiconductor devices shrink down to 20 nm (and smaller) it is critical to produce thin specimens of less than 20 nm in thickness. This is necessary to avoid overlapping individual structures and therefore blurring the STEM image of an individual device. Additionally, inorganic carbon materials (primarily carbon nanotubes and graphene) and organic polymeric materials are being widely investigated as viable post-CMOS device materials. The abovementioned materials share the STEM specimen problems of electron beam-induced structural disorder (due to knock-on damage) and a reduction of STEM image contrast due to the small ratio of elastic to inelastic scattered electrons and the narrow angle of the scattered electrons. Low energy microscopy reduces beam-induced damage and enhances scattering contrast, but also reduces resolution as the electrons’ wavelength increases. Accordingly, it is necessary to determine methods for reducing specimen damage and improving contrast without loss of resolution. To specifically address that need, research institutions, academia, and private industry have collaborated to develop and commercialize a novel low energy FE-SEM/STEM. [1-4].

The imaging resolution of a 30 kV FE-SEM with a STEM function has recently been improved, thanks to a new lower spherical aberration objective lens, and a high stability and coherency (ΔE) electron gun. The cold field emission scanning electron microscope (CFE-SEM) with an in-lens type objective lens, Hitachi SU9000, provides all the above mentioned benefits and made possible the development of our high resolution STEM technique. Through using this new method it is possible to
routinely achieve lattice resolution, showing the lattice fringes of the graphite (002) plane with a spacing of 0.34 nm [5].

In this study, we describe the observation conditions for obtaining lattice resolution enhancement in STEM imaging at an accelerating voltage of below 30 kV.

**Experimental method**

The SU9000 CFE-SEM’s accelerating voltage range is from 500V to 30 kV, making it possible to attempt lattice imaging below 30 kV without special equipment. We selected a single-crystal silicon specimen to illustrate how our low accelerating voltage STEM technique would apply to the analysis of current semiconductor devices. The sample was thinned by an Ar⁺ ion milling technique, producing a final sample thickness of about 20 nm.

Theoretical resolution is limited by electron diffraction and aberration effects. For every SEM, there is an optimum beam half angle for minimum theoretical resolution. In the SU9000 FE-SEM’s case, this optimum beam half angle is approximately 10 mrad at 30 kV (maximum accelerating voltage). Normally this optimum beam half angle is selected when observing the real objective image because the image resolution is best at this condition. On the other hand, for lattice image observation is necessary to set the beam half angle to \( \alpha > 2\theta_B \). In order to observe the Si (111) lattice fringes (\(d=0.314\) nm) at accelerating voltages of 30 kV and 15 kV, the beam half angles needed to be set to \( \alpha > 22.5\) mrad and \( \alpha > 32.5\) mrad respectively. Therefore, in order to obtain the lattice image of Si at 30 kV and 15 kV, we adjusted the objective aperture, 2nd condenser lens, and objective lens to produce a larger beam half angle than the one for optimum resolution.

The objective lens spherical aberration coefficient (Cs) increases as the amount of defocus between STEM structural imaging and STEM lattice imaging also increases. The optics of the SU9000 need a great amount of defocus for optimizing lattice fringe imaging and clearly presents a significant challenge. For this reason, it is difficult to observe the structural and lattice fringes information of the specimen simultaneously. However, it is possible to reduce Cs by reducing the focal length which results in a short working distance. Therefore, we made an experimental sample stage shown in Fig.1 to reduce the distance between the objective lens and the actual sample. This resulted in a reduction of Cs from about 2 mm to 1 mm.

![Fig.1 Schematic images of the experimental sample stage](image)

**Results and Discussion**

Fig.2 shows a comparison of image resolution and amount of defocus using standard optics and optimized optics. We observed BF-STEM images of graphite lattice fringes in 3 positions. The working distance and Cs according to computation are 3.0 mm, 2.0 mm at sample position (a), 2.7 mm, 1.7 mm at (b), 1.8 mm, and 1.0 mm at (c). Sample position (a) is the “standard” STEM observation.
position and sample position (c) is upper limit at an accelerating voltage of 30kV setting. We calculated the amount of defocus difference ($\Delta f$) between the lattice and in focus conditions by noting the value of the lens’ digital analog converter (DAC) at each focus point. The amount of defocus difference is reduced as the sample position rises. The ensuing reduced Cs at sample position (c) (25 nm defocus) makes it possible to clearly observe the structural image and lattice image simultaneously.

Next we investigated the image resolution when changing the amount of defocus. We observed the BF-STEM images of single crystal Si<011> by a through-focus method at an accelerating voltage of 30 kV. Fig.3 shows the widest lattice image and narrowest single crystal Si<011> lattice image (with inset Fourier transform image) which could be observed by the SU9000. Amount of defocus ($\Delta f$) was based on using (a) as 0-reference. As $\Delta f$ was changed, the BF-STEM image at $\Delta f = -341$ nm provided the best result showing the Si (222) plane, which has a spacing of 0.157 nm. The inset Fourier transform image confirms the Si (222) reflection spot. The BF-STEM and Fourier transform images at $\Delta f = -223$ nm show the Si (220) plane, which has a spacing of 0.192 nm. This result emphasizes that high resolution imaging of less than a 200 pm is possible without aberration correctors, and creates new possibilities and applications for low energy STEM.

Fig.4 shows a high resolution BF-STEM image with its inset Fourier transform image, observed along the Si<110> zone axis at an accelerating voltage of 15 kV. The BF-STEM image shows the Si (111) plane, which has a spacing of 0.314 nm and Fourier transform image shows the Si (111) reflection spot, corresponding to 0.314 nm. These results reveal that we can observe lattice images at below 30 kV by using a high-performance in-lens CFE-SEM such as the SU9000. This method has the potential for high contrast visualization, less beam damage, and high resolution imaging on the latest semiconductor devices and carbon materials.
Conclusion
We reported a method for STEM image observation of lattice fringes in silicon, with a minimum lattice spacing of 0.157 nm, by using an in-lens type FE-SEM without aberration corrector at an accelerating voltage of 30 kV. We also observed lattice fringes in silicon (Si (111) lattice spacing of 0.314 nm) at an accelerating voltage of 15 kV. These results reveal the potential for high contrast visualization without loss of resolution for the latest semiconductor devices and any carbon-based materials with minimal radiation beam damage.

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