Observation of carrier distribution in GaAs semiconductors using phase-shifting electron holography

H Sasaki\textsuperscript{1,2}, K Yamamoto\textsuperscript{2} and T Hirayama\textsuperscript{2}
\textsuperscript{1} Yokohama R&D Lab, The Furukawa Electric Ltd., 2-4-3 Okano, Nishi-ku, Yokohama, 220-0073, Japan
\textsuperscript{2} Japan Fine Ceramics Center, 2-4-1 Mutsuno, Atsuta-ku, Nagoya, 456-8587 Japan

Abstract. Off-axis electron holography was used to map carrier distributions in GaAs semiconductors. After preparing cross-sectional transmission electron microscopy (TEM) specimens by a rotating-microprobe technique and a focused ion beam (FIB) system equipped with a low-voltage Ar ion beam for removing FIB damaged layers, we observed the different carrier concentration in GaAs specimens and evaluated the carrier diffusion at the interface between material with different carrier concentrations.

1. Introduction
The measurement of carrier distributions in semiconductor devices is very important in the development of new devices and for the analysis of device failure. The development of a technique for measuring the carrier distribution across a p-n junction in silicon has resulted in electron holography being widely used to measure the carrier distributions in silicon semiconductors [1–3]. This is generally done using transmission electron microscope (TEM) specimens prepared using focused ion beam (FIB) milling, which enables specific parts to be cut from a bulk sample. A high-voltage Ga ion beam, however, damages the surface layers of TEM specimens of compound semiconductors and thereby complicates the phase distributions measured by electron holography [4]. In this study we therefore used a low-voltage Ar ion beam in a FIB system to prepare GaAs specimens and used off-axis phase-shifting electron holography to map the GaAs carrier distribution with high spatial resolution and high phase-measurement sensitivity.

2. Experimental Details

2.1. GaAs sample
We examined p-n thin films grown on a GaAs substrate by MOCVD. A structure of a TEM specimen is shown in Fig. 1. Silicon and zinc were used as n-type and p-type dopants, and the samples were designed for testing the sensitivity of electron holography to different carrier concentrations.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure1.png}
\caption{Structure of GaAs TEM specimen with p-type and n-type regions.}
\end{figure}
2.2. **FIB preparation**

The procedure for preparing TEM specimens is shown in Fig. 2. The specimen was prepared using a SIINT SMI3050TB (Triple Beam) FIB system, which combined a Ga ion beam, a scanning electron microscope (SEM) for process monitoring, and an Ar ion beam to remove the FIB damaged layers [5]. We used a rotating microprobe so that a TEM specimen whose thickness is uniform along the x-direction shown in Fig. 1 could be prepared by irradiating it with Ga ions from the y-direction. First, a carbon layer was deposited on the surface of the sample to protect its surface from the Ga ion beam. As shown in Fig. 2(a), a small cross-sectional specimen with p-n junction was lifted out from the GaAs bulk sample using a microprobe. Figure 2(b) shows a scanning ion microscope (SIM) image of small specimen being held by the microprobe. As shown in Fig 2 (c), the microprobe was rotated by 98 degrees which resulted in changing the side surface to the top surface due to the configuration of microprobe in the FIB system. It could therefore be FIB-milled along the y-direction. Figures 2(d) and 2(e) show how the specimen was fixed to the thin Si grid by carbon deposition. The specimen suitable for TEM observation was thinned to 300 nm in the FIB system. Finally, the FIB damaged layer was removed by milling the sample surface for 5 minutes with a 1.0-kV Ar ion beam in the FIB system. Figure 2(f) shows a SEM image of a TEM specimen after Ga ion and Ar ion milling.

![Figure 2. Preparation TEM specimens.](image)

**Figure 2.** Preparation TEM specimens. a) SEM image of lifted TEM specimen. b) SIM images of specimen held by microprobe, c) microprobe and specimen after rotation, d) specimen close to Si grid. SEM images of e) specimen fixed to Si grid by carbon deposition, f) TEM specimen after Ga ion and Ar ion milling.

2.3. **Phase reconstruction by phase-shifting method**

Phase-shifting electron holography [6] was used to reconstruct the phase images because it produces a phase map that has higher spatial resolution and phase-measurement sensitivity than a phase map produced by the Fourier transformation method. In the conventional Fourier transformation method, the spatial resolution is determined by the radius of the low-pass filter used to get one of the sidebands and is consequently about three times the fringe spacing. The spatial resolution of an image reconstructed using the phase-shifting method, in contrast, is determined by the pixel size of the electron detector at a low to medium magnification. The spatial resolution in our experiment was approximately 2 nm, and the sensitivity of the phase measurement was estimated to be approximately 0.02 rad [6].

Electron holographic observations were made with a JEOL-3100F TEM operating at 300 kV, and equipped with a Schottky field-emission electron gun and an electron bi-prism. Digital holograms with 1024×1024 pixels were recorded using a GATAN 794 slow-scan CCD camera system. In this experiment, 13 holograms with different initial phases were used for phase reconstruction.
3. Results and Discussions

Figure 3 shows the reconstructed phase image. The p- and n-type regions are clearly seen as areas of dark and bright contrast, and some differences of changing dopant concentrations can also be seen.

The averaged phase profiles across the p-n junction were obtained from the phase image. The line profile of p-type regions is plotted in Fig. 4(a), and the corresponding profile of Zn dopant concentrations measured by secondary ion mass spectrometry (SIMS) is shown in Fig. 4(b). The interface between the $1 \times 10^{19}$ cm$^{-3}$ region and $1 \times 10^{18}$ cm$^{-3}$ regions is clearly seen in both profiles, but that between the $1 \times 10^{17}$ cm$^{-3}$ and $1 \times 10^{16}$ cm$^{-3}$ regions is not evident, because charges are trapped on specimen surfaces and the low-dopant-concentration regions are almost depleted in TEM specimen [7].

The averaged phase profile of n-type areas is shown Fig. 4(c), and the corresponding profile of Si dopant concentrations is shown in Fig. 4(d). In contrast to what is seen in the profiles for the p-type areas, the $1 \times 10^{19}$ cm$^{-3}$ and $1 \times 10^{18}$ cm$^{-3}$ regions are not clearly distinguished in the phase profile. This is attributed to dopant diffusion at this interface (indicated in the SIMS profile). In other words, carrier diffusion at the interface between $1 \times 10^{19}$ cm$^{-3}$ and $1 \times 10^{18}$ cm$^{-3}$ regions can be evaluated by these phase profiles. The contrast is almost the same in the regions with dopant concentrations below $1 \times 10^{17}$ cm$^{-3}$, and this is also due to carrier depletion in thin TEM specimens.

![Figure 3. Phase image of GaAs specimen reconstructed by phase-shifting method.](image)

**Figure 4.** a) Line profile of phase image in p-type region. b) Zn SIMS profile. c) Line profile of phase image in n-type region. d) Si SIMS profile.
4. Conclusion
We prepared TEM specimens by a FIB system equipped with a rotating microprobe and a low-voltage Ar ion beam that makes it easy to remove FIB damaged layers on the specimen surface. We clearly observed the p-n junction and the difference in dopant concentrations between $1.0 \times 10^{19} \text{ cm}^{-3}$ and $1.0 \times 10^{18} \text{ cm}^{-3}$ regions and could also evaluate the carrier diffusion at the interfaces between regions with different carrier concentrations. This study indicates that these techniques will be highly useful for analyzing compound semiconductor devices.

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