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# Minimum detection limit and spatial resolution of thin-sample field-emission electron probe microanalysis

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ABSTRACT

The minimum detection limit and spatial resolution for a thinned semiconductor sample were determined by electron probe microanalysis (EPMA) using a Schottky field emission (FE) electron gun and wavelength dispersive X-ray spectrometry. Comparison of the FE-EPMA results with those obtained using energy dispersive X-ray spectrometry in conjunction with scanning transmission electron microscopy, confirmed that FE-EPMA is largely superior in terms of detection sensitivity. Thin-sample FE-EPMA is demonstrated as a very effective method for high resolution, high sensitivity analysis in a laboratory environment because a high probe current and high signal-to-noise ratio can be achieved.



## 1. Introduction

Electron probe microanalysis (EPMA) together with wavelength dispersive X-ray spectrometry (WDX) is a very powerful method for analyzing the distribution of trace elements in materials with high precision [1–3]. In 2004, an EPMA system using a Schottky field emission (FE) electron gun was developed by Kimura et al. and is now commercially available through JEOL Ltd. [4–6]. The use of a FE electron gun allows superior spatial resolution compared to that available with a conventional W or $LaB_6$ electron gun, especially at lower acceleration voltages [4–6]. However, even the resolution of FE-EPMA, which is approximately 200 nm, does not meet recent demands for the microstructural analysis of materials and devices [7]. The spatial resolution is mainly determined by the larger of the diameter of the electron beam on the sample surface and the lateral width of the characteristic X-ray generation region in the sample [1]. In most cases of FE-EPMA, the X-ray generation region is much larger than the electron beam size [4,5]; therefore, high-resolution analysis is generally performed at a lower acceleration voltage. However, the resolution thus obtained is also approximately 200 nm, and the detectable elements are limited due to the critical voltage [1]. Another way to achieve high resolution analysis is to thin the sample and use a higher acceleration voltage [8,9]. The concept of spatial resolution improvement by sample thinning is schematically presented in Scheme 1. This scheme also shows how thinning may worsen the detection sensitivity of FE-EPMA as the sample volume is reduced. Sample thinning can lead to a resolution of several tens of nanometers, according to theoretical calculations, and overcome the critical voltage limitation because the analysis is performed at a higher acceleration voltage. Thus, greater improvement in resolution can be expected through sample thinning in the case of FE-EPMA than that for W– or $LaB_6$–EPMA because the diameter of the electron beam can be very small [4,5]. However, sample thinning for FE-EPMA has seldom been employed in the laboratory. It is assumed that this can be attributed to the generally accepted view that sample thinning significantly reduces the characteristic X-ray signals and worsens the detection sensitivity. However, to our knowledge, there have been no systematic investigations of the detection limit and spatial resolution of thin-sample FE-EPMA. Therefore, in the present study, the minimum detection limit and the spatial resolution of thin-sample FE-EPMA is determined using a semiconductor sample. By comparing the FE-EPMA results for a thinned sample with those obtained using energy dispersive X-ray spectrometry on a scanning transmission electron microscope (STEM-EDX), the superiority of FE-EPMA is confirmed in terms of the detection sensitivity. We demonstrate that thin-sample FE-EPMA is a very effective method for high resolution, high sensitivity analysis because a high probe current and high signal-to-noise (SN) ratio can be achieved.

## 2. Materials and methods

### 2.1. Sample preparation and mounting method

Gallium indium phosphide (GaInP) epitaxial films on gallium arsenide (GaAs) substrates were used for the present experiments.

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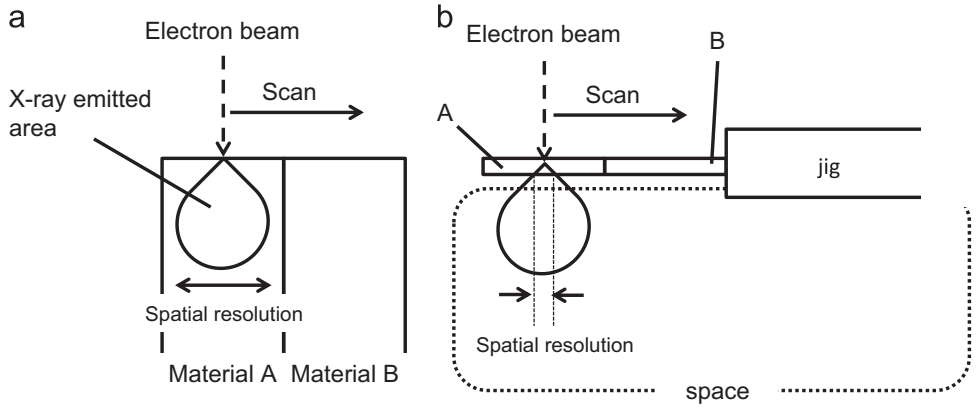
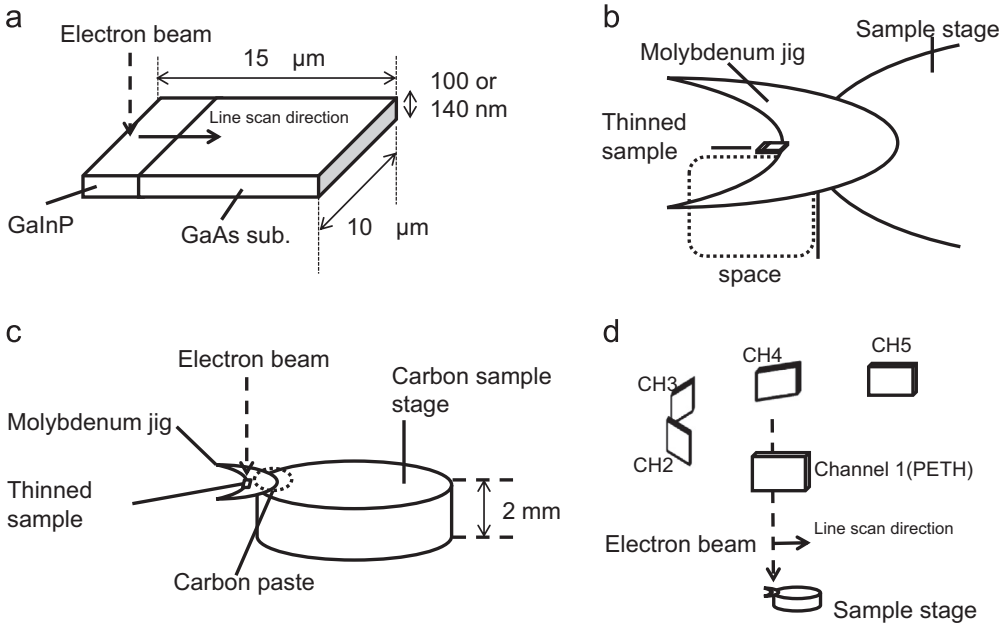



**Scheme 1.** Concept of spatial resolution improvement in FE-EPMA by sample thinning. (a) Bulk-sample FE-EPMA and (b) thin-sample FE-EPMA. From (a) and (b), it is evident that sample thinning improves the spatial resolution and the detection sensitivity is worsened due to the reduction in sample volume.




**Scheme 2.** (a) Schematic diagram of a thinned sample prepared using a focused ion beam. GaInP/GaAs samples were thinned perpendicular to the layer structure. In the present experiments, all line scans using FE-EPMA and STEM-EDX were conducted across the GaInP/GaAs interface as indicated. (b, c) Mounting method for a thinned sample. The sample is horizontally glued to the molybdenum jig as shown in (b). For STEM-EDX analysis, the jig is attached to a commercial sample holder (Hitachi HD-2700). For FE-EPMA, the jig is fixed onto a carbon sampling stage using carbon paste, as shown in (c). The molybdenum jig is set to be a certain distance below the sample to reduce the background signal from the sampling stage, as depicted in (b). In all the line scans, the scan direction is set parallel to the PETH analyzing crystal used to exclude edge effects, as indicated in (d).

The Ga/In atomic ratio in the GaInP layer was confirmed to be approximately 1 using the ZAF (atomic, absorption, and fluorescence) correction method [10] for FE-EPMA. Scheme 2(a) shows two GaInP/GaAs samples were thinned perpendicular to the layer structure to thicknesses of approximately 100 and 140 nm using a gallium focused ion beam, which was confirmed by secondary electron images of sample cross-sections. The thinned samples were horizontally attached to the edge of a molybdenum jig (Scheme 2(b)). The two thinned samples were then analyzed using FE-EPMA and STEM-EDX. These samples are denoted as Sample A (100 nm thick), sample B (140 nm). The cleaved cross-section of a bulk GaInP/GaAs sample was analyzed as a reference and is referred to as the Bulk sample.

For STEM-EDX analysis, the molybdenum jig was attached to the sample holder of a commercial STEM. For FE-EPMA, the jig was fixed on a carbon sampling stage using carbon paste (Scheme 2(c)). The molybdenum jig was set at a certain distance below the sample to reduce the background signal from the sampling stage, as shown in Scheme 2(b). To avoid charge-up, the sample was coated with carbon.

### 2.2. FE-EPMA

Indium L$\alpha$ X-ray line profiles and energy dispersions around In L$\alpha$ were measured using a FE-EPMA system (JXA-8530F, JEOL Ltd.) with wavelength dispersive X-ray spectrometry (WDX). In all the measurements, the working distance was approximately 11.0 mm and the takeoff angle was 40°. The radius of the Rowland circle was 100 mm (H-type spectrometers in JEOL microprobes) and the used analyzing crystal was PETH. X-rays were introduced through a 0.5 mm slit and then detected with a Xenon-sealed proportional counter. The measurement conditions during FE-EPMA with a