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Lateral Resolution of EDX Analysis with Ultra Low Acceleration Voltage SEM

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The spectral imagings were performed in order to examine the lateral resolution of the EDX analysis which is attached in an ultra low acceleration voltage SEM. The spectral imagings were performed for the cross section of the Ni coating on Al, which was cut using microtome, and the cross section of GaAs/AlAs super-lattice, which was cleavaged, using a ultra-low-acceleration-voltage SEM and EDX. The line scan of Al and Ni across the interface of the Ni coating and the Al substrate shows that the lateral resolution measured under the acceleration voltage of 7 kV is less than 100 nm. All of four layers with a thickness of about 25 nm of the GaAs/AlAs super-lattice are apparently observed in the spectral mapping of Ga and Al, which is measured under the acceleration voltage of 3 kV. This high lateral resolution of the EDX analysis can be resulted from the followings: (1) The area of the characteristic x-rays generation becomes small for low acceleration voltage of the primary electron. (2) The electron beam with the small diameter and the high current is obtained even under the ultra low acceleration voltage using an optimum electron optics.

INTRODUCTION

It is important to determine lateral distribution and/or depth distribution of elements in materials. When higher lateral resolution is required, TEM-EDX or EELS analysis of cross-section of sample, AES or XPS, SIMS depth profiling are commonly used. Although TEM techniques have high lateral resolution of less than several nm, sample preparation is sometimes very difficult. In the case of the surface analysis techniques such as AES, it is very difficult to analyze buried structure. When the medium lateral resolution of one micron order is required, EPMA (Electron Probe microanalysis). or SEM (Scanning Electron Microscope)-EDX (Energy dispersive X-ray spectroscopy) is commonly used.

Moreover, mappings using EDX which is attached to SEM are widely used for daily analyses, since the acquisition time is shorter than that using EPMA. Recently, it is reported that the lateral resolution using FE-EPMA is about 100 nm with decreasing the acceleration voltage and that the value is beyond the typically believed value (micron order) for the analysis under high acceleration voltage [1].

Ultra low voltage SEM with a new optics and Schottky-type FE gun have advantages on high resolution and high current density even in the low voltage of 100 V. These results suggests that lateral resolution using ultra low voltage SEM-EDX may be smaller than that as supposed. Additionally, the EDX spectral imaging, which is obtained as the result of the comparing with each spectrum measured in the whole scanned area and the standard spectra of elements, gives the quantitative distribution of elements, even if the energy of an element is close to that of another element.

Thus, the spectral imaging of Ni coated on Al and GaAs-AlAs superlattice were performed in order to examine the lateral resolution of the EDX analysis which is attached in an ultra low acceleration voltage SEM.

Fig. 1 SEM image and spectral mapping of Ni and Al in Ni-P coating on Al. Acceleration voltage was 7 kV. Line scan was performed at line displayed in SEM image. At arrow displayed in SEM image, line analysis is performed.

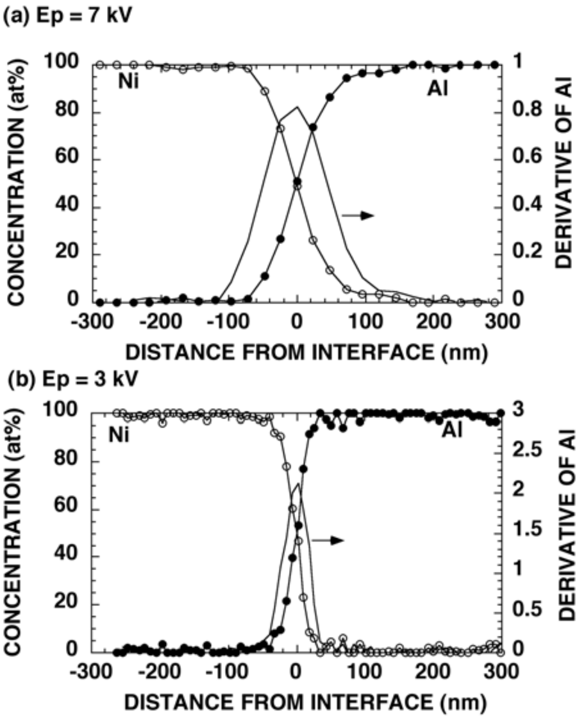

Fig. 2 Line scan of Ni and Al of Ni-P coating on Al, which were performed at line displayed in SEM image. Acceleration voltage was 7 kV (a) and 3 kV (b).

EXPERIMENTAL

A Ni-P coating on Al and a GaAs/AlAs super-lattice standard sample (NIMC CRM5201-a) were used for the EDX analyses. Cross section of the both specimen were prepared. The cross section of the Ni-P coating on Al was cut using microtome. The cross section of GaAs/AlAs superlattice was cleaved.

The specimens were analyzed using EDX (VANTAGE, Thermo Electron) which is attached in an ultra low acceleration voltage SEM (ULTRA55, ZEISS). Spectral imaging of Ni coating on Al were performed using Al and Ni with the acceleration voltage of 7 and 3 kV. For the GaAs/AlAs superlattice, Ga, As, and Al were analyzed with the acceleration voltage of 3 kV. Sample drift was corrected by moving to original position, after comparing with the present SEM image and the stored referencing image. As the referencing image which specifies the original position, accidentally introduced scars or particles on the sample near the analysis area were chosen.

RESULTS AND DISCUSSION

EDX line scan for Ni coating on Al

Figure 1 shows the results of spectral mapping for Ni and Al in the Ni-P coating on Al. The acceleration voltage was 7 kV. Figure 2 (a) shows line scan of Al and Ni across the interface between the Ni coating and the Al substrate. The atomic concentration is displayed as the normalized content by Ni and Al. The derivative of Al concentration versus distance is also displayed.

Each profile of Ni and Al shows a sharp interface. The lateral resolution, which is defined as the width from 16% to 84% of the line scan, is 82 nm. The distribution of Al observed in Ni coating layer is very close to that of Ni observed in Al substrate within the area near the interface, in which the content of Al is 5% to 95%. Additionally, the tailing of Ni in Al substrate is observed compared with Al in Ni coating layer.

The same Ni-P coating on Al was analyzed under the acceleration voltage of 3 kV. The line scan of Ni and Al is showed in Fig.2(b). In this case, the lateral resolution, which is defined as the width from 16% to 84% of the line scan, became 31 nm. The result shows that the lateral resolution for EDX analysis can be improved by the lowering the acceleration voltage. The lateral resolution clearly increases with decreasing the acceleration voltage.

Spectral mapping of GaAs-AlAs superlattice

AES depth profile of the GaAs/AlAs superlattice on GaAs is shown in Fig. 3. The depth profile was measured using Ar^+ ion sputtering with the

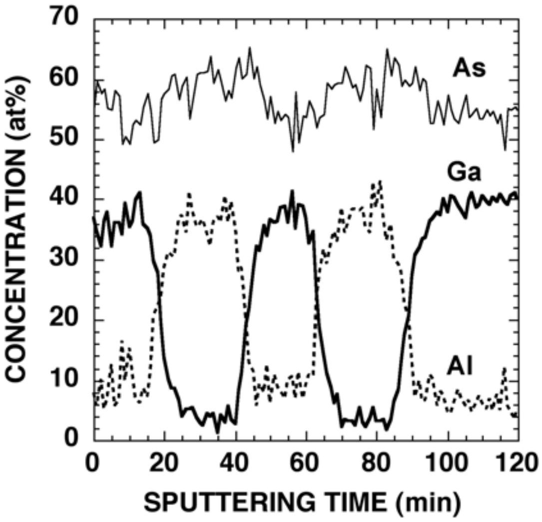

Fig. 3 AES depth profile of GaAs/AlAs superlattice on GaAs. Acceleration voltage of Ar^+ ion for depth profile was 2 kV.

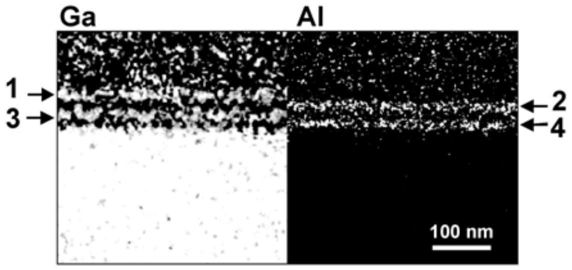

Fig. 4 Spectral mapping of Ga and Al for GaAs/AlAs superlattice on GaAs. Acceleration voltage of the primary electron was 3 kV.

acceleration voltage of 2 kV without the sample rotation. Four layers of two GaAs layers and two AlAs layers were observed.

Figure 4 shows the result of the spectral mapping of Ga and Al for the GaAs/AlAs superlattice on GaAs. The acceleration voltage of the primary electron was 3 kV. All of two cycles of GaAs layers and AlAs layers about 25 nm thickness, respectively, are clearly observed in this spectral mapping. This result means that lateral resolution is improved to about 25 nm with decreasing the acceleration voltage. The result means that the structure having the size of less than 25 nm can be analyzed using EDX under the acceleration voltage of less than 3 kV.

Lateral resolution of EDX analysis

These results of the EDX analysis for the Ni coating on Al and GaAs/AlAs superlattice on GaAs show that the lateral resolution for the analysis of characteristic x-rays measured using FE source is rather small. Castaing described that the lateral resolution for the analysis using characteristic x-rays is the sum of the diameter of the primary electron and the production range of the characteristic x-rays [2].

Figure 5 shows the range of the characteristic x-rays for Al and Ni. The plots show the sum of the diameter of the primary electron and the x-ray generation area (line: x-ray generation area given by Castaing [2], dotted line: six times effective attenuation length (EAL), which is the inelastic mean free path [3] corrected using elastic mean free path [4]). The circle is experimentally obtained value.

The range for the x-rays production area defined by Castaing is 729 and 136 nm for Al K line in Al for 7 kV and 3 kV acceleration, 293 and 66 nm for Ni L line in Ni for 7 kV and 3 kV, respectively. The range calculated using Castaing’s equation is larger than that the obtained lateral resolution (82 nm for 7 kV, and 31 nm for 3 kV). The result suggest that the real production area for the characteristic x-rays is much smaller than that derived by Castaing, which they

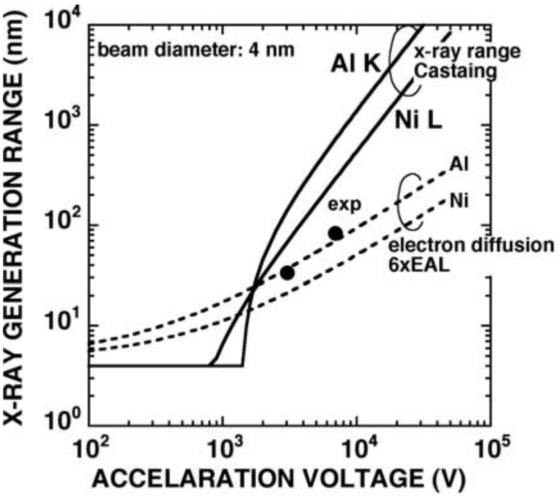

Fig. 5 Range of characteristic x-rays generation for Al and Ni. Plots show sum of beam diameter and the x-ray generation area (straight line: given by Castaing [2], dotted line: six times of effective attenuation length (inelastic mean free path [4] corrected using elastic mean free path [5]. Circle is experimentally obtained lateral resolution.)

showed that their equation is good for the range of 10 – 30 kV. Until the energy is fully lost, the electrons travel in the material for a length which is several times the effective attenuation length, and the characteristic x-rays are emitted. The length is much decreased with the lowering acceleration voltage.

Moreover, the improvement of the lateral resolution with the decreasing acceleration voltage means that the diameter of the primary electron beam is quite small. The diameter of the primary electrons for this EDX analysis was about 3 nm for 7 kV, and 4 nm for 3 kV. The small diameter of primary electron beam and the sharp distribution even under the ultra low acceleration voltage are obtained by an optimum electron optics employed in the present FE-SEM [5, 6].

Moreover the tailing of Ni in the Al substrate is observed within about 200 nm from the interface. The attenuation depth of electron in Al is larger than that in Ni L, and the range of production of Al K line (excitation energy: 1559 eV) is larger than that of Ni L (excitation energy: 853 eV). The electron, which is irradiated at Al substrate near interface, can travel to the Ni layer beyond the interface, and it can excite Ni and Ni L characteristic x-rays can be emitted. Ni can be detected even when the primary electron irradiates the Al substrate. This tailing of Ni in the Al substrate is due to the emission of Ni L line excited by the scattered electron in Ni-P coating to the Al substrate. Additionally, the mean free paths of Al K characteristic x-rays (910 nm in Al, 870 nm in Ni) as the inverse of the linear total absorption coefficient of Al K characteristic x-rays are larger than the width for this tailing of Ni (about 200 nm). Ni L fluorescent x-rays excited by Al K characteristic x-rays, which are generated in the Al substrate and travel to the Ni-P coating layer, will not affect this tailing.

Thus, both the small size of the primary electron beam, and the small characteristic x-rays generation area give higher lateral resolution for the ultra low voltage SEM-EDX analysis than that widely believed. The lateral resolution for 7 kV acceleration becomes one third of that for 20 kV which is commonly used in conventional EPMA or FE-AES, and the resolution for 3 kV is one fifth of that at 20 kV.

CONCLUSION

The spectral imaging were performed in order to examine the lateral resolution of the EDX analysis. Spectral imaging were performed for the cross section of the Ni coating on Al, which was cut using microtome, and the cross section of GaAs/AlAs super-lattice, which was cleaved, using a ultra-low-acceleration-voltage SEM and EDX. The line scan of Al and Ni across the interface of the Ni coating and the Al substrate shows that the lateral resolution measured under the acceleration voltage of 7 kV is less than 100 nm. All of four layers with a are apparently observed in the spectral mapping of Ga and Al, which is measured under the acceleration voltage of 3 kV. This high lateral resolution of the EDX analysis can be resulted from the followings: (1) The area of the characteristic x-rays generation becomes small with the lowering acceleration voltage of the primary electron. (2) The electron beam with the small diameter and the high current is obtained even under the ultra low acceleration voltage by an optimum electron optics.

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