Original Paper

Lateral Resolution of EDX Analysis with Low Acceleration Voltage SEM

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Spectral imaging mappings for a cross section of a Cr/W superlattice (Cr/ W/….W/Cr/ TaO_x layers on a Si wafer) were performed in order to examine the lateral resolution of the SEM-EDX analysis. A cross section of the Cr/W superlattice was analyzed using EDX which is attached to an Ultra Low Voltage SEM (ULV SEM) using an acceleration voltage of 2.7 -30 kV. Both the W layers with a thickness of 12 nm and the Cr layers with a thickness of 35 nm are clearly observed in the spectral imaging mappings when measured under the acceleration voltage of less than 3 kV. These results show that the lateral resolution for the analysis of characteristic X-rays using EDX is about 30 nm, and this value is smaller than the widely believed value. This high lateral resolution of the EDX analysis results from the following: (1) the area of the characteristic X-rays generation becomes small with the lowering acceleration voltage of the primary electron, (2) the electron beam maintains a small diameter and the high current even under the ultra low acceleration voltage by the optimum electron optics employed in the ULV-SEM.

1. Introduction

It is important to determine the lateral distribution and/or the depth distribution of elements in materials. When high lateral resolution is required, the TEM-EDX or EELS analysis of the cross-section of the sample, or the depth profiling of AES, XPS, or SIMS are commonly used. New TEM microscopes with aberration correction give high lateral resolution of about 1 nm [1], but the sample preparation for TEM is very difficult. In the case of the surface analysis techniques such as AES, it is very difficult to analyze a buried structure. When the medium lateral resolution of one micron is required, EPMA (Electron Probe microanalysis), or SEM (Scanning Electron Microscope)-EDX (Energy dispersive X-ray spectrometry) is commonly used.

It is reported that the lateral resolution using Ultra Low acceleration Voltage SEM (ULV-SEM)-EDX with new optics and a Schottky-type FE gun is 31 nm with a low acceleration voltage and that the value is beyond the typically believed value (on the order of a micron) for the analysis under high acceleration voltage (typically 10-30 kV) [2]. Further, it is reported that the lateral resolution using FE-EPMA is about 100 nm with low acceleration voltage and that the value is beyond the typically believed value (on the order of a micron) for the analysis under high acceleration voltage [3].

Thus, the spectral imaging mappings of the Cr/W superlattice were performed in order to examine the lateral resolution of the EDX analysis which is attached in an ultra low acceleration voltage SEM.

2. Experimental

A Cr/W superlattice, which was prepared using the sputter deposition, was used for the EDX analyses. The thickness of W was 12 nm and that of Cr was 35 nm, respectively. Cr and W layers were prepared on top of a 350 nm Ta oxide film coated Si wafer. The W/Cr superlattice was cleaved for the analysis of the cross section.

The specimens were analyzed using EDX (NSS 300, Thermo Fisher Scientific) which is attached in an ULV-SEM (ULTRA55, Carl Zeiss). The spectral imaging mappings of the Cr/W superlattice were performed with the acceleration voltages of 2.7, 3.0, 5.0, 10, and 30 kV, the current of about 2 nA, and the diameter of the primary beam of about 4nm. The pixel numbers for the imaging were 256 x 192, and the size of one pixel was 5 nm x 5 nm. The measurement time was about 6 hours for 2.7 and 3 kV, and about 1 hours for 5, 10, and 30 kV. An EDX spectrum was stored at every pixel in the measured imaging data. Sample drift was corrected by moving the beam shift after comparing the current observed SEM image and the stored initial image. Cr L, W M, Ta M, O K, Si K characteristic X-rays were analyzed when the acceleration voltage was less than 10 kV. Cr K, W L, Ta L, O K, Si K characteristic X-ray were analyzed when the acceleration voltage was more than 10 kV. The intensity of each line was displayed as the net intensities, which is calculated using the least-linear-square fitting for the measured spectrum using the stored standard spectra after the background is subtracted [4]. This fitting can separate elemental contribution to the peaks in the spectrum when the elemental photon energies are close. In this article, we defined the spectral mapping as the measured mapping and the whole of the above procedure, and the spectral imaging mappings as the calculated mapping from the measured one. The line profiles of the intensities of the characteristic X-rays were calculated from the spectral imaging mapping data.

3. Results and discussion

3.1 Spectral mapping of Cr/W superlattice

Figure 1 shows the spectral imaging mapping of Cr L, W M, Ta M, O K, and Si K for the Cr/W superlattice on Ta oxide coated Si wafer. The acceleration voltage of the primary electron was 2.7 kV. A SEM image is also displayed.

The SEM image shows that brighter thin layers and darker thin layers on a relatively bright thick layer. The brighter thin layers correspond to W layers, the darker thin layers correspond to Cr layers, and the thick layer is Ta oxide. The SEM image shows that the thickness of W layer is thinner than that of Cr layer. Both of the W layers with a thickness of 12nm, and the Cr layers with a thickness of 35nm are clearly observed in the maps. The profile of Cr L, Ta M, O K, and Si K is sharp at the interfaces between Cr and Ta oxide, and the interface between Ta oxide and Si wafer.

Figure 2 shows the result of the spectral imaging mapping of Cr L, W M, Ta M, O K, and Si K for the Cr/W superlattice, which were measured with the acceleration voltage of 10 kV. Each Cr and W layer is not separated. Both the interface between Cr and Ta oxide, and the interface between Ta oxide and Si wafer is broader than that seen in fig. 1.

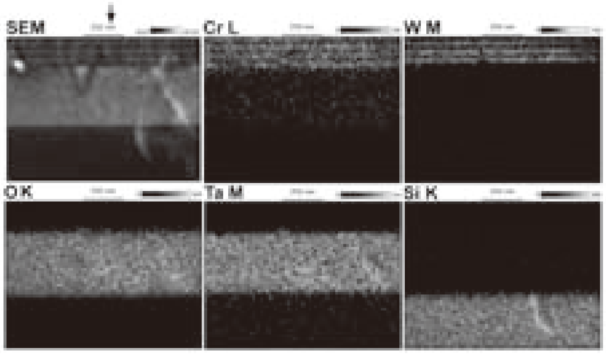

Fig. 1 SEM image and spectral imaging mapping of Cr/W superlattice on Ta oxide formed Si wafer. Acceleration voltage is 2.7 kV. Position of line profile in Fig. 3(a) is displayed by arrow.

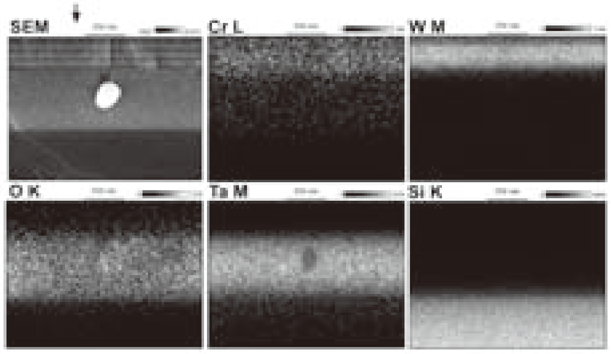

Fig. 2 SEM and spectral imaging mapping of Cr/W superlattice on Ta oxide formed Si wafer. Acceleration voltage is 10 kV. Position of line profile in Fig. 3(b) is displayed by arrow.

3.2 Lateral resolution of EDX analysis

The line profiles of Cr L, W M, Ta M, O K, and Si K for the Cr/W superlattice with the acceleration voltage of 2.7 kV and 10kV are shown in Fig. 3 (a) and (b). The intensities of Cr L and W M shows a complementary distribution in the profile of 2.7 kV. It explains that Cr and W are formed layer by layer on the Si wafer. Moreover, the decrease of intensity of the O K characteristic X-rays at the interface between Ta oxide and Si wafer occurs at a slightly deeper position than that of the Ta M characteristic X-rays. This distribution of the O K intensity may mean that there is a surface Si oxide on the Si wafer.

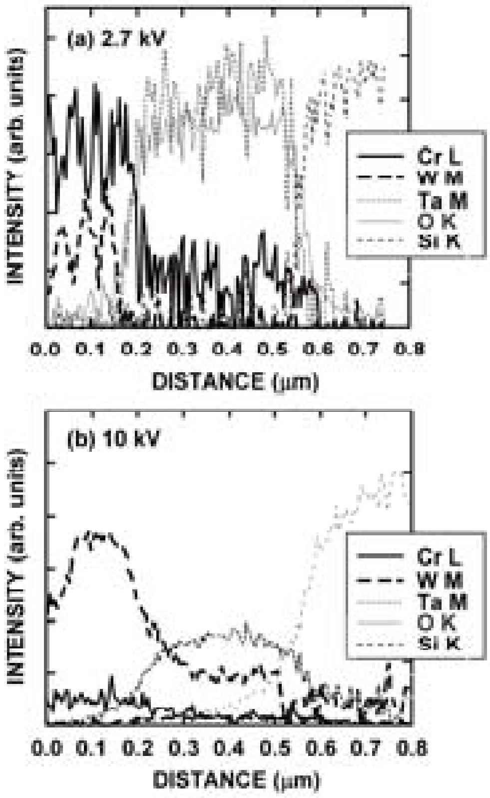

Fig. 3 Line profile of Cr/W superlattice on Ta oxide formed Si wafer. Acceleration voltage is (a) 2.7 and (b) 10 kV. Positions of line profiles in Fig. 1 and 2 are displayed by arrow.

On the other hand, the oscillation, which is observed for 2.7kV, is almost invisible in the results of 10kV. The widths of the interface between Cr and Ta oxide, and that between Ta oxide and Si for 10kV are clearly broader than those of 2.7kV.

Even in the case of 2.7 kV, the plateau of the intensity in the Cr layers and W layers was not obtained, and the minimum of the intensities are higher than that of background level. This means that the lateral resolution is larger than that of the thickness of the Cr and W layers. Therefore, the lateral resolution is expressed as the ratio of the difference in the intensities of the top and the bottom for Cr L and W M against the intensity of the top, instead of the width at the interface. The intensity ratio of this peak height is shown in Fig.4. The lateral resolution is also estimated using the width of the line profile for the Ta M characteristic X-rays at the interface between Ta oxide and Si wafer. The width is shown in the fig. 5. The resolution of Ta M with the acceleration voltage of 2.7 kV is about 30 nm. The ratio of the peak height for each Cr L and W M increases as a function of the decrease of the acceleration voltage, and the width at the interface of Ta M decreases with the acceleration voltage. Both results prove that the lateral resolution of the mapping and line analysis using characteristic X-rays increases with the lowering of the acceleration voltage.

Castaing showed that the lateral resolution for the analysis using characteristic X-rays is expressed as the sum of the diameter of the primary electron beam and the production range of the characteristic X-rays [5]. Figure 6 shows the range of the characteristic X-rays for Cr L in metallic Chromium, W M in metallic tungsten, and Ta M in Ta_2O_5. The plots show the sum of the diameter of the primary electron beam (4 nm) and the X-ray production range (line: X-ray generation area [6] modified the equation by Castaing [5]). The circle is experimentally obtained 20-80% width at the interface TaOx layer and Si wafer.

The production range calculated using Castaing is larger than that the obtained lateral resolution. The results suggest that the real production area for the characteristic X-rays is much smaller than that derived using the Castaig's equation, which is good for the range of 10 – 30 kV.

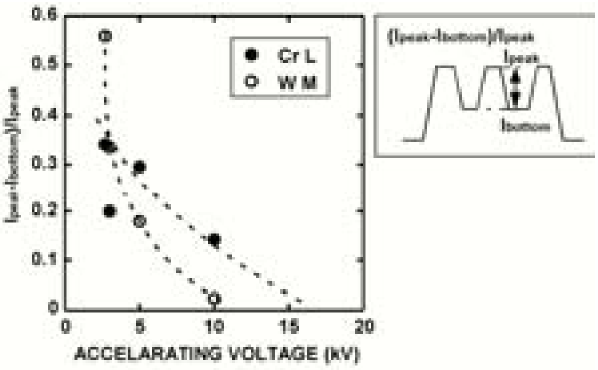

Fig. 4 Ratio of difference in intensities of top and bottom for Cr L and W M against the intensity of the top as a function of acceleration voltage.

On the other hand, the electrons travel in the material for a length which is several times the effective attenuation length, which is the inelastic mean free path [7] corrected using elastic mean free path [8]. The characteristic X-rays are emitted during the travel. So it is expected that the generation area of the characteristic X-rays is close to the effective attenuation length. The

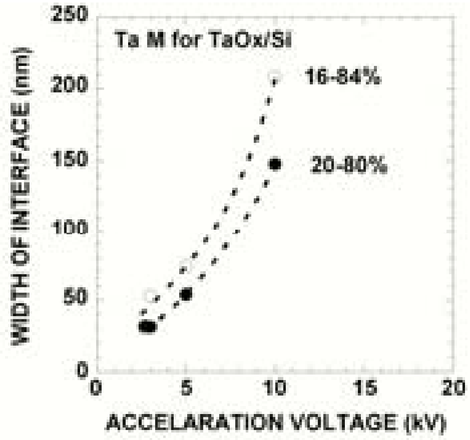

Fig. 5 Width of line profile for Ta M characteristic X-rays at the interface between Ta oxide and Si wafer.

six times effective attenuation length (EAL) is displayed in the dotted line of Fig.6. This value is close to the experimentally obtained width. The several times of effective attenuation length may be a rough guide to estimate the lateral resolution of EDX analysis with the low 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 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 [9,10].

Lastly, the lateral resolution may be affected by the sample drift. Although the sample drift is corrected in this experiment, the broadening of about 10 nm may be occurred.

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. SEM-EDX with low acceleration voltage has a potential to characterize the nano-scale structures with ten nanometer size.

4. Conclusion

The spectral imaging mappings were performed in order to examine the lateral resolution of the EDX analysis. The spectral imaging mappings were performed for the cleaved cross section of a Cr/W superlattice using an ultra-low-acceleration-voltage SEM and EDX. Both the

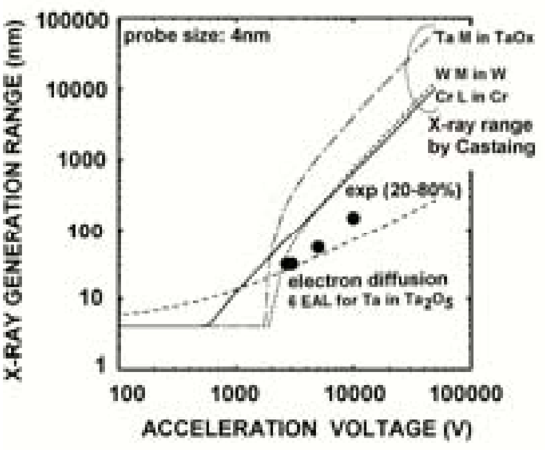

Fig. 6 Range of characteristic X-rays generation for Cr, W, and Ta. Plots show sum of beam diameter and the X-ray production range (straight line: given by Castaing [5], dotted line: six times of effective attenuation length of TaM in Ta_2O_5 (inelastic mean free path [6] corrected using elastic mean free path [7]), Circle: experimentally obtained lateral resolution at interface TaOx layer and Si wafer).

W layers with a thickness of 12 nm and the Cr layers with a thickness of 35 nm are clearly observed in the spectral imaging mappings when measured using the acceleration voltage of less than 3 kV. On the other hand, the separation of the layers was not clear in the mappings using an acceleration voltage of more than 10 kV.

These results show that the lateral resolution for the analysis of characteristic X-rays using EDX is smaller than that widely believed value, the micron meter scale. 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 a small diameter and high current is obtained even under the ultra low acceleration voltage by an optimum electron optics employed in the ULV-SEM.

5. References

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