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Comparison of Detection Limits for Elemental Mapping by EF-TEM and STEM-XEDS

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The elemental mapping approach can be the best way to analyze nano-scale features in materials such as fine precipitates and interfaces/boundaries, since two dimensional fluctuation in composition around such small features, which may be easily missed by point or line-scan analyses, can be revealed in images of elemental distributions. Currently, such elemental distributions can be obtained by a transmission electron microscope equipped with an energy filter (EF-TEM), or a scanning transmission electron microscope (STEM) with an X-ray energy dispersive spectrometer (XEDS) and/or an electron energy-loss spectrometer (EELS). Although the spatial resolution of the elemental mapping can reach the sub-nanometer level in EF-TEM and STEM-EELS mapping, and few nanometers in STEM-XEDS mapping, the analytical sensitivity becomes proportionately worse. This is true especially in the STEM since the acquisition time at a single pixel (dwell time) is much shorter than for point or line analyses. So, the intensity fluctuation in the elemental maps may not reflect the compositional fluctuations but could be lost within the noise level. In this study, comparative elemental maps of dilute amounts of Mn in Cu have been taken by EF-TEM and STEM-XEDS, to analyze the detection sensitivities of elemental mapping with statistical accuracy.

To acquire STEM-XEDS elemental maps, a VG HB 603 STEM at Lehigh University has been used. Figure 1A shows an X-ray spectrum from a homogeneous, 50-nm-thick Cu-0.98wt%Mn thin specimen and the window settings for background subtraction. The maps of peak and background intensities have been gathered with 128x128 pixels for a dwell time of 0.2 s (total acquisition time: ~1 hr without dead time). The size of a single pixel is 1.25x1.25 nm^2 and this is smaller than the incident beam diameter (~1.6 nm). The EF-TEM elemental maps have been obtained in a JEM-2010FEF at Kyushu University equipped with an in-column omega-type energy filter. The EELS spectrum around the Mn $L_{2,3}$ edge is shown in Fig. 1B with window settings for 3-window background subtraction. Each map has been acquired for 200 s by a slow-scan CCD camera with 8x8 hardware binning of 1kx1k pixels. So, the acquired maps are 128x128 pixels with a single pixel dimension of 0.9x0.9 nm^2 (which closely matches that in the XEDS maps).

In an image with a relatively low signal intensity, one of the most important parameters to evaluate is the signal-to-noise ratio (SNR). The SNR for an image can be calculated as $(SNR)_{img}$ = (signal intensity) / (variance of the signal intensity)$^{1/2}$ [1]. Since homogeneous specimens have been used in this study, the variance of the elemental map is not due to the difference in composition. The $(SNR)_{img}$ values for the XEDS Mn Kα maps and EF-TEM Mn $L_{2,3}$ maps as a function of the specimen thickness are shown in Figs. 2A and B, respectively. The specimen thickness has been determined by the ζ-factor method [2] for the XEDS maps and by the log-ratio method [1] for EF-TEM maps. The error bars indicate the 99% confidence limit ($\pm 3\sigma$). The dashed line at SNR = 5 represents the Rose criterion below which the signal of the image cannot be distinguished from the noise [3,4]. So, the open symbols in Fig. 2 indicate satisfaction of the Rose criterion. In both the mapping methods, the $(SNR)_{img}$ from a single pixel (1x1) is well below the Rose criterion. The $(SNR)_{img}$ value can be improved by adding several pixels together (eg. 4x4 and 8x8 pixels) although the spatial resolution has then to be sacrificed. For XDES maps, the $(SNR)_{img}$ increases linearly as the specimen thickness increases since the signal intensities are higher from thicker regions. In contrast, the $(SNR)_{img}$ values from the EF-TEM maps are at a maximum around a specimen thickness of 30-50 nm. The decrease of $(SNR)_{img}$ from thicker regions is mainly because the background levels significantly increase and degrade the weak signal intensities. To discuss the detection limits for elemental mapping quantitatively, therefore, the effect of the background intensities should also be taken into account, in the same manner as the detection limits for XEDS [5] or for EELS spectra [3,6]. However, it was found that the Rose criterion can be more severe than the definition of the detection limit for spectra in the most conditions, especially for XEDS maps.

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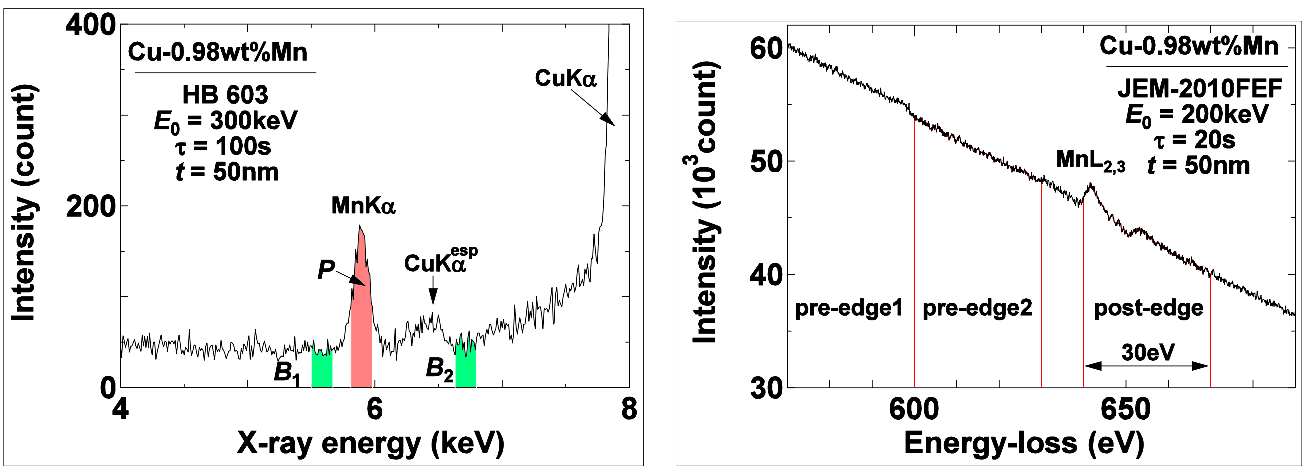
1A **1B**



2A **2B**

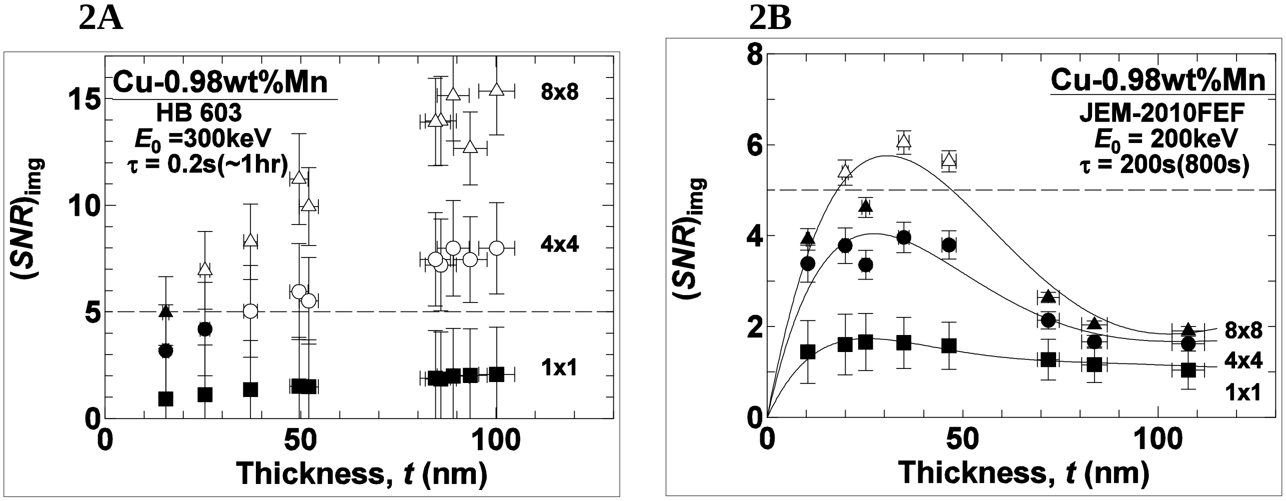


Fig.1 XEDS (A) and EELS (B) spectra from a 50-nm thick Cu-0.98wt%Mn thin film. Energy windows for background subtraction are also shown.

Fig.2 Signal-to-noise ratio plotted against the specimen thickness for STEM-XEDS maps (A) and EF-TEM maps (B). The error bars represent the 99% confidence level and open symbols indicate above the Rose criterion, SNR = 5 (dashed line).

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