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Monte Carlo simulation and theoretical calculation of SEM image intensity and its application in thickness measurement

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ABSTRACT

The intensity profiles of backscattered and secondary electrons from a pure Mg sample have shown a variation with sample thickness and acceleration voltage in the range of 5–30 kV, depending on the specimen holder used. The intensities of backscattered electron (BSE) and secondary electron (SE) signals increases with the sample thickness until saturation when using a scanning transmission electron microscopy (STEM) holder with a closed tube below the sample. However the SE signal increases to the maximum and then decreases with the sample thickness when using a transmission Kikuchi diffraction (TKD) holder with no shielding below the sample whereas the BSE signal again increases until saturation. The influence of the holder on the SE signals is caused by the fact that secondary electrons emitted from the bottom surface could be detected only when using the TKD holder but not the STEM holder. The experimental results obtained are consistent with the Monte Carlo simulation results. Application of the magnitude of the SE and BSE signals to measurement of sample thickness has been considered and the BSE image profile shows a reasonably good accuracy.

1. Introduction

STEM-in-SEM reveals the internal structure of thin foil samples with high contrast and resolution due to the low voltage and thin sample utilized, which increase the electron scattering cross-sections and reduce the interaction volume [1]. In the last few years, thin foil samples have also been widely used for diffraction study in SEMs. The so-called transmission Kikuchi diffraction (TKD) technique employs the traditional EBSD detectors but very thin samples [2]. Compared with bulk sample, it is more complicated for the SEM imaging of thin samples as the image contrast may vary with thickness, as well as composition and topography. So it is quite important to understand the expected contrast seen in both secondary electron (SE) and back-scattered electron (BSE) modes. A combination of Monte Carlo (MC) simulation and experiments helps to interpret the image contrast and how it varies with imaging conditions, such as sample thicknesses and voltages.

On the other hand, it is important to obtain accurately the thickness of thin samples when the density of microstructure features, such as precipitates, dislocations and dispersoids is needed. It is also important for absorption and fluorescence effect corrections when energy dispersive spectroscopy (EDS) is used for composition analysis. Focused ion beam (FIB) can be used to cut the sample to reveal the cross section and then directly measure the sample thickness. However, this method is destructive. Electron energy loss spectroscopy (EELS) and convergent beam electron diffraction (CBED) have also been used widely to determine the sample thickness. For EELS, the thickness calculation is based on a simple relationship between the log-ratio intensity distribution and the ratio of mean free paths of electron inelastic scattering to sample thickness, that is $t = \lambda \ln(I/I_0)$, where t is the sample thickness, λ the mean free path of inelastic scattering, I the total intensity of the zero loss peak and the plasma peak and I_0 the intensity of the zero loss peak [3]. In the CBED [4], the sample thickness can be linked to the fringe minima observed as $(s_i^2 + \frac{1}{\xi_g^2})t^2 = n_i^2$, where s_i is the deviation of the ith minimum from the exact Bragg position, ξ_g is the extinction distance and n_i an integer. Thickness can be determined from the slope of the plot of ${s_i}^2$ versus ${n_i}^2$. Both EELS and CBED methods can give a reliable thickness. Especially for CBED, in which case the relative error is better than 5% even with the above simple version of the formula [5]. Even better accuracy can be obtained by quantitative many-parameter fits to the intensity profiles of the CBED discs [6]. However, both methods are time consuming, especially when many thickness measurements are needed.

Since the BSE coefficient and SE yield have been widely researched using experiments and theoretical calculations and for thin samples, both depend on the thickness [7]. There is a potentially more efficient way to determine the thickness of a specimen

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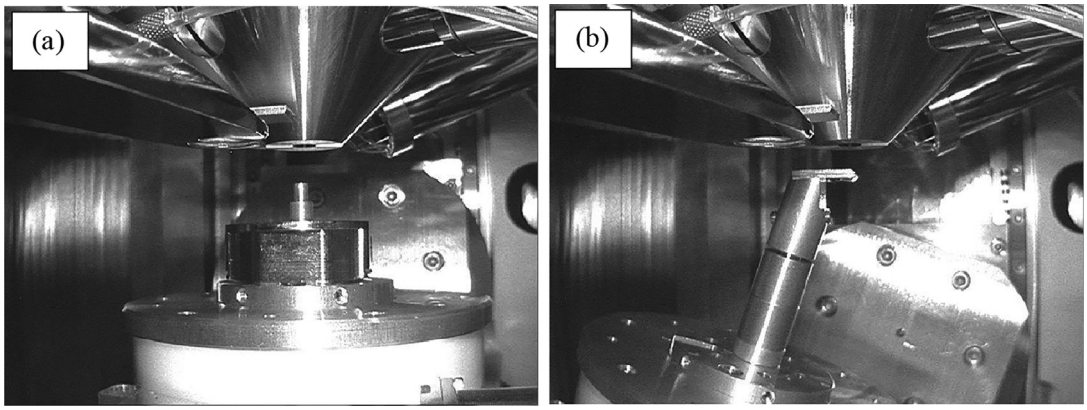

Fig. 1. Two types of holders used in this study: (a) STEM holder and (b) TKD holder.

based on the BSE coefficient and SE yield obtained. In this paper, the intensity profiles of both BSE and SE images were measured experimentally vis-à-vis Monte Carlo (MC) simulations and theoretical calculations, and the application to thickness determination is discussed.

2. Experimental procedure and simulation

Pure Mg TEM samples of 3 mm diameter were prepared by twin-jet polishing using a solution containing lithium chloride 8.8 g, magnesium perchlorate 19.3 g, methanol 833 mL and butoxyethanol 167 mL, at a voltage of 70 V and a temperature of −45 °C. Both BSE and SE images were taken at different acceleration voltages (HV) of 5, 10, 15, 20, 25 and 30 kV using two different sample holders (an STEM holder and a TKD holder) in a TESCAN Mira microscope (the configurations are shown in Fig. 1). The SE and BSE detectors used are an Everhart Thornley detector with standard grid bias and a YAG detector with a single annular scintillator, respectively. The other imaging conditions were kept the same. The image intensity profiles versus distance from the edge of the hole were obtained from defined positions. The sample was finally cut apart using the FIB at these defined locations to directly measure the thickness of the sample.

MC simulation of the SE and BSE yields was done using CASINO version 3.3 [8] with a thickness step of 0.1 μm from 0.1 μm to a thickness when no electrons can get through the sample and with different energies from 5 to 30 keV in steps of 5 keV. At 5 keV, the initial thickness was set as 10 nm with a step size of 10 nm. In every case, 1,000,000 electrons were considered. The cut-off energy was set to 50 eV for all the conditions. It has been shown that the mean penetration depth in aluminum is less than 1 Å when the cut-off energy changed from 100 eV to 20 eV [9]. So the effect of cut-off energy used in the current work on the SE and BSE signals and therefore the thickness determination was regarded as negligible. The modified Bethe formula given by Joy and Luo [10] and Lowney [11] was used to determine the stopping power and the scattering cross section was calculated by ELSEPA model [12].

The BSE coefficient of bulk samples has been widely researched using both experiment and theoretical calculation as it is the foundation of the BSE imaging. Hunger and Küchler [13] measured the BSE coefficients of 28 elements and derived an analytical expression of the dependence of BSE coefficient on the electron energy and the atomic number. For light elements with Z smaller than about 50, the BSE coefficient decreased with increasing incident electron energy and vice versa. Everhart [14] derived a formula which showed that the BSE coefficient was independent of the incident electron energy, which is consistent with the MC simulations using both single and plural scattering models. Joy [7] suggested the inconsistencies between the experimental results [13] and computed data [14] probably arose from the variety of methods used to measure the BSE coefficient. Compared with bulk samples, BSE coefficients of thin samples have received little attention with regard to the relationship between BSE coefficients and thickness. Niedrig [15] reported a linear relationship between BSE efficiency and the sample thickness for most elements (except those with low atomic number) in the low thickness region which is much smaller than the penetration depth of the incident electrons, and proposed a model to interpret the experimental results. Nakhodkin et al. [16] extended the Everhart model to films with thicknesses between 0 and $R/2$, where R is the maximum penetration depth. Using a simple potential between electron and atom, Kanaya and Okayama [17] obtained an equation which can be used to calculate the BSE coefficient over the whole range 0 to R, the predicted BSE coefficients were much bigger than the experimental results [18]. MC simulation has therefore been carried out to determine the dependence of BSE coefficient on the thickness.

In contrast to BSE coefficients, SE yields do not depend upon the atomic number, while strongly depend on the incident electron energy [19]. Baroody [20] formulated a theory based on the Sommerfeld model [21] and pointed out that the dependence of SE yield on incident electron energy can be described using a single curve. However, the calculated data were lower than the experimental results [20]. After that, several theories were proposed to calculate the SE yield, e.g. by Seiler [22], Dionne [23]. Lin and Joy [24] thoroughly examined the correlation between SE yields (δ) and the primary electron energy (E_{PE}) for 44 elements and proposed a semi-empirical law to describe the correlation

$$\frac{\delta}{\delta^m} = 1.28\left(\frac{E_{PE}}{E_{PE}^m}\right)^{-0.67}\left\{1-\exp\left[-1.614\left(\frac{E_{PE}}{E_{PE}^m}\right)^{1.67}\right]\right\}$$

where δ^m is the maximum SE yield and E_{PE}^m the corresponding energy for the maximum SE emission. For Mg, the parameters were set as 0.8 and 240 eV respectively, which agree reasonably well with the experimental results of 0.8 and 300 eV and the calculated results of 0.67 and 280 eV by Kanaya and Kawakatsu [25]. Only those SE excited near the surface can reach the surface and escape from it. The escape depth had been revealed by MC simulation, indicating that the escape depth in Cr for 20 keV electrons was about 3 nm [26]. This is consistent with the result of Seiler [22], which showed that the escape depth of SE is about 5λ where λ is the mean free path of SE and of the order of 5 nm and 75 nm for metals and insulators, respectively. This can be used to explain why the SE intensity profiles had a step at the edge of the holes. As the sample thickness is much greater than 5 nm, the SE yields by PE saturated immediately even at the edge.