doi:10.1017/S1431927618003392
Microsc. Microanal. 24 (Suppl 1), 2018

A Simple Method to Estimate Local Absolute Thickness Using Scanning Transmission Electron Microscopy

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In analytical scanning transmission electron microscopy (STEM), it is often important to know the local absolute thickness of the thin specimen, especially when it comes to X-ray analysis and electron energy loss spectroscopy (EELS) for quantification, as well as for achieving higher spatial resolution by deconvolution of beam spreading/broadening effect. This study presents a simple method for estimating the local absolute thickness using the intensity of STEM high-angle annular dark-field (HAADF) images from a TEM specimen. Although a number of factors can contribute to the intensity of STEM HAADF images, including atomic numbers of elements, specimen thickness, crystal orientation, geometry of detector, etc., this study makes the assumption of $I \sim t{\cdot}Z^{\alpha}$, where I is the intensity obtained from the HAADF image, t is the local absolute thickness, Z is the average atomic number and α is a function of camera length (and hence a function of collection angle of HAADF detector) and is in the range of 1.6-1.9 for most cases [1].

The sample investigated in this study is an RF-sputtered amorphous $La_{0.8}Sr_{0.2}MnO_3$ (LSM) thin film with thickness of ~180 nm on 8 mol% Y_2O_3 stabilized ZrO_2 (YSZ) single crystal substrate with a surface normal of (100). The TEM specimen was prepared using a standard focused ion beam (FIB) lift-out process where Pt was deposited to protect the film from damage by Ga ions in FIB. Prior to FIB lift-out process, a thin layer (~ 10 nm) of Pt was sputtered on the surface of the sample using a commercial sputtering coater. The STEM HAADF imaging was carried out in an FEI Titan G2 80-300 TEM/STEM. The specimen was first tilted off-zone for YSZ single crystal to minimize channeling effect, and STEM HAADF images were then recorded with various camera lengths while keeping all the other experimental conditions unchanged (Figure 1). Intensities of the STEM HAADF images were integrated along a line (with a width of 100 nm) starting from Pt across LSM layer into YSZ substrate, as shown in Figure 2a. By dividing the intensity profile with corresponding Z^{α}, a profile can be obtained with a linear relationship with local absolute thickness of the specimen. EELS spot analysis of the zero-loss region was carried out at multiple spots along the line in the HAADF image where intensity was integrated, to measure the relative thicknesses of the spots. The absolute local thicknesses were then obtained with estimated inelastic mean free path using the method described in reference [2].

The continuous absolute thickness profile calculated using STEM HAADF intensity shows excellent agreement with absolute thickness measured by EELS (Figure 2b). The profile was calibrated using three absolute thickness values measured using EELS at Pt, LSM and YSZ regions. In practice, it is best to record the STEM HAADF image at small camera lengths (or large collection angles) to estimate local absolute thickness of a specimen using the method described above. Although the precise value of α can be experimentally measured at each camera length, it is found that α=1.8 is a fairly good estimation at small camera lengths [3].

References:

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[3] This technical effort was performed in support of the National Energy Technology Laboratory's solid oxide fuel cell research activity under the RES contract DE-FE0004000. The authors acknowledge use of the Materials Characterization Facility at Carnegie Mellon University supported by grant MCF-677785.

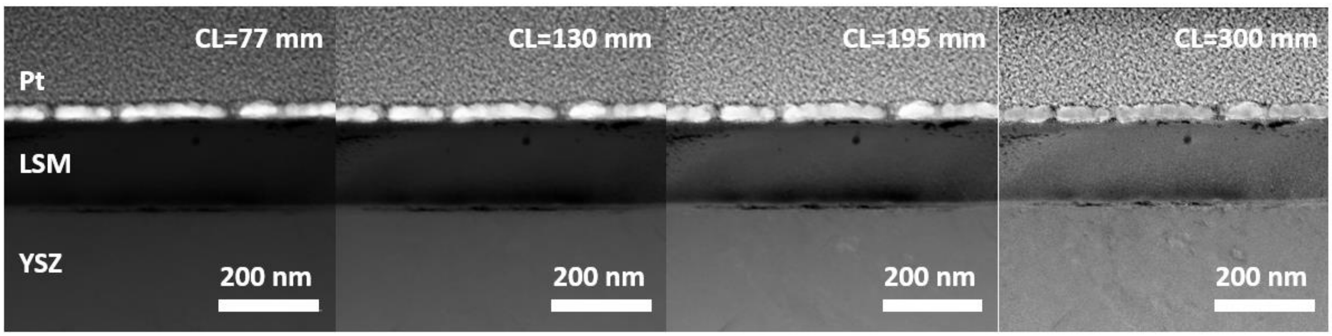

Figure 1. STEM HAADF images taken at various camera lengths at the same location.

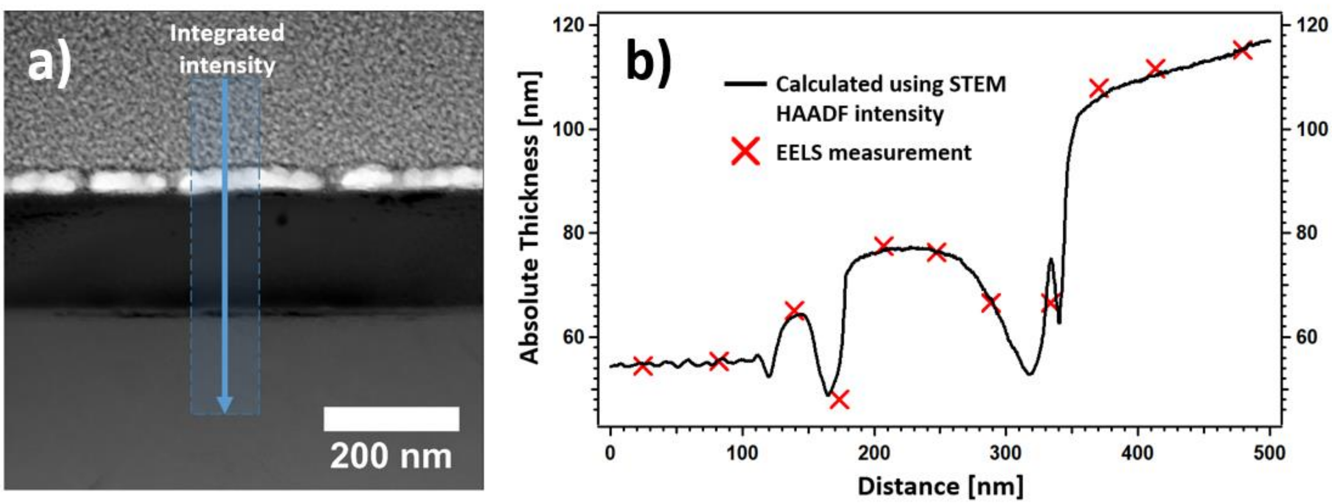

Figure 2. a) STEM HAADF image showing region where integrated intensity was obtained and b) comparison of local absolute thicknesses obtained using STEM HAADF intensity (CL=130 mm) and EELS.

https://doi.org/10.1017/S1431927618003392 Published online by Cambridge University Press