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Measuring Relative Performance of an EDS Detector Using a NiO Standard

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Abstract

A method for measuring the relative performance of energy dispersive spectrometers (EDS) on a TEM is discussed. A NiO thin-film standard fabricated at Sandia CA is used. A performance parameter, ξ, is measured and compared to values on several TEM systems.

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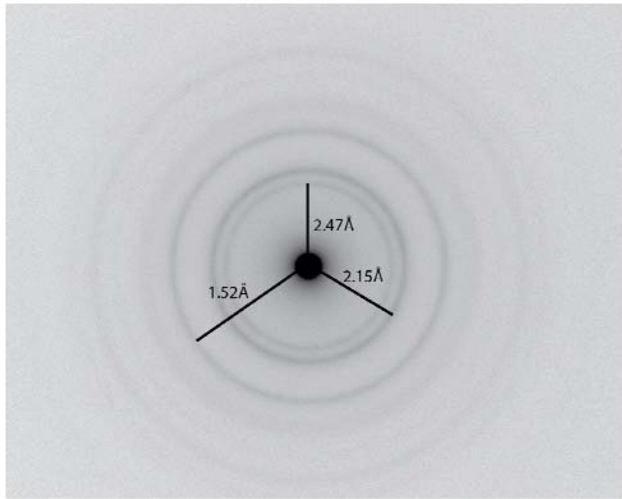
1. MEASURING RELATIVE PERFORMANCE OF AN EDS DETECTOR USING A NIO STANDARD

It is difficult to measure the solid angle of an EDS detector in the TEM. It requires knowing some information about the detector that the operator may not always know. This document outlines a method for measuring a parameter, ξ, which describes the relative efficiency of an EDS detector and has units of $\text{counts}/(\text{nA} \cdot \text{sec} \cdot \text{nm})$. The comparison of this parameter on several instruments yields an idea of the relative performance of each system.

Thin films of NiO were sputtered onto single crystal NaCl crystals coated with 8 nm C (8 nm C measured by AFM). The total pressure in the sputtering chamber was brought to 4 mtorr by the introduction of O_2 gas. The films were sputtered with a plasma power of 300 watts. I confirmed with electron diffraction that the samples are NiO.



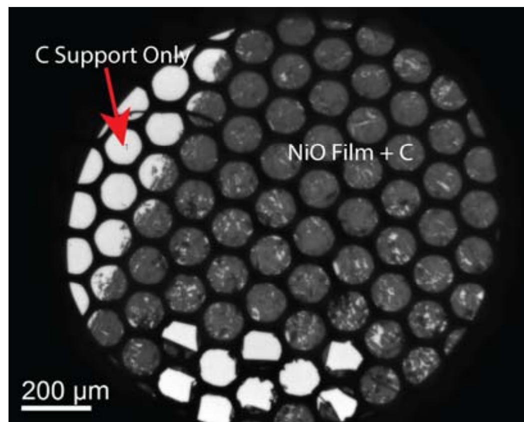
hkl	D_{hkl} (Å) Calculated NiO
111	2.408
200	2.085
220	1.474
311	1.257
222	1.204
400	1.043

The d-spacings are slightly larger than the expected values for perfect NiO. I believe this is because the NiO films are slightly strained. The NiO films are floated off of the NaCl crystals in distilled water. When they dry on the grid, I believe they have a tendency to bow (convex or concave) over the holes in the Moly TEM grid. This bowing strains the films and makes the d-spacings slightly larger than expected.

In order to measure ξ, we need a measurement of the electron probe current, the NiO thickness, and the counts from the Ni K x-ray emission. The electron probe current was measured with a picoammeter and a Faraday cup in Gatan 626 holder. The results for a JEOL2010F are below Note: Probe sizes are not “true”, they are just labels.

“1.6 nm probe”, 50 μm condenser aperture	.92 nA
“1.0 nm probe”, 30 μm condenser aperture	.12 nA
“0.7 nm probe”, 30 μm condenser aperture	.07 nA

The thickness of the NiO film was measured using the EELS log-ratio technique. The moly grid has a C support film. So, we must measure the thickness of the C support film and the NiO. To do this a measurement was made on the C support film alone, and then another measurement was made on the C support film + NiO. The NaCl crystals had a 8 nm film of amorphous C deposited on them (I did this in an SEM coater) because this helped eliminate a texture that developed in the NiO film when deposited directly on NaCl. I also think this helps prevent tearing of the NiO film during drying. The image below shows regions of the C film alone and the C support plus NiO.



Several measurements of t/λ were averaged for the absolute thickness measurements. A value of λ_C of 160 nm (R.F. Egerton, S.C. Cheng, Ultramicroscopy, 55 (1994) 43-54.) and λ_{NiO} (K. Iakoubovskii, K. Mitsuishi, Y. Nakayama, K. Furuya, Physical Review B, 77 (2008).) of 115 nm were used.

Sample description	Relative thickness (t/λ)
Sample #30 C only	.19
Sample #30 C+NiO	.78
Sample #31 C only	.185
Sample #31 C+NiO	.69

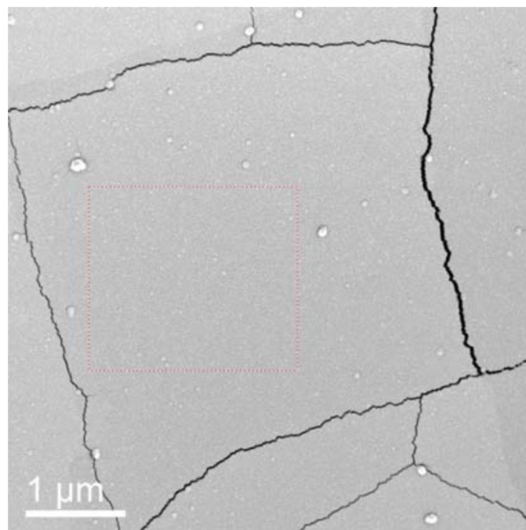
Based on these measurements, the C support film is ~30 nm thick. That makes the total thickness of the C 38 nm (because of the added 8 nm C film on the NaCl crystal). Then I could calculate the NiO thickness. I assumed that t/λ adds linearly.

$$t/\lambda_{total} = t_c/\lambda_C + t_{NiO}/\lambda_{NiO} \Rightarrow\Rightarrow\Rightarrow t_{NiO} = \frac{\left(t/\lambda_{total}\right)\lambda_C\lambda_{NiO} - t_C\lambda_{NiO}}{\lambda_C}$$

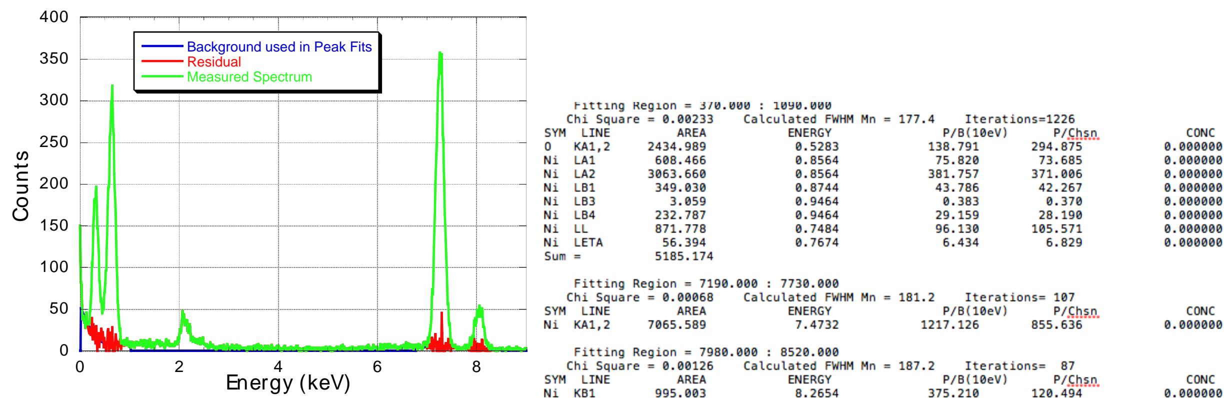
$$= \frac{t_c\lambda_{NiO} + t_{NiO}\lambda_C}{\lambda_C\lambda_{NiO}}$$

So the C thickness is about 38 nm based on a t/λ of ~0.2 for C alone and the added 8 nm film on the NaCl. The result is a NiO thickness of 62 nm for sample #30 and 52 nm for sample #31.

Finally, the EDS spectra are measured. The EDS spectra are measured with a very short time constant so that the dead times are very low. The goal is to count as many counts as possible in the x-ray detector. Then a small area of the sample is scanned (where there are no cracks or large deformities in the film) and the EDS spectrum is recorded. Note that there are places in the NiO film where it has folded on itself so that it is 2x thicker. This is easy to see in HAADF if you compare the contrast from neighboring regions at low mag. Make sure the region used for the calculation is the same region the thickness was measured from. An example HAADF image and scan box is shown below.



The EDS spectra were measured with an integration time of 120 sec. The livetime was slightly less because of dead time. The spectra were then imported into the NIST software old DTSA. The Ni K and L and O K peaks were then fit using a simplex fit. An example of the results is shown below.



The detector performance parameter can now be calculated.

$$\xi = \frac{Ni_{K\alpha} + Ni_{K\beta}}{\text{probe current } \cdot \text{NiO thickness } \cdot \text{livetime}} \left[\frac{\text{counts}}{\text{nA} \cdot \text{sec } \cdot \text{ nm}} \right]$$

The measurement on our FEI G2 Titan 80-200 with ChemiSTEM (nominal 0.7 str solid angle) yields a value for ξ of approximately 80 counts/nA sec nm (with a different NiO sample). The results of 3 measurements on our JEOL 2010F with Oxford SiLi EDS detector (nominal solid angle 0.11 str) and Gatan double-tilt analytical holder are in the table below. Additionally, relative results from the FEI Tecnai F30-ST are shown.

Measurement Condition	ξ (counts/ nA sec nm)	Value Relative to Titan (nominally 0.11/0.7=.157)
Sample #30; 0.7 nm probe; 50 μm condenser aperture	7.98	.0997
Sample#31; 1.6 nm probe; 30 μm condenser aperture	9.95	.124
Sample#31; 1 nm probe; 30 μm condenser aperture	10.98	.137
Tecnai F30-ST, 0.1sr Si(Li)	~7	.095

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