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Low Voltage Analysis: How accurately do you need to know your coating thickness?

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EPMA at low accelerating voltages and/or low over-voltages in the reach for ever higher resolution means that any surface coatings on our samples can no longer be treated as an insignificant part of the analysis volume. For example, absorption and fluorescence due to the film can become significant components of the correction calculation. Also, energy loss of the primary electrons within the film reduces the energy available to fluoresce X-rays in the sample. This is particularly relevant for low over-voltage analyses. Unfortunately, determining even the relatively trivial parameter of the thickness of a pure element coating can be prone to significant errors. The results presented here show significant systematic differences depending on how the film thickness is determined.

To test the accuracy and repeatability of coatings measured with a film thickness monitor (FTM), polished samples of Bi metal were coated with a range of thicknesses of Cu. For each sample a fixed 5nm target thickness was set and nominal thicknesses of 5, 10, 15 and 20nm coatings deposited by applying single or multiple coatings. The Cu density used for the FTM settings was taken to be 8.96 gcm^{-3}, the bulk elemental density. To minimize any errors from the difference in position between the samples and the FTM sensor, a coater model was chosen which co-located the FTM sensor with the samples being coated.

Experimental k-ratios for Cu;Lα and Bi;Mα were acquired using EPMA at 5, 7, 10 and 15kV, calibrating each coating against uncoated pure Cu and Bi metal reference materials. The derived k-ratios were converted directly into modelled film thicknesses using GMRFilm, and indirectly using film thickness versus k-ratio relationships derived using DTSA II. The resulting values are summarized in Table 1. Film thickness versus k-ratio data was also generated with GMRFilm to allow the results from the two software packages to more directly be compared. For both software packages the default settings were applied, and the film density used was 8.96 gcm^{-3}

The resulting plots of film thickness versus k-ratio, Figure 1, show linear relationships over the range of thicknesses calculated. GMRFilm appears to produce similar but systematically lower k-ratio values than DTSA II for a given film thickness, with the difference increasing with decreasing accelerating voltage. The comparisons between the FTM quoted thicknesses and the two sets of Monte Carlo calculated thicknesses also show systematic differences, with the modelled films being consistently 4 – 5% thicker than the FTM values (Table 1).

In order to assess the analytical impact these discrepancies could be expected to have, GMRFilm was used to calculate the Bi;Mα emitted k-ratios at 5, 7, 10, and 15kV, using the FTM and GMRFilm thicknesses for the thinnest and thickest Cu films. The results summarized in Table 2 show that, at 15kV the difference in k-ratio between the FTM and GMRFilm thicknesses is less than 1%. However, at 5kV even the thin coat yields a difference of nearly 3%, whilst for the thick film the difference is ~6.5% and we could expect this magnitude of 'error' to propagate to our analyses under these conditions. This, of course, assumes that the Monte Carlo results are closer to the true film thickness than the FTM.

Since the thicker films were deposited using multiple coatings of nominally 5nm the highly linear correlations for the set of four film thicknesses shown in Figure 2 indicate that the coating depositions have at least been very repeatable. This gives us the expectation that if we can calibrate our FTM we could expect to get improved thickness measurements.

Cu on Bi

	DTSA II		GMRFilm	
FTM	Mean	*Std. Dev.*	Mean	*Std. Dev.*
5.46	5.52	*0.15*	6.40	*0.10*
10.96	11.42	*0.34*	12.51	*0.14*
16.43	17.62	*0.88*	18.70	*0.29*
21.92	22.46	*1.58*	23.61	*0.45*

Table 1 FTM-measured film thicknesses and Monte Carlo calculated film thicknesses from the experimentally derived k-ratios for the Cu on Bi samples. The mean and standard deviation DTSA II and GMRFilm values are derived from 48 analysis points for each sample (12 points at each of the four accelerating voltages 5, 7, 10, and 15kV).

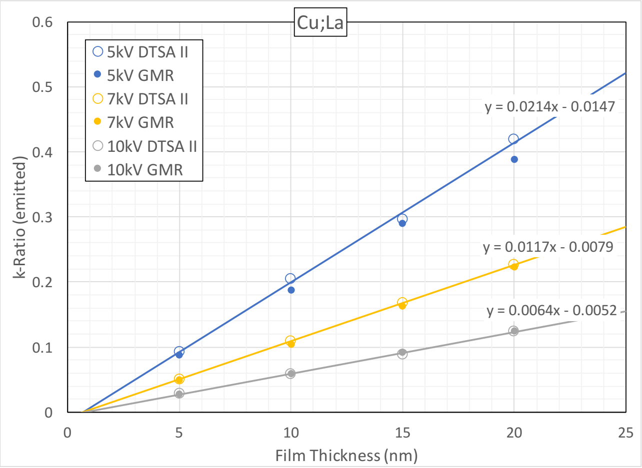

Figure 1 Monte Carlo derived film thickness versus k-ratio relationships for Cu films on Bi from both DTSA II and GMRFilm.

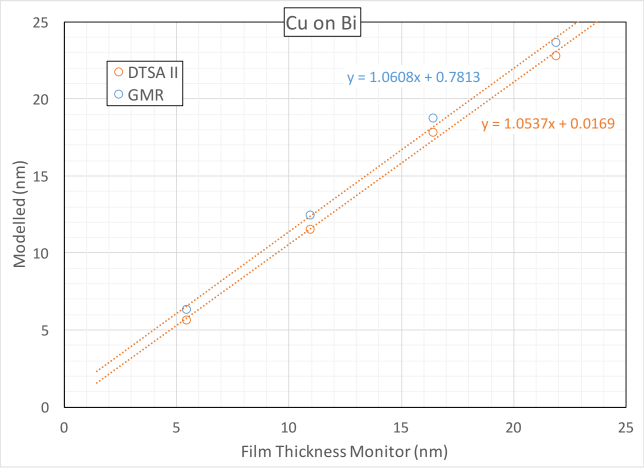

Figure 2 Comparisons of the film thicknesses reported by FTM and Monte Carlo modelled thicknesses derived from experimental k-ratios.

a)	Bi;Mα Thin Cu Film		
kV	nm	k-ratio	% Diff.
5	5.46	0.8639	2.97
	6.40	0.8383	
7	5.46	0.9333	1.35
	6.40	0.9207	
10	5.46	0.9645	0.67
	6.40	0.9581	
15	5.46	0.9797	0.36
	6.40	0.9761	

b)	Bi;Mα Thick Cu Film		
kV	nm	k-ratio	% Diff.
5	21.92	0.4780	6.45
	23.61	0.4472	
7	21.92	0.7018	3.21
	23.61	0.6792	
10	21.92	0.8431	1.55
	23.61	0.8300	
15	21.92	0.9150	0.75
	23.61	0.9081	

Table 2 The effect of the difference in film thicknesses reported on GMRFilm calculated Bi;Mα k-ratio values over a range of analysis voltages for both the thinnest (a) and thickest (b) Cu coatings measured.