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Ultra-thin Iridium as a Replacement Coating for Carbon in High Resolution Quantitative Analyses of Insulating Specimens

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Insulating specimens are traditionally coated with a thin conductive layer of carbon in preparation for quantitative microanalysis. Results are insensitive to exact carbon coat thickness for energy lines greater than 1 keV and beam voltages greater than 10 keV. However, for specimens that contain carbon (e.g. where quantitative analysis is desirous), an alternative conductive coating needs be applied. Traditional substitute coatings, such as Au, Cr, Al, or Au-Pd, require standards to be coated similarly or require application of significant corrections, which can result in significant errors [1,2].

In this study, iridium was tested for usefulness as a reasonable alternative coating for quantitative analysis. Previously, elements like Pt and Ir had been found to be an excellent coating for high resolution imaging [3] and layers of a few nm of Ir were found to provide adequate protection from beam damage with sufficient conduction ~0.5 nm thick [4]. We investigated thin coatings of iridium from 0.2 to 6 nm, sputtered onto various substrates (silicon, copper, carbon, aluminum alloy, and glass) using a commercial bench-top magnetron sputter coater, and analyzed by EPMA and FE-SEM.

Offline correction using $\phi(\rho z)$ algorithms and Monte Carlo simulations was employed to determine the Ir-coating thickness and estimate the effect on emitted x-ray intensities from the substrate. The two procedures give good agreement for <6 nm Ir-film thicknesses. There is a monotonic relationship between the iridium thickness and the k-ratio that can be expressed by a 2^{nd} order polynomial (Fig. 1). For thicknesses of iridium 1 nm or less, the effect on x-ray intensity is less than 5% for soft x-rays and less than 2% for most lines (Fig. 2).

Results of this study indicate that an Ir-coat of 0.4 nm thickness is sufficient to provide sufficient conductivity on an insulating sample for analysis; although for samples containing beam-sensitive materials, a minimum thickness of 1 nm is necessary to avoid element migration (Table 1). Coatings of less than 2 nm require minimal or no additional offline corrections, making this an ideal substitute for traditional carbon coatings and require no special preparations of standards (i.e. uncoated or carbon-coated standards can be directly compared). However, the K-lines of boron to oxygen and the L-lines of calcium to chromium are heavily absorbed by iridium, requiring correction for Ir thicknesses over 1 nm.

References

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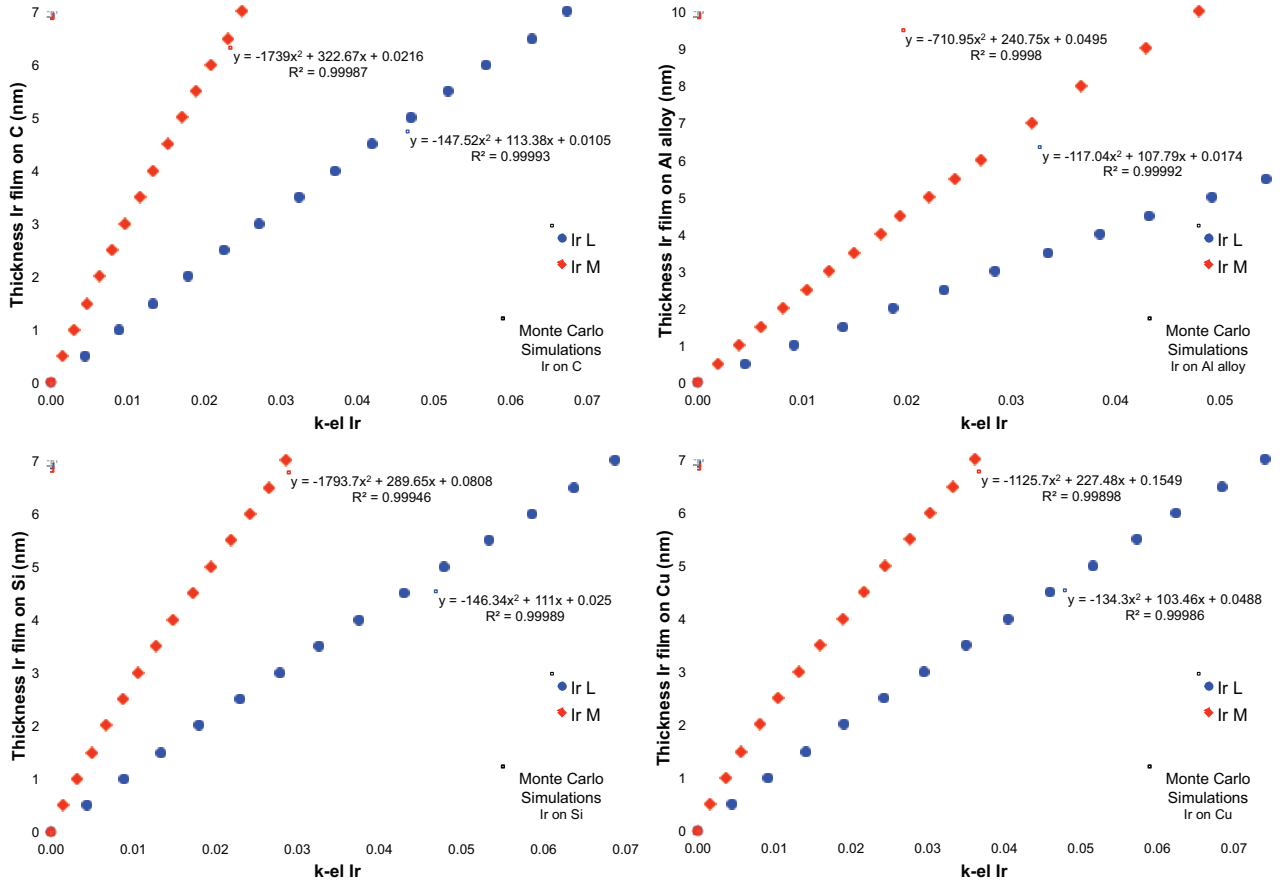

Figure 1. Monte Carlo simulations of iridium coating thickness on substrates of various composition.

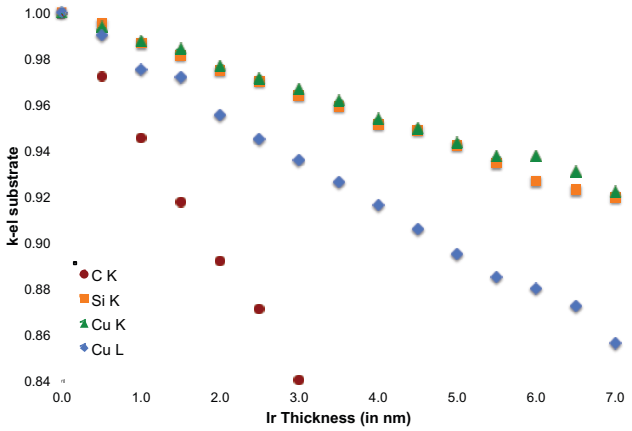

Figure 2. The calculated loss of intensity in pure element substrate as a function of Ir-coat thickness using Monte Carlo simulations.

Measured Composition of Ir-coated Glass					
Thin Film Calculations using k-el Ir M					
EDS	C-coat		Thickness Ir-coated sample		
El wt%	~15 nm		0.4 nm	1.0 nm	1.8 nm
Na	9.13		**8.81**	9.09	9.19
Mg	2.39		2.41	2.37	2.37
Al	0.34		0.32	0.33	0.33
Si	34.59		34.04	34.44	34.56
K	0.19		0.31	0.20	0.18
Ca	5.91		5.95	5.92	5.91
O	47.45		48.00	48.17	48.53
Total	100.00		99.84	100.51	101.07

Table 1. Measured composition of Ir-coated glass using EDS compared to glass coated with carbon. No correction needs to be made for the Ir-coat.

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