878 CD
DOI: 10.1017/S1431927606064403
Microsc Microanal 12(Supp 2), 2006

Multi-Voltage EPMA of Thin Films or Nano Science in the Z Dimension

John J. Donovan

Dept. of Chemistry, University of Oregon, donovan@uoregon.edu

Quantitative EPMA analysis of thin (sub-micron) films on substrates is a useful and efficient way to characterize nano-scale materials for both chemistry and thickness in the z dimension. Previously when sufficiently thick films were being characterized, it was sometimes feasible to reduce the operating voltage of the instrument to keep the x-ray interaction volume entirely within the film overlying the substrate. However, as interest on ultra-thin films for nano-science applications increases, the thickness of these films decreases to the point where they can no longer be treated as a “bulk” analytical situation.

To obtain quantitative results for thin films on arbitrary substrates requires the acquisition of x-ray intensities at several operating voltages. These voltages are usually selected to cover a range of intensity emission where a moderate change in operating voltage produces a significant change in the x-ray emission of elements contained in the thin film. At the same time, the minimum operating voltage should be sufficient to penetrate into the underlying substrate. This is critical not only for the measurement of the substrate x-rays, which can then be used to quantify the thin film mass thickness (or linear thickness if the density is known), but also for proper treatment of multi-layered elemental depositions as homogeneous compositions. See Tables 1 and 2.

For example, thin films less than 100 nm will require typically a reduction in the range of operating voltages requiring the use of softer x-rays including L and M series lines (Fig 1.), while thicker films benefit from a higher range of operating voltages (Fig. 2). As Figure 1 reveals, it is necessary to help constrain the iterative model by utilizing operating voltages that produce a range of intensity variation for the film in question.

Additionally a robust and flexible data processing software is critical for maximizing efficiency, which means that the software must be able to automatically iterate both film thickness and composition simultaneously. Also essential is the software’s ability to correct for characteristic and continuum fluorescence between the substrate and the film layers and between the film layers themselves. This greatly limits the field of useful thin film processing packages since only a few products such as GMR Film and StrataGem are able to do this and only StrataGem can import data from a variety of sources.

With the increasing reliability of automated acquisition at multiple accelerating voltages and the growing sophistication of thin film data processing, the electron microprobe provides a powerful tool for accurate and rapid analysis of arbitrary chemistry and geometry. In the analytical situation where the same element exists in both the substrate and the film (or in multiple film layers), the use of XRD to constrain thickness or grazing XRD to constrain density can provide the analyst with the necessary leverage to obtain excellent quantitative results for thin films down to 10 nm or less.

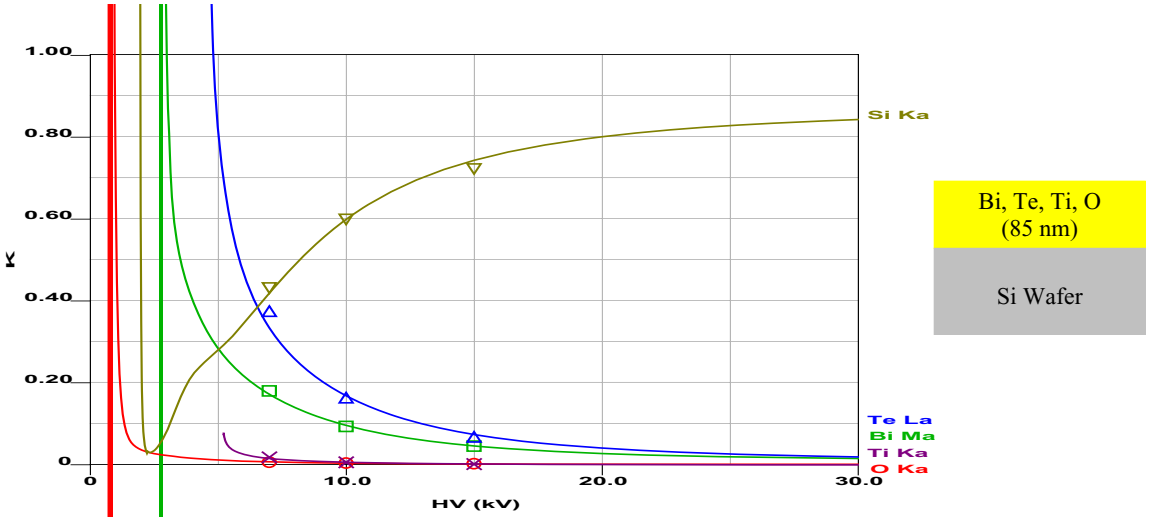

Figure 1. Operating voltage vs. K-ratio intensity for Bi, Te, Ti on Si at 7, 10, 15 keV

SAMPLE DESCRIPTION

Layer #	Element	Concentration	(# atoms)	Mass Depth (μg/cm²)	Thickness (nm)	Density
1	Bi	0.3775	0.2510	42.700	85.4	5.0
	O	0.0047	0.0408			
	Te	0.5982	0.6514			
	Ti	0.0196	0.0569			
Substrate	Si	1.0000	1.0000			

Table 1. Quantitative results for above system after iteration of composition/thickness.

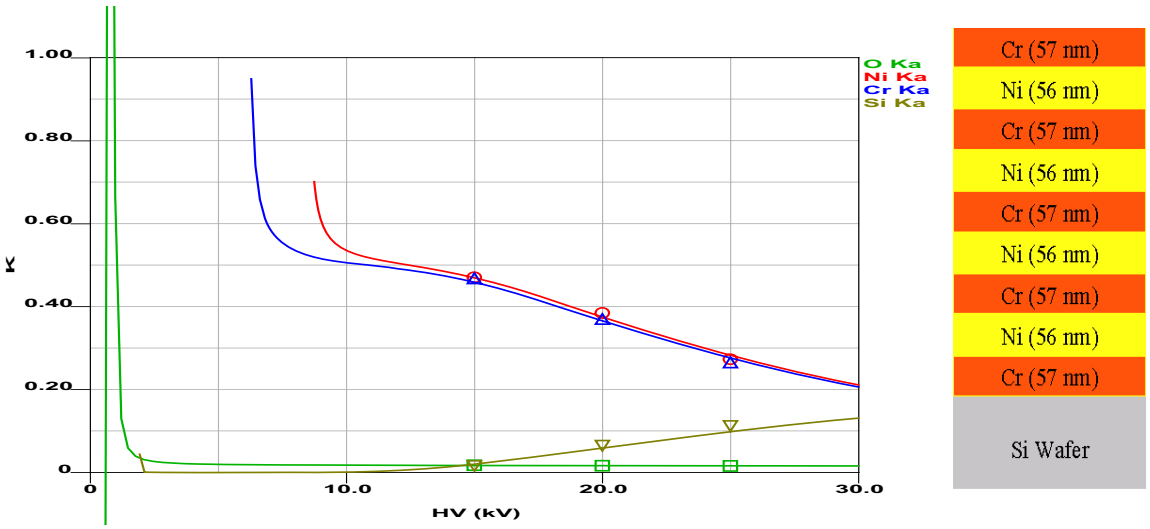

Figure 2. Operating voltage vs. k-ratio for NIST 2135c thin film at 15, 20, 25 keV

SAMPLE DESCRIPTION

Layer #	Element	Concentration	(# atoms)	Mass Depth (μg/cm²)	Thickness (nm)	Density
1	O	0.0056	0.0189	425.401	541.9	7.8
	Ni	0.5081	0.4715			
	Cr	0.4864	0.5096			
Substrate	Si	1.0000	1.0000			

Table 2. Quantitative results for above system after iteration for composition/thickness.

https://doi.org/10.1017/S1431927606064403 Published online by Cambridge University Press