Thin Film Analysis

GMRFILM is a research grade, shareware DOS program for thin film analysis (created by Richard Waldo of General Motors Research Labs). It can be used to estimate film thicknesses using the experimentally determined K-ratios, or to calculate compositions given known thickness and K-ratios. Thin films are increasingly important in the EPMA lab, e.g. as specific research objects (creating thin film superconductors), AND because many samples have either intentional (conductive) thin films laid down on them, or accidental ones (e.g. oxide coat).

You run it on an IBM compatible PC, in a DOS window.

The software is on a DOS format diskette; first load the 2 files (gmrfilm.abs is a short abstract, test.exe is the compressed program) into a subdirectory on your hard disk, then type **test.exe** to uncompress the program and all files (there is a readme.1st file with more info about the program). Type **gmrfilm** to run it.

Given conditions: Generally take the defaults, but especially the following:

<u>Film</u> analysis <u>PAP</u> phi rho z model $E_0 = \underline{15} \text{ kV}$

<u>40°</u> take off angle K lines for O, Mg, Si, Ti ; L lines for Zr, Hf; M line for Au

-- it will ask for an approximate density for layer.Use what you find in the literature, unless I give you something to use.

-- take defaults, EXCEPT do include the continuum fluorescence correction.

Enter all element and x-ray data in format ELXR, s where EL is the Element, XR is the Xray line, and s is the switch: e.g. TiKa or TiKa, m (both equivalent) or C Ka or O Ka, c (note space after single letter element)

switches: if the element is in more than one layer, use m -- if standard used is pure element

use n -- if standard used is a compound

if the element is in only one layer, use c -- if standard is a compound use no switch if pure element standard

1. You want to determine the thickness of an oxide coating (TiO_2) that has formed on your "pure" Ti standard (no carbon coat). Calculate the TiO₂ thicknesses from the experimental k-ratios:

Ti .9738 O .0180 Assume TiO2 density is 4.2 g/cm3.

You will input guessed thicknesses of the thin film TiO2 coating (hint: somewhere between 10 and 1000 A), and then compare the resulting k-ratios with those you are given, trying to find the best match for each case, for both k-ratios. NB: for the element that is in both layers (Ti), the program splits out the contribution from each layer. Obviously, your experimental k-ratio is the <u>sum</u> of both. You may not get both elements to exactly match the above k-ratios: give you best/closest estimate.

2. Calculate the relative error on each Mg, Si and O Ka (K-ratios) in an analysis of your unknown Mg2SiO4 if your standard has 250 A of Carbon whereas the unknown has 150 A (assume simple case elemental standards; assume C film density 1.6 g/cm3).

3. You have a zircon (Zr2SiO4, with slight substitution of Hf for Zr) that has been ion probed, which means it has been gold coated. You want to do an epma analysis, but all you have are your normal carbon coated zircon standards. You go ahead, calibrate, and give the analysis a shot....and get an analysis with a total of about 91 wt%:

K-ratio Element wt% (after normal matrix correction)

Zr 0.3652 43.73

Hf 0.0066 0.95

Si 0.1316 14.46

O (by stoic) .0802 31.98

Then you recall you have GMRFILM on your computer, and figure you'll plug in the k-ratios, with some guesses at the Au coating thickness, to see if you can extract a halfway decent chemical analysis. Can you? What is your estimation of the Au thickness (assume density of 19 g/cm3)?