Investigations of the quantitative analytical depth resolution of field emission electron probe microanalysis

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For many applications in material analysis, an EDX- or WDX-element analysis (EPMA - electronprobe microanalysis) with high spatial resolution is of great importance. Hereby, not only the qualitative resolution, but especially the quantitative resolution, which delivers the exact element composition of small features, is in the focus of interest. So far we investigated the lateral analytical resolution whereby 'lateral' indicates the horizontal x-/y- directions [1]. In accordance with many other published results, we measured the best value for the qualitative lateral resolution to be 26 nm. The value has been determined from 16 to 84 % intensity rise of the Au-signal in a linescan at a Si-Au interface. To determine the quantitative lateral resolution, different thin metallic layers were generated on Si wafers by vapour deposition to be analysed at FIB-prepared cross-sections. The layers can be resolved quantitatively if the source volume of the emitted X-ray is completely inside the layer and no X-ray signal from the adjacent layers is detected. Thus, quantitative lateral resolutions of 140 to 170 nm for Au layers, 150 to 210 nm for Ag layers and 260 to 280 nm for Al layers were proved [2, 3].

The aim of this work, now, is to determine the quantitative depth resolution of EPMA at thin surface films and to compare measured, simulated and calculated data. Specimens are generated by evaporation of thin metallic layers onto silicon substrates again. In the first step, seven aluminum layers are deposited with thicknesses from 3850 down to 120 nm. The surface of each of these layers is analysed quantitatively using the 3850 nm thick layer as reference standard. For EPMA, a field emission electron probe microanalyser (FE-EPMA) with wavelength dispersive X-ray spectrometers (WDX) is used. The accelerating voltage is varied from 15 to 3 kV. A quantification result of 100.0 +/- 0.5 wt % Al and less than 0.5 wt % Si indicates that the source volume of the emitted X-rays is completely inside the layer. For comparison the volume of generated X-ray is estimated by Monte Carlo (MC) simulations (casino v2.48) of the electron scattering in Al for electron energies between the primary energy and the critical ionisation energy of the Al Ka X-ray line. Furthermore, the quantitative depth resolution is calculated as the ionisation range of electrons [4].

The thinnest Al layer, for which a precise element quantification is still possible, is 150 nm thick measured with 3 keV electron energy. For comparison, the thinnest Al layer, which could be resolved lateral, was 280 nm thick. Regarding the simulated and calculated resolutions, there is a good correspondence with the measured data, except at 3 keV, where the measured resolution is worse (Fig. 1). We noticed the same discrepancy for the lateral resolution measurements already. The reason for this is not clear yet. Since the effect is the same for the lateral and for depth resolutions, any drift effect can be excluded. In the next step, depth resolution measurements will be extended to Ag and Au.

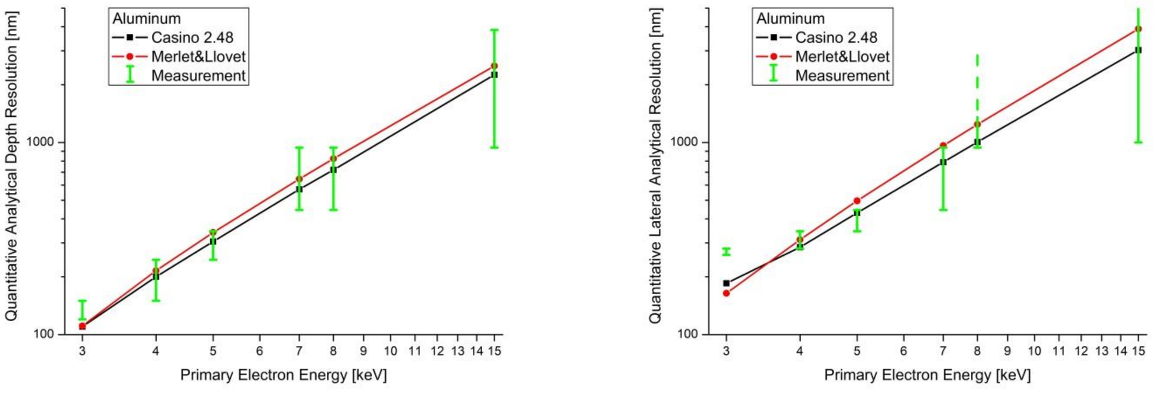

Figure 1. Comparison of simulated, calculated and measured depth and lateral resolutions at evaporated Al layers

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[3] Berger D and Nissen J 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **304** 012002 doi:10.1088/1757-899X/304/1/012002

[4] Merlet C and Llovet X 2012 *IOP Conf. Ser.: Mater. Sci. Eng.* **32** 012016 doi:10.1088/1757-899X/32/1/012016