# Functional Materials

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## Beam size and analytic resolution of the FEG-EPMA JEOL JXA-8530F

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The spatial resolution of EDX- and WDX- element analysis in the SEM is limited mainly by the size of the interaction volume. Therefore an Electron Probe Microanalyser (EPMA) with a thermionic electron emitter is sufficient in many cases. To increase the spatial resolution necessary for modern applications with nanostructures it is required to work with lower acceleration voltages (below 5 kV) of the primary electrons. Then the interaction volume is decreased efficiently and a higher spatial resolution is expected. But it should be kept in mind that electron beams from a thermionic source might not be focused very well for low acceleration voltages. Consequently, element analysis with the Field Emission Electron Probe Microanalyser (FEG-EPMA) is the method of choice.

Even for a FEG, the minimum achievable beam diameter is inversely proportional to the acceleration voltage. But the size of the interaction volume increases linearly with it. Therefore the acceleration voltage should be chosen very carefully to balance both effects and to achieve the best possible spatial resolution. Moreover the beam size depends on the beam current.

To get reliable information on the beam size of our FEG-EPMA JEOL JXA-8530F we recorded SE- and BSE-images of a gold-on-carbon high resolution sample by varying the accelerating voltage (3, 5, 8, und 15 kV) and the probe current in steps from 10 pA to 100 nA. Using the FEI-programme “Image”, we measured the contrast profile of the gold insulars edges in the SE- und BSE-images. The rise of these profiles (1 - 99 % criterion for comparison with Monte Carlo simulations) gives information on the probe diameter, e.g., 15 nm for 15 kV at 10 pA or 31 nm for 8 kV at 1 nA. Usually resolution measurements using profile scans are evaluated using the 16% - 84% criterium. But for the comparison with Monte Carlo simulations, the 1% - 99% criterium has to be chosen, since Monte Carlo simulations assume complete absorption of the electrons inside the sample. Results of the measured probe diameters are shown in “Figure 1”. These results accord with the data provided by JEOL.

In the next step the size of the interaction volume has to be regarded. Obviously it can not be measured directly. Therefore we made measurements on especially crafted model samples. For the first sample a silver layer (500 nm) was sputtered on a silicon wafer substrate. Furthermore, we sputtered a gold layer of 75 nm thickness and another 500nm Ag-layer (“Figure 2”). On the layers cross-section, a profile scan with 15 kV and 1nA was measured at a probe diameter of 22 nm. Considering the 1/99 criterion, we get a spatial resolution of 360 nm for the AuMα-signal (2.12 keV) and 200 nm for the AuLα-signal (9.68 keV) (“Figure 3.”).

Radial Xray distribution curves simulated with the Monte Carlo program Casino (version 2.48) confirm these results (“Figure 4.”). Decreasing the voltage to 5 kV, the current was 1 nA, we get an enhancement of resolution to 120 nm for the AuMα-signal. The estimated resolution using Monte Carlo simulation, however, is about 60 nm. Considering the probe diameter of 38 nm, we expect a resolution of about 100 nm. The measured resolution is worse due to fluorescence of AgLα-Xray (2.98 keV) widening the Au-Mα-signal ($M_{abs}$: 2.31 keV). In another test, we sputtered a 75 nm gold layer on a silicon wafer substrate. An additional 275nm thick carbon layer was deposited on the gold (“Figure 5”). In this case the cross-section profile scan was measured with 5 kV (1 nA) only. Having no fluorescence from Si-Kα (1.74 keV) we should get a better resolution and indeed 90 nm was achieved (“Figure 6”). Thus, measurement and Monte Carlo simulation (“Figure 7”) nearly coincide.

In conclusion, the FEG-EPMA has a very good analytical spatial resolution smaller than 100 nm significantly increased compared to EPMAs with thermionic electron emitter. To achieve the optimum analytical resolution the minimum beam diameter at low acceleration voltages and the actual size of the interaction volume has to be taken into account. The latter might be derived from measurements at model systems or by Monte Carlo simulations.

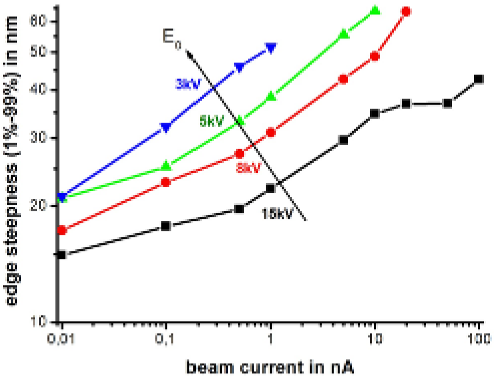


**Figure 1:** Probe diameter as a function of beam current and HV

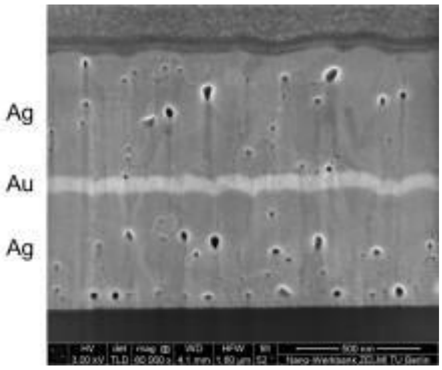


**Figure 2.** FIB-cross-section of Ag/Au/Ag-layers

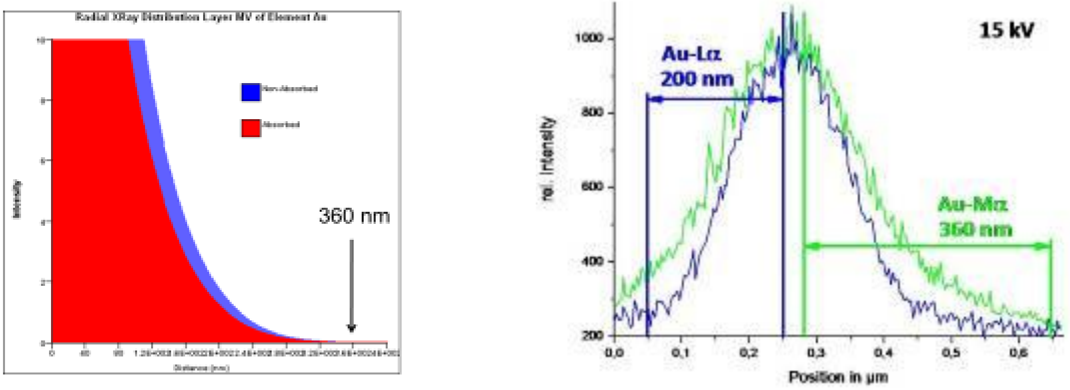
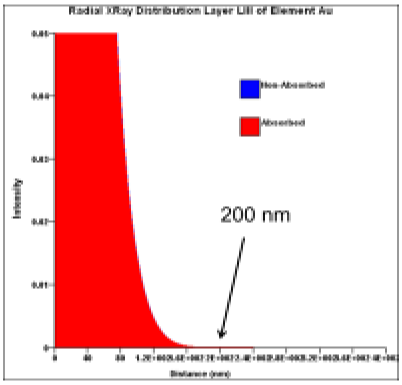


**Figure 3.** Profile scan on the Ag/Au/Ag-layers



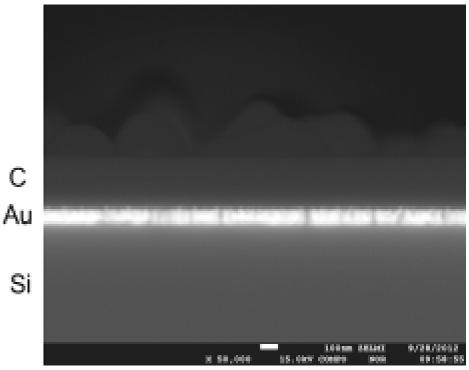
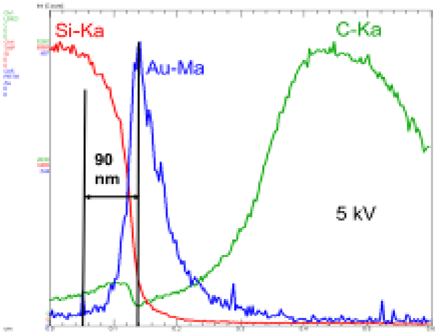


**Figure 4.** Radial Xray Distribution (RXD) simulation curves of AuMα and AuLα at 15 kV

**Figure 5.** Cross-section of Si/Au/C-layers



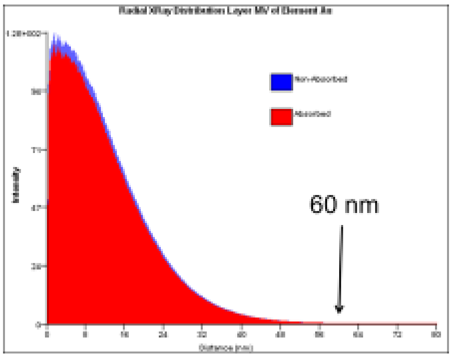


**Figure 6.** Profile scan on the Si/Au/C-layers

**Figure 7.** RXD simulation curve of AuMα at 5