In Situ TEM: The Performance of EDXS at Elevated Sample Temperatures

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Over the last years, *in situ* experiments in transmission electron microscopy (TEM) significantly increased thanks to the introduction of specimen holder systems using micro-electro-mechanical system (MEMS) chips to mount the samples [1]. One major improvement over furnace holders for heating is that they allow for analytical methods such as energy-dispersive X-ray spectroscopy (EDXS). Detectors can now collect information under low take-off angles, because of the wide field of view onto the sample. However, EDX semiconductor detectors are naturally susceptible to infrared radiation (IR), as the excitation of electrons to the conducting band requires only the energy of thermal photons. Thus - although MEMS devices are very efficient heaters for *in situ* TEM - the influence of the emitted heat on background signal, energy resolution, elemental quantification and mapping in EDXS is of great interest.

In order to determine the impact of IR on the spectral quality, we tested a windowless SDD (four quadrant Super-X inside an FEI Titan G2) in combination with two specimen holders, a Wildfire D6 and a Wildfire S3 by DENSsolutions. We did so by collecting the signal without an electron beam for a series of temperatures and holder tilt angles (Fig. 1). To evaluate the quality of EDX spectra at elevated sample temperatures, we prepared a specimen consisting of a 10 nm thin AuPd film, sputtered onto the supporting silicon nitride membrane of the MEMS chip. The system of AuPd on Si_3N_4 provides a sufficient number of characteristic lines that we examined for their stability. Avoiding the directional infrared radiation from the chips' surfaces [2] works by tilting away from the detector. We therefore simulated the shadowing behaviour of the specimen for characteristic X-rays based on the model by Kraxner et al. [3].

Starting at 580°C, infrared radiation began to prevail X-rays in terms of incoming counts on the detector system. At 650°C dead times exceeded 40%, rendering further heating unfeasible. Tilting towards the detectors would result in a lowering of those limits by around 20°C. Between 500°C and 600°C, the Si-K, Au-M and Pd-L lines broadened by up to 35% for the single-tilt holder (Fig. 2) and 60% for the double-tilt version. Especially in the low energy range, energy resolution decays at those temperatures and renders light elements undistinguishable from the background signal. Simulations for the shadowing of the single tilt holder predicted full illumination of the detectors for a convenient range of tilt angles (Fig. 3). The double tilt holder, where the MEMS chip lies embedded in a two-axis suspended cradle, is subject to improvement for EDXS.

References:

[1] Allard, L. F. et al. (2009), Microsc. Res. Tech., 72: 208 - 215.

[2] Weise, S. et al. (2016), Proc. SPIE 9752, Silicon Photonics XI, 97521E.

[3] Kraxner, J. et al. (2017). Ultramicroscopy, 172, 30 - 39.

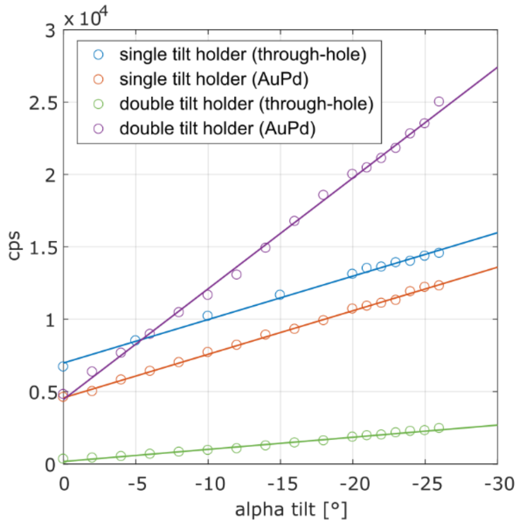

Figure 1: IR induced counts on detectors Q3 and Q4 without electron beam (T = 600°C).

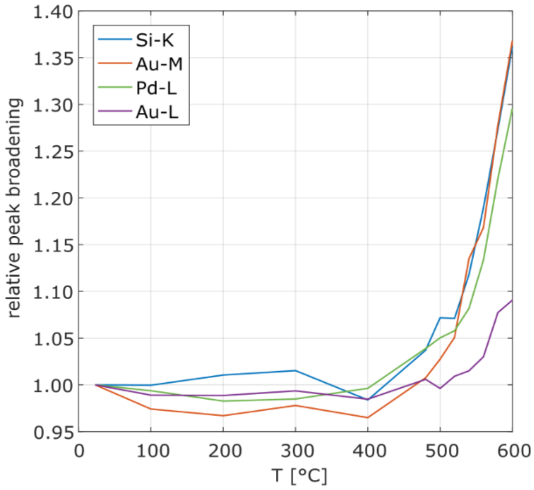

Figure 2: Relative spectral peak FWHM increase with respect to room temperature (detectors Q3 and Q4, single tilt holder, alpha tilt: -25°).

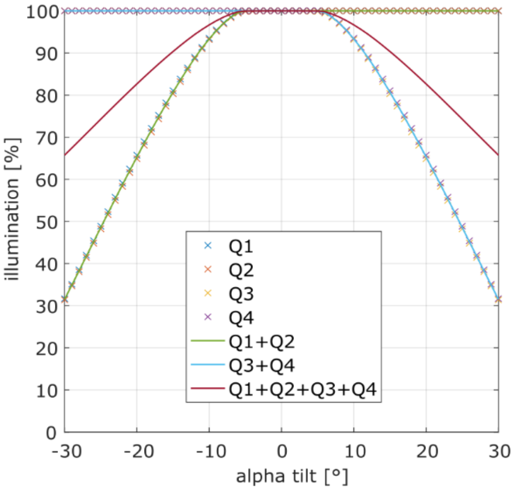

Figure 3: Simulated detector quadrant X-ray illumination for a series of holder tilt angles (single tilt holder).