Composition Analysis by EDS at Elevated Temperatures and More

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We discuss possibilities and challenges of energy dispersive X-ray spectroscopy (EDS) and EDS element mapping at elevated temperatures in SEM and STEM. An electron transparent Pd-Au-nanoparticle test structure on carbon film investigated in conventional STEM is one example. The agglomeration and evaporation of its nanoparticle material during heating from room temperature up to 1000°C were studied. Chemical phase analysis and simple statistical methods were applied to quantify the areal coverage of different species and its change with rising temperature. The stability of the heating holder in Z-direction during temperature change ensured very low drift and successful element mapping and phase tracking [1].

Heat radiation at elevated temperatures changes the low energy part of EDS spectra. We demonstrate, that element identification below 2kV and chemical phase analysis are still possible within certain limits. E.g. nitrogen can be detected up to 900°C (Fig.1). The results of phase and particle analysis (Fig.2) demonstrate the capabilities of in situ EDS in combination with statistical methods. Material agglomeration and evaporation of one involved species, the gold, during heating could be quantified by areal coverage values.

Further options of using EDS for in situ analysis are discussed. Speed of analysis defines, which processes can be usefully analyzed in situ by EDS. Optimum photon detection geometry is crucial for efficient, and thus fast, EDS in the electron microscope. Large solid and a high take-off angles for photon collection, proper collimation and suitable sample holders are desired. Multi-detector arrangements enable large solid angles at high take-off angles particularly well. The specimen holders should not shadow the detector(s). Holder and window materials and even reaction species sticking to them must be well known to ensure correct quantitative analysis of spectroscopic data, as e.g. in case of reaction cells holding liquid samples between electron transparent membranes. Just geometric optimization in combination with high brightness electron sources and aberration correction allows the identification of single atoms at low accelerating voltages in STEM within a few seconds during tracking [2]. This looks promising thinking of new challenging in situ experiments. Additionally, streams of constantly changing data require new ways of data acquisition and processing, which will be discussed.

References:

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[2] R M Stroud et al. Appl. Phys. Lett. **108** (2016) 163101.
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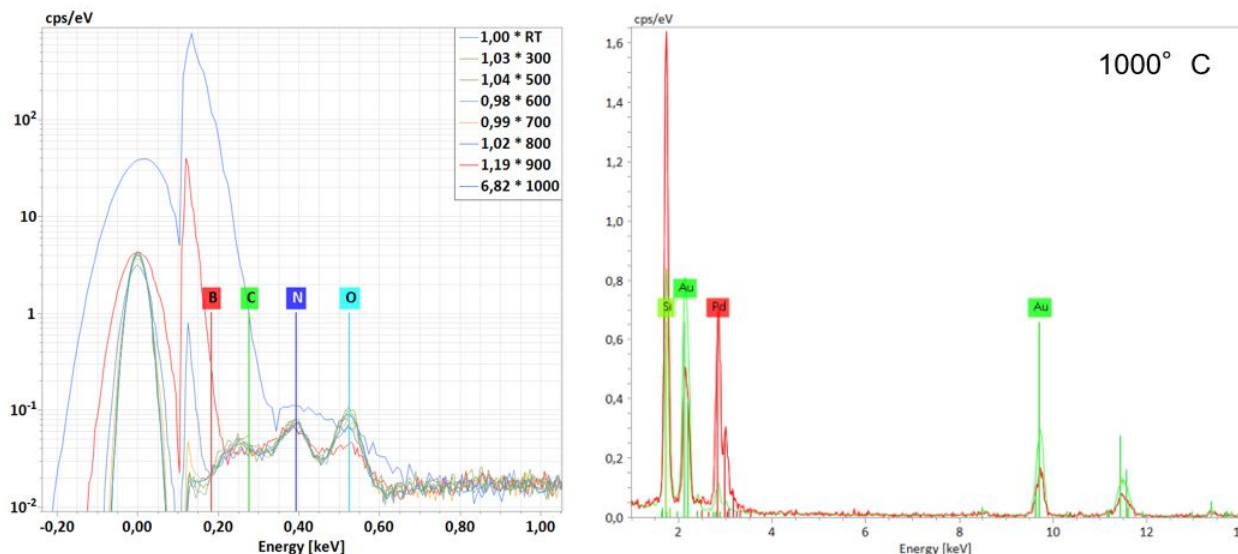
Figure 1. Left: The low energy part of EDS spectra from room temperature to 1000°C normalized to a background region around 1kV. Nitrogen (part of the substrate) can be identified up to 900°C. At 1000°C the elevated background intensity obscures the oxygen signal; Right: Spectra of the two phases of the test structure, Au = P2 (green) and Pd = P3 (red) at 1000°C. Si is part of the specimen holder.
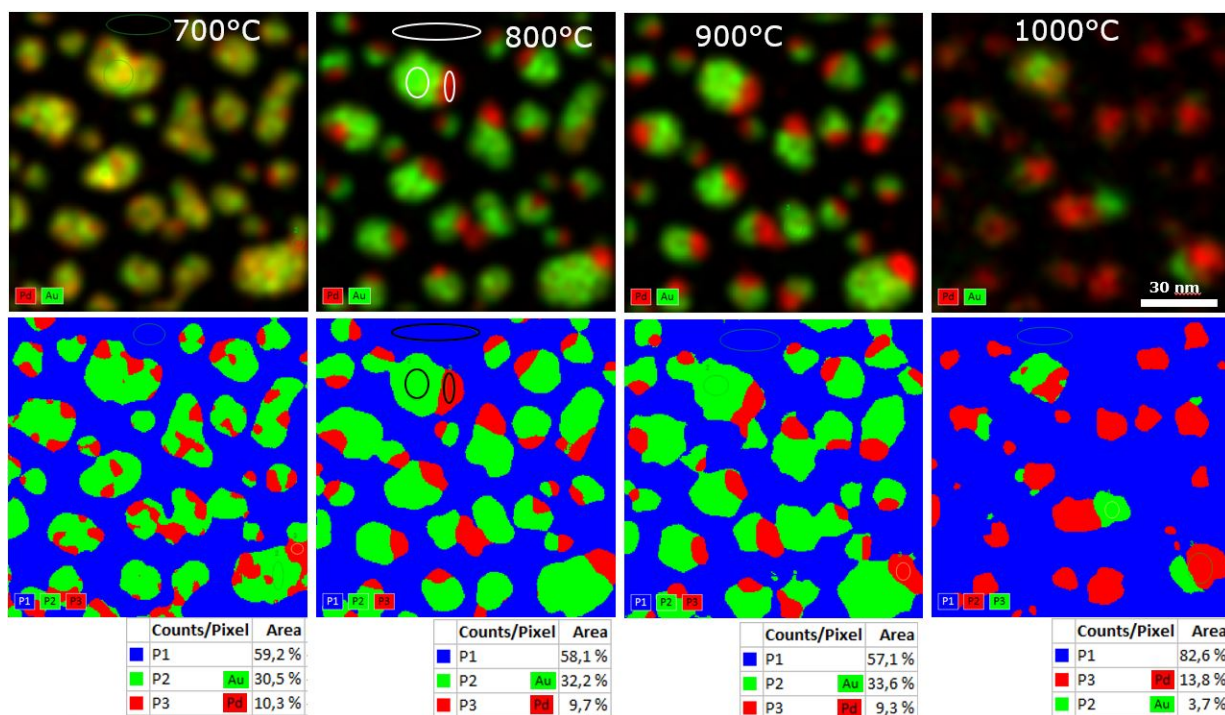

	Counts/Pixel	Area
P1		59,2 %
P2	Au	30,5 %
P3	Pd	10,3 %

	Counts/Pixel	Area
P1		58,1 %
P2	Au	32,2 %
P3	Pd	9,7 %

	Counts/Pixel	Area
P1		57,1 %
P2	Au	33,6 %
P3	Pd	9,3 %

	Counts/Pixel	Area
P1		82,6 %
P3	Pd	13,8 %
P2	Au	3,7 %

Figure 2. Element distribution and respective phase areal coverage at various temperatures. The regions used to define the three phases: exposed carbon film (P1), gold on carbon (P2) and palladium on carbon (P3) are marked in the second image. The tables below the images give the relative areal coverage of the identified chemical phases. Agglomeration takes place. Au starts to evaporate at higher temperatures, so its areal coverage is reduced at 1000°C while the exposed carbon film (P1) area is growing. The data show, how phase and particle analysis can be used to quantify in situ data.