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Fast STEM EELS Spectrum Imaging Analysis of Pd-Au Based Catalysts

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Metals such as Pd and Pt have been extensively used in the automotive industry as exhaust gas catalyst for pollution control. Until recently Au has never been used due to its thermal instability. In particular, Au particles at the nanometer scale show a melting point at relatively low temperatures that are much lower than those experienced during the catalytic process. However, Pd-Au alloys have attracted a lot of interest due to their resistance at high temperature and this explains their use in several fields such as the CO and hydrocarbon oxidation, the synthesis of Vinyl acetate monomer, hydrocarbon hydrogenation and many others. Pd is the catalytic center whereas the Au has the effect of changing the chemical properties at the surface of the Pd-Au alloy. These changes affect the catalytic properties. Hence, the study of the chemistry and the elemental distribution is important in order to understand the properties of the whole catalytic system. TEM related techniques have proved to be valuable tools for characterizing such materials.

Traditionally Pd-Au alloys or other particle systems containing heavy elements such as Au or Pt have been mainly characterized using pure STEM imaging and EDS for extracting some elemental distribution information. EELS has not mainly used as heavy elements shows they characteristic edges in the EELS spectrum at high-energy which results in a decreased signal-to-noise ratio therefore prolonged acquisitions would be required. However it is clear that analyzing such materials containing heavy elements using EELS would be beneficial given the wealth of information in the EELS spectrum over the EDS one. In addition of the simple elemental distribution which is also available with EDS, EELS is capable to deliver information such as absolute thickness, chemical phase, oxidation state, absolute composition, optical via the detection of surface plasmons, electrical conductivity, band gaps etc. These cannot be obtained using EDS. Over the past few years, there has been huge improvement in the instrumentation. Aberration corrected microscopes are now capable to generate so much current in a narrow probe that elemental maps from heavy elements can be acquired in a matter of seconds. The latest generations of EELS spectrometers are equipped with a camera that is so sensitive that EELS spectra from heavy elements can be acquired in a matter of milliseconds [1]. The extended dynamic range that can be obtained with the simultaneous acquisition of two different regions of the EELS spectrum suing the DualEELS™ [2] capability allows collect any information in the EELS spectrum.

To illustrate these advances, data were acquired at FELMI TU-Graz in Austria using a probe corrected FEI Titan G2 STEM operating at 300kV. The microscope is also equipped with a fully upgraded GIF Quantum ERS as EELS spectrometer. The sample analyzed consists of Pd-Au catalyst nanoparticles deposited on a carbon film supported on a Cu mesh TEM grid. EELS data were acquired in DualEELS™ mode and the regions of the EELS spectrum from 200eV to 2200eV and 1800eV to 3800eV were acquired using acquisition times of 7ms and 30ms respectively. Figures 1a,b show EELS spectra extracted from a region of the sample across a grain boundary. The EELS low and high core-loss spectra appear to be virtually noiseless due to the very high counting statistics in the data. The Pd $M_{4,5}$-edges at 335eV appear to be very strong as well as the Au $M_{4,5}$-edges at 2206eV in the high core-loss spectrum. It is also interesting to note there is sufficient signal that the high energy Pd $L_{2,3}$-edges at 3170eV are also well above the background and can be used for analysis if needed. Figure 2 shows the EELS colorized elemental map of Pd and Au. The quality of these maps is high enough that details such as the Pd diffusion into the Au can be observed.

These preliminary results demonstrate that when the EELS and STEM systems are properly configured, high-energy edges can be recorded with high signal-to-noise ratio using short exposure times. This illustrates the suitability of the EELS technique to study catalyst and materials containing heavy elements such as Au or Pt.

References:

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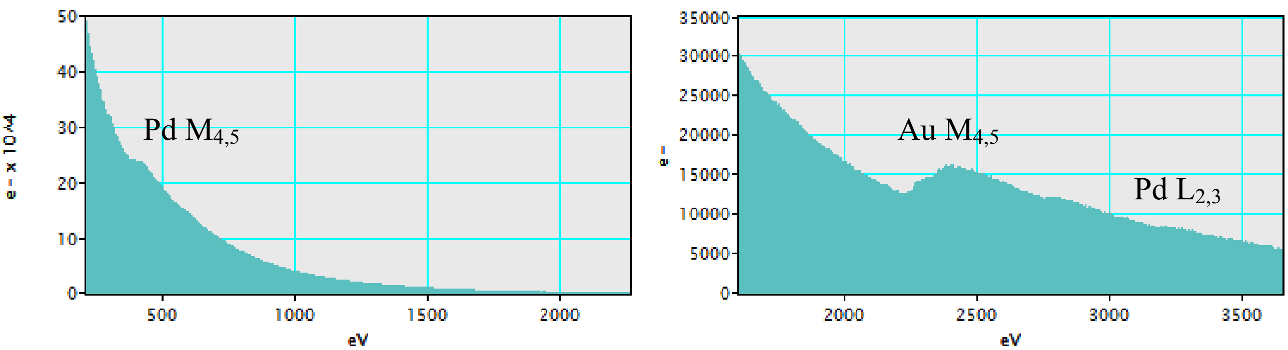
[2] A.J. Gubbens et al., Ultramicroscopy, 110, 962-970, 2010

[3] TEM specimen was provided by Professor Jianfang Wang's group at the Chinese University of Hong Kong



Figures 1a,b: Low and High core-loss EELS spectra extracted from the region in the sample across the gran boundary between two particles.

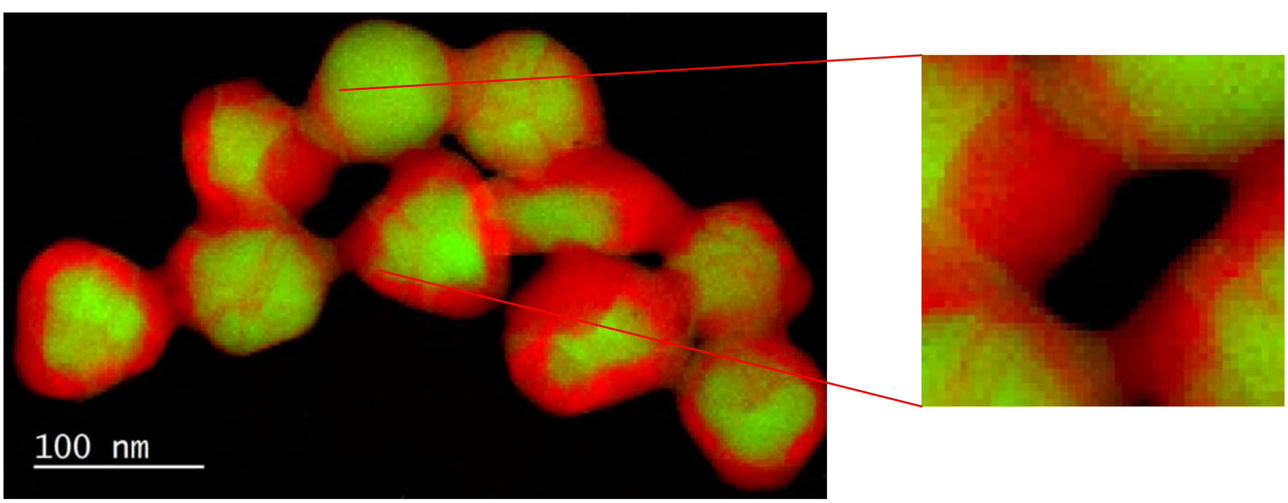


Figure 2: Colorized elemental maps of Pd in red and Au in green acquired using EELS. The inset is the enlarged image of the region in the box. The contrast shown in these maps is very high.

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