Multimodal and multidimensional EELS and EDX for investigations of catalysts at the nanoscale

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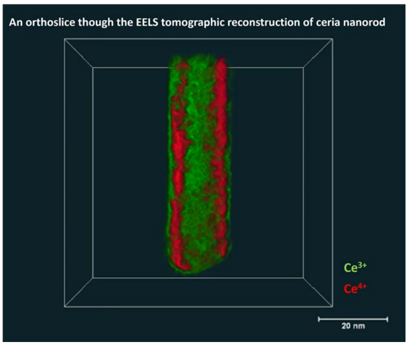
Ceria-based materials find their applications in various fields. Due to their oxygen deficiency at the surface the ceria nanoparticles are especially attractive catalysts [1]. By introduction of dopants into the ceria lattice the catalytic performance of ceria nanoparticles can be even further increased. To fine-tune the catalytic performance though the insights into the structural-properties relationships in the materials advanced TEM with spectroscopy should be applied. 2D and 3D multimodal STEM is of crucial importance to understand the structure and chemistry of doped catalysts at the nanoscale and to enable the targeted approach to the materials development.

In this work we combined EDX, EELS and electron tomography to investigate the local chemistry of Cu-doped ceria (Cu:CeOx) catalyst exhibiting superior performance for low temperature CO oxidation with respect to the undoped CeOx material. EELS both in 2D and 3D evidenced the elegant double core-shell structure of the undoped CeOx nanorod (see the figure), most likely related to the large amount of voids inside the particle: a shift of the Ce M4,5 edge onset as well as the white line ratio variation was clearly observed in the EELS spectrum allowing to distinguish between Ce4+ and Ce3+. In contrast to the previous work [1] where to gain the similar insights the monochromated 120kV setting was applied in the present study the experiment did not require the use of the excited monochromator and the lowered HT making the experimental work easier. Simultaneous EELS-EDX investigation was performed to study the Cu:CeOx material. Even though the CuL2,3 edge is visible in the EELS spectrum, its overlap with the fine structure of Ce inhibits the reliable STEM-EELS mapping of Cu in Cu:CeOx. EDX in this case clearly located Cu locations within the nanorod while STEM-EELS evidenced the Ce3+ oxidation state of cerium, highlighting the high amount of oxygen vacancies which can be correlated to the superior performance of the doped ceria catalyst. To sum up, both EELS and EDX giving the access to the local chemical information, in combination with the electron tomography, giving the access to the 3D information, are of great help while investigating catalytic nanoparticles. We highlighted here the unique insights of the multimodal and multidimensional approach by an example of doped ceria catalyst investigation.

Graphic:



Keywords:

EELS, EDX, Multimodality, Catalysis

Reference:

[1] Maria Meledina, Stuart Turner, Vladimir V Galvita, Hilde Poelman, Guy B. Marin, Gustaaf Van Tendeloo F, Nanoscale 7 (2015), p. 3196-3204. https://doi.org/10.1039/C4NR06060A