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Quantitative Evaluation of Supported Catalysts Key Properties from Electron Tomography Studies: Assessing Accuracy Using Material-Realistic 3D-Models

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Abstract : The ability of Electron Tomography to recover the 3D structure of catalysts, with spatial resolution in the subnanometer scale, has been widely explored and reviewed in the last decades. A variety of experimental techniques, based either on Transmission Electron Microscopy (TEM) or Scanning Transmission Electron Microscopy (STEM) have been used to reveal different features of nanostructured catalysts in 3D, but High Angle Annular Dark Field imaging in STEM mode (HAADF-STEM) stands out as the most frequently used, given its chemical sensitivity and avoidance of imaging artifacts related to diffraction phenomena when dealing with crystalline materials. In this regard, our group has developed a methodology that combines image denoising by undecimated wavelet transforms (UWT) with automated, advanced segmentation procedures and parameter selection methods using CS-TVM (Compressed Sensing-total variation minimization) algorithms to reveal more reliable quantitative information out of the 3D characterization studies. However, evaluating the accuracy of the magnitudes estimated from the segmented volumes is also an important issue that has not been properly addressed yet, because a perfectly known reference is needed. The problem particularly complicates in the case of multicomponent material systems. To tackle this key question, we have developed a methodology that incorporates volume reconstruction/segmentation methods. In particular, we have established an approach to evaluate, in quantitative terms, the accuracy of TVM reconstructions, which considers the influence of relevant experimental parameters like the range of tilt angles, image noise level or object orientation. The approach is based on the analysis of material-realistic, 3D phantoms, which include the most relevant features of the system under analysis.

Keywords : electron tomography, supported catalysts, nanometrology, error assessment
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