Transmission Electron Microscopic Characterisation of Catalysts

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Transmission electron microscopy (TEM) is undoubtedly one of the most important tools for visualizing the morphology, extracting crystallographic information and for local chemical analysis of industrial heterogeneous catalysts. It is nowadays possible to directly image the complex nanostructure of catalytic materials. Spectroscopic measurements performed in situ within the microscope provide elemental analysis and information on oxidation state and bonding. The development of in situ, controlled atmosphere instruments allows the study of working catalysts. The as a tool for catalyst characterization will be presented. A short preview is given in the following.

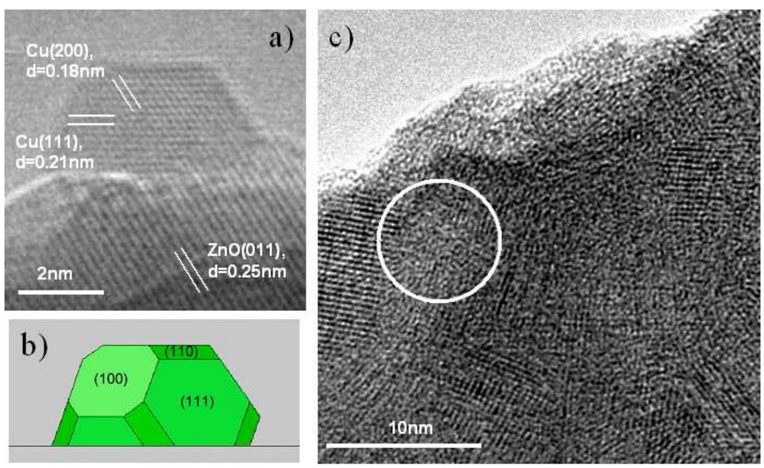

Figure 1: a: Lattice fringe image of metallic Cu particle supported on ZnO. b: Wulff construction of the imaged Cu crystal. c: Lattice fringe image of nanocrystalline molybdenum oxide. The circle emphasizes noncrystalline material embedded the nanocrystals.

The information of surface construction of metal particles is essential to understand the catalytic performance of the supported metal catalysts. As particles get larger than 20 Å, it becomes possible to discern their shapes and the nature of surface facets. In Figure 1(a) a so-called lattice fringe image of a Cu/ZnO catalysts for methanol synthesis is shown [1]. The lattice planes are clearly revealed in the Cu particle and ZnO support making it possible to assign the well-defined facets of the Cu crystal and the interface between the metal particle and the oxide support to low-index surfaces of Cu. From this information a Wulff construction is performed (Figure 1(b).

Another example of structural information which TEM can provide is shown in Figure 1(c). Nanocrystalline molybdenum oxides used as precursor to a catalyst for selective oxidation of propene is clearly revealed in the lattice fringe image. The image provides information on the local arrangement of the nanocrystals as well as the noncrystalline material observed to embed the molybdenum oxide nanocrystals [2].

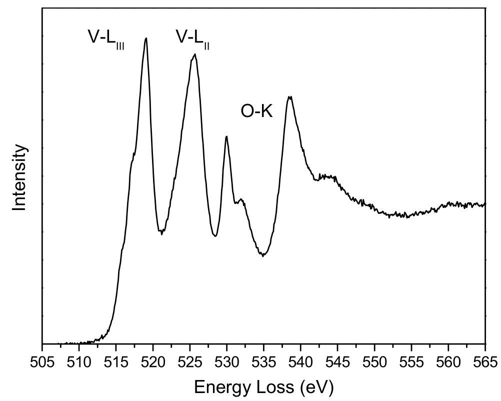

Figure 2: Electron energy loss spectrum of β-$VOPO_4$ showing the V L-ionization edges together with the O K-ionization edge.

Information on the electronic structure of catalysts can be obtained by electron energy loss spectroscopy (EELS) a well-developed technique equipped on TEM. The acquired spectra are complementary to EXAFS and provide similar information, but with high spatial resolution. An example of the type of information obtained can be seen in Figure 2 showing the vanadium L-ionization edges and the oxygen K-ionization edge of a β-$VOPO_4$-phase detected in catalysts used for selective oxidation of n-butane [3]. The fine structure (known as ELNES) observed at the ionization edges is correlated to the local density of empty states of the probed material and thereby provides information on the local electronic environment.

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References

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