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Porosity and Wetting Behavior in Model Systems for Hydrogen Storage

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Nanocomposites prepared by impregnation of activated carbon fibers with magnesium boronates or alanates doped with titanium catalysts are very promising materials for hydrogen storage. They exhibit a high hydrogen storage capacity and fast loading/unloading kinetics [1]. The reaction kinetics depend on the catalyst [2] as well as the morphology of the composite and the porosity of the support material. We have been using aberration corrected high resolution TEM and electron tomography [3] to characterize the porosity of the activated carbon fiber support. At the surface of the activated carbon fibers, we observed a highly disordered structure, presumably due to ball milling of the fibers. In contrast, electron tomography and HRTEM of the bulk material indicated extended linear pores in agreement with the models assumed for BET analysis (Fig. 1).
The stability of the boronates and alanates does not allow for a detailed morphological study without significant beam damage. Therefore we were using nickel (by reduction of nickel nitrate) and aluminum (by thermal decomposition of organo alumino compounds) composites to study the morphology and wetting behavior. Electron tomography and STEM-EDX analysis revealed a very uniform distribution of nickel nitrate and aluminum inside the pores of the activated carbon fibers after initial processing at mild temperatures. No crystalline structures could be observed for the nickel nitrate and the (oxidized) aluminum. However, during reduction by H_2 at 500C, the nickel has a very high mobility leading to the formation of crystalline nickel particles well dispersed inside the pores of the activated carbon fibers (Fig. 2). The average diameter of the nickel particles is ~ 3.7 ± 1.1 nm and thus significantly larger than the average pore diameter of ~ 2 nm. A quantitative analysis of the STEM-EDX results and the tomographic reconstructions is in good agreement with the nominal elemental composition of the composite assuming a complete penetration of the pore system by the aqueous/organic solution used during impregnation.

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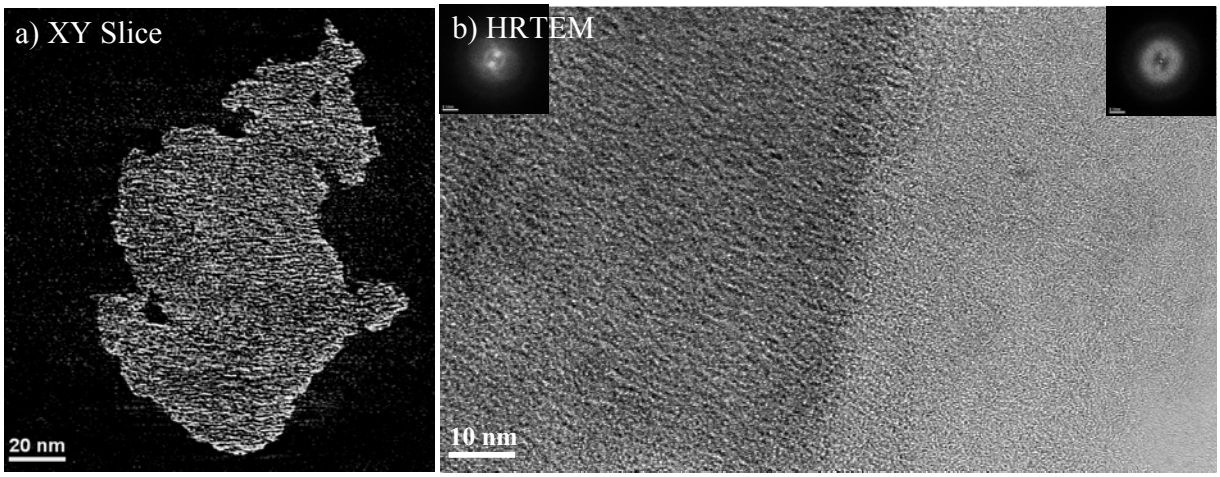

FIG. 1. a) XY slice through the tomographic reconstruction of an activated carbon fiber fragment revealing aligned extended pores with a diameter of 1~-3 nm,
b) HRTEM image at the edge of a fiber fragment exhibiting a disordered carbenous structure at the surface (right side) and aligned pores in the thick bulk part of the fiber (left side).

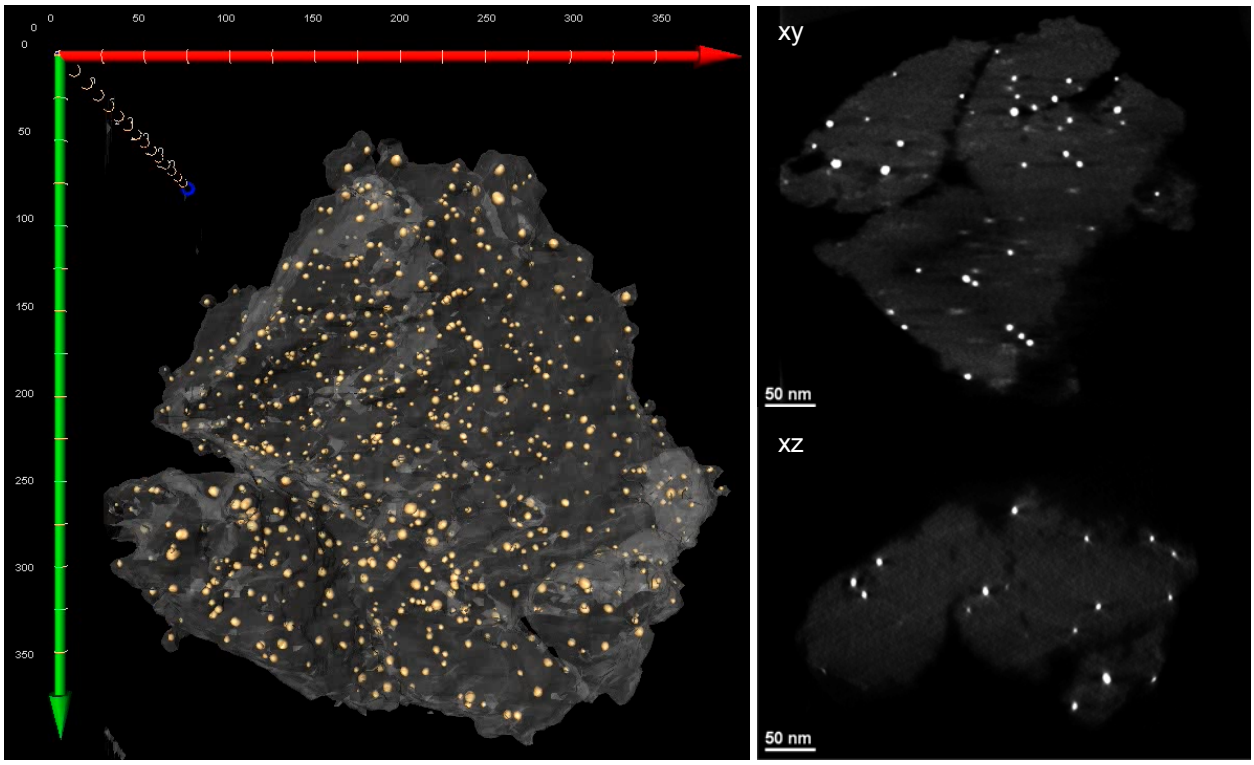

FIG. 2. HAADF-STEM tomographic reconstruction of the 3D distribution of nickel particles (average diameter of ~3.7 nm) inside an activated carbon fiber. In addition, some cracks and larger extended pores are also visible in the 3D reconstruction.

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