COMBINED MICRO-XRF/XRPD TOMOGRAPHY FOR THE CHARACTERIZATION OF FeCrAlY-FOAM-BASED CATALYSTS

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Steam reforming (SR) of hydrocarbons (i.e. natural gas, biomasses, alcohols) is the most commonly used method to produce hydrogen (H_2) on an industrial scale. Since this is an endothermic reaction, temperatures of about 1173-1273 K have to be maintained. These hard conditions have a destructive effect on the reactor tube material and shorten its life time. Catalytic partial oxidation (CPO) can be used instead of SR for small-scale H_2 production. This slightly exothermic reaction has to be performed at ultra-high space velocity to prevent total oxidation. Metallic foams such as FeCrAlY are promising catalyst supports for both SR (because of its thermal stability and conductivity) and for CPO (because of its high porosity).

The catalyst can be deposited on this support in two ways: (a) wash-coating of calcined foam with bohemite in HNO_3, followed by impregnation with a hydrotalcite-type (HT) of catalyst precursor (b) direct electrochemical deposition (ECD) of the HT precursor, a method used for the preparation of modified electrodes.

A combination of X-Ray Fluorescence and X-Ray Powder Diffraction on a micro scale has been employed to visualise in a completely non-invasive manner the layered structure of the catalyst after impregnation and its distribution on the foam-walls in both methods. XRF and XRD tomograms show virtual two-dimensional cross-sections of foam-pillars in which the elemental and crystal phase composition of the catalyst layers are visualized. In order to obtain these maps, Pawley fitting of thousands of two-dimensional diffraction patterns collected with an area detector was performed. During this procedure, the relative intensities of diffraction lines belonging to one crystallographic phase are kept constant and the remaining phase scaling factors allow to visualize the distribution of that phase after tomographic reconstruction with an MLEM algorithm. XRF spectra that are recorded simultaneously with the XRD data by means of two SDD-detectors are fitted to an analytical model in order to extract the net intensities of the element specific X-ray lines; these net data are used to construct element specific tomograms.

In all cases, it could be observed that an Al_2O_3 layer was formed between foam and catalyst, as intended in order to ensure catalyst phase dispersion and protection of the metallic foam under industrial conditions from corrosion, oxidation effects, etc. The formation of the active catalyst phase [$Ni(Mg)(Al)_2O_4$, $Ni(Mg)O$] from the HT precursor could be demonstrated. By means of the ECD method, thinner layers of the catalyst could be deposited than by means of the conventional wash-coating approach.