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MS-1-P-5833 Combining electrons and ions: a (S)TEM-FIB microscopy application to unveil the final surface structure of a washcoated monolithic catalyst

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The use of structured materials consisting of catalyst-coated honeycomb-type monoliths offers some advantages in comparison with the powder catalysts: e.g. higher exposed surface area and/or improvement of the active site-reactant contact. The dip-coating is the most extended procedure to load the powdered catalyst onto the monolith. Although the structural and chemical characterization at the nanometer scale of powder catalysts by (scanning) transmission electron microscopy, (S)TEM, techniques is in most cases well established, the characterization at such scale of catalytic devices, as it is the case of coated monoliths, poses currently a few challenges, some of them related to basic strategies to obtain representative information from the observations and others related to the sample preparation steps. In any case the approach is mandatory to determine the influence of the preparation procedure used to load the monolith on the final structure of both the active catalyst powder and of the coating itself.

There are several techniques to characterize devices at the micrometer scale but they present some disadvantages related to get a sample without mechanical damages. More recently, Focused ion beam (FIB) offers a solution of this particular (S)TEM characterization challenge, because it allows extracting precisely positioned, nanometer-sized sections of this type of devices, suitable for high resolution studies by different (S)TEM techniques. In this contribution we report how the combination of scanning electron microcopy (SEM)+(S)TEM + FIB studies yields valuable information from specific areas of a Co3O4/La-modified-CeO2 catalyst deposited by means of washcoat techniques on honeycomb cordierite (400 cells/in2) and Fe-C-based monoliths, designed for catalytic combustion of volatile organic compounds.

X-EDS maps in STEM-mode provided detailed information about the spatial distribution of these components. Remarkable, a Co3O4 layer was detected the external surface of the washcoat surrounding an inner core made up of an ensemble of the nanosized CeO2 support crystallites, suggesting that during the washcoating process the two components of the initial active catalysts, a ceria-supported cobalt oxide, segregate in space giving rise to a stratified structure in the coating layer of the catalytic device, far too different from the initial powder in which the two components were intimately mixed. These findings were confirmed in both cases, ceramic or metallic monolithic-supports. We remark the potential of the combined FIB-STEM characterization to give detailed important information about the surface of the monolith, which it is not possible to obtain by macroscopic characterization techniques and even by Electron Microscopy techniques.

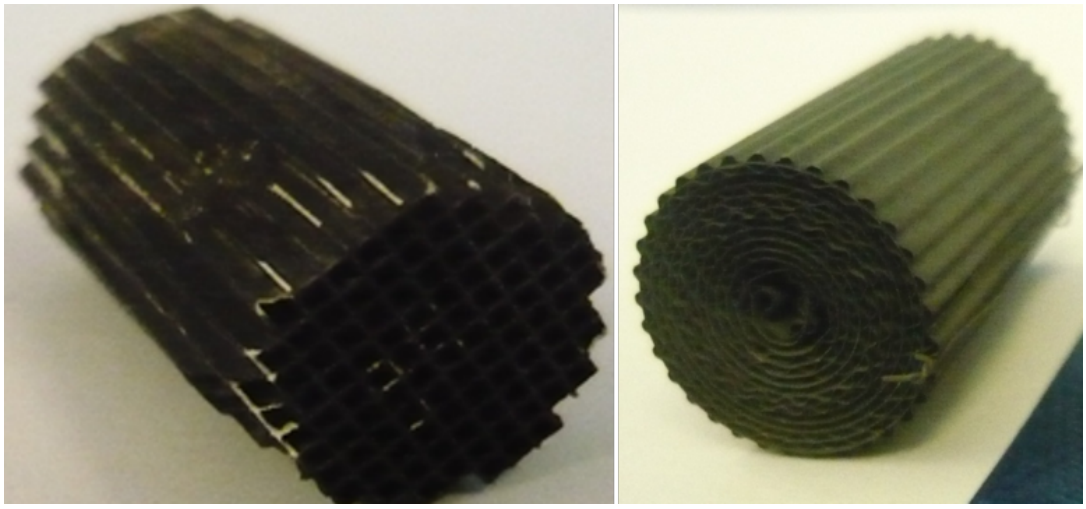

Fig. 1: Catalyst-coated ceramic (left) and metallic (right) honeycomb-type monoliths.

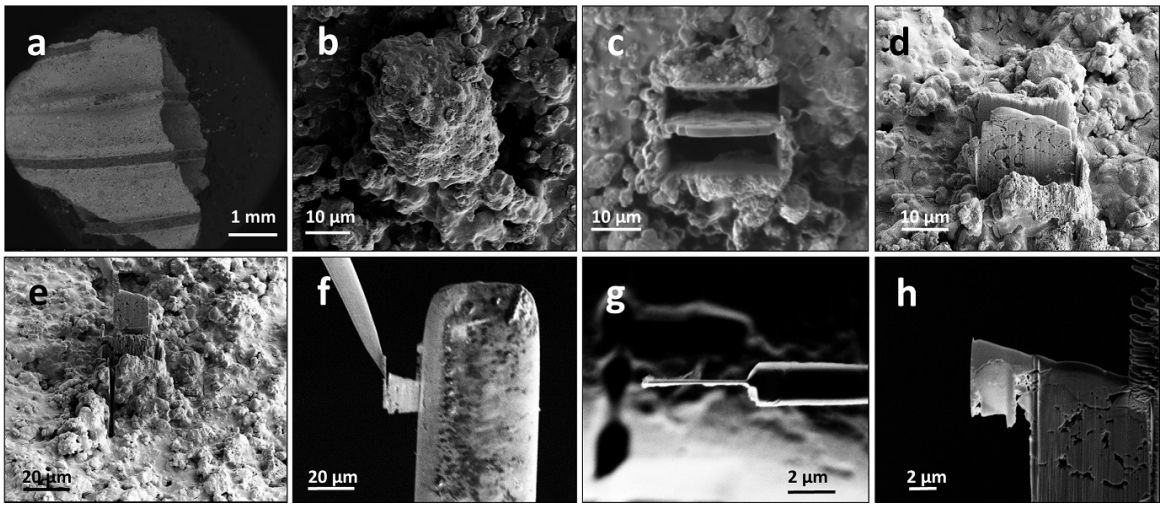

Fig. 2: SEM images recorded at the different steps of the preparation of the FIB sample of the monolith washcoated. Adapted from Hernandez-Garrido et al. J Phys Chem C, 117(25):13028. Adapted with permission.

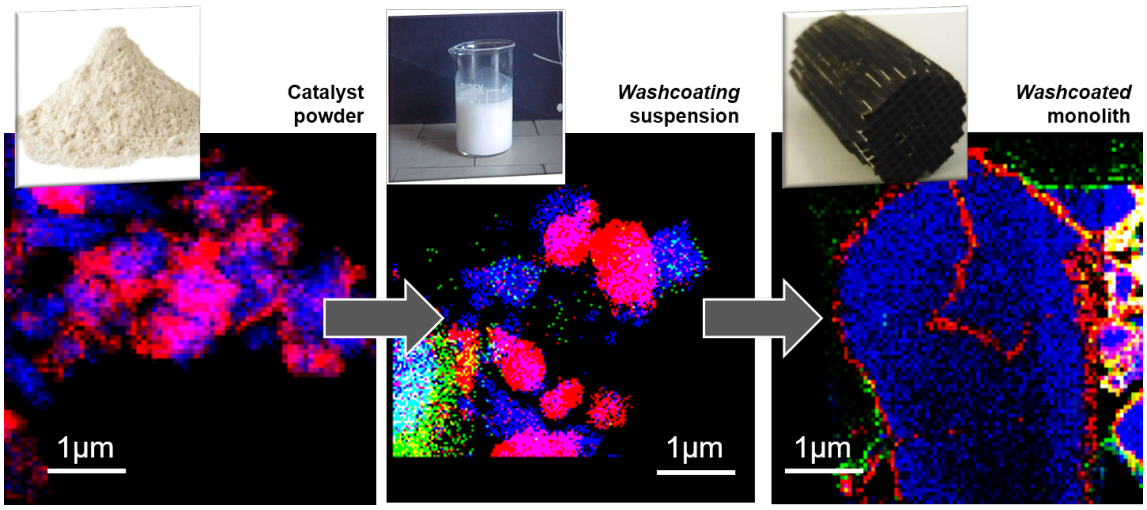

Fig. 3: X-EDS element (Co, Ce and Al) distribution maps recorded on the catalysts powder (a), the waschcoating suspension (b) and the final FIB sample from the ceraminc monolith. Adapted from Hernandez-Garrido et al. J Phys Chem C, 117(25):13028. Adapted with permission.