	Experiment title: Characterization of a novel industrial cracking catalyst for oil refinery via near-field ptychographic X-ray Computed Tomography	**Experiment number**: ma5295
Beamline: ID16A	**Date of experiment**: from: 02/03/2022 to: 06/03/2022	**Date of report**: 22/03/2023
Shifts: 12	**Local contact(s)**: Federico MONACO	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
* Julio César DA SILVA; Institut Néel (CNRS), Grenoble, France
* Anico KULOW; Institut Néel (CNRS), Grenoble, France
* Mohamed Redhouane BOUDJEHEM; Institut Néel (CNRS), Grenoble, France
Nils BLANC; Institut Néel (CNRS), Grenoble, France
Alla DIKHTIARENKO; KAUST Catalysis Center, Thuwal, Saudi-Arabia
Jorge GASCON; KAUST Catalysis Center, Thuwal, Saudi-Arabia
Samy OULD-CHIKH; KAUST Catalysis Center, Thuwal, Saudi-Arabia
Tuiana SHOINKOROVA; KAUST Catalysis Center, Thuwal, Saudi-Arabia

Report:

Objective of the beamtime

The objective of this beamtime was the investigation of the microstructure and pore network of the new ACM-101 Fluid Catalytic Cracking (FCC) catalyst for oil refinement with and without the addition of ZrO_2, in three different states (fresh, spent and regenerated) via near-field ptychographic X-ray computed tomography (nf-PXCT). This method has the invaluable advantage that large fields of view can be measured with a high resolution in the range of tens of nanometers. The information obtained by these measurements helps to better understand the processes during the catalysis, for example, the formation of coke. The catalysts are composed mainly of ZSM-5 and USY zeolites, Al_2O_3 binder, kaolin clay, and SiC additives. The addition of ZrO_2 allowed to increase in the performance of the catalyst in terms of selectivity to light olefins and hindering unwanted coke formation during use.

Measurements

We have measured six single particles of ACM-101 mounted on tomography pins by nf-PXCT, which allowed us to image the whole particles of diameters between 30 µm and 70 µm with pixel sizes between 25 nm and 40 nm. Additionally, we have performed an nf-PXCT scan on a classical, commercially available FCC catalyst, and an X-ray fluorescence tomography scan of a small region of a spent ACM-101 catalyst particle with added ZrO_2. Before each measurement, test scans were performed to optimize the parameters. Each nf-PXCT consists of the acquisition of 16 inline holograms of the sample at 1400 to 1800 different angles, divided into 4 sub-tomograms. Leftover time was used to perform Holotomo-Scans on two different samples.

Data Analysis

Several data analysis steps are necessary to obtain the measured samples' quantitative electron density volumes. The acquired data set consists of diffraction images that are used to reconstruct the sample's complex transmittance, which was done with PtyPy, a freely available, worldwide-used python software for ptychography. Further, we worked with the phase images obtained in this way. After careful phase ramp removal, phase unwrapping and alignment, the tomographic reconstruction is performed with Toupy, a python software package designed for the tomographic processing of pytchographic data sets. In this way, we obtained the 3D volumes of the six different ACM-101 catalyst particles (three ortho slices through the delta-tomogram of the ACM-101 spent are shown in Figure 1) with resolutions around 60 nm.

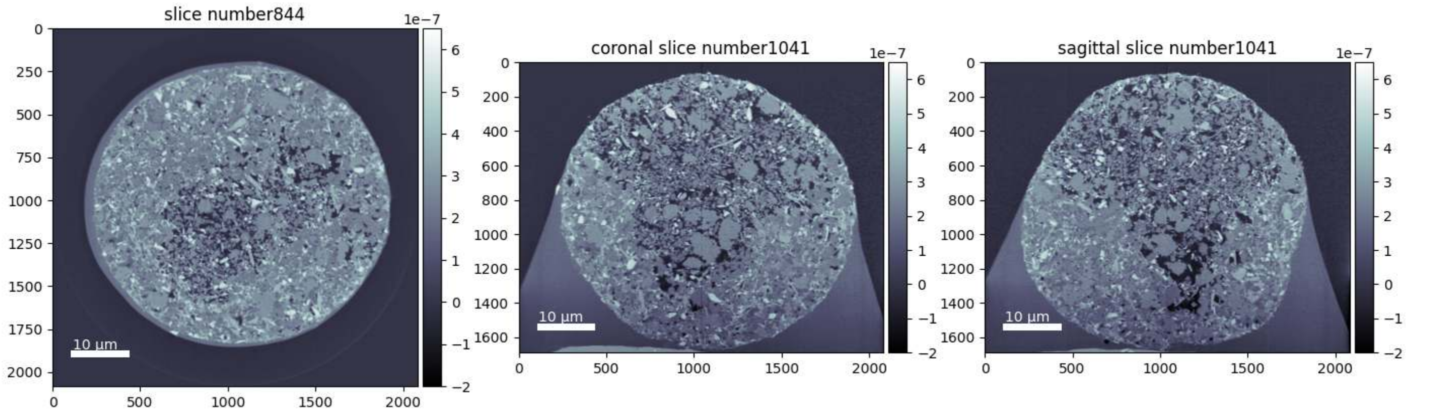

Figure 1: Orthoslices through the delta tomogram of the ACM-101 spent. The glue that has been used to mount the catalyst particle on the tomographic pin is clearly visible at the bottom edges of the sample. The microstructure in the sample is clearly visible, although at some places, the glue has penetrated.

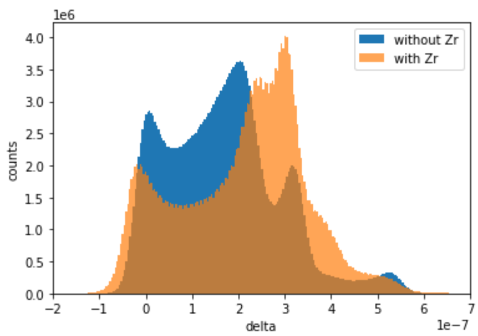

Figure 2: Delta histograms of a region of the delta tomogram of the ACM-101 regenerated in a region without glue with (orange) and without (blue) added ZrO_2.

A first analysis of the delta values of the regenerated ACM-101 with and without added ZrO_2 shows that the delta values (which are proportional to the electron density) do change considerably for the two considered catalyst particles. Peaks of four different components are discernible (most probably pores, zeolites, Al_2O_3 binder and kaolin clay), that are affected differently by the addition of ZrO_2, showing a preference for this additive to bind to one component. The non-apparition of an additional peak at high delta values is a clear hint that ZrO_2 does not build clusters/agglomerations with sizes of the voxel size or larger.

More detailed analysis, such as quantitative pore volume analysis, and the comparison between the particles at different lifetimes, is in progress with the segmentation of the delta tomograms.