Agglutination of Single Catalyst Particles during Fluid Catalytic Cracking as observed by X-ray Nanotomography

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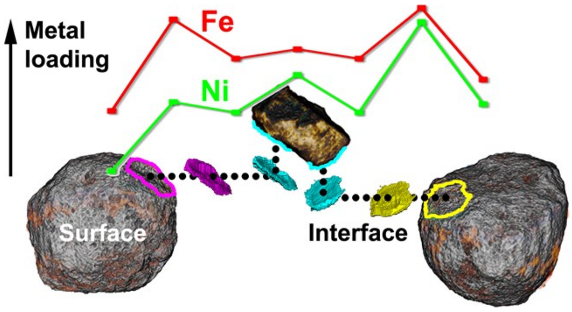
Supporting information

Movie S1:
Animation showing the TXM tomography data. Optical densities of the two large particles are rendered using a grey to white color scale, the middle particle using a black to grey color scale. The red to white color map represents relative Fe concentrations; the blue to green color map indicates relative Ni concentrations.

Movie S2:
Zoom of the interface region between left, middle, and right particle. The left and right particles are cut for better visibility of the interface area and rendering colors are set as in movie S1. The movie clearly shows the enhanced Fe and Ni concentrations at the surface of the left and right particle as well as throughout the middle particle. In the last third of the movie the middle particle is completely removed to uncover the enhanced metal concentrations around the contact areas on the surface of the left and right particle.

Table of contents figure:
X-ray nanotomography of a whole FCC particle cluster suggests that elevated interfacial metal concentrations are responsible for E-cat particle agglutination.



Differential absorption contrast imaging

The image contrast obtained in transmission X-ray microscopy (TXM) is a function of the X-ray attenuation coefficient. The value of this attenuation coefficient is determined by X-ray energy, the elemental composition of the sample, and its density. At element specific X-ray energies the X-ray absorption shows an abrupt increase that is known as the X-ray absorption edge jump of that specific element. The magnitude of this edge jump, measured as the difference between the X-ray absorption below the edge jump and the X-ray absorption above the edge jump, is directly proportional to the elemental concentration (i.e. the number of absorbing atoms) and thus can be utilized for element specific imaging. A map of the relative elemental concentration is obtained by subtracting 2D images or 3D volumes collected above and below the X-ray absorption edge of a specific element. The resulting image contrast is then exclusively caused by the concentration of the element of interest and in this way provides a direct measure of the relative (2D or 3D) elemental concentration distribution within the sample[9]. The elemental concentrations are relative and not absolute (i.e. they cannot be provided in units of mass per volume (mass concentration, kg/m3) or number concentration (e.g. ppm), as this would require a calibration of the instrument (as, for example, done in quantitative XRF analysis). However, the relative elemental concentrations can reliably be compared with each other, and provide, for example, a measure of Fe to Ni ratios or changes in elemental concentrations across the sample.

In this work, data collection was performed in mosaic tomography mode and the whole cluster was imaged with 180 2D projection images using single angle steps. Projection images were recorded using a dwell time of 1 second and 10 repetitions. The set of projection images was subjected to tomographic reconstruction using an iterative algebraic reconstruction technique (i-ART)[S1]. X-ray nano-tomography was performed below and above the Fe K edge (7.100 keV and 7.132 keV, respectively) and below and above the Ni K edge (8.325 keV and 8.350 keV, respectively). Relative 3D elemental concentration distributions were then determined as the difference in absorption contrast between the data collected below and above the respective X-ray absorption edge.

References:

S1. (a) Y. Liu, F. Meirer, P. A. Williams, J. Wang, J. C. Andrews, P. Pianetta, *J. Synchrotron Rad.*, 2012, **19**, 281–287. (b) Y.J. Liu, P.P. Zhu, B. Chen, J.Y. Wang, Q.X. Yuan, W.X. Huang, H. Shu, E.R. Li, X.S. Liu, K. Zhang, H. Ming, Z.Y. Wu, *Phys. Med. Biol.,* 2007, **52**, L5–13.