Rapid Tomography in Environmental TEM: Solutions for a Fast Analysis of Nano-Materials in 3D under In-Situ conditions

Siddardha Koneti\textsuperscript{1}, Lucian Roiban\textsuperscript{1}, Thomas Grenier\textsuperscript{2}, Voichita Maxim\textsuperscript{2}, Anne-Sophie Gay\textsuperscript{3}, Florent Dalmas\textsuperscript{1} and Thierry Epicier\textsuperscript{1}

\textsuperscript{1}. Univ Lyon, INSA-Lyon, Université Claude Bernard, CNRS UMR 5510, MATEIS, Bât. Blaise Pascal, 69621 Villeurbanne, France.
\textsuperscript{2}. Univ. Lyon, INSA-Lyon, CNRS UMR 5220, Inserm U1044, CREATIS, Bât. Blaise Pascal, 69621 Villeurbanne, France.
\textsuperscript{3}. IFP Energies nouvelles –Rond-point de l'échangeur de Solaize - BP 3, 69360 Solaize - France

Environmental Transmission Electron Microscopy (ETEM) in a dedicated instrument has been the subject of recent considerable developments allowing to follow chemical reactions under environmental, e.g. gas and temperature conditions even at atomic resolution [1]. A typical domain of applications concerns catalysis, where supported nanoparticles (NPs) can be followed during synthesis and evolution (activation / de-activation) in the presence of various gases. Whereas numerous works have now been published in conventional imaging, that is, 2D projection, little work is reported on 3D investigations performed under in situ. It is easy to understand why such an approach is difficult: since TEM tomography consists in reconstructing numerically a tilt series of images projected over a wide angular range, the time to acquire these data is generally too important as compared to the speed of the sample evolution, or the kinetics of the studied chemical reaction. The duration of the conventional acquisition step is typically one hour, or a fraction of one hour, making it impossible to get tilting sequences where the object does not experience any significant shape change which obviously hampers 3D reconstruction.

In this work we aimed at understanding the possibilities and limitations for acquiring fast tilt series under BF-TEM conditions in terms of speed and proper signal to noise ratio with the help of equipments available today. The main interest is to reduce the acquisition time from few tens of minutes to few mins or even to seconds. Vivid nano-materials were studied to estimate and discuss the possibilities for rapid tomography under environmental conditions. Firstly, simulations performed on a ghost model will address the question of the blur induced by a continuous rotation while recording images. Secondly tomographic results on alumina catalysts will be shown to understand the capabilities of a rapid tomography approach (Palladium nano particles grafted on alpha and delta alumina, see Figure 1). Thirdly the possibility and reliability of using rapid tomography approach under environmental condition will be demonstrated with the example of nanoactylasts for nano-particulate reduction in exhaust gases from car Diesel engines [2]. The 3D evolution of soot onto ZrO\textsubscript{2} catalysts is followed during its consumption under 1.7 mbar of oxygen at different temperatures.

This fast tomography approach open the way to study beam sensitive materials like polymers and biological samples even at 300 keV without any special treatments. As a further illustration, tomographic studies performed on polymer (Mg\textsubscript{3}AlCO\textsubscript{3} LDH nano-platelets dispersed in Latex shown in fig 2) and on biological sample (Magnetotactic bacteria) will also be shown.
Figure 1. TEM reconstructed volumes of δ-alumina supporting Pd NPs (shown in color). Left: conventional bright field tomography acquired in 45 minutes; middle: fast tomography sequence acquired in 150 seconds with a continuous tilt approach (both sequences recorded under high vacuum over an angular amplitude -74 to 66°). Right: comparison of the very similar Pd particle size 3D measurements (mean diameter respectively 4.1 and 3.9 nm for the conventional and fast tomography).

Figure 2. “Fast tomography” reconstructed volume of a LDH-filled latex nanocomposite: a) 3D model, b) extracted orthoslice and high resolution details of c) crystallographic structure and b) interlayer spacing of LDH platelets dispersed in the latex matrix.

References


Acknowledgements

We thank the INSA BQR “SPEED3D” and ANR “3DClean” (n°15-CE09-0009-01) projects for financial support. This work was supported by the LABEX iMUST (ANR-10-LABX-0064) of Université de Lyon, within the program "Investissements d'Avenir" (ANR-11-IDEX-0007) operated by the French National Research Agency (ANR).