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IT-10-O-2812 Fast tomography acquisition for in situ 3D analysis of nanomaterials under variable gas and temperature conditions in Environmental-TEM

EPICIER T.[1, 2], ROIBAN L.[1], LI S.[2], AOUINE M.[2], SANTOS AIRES F. C.[2], TUEL A.[2], FARRUSSENG D.[2]

[1]MATEIS, INSA de Lyon, Université Lyon I, 69621 Villeurbanne Cedex, France,
[2]IRCELYON,Université Lyon I, 2, Av. A. Einstein, 69626 Villeurbanne Cedex, France

Email of the presenting author: lucian.roiban@insa-lyon.fr

In the last two decades, tilted tomography in a transmission electron microscope (TEM) has become a widely used approach in order to quantify the three dimensional (3D) distribution of features in materials and nanomaterials[1, 2]. During the tilt series acquisition, a projection of the area of interest is recorded at each angle over a large angular amplitude, the final resolution along Z axis being directly related to the maximal tilting angle. The tilt series acquisition is usually performed automatically; depending on the employed acquisition method (automatic focusing, and cross-correlation based tracking), the total acquisition time typically ranges between 30 minutes to several hours. Such conditions are totally incompatible with in-situ experiments, where the materials are subject to changes under external mechanical or electrical solicitations as well as variable temperature and gas flow. Following the 3D evolution in such a context can be attempted by a ‘before/after’ strategy, where a first tomography analysis is performed on the object prior to any solicitation, then a second one after the solicitation as performed to track fuel cell nanocatalysts during electrochemical aging [3]. The recent development of commercial Environmental TEM (ETEM) [4] offers a wide range of in situ environmental studies of nanomaterials, such as oxidation / reduction at high temperature: this opens new opportunities to (try to) investigate in situ the 3D structure of nanomaterials. In this context, we are currently optimizing a fast acquisition method for tomography studies, based on video acquisition of tilted series in less than 1-4 minutes. We have applied this approach to the study of metallic Ag nanoparticles (NPs) encaged in silicalite hollow shells (silica-cages) for application in selective catalysis [5]. Single-tilt tomography and ETEM experiments were performed on a Cs-corrected TITAN ETEM, 80-300 kV, recently installed at CLYM in Lyon. Results are illustrated by figures 1 (fast acquisition performed over an angular amplitude of 116° in 3 minutes and 40 seconds) and figure 2 (ETEM experiments up to 700°C and oxygen partial pressure of 2 mbar). **References** [1] P.A. Midgley, R.E. Dunin-Borkowski, Nature Mat., 8 (2009) 271-280. [2] T. Epicier, chap. 3 ‘Imagerie 3D en mécanique des matériaux’, ed. J.Y. Buffière, E. Maire, Hermès - Lavoisier, Paris, (2014). [3] J. Jinschek, Microscopy and Analysis, Nanotechn. Issue November (2012) 5-10. [4] Y. Yu, H.L. Xin, R. Hovden, D. Wang, E.D. Rus, J.A. Mundy, D.A. Muller, H.D. Abruña, Nano Lett., 12 9 (2012) 4417-4423. [5] S. Li, L. Burel, C. Aquino, A. Tuel, F. Morfin, J.L. Rousset, D. Farrusseng, Chem. Comm. 49 (2013) 8507-8509.

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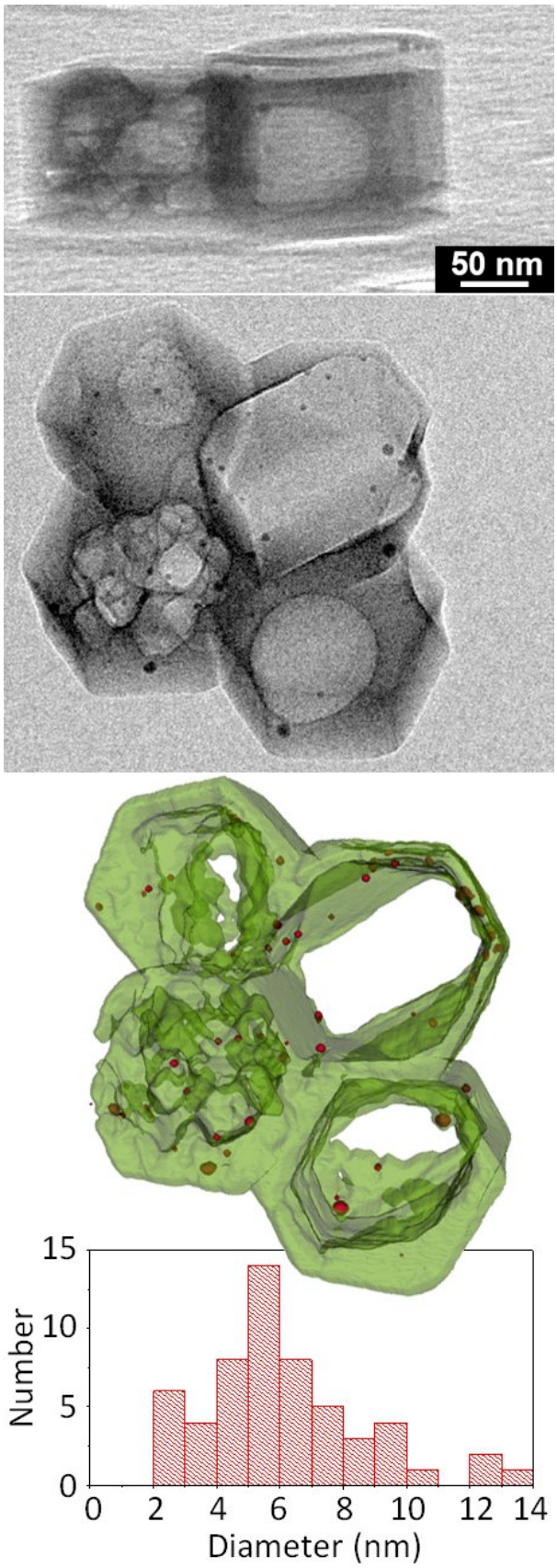

Fig. 1: Fast single-tilt tomography; a-b): video frames extracted at 78° and - 38.5° from a continuous tilting series acquired in bright field mode in less than 4 minutes; c): surface rendering of the silica-cages (green) and size histogram of Ag NPs (red); only 3% are outside of the silica cages. Acquisition conditions: high vacuum, 20°C, 300 kV.

Fig. 2: a): Assembly of silica cages containing Ag NPs at 20°C under high vacuum; b): same area at 700°C under high vacuum: the Ag NPs have grown but are mostly still inside the silica-cages; c): other area at 450°C under 2 10-2 mbar of O2 flow: note that all Ag NPs are out of the cages on the carbon supporting film, contrarily to b).