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TOWARDS ONE HERTZ ELECTRON TOMOGRAPHY OF DYNAMIC PROCESSES UNDER ENVIRONMENTAL CONDITIONS: EXPECTATIONS AND LIMITATIONS DUE TO BLUR EFFECTS

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Summary: The final goal of the present work is to improve the speed of acquisition of Electron Tomography tilt series down to one Herz, in order to follow in situ and in 3D the dynamic evolution of nanomaterials, e.g. nanocatalysts in an Environmental Transmission Electron Microscope. In this perspective, we focus here on the evaluation of the blur effects within the projections inevitably induced by the high rotation speed of the goniometer.

1. INTRODUCTION

Transmission Electron Microscopy (TEM) is one of the most powerful tools to study the crystallographic structure, chemistry and morphology of nanoparticles (NPs) down to the atomic level. With the development of Environmental instruments [1], the samples can now be heated in situ under a gaseous atmosphere. Environmental TEM (ETEM) allows chemical reactions to be followed even at atomic resolution, and such studies are particularly suitable to the analysis of NPs to be used for catalytic applications. In parallel, the development of Electron Tomography (ET) has allowed the access to the 3D structure of materials, and it is then worth applying ET to the study of nano-catalyst systems. As a typical example, the distribution of supported NPs evolves with time and temperature (such as during calcination, reduction or oxidation treatments), and measuring this distribution in 3D is required for a good correlation with the catalytic activity of the system. That is where a major difficulty arises: the acquisition of tilted projections series needed for a volume reconstruction takes a fraction of hour or more than one hour in conventional approaches, making them very inappropriate with respect to the speed of the processes to be followed.
The main objective of our project is then to speed up the acquisition step of the tomography approach. To do so, a fast continuous rotation is applied to the goniometer and the projections are acquired in the bright field mode using a high speed and sensitive camera generating at least 25 fps in 4K and up to 100 fps in a binned 2K mode (Oneview from Gatan, www.gatan.com). Under such conditions, tilting series of projections can easily be acquired in a few seconds over an angular range up to 140°; however, due to such fast rotations, blur effects are likely to occur and one main concern here is to evaluate the influence of these blur effects on the final quality of the reconstructions.

2. EXPERIMENTAL METHOD

We will report some experimental results obtained on a dedicated Environmental TEM, FEI TITAN ETEM 80-300 kV equipped with an objective aberration-corrector. They concern various nanocatalysts such as zirconia (ZrO_2)-based particles for soot oxidation [2]. Reasonably good reconstructions will be presented of nano-objects tilted in an angular range up to -73 to +73° in less than 5 seconds under environmental conditions, e.g. at high temperature (up to 600°C) under a partial pressure of up to 2 mbar of oxygen or air.
In addition, we will study the effect of blurring by means of numerical reconstructions of a dedicated ghost model deduced from a tomographic experiment concerning Pd NPs supported on alumina. The reconstructions were performed using our GPU-based implementation of SIRT algorithm. The motion filter available in MATLAB was

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used to simulate the blurred images. These blurred images were chosen randomly and different magnitudes of blurring were set in order to mimic mechanical instabilities of the goniometer during the fast rotation.

3. RESULTS

Results illustrated in figure 1 will be discussed in terms of the blurring conditions, that is: amplitude, frequency, etc… Ongoing work will be undertaken to develop blur corrections algorithms to provide better reconstructed volumes in cases where the acquisition has necessarily to be run very fast due to severe environmental conditions.

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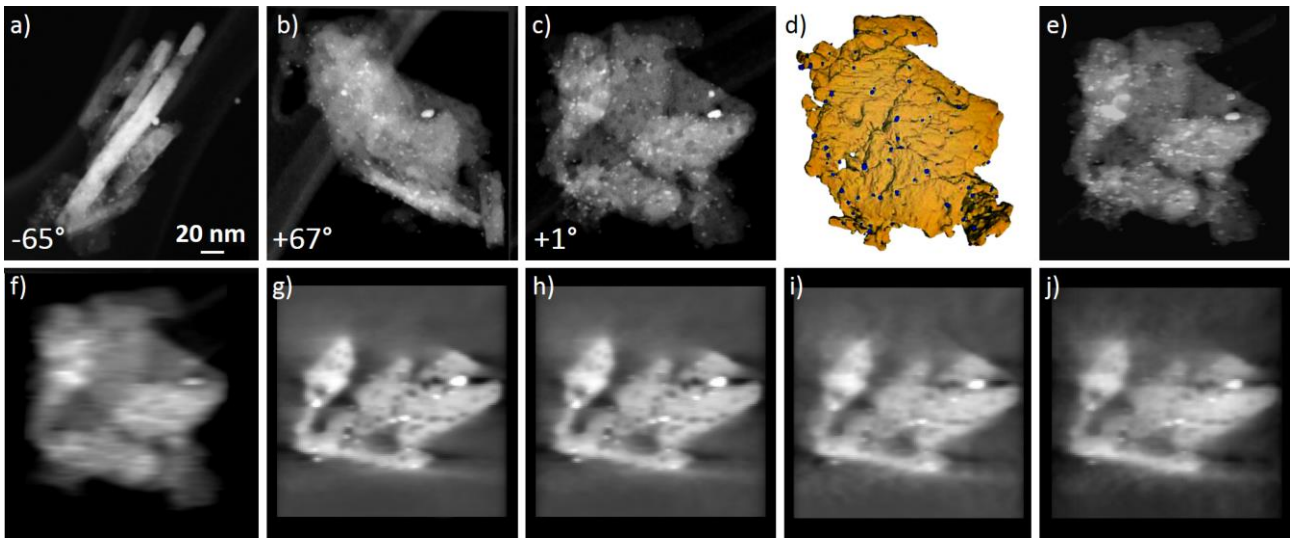

Figure 1: (a-c): experimental ETEM projections (STEM mode) extracted from a -65/+67° ET series of a collection of Pd NPs supported on a delta-alumina grain; (d): reconstructed 3D model; (e) re-projected image from (d) corresponding to the orientation shown in (b); (f) same as (e) with a translational blur of 20 pixels along Ox (field of view 200 pixels); (g)-(j): central sections of the reconstructed model from 210 projections such as in e) calculated over a tilting range of 140°: (g) without any blurred image, (h) with Ox translation blurs (random amplitude between 10 and 20 pixels) applied randomly to 10% of the projections; (i-j) 50% of the projections are blurred with translation blurs of amplitude between 10 and 20 pixels in (i) and 20 and 40 pixels in (j).