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A Machine Learning pipeline to track the dynamics of a population of nanoparticles during in situ Environmental Transmission Electron Microscopy in gases

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Tracking the dynamics of supported nanoparticles (NPs) during the conditioning and under reactive conditions of heterogeneous nanocatalysts is an essential key for the optimization of their activity and durability [1]. Owing to its spatial resolution, Conventional Transmission Electron Microscopy (TEM) has always been a privileged technique for these studies, but the availability of Environmental techniques, either in a dedicated ETEM of in close cells, increases the potential of TEM experiments since they provide a way to follow directly such an evolution under gas and at high temperature. Features that are of significant importance are typically the mobility of NPs (surface diffusion, anchorage effects on their support), their size evolution (e.g. growth by coalescence or Ostwald Ripening, disappearance by dissolution or sublimation).

Probably one of the main concerns is, as always, the poor representativity of TEM results in terms of statistical meaning. This issue is of stringent importance when video sequences are acquired during in situ experiments, since the production of large sets of data renders their analysis and interpretation very tedious and time consuming. This is however a reasonable strategy to obtain more statistics.

While detailed treatments can confidently be performed manually when analyzing a few NPs from relatively narrow regions of interest (see for example [2-3]), new automation approaches are required to achieved a reasonable statistical relevance. This becomes to be more and more possible owing to the constant development of easy-to-use machine learning routines as already performed in this field by few groups (e.g. [4-5]).

This contribution will present a complete pipeline dedicated to the tracking of a NP population evolving under in situ gas and temperature conditions, with the aim of enabling a thorough analysis of their evolution according to the previously mentioned interactions and features regarding mobility and size evolution.

It consists in several steps which (see figure 1) will be detailed and illustrated in the case of Scanning TEM imaging of a Pd-delta-Alumina catalytic system followed during in situ calcination under oxygen in a dedicated FEI-Titan ETEM microscope [6]:

(i) Proper registration of successive images (frames) from continuous sequences

(ii) Robust detection of NPs using the well-known Convolutional Neuronal Network (CNN) U-Net [7] assisted by a verification of the ‘Treacy-Rice’ analysis of scattered intensities [8] after a background subtraction routine performed locally in order to account for intensity heterogeneities of real supports with roughness or topography

(iii) Dedicated training and fine tuning of the CNN using large quantities of realistic annotated simulated images

(iv) Identification of trajectories using an energy criteria-based approach (referred to as Multiple Object Tracking [9]) derived from the continuous energy minimization tracking developed by [10]

(v) Automatic analysis of 'fusion' events (i.e. NPs coalescence) [11].

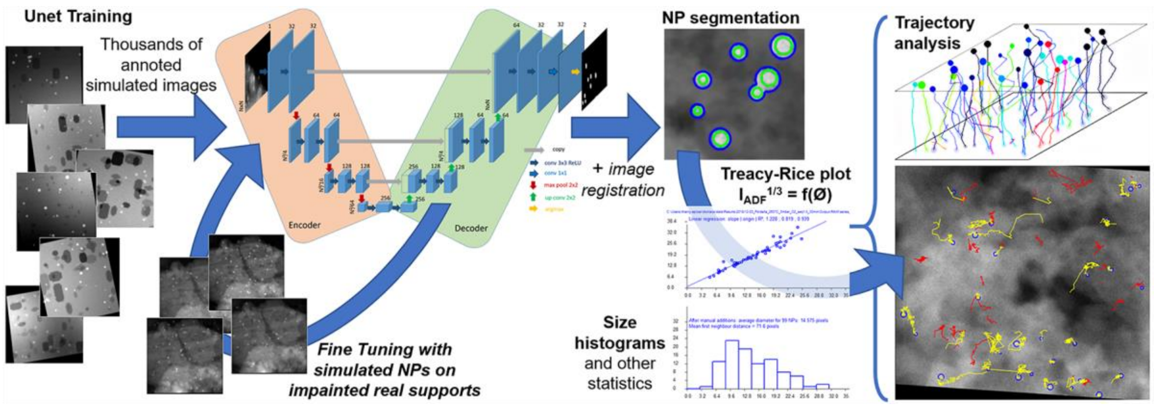

Figure 1. General synoptic of the NP tracking analysis.

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