doi:10.1017/S1431927622002811
Microsc. Microanal. 28 (Suppl 1), 2022

Fast Automatic Analytical Particle Analysis Using an AI Guided Smart Scan Strategy

Pavel Potocek[1], Maurice Peemen[1], Bert Freitag[1]*, Philipp Müller[2] and Thorsten Wieczorek[2]

[1.] Thermo Fisher Scientific, Eindhoven, Netherlands.
[2.] BASF SE, Ludwigshafen am Rhein, Germany.
* Corresponding author: bert.freitag@thermofisher.com

The shape, size, chemical composition and distribution of nanoparticles influence their properties and function - especially in catalysis applications. The relevance of such measurements depends greatly on a statistically significant analysis on sufficiently large numbers of particles. Therefore, an automatic, fast, and reliable way of characterizing these parameters is pivotal in nanoparticle research. We demonstrated a relative speed up of two orders of magnitude compared to the classical procedure based on full frame XEDS mapping.

The challenges with automated analytical particle analysis have several causes. Particles embedded in a matrix material are hard to automatically identify and measure due to varying background contrast. Mapping times for compositional information increase dramatically when thousands of particles in slow full frame acquisition have to be mapped chemically. Last but not least, data analysis can still be improved for such use-cases. Trained neural networks can identify the position and shape of individual particles of interest automatically. This identified position and shape information can be used for a smart scan of only the relevant areas to speed up the analytical measurement. We developed a novel deep learning-driven workflow that spends most acquisition time on particle regions, discards the less interesting regions and provides an automated analysis of the particles. An overview of this workflow is illustrated in figure 1. The most important steps to enable such a workflow are following:

i) the acquisition of fast overview image with a fast detector (HAADF)
ii) identify the particles of interest (position and shapes) in composite materials via AI
iii) visit the identified particles within this case analytical (XEDS) measurement in a second slower scan
iv) build statistical information with an automatic analysis

The critical part of the workflow is the particle detection algorithm. We applied our workflow to challenging conditions for AgPd particle detection in a highly complicated background typical for catalyst materials. An ensemble of six convolutional neural networks (CNN) is used to identify the positions and shapes of the individual particles in matrix materials. The individual Unet [1] CNNs have different configurations for number of layers, number of features, positive or negative input image as well as batch size. The resulting particle detection is using a simple mean value from all individual networks. A set of 32 manually labeled images with particles was used for the training the networks. Individual labeled image size was 2048x2048, the training batch size was either 256 or 512 squared pixels.

Visiting individual particles with the XEDS measurement is enabled by an experimental software adaptation of the existing digital scanning device on a TalosF200 tool utilizing a custom scan pattern functionality. The same adaptations can be applied on Themis or Spectra type columns. The custom scan pattern functionality enables visiting individual pixel positions with precision higher than the original

reference image pixel size. There are multiple options to control the XEDS scanning like visit only particle centers, or visiting small geometric areas (rectangle, circle) or scan the complete particle shape. The workflow assumes there is no drift between steps i) and iii) and/or during iii). One should keep track of this condition, which can significantly influence the results. Note that step iii) can be extended with a drift compensation algorithm in the future. The same is true for specimen shrinkage e.g. due to electron overdose.

We applied this adaptive scan particle workflow to characterize AgPd particles in a titaniumoxide matrix and determined on more than 4000 particles their size, shape distribution and chemical variation. (Figure 2) Full mapping of the selected area in conventional XEDS mapping would have taken more than a day. The new method allowed to obtain the results within 23 minutes which enables statistical relevant analysis in a reproducible way on the nanoscale. We applied the method to other similar problems and will present them during the contribution.

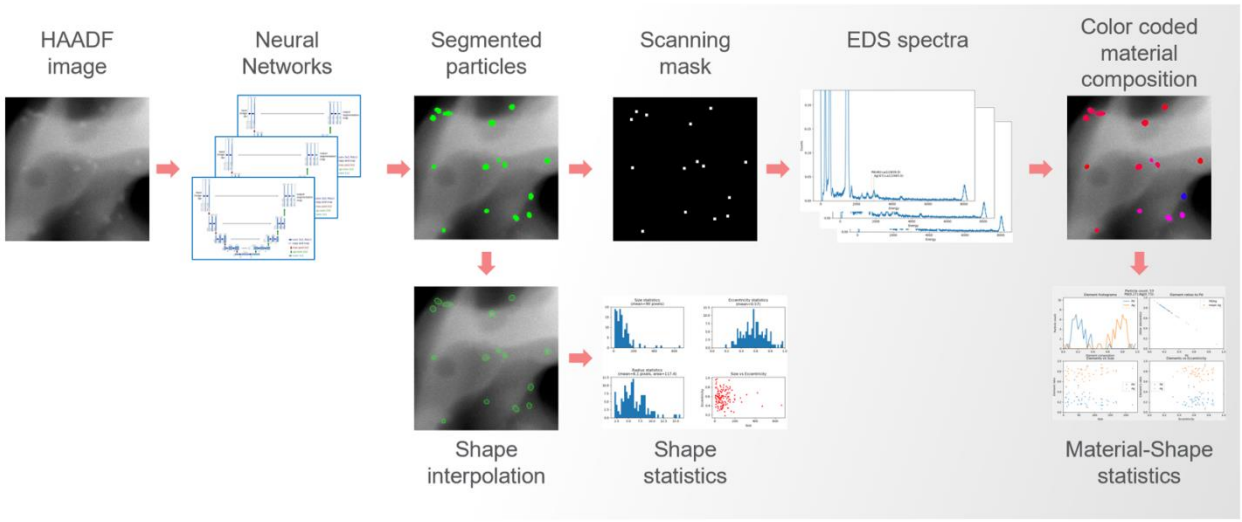

Figure 1. Workflow diagram of our adaptive scan acquisition. A single trained CNN could not achieve sufficient particle detection precision. We boosted the accuracy and robustness by using a combination of several CNNs. The detection quality depends on the training data. This means that retraining is required when the application is applied to other conditions (e.g. different substrates, particle shapes, detectors, …).

https://doi.org/10.1017/S1431927622002811 Published online by Cambridge University Press

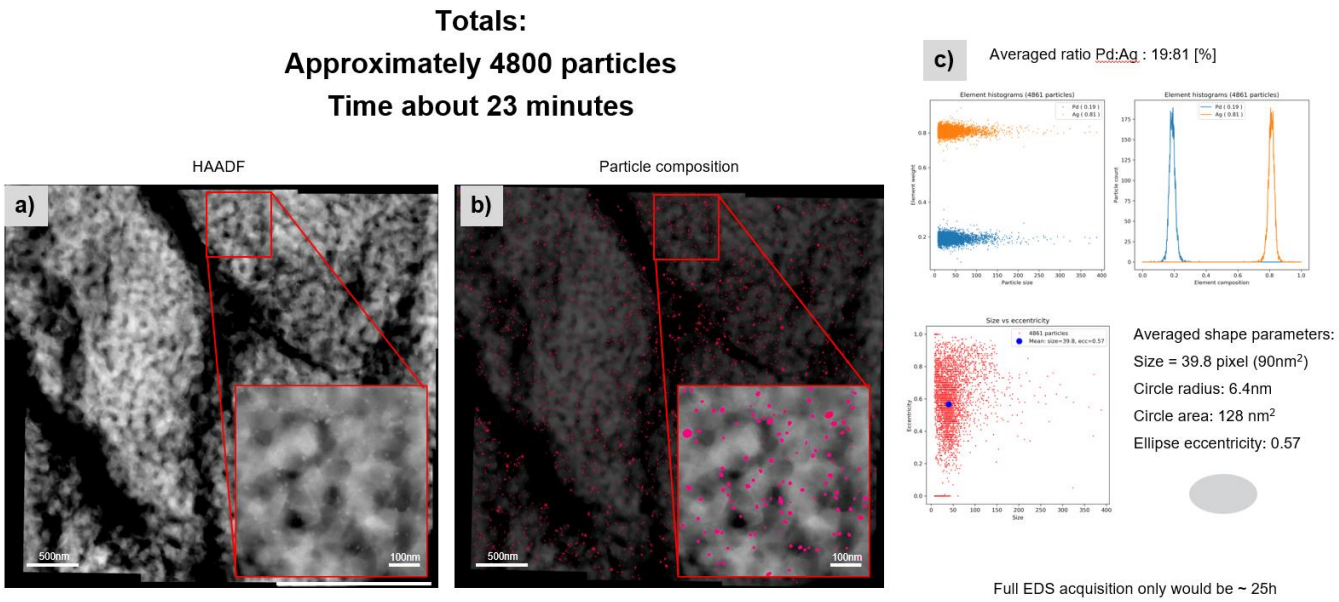

Figure 2. (a) 3k x 3k HAADF image of AgPd particles in matrix material. (b) Overlay of identified particles using the trained network. (c) automatic statistical analysis of shape size, morphology, and composition of 4000 particles within 23 minutes total workflow time.

References:

[1] Ronneberger O., Fischer P. and Brox T., (2015) U-Net: Convolutional Networks for Biomedical Image Segmentation. In: Navab N., Hornegger J., Wells W., Frangi A. (eds) Medical Image Computing and Computer-Assisted Intervention – MICCAI 2015. MICCAI 2015. Lecture Notes in Computer Science **9351**. (Springer, Cham). https://doi.org/10.1007/978-3-319-24574-4_28

https://doi.org/10.1017/S1431927622002811 Published online by Cambridge University Press