Influence of defocus on electron tomography in TEM mode: application to soot aggregates

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1. INTRODUCTION

The significant impact of atmospheric aerosols on regional and global climate is now widely recognized, and atmospheric models - used in the assessment of radiative forcing – have been improved to include important aerosol species [1]. Among these, soot aerosol particles are major contributors to light absorption and consequently have a direct impact on the radiative balance of the atmosphere. They are in fact aggregates, which have a complex morphology usually described through its fractal dimension, and are made of spherical primary particles having a diameter typically ranging from a few nanometres to dozens nanometres [2]. Soot aggregates radiative and transport properties are influenced by their morphology that is commonly characterized from 2D images produced by TEM. To date, only a few studies have been performed with electron tomography in order to characterize the 3D shape of these aggregates [3-5] and we show in this work that one has to pay attention to the defocus during the acquisition of tilted series to avoid artefacts and ensure a realistic reconstruction. The impact of the defocus on the reconstruction of soot particles is investigated in details and a procedure ensuring a realistic reconstruction is proposed and validated with numerically generated spheres.

2. RESULTS

2.1 Influence of defocus

Soot particles are collected with a probe located at 5 cm behind a turbofan engine mounted on a test bench and holey carbon TEM grids are used as supports for the collected soot aggregates. They show submicronic complex shapes and are composed of primary particles that have a lognormal size distribution with a geometric diameter of 15 nm.

Soot aggregates are analysed using a FEI Tecnaï G2 TEM operating at 200kV. Bright field images are acquired by tilting the specimen from typically -70° to +70° (angle step: 1°). Tilted series are then processed using a Sequential Iterative Reconstruction Technique (SIRT) with 30 iterations and the aggregates volume is segmented following the method developed by Adachi et al. [4]. Measurements of the surface area and volume are done using the Amira software suite. Due to the low contrast of this carbonaceous matter, it is very common to acquire defocused images. Thus we acquire several tilted series of the same aggregate with different defocus values $\Delta f=0$ to $-1 \mu m$ (figure 1) in order to evaluate the influence of this parameter on the reconstruction and the 3D determined parameters. We determine surface area values varying from 1.85×10^5 nm² to 4.00×10^5 nm² depending on the defocus.

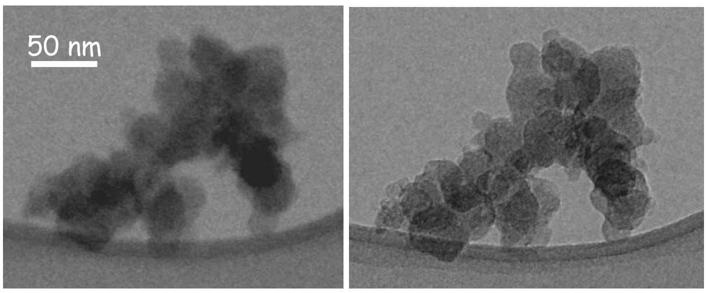

Figure 1. Projected image at 0° tilt with no defocus (left), -0.5 µm defocus (right)

We show that a detailed analysis of the tomograms intensity profiles as a function of the defocus allows pointing out noticeable differences on the shape of reconstructed primary particles. By increasing the defocus, a curved intensity profile centered on each particle occurs with increasing amplitude. We also observe undulations for $\Delta f=-1 \mu m$ (figure 2). These changes are linked to the Fresnel fringes generated with defocus on the particles edge (figure 3).

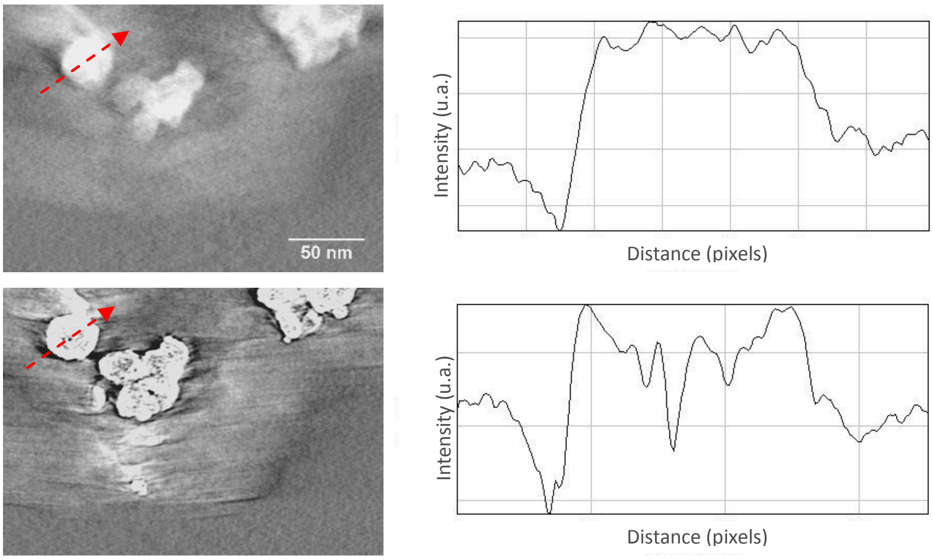

Figure 2. Negative Slice (xy) perpendicular to the electron beam for *i)* a non defocused tilt serie (top left) and *ii)* a tilt serie with a defocus of -1 µm (bottom left). Intensity profiles measured along the red arrows (right).

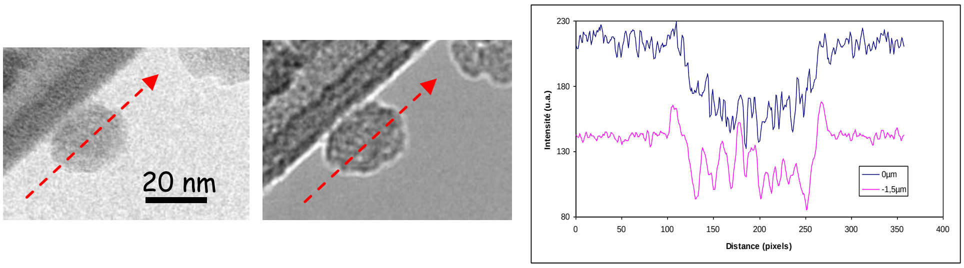

Figure 3. TEM bright field images of an isolated primary particle with Δf=0 (left) and Δf=-1.5µm (middle). Intensity profiles measured along the red arrows (right).

2.2 Validation of the method on a numerically generated particle

The same procedure (reconstruction, segmentation, surface area measurement) is applied on a numerically generated sphere having a diameter of 30.4 nm (Figure 4). We determine a surface area of 2819 nm² from the reconstruction, which is in good agreement with the theoretical surface area value of 2903 nm².

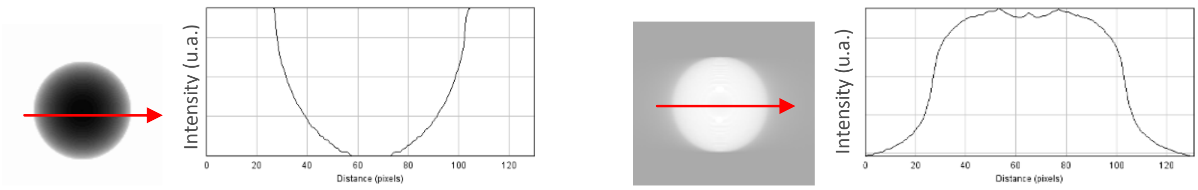

Figure 4. Left: Simulated TEM bright field image at tilt 0° with no defocus and associated intensity profile. Right: Reconstructed central xy slice and associated intensity profile.

3. CONCLUSION

Electron tomography is a suitable tool to determine 3D morphological characteristics of soot aggregates. However one has to pay attention to the defocus for tilted series acquisition in TEM mode as it can drastically modify the shape of reconstructed particles. Indeed the presence of Fresnel fringes linked to defocus change the intensity profile of particles, leading to erroneous 3D quantitative measurements. We propose here a method to evaluate 3D parameters such as surface area and volume of soot aggregates and we show its validation on numerically generated spheres.

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