doi:10.1017/S1431927616007108
Microsc. Microanal. 22 (Suppl 3), 2016

Low Dose Electron Microscopy of Cobalt Oxide Heterostructures, the Genuine Atomic Structure and Dose Limit

Hector A. Calderon[1], Oscar E. Cigarroa-Mayorga[1] and C. F. Kisielowski[2].

[1.] Depto. Física, ESFM-IPN, Mexico City, Mexico.
[2.] NCEM-LBNL, Berkeley CA, USA.

Heterostructures are being developed for solar energy recovery. They can be part of a device to generate a fuel having sunlight as a component to decompose water and chemically reduce carbon dioxide. In this investigation, a cobalt oxide nanoparticle attached to a nanobar is observed by means of transmission electron microscopy in order to determine its genuine atomic distribution i.e., undisturbed by the electron beam. This is important in order to relate the catalytic activity to atomic distribution on the surface of the nanoparticle since this determines efficiency of the component and durability. Previous efforts to determine the genuine atomic distribution have been published in the literature by using low dose electron microscopy. Here further work is reported together with results related to the dose limit that this material can withstand. It is known that the electron dose rate is rather important to control beam sample interaction but also that there is a limit of the total dose that a material can withstand. Transmission electron microscopy has been performed in the TEAM 05 microscope (80 KV, NCEM-LBNL) in conditions of low dose rate in TEM mode together with a routine in MacTempas ® in order to apply the exit wave reconstruction (EWR) procedure with 40 or more experimental images.

A summary of the results is given in Figures 1-3. Figure 1 shows an experimental image taken at approximately 20 $e^-/Å^2s$ together with the phase image from ERW and the corresponding diffraction pattern. The structure is undisturbed by the electron beam since phase images obtained from the first 20 images is identical to that determined with the last 20 images. Figure 2 shows results of different focal series (FS) a total of 17 FSs have been evaluated at different dose rates. Initially the dose rate is kept constant at 20 $e^-/Å^2s$ and five series are taken, as shown in Fig. 2b, little change is seen in the phase image but there is an structural change that can be detected in the corresponding diffraction pattern, the variation is rather subtle but important. Fig 2c shows results after FS6 with a dose rate of 10 $e^-/Å^2s$. Then, the dose rate is increased to 45 $e^-/Å^2s$ (Fig. 2d) and clear changes around the particle surface can be detected, some atoms detach from the original position and reattach at different locations. This process continues in the subsequent focal series taken at a higher dosage. Experimentally increasing the dose rate is followed by a focal series taken at a low dose in order to record possible changes due to the beam interaction with the sample. Fig. 2e shows a phase image taken at around 1000 $e^-/Å^2s$, the image clearly shows the effects of the higher dosage i.e., repositioning of the atomic species and structural changes (see diffraction patterns in Fig. 3). Comparison of the phase image in Fig. 1c and that in Fig. 2f give a clear view of the changes that a higher dose can produce. On the other hand, 6 FSs have been taken in low dose (around 20 $e^-/Å^2s$) representing a total of 240 images without an appreciable motion of atoms (repositioning). Nevertheless small variations are recorded that change and increase the crystallinity of the nanoparticle have been also detected.

References:

[1] The authors acknowledge funding from the IPN, CONACYT and COFFA IPN. The use of TEAM 05 at NCEM-LBNL is supported by the Molecular Foundry, which is supported by the Office of Science,

the Office of Basic Energy Sciences, the U.S. Department of Energy under Con- tract No. DE-AC02-05CH11231.

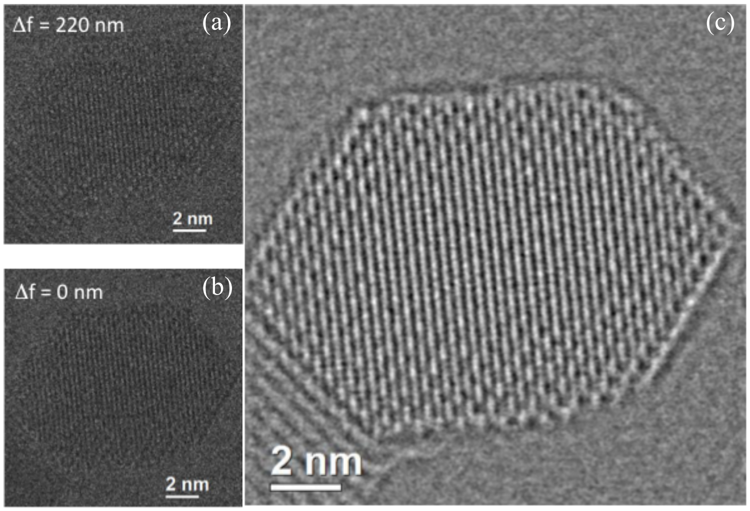

Figure 1. Co oxide nanoparticle. (a,b) Experimental images with a dose rate of 20 $e^{-}/Å^{2}s$. (c) Phase image.

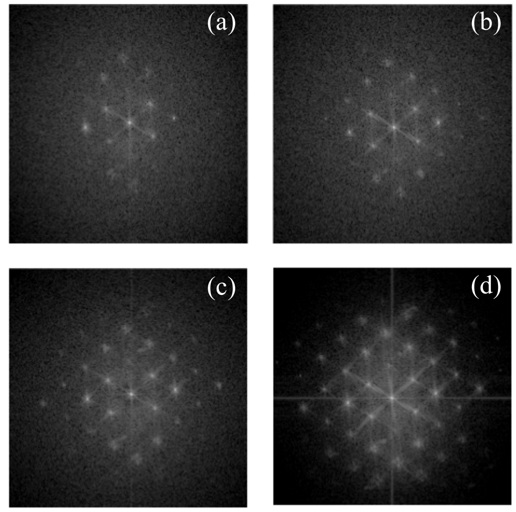

Figure 3. Diffraction patterns. (a) After FS 1, (b) FS 17, (c) FS 10, (d) FS 12.

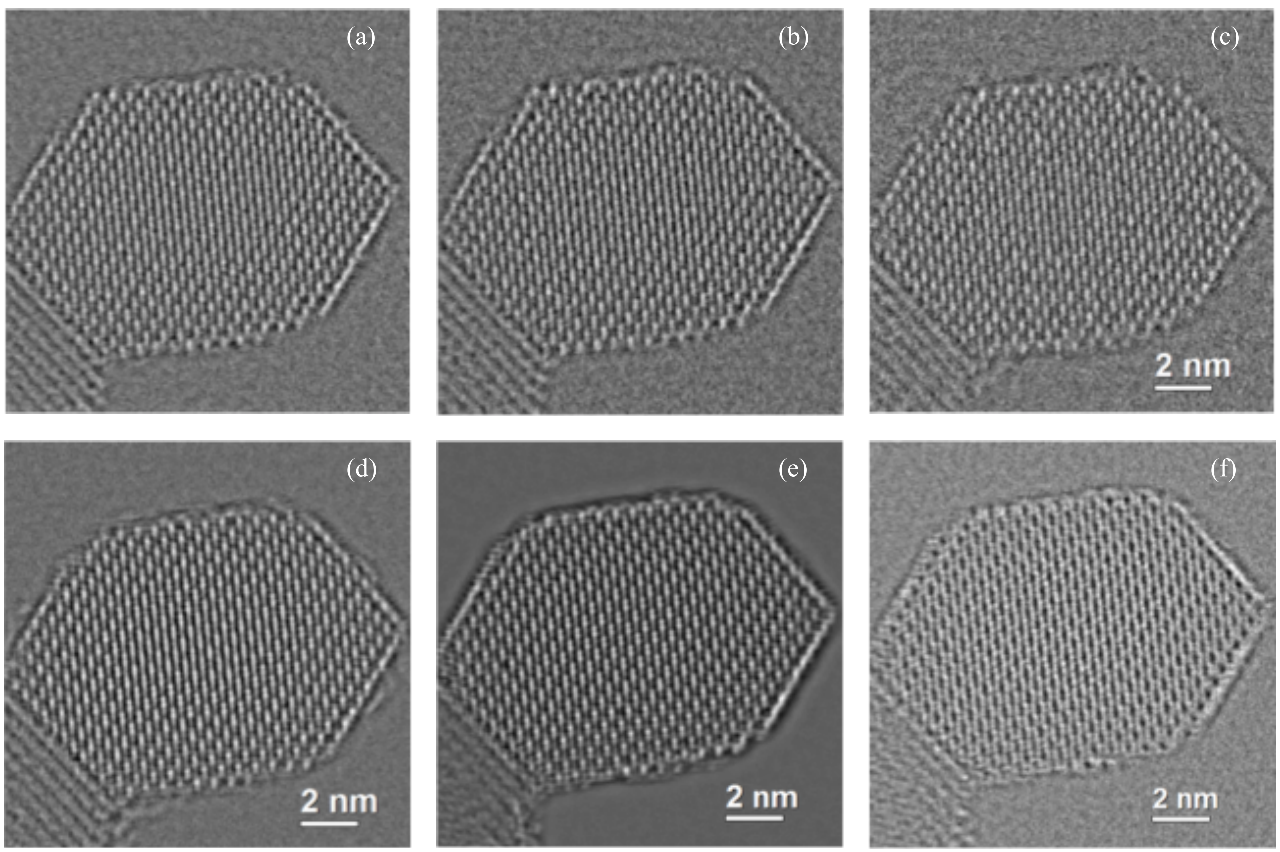

Figure 2. Phase Images (a) After FS 4, (b) FS 6, (c) Fs 8, (d) FS 10, (e) FS 12, (f) FS 17. The dose rate is different for each Focal Series (FS).

https://doi.org/10.1017/S1431927616007108 Published online by Cambridge University Press