Effect of the environment on the elastic properties of CeO_2 nanocubes studied by *in situ* ETEM nanocompression

L. Joly-Pottuz[a*], T. Epicier[a], M. Cobian[b], T. Albaret[c], D. Stauffer[d], K. Masenelli-Varlot[a]

a. Univ Lyon, INSA-Lyon, MATEIS UMR5510, Bât. Blaise Pascal, 7 Avenue J. Capelle, 69621 Villeurbanne Cedex, France
b. Univ Lyon, ECL Ecully, LTDS UMR5513, 36 Avenue de Collongue, 69134 Ecully, France
c. Univ Lyon, UCB Lyon 1, ILM, CNRS UMR 5306, 69621 Villeurbanne, France
d. Bruker Nano surfaces, Hysitron Inc, 9625 W 76th St., Minneapolis, MN 55344, USA
* lucile.joly-pottuz@insa-lyon.fr

Characterization of nanomaterials or materials at the nanoscale has drastically been improved during the last decades. A challenge lies in the *in situ* microstructural characterization of such materials as it can give access to valuable information regarding the microstructural changes induced by their use in working conditions (for instance during compaction process in the case of ceramic nanoparticles). The availability of dedicated TEM (Transmission Electron Microscopy) holders equipped with nano-indenters, e.g. hard tips, is of very high interest to test *in situ* the mechanical properties of nanometer-sized objects [1]. In the case of plastic deformation of crystalline nano-objects, Molecular Dynamics simulations have shown that dislocation nucleate at the surface [2, 3]. Therefore, it might be interesting to investigate such mechanical aspects with experiments in a controlled environment (i.e. under gas pressure) which reproduces the real one.

A Hysitron PI 95 Picoindenter has recently been installed on a Cs-corrected FEI Titan ETEM (Environmental TEM) microscope. It opens the possibility of performing *in situ* compression under gas pressure, with high resolution imaging capabilities. We will present a comprehensive study on CeOx nanocubes. Nanocubes are compressed either under vacuum or under air pressure, with different irradiation doses to obtain various compositions ($1.5 < x < 2$). In order to minimize data dispersion due to mechanical accommodation for instance, we focus on the elastic regime. This way, it is possible to compare the elastic moduli deduced from a Digital Image Correlation based analysis on the same nanocube but with different oxygen contents. To better understand the reduction/oxidation process and its effect on the mechanical properties, we present DFT+U simulations on bulk systems with various compositions. The stability of each crystallographic phase is in agreement with the literature [4] and with experimental observations. Significant changes in the calculated elastic moduli are obtained and compared with experimental results.

[1] Q. Yu, *et al.* MRS Bulletin 40, 62-70 (2015); [2] S. Lee, *et al.* Nat. Commun. 5:3033 (2014); [3] I. Issa *et al.* Acta Mater. 86, 295-304 (2015); [4] J.L.F. Da Silva, Phys. Rev. B 76 (2007), p. 193108.

Acknowledgements: LABEX iMUST (ANR-10-LABX-0064) of Université de Lyon, within the program “Investissements d’Avenir” (ANR-11-IDEX-0007) operated by the ANR, CLYM (www.clym.fr) for the access to the microscope and A.K.P. Mann, Z. Wu and S.H. Overbury (ORNL, USA) for having provided the samples.