
In situ compression of oxide nanocubes in Transmission Electron Microscopy

Rongrong Zhang¹, Karine Masenelli-Varlot¹, Gaétan Laurens², Tristan Albaret², David Dunstan³, Frederic Chaput⁴, and Lucile Joly-Pottuz*¹

¹Materiaux: Ingénierie et Science – Institut National des Sciences Appliquées (INSA) - Lyon, CNRS : UMR5510 – France

²Institut Lumière Matière [Villeurbanne] – Université Claude Bernard-Lyon I - UCBL (FRANCE) – France

³School of Physical and Chemical Sciences, Queen Mary University of London, – United Kingdom

⁴Laboratoire de Chimie – École Normale Supérieure - Lyon – France

Abstract

The behaviour of materials at the nanometre scale may strongly differ from their bulk counterparts because of the small size of the stressed volume, and because of the large surface-to-volume ratio. In addition, oxide materials contain ionic bonds, which may strongly affect the deformation mechanisms. However, the literature still lacks information regarding plasticity mechanisms in many oxides.

In situ mechanical testing in Transmission Electron Microscopy (TEM) is a convenient technique to study plasticity in nano-objects as it provides force measurements associated with live video recording up to atomic resolution. The experimental set-up and the procedure used for data processing will first be described. We will show results obtained on cerium oxide (CeOx) nanocubes, with sizes in the range 20-130 nm. Importantly, CeOx can be reduced during analysis due to the electron beam. This process leads to the transformation from CeO₂ (fluorite) to CeO_{1.5} (bixbyite) (1). This transformation is reversible, the fluorite phase being recovered after beam blanking thanks to the presence of residual oxygen gas. It is therefore crucial to control the experimental conditions to understand the acquired data.

Experiments under different conditions (dose rate, environment gas pressure) will be shown. On fluorite, the values measured for the Young modulus on single nanocubes under different conditions will be compared with theoretical values and discussed (2). The plasticity mechanism will also be determined and compared with simulation results. On bixbyite, the active slip systems will be determined. The appearance of stacking faults in bixbyite but not in fluorite will be shown experimentally, and discussed. Finally, we will investigate the effect of the nanocube size on the yield stress, and discussed the value of the exponent in light of the literature.

Acknowledgements

This work was funded by ANR (projet ANR-18-CE42-0009) and CSC. The Consortium Lyon Saint-Etienne de Microscopie (CLYM) for the access to the microscope and D.D. Stauffer

*Speaker

(Bruker) for his help.

References

- (1) A.C. Johnston-Peck *et al.*, *Ultramicroscopy* 170 (2016), 1-9.
- (2) L. Joly-Pottuz *et al.*, *JOM* 76 (2024), 2326-2335.