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In situ observation of dislocation evolution in cerium oxides nanocubes in an environmental TEM

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Characterizing the mechanical properties and deformation mechanism of ceramic materials at the nanoscale is of much interest to improve their processing and properties. At the nanoscale, plastic deformation have already been observed in alumina and magnesium oxide nanoparticles at room temperature. Cerium oxide, as one of the most important ceramic materials, is widely used in many applications, such as in solid oxide fuel cell electrodes, catalysis, or gas detection. However, there are few experimental pieces of evidence of the evolution of defects, which have significant impact on many material properties, especially on mechanical behavior. TEM observations of CeO2 nanocubes demonstrated a beam sensitivity with reduction under high electron dose. Reduction may have an impact on the mechanical properties. The development of TEM nanocompression experiments inside an environmental transmission electron microscope (ETEM) is of much interest to study the mechanical properties of ceramic particles and the effect of the reduction on them. High resolution imaging may also be very useful to fully study the defects formation (dislocations, stacking faults…) during compression. In situ nanocompression under gas and at high resolution in an ETEM were developed on CeO2 and are reported in this work.

Cerium oxides nanocubes (20-50nm size) were compressed using a dedicated Hysitron PI 95 sample holder in an environmental transmission electron microscope (ETEM). By controlling the electron dose and by using a small pressure of air in the ETEM, different CeOx structures (x between 1.5 and 2) were tested to compare their mechanical properties and their deformation mechanisms. Displacement-controlled in situ compression tests were performed in a Titan ETEM 80-300 kV microscope equipped with a Bruker PI95 picoindenter. The loading direction was [100]. Different imaging techniques have been used to identify the slip systems in CeO2. High resolution imaging has also been performed during compression of Ce2O3 nanocubes to observe dislocations, stacking fault or nano-twin formation. Simulation calculations (DFT, DM) are very useful to complete/understand the in situ nanocompression experiments and were developed on CeOx material, even if computing time might be long due to the complexity of the structures.

CeO2 has a fluorite structure (space group Fm-3m). Their reduction under the electron beam creates oxygen vacancies in the structure, which leads to a fcc bixbyite structure (space group Ia-3) with a double cell parameter compared to fluorite. In the case of CeO2, deformation mechanism is found to be similar to those observed in other fluorite structure, with <110> {111} slip systems. The evolution of the yield stress with the nanocube size reveals to be similar to the one already observed for other fcc structures. In the case of Ce2O3 bixbyite structure few studies have reported the deformation mechanism and the slip systems so far. Oxygen vacancies may play a role in defect formation. To observe the deformation mechanisms accurately, high resolution imaging could be performed during in situ nanocompression. Dissociation of perfect dislocations into partials and stacking faults has been observed, which is unusual in oxides. The results obtained for both structures are discussed and compared to simulations works. Compression cycles carried out on the same nanocube under different irradiation conditions (thus either in the fluorite or in the bixbyite structure) will be shown to highlight the differences between both mechanisms.

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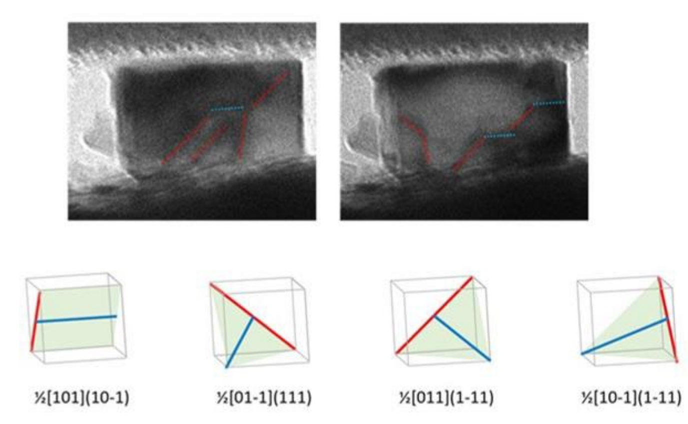
In situ nanocompression experiments have been used to study and compare the deformation mechanism of CeOx nanocubes. On single nanocubes, different mechanisms have been evidenced depending on the irradiation conditions and thus the crystallographic phase. When compressed along the <100> direction, fluorite deforms at room temperature via <110> {111} perfect dislocations and the effect of the nanocube size on the yield stress is similar to what is observed in other fcc materials. In bixbyite, the dislocations dissociate and form stacking faults. This process is reversible.

Graphic:



Keywords:

In-situ, Nanocompression, ETEM, Oxides