Nanometer sized objects are attracting large attention nowadays due to their breakthrough mechanical properties such as high hardness, crack propagation resistance and high elastic limit in comparison to the bulk of their counterparts [1]. In situ TEM nanoindentation is a particularly well suited technique for the mechanical testing of nano-sized objects. Images of the deforming sample and force-displacement curves can simultaneously be acquired. The challenge remains in the identification of the material mechanical behavior, namely the constitutive law with the intrinsic parameters – Young modulus, yield strength, Poisson ratio – as well as the understanding of the deformation mechanism. In this study, we propose an innovative method for a complete mechanical analysis of nanoparticles in the size range [30 nm-300 nm]. This protocol consists in coupling of in situ TEM nano-compression tests of isolated nanoparticles, image analysis and mechanical simulations. After the experiments, the load-real displacements curves are measured by Digital Image Correlation. Then a constitutive law is obtained through an inverse Finite Elements simulation. The determination of a constitutive law includes the determination of the material intrinsic parameters such as Young modulus, Yield stress, hardening coefficient, and stress at fracture. In this presentation, the method will be presented through the analysis of transition alumina nanoparticles. It will be shown that such ceramic nanoparticles can undergo large plastic deformation, which is not observed in the bulk (Fig1). The parameters of the constitutive law will be discussed in the light of the literature, and especially the work from K. Zeng et al. [1]. They showed that the electron beam, during in situ TEM nano-compression tests of silica nanoparticles, creates structural and bonding defects throughout the entire sample and facilitates the plasticity of the nanoparticles. The deformation mechanisms will be investigated through performed compression experiments in a Diamond Anvil Cell, at room temperature and in the absence of electron beam. We will present the results obtained from HRTEM observations of thin foils extracted from samples compacted at various uniaxial pressures. We will show that plastic deformation occurs also in this case (Fig2). Moreover, the appearance of a nanoparticle preferential orientation will be evidenced (Fig3). This point will be discussed in function of the possible slip systems. Finally, we will demonstrate that such HRTEM analysis gives interesting pieces of information, which permit to better understand how the nanoparticles behave and deform during the in situ experiments.


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Fig. 1: TEM in situ nano-compression force-displacement curve. The simulations using DIC-FE (red) or the analytical method (blue). A good agreement is found for both simulations with the experiment, especially for the DIC-FE method which takes into account the plastic regime, contrary to the analytical method which is valid only in the elastic domain.

Fig. 2: (Left) TEM image revealing the plastic deformation of transition alumina nanoparticle compacted in a DAC at 5 GPa uniaxial pressure. (Right) TEM image of compacted transition alumina in DAC at 20 GPa. It reveals the oriented crystallographic texture with respect to the compression axis.

Fig. 3: (a) HRTEM image of the FIB thin foil of a zone of contact between two alumina nanoparticles. (b) Fourier Transform of the dotted zone of the deformed particle.