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Room temperature plasticity and phase transformation of nanometer-sized transition alumina nanoparticles under pressure

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ABSTRACT

A powder of transition alumina nanoparticles (including γ and a so-called δ -type) is compacted at room temperature in a diamond anvil cell (DAC) under pressures ranging from 5 GPa to 20 GPa. Characterization carried out on thin foils prepared by focused ion beam (FIB), from the compacted powder, unambiguously reveals plasticity. High resolution transmission electron microscopy (HRTEM) and electron diffraction also evidence phase transformation of nanoparticles under high pressure and nanoparticles show faceting parallel to the loading direction with a preferential crystallographic orientation of the facets corresponding to $\{220\}$ planes of γ - Al_2O_3 . It can also be deduced, from the comparison between the DAC experiments and *in situ* TEM nano-compression tests on single particles performed in a preceding work, that plasticity is driven by slip bands corresponding to $\{111\}$ slip planes, common for a spinel structure, such as, for instance, γ - Al_2O_3 .

We also demonstrate that at ambient temperature the transition alumina phase is also prone to structural change under high pressures with the following sequence γ - $\text{Al}_2\text{O}_3 \rightarrow \delta^*$ Al_2O_3 , these two phases coexisting, sometimes, in the same nanoparticle after compaction. Plasticity at room temperature in alumina nanoparticles and the subsequent phase transformation under pressure may have strong impacts on the process of alumina nanostructured ceramics.

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1. Introduction

Ceramics have always been considered as brittle materials at room temperature. Nevertheless, recent studies show that they can present a plastic behavior at the nanometer scale [1,2]. This peculiar behavior for ceramic materials may have a strong impact either on their processing [3] or their future use, since it would open the door towards low-temperature forming, followed by much more moderate sintering temperatures. Nanometer-sized ceramic

nanoparticles are nowadays mostly used for the processing of bulk ceramics like, for instance, nanocomposites for biomedical applications [4–6]. The aim of using nanometer-sized particles is to obtain nanometer-sized grains after sintering. The so-called nanoceramics exhibit extraordinarily good mechanical properties like high wear resistance, low friction, and high strength and hardness [7]. In the case of alumina, the absence of porosity and the nanometer grain size are key points to enhance transparency [8–11]. Thanks to these outstanding properties, many other applications could be considered [12,13]. Understanding the plastic behavior of nanometer-sized ceramic nanoparticles is one possible research axis in order to keep their nanometer size during processing. Our first results obtained during *in situ* nano-solicitation experiments in Transmission Electron Microscopy (TEM) revealed a surprising behavior for transition alumina and MgO nanoparticles: plasticity at room temperature [1,2]. In the case of MgO, this was also observed by Korte and Clegg on micropillars previously prepared by Focused Ion Beam (FIB) [14]. Kiani et al. also used FIB sample

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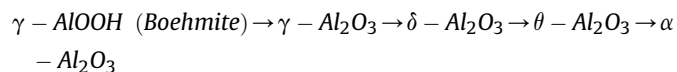
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preparation to study room temperature plasticity in ZrC pillars [15]. The advantage of such *in situ* tests in the case of alumina lies in the fact that it is also possible to test transition phases (metastable phases of alumina) which do not exist at the microscale and as bulk material. Indeed, α -alumina is the only phase of alumina that is stable at bulk, under a wide range of pressures and temperatures [16] while nanometer-sized powders generally consist in a mixture of transition alumina. Studying the behavior of transition alumina is of much interest. Liao et al. [17] showed that the strong compaction of transition alumina (perfectly spherical nanoparticles) improves the green density up to 83% at a pressure of 8 GPa. They concluded, without evidence, that the high density could be linked to a possible partial plastic deformation of transition alumina nanoparticles.

In addition to pressure-assisted plasticity, structural changes may also occur during compression. To investigate possible phase transformation, the different crystallographic phases of alumina can first be briefly introduced. The structure adopted by α -Al₂O₃ can be described by oxygen anions in a near hexagonal compact arrangement and cations filling 2/3 of the octahedral sites. As a consequence, the α -Al₂O₃ structure contains vacancies that are present in 1/3 of the octahedral sites in an ordered array and well-known sequence along a rhombohedral structures axis (2Al³⁺-vacancy- 2Al³⁺). Transition alumina phases (γ , δ , η , θ) are described by the spinel structure defined by the chemical formula AB₂O₄, here Al₃O₄. However as far as stoichiometry is concerned, alumina is described by the chemical formula Al₂O₃. This means that the structure contains Al³⁺ vacancies. In all transition alumina phases, the oxygen anions are nearly in a face-centered cubic arrangement and cations are occupying octahedral and tetrahedral sites. Each phase of transition alumina presents a specific percentage of cations occupation of the octahedral sites over the tetrahedral sites, inducing a specific cations arrangement in each transition alumina phase. For instance, the cation arrangement can be described by cubic and tetragonal structures for γ -Al₂O₃ and δ -Al₂O₃ respectively, with a certain number of randomly distributed vacancies in each unit cell. The δ -Al₂O₃ tetragonal variety is a quasi-supercell of γ -Al₂O₃ parameters a, b of its cell are very close to those of the γ phase divided by $\sqrt{2}$ and its c parameter is close to 3 times that of the γ one (JCPDS file 46-1131). Therefore, the main diffraction peaks of tetragonal δ -Al₂O₃ are in quasi-coincidence with those relative to the γ phase. Only weak supplementary peaks for high inter-reticular distances are present in the δ phase. Other transition alumina phases are also found in the literature. In particular, the Al³⁺ cations can be arranged in an orthorhombic structure, leading to the phase δ^* -Al₂O₃ [18], also called "orthorhombic δ -Al₂O₃" by Levin et al. [19]. This phase can be quite easily distinguished from the γ one by X-ray diffraction (see Fig. 1).

For surface areas lower than 125 m²/g, the α -phase is the most stable one, whereas transition phases are metastable [20]. A sequence of high temperature processes that enables phase transformations from boehmite towards transition alumina and then to α -Alumina is given by Refs. [19,21]:



These transformations are not reversible as each phase obtained after a transition is preserved when temperature is decreased down to room temperature. As mentioned previously, some other intermediate phases (δ^* , δ'), not present in this sequence, may also be found. As a consequence, it has been suggested that transformation from γ -Al₂O₃ (cubic) to δ^* -Al₂O₃ (orthorhombic) occurs before the cubic spinel collapse and exhibits a tetragonal character (δ -Al₂O₃).

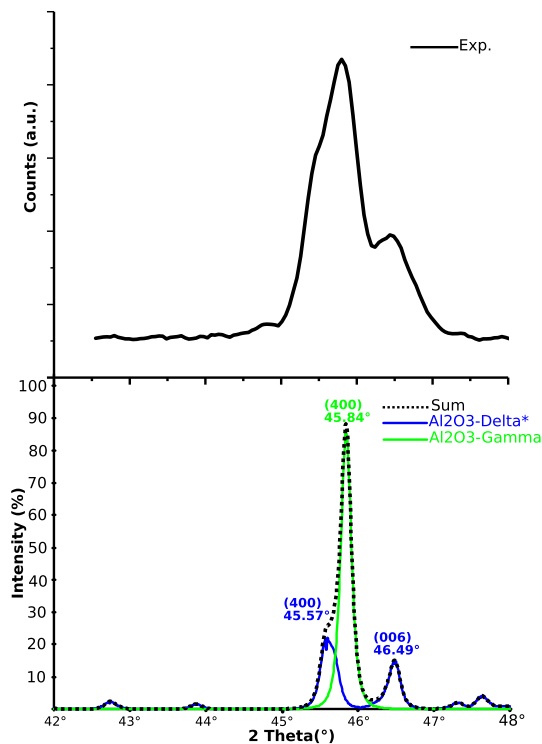


Fig. 1. Phase indexation in the native nanoparticles. Upper part: experimental X-Ray diffractogram. Lower part: simulated XRD diffractogram for a mixture of γ -Al₂O₃ (70 wt.%) and δ^* -Al₂O₃ (30 wt.%) calculated with the software of Carine Crystallography 3.1.

This is followed by a monoclinic structure (θ -Al₂O₃) transformation and finally at higher temperature the trigonal structure α -Al₂O₃ is obtained (without any occupancy of the tetrahedral sites by cations) [19]. It has to be mentioned as well that although some other sequences have been proposed in the literature, for instance by Macêdo et al. [22] or by Paglia et al. [23], there is a lack of information about the physical parameters involved into these phase transformations. For example, temperature is known to trigger phases transformations [17,23], but α alumina is also sensitive to structural changes at high pressure (90 GPa) [24].

As the fabrication of bulk nanostructured alumina includes the compaction of green samples made of transition alumina nanoparticles, which turned out to exhibit a plastic behavior at room temperature, it is necessary to understand better the behavior of the nanoparticles during compaction. In this study, we aim at using a diamond anvil cell (DAC) to compact a powder of transition alumina nanoparticles at room temperature. DAC allows compacting the powder with relatively high pressures (20 GPa in our study), and we may expect locally high stresses which may trigger plasticity and/or phase transformation at ambient temperature. In addition to a deep characterization of the compacted powder, our objective is also to correlate the results obtained with *in situ* previous nano-compression experiments in TEM, where plastic behavior of individual crystalline particles was observed in real time [1,25].

2. Materials and methods

The transition alumina nanoparticles used in this study have been produced by Nanophase Technologies Corporation (Romeoville, IL, USA). It is thermally treated at 900 °C to improve its crystallinity. The particles are spherical and the size distribution lies

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