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In Situ Study of CeO_2 Microspheres Sintering Using HT-ESEM

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Sintering could be defined as the transformation of a powdered compact into a cohesive material under heating at high temperature. It appears as a key-step in the preparation of ceramic materials such as UOx and MOx nuclear fuels. The sintering is usually described through three different stages. The initial stage involves the elaboration of necks between the grains and leads to the cohesion of material while the intermediate and final stages are dedicated to the elimination of porosity between the grains by the means of grain growth mechanisms [1]. Presently, only few experimental works are devoted to the kinetics of necks elaboration (i.e. first stage of sintering), and this stage is mainly described through numerical simulation of 2 to 4 spherical grains in contact [2].

In the present study, we report the first experimental observations of the initial stage of sintering of CeO_2 microspheres using an Environmental Scanning Electron Microscopy at high temperature (HT-ESEM). Actually, the use of HT-ESEM allowed the in situ observation of the samples during long term heat treatments up to 1400°C under various atmospheres [3]. In a first step, CeO_2 spherical grains were synthesized to investigate similar systems to those modeled. Then, the HT-ESEM was used to investigate the first stage of sintering. In this aim, three different systems (single grain, two and three grains in contact) were investigated between 900°C and 1200°C:

- Monitoring of a single grain led to the evolution of the number of crystallites included in the sphere. From the micrographs series, the time necessary to reach a spherical single crystal through the growth of crystallites was determined, as well as the mechanisms involved and the associated activation energies (Figure 1) [4].
- The observation of the morphological modifications of two and three grains arrangements then led to assess the evolution of several parameters of interest such as neck size, dihedral angles between the spheres or distance between the grain centers. From the micrographs series, it was possible, for the first time, to identify experimentally the mechanisms of necks growth between the grains and to compare the behaviour of near ideal single-crystal systems with polycrystalline samples (Figure 2 and 3) [5].

The use of HT-ESEM observations appears of a great interest for the study of sintering phenomena. Image processing allows determining original and fundamental experimental data, such as the mechanisms of necks growth, characteristics of the processes occurring during the initial stage of sintering.

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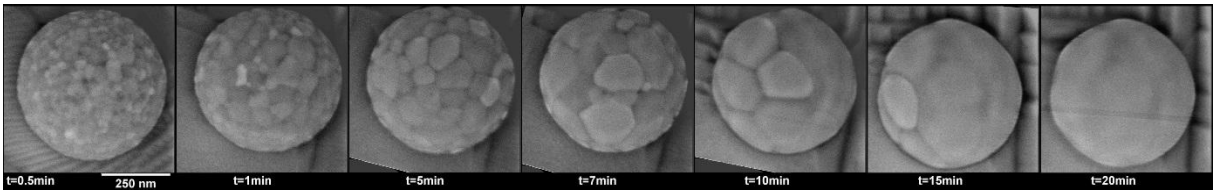

Figure 1. *In situ* HT-ESEM micrographs series of CeO_2 nanospheres showing the evolution of the nanostructure of the nanospheres at T=1100°C. The contrasts of the images have been enhanced by image processing to better see the cristallites.

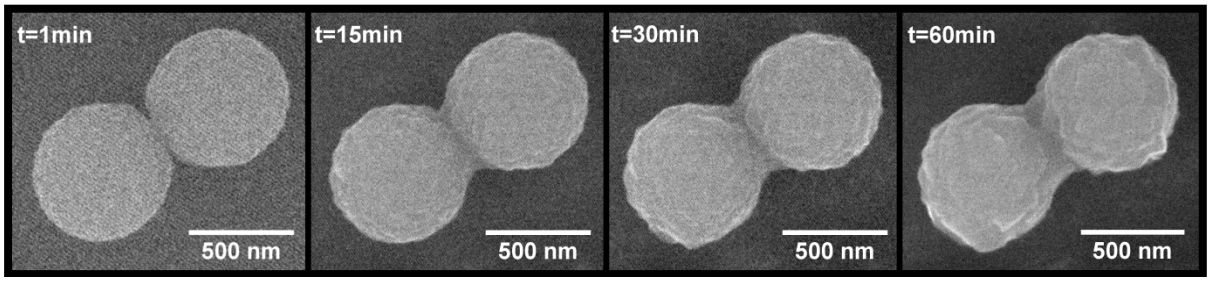

Figure 2. *In situ* HT-ESEM micrographs series of CeO_2 nanospheres showing the evolution of neck size, dihedral angles and porosity between the grains at T=1100°C in the two spheres systems (polycristalline grains).

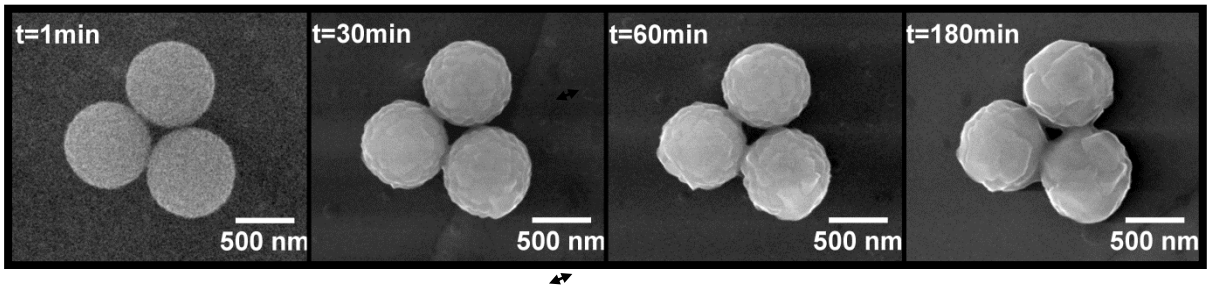

Figure 3. *In situ* HT-ESEM micrographs series of CeO_2 nanospheres showing the evolution of neck size, dihedral angles and porosity between the grains at T=1100°C in the three spheres systems (polycristalline grains).

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