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First Stage of Sintering of ThO_2 Microspheres: a HT-ESEM and HT-HRTEM Study

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The behavior of ThO_2 microspheres [1] during heat treatments at high temperature (1000-1300°C) was investigated by electron microscopy, including *in situ* and *ex situ* ESEM [2] as well as HT-HRTEM with high temporal resolution, and further advanced image processing.

Polycrystalline microspheres were put in contact to investigate the formation of a neck during heat treatment (i.e. sintering) by HT-ESEM (Fig. 1). This process was found to deviate from the expected trend, with the formation of intermediate large-size crystallites between the initial microspheres. Such modification was mainly assigned to the initial microstructure of the particles, which combined nanoscale crystallites with a low amount of residual porosity that precludes any significant contribution of oriented attachment over the global mechanism.

In parallel, HT-HRTEM experiments were performed at 1000°C on a similar system in order to focus specifically on the neck zone (Fig. 2). At low temperature (from RT to 300°C), crystallites orientate preferentially without growth, thus leading mosaicity to decrease within each grain. Fast reorganization of the crystallites orientation was further evidenced at the grain boundary at high temperature (1000°C). This phenomenon yields to the formation of larger crystallites and to the increase of neck size. The growth of the crystallites seems to be the reason for the formation of intermediate large-size crystallites that is also observed using HT-TEM. Also, onset of this last step is strongly dependent on the illumination by the electron beam that can initiate and/or accelerate the sintering.

This study clearly illustrates the interest to combine both HT-ESEM and HT-HRTEM techniques in order to describe precisely the first stage of sintering of metallic oxides. The possibility to record TEM images with a high temporal resolution is of major importance to describe local and fast phenomena. [3]

References:

[1] L Wang *et al*, Cryst Eng Comm **16** (2014), p.10469.
[2] GI Nkou Bouala *et al*, Journal of the European Ceramic Society **37** (2017), p. 727.
[3] The authors acknowledge funding from the Materials Federative Project included in the NEEDS program (Nucléaire, Energie, Environnement, Déchets, Société) of CNRS and the METSA network.

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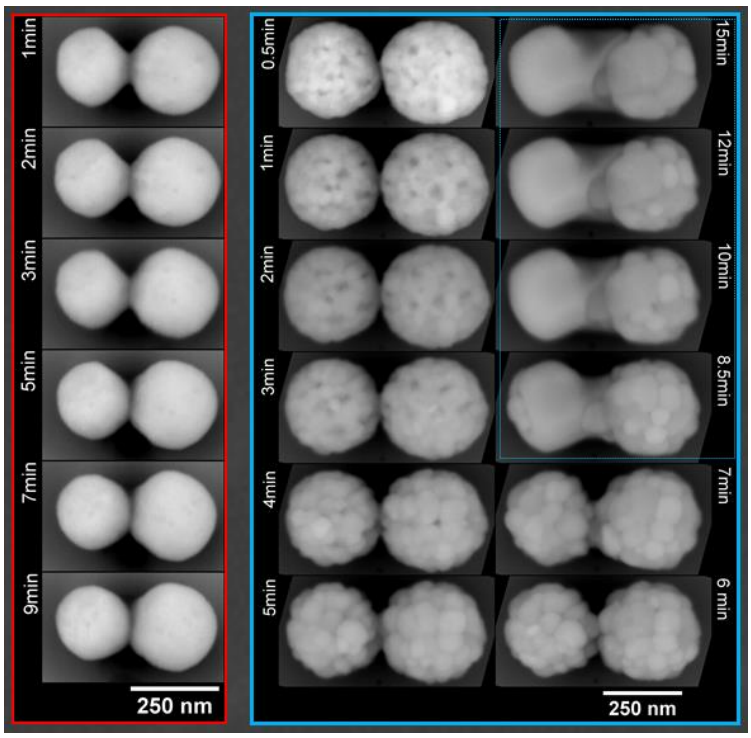

Figure 1. Mono- (red) and Poly- (blue) crystalline grains observed at T=1275°C in the HT-ESEM.

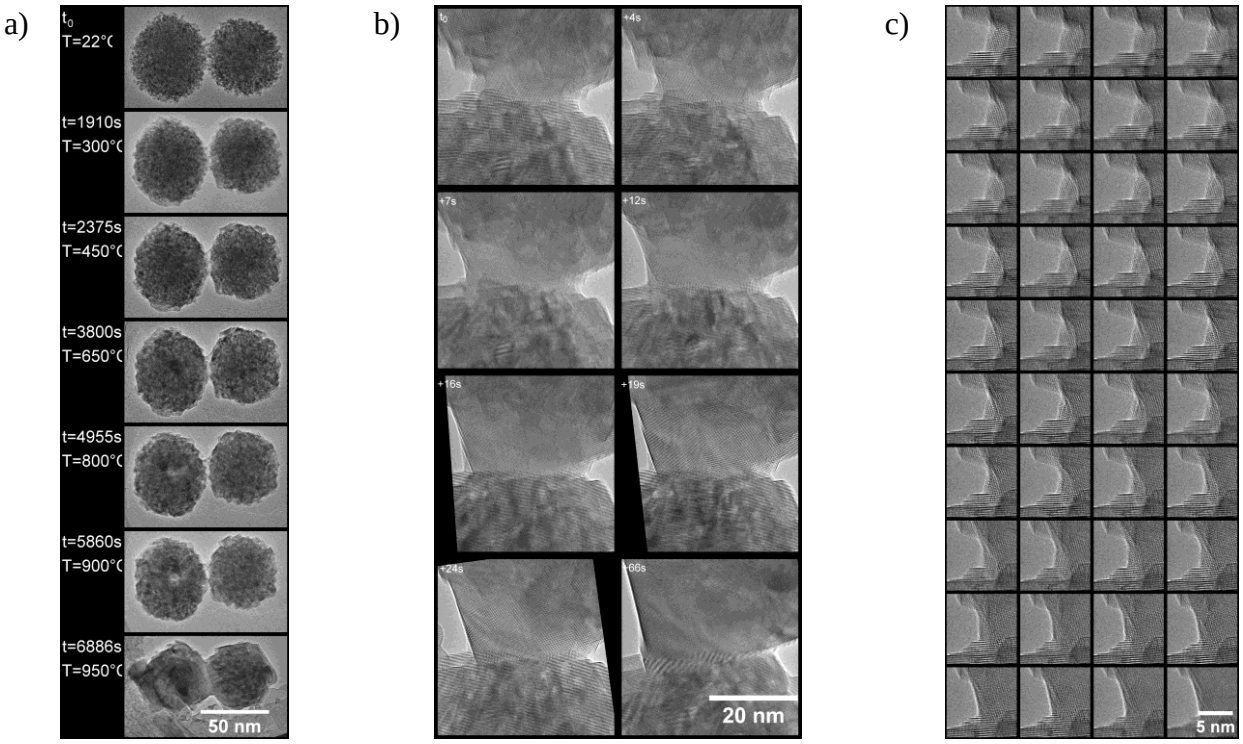

Figure 2. "Long-term" experiments were performed with 143ms time resolution at 950°C in the HT-HRTEM. (a) Duration: 2 hours / Effect of heating, (b) Duration: 1 minute after 2h12 heating / Crystallites growth in one grain, (c) Duration: 4 seconds after 2h12 heating / Crystallites reorganization on the neck.

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