# Use of HT-ESEM for the direct observation of ceramic sintering

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The final stage of sintering involves two major phenomena occurring simultaneously, *i.e.* pore shrinkage and grain growth. The microstructure evolution of a solid during sintering is then related to the kinetics of change in the grain size and pore distributions. These modifications are directly linked to diffusion processes (and more generally to mass transfers) that generate displacements of grain boundary and pores. Various models were developed to predict these processes but associated direct observations remained very rare. Indeed, even if the knowledge of experimental data is of great importance, only very few direct determinations of kinetic parameters are available, due to some difficulties to develop *in situ* observation methods that allow the visualisation of high temperature processes at the grain scale [1, 2].

Here we will report the direct observation of the microstructure evolution of a ceramic pellet during sintering by the means of a new generation scanning electron microscope (FEI Quanta 200 ESEM-FEG) equipped with a high temperature stage (HT-ESEM). This technique allows recording SEM images while heating the sample up to 1400°C for few hours [3]. Approximately 10 nanometers spatial resolution images are recorded each 1 to 15 minutes (depending on the temperature considered). Then, these images are stacked into videos and exploited image by image to quantify local kinetic phenomena such as grains reorganization, grain size evolution, grain boundaries displacements and porosity elimination.

In this work, the morphological modifications of a unique grains population of oxide ceramic were directly observed in the temperature range 1000 to 1400°C during long-term experiments by the means of HT-ESEM. The evolution of grain growth can thus be plotted through a one-shot experiment for a given temperature. The correlation between the obtained *in situ* data with *ex situ* experimental data validates the accuracy of HT-ESEM runs. The main interest of this method lies in the possibility to follow the microstructure modifications of a unique zone of the material at the grain scale over long periods, with a one minute time resolution (Figure 1). This led to access kinetic parameters, such as pores or grain boundaries mobility, generally unreachable by other techniques. We will compare the experimental data with videos obtained from calculation.

Several examples corresponding to the study of $CeO_2$ or $ThO_2$ will be used to highlight the possibilities offered by the HT-ESEM imaging applied to the study of ceramic sintering process. First, the mass transfer from grain to grain will be directly measured from mathematical treatments on image series. The activation energy of the mass transfer controlling mechanism will be determined from Arrhenius plot of the data. Second, the grain boundary velocities will be determined for each temperature. Finally, two original methods for the rapid determination of sintering maps will be presented: the first one is based on the correlation between data coming from dilatometry and ESEM, the second one being only based upon ESEM image treatment. This original approach offers a new vision of solid state sintering of ceramics that can be extended to a broad variety of microstructure modifications occurring in materials during heat treatment.

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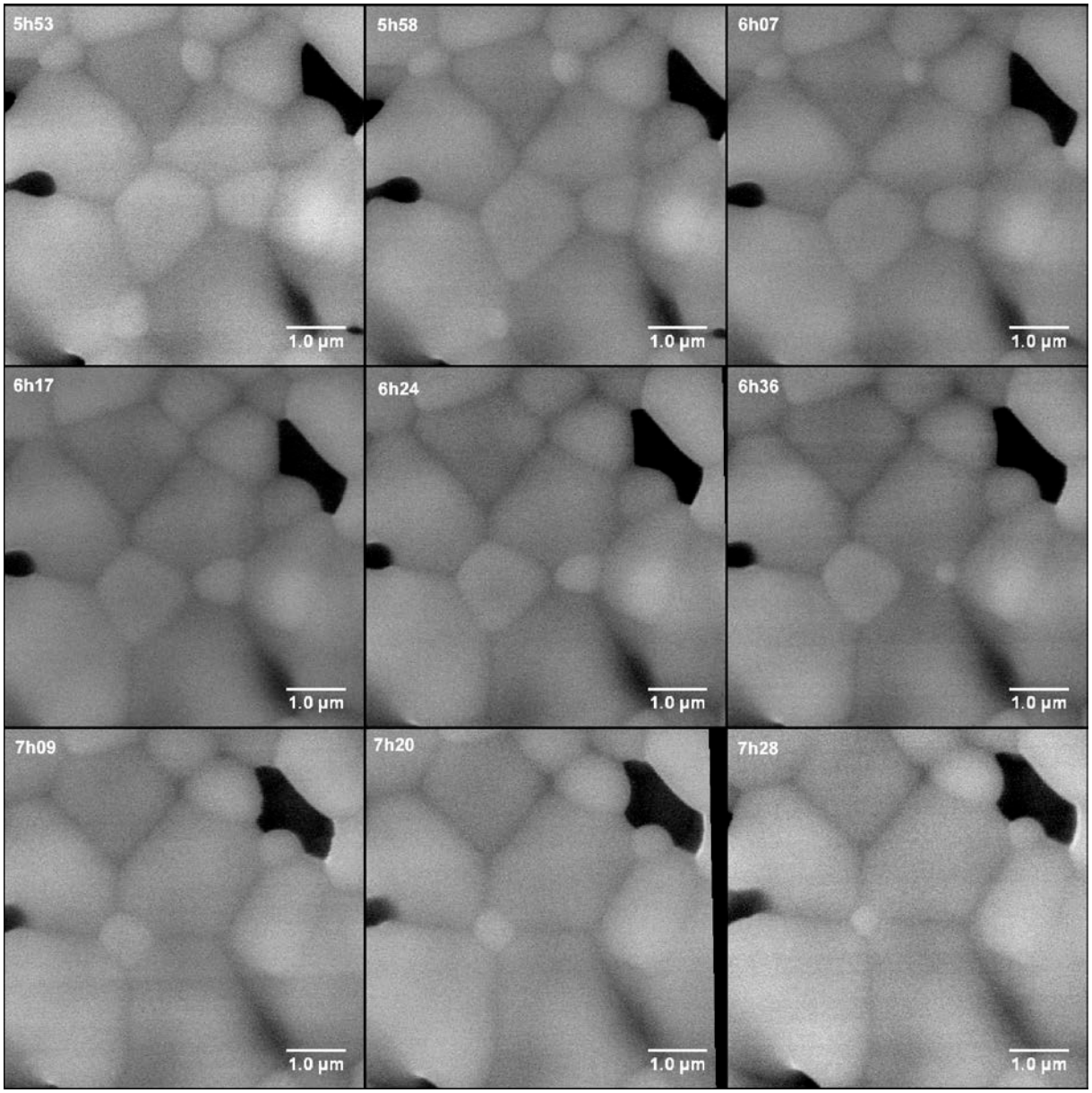


***Figure 1.*** *HT-ESEM image series recorded in situ during the heat treatment of $CeO_2$ at T = 1300°C*