ESRF	**Experiment title:** In situ nanotomography of the sintering of agglomerated ceramic powder	**Experiment number**: MA-5301
Beamline: ID16B	**Date of experiment**: March 1-2, 2022	**Date of report**: 30/06/2022
Shifts: 3	**Local contact(s)**: Julie Villanova	*Received at ESRF:*

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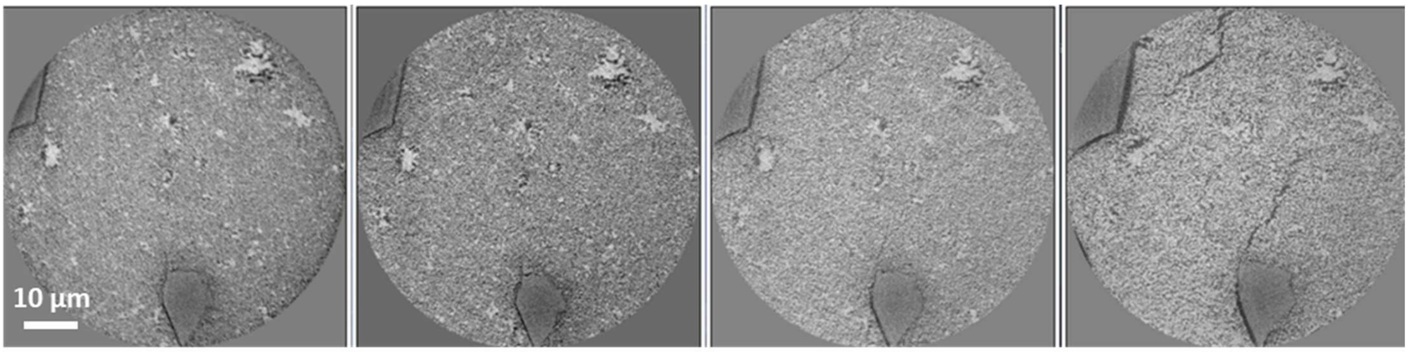
Report:

The selected material is a zinc oxyde powder composed of submicronic particles that are naturally agglomerated in the dry state. This powder was used either alone or mixed with 20 vol.% of alumina particles of about 20 µm size, which are supposed to be inert at ZnO sintering temperature. The powder or powder mix was pressed in a die at room temperature. The resulting compacts were broken into tiny fragments of about 100 µm diameter for in situ sintering experiments.

At the ID16B beamline, we used a phase sensitive nano holo-tomography approach. It generates image contrast stemming from the X-ray phase shift induced by the sample and provides more sensitivity than absorption imaging, as in the case of conventional X-ray CT. The beamline is equipped with a Kirkpatrick-Baez mirror system which focuses the incoming parallel X- ray beam to a small size at the focal plane. The consequent conic beam geometry then provides a large geometrical magnification. Data acquisition is performed at four different sample-to-focus distances, by moving the sample relative to the focal plane. At each distance, the sample is rotated to obtain a complete tomographic scan of over 2000 projections. These collected tomographic projection datasets are used as an input for the phase retrieval to reconstruct the phase shifts and to create 2D phase maps in all angular projections. The retrieved phase maps are then made to undergo a 3D reconstruction procedure using a filtered back projection algorithm implemented in the ESRF PyHST2 software package. Subsequently, the reconstructed image stack is post-processed to remove defects like ring artefacts. Detailed morphological features are revealed, thanks to the extremely brilliant resolution and a large field of view ((64x64x54 µm3) resulting from the multiple distance phase retrieval. The acquired images had a voxel size of 25 nm.

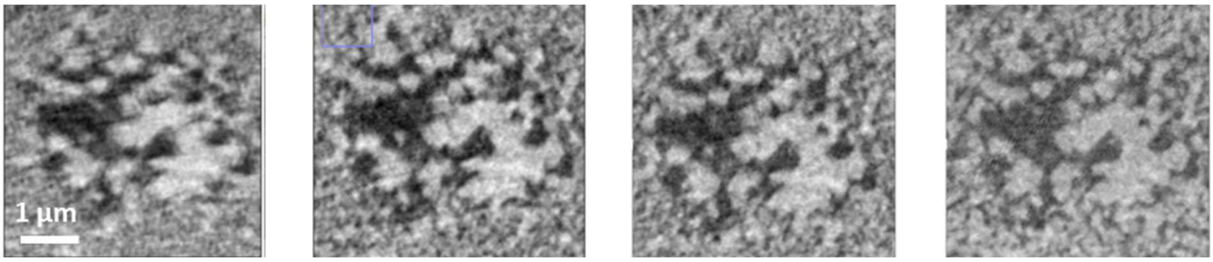
This resolution cannot allow us to accurately identify elementary ZnO particles and interparticle pores but it should provide relevant information on the presence and evolution of larger pores that may be located between agglomerates or close by alumina inclusions or on the formation of cracks during sintering. Following these pores is particularly meaningful because it is most likely that they will remain until the end of sintering and they will thus be detrimental to the properties of the final material.

It was planned to conduct two in-situ tests (one with a ZnO compact and one with a ZnO+Al_2O_3 compact) by using a furnace available at ID16B. The experimental procedure was as follows. The furnace was installed in the hutch on a vertical translation above the turntable. The sample to be studied was mounted on the measuring stand (alumina rod placed on the turntable) and the furnace was lowered around the sample. Then the temperature inside the furnace was increased. At predefined temperature, the sample was moved vertically to a less hot area so as to freeze its microstructure and it is imaged using the same 4-distance holo-tomography technique. It was then reintroduced into the hot zone of the furnace and the process was repeated for several temperature. Unfortunately problems of temperature control perturbed the test run with ZnO compact. Thus only the one with ZnO-alumina mixture has been usable. During this test, 5 sets of image scans have been acquired at room temperature and at 700, 800, 900 and 1000°C, respectively. Virtual cross-sections of these images are displayed in Figure 1. One can identify the matrix composed of partially sintering ZnO particles, ZnO aggregates (in light grey) with porosity inside and around, alumina particles (in dark grey) with porosity around them and cracks that developed in the course of sintering. Details of the evolution of the porosity induced by an aggregate appear in Figure 2. These images are being quantitatively analysed to draw meaningful information from them. It includes the specific investigation of each of the three kinds of “harmful” pores that have been previously identified.



Room temperature 700° 800°C 900°C

Figure 1: Virtual cross-section of 3D images acquired at various temperatures during in situ experiments on ZnO-alumina mixture



Room temperature 700° 800°C 900°C

Figure 2: Local observation of the evolution of porosity induced by particle aggregates

The experiment has thus been very successful, as it provided unique 3D images whose analysis will be very fruitful in understanding the reasons for the presence of defects in sintered nanoceramics. The obtained results will be compiled in the report of Aatreya Venkatesh's PhD. thesis, which will be defended in late 2022. They will be presented at sintering conferences and one or two papers will be written for submission to specialist journals.