ESRF	**Experiment title:** Evolution of porosity and determination of porosity gradient in ceramics at micrometer-scale during sintering	**Experiment number:** HS-892
Beamline: ID19	**Date of experiment:** from: 26-04-99 7:00 to: 27-04-99 7:00	**Date of report:** 11-08-99
Shifts: 3	**Local contact(s):** J. BARUCHEL BOLLER Elodie, LUDWIG Wolfgang (PLUO E)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*HEINTZ Jean-Marc, ICMCB-CNRS, F-33608 PESSAC
*GENDRON Damien, ICMCB-CNRS, F-33608 PESSAC
*BERNARD Dominique, ICMCB-CNRS, F-33608 PESSAC
*VIGNOLES Gérard, LCTS, F-33608 PESSAC
BARUCHEL José, ESRF ID 19 line
BOLLER Elodie, ESRF ID 19 line
LUDWIG Wolfgang, ESRF ID 19 line

Report:

Three shifts were allocated for preliminary investigations of X-ray computerised microtomography of partially densified alumina ceramics. Thanks to the very high efficiency of the ID19 beamline (team and equipment), we were able to scan each specimen at two different resolutions (0.8 µm and 1.8 µm) which was more than initially planned. The samples were examined using a monochromatic beam (17.5 keV) and a bent multilayer device (which decreases strongly the acquisition time). 900 projections were acquired each time using a Gd_2O_3S:Tb scintillator, a light amplification setup and a FRELON CCD camera with 1024x1024 square elements and 14-bit dynamics [1]

The powder under investigation was a pure alumina powder (Baikowski DF 1200) and the average grain size was estimated to be 4 µm. The reference sample which corresponded to the initial state was obtained by cold pressing this powder. A slight sintering was then applied (1300°C, 1 min.) in order to get a minimum of cohesion without any significant change of the microstructure. Two types of sintered samples were studied : i) a powder naturally sintered (= free sintering), ii) a powder layer deposited on an already dense alumina (i.e. sapphire) substrate, and sintered (= constrained sintering). The sintering temperature was fixed to 1600°C and the sintering times were 15 min, 30 min and 60 min. Due to the large initial grain size, relative density of the free samples remained low and was 62.5 %, 67 % and 68 % respectively. The constrained samples were assumed to exhibit even lower relative densities [Letullier 94, Ph.D. thesis, Bordeaux].

Measurements and results

Reference specimen : this sample was examined at 0.9 µm and 1.9 µm resolution.

Sintered specimen : in a first run, for each sintering time, free and constrained samples were put together on the sample holder and examined simultaneously at a 0.9 µm resolution. As a matter of fact, it appeared that reconstruction of these objects could be quite difficult due to the non-axisymmetry of the whole sample (these scans could be considered as peculiar local tomography experiments). So, taking advantage of the low acquisition time, the three samples of each series were then examined individually at both resolutions. A last experiment consisted in scanning the 60-min free-sintered specimen using 1200 projections instead of 900.

As a whole, 18 scans were performed on these alumina ceramics.

During the experiments, a control section was reconstructed systematically for each sample giving us an idea of the quality of the acquired data. Examples of samples sintered during 30 min. are given in Fig. 1 (free) and 2 (constrained). Unfortunately, phase contrast artefacts linked to the low grain size (large number of solid-gas interfaces) superimposed themselves on the grey level image, preventing an easy separation of the two phases (pore, matter) at the micrometer scale.

Forthcoming work :

Due to the large effect of the phase contrast artefact, no sintering simulations based on 3D reconstructions will be considered. However, the comparison of free and constrained samples will be attempted, using numerical analysis of the signal in order to extract information on porosity gradients (estimation of the effect of the substrate on sintering). Image analyses and 3D reconstructions will be realised in the frame of the CM3D research network (4 laboratories in Bordeaux) in collaboration with CREATIS and ESRF (ID19).

A new proposal will be presented, taking into account this preliminary work. Samples will be prepared from a glass powder with a higher grain size (~ 50 µm). Experimental minimisation of phase contrast can also be tried by adjusting the distance of the sample from the detector or by interposing a rotating disk containing holes to obtain a decoherence of the incident beam.

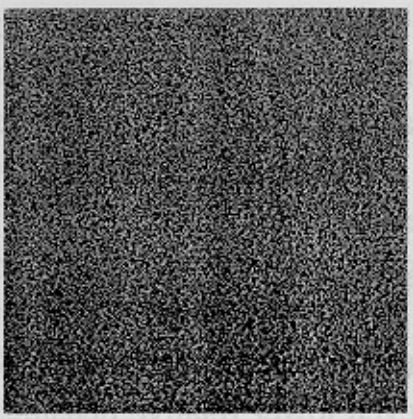

Fig. 1 : free sintered sample

2D slice of tomographic scans of alumina ceramics sintered during 30 min. at 1600°C
Image size : 512x512 pixels
Pixel size : 0.9 μm^2

On figure 2, the uniform layer on the left part is the sapphire substrate

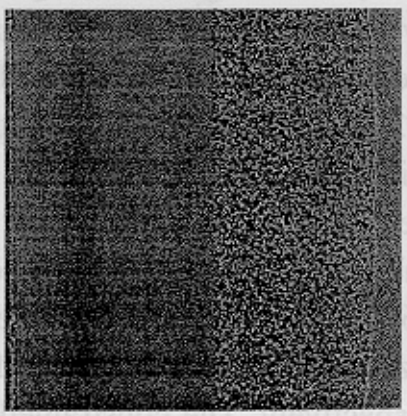

Fig. 2 : constrained sintered sample.