ESRF	**Experiment title:** Pair Distribution Function analysis of densified size-stabilised nano-materials for solid oxide fuel cells	**Experiment number**: HS-3654
Beamline: ID31	**Date of experiment**: from: 12/11/2008 to: 16/11/2008	**Date of report**: 2/2/2010
Shifts: 12	**Local contact(s)**: Dr. Michela Brunelli	*Received at ESRF:*

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Report:

Recently, the possibility of obtaining dense materials with nano - sized structure is of great technological interest in the field of structural materials, where the reduction in particle size has been shown to be able to improve several mechanical properties, as well as in the field of functional materials. The densification of ceramic nanopowders to achieve relative densities >95% maintaining a nanometric grain size has proven to be extremely challenging, particularly when the grain size of the final product is required to remain <20 nm. Ceramic materials, in fact, require usually high temperatures in order to achieve a good degree of densification. However, nanopowders present a very large driving force for grain growth. As a result, high levels of densification can usually be achieved only at the cost of massive grain growth that drives the grain size out of the nanometric range. Recently a relatively new densification technique has shown the potential to overcome most of the difficulties related with the densification of nanopowders. This technique is usually referred to as Field Assisted Sintering (FAST) or Spark Plasma Sintering (SPS)

This is a continuation of experiment HS3611, in which size stabilised powders of ZrO2 and TiO2 were analysed through total scattering and Pair Distribution Function analysis. In the case of densified materials, however, we are planning to concentrate only on ZrO2, synthesised in two different ways and densified in different SPS conditions, as ZrO2 is the most interesting from the technological point of view.

The aim of this experiment is two-fold: on one side we would like to obtain good PDF data at room temperature for the densified materials, in order to check for differences in their local and the average structure with respect to the corresponding powders (which were measured in experiment HS3611). Moreover, we would like to check for the influence of the powder synthesis method on the structural characteristics of the final densified materials. Different SPS conditions will also be checked. On the other side, we would like to investigate the kinetics of the polymorph transformation (from

cubic/tetragonal to the thermodynamically stable monoclinic polymorph). This should lead to an evaluation of the activation energy of the reaction, together with its mechanism.

Many different samples (whole pellets) were measured in total scattering experiments, with a short wavelength (about 0.4 Å), with the aim of obtaining the atomic Pair Distribution Functions of the material densified in different conditions (temperature, pressure and holding time).
The theoretical Qmax with a wavelength of 0.4 Å and a maximum diffraction angle of 115° 2theta is about 27/Å. However, the Qmax actually used for obtaining the PDFs was 22-23/Å. This was due to the fact that, even though data collection times were much longer at higher angles, the counting statistics did not allow the use of data so far away in the reciprocal lattice. This was more than enough to get perfectly usable PDFs.
Each total scattering experiment was of about 5 hours: longer collecting times were tried (up to 9 hours), but they did not provide enough counting statistics to justify an almost double counting time.

We were very happy about the experimental setting at the beamline, in particular of the sample-changing robot that allowed us more sleeping time than is usual at ESRF!