ESRF	**Experiment title:** Local strains probed by Laue microdiffraction into zirconia containing refractory materials	**Experiment number**: 32-02-765
Beamline: BM32	**Date of experiment**: from: 26.09 to: 30.09	**Date of report**: 26.11
Shifts: 12	**Local contact(s)**: Odile Robach	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

René Guinebretiere[a,*], Olivier Castelnau[b,*], Vincent Michel[b,*], Fanny Gouroud[a,*], Taylan Örs[b, c, *]

[a] CNRS UMR 6638 SPCTS ENSCI 47 avenue Albert Thomas - 87065 Limoges, France

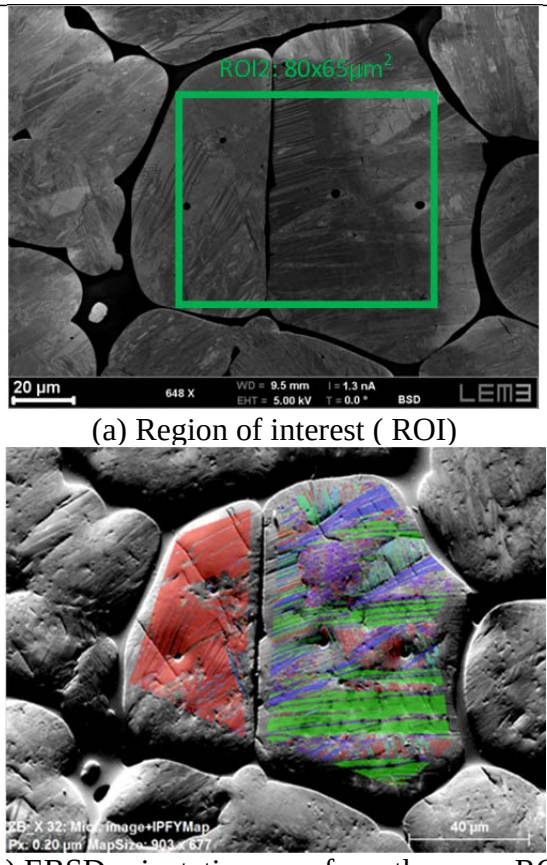
[b] Arts et Metiers ParisTech - CNRS UMR 8006 Laboratoire PIMM 151 Bd de l'hopital - 75013 Paris, France

[c] LEM3 UMR CNRS 7239, Ile du Saulcy - 57045 Metz, France

Report:

Aim

The experiments were conducted within the ANR project called ASZTECH. The project aims to improve the mechanical behaviour of new high performance ZrO_2 based refractory materials at elevated temperatures, which are used as bricks for glass making furnaces by the industrial partner of the project, the St. Gobain company. During the brick manufacturing process ZrO_2 undergoes two different phase transformations upon cooling. Of these, the second one (tetragonal to monoclinic) implies a volumetric expansion of 4%, which leads to internal stresses within the material. The aim of this experiment was then to measure the residual stress at the scale of phase transformation induced strain, i.e. at microstrain levels by Laue microdiffraction (LMD) set-up available in the beamline BM32 at ESRF.



(a) Region of interest (ROI)

(b) EBSD orientation map from the same ROI

Fig. 1. BSE image of sample ZB-A1

Experiment

Due to size and weight constraints it is not possible to measure an entire brick (450×400×250 mm^3 in size, weighing 250kg). Specimens, typically 5x5 mm^2, that are cut out of such bricks were measured instead. Six different samples with a fixed composition (ZrO_2: 95%, SiO_2: 4%, Al_2O_3 < 1%, B_2O_3 < 1%, labelled "ZB") were taken from two different locations (surface and interior parts,

labelled “A” and “B” respectively) of the bricks. These pieces were then subjected to different conditions which are labelled as “1” for no treatment (as-cast) and “2” for heat treatment at 1500°C followed by controlled cooling to room temperature. The treatment labelled as “3” has the same time-temperature profile as “2”, however the cooling is done under compressive stress (2MPa), applied to the centre of the disc shaped specimen. A sample with a different composition as well as a powder sample of the original “ZB” composition was also measured, making a total of eight samples.

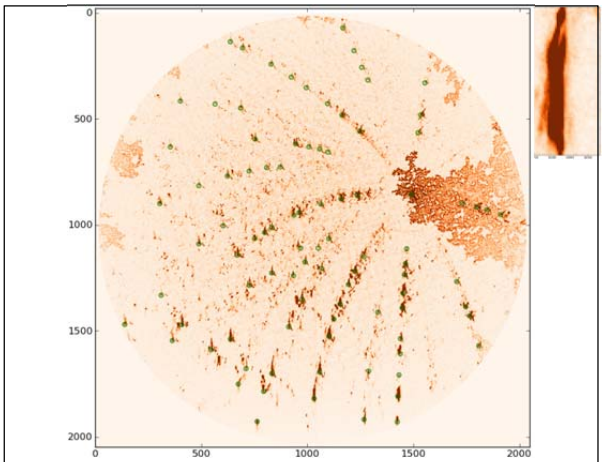
As a preparation for the LMD experiment, scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD) measurements were carried out. During these studies the microstructure of the specimens were investigated and various parts were selected for further analysis, named regions of interest (ROI) (see Fig.1). Needle- or plate-like monoclinic crystallites, or crystallographic domains, have sizes varying between 0.1 to 20 µm and are located within larger zones, which can be as large as couple of hundreds of microns. Further measurements carried out by the EBSD method give important information about the crystallite orientation on the surface (Fig.1b). These data will be used as input for the LMD analysis later on.

Before the LMD measurements on the samples, calibrations on the set-up geometry were done with a Ge single crystal of known crystallographic orientation. The LMD measurements were conducted as step scans of the previously selected ROIs. The beam size used was approximately 0.3x0.3 µm^2. Depending on the measurement, step sizes of 0.5 or 1 µm were used. By this method ROIs between 10-100 µm were measured. Two different sample-to-detector distances were used: 65 mm as standard distance and a further distance of 80 mm to resolve closer Bragg peaks. About 20 ROIs were investigated and more than 30000 images (Laue patterns) were collected from these eight samples.

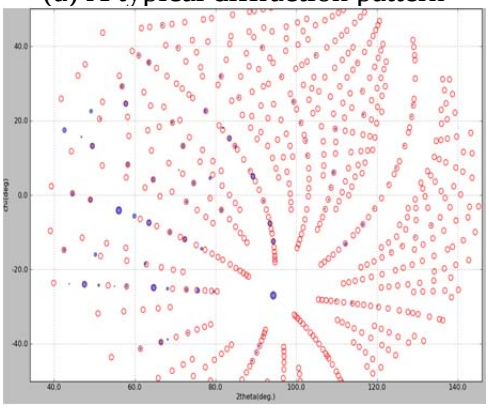
Preliminary Results

A typical image is given in Fig.2a. Most of the analysed points (steps) appear to contain many crystal domains in the diffracting volume, even though a very small beam size (0.3x0.3µm^2) was used. This means the average size of the monoclinic crystallites are even smaller in the submicron level.

The software “LaueTools”, written by BM32-CEA staff, is used for the quantitative data analysis. The main challenge during the analysis of the current data is to have a reliable peak list. As seen in in the insert Fig.2a, the peak shapes are quite distorted. This is caused by the small crystallites of slightly differing orientations. It is then a non-trivial task to obtain a peak list belonging to the same crystal orientation. Once one orientation of the crystallites is singled out in the pattern, indexing could be performed. In a typical pattern around 50 peaks can be indexed (Fig.2b), with a mean deviation of less than 2 pixels. Subsequent strain refinements on these samples yield a variety of deviatoric strain values ranging from 0.1x10^{-3} to 5x10^{-3} depending on the sample and direction.



(a) A typical diffraction pattern



(b) Indexing of the same pattern

Fig. 2. Laue microdiffraction patterns of the ZB-A1 sample.