ESRF	**Experiment title:** The effect of plastic deformation on the distribution of elastic strain in high purity zirconium	**Experiment number:** 02-2-635
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Report:

The present experiment aims at finalizing the PhD work of Nils Letouzé (2001-2005), done in the framework of collaboration between the French CEA, EDF, and CNRS ("CPR SMIRN", a Research Program Contract for the Simulation of Materials for Nuclear Reactors, which supports about 15 theses). Our contribution consists in studying the plastic mechanical behaviour of high purity polycrystalline zirconium by experimental techniques (mostly X-ray and neutron diffraction) completed by theoretical investigations (homogenization methods).

Zirconium polycrystalline alloys are widely used in the nuclear industry, especially as cladding tubes and guide tubes for Pressurized Water Reactors. From the mechanical point of view, zirconium alloys can be considered as composite materials, because they are made of grains with soft and hard crystallographic orientations for plastic deformations. A better understanding of the effective (overall) mechanical properties of these materials is suited for the improvement of alloys actually in use, for the development of new alloys, and for the optimization of elaboration processes. For this, a good knowledge of the mechanical behaviour of individual grain, as well as adequate scale transition models (from the grain to the polycrystal) are necessary.

The plastic deformation of zirconium is strongly anisotropic since dislocations mostly glide on prismatic planes. After plastic deformation, the local residual stress created by the interaction between grains may reach the macroscopic yield stress. Furthermore, a few percent of plastic deformation makes the density of dislocations to increase by several orders of magnitude (hardening effect), which is another source of elastic strain since dislocations distort the crystallographic lattice.

Due to the local anisotropy, the created field of residual stress is mostly dependent of the local orientation of the grain. That is, the distribution of elastic strain in the Euler space can be considered as the signature of the local deformation processes. Its precise characterization is thus essential.

The average mechanical behaviour of grains was investigated by high-resolution X-ray diffraction at the CRG beamline D2AM. The sample was mounted in the 7-circles goniometer. Diffraction in Bragg geometry has been carried out at an energy of 15.5keV. The corresponding attenuation length is 71.4 microns for Zr, i.e. about 4 times larger then the grain size, so that not only the surface grains are measured. A large beam size ($1.5 \times 0.5\,\mathrm{mm}^2$, H $\times$ V) was selected, and the samples were rocked about the theta circle during acquisition (between 0.5 and 2 deg. total amplitude) in order to increase the number of grains scanned simultaneously. Data evaluation reveals that the selected rocking amplitude is the lower limit to obtain statistically representative measurements of the bulk material as they should be for characterizing the volume average distribution of strain in the orientation space. Unfortunately, the acquisition time at D2AM was strictly determined by the rocking amplitude (20 sec. for 2 deg.), and this has strongly limited (by a factor of 5 or so) the amount of collected data. High resolution was achieved by using a Ge 111 crystal analyser and the Ge solid state detector of the beamline.

First, two reference powders (LaB6 - SRM 660b, and Zr) were measured in order to characterize the resolution of the setup. Next, two Zr specimens could be measured. The first specimen has been cooled from 800 deg.C down to room temperature; this treatment creates residual stress due to the strong anisotropy of the dilatation coefficient of the grains. The other specimen was subjected to additional plastic deformation under creep in uniaxial tension at 50 MPa and 400 deg.C (2.5% total strain).

$\{10\bar{1}0\}$, $\{20\bar{2}0\}$, $\{0002\}$, $\{0004\}$, $\{10\bar{1}1\}$, and $\{20\bar{2}2\}$ reflections were measured for 10 different orientations of the specimens with respect to the beam by performing 2-theta scans of about 0.5 deg. amplitude in about 200 steps. Both the position of the diffraction line which characterizes the average strain in the diffracting volume, and the line shape (via the analysis of its Fourier transform) which characterizes the standard deviation of strain in the same volume are of interest for this study, the second quantity being associated with the work hardening of the specimens. It has been found out that the obtained data are of good quality and that the beamline well matches the requirements of this study. The interpretation of data is still not completed at present. The first comparison to the prediction of micromechanical models is however promising. The obtained results will be of major importance for the PhD of N. Letouzé, to be defended in 2005. Final results will be published as soon as possible in international journals.