ESRF	**Experiment title: Single grain analysis of intracrystalline hardening in tensile deformed zirconium polycrystals by X-ray microdiffraction**	**Experiment number**: HS-1057
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Report:

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 is necessary.

The plastic deformation of zirconium is strongly anisotropic since dislocations mostly glide on prismatic planes. During plastic deformation, a strong field of residual stress is created by the interaction between grains. 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 depends mainly on the local orientation of the grain, but also on the microstructure of the material (grain shape, surrounding of the investigated grain, etc…). 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 aim of this experiment was to characterize, with a spatial resolution of a few tenth of microns, the stress state in individual grains of a deformed Zr polycrystal. The detailed experimental setup, developed by our group in collaboration with ID22 scientists, is explained in details in the report of experiment HS-578. Basically, the monochromatic X-ray beam (16 keV) was focussed by a Compound Refractive Lens down to about 10 microns diameter. The specimen was mounted on xyz translation stages inside the 6-circle goniometer that was available at ID22. Single grain diffraction was carried out in Bragg geometry, and the diffraction pattern were recorded by a 2D gas filled detector developed at the ESRF.

For several diffraction peaks to be recorded successively on a given grain, the sample needs to be rotated around that grain with the present setup. In consequence, the spatial resolution of the setup is limited by the size of the sphere of confusion of the goniometer, which was here quite small (about 30 microns) but significantly larger than the beam cross section. However, the main advantage of the proposed setup is that high resolution diffraction can be carried out owing to the use of monochromatic beam. A better compromise between spatial resolution and high-resolution diffraction will probably be attained by the white beam microdiffraction setup actually under development at BM32 (and already available at ALS-7.3.3).

The technique was applied to a zirconium alloy (Zircaloy-2) deformed plastically under traction up to 15% axial strain. 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. The most instructive result obtained here is presented in figure 1, which shows the diffracted intensity recorded with the 2D detector. If the specimen is stress free (like powders), the pattern should be a nice conical (intersection of the diffraction cone and the detector plane), which local radius can be easily calculated and the data corrected from this geometrical effect. For the plastically deformed specimen, it is clear that different part of the same grain, slightly desoriented by 1 or 2 degrees, exhibit very different stress regimes since large variations in the Bragg angle are clearly observed. It can be evaluated that the *intragranular* fluctuation of internal stress in the specimen is of the order of 100 MPa, i.e. of the same order than the macroscopic yield stress.

This experiment has been presented several time at french and international conferences [1-6]. Based on the obtained results, it is planned to pursue similar measurements with a white beam setup in order to favor the spatial resolution (since this was a limitation for the interpretation of our data).

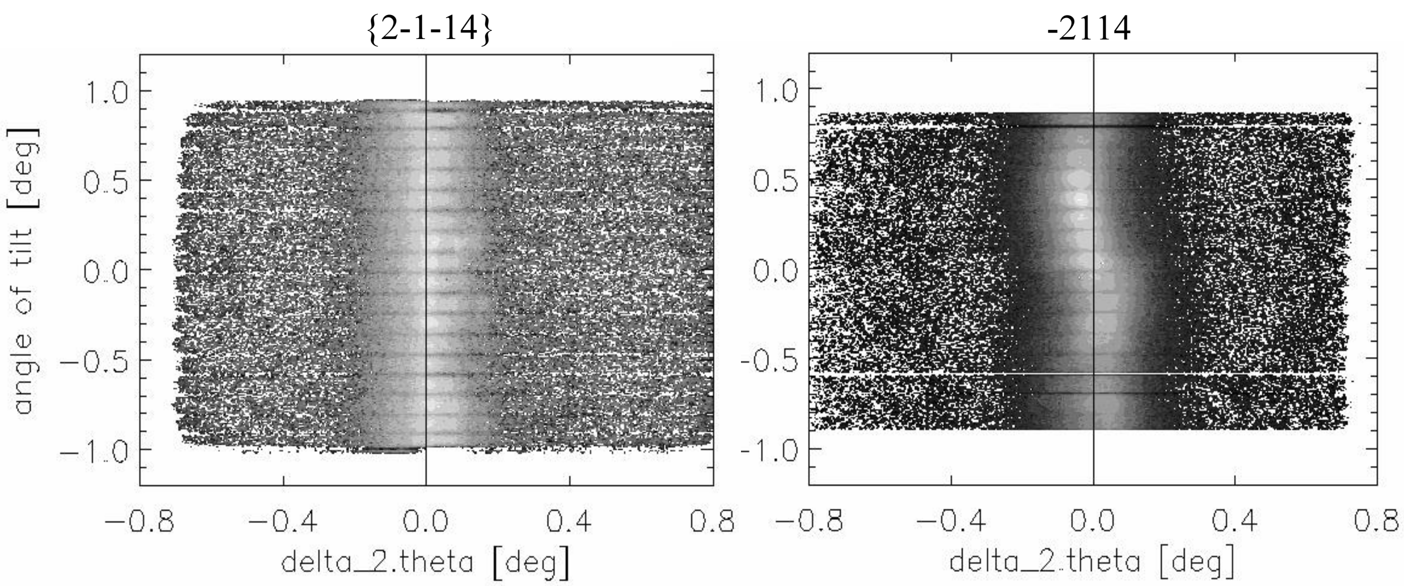

Figure 1. Diffraction pattern recorded on the 2D detector. Left : a reference Zr powder. Right : a single grain of the polycrystalline Zr specimen; here, the deviation of 2-theta with respect to the tilt angle correspond to residual stress of the order of 100 MPa.

[1] Castelnau O., R. Brenner, T. Chauveau, J.L. Béchade, T. Ungar, M. Drakopoulos, A. Snigirev, I. Snigireva, Single grain analysis of the plastic behavior of a polycrystalline Zr alloy with a X-ray microdiffraction technique, 10th ESRF User's Meeting, 10 fév. 2000, Grenoble (France).

[2] Castelnau O., J.L. Béchade, R. Brenner, T. Chauveau, B. Bacroix, T. Ungar, M. Drakopoulos, A. Snigirev, I. Snigireva, Single grain analysis of the plastic behavior of a polycrystalline Zr alloy with a X-ray microdiffraction technique, Europ. Conf. on Adv. in Mechanical Behaviour, Plasticity and Dammage EUROMAT 2000, Tours (France), 7-9 nov. 2000, Eds. D. Miannay, P. Costa, D. François, A. Pineau, p. 911-916.

[3] Castelnau O., J.L. Béchade, R. Brenner, T. Chauveau, B. Bacroix, T. Ungar, M. Drakopoulos, A. Snigirev, I. Snigireva, Analyse grain à grain du comportement plastique d'un alliage de zirconium par microdiffraction X à haute résolution, Colloque MECAMAT, Multi-approche en mécanique des matériaux, Aussois (France), 28/01-01/02 2001.

[4] Castelnau O., J.L. Béchade, R. Brenner, T. Chauveau, T. Ungar, M. Drakopoulos, A. Snigirev, I. Snigireva, Analyse grain à grain de l'écrouissage intracristallin d'un alliage de zirconium par microdiffraction des rayons X à haute résolution, Réunion du GFAC, St-Nazaire (France), 22-23 mars 2001.

[5] Castelnau O., J. Crépin, V. Ji, A.L. Hamon, A.F. Gourgues, G. Régnier, Microdiffraction en faisceau blanc et/ou monochromatique pour la mécanique des Matériaux, Atelier Imagerie - Synchrotron SOLEIL, Orsay (France), 26-28 nov. 2003.

[6] Castelnau O., La microdiffraction, un outil indispensable pour l'analyse mécanique locale des polycristaux, Colloque Micro-diffraction, Orsay (France), 28-29 juin 2004.