ESRF	**Experiment title: In-situ X-ray diffraction studies on formation of recrystallised duplex structures**	**Experiment number**: ME-1191
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Report:

Annealing of deformed metals may result in the nucleation of strain-free, recrystallised grains with a low dislocation density, which grow and eventually consume the surrounding matrix. As the final material properties depend on grain size and texture, it is important to control this process. Fully recrystallised extruded aluminium profiles usually display a duplex-type grain structure with distinct differences between surface and bulk regions. Surface regions normally consist of randomly oriented grains, while a very coarse and strongly textured grain structure (cube) is found closer to the profile centre. These duplex microstructures are the result of different nucleation mechanisms operating in the surface and centre of the profiles, and can in turn be related to through thickness-variations in (i) deformation conditions (temperature and strain rate), (ii) deformation mode (centre: plane strain compression, surface: shear deformation), (iii) texture development as well as possible variations in (iv) stored energy and (v) microchemistry (solute level, and size, density and spatial distribution of precipitates) during extrusion.

The overall objective of this experiment was to investigate the competing growth kinetics that eventually determine the extent of the recrystallised surface region. Samples for ME-1191 were prepared from a 6082-alloy which was subjected to immediate cold water quenching at the output of the extrusion tool. The quenching had been tested and documented to work with no recrystallisation taking place, i.e. the as-extruded deformation energy was to a large extent conserved in the material. This opened for realistic studies of recrystallisation and the evolution of duplex structures through isothermal post extrusion annealing experiments. Supplementary to the investigation on extruded profiles, nucleation and growth kinetics were also studied in an ultra-fine grained (UFG) 6005- and 6060-alloys, since the bulk structures in UFG-variants in many ways resemble the surface deformation structure of extruded aluminium profiles.

The XRD experiments were carried out in a Laue transmission geometry. Since isothermal recrystallisation studies were performed, special care had to be taken both during sample preparation and the design of the furnace; i.e. the furnace had to heat the samples rapidly to the annealing temperature. As a consequence samples with a thickness of only ~300 μm (and cross sections of ~3.0 x 2.5 mm^2) were prepared by spark erosion. A custom made furnace was constructed, consisting of two Al-90wt%Cu metal blocks with Ø0.5mm Pt-resistance wires thread into internal channels and powered by 20V/10A DC. Ceramic wool covered with Al-sheets contributed to reduce heat loss to the surroundings. The furnace was mounted on a small Al-rig fixed onto the combined xyz-ω Huber translation-rotation goniometer sample stage at the beamline, and during the experiment samples were brought into position through a channel in the pre-heated

furnace by a motorized insertion device. Free paths for the incident and diffracted beams were secured by rectangular and conical openings at the entry and exit sides of the furnace, respectively. After insertion, the samples were found to reach the measurement temperature within 1-2 s. Simultaneous to sample insertion, the collection of *in-situ* diffractograms were initiated, employing a FReLoN 2000 CCD detector. The energy of the incident monochromatic beam was 27 keV, while the beam cross section was varied from 0.2x0.2 to 0.5x0.5 mm^2.

Conversion of raw diffraction intensity data to grain volumes was carried out employing a set of in-house developed software routines based on standard procedures for the handling of diffraction data. In the analysis grain volumes has been extracted from the processed intensities, assuming fully kinematical diffraction conditions. All the investigated variants were significantly textured, and a majority of the growing grains tend to fall into these preferred orientations. It has therefore been quite demanding to arrive at a automatic procedure that can separate the diffraction signals corresponding to initial stages of the nucleation and growth of the recrystallised grains from the local and time-varying background which reflects annealing of the initial subgrain structure. With a few attempts it has nevetheless been possible to assign fully automatic procedures for this purpose, although detecatbility towards the earliest stages of growth has been sacrificed for statistical significance. Problems with data extraction due to sample texture was foreseen prior to the experiment, and was the main reason to choose a rather low X-ray energy in order to be able to resolve individual grains. With little or no deformation texture higher X-ray energies would be preferential as that would provide better statistics in terms of observations of individual grains.

Data analysis is soon complete, and the results will be processed for a manuscript in a refereed journal. In the UFG-variants, the intensity-peaks are much sharper, and generally more individual observations are available. From that part of the study a paper has already been written and submitted to the conference Rex & GG III, and another paper will be prepared for a journal submission.

Individual growth curves for grains with <111> and <200> ‖ the extrusion direction (ED) during annealing of UFG-6060 are given in Fig.1 below. Both the average behaviour and the growth characteristics of each grain were extracted from the data, and not surprisingly the grains are associated with individual growth kinetics. It is interesting to notice that while some of the grains begin to grow immediately, others need a certain "incubation time" before the onset of growth.

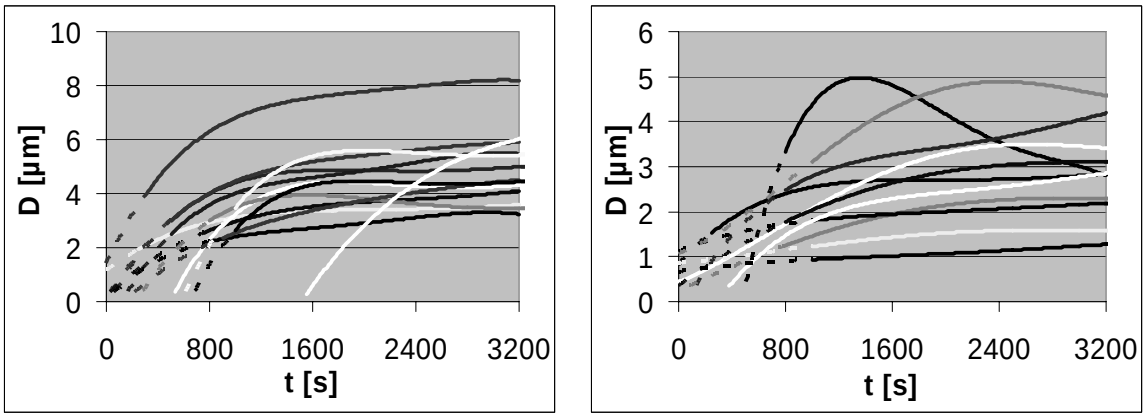

Figure 1: Gain growth in UFG-6060 during annealing at 270°C for individual grains aligned with a) <111> ‖ ED, b) <200> ‖ ED, c) <220> ‖ ED. Note that the degree of rotation around ED is varying for different grains.