ESRF	**Experiment title:** Kinetics of grain boundary motions in nickel during recrystallization	**Experiment number:** HS 25
Beamline: ID 11	**Date of experiment:** from: 1l/12/96 to: 16/12/96	**Date of report:** 24/08/97
Shifts: 15	**Local contact(s):** H.Graafsma	*Received at ESRF:* 3 SEP. 1997

Names and affiliations of applicants (* indicates experimentalists):

S. Garbe[1]*, H. F. Poulsen[1]*, D. Juul Jensen'*, N.C. Krieger Lassen[1]*, P.B. Olesen[1]*, U. Lienert[1,2]*, H. Graafsma[2]

[1]Risø National Laboratory, Materials Department, P.O.Box 49, DK-4000 Roskilde
[2]ESRF

Report:

Aim: The aim was to test a novel method for three dimensional characterization of grain boundaries in polycrystalline materials. The method is based on the use of high energy synchrotron radiation in transmission mode. A combination of a collimated incoming beam and a conical slit is used to define a small gauge volume in the bulk of a specimen. A Copper specimen (3x3x1 mm^3), where the surface previously had been characterized by electron microscopy (EBSP), should be investigated in 3D. In addition the aim was to study kinetics of selected grain boundaries in Nickel.

Set-Up: A mosaic (20") $SrTiO_3$ monochromator providing 50 times more intensity than the standard Si monochromators was installed. The energy was tuned to 87.81 keV, in order to match the (111) and (200) reflections of Copper with the opening angles of the conical slit system /l/. The incoming beam was defined to 20 µm x 20 pm by two cross slit systems. The sample was mounted on x,y,ω-stage behind the slit systems. The gap of the conical slit was 50µm, leading to a 20x20x500 μm^3 trapezoidal gauge volume. Behind the conical slit the diffraction patterns were registered with a tapered CCD-camera. The collimated beam was aligned with the copper surface to within 0.01". As reference positions, the edges of the sample were determined with a precision of 10µm. We report here only on near-surface measurements where direct comparisons to the EBSP technique can be done.

Results: The determined grain orientations and grain morphology are shown in Fig. 1 /2/. At the synchrotron a grid with a step-size of 20μm was performed for each grain. Boundaries were determined by the intensity half-point. To increase accuracy several boundaries were mapped with more than one reflection. The deviation in grain orientations between the two techniques is of order OS", which is the uncertainty of the EBSP technique. The maximum deviation in grain boundary positions were 25μm. Grain boundary positions based on reflections from each of the two neighboring grains - cf. grain P in Fig. 1 - corresponded within 20μm. Due to the time consuming experimental set-up, measurements on Nickel were not attempted.

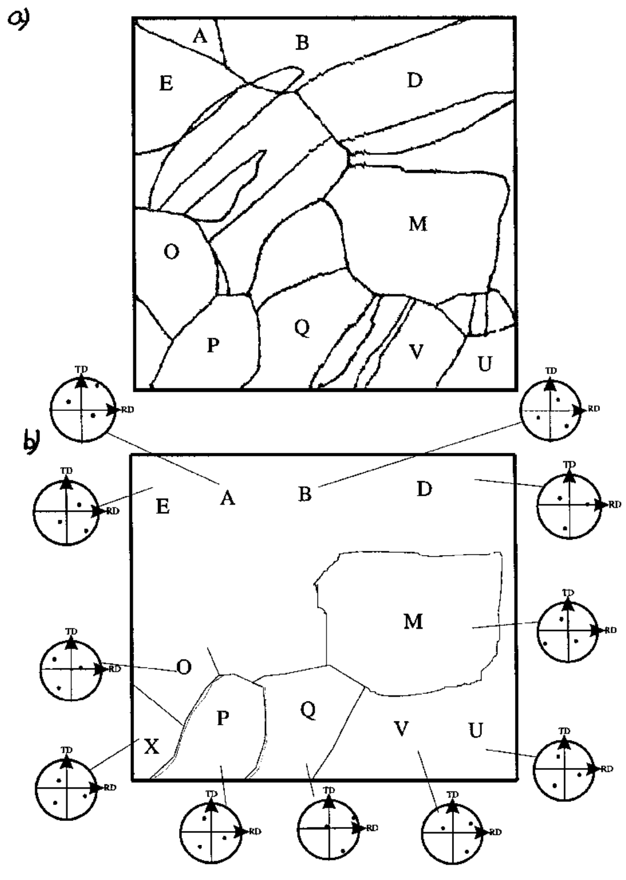

Fig.1: The grain morphology map of the copper sample shown the EBSP-results in a) and the synchrotron results in b). In the pole figures RD describes the rolling dirction and TD the transversal direction of the copper. The two lines at the P grain boundary correspond to a data analysis performed on reflections from the P grain and reflections from neighbouring grains, respectively.

Outlook: The necessary instrumental developments needed to fully exploit this technique were discussed at the ESRF workshop on 'Local characterization of materials'. Conical slits allowing the definition of local volumes of order $5x5x50\,\mu m^3$ are in development.

Ill H.F.Poulsen, **S.Garbe,** T.Lorentzen, D. Juul Jensen, F.W. Poulsen, N.H. Andersen, T.Frello, R.Feidenhans'l H.Graafsma; Journal of Synchrotron Radiation, 4 (1997) 147 - 154

/2/S. **Garbe,** CThomsen, H.Graafsma, H.F.Poulsen, D. Juul Jensen; in preparation for Phil. Mag. A