ESRF	**Experiment title:** Inhomogeneities, structural defects and growth in quasicrystals investigated by diffraction, absorption, and phase imaging	**Experiment number:** HS-686
Beamline: BL16/ID19	**Date of experiment:** from: 23/09/98 to: 29/09/98	**Date of report:** 28/02/99
Shifts: 18	**Local contact(s):** HÄRTWIG Jürgen, MANCINI Lucia	*Received at ESRF:*

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Report:

This report deals with experiments performed during the beam time allocated for the last six months of 1998. Actually, these experiments are part of a long-term project, which was not accepted as proposed, but we are submitting again in a modified version. The project concerns the study of defects in real quasicrystals by X-ray imaging techniques (see attached proposal). The experiments related hereafter aimed to:

1) analyze the effects of high temperature (up to 750 C) annealing on the structural perfection of samples cut into centimeter-sized Al-Pd-Mn single grains, grown by the Czochralski method;

2) check if the defects previously observed [1-4] (holes, precipitates and loop-shaped defects) in grains of the most studied quasicrystalline alloys (Al-Cu-Fe and Al-Pd-Mn) are also present in grains of a new alloy: Zn-Mg-Y.

Annealing effects

The investigations were performed at room temperature, before and after annealing the samples, by combining X-ray diffractometry with X-ray imaging techniques (X-ray diffraction topography and phase radiography). They show that the lattice perfection is strongly dependent on the presence and characteristics of inhomogeneities (faceted micro-holes and/or precipitates) we observed in the QC matrix. We could also demonstrate that holes are related to two-lobes contrasts visible on the monochromatic-beam topographs. Indeed, we observed that the global quality of the investigated Al-Pd-Mn sample, which after annealing did not contain precipitates, turned out to be improved (FWHM > 20 arcsec) with respect to the as-grown state (FWHM > 1' of arc). In this case, we also recorded a simultaneous disappearance of the two-lobes contrasts [1, 3] and a perfect visibility of the faceted holes on the topographs (fig. 1). This phenomenon indicates that the strain-field associated to the presence of holes in the QC matrix is partially (or totally) relaxed, resulting in a better structural quality of the sample. In some case, this relaxation produces the formation of loop-shaped contrast around the hole image [in preparation]. Moreover, the nice contrast of holes on the topographs indicates that this perfection is very high at least in a region corresponding to the holes size (> 20 μm^3). Diffractometry measurements have been performed after annealing. The annealed sample was not polished after the thermal treatment. When any precipitates formation was detected, a linear dependence of FWHM on $Q_\perp$ has been found, as already reported for cleaved and/or polished samples studied in reflection geometry [8], but with a strong decrease of the slope with respect to the as-grown state. This indicates a clear influence of the phason component on the annealing process [in preparation]. On the contrary, we recorded a deterioration of

the global quality of annealed QC samples showing the precipitation of a secondary phase in the matrix [2, 3, 5].

Zn-Mg-Y observations

We investigated by X-ray diffraction and phase radiography a millimeter-sized QC sample of Zn-Mg-Y, as-grown by the flux technique [9] and cut along a fivefold axis. This new icosahedral alloy is interesting for the scientific community, its structure being very different from that of Al-Pd-Mn QCs. For the analyzed sample, phase radiographs did not show the presence of faceted micro-holes in the sample volume, or of precipitates. Monochromatic-beam diffraction topographs show several types of contrasts (fig. 2)): a) loop-shaped contrasts similar to that observed in the topographs of Al-Pd-Mn samples; b) straight lines starting from the seed image (like for crystals grown from solution), oriented along the five twofold axes of the QC and similar to growth-sectors; c) 5 bunches of black straight lines similar to dislocation lines. All these contrasts are under analysis, but these first experiments appear very promising. The FWHM values recorded for this sample are larger (several minutes of arc for the whole sample and 80 arcsec by using a 250x250 μm^2 pinhole) than that measured on as-grown Al-Pd-Mn grains [in preparation].

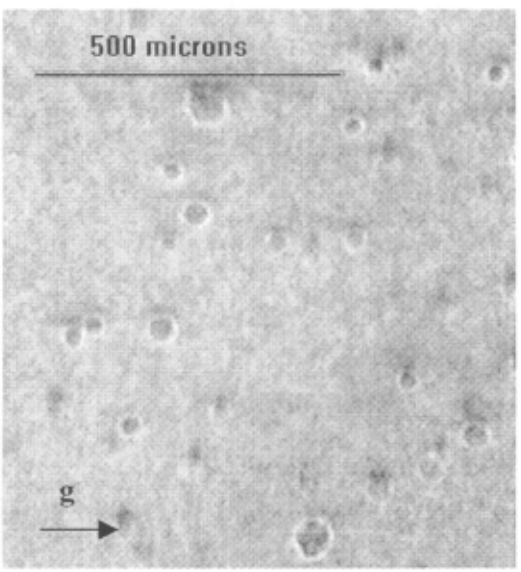

Fig. 1: Monochromatic-beam topograph (E=24 keV) Of an Al-Pd-Mn QC sample after annealing (96 h at 750° C). Faceted holes are well visible on the image.

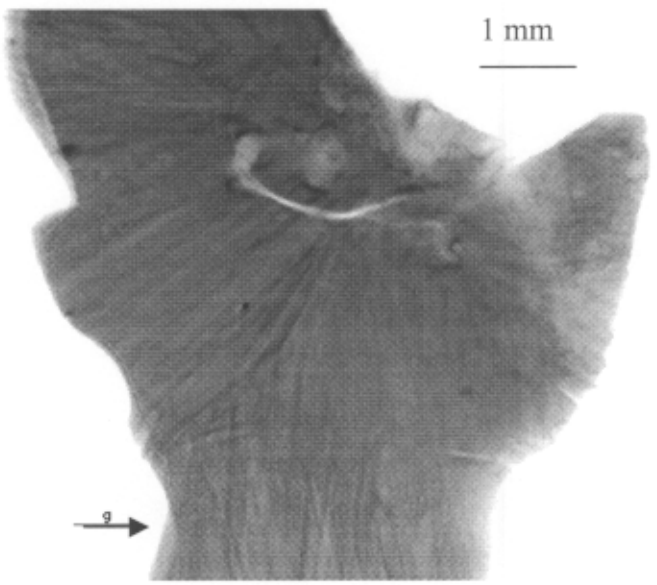

Fig.2: *Integrated* ($\Delta\omega = 0.14°$) monochromatic-beam topograph (E = 50 keV) of a Zn-Mg-Y QC sample in the as-grown state. Several contrasts are visible.

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