ESRF	**Experiment title:** Inhomogeneities, structural defects and growth in quasicrystals investigated by diffraction, absorption, and phase imaging	**Experiment number:** HS556
Beamline: BL16/ID19	**Date of experiment:** from: 04/04/98 to: 07/04/98	**Date of report:** 30/07/98
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Report:

The allocated beam time was used to study structural defects and inhomogeneities in real icosahedral quasicrystals (QCs) we previously observed by X-ray topography and phase contrast radiography in AlPdMn grains [1, 2, 3, 4]. Concerning **structural defects,** it has been proposed, theoretically, that the strain field around QC defects can be considered as the sum of two components refered to two subspaces, being the components of a periodic hyperspace: the physical subspace $E_{//}$ (phonon strain) and the perpendicular one $E_{\perp}$ (phason strain). The mechanical treatments on QCs are expected to result in deformations which are very different from those of the crystals. The first results, obtained on claved and/or polished samples at the D2AM beamline in the reflection geometry [5], showed that the full width at half maximum (FWHM) of the X-ray rocking curves varied as a function of the modulus of the reciprocal-space vector Q components in the parallel space ($Q_{//}$) and in the perpendicular one ($Q_{\perp}$). A linear dependence of FWHM on $Q_{\perp}$ has been found, indicating the presence of a distribution of phasons in the sample, without any particular dependence on $Q_{//}$.

Experiments for the determination of the strain components in a mechanically treated (cut and polished) as-grown sample have been performed at the ID19 beamline of the ESRF in the transmission geometry. The same kind of dependence of FWHM on $Q_{//}$ and $Q_{\perp}$ than in the reflection geometry has been found in this case. These measurements have been repeated, on the same sample, after a high temperature (up to 750°C) annealing treatment. The annealed sample was not polished after the thermal treatment. Again a linear dependence of FWHM on $Q_{\perp}$ has been found. The global quality of the investigated sample, which after annealing did not contain second phase precipitates, turned out to be improved (FWHM >≈ 20'' of arc) with respect to the as-grown state (FWHM >≈ 1' of arc). On the contrary, we recorded a deterioration of the global quality of annealed grains showing the precipitation of a secondary phase in the QC matrix [3,4]. We plan to measure the effect of a controlled mechanical deformation (tribology experiment), on this sample starting from the reference values obtained both in the as-grown and as-annealed states.

We also used the high coherence properties of the ESRF X-ray beams to study **inhomogeneities** (holes and precipitates) in the QC grains. The first reported evidences for the existence of faceted dodecahedral holes in QCs have been obtained using Scanning Electron Microscopy (Beeli and Nissen 1993) and Transmission

Electron Microscopy (Audier, Guyot and Brechet 1990). They correspond to microholes essentially located at the surface of the grains. We showed, by using phase contrast radiography, that dodecahedral holes were present in the volume of all investigated QCs grown along a twofold axis. Faceted holes having a dodecahedron shape, all similarly oriented and having variable sizes, are observed in the as-grown grains phase images. The holes distribution is homogeneous as a function of the depth with respect to the surface. They occupy about the 0.2% of the grain volume. We observed the presence of three families of holes, with a non homogeneous distribution of sizes (-20, -5 and >≈1 µm), which appear to be related by a factor involving τ (the golden mean). This suggested that they could be an intrinsic property of QCs, which could be essential for the quasicrystalline growth and stability.

Thus, we realized phase contrast tomographic experiments on a QC as-grown grain. At first, this study was performed with a spatial resolution of about 6.5 µm. Most of the results we found were in good agreement with our model. In particular the measured hole sizes and their spatial distribution. Nevertheless, the spatial densities measured for the different families of holes, are bigger then that foreseen from the model, and the distance distribution for neighbouring holes (of the bigger family) is flattened (fig. 1). We could explain this discrepancy by the bad thermal conductivity of QC [5]. In order to get a deeper understanding, the same analysis was performed with a spatial resolution of about 1.9 µm (fig. 2).

In order to precise the origin of the holes a theoretical model going in this way, and predicting sizes, densities, mean distances and relative positions of the holes, has been formulated [6]. Phase tomography, through the production of 3D reconstructions of the holes within the quasicrystalline matrix, appears as an unique tool in order to check these models. Further elaboration and investigations are needed to unambiguously determlne the origin of the observed holes and the associated strain field.

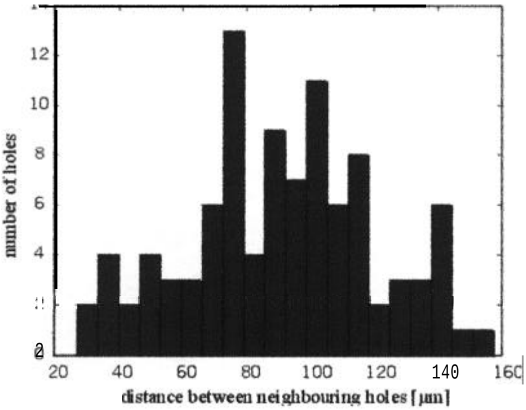

Fig. 1: The distance between nearest-neighbour holes for an as-grown QC AlPdMn single-grain

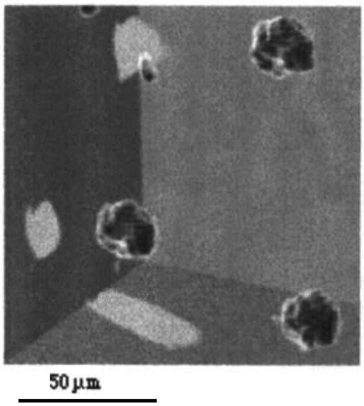

Fig. 2: Phase tomography 3D reconstruction of a part of an as-grown QC AlPdMn grain: 2 families of dodecahedral holes are visible.

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