ESRF	**Experiment title:** Inhomogeneities, structural defects and growth in quasicrystals investigated by in-situ and real time X-ray diffraction, absorption and phase imaging	**Experiment number**: HS-1030
Beamline: ID19	**Date of experiment**: from: 29.09.99 to:15.02.00	**Date of report**: 29.08.00
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Report:

This report deals with experiments carried out during the beam time allocated in the first year of a long term project. The aim of this project is to better understand defects previously observed in icosahedral AlPdMn quasicrystals [1-4]. Defects are believed to have a great impact on the unusual physical properties of quasicrystals. The defects studied are pores and defects that give rise to loop-shaped contrasts in X-ray topography. The loop-shaped contrasts were studied to determine the nature of the defects associated to the contrasts, their geometry and their behavior during annealing. This included a systematic study of the contrast formation mechanisms in synchrotron radiation topography. The pores were examined in a large variety of icosahedral AlPdMn samples in order to determine their origin.

Systematic study of loop shaped contrasts

We carried out a systematic study of typical defect contrasts in X-ray topographs of various single grains of icosahedral Al-Pd-Mn quasicrystals by X-ray topography. In white beam and in monochromatic topography the defect contrasts were studied as a function of the sample to detector distance. Additionally, we recorded monochromatic topography images at different working points on the rocking curve and using different harmonic reflections.

Some general results are the following:

i) Despite the extreme geometrical resolution at ID19 white beam topography showed reasonable (not blurred) contrast only for very short sample-to-detector distances (<4cm) (figure 1).

ii) Depending on the defect type, in synchrotron double crystal topography the defect contrast may change considerably as a function of sample-to-detector distance and of the position of the working point on the rocking curve. A transition from loop shaped contrasts to line shaped contrasts was observed.

iii) Under the special conditions chosen, the dependence of contrast shape and size on the harmonic reflection used was rather weak.

The defects resulting in those types of line- and loop-shaped contrast were precipitates. These results and their consequences on the modelling of the strain field around the defects are currently being analysed.

Porosity

The investigations were carried out at room temperature with phase sensitive radiography. Pores have been observed in all samples. Most of them are faceted with a dodecahedral shape, which reflects the point symmetry of the icosahedral quasicrystal. In the different samples the sizes of the pores fall into one, two or even three families of preferred sizes. There is no evidence of a dependence of the volume fraction of the pores neither on the chemical composition, nor on growth parameters such as growth velocity or the growth

method. However, samples grown under an Argon atmosphere have larger volume fractions of pores. This could indicate a possible influence of Argon on the porosity, a hypothesis that is currently being checked.

Borrmann effect

The perfection of one of the quasicrystal grains with the highest degrees of perfection was checked by means of X-ray diffractometry and X-ray topography. Full widths at half maximum of de-convoluted reflectivity curves were close to the theoretical values and a partial Borrmann effect was clearly visible in the measured reflection and transmission curves, whereas the topographs showed still rather strong contrast, what means that strong deformations (defects) exist in the grain. Consequently, dynamical effects like anomalous transmission may exist in selected quasicrystalline grains, but even then the structural quality is still far from being "highly perfect".

In-situ observations

The observations were performed by a combination of X-ray topography and phase sensitive radiography. The experiment consisted in two short and one long annealing cycles at 750 °C. The black-white double-lobes topographic contrasts related to the pores [4] disappeared during the heating in the first cycle and reappeared as an inverted (white-black) double-lobe contrast after the cooling down in the third cycle (figure 2). During this third cycle we also observed the nucleation of precipitates. This result is complementary to the result of a previous ex-situ annealing at 750ºC of another icosahedral Al-Pd-Mn sample. In this case no precipitates were observed after annealing and the double-lobe contrast disappeared without reappearing with inverted intensities. Both experiments indicate that the strain around the pores, which is the origin of the contrasts, is modified during annealing and depends on the nucleation of precipitates.

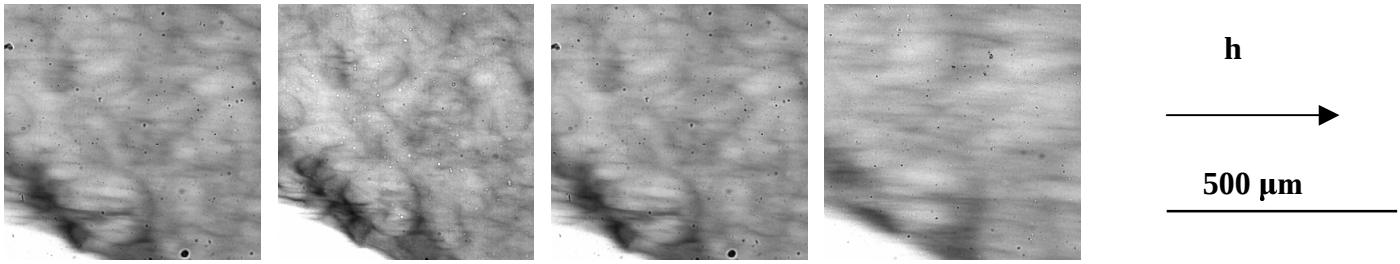

Fig.1: White beam X-ray topographs of a Al-Pd-Mn specimen. The images corresponding to the same defect contrast were recorded at 1.8, 8.5, 15 and 35 cm (left to right) from the sample. At higher distances, the contrast becomes blurred.

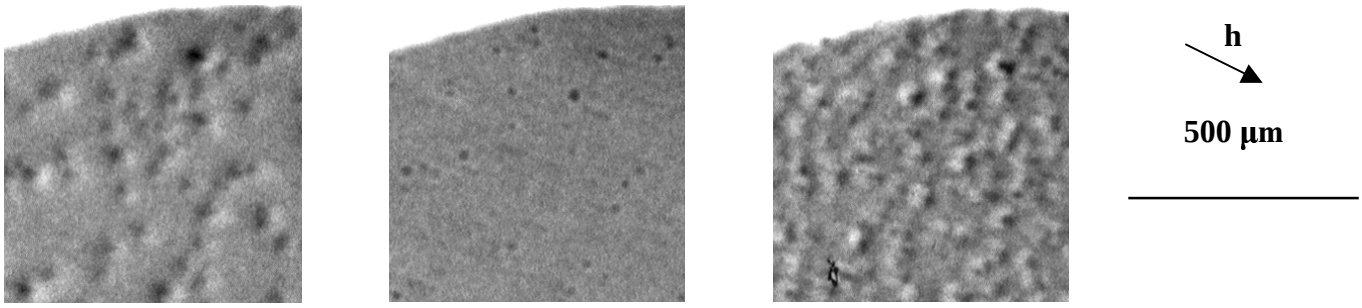

Fig. 2: Monochromatic X-ray topographs (24 keV, 25cm) recorded at room temperature before the first and after the third annealing cycle (left and right) and at 750°C at the beginning of the long annealing (centre).

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