



ESRF

**Experiment title:**

**INVESTIGATION OF QUASICRYSTAL DEFECTS  
BY X-RAY TOPOGRAPHY**

**Experiment  
number:  
HC532**

**Beamline:**

**BL16/ID19**

**Date of experiment:**

from:20/5/96 to:21/5/96 and from: 16/6/96 to: 21/6/96

**Date of report:**

23/07/96

**Shifts:**

15

**Local contact(s):**

**BARUCHEL** José

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### **Report:**

The purpose of this experiment was to continue the synchrotrons X - ray topographic investigation of the microstructure and defects in quasicrystal, we successfully started at LURE, by taking advantage of the ESRF synchrotrons beam, particularly the large number of photons and the low emittance which increases the spatial resolution of the images.

Accordingly we performed both White and Monochromatic beam studies, at room temperature, of the loop shaped defects we observed previously either in AICuFe or in AIPdMn quasicrystal [1]. We focused our investigations on AIPdMn grains grown by the Czochralski technique at the L. T. P.C.M. of Grenoble, in order to determine the influence of further annealing on loops arising directly from growth. Therefore we examined two grains : the first one immediately after growth and then after successive annealing at 750°C and various annealing time, the second one after a 4 x 24 hours annealing at 750°C.

As a general trend the obtained results confirm the existence of loop shaped defects in quasicrystal. They reveal that loops are very small and isolated after growth (from 1  $\mu\text{m}$  to a few 10 pm) and they increase in size and become entangled during further annealings. Such loops have never been observed at a lower level of size, by electron microscopy, neither by us in the same samples, nor by other authors.

White beam investigations give an overview of the whole network of large loops after annealing, but they fail in revealing the small loops in as grown grains. They show that these defects are invisible for some reflections without enabling us to apply unambiguously the extinction rules established for quasicrystal defect images [2].

In the case of monochromatic studies we actually benefit of the exceptional quality of the ESRF synchrotrons beam. On one hand the increased spatial resolution of the images was very efficient to distinguish the small loops in as grown grains and to have an insight of the geometry of the loop entanglement after annealing in which appears necklace shaped figures (fig. 1). On the other hand the high flux allowed us to record tomographs at the feet of diffraction peaks (“weak Beam” imaging) and thereby to gauge the level of distortion in the vicinity of the loops and at their intersections. It was very surprising to discover that these loops have regions, highly disoriented, up to about 6 min. of arc, looking like double streaks or elongated thin loops in case of the grain annealed 4 x 24 hours at 750°C (fig. 2).

Despite all the collected results are still under analysis because the last experiment was ended only three weeks ago, we can already say that they appear as highly promising for the identification of loop shaped defects, particularly the imaging along the rocking curves in the monochromatic mode. Therefore we can expect to identify the strain field around the loops and determine if they are pure phasons loops or dislocations having a strong phason component (strong bl). Moreover the opportunity of “weak beam” imaging (the exposure time being short relatively to other synchrotron sources: about 30 min.) will allow to localize surely the high distortions and to check if they correspond to the phonon component of the loops or (and) to their interaction at their crossing level in the entanglements.

Nevertheless further investigations recording the images produced using several diffraction vectors  $g$  are still necessary in order to precise these results (see the new application).

### REFERENCES

- [1] Gasta. ldi J., Reinier E., Jourdan C., Grange G., Quivy A. and Boudard M. Phil Msg. Lett. 72 (1995) 311.
- [2] Wollgarten M., Gratias D., Zhang Z. and Urban K. Phil. Msg. **A64** (1991) 819.

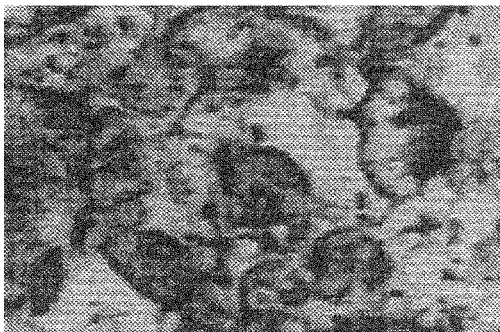


Fig. 1: Loop entanglement appearing as a necklace figure in an AlPdMn grain annealed 4x 24 hours at 750°C. Imaging at the top of the rocking curve.

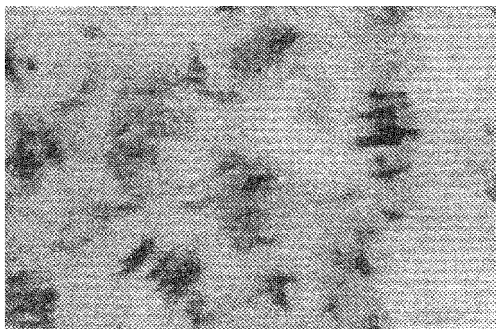


Fig. 2 :“Weak beam” imaging of the loop entanglement of fig. 1. at 196 arc. sec. from the top of the rocking curve.