ESRF

Experiment title: Atomic structure of the AlPdMn icosahedral phase and its crystalline approximant: anomalous diffraction study at the Pd edge

Experiment number: HC-51

Beamline: ID1l-BL2

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Report:

The AlPdMn icosahedral phase belongs to the class of 'perfect' quasicrystals, i.e. the quality of the long range order of these quasicrystals is similar to what is observed in crystalline alloys. The aim of the experiment was to carry out an anomalous experiment at the Pd edge on the quasicrystalline phase, with particular emphasis on weak reflections, to improve the number of reflections measured with respect to a previous experiment carried out at the Pd edge at Brookhaven. It was shown that the partial Pd structure factor could& extracted (l).

In order to assess the data treatment the same measurement has been carried out on a crystalline approximant phase, called the ξ phase: It is an orthorhombic phase with parameters equal to 12.36, 16.57, 23.45A and space group Prima. Its atomic composition is $Al_{71}Pd_{24}Mn_5$ to be compared to $Al_{68.7}Pd_{21.7}Mn_{9.6}$ for the icosahedral phase. Studies on a standard X-ray diffractometer showed that about half of the reflections are too weak to be measured. A model has been proposed, based on this reduced set of data (2).

The experimental procedure was the following:

We used spherical single grains of diameter 100 μm for the crystalline phase and 160 μm for the quasicrystalline one. 2 image plate were exposed altemativell y, a MD and a Fujy one. The distance crystal-film was set to 14 cm. Because of the large dynamical range two set of data have been recorded a each energy: the first one had oscillations of 4° in 50s with a 1° overlap, the other one 7° oscillation in 10s for recording of the strongest reflections, both covering a 90° sector.

For both sample data have been recorded at 22.959 ev and 24320 eV (Pd edge at 24340 eV) corresponding to f' value of -6 and -2. For the crystalline phase, a set of data was recorded 25 eV

above the Pd edge to check for a possible lack of centrosymmetry. For the icosahedral phase a set of data was recorded at an intermediate energy (E=24203eV, f'=-4), to be able to extract the partial Pd structure factor. Finally a set of still photographs were recorded around the principal symmetry axes of the icosahedral phase. The icosahedral phase was previously oriented, the final tuning tuning done on the beam line and manually on the goniometer head (no motorisation available at that time).

Altogether this lead to more than 250 image plates recorded!

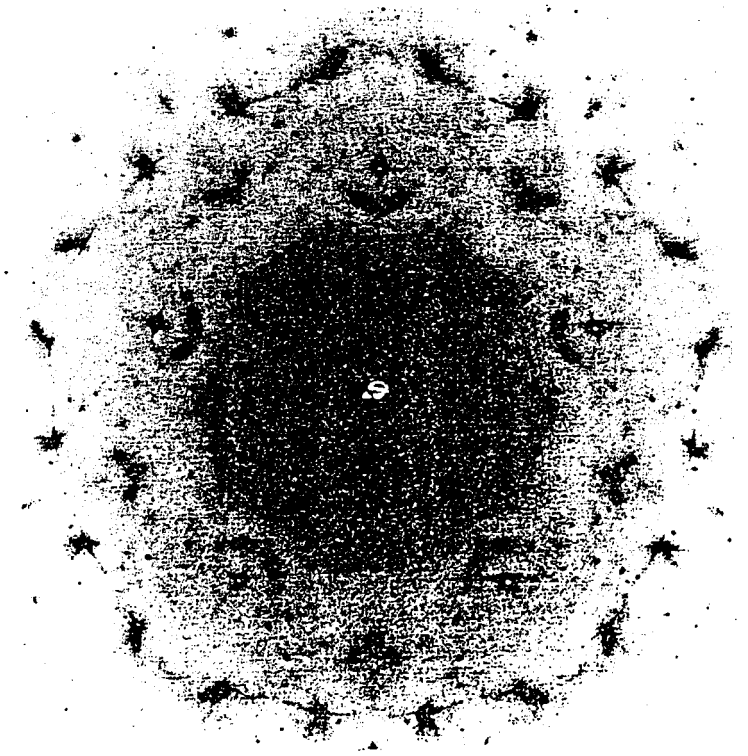
The data treatment was first tempted on the crystalline data set with the Denzo software. Various trial of indexing were unsuccessful. This is rather surprising since the same crystal could be oriented without any difficulties in centring 10 reflections on a standard diffractometer. This might be due to the very large dynamical range of the observed Bragg reflections: there are very strong reflections which produces a pseudo decagonal symmetry.

For the integration of the icosahedral reflections a software is being developed at Zurich by Steurer and coworkers. Recording of reflections have been done on a rotating anode and are being currently analyzed. This software will be used in the near future on the previous data set.

The most straightfoward result has been obtained with the still photograph. In particular the diffuse scattering, already studied with neutron, could be nicelly evidenced. An example is shown on the figure, with a 5-fold symmetry. This shows that the diffuse scattering preserved the icosahedral symmetry, as seen on the figure. Moreover, small rotations close to this 5-fold axis, showed that the majority of the diffuse scattering is related to Bragg reflections, as previously shown.

References

(1) de Boissieu M, Stephens P, Boudard Met al 1994 Phys. Rev. Lett- 723538.
(2) Klein H, de Boissieu M., Boudard M. et al. , Aperiodic 94, in press
(3) de Boissieu M., Boudard M., Hennion B. et at., Phys. Rev. Lett. 95, 3July



Still photograph with a 5-fold axis in the beam. 2 mn exposure time The contrast has been enhanced to show the diffuse scattering. Note that it respects the 5-fold symmetry.