ESRF	**Experiment title: Grazing incidence *X* ray diffraction at the quasicrystal AlPdMn surface perpendicular to 5 fold axis**	**Experiment number:** S1317
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Names and affiliations of applicants (* indicates experimentalists):

J. Chevrier*, G.Cappello*, A. Dechelette*, LEPES CNRS Grenoble BP 166 38042 Grenoble cedex 9 and ESRF

A. Goldman*, Ames Laboratory and Iowa State University (Iowa USA)

M. de Boissieu*, LTPCM/ENSEEG INPG Grenoble

A. Stierle, V. Formoso, F. Comin, L.Ortega, ESRF

Report: Surface diffraction at grazing incidence and x-ray reflectivity have been used to probe the 5 surface of the AlPdMn quasicrystal. A major issue for this experiment was to investigate the long range organization of the surface in connection with the quasicrystalline bulk state after surface preparations based on ion sputtering and annealing at different temperatures. Large effects such as a large departure from the nominal composition after sputtering and recovery after annealing at temperature higher than 700K are well established by LEED and by electron spectroscopy (XPS and Auger). Combination of high resolution surface synchrotron techniques and of surface microscopies (STM and AFM) is required to probe the long range organization of the surface. In this experiment, on the ID32 beam line, a 5 surface of a AlPdMn quasicrystal has been prepared in situ on the transferable UHV chamber. During the week before the synchrotron experiment, the MEED implemented on this chamber could be extensively used to characterize the surface preparation. After a UHV surface preparation identical to the one leading to a well established LEED pattern at a temperature around 750K, a MEED pattern could be reproducibly observed. After transfer on ID32 diffractometer, this surface was refreshed and analyzed by mean of the ID32 X ray beam. This surface presented a broad reflectivity (from a a scan) indicating a rather rough surface. In the reflectivity curve, a well defined bump appeared at twice the critical angle.

A rod scan measured first after sputtering and then after annealing, showed no evidence for a well defined surface signal: the signal between Bragg peaks was even enhanced after sputtering. Finally, well defined “wings” around the specular rods revealed what could be classically analyzed as a faceting of the surface. In conclusion, after annealing at a temperature just above 700K, at lengths probed by the synchrotron radiation, the surface appeared rough, and possibly faceted. From the experimental conditions during surface preparation, such a surface is strongly expected to produce a LEED pattern and a spectroscopic signature of the quasicrystalline surface.

After annealing at temperature close to 900K, a complete, irreversible and rapid change occurred. The bump in the surface reflectivity disappeared. Wings characteristic of faceting were no longer observed. A strong surface signal was observed in the rod scan as shown in figure 1. By continuously scanning across this rodscan at position 1=0.2 during ion bombardment, we could even follow as shown in figure 2 the surface roughening induced by sputtering. All these results are a signature of a flat and well organized surface. The in plane diffraction peaks at incidence lower than the critical angle provided a clear five fold symmetry. Their position was the one expected from the bulk Bragg peaks. Their width gave a surface coherence length of about 400Å. No evidence for a systematic change in their width or position could be identify depending of the peak position in 6D reciprocal space. This experiment then shows that, after annealing at temperature close to 900K, we observe a flat and well organized surface. From all of our results, we have no evidence for a surface reconstruction. The bulk termination of the 5 surface of the AlPdMn quasicrystal provides a reasonable model for the analysis of the present results.

A major point of this experiment is therefore an unexpected change of the surface morphology at high temperature (around 900K from this experiment). Such an experimental observation defines an irreversible surface transformation which asks for a detailed analysis of the surface morphology and of the surface structure in both states. This statement puts our work at the epicenter of the present analysis of the quasicrystalline surfaces.

Figure 1: Rodscan (hkl) on the ordered surface (-) and on the sputtered surface (----) after annealing at a temperature of about 900K.

Figure 2: ω-scan across the rodscan presented in figure 1. The l-value is 1=0.2. Continuous decrease of the surface signal could be followed. Ordered surface (-); sputtered surface (--).