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Report:

In a previous experiment we observed, that annealing of the icosahedral Al-Pd-Mn quasicrystal (QC) in UHV induced a structural change in the surface vicinity [1]. Actually, we observed a progressive Bragg peak splitting induced by annealing on all studied samples. Aim of the experiment on D2AM was then to study the so defined structural changes in detail. We compared therefore the structure of samples cut in the same single grain but exposed to different temperature treatments.

Descriptions of the Al-Pd-Mn QC are based on a 6D hyperscape. The high number of different reflections along one direction (an intrinsic property of QC) allowed us to determine the 6D lattice parameter with very high precision. In fact, the 6D lattice parameter a can be expressed as follows ((N,M) characterize the Bragg reflection, λ is the X-ray wave length, Θ_{Bragg} the Bragg angle):

$$a = \frac{\lambda}{2\sqrt{2(2+\tau)}} \cdot \frac{\sqrt{N+M\tau}}{sin\Theta_{Bragg}} = \underbrace{\frac{\lambda\sqrt{N+M\tau}}{2\sqrt{2(2+\tau)}}}_{\Lambda(N,M)} \frac{1}{sin\Theta_{Bragg}} = \frac{\Lambda(N,M)}{sin\Theta_{Bragg}} \qquad (1)$$

We collected the data using the D2AM 7-circle diffractometer. In addition we mounted a Si crystal using the Si(111) reflection to analyze the reflected beam. This set-up corrected slight sample misalignments. Our tests showed the thus increased precision in the lattice parameter determination. In the experiment, we used only one incidence wavelength and tilted the sample in order to excite the different Bragg reflections. An intrinsic consequence of this experimental approach is a strong variation of the penetration depth and therefore of the probed sample thickness.

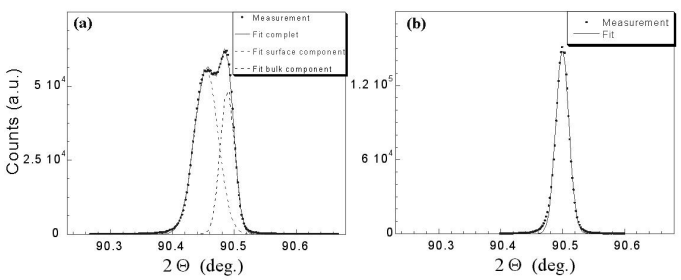

Fig1.: 147-236 Bragg reflection on transformed sample (a) and after strong repolishing (b).

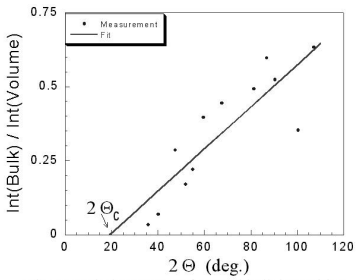

Fig. 2: Ratio between surface and bulk intensities of different Bragg reflections versus X ray incidence angles.

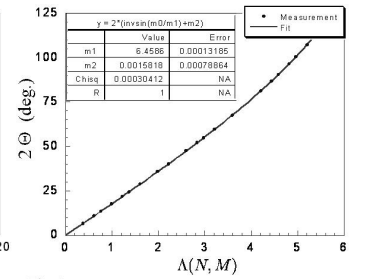

Fig. 3: Lattice parameter determination of the surface structure on the transformed sample. We trace the Bragg peak positions versus the reduced variable Λ(N,M)

In fig. 1 (a) we show the 147-236 Bragg reflection on the transformed sample. We identify clearly two structural components which fit to the sum of two gaussians. We obtained similar results for the other measured reflections on the same sample, when the X ray penetration depth is large enough. Though the shapes of the measured spectra differ between the different reflections, they all fit excellently to two Gaussians. For lower penetration depths we measured however only one feature in the spectra. In fig. 2 we trace, the integrated ratio between both features, when the penetration depth (incidence angle) is varied. We determine thus a critical angle $\Theta_C = 10°$. For lower angles we cannot see the right feature (bulk feature). This is a clear indication, that the structural change is located in surface vicinity. Considering the penetration depth corresponding to the critical angle Θ_C, we determine the thickness of the changed 'surface' structure region to $6\mu m$. A further verification of the surface character of the structural transformation yielded another experiment. In fact, after re-polishing of the transformed sample the surface feature disappeared for all Bragg reflections (cf. fig. 1 (b)). Considering other experiments probing the surface morphology after temperature treatments, we can identify the changed structure as a zone depleted in structural vacancies. In fact, we observe, that annealing of i-AlPdMn samples induces a strong vacancy condensation towards the surface [2].

In order to determine the 6D lattice parameter perpendicular to the surface, we fitted the exact Bragg peak positions of the different structures to the Bragg's law for QC (cf. eq. 1). This is exemplified in fig. 3 for the surface structure of the transformed sample. We determined thus the 6D lattice parameter a of surface and bulk region to $6.4586 \pm 0.0002\mathring{A}$ and $6.4568 \pm 0.0003\mathring{A}$ respectively. For the different other studied samples we determined also lattice parameters with high precision. Statements about strain fields in the different samples are also possible, need however further detailed analysis.

We have already presented these results on the international conference of Quasicrystals in Sendai (Japan) in September 2001. Furthermore, we describe the results in detail in a paper which we will submit to PRL [2].

References

[1] F. Schmithüsen, G. Cappello, S. Decossas, G. Torricelli, T.-L. Lee, M. de Boissieu, Y. Calvayrac, T. Lograsso, F. Comin and J. Chevrier, to appear in MRS Symp. Proc., Boston (2000)

[2] F. Schmithüsen, J. Chevrier, F. Comin, M. de Boissieu
to be submitted to PRL