# X-Ray diffraction studies on 3D magnetic nanoparticle assemblies

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Fundamental research on magnetic nanostructures is an important part of today's scientific effort in information technology [1].In particular, self-assembled structures of magnetic nanoparticles are candidates for a new generation of improved magnetic storage media [2]. For optimizing the performance and reliability of such devices it is important to understand and control structure and shape of individual nanoparticles as well as of 2D and 3D self-assembled particles inside ordered superstructures. The latter is particularly important for applications as self assembling processes are applicable for low-cost mass production of very small structures. Therefore it is of great interest to study the superstructure of nanoparticles in a single, higher dimensional arrangement. The samples that were investigated contain spherical iron oxide nanoparticles with narrow size distribution (ca. 10 nm diameter). The nanoparticles have been deposited on Si substrates by a drop casting method in a magnetic field to achieve a highly ordered arrangement. These 3D ordered arrangements of nanoparticles are called mesocrystals, in anaology to crystals formed by atoms. The mesocrystals grow as pillars on a flat silicon substrate (Fig. 1). The samples under investigation were single mesocrystals separated using a focused ion beam (Fig. 2a).

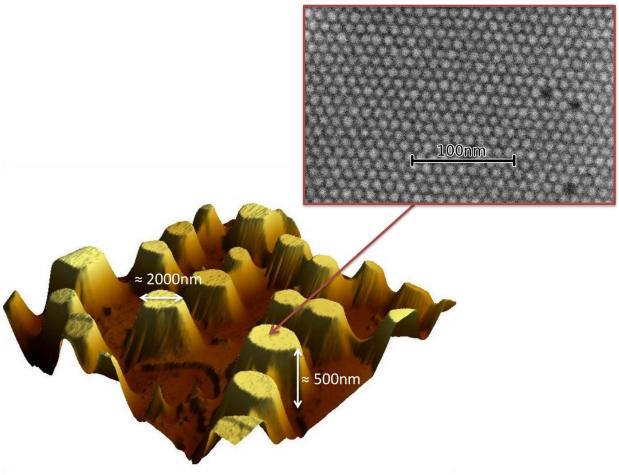


Fig. 1 SEM measurement from the top of a mesocrystal and overview of the arrangement of mesocrystals on a substrate with AFM

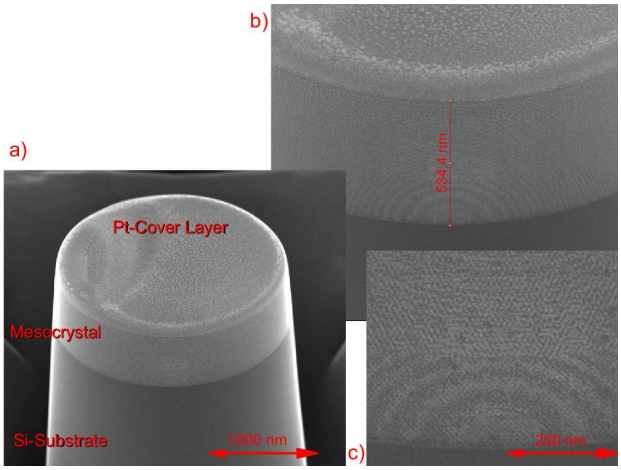


Fig. 2 Single mesocrystal extracted from a structure as in Fig. 1. The size is 2000 nm in diameter and 500 nm in height. The zoom (c) shows a perfect mesocrystalline order.

The mesocrystals were about 2000 nm in diameter and 500 nm in height. They were deposited on silicon substrates and have Pt-cover layers. The super structure of the nanoparticles was prechar-acterized using SEM (Fig. 2c). The purpose of this study was to use a completely new approach: Performing a diffraction experiment on a single isolated mesocrystal with a microfocused x-ray beam. This should make it possible to measure the mesocrystal peaks without powder average and without background from disordered regions, as was the case in previous experiments.

A diffraction study under small diffraction angles was performed at the P08 instrument in order to get information on the structure of a single highly ordered Mesocrystal. A special setup was built to fix the single Mesocrystals on the sample holder (Fig. 3) and using goniometers would be adjusted such that the (001) direction of the Mesocrystal is parallel to the axis of the phi circle (vertical to

the beam), in order to rotate around the z-axis to reach different hk-directions in the plane while keeping the l-component fixed. The detector position was fixed at 1 m distance from the sample and the energy to 22,118 keV to have sufficient q-range, to reach many reflexes at a time and to avoid beam damage to the sample. Optic's elements in the optic hutch (far away from the sample position) generate a vertical microfucus of 20 $\mu$m with a focus far away from the sample position.
On the first day the experimental setup was built, the beamline was aligned and a test of the newly installed ccd detector (a replacement) was performed. The sample was subsequently installed in the first night. For the rough positioning of the sample position, we used a NaJ point detector measuring the beam attenuation due to the Cu sample holder (attenuation due to the small mesocrystal or silicon substrate was too weak to be detected). The next step was to carry out a precise adjustment with the Si (400) reflection to define the exact out of plane direction. To locate the small silicon substrate (5x10x10 $\mu m^3$) and it's Si(400) reflection, the ccd detector was used. However, there were several technical problems with the newly installed ccd detector resulting in extremely frequent crashes of the ccd server software necessitated hardware resets inside the hutch. Above-average beam drops at Petra III and suboptimal beam stability further complicated the search for the (400) reflection and resulted in a large loss of time. Despite of these difficulties, we eventually found the Si(400) reflection Fig. 4. In the next step after the adjustment we lost the reflex and by going back to same position, which was tuned before, no reflection was present before adjusting for a beam movement. Even after readjusting the Si reflection further improvement of the sample alignment was not possible as the positioning was not reproducible because of the low beam instability (The sample is less 2 $\mu$m in diameter). On this account it was not possible to measure reflections from the mesocrystal itself, although we were close to finding the mesocrystal when the time ran out.
Besides all obstacles the test experiment could show, that it is possible to align such small samples in the x-ray beam and that the intensities are quite reasonable (The (5x10x10 $\mu m^3$) Si crystal reflection saturated the ccd detector). With an improved beam stability and the use of lower x-ray energies, to make it possible to use the microfocussing optics, this stability could be achieved which would make this interesting and challenging experiment suitable.

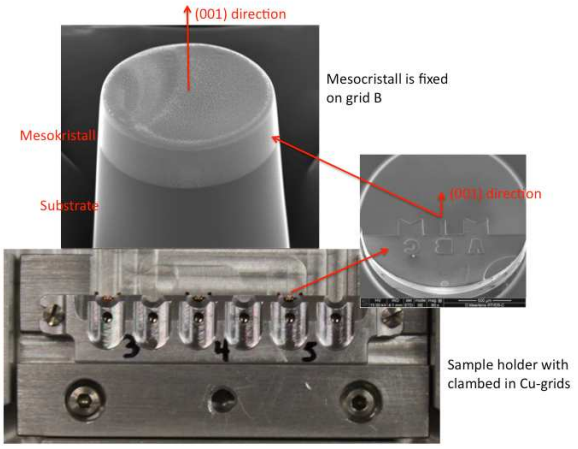


Fig. 3 sample holder and alignment of the sample

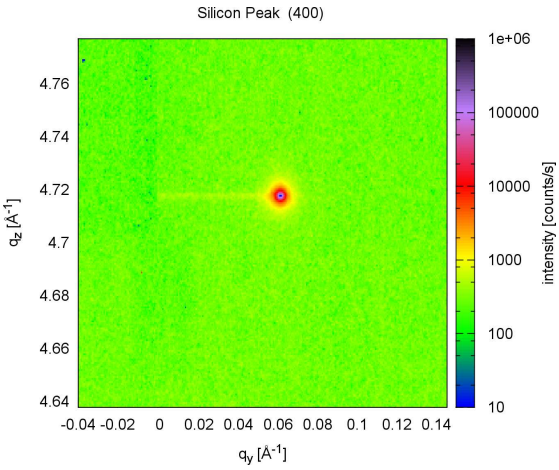


Fig. 4 (400) Silicon Peak of the substrate

# References


[1] A. Moser, K. Takano, D.T. Margulies, M. Albrecht, Y. Sonobe, Y. Ikeda, S.H. Sun, E.E. Fullerton, Journal of Physics D-Applied Physics 35, R157-R167 (2002).

[2] F.M. Fowkes, Y.C. Huang, B.A. Shah, M.J. Kulp, T.B. Lloyd, Colloids and Surf 29, 243 (1988).