The mechanism of crystal growth of nanosized zeolites with LTA and FAU type structures in colloidal solutions and on glass substrates has been investigated by applying a new strategy for *in-situ* GID measurements using synchrotron X-ray radiation. This approach has been developed at ID1 and allows investigation of the self-assembly process of nano-structures in thin films with different thickness on various planar substrates.

A reactor for *in-situ* synthesis of zeolites has been build and inserted in the 4-circle diffractometer of ID1 beam line (**Fig. 1**). For convenience five reactors have been made, which allows investigation of zeolite crystal growth at different temperatures, from different precursor solutions, and on various substrates. The X-ray energy was set to 15 keV, resulting in a transmission of 60 % through 1 cm of the solution.

The zeolites were synthesized *ex-situ* at 60 °C and 90 °C in a conventional oven placed next to ID01, and the X-ray patterns were collected after defined crystallization time. The glass substrates seeded by spin-coating of stable colloidal suspensions were fixed vertically in the synthesis container by 6 Teflon posts (see **Fig. 1**).

Experimental results for LTA zeolite grown in solution and on glass plate are shown in **Fig. 2**. In the angular range, 2θ =3-10^o, all Bragg reflections characteristics for LTA type structure were found in the solution, while only two peaks indexed as *(200)* and *(220)* were collected for the film grown on glass. It is clear that a large fraction of the crystals in the film are oriented with their *c* axis directed perpendicular to the substrate surface, since all measured reflections have *(hk0)* values (**Fig. 2**).

Depth-dependent structural information on a certain lattice plane was obtained by keeping the corresponding $2\theta_{(hkl)}$ constant and varying α_i. The best depth sensitivity is reached when the intensity distribution along the exit angles is investigated, and α_i and 2θ is kept constant and the α_f-intensity distribution is recorded. The intensity of the two Bragg reflections at 2θ = 3.86 and 5.47^o in the LTA film and solution are plotted as a function of the exit angle, α_f (**Fig. 3**). The curve with a pronounced maximum at the critical angle α_c=0.02^o, characteristic for single crystals, is measured for the film, which looks completely different from bulk sample due to the fact that the crystals are growing and distributed randomly in the solutions.

In addition, many pure silica and Fe-containing mesoporous films have been prepared by evaporation-induced self-assembly *via* spin coating. They have been investigated in grazing-incidence geometry in order to determine the orientation of the mesoporous channels. The results suggest that mesoporous films possess three-dimensional organization of mesopores with domains showing orientation normal to the substrate.

The *in-situ* GID study of the zeolite crystal growth in both bulk and film has been successfully performed for the first time and provide more insight into the crystallization phenomena.

The experiments were carried out recently (end of January 2002) and the data are currently under evaluation. Based on the obtained results several papers will be prepared and submit in the near future. Some of these results have been shown at the 14 DZT-Frankfurt, and also they will be reported at the EMRS 2002-Strasbourg, symposium I 'Synchrotron radiation and materials science'.

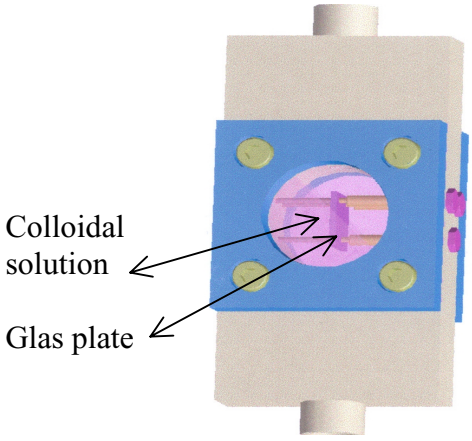

Fig. 1. Container for in-situ X-ray measurements of zeolite crystal growth.

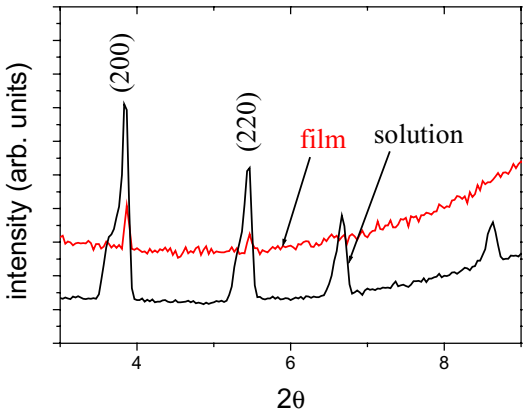

Fig. 2 Radial (2θ)-scans of LTA zeolite in solution (black) and on glass plate (red) in the 2θ range 3-10°.

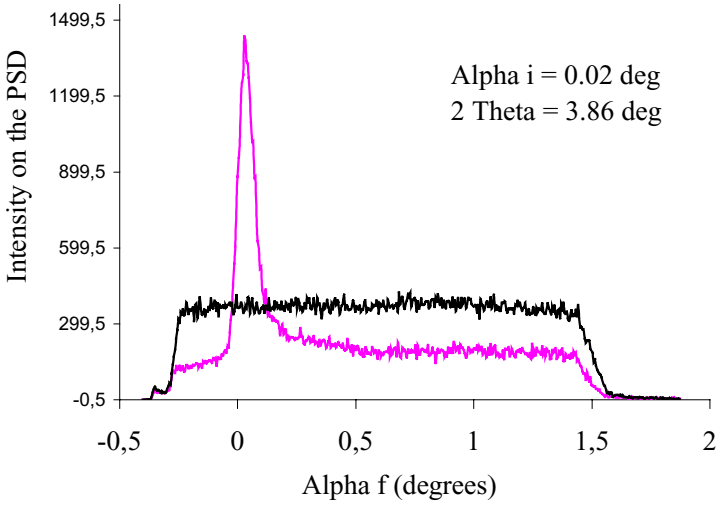

Fig. 3 Intensity of Bragg reflection as a function of α_f at the (200) peak of the film (pink) and bulk sample (black), respectively. Only the film sample shows the expected maximum at the critical angle α_c=0.02°.