2.14 Layer-by-layer growth of thin epitaxial Fe_3Si films on GaAs(001)

Molecular-beam epitaxy (MBE) allows the controlled fabrication of epitaxial layer sequences with very sharp interfaces. The present work aims at the in-situ characterization of the Fe_3Si epitaxial growth process in the layer-by-layer growth mode by x-ray surface diffraction methods in order to achieve high-quality interfaces. The long-range ordering in the films is monitored by measuring different Fe_3Si superlattice reflections. The Fe_3Si films were grown on GaAs(001) templates by solid-source MBE in a chamber built into the six-circle diffractometer at the wiggler beamline U125/2 KMC (PHARAO) at the storage ring BESSY in Berlin.

The GaAs(001) templates were prepared in a separate III-V growth chamber using standard GaAs growth techniques. After capping the samples by As, the samples were transferred into the system at BESSY for the Fe_3Si deposition by means of an ultra-high-vacuum (UHV) shuttle. The As cap was removed by annealing the sample in the preparation chamber before transferring it into the growth chamber. The Fe_3Si layers were grown on the As-rich GaAs surface at different substrate temperatures T_s near 200 °C, which is the optimum growth temperature for structural and interfacial perfection and a high degree of long-range atomic order. The Si and the Fe cell temperatures were tuned in order to achieve perfect lattice match of the Fe_3Si to the GaAs substrate and, at the same time, full stoichiometry of the films. During this process, the Si content of the film was determined from the position of the Fe_3Si layer peak.

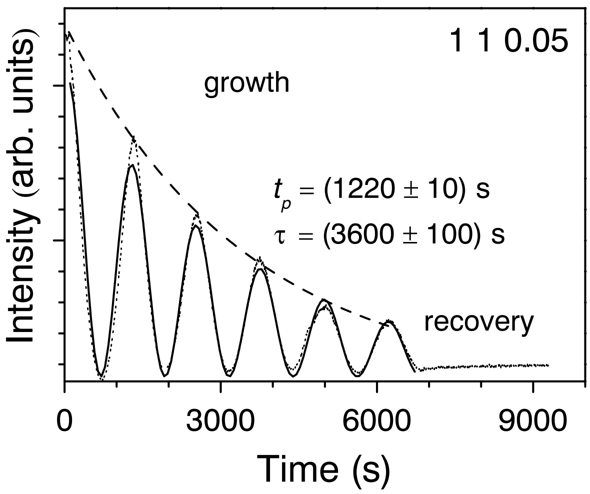

Fig. 36. X-ray oscillations (dotted line) with a period t_p observed by grazing-incidence diffractometry during the MBE growth of an Fe_3Si epitaxial layer on GaAs(001) at $T_s = 220$ °C. For the determination of the period t_p and the decay time τ, the measured curve is approximated by a function proportional to $\sin^2(\pi t/t_p)\ \exp(-t/\tau)$ (solid line).

We obtained optimum temperatures of 1239 and 1370 °C for the Fe and the Si cells, respectively, and achieved an almost perfect stoichiometry of the samples during and after growth. Annealing of the surface at 310 °C for about 1 h, while growing one monolayer (ML) of Fe_3Si, improved the surface quality considerably, as determined in situ by the increasing intensity of the x-ray surface reflections. The x-ray intensity oscillations shown in Fig. 36 were obtained after such an annealing procedure, demonstrating that Fe_3Si grows layer by layer. The intensity oscillations exhibit damping, indicating that several terrace levels contribute to the diffracted intensity. This is confirmed by subsequent atomic-force-microscope (AFM) measurements. One ML is grown in $t_p = 1220$ s. The time constant of the damping is $\tau = 1$ h. The growth rate was confirmed by comparison of rod scans with dynamic simulations using the layer thickness as a fitting parameter. The recovery of the diffracted intensity after the

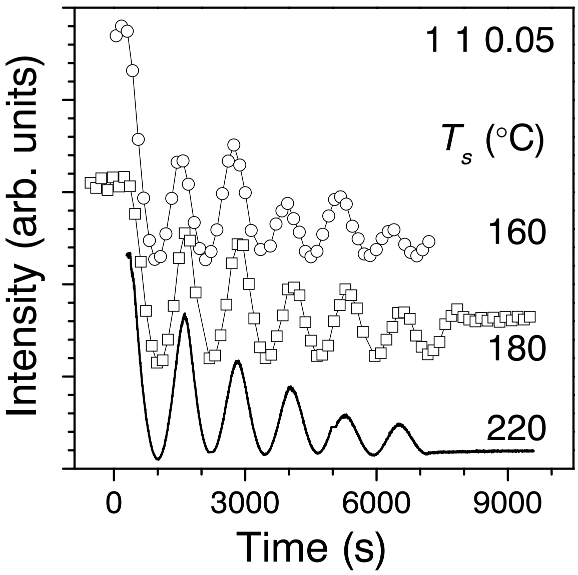

Fig. 37. Comparison of the x-ray oscillations measured during Fe_3Si growth at $T_s = 160$, 180, and 220 °C. Each open symbol is derived from a measurement of a complete rocking curve. The data for 220 °C are a direct measurement of the time-dependent diffracted intensity.

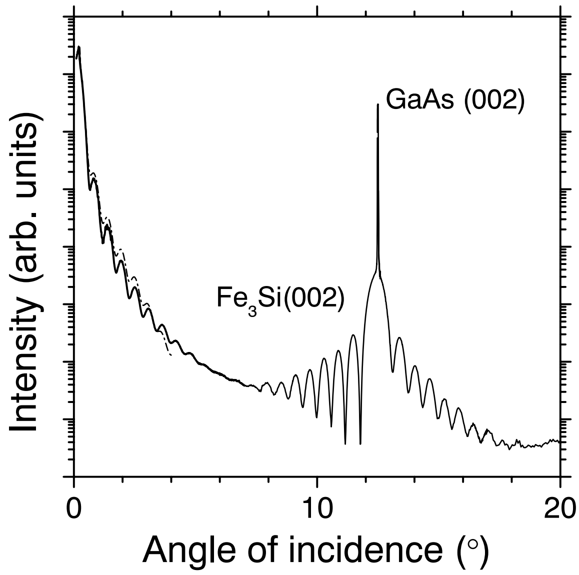

Fig. 38. Diffraction reflectivity curve along the (00) rod together with the 002 reflections of the GaAs substrate and the Fe_3Si film (solid line). The 20-monolayers-thick film is almost perfect with low surface and interface roughness as confirmed by reflectivity simulation (dash-dotted line).

growth is very slow.

Figure 37 shows a comparison of the x-ray oscillations measured during Fe_3Si growth at $T_s = 160$, 180, and 220 °C. Each open symbol was derived from a measurement of a complete rocking curve and represents its maximum. The data show a decay of the oscillations. At the lowest temperature, the maxima alternate between lower and higher values. Two length scales are found from fits by two Lorentz functions to each of these rocking curves. These findings are confirmed by a direct comparison with AFM measurements. On the reference sample, we observe pronounced terraces of about 50 nm width on the Fe_3Si surface. The step edges of the terraces are rough with many kinks so that we can attribute another length scale below 10 nm to the widths of the dendritic features of the edges and to some islands on the terraces.

Figure 38 shows an extended reflectivity curve including the 002 diffraction maximum (solid line), which is due to the epitaxial film and the substrate. The film is almost perfect with low surface and interface roughness as confirmed by reflectivity simulation (dash-dotted line). Only the epitaxial Fe_3Si film is present under UHV conditions, whereas an additional oxide layer is observed after the sample is exposed to air. The 002 reflection of GaAs is quasi-forbidden and thus very narrow. It can be distinguished from the 002 maximum of Fe_3Si, which is broad due to the small thickness of the film. The Fe_3Si 002 peak is a superlattice reflection which is sensitive to disorder. The relatively high intensity of the Fe_3Si maximum reveals a well ordered film.

(B. Jenichen, V. M. Kaganer, W. Braun, J. Herfort, R. Shayduk, C. Herrmann, K. H. Ploog)