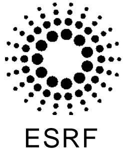

ESRF

Experiment title:

Potential Controlled Stripping of an Amorphous As-Layer on GaAs(001) in Electrolyte: An In-situ X-ray Scattering Study

Experiment number: SI-239

Beamline: ID11

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Report:

In this experiment we demonstrate that it is possible to remove **a** protective α-As layer from a GaAs(001) wafer in an electrolyte (here H_2SO_4), and to **obtain a smooth surface with** well defined stoichiometry at the solid/liquid boundary. Using an electrochemical cell of new design, we monitor the reductive etching of the As cap at sufficiently negative potential by x-ray diffraction. For the experiments, n-GaAs(001) wafers with a 1 μm thick homoepitaxial n-type film grown by MBE and protected by a smooth 50 nm thick α-As layer were used. The doping level in film and substrate was 1 $\times 10^{18}$ cm^{-3} Si. The samples were cut to a size of $\approx$ 2 x 2 mm^2 and ohmic contact was made with an In/Zn alloy annealed to the back of the sample. Using epoxy resin, the samples were mounted in a way that only the arsenic capped face was exposed to the electrolyte (0.5 M H_2SO_4). The experiments were performed under potentiostatic control in a three-electrode setup with a saturated calomel reference electrode. The As stripping is performed at an electrode potential of -1.2 V (SCE), where hydrogen is generated strongly at the GaAs surface and a thin-layer electrochemical cell, typically employed for x-ray diffraction measurements', could not be used. Our cell features a hemispherical quartz dome of 1 cm diameter and wall thickness of 100 μm which contains the electrolyte and serves as x-ray window. The x-ray diffraction measurements were performed on the "big kappa" diffractometer at ID11 at 20 keV where a transmission of 50% was obtained. In Fig. 1 the intensity of the x-rays diffracted by GaAs(220) planes which are normal to the GaAs(001) surface is plotted as a function of incident glancing angle α_i at a fixed angle α_f = 0.5" of the outgoing diffracted wave (see inset in Fig. 1). Total reflection of the incident x-ray wave leads to a rapid decay of the diffracted intensity for angles smaller than the critical angle, as shown in Fig. 1. For incident angles $\alpha_i > \alpha_c$ the q'_z is real and its value increases with α_i. Because the (220) Bragg point lies in the surface plane, and increasing value of q'_z means a movement along the (221) crystal truncation rod. This is the reason of the slow decay of the intensity on the right side of the intensity profile shown in Fig. 1.

In order to monitor the stripping of the As cap, the incidence angle was fixed at $\alpha_i - \alpha_0$ = -0.05"

on the rapidly decaying part of the curve (see arrow in Fig. 1) and the electrode potential set to -1.2 V (SCE). The intensity of the GaAs(220) surface reflection was recorded as a function of time, as shown in Fig. 2(a). A linear increase, a broad maximum and a final decrease of the intensity is observed. Figure 2(b) displays the current across the interface recorded simultaneously with the intensity/time plot from Fig. 2(a).

The reason for the increase in intensity of the (220) surface reflection, shown in Fig. 2(a), is the stripping of the arsenic layer with time. Since the penetration depth d_e of the x-rays in the a-As cap is constant, the x-ray intensity increases at the GaAs:As interface with decreasing thickness of the layer, thus leading to higher scattering amplitudes from the GaAs(001) substrate. The explanation for the observed maximum in the intensity is based on the structural properties of the interface between the GaAs(001) surface and the arsenic film. Whereas the bulk of the arsenic film is known to be amorphous [2], the first As layers at the interface may be epitaxially ordered. These layers are thought to be more stable than amorphous As, and will dissolve less easily. Since the thickness of the ordered fraction of the As cap is very small, it will not affect the penetration depth d_e to a significant extent; however, etching of the underlying GaAs is inhibited until the film is completely dissolved.

This explanation is corroborated by the current/time behavior of the etching process (Fig. 2(b)). For comparison, a curve obtained under the same electrochemical conditions in our home lab is shown. The current increases with a nearly constant slope for the rising part of the x-ray intensity curve. When the scattering intensity is almost constant, the current increases at a much higher rate, indicating that strongly passivating material is removed from the interface. After the complete removal of the arsenic cap, the electrode current saturates. While the electrical properties remain constant, the morphological properties of the interface change due to some surface roughening, i.e., the scattered intensity goes down.

1. J. Zegenhagen et al., Surf. Sci. 352-354, 346 (1996).
2. U. Resch et al., Surf. Sci. 269/270, 797 (1992).

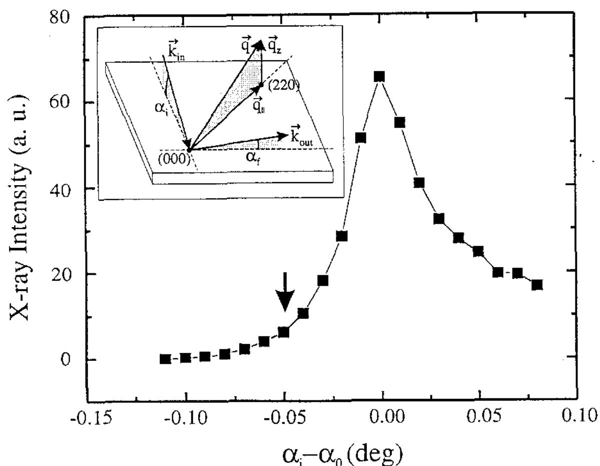

Fig. 1: Intensity of the (220) surface reflection as a function of glancing angle α_i. Experimental points of the curve represent integrated intensities. The inset shows the scattering geometry at the GaAs surface. $\vec{k}_{in}$ is the propagation vector of the x-ray beam penetrating the **α-As** layer, the layer itself is omitted for clarity.

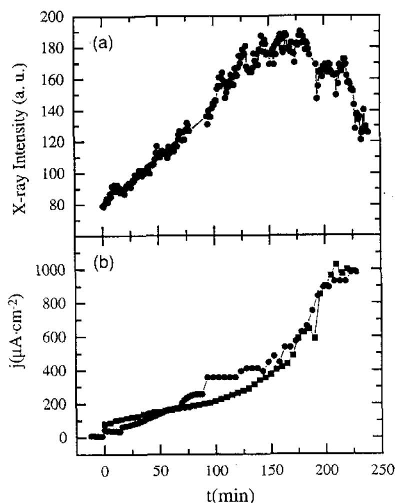

Fig. 2: (a) Intensity of the (220) surface reflection vs. time during etching at -1.2 V (SCE), with $\alpha_i - \alpha_0$ = -0.05' (see arrow in Fig. l), and α_f = 0.5". (b) Current/time behavior of the GaAs:As electrode during etching. measured simultaneously and independently in the laboratory (square).