Applied Surface Science 367 (2016) 391–400

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

A detailed look beneath the surface: Evidence of a surface reconstruction beneath a capping layer

D. Krull[a,*], M.F. Tesch[b,1], F. Schönbohm[a], T. Lühr[a], C. Keutner[a], U. Berges[a], H.-Ch. Mertins[b], C. Westphal[a]

[a] Lehrstuhl für Experimentelle Physik I and DELTA, Dortmund University of Technology, Otto-Hahn-Str. 4, 44221 Dortmund, Germany
[b] University of Applied Sciences Münster, Stegerwaldstr. 39, 48565 Steinfurt, Germany

ARTICLE INFO

Article history:
Received 13 October 2015
Received in revised form 18 January 2016
Accepted 18 January 2016
Available online 22 January 2016

Keywords:
Internal interface
Surface beneath a capping layer
XPD
T-MOKE
Semiconductor
Ferromagnet

ABSTRACT

Many physical effects are strongly depending on the composition of the interfaces between separating layers. Hence, the knowledge of the interfacial characteristics such as structure, chemical bonds, or magnetic properties of the corresponding materials is essential for an understanding and optimization of these effects. This study reports on a combined magnetic and structural analysis using X-ray photoelectron diffraction (XPD) and transverse magneto-optical Kerr effect (T-MOKE). The information depth of these methods is demonstrated by investigating the uppermost GaAs(001) layer beneath a Fe-film and the interfacial regimes of Fe/GaAs(001) beneath an MgO capping layer.

Iron was prepared on a clean GaAs(001) surface and a GaAs(001)-(4 × 2)-reconstructed surface. Beneath the Fe-film, the (4 × 2)-reconstruction is not lifted, which is clearly shown by the diffraction pattern of the GaAs(4 × 2)-Fe surface. It is shown that Fe inter-diffusion, resulting in an amorphous interface, is almost prevented by the Ga-rich reconstruction. The magneto-optical measurements with T-MOKE clearly demonstrated the Fe-interlayer in a ferromagnetic state. We find no evidence for magnetic properties neither within the signal of the GaAs-substrate nor the MgO-film.

1. Introduction

Surfaces and interfaces are of great importance, e.g. in the field of electric semiconductor components, due to their different characteristics compared to the volume substrate. A perfect crystalline interface is essential to achieve epitaxial films on a semiconductor and hence to increase the efficiency of the effects arising at interfaces [1–3].

For example, the Tunnel Magneto Resistance (TMR) [4] and Giant-Magneto Resistance (GMR) [5,6] are in the focus of present research [7–14]. These effects are strongly depending on the interface structure [15,16] and an epitaxial film-growth on the substrate surface is essential. Further materials are under suspicion of structure phase transitions if prepared on a substrate. For multilayer film growth, some elements are known showing a phase transition as a function of film thickness resulting in strong structural changes at the interfaces [17–21]. An imaging or a two dimensional diffraction image on an individual internal layer was not possible in the past. Further, it is experimentally extremely difficult to measure the intermixing at the interface.

In this study we demonstrate that X-ray photoelectron diffraction (XPD) and transverse magneto-optical Kerr effect (T-MOKE) allow a detailed look into the structure of an internal film at a surface. As an example, we present a close-up view of the GaAs(001) surface beneath an Fe-film with capping MgO-layers. Additionally, a detailed view into the interface structures is presented. The three-layer system GaAs(001)-(4 × 2)-Fe—MgO was chosen, because structure information to the layers of this system is already available [22]. Therefore, this system is a good candidate for studying internal interfaces and for investigating the substrate surface structure beneath a film.

In literature the GaAs surface-reconstruction was suspected being involved in the film growth structure of Fe on GaAs [23,24,22,25–27]. Due to the limited access to an internal interface and especially to the substrate surface the proposed inter-diffusion between GaAs and Fe was not resolved in the past. For the GaAs surface several reconstructions are known. They can be characterized by their gallium and arsenic concentration. In this study, a clean and a reconstructed GaAs-surface were prepared in order to examine the influence of the substrate on the epitaxial Fe-growth.

* Corresponding author.
E-mail address: dominique.krull@tu-dortmund.de (D. Krull).
[1] Now at: Helmholtz Zentrum Berlin für Materialien und Energie Freie Universität Berlin, Albert-Einstein Straße 15, 12489 Berlin, Germany.

http://dx.doi.org/10.1016/j.apsusc.2016.01.162
0169-4332/

Arsenic is under suspicion for interacting stronger to Fe than Gallium [28,29], and the (4×2)-reconstruction was chosen here as a representative of the most Ga-rich reconstruction. A mixed bcc/fcc structure of an iron film due to a possible phase transition [30] is discussed in literature. We expect that transition being clearly apparent in a photoelectron diffraction pattern and thus this system is investigated in this study.

In order to analyze the interface structure X-ray photoelectron diffraction (XPD) was applied. XPD combines core-level photoelectron spectroscopy (XPS) with intensity variations as a function of polar- and azimuth-angle [31]. The out-going photoelectron wave of the emitting atom is scattered at the neighboring atoms and results in intensity variations of the spectra. Additional XPS-spectra allow a detailed analysis for each element and its chemical environment [32]. The spectral components contained in the photoelectron signals provide information of the local bonding, e.g. whether the emitter is located as a part of a dimer at the surface or located within a bonding beneath the surface. We compare experimental and simulated XPD pattern for various models.

The chemical composition and structure of the Fe/GaAs(001) interface was analyzed and yielded an epitaxial growth for a reconstructed substrate. Furthermore, the surface reconstruction prevented an inter-diffusion between the GaAs-substrate and the Fe-adsorbate. The magnetic properties of this system were verified by magneto-optical spectroscopy. It provides an important tool for the investigation and characterization of magnetic materials [33,34]. The magneto-optical measurements using the transversal magneto-optical Kerr Effect (T-MOKE) revealed a ferromagnetic Fe-interlayer only.

2. Experiment

2.1. Setup

The preparation and XPD measurements were performed in an ultra-high vacuum chamber (UHV) with a base pressure of $p = 5 \times 10^{-11}$ mbar at the PGM beamline 11 at the storage ring DELTA in Dortmund, Germany. The chamber is equipped with a hemispherical electron analyzer, an electron beam evaporator, a sputter gun, and a LEED (*low energy electron diffraction*) system. The sample can be positioned by a manipulator in all three spatial directions as well as in azimuth and polar angle rotations. The overall energy resolution is around 90 meV and the angular resolution is limited by a geometric aperture with a diameter of 5 mm. Within the XPD measurements both polar and azimuth angles were changed evenly, with polar and azimuth angle range of $0^\circ \leq \theta \leq 60^\circ$ and $0^\circ \leq \phi \leq 360^\circ$, respectively. The step-increment of $\Delta\theta = 2^\circ$ and $\Delta\phi = 1.8^\circ$ was kept constant for both angles.

The T-MOKE measurements were carried out in an UHV chamber designed for T-MOKE experiments at the TGM beamline 12 at DELTA. The T-MOKE experimental set-up contained a magnetization device based on NdFeB permanent magnets that provided homogeneous magnetic fields with a flux density up to 500 mT. A detailed report on the set-up can be found in [35,36].

Here, two substrates were prepared for our investigations, a non-reconstructed GaAs(001) surface and the Ga-rich (4×2)-reconstruction. GaAs(4×2) was discussed as a suitable substrate for crystalline Fe-films [25,26]. The procedure for a successful (4×2) surface reconstruction has been discussed in detail previously [22]. In order to degas the samples flash annealing at 600 °C was applied for about 1 min, following keeping the temperature constant at 540 °C for approximately 60 min. The preparation of the clean GaAs(001) and GaAs(001)-(4×2) surfaces were carried out by several sputter and annealing cycles. The preparation parameters are summarized in Table 1.

Table 1
Preparation parameters of the GaAs(001) surfaces.

	Sputter	Anneal
GaAs(4×2)	$E_{Ar^+} = 1000$ eV, 20 min	550 °C, 45 min
GaAs clean	$E_{Ar^+} = 1000$ eV, 30 min	300 °C, 45 min

The clean sample was verified by XPS survey spectra showing the GaAs core level signals only without any indication of a surface structure with long-range order in the LEED pattern. Accordingly, the reconstructed surface was verified by XPS survey spectra and a (4×2) LEED pattern.

In order to prepare the iron layer an iron rod was evaporated using an electron beam evaporator. During the evaporation of 15 min the GaAs sample was kept at room temperature and was evenly rotated around its surface normal to support a smooth growth. The MgO film was obtained from direct evaporation of an MgO crystal for 15 min. Again, the substrate was kept at room temperature and rotated around its azimuth during the evaporation process. The layer thicknesses were calculated from the XPS and XPD data [37,38] being $d_{Fe} = 18$Å and $d_{MgO} = 4.6$Å for the prepared Fe-film and MgO-film, respectively.

2.2. X-ray photoelectron spectroscopy and diffraction (XPS/XPD)

X-ray photoelectron spectroscopy (XPS) and diffraction (XPD) are based on the photoelectric effect [39–41]. Due to elastic scattering and diffraction the intensity of the photoemission signal varies as a function of emission direction [42]. Hence, by varying the detection angles at constant excitation energy the symmetric intensity modulations can be attributed to the symmetries of the sample structure. Detailed review articles are references [31,43,44], for example.

In all spectra the background was removed by a Shirley-function [45]. High-resolution spectra recorded at $\theta = 0^\circ$ and $\theta = 60^\circ$ allowed identifying spectral components related to atoms in bulk and interface regions, respectively. The high-resolution spectra were analyzed using a Gaussian function $G(E)$, where A_i denotes the complex amplitude, σ_i the full-width half-maximum (FHWM), and h_{LS}, f_{LS} include the height ratio and the energy shift caused by the spin-orbit-coupling (SOC).

$$G(E) = \sum_i \left[A_i \cdot \exp\left(\frac{1}{2} \left[\frac{E - E_i}{\sigma_i} \right]^2 \right) + h_{LS} A_i \cdot \exp\left(\frac{1}{2} \left[\frac{E - (E_i - f_{LS})}{\sigma_i} \right]^2 \right) \right] \quad (1)$$

One XPD pattern consists of approximately 6000 individual recorded spectra. The analysis of the experimental diffraction pattern has been carried out by comparing these to simulated pattern obtained for various atom structure models. The simulations used the MSPHD program (*full multiple scattering code for low energy photoelectron diffraction*), which is an excellent tool for structure investigations [46–48]. The angular momentum cut-off was set to $l_{max} = 6$ and the atom cluster size was about 100 atoms. A quantitative comparison is provided by using an *R*-factor, defined as

$$R = \frac{\sum_i (\chi_{\exp_i} - \chi_{sim_i})^2}{\sum_j (\chi^2_{\exp_j} + \chi^2_{sim_j})}. \quad (2)$$

In this definition [49] an *R*-factor of 2 corresponds to an anti-correlated intensity distribution, whereas a perfect agreement is indicated by an *R*-factor of 0. Thereby, the anisotropy function is given by $\chi(\theta, \phi) = [I(\theta, \phi) - I_0(\theta)]/I_0(\theta)$, where $I(\theta, \phi)$ and $I_0(\theta)$ denote