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3D Auger quantitative depth profiling of individual nanoscaled III–V heterostructures

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ABSTRACT

The nanoscale chemical characterization of III–V heterostructures is performed using Auger depth profiling below decananometric spatial resolution. This technique is successfully applied to quantify the elemental composition of planar and patterned III–V heterostructures containing InGaAs quantum wells. Reliable indium quantification is achieved on planar structures for thicknesses down to 9 nm. Quantitative 3D compositional depth profiles are obtained on patterned structures, for trench widths down to 200 nm. The elemental distributions obtained in averaged and pointed mode are compared. For this last case, we show that Zalar rotation during sputtering is crucial for a reliable indium quantification. Results are confirmed by comparisons with secondary ion mass spectrometry, photoluminescence spectroscopy, transmission electron microscopy and electron dispersive X-ray spectroscopy. The Auger intrinsic spatial resolution is quantitatively measured using an original methodology based on the comparison with high angle annular dark field scanning transmission electron microscopy measurements at the nanometric scale.

1. Introduction

III-V semiconductors are foreseen materials for the downscaling of metal oxide semiconductor field effect transistors (MOSFETs) below the 16 nm technological node [1–4]. Their integration in new architectures such as quantum well field effect transistors (QWFETs) is also considered [5]. Indium based semiconductors, such as InGaAs, are promising high mobility channels to improve the speed of operation. In QWFETs, the carrier mobility is enhanced by taking advantage of quantum electron confinement in a multilayered channel. The quality of the interface between these complex channels and the gate oxide is of crucial importance to preserve the electrical properties of the device. A thin layer, InP for instance, can be inserted to distance the channel from interfacial defects located at the high-K/III-V interface. Another important issue is to grow high quality III–V materials on Si wafers. For this purpose, a partially relaxed buffer layer, such as GaAs, is used to reduce the defect density near the channel. To further improve the quality of the III–V materials near the top surface, the deposition is made on patterned SiO_2 trench arrays using the aspect ratio trapping method [6,7]. With this solution, dislocations that are generated at the interface with the Si substrate are trapped in the SiO_2 walls.

Abbreviations: MOSFETs, metal oxide semiconductor field effect transistors; QWFETs, quantum well field effect transistors; QW, quantum well; RSFs, relative sensitivity factors; AES, Auger electron spectroscopy; SIMS, secondary ion mass spectrometry; PL, photoluminescence; STEM-EDX, scanning transmission electron microscopy combined with electron dispersive X-ray spectroscopy; HAADF-STEM, high angle annular dark field scanning transmission electron microscopy; MOCVD, metal organic chemical vapor deposition; RBS, Rutherford backscattering spectrometry; FIB, focused ion beam.

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MOSFET downscaling needs the development of complex non-planar III–V architectures. A precise analysis of their chemical composition is mandatory for process control and optimization [8,9]. In particular, all the interfaces are critical for the final device electrical and optical performances. We need to evidence possible interfacial mixing and/or interdiffusion phenomena. Another important issue is to control the indium content in the InGaAs QW for a precise bandgap tuning. Measuring elemental depth distributions is thus of crucial importance. However, chemical depth profiling of such complex 3D architectures with high aspect ratio and low lateral dimensions is very challenging. Advanced characterization methods that combine high lateral and depth resolutions are needed, thus allowing direct analysis of individual III–V heterostructures.

Quantitative Auger compositional depth profiling is of real interest because it has sufficient lateral and depth resolutions to investigate such complex 3D architectures [10–13]. New generation Auger nanoprobes can provide enough sensitivity and long term stability to investigate the elemental composition of nanostructured materials [14]. In this paper, we thus highlight the possibility of Auger electron spectroscopy (AES) through depth profiling measurements to obtain quantitative in-depth chemical information of planar and non-planar III–V heterostructures designed for hybrid integration on Si. This approach was already proven to be efficient to get the indium distribution in planar InGaAs/GaAs quantum wells (QWs) [15]. But no indium quantification was provided. Here, we obtain reliable indium quantification through the use of adequate relative sensitivity factors (RSFs) evaluated on reference samples. We also make one more step to measure the indium content in InGaAs QWs for patterned III–V heterostructures, going from 2D towards 3D chemical depth profiling. The Auger lateral resolution allows individual trenches to be analyzed with clear identification of the InGaAs QWs inserted between AlAs barriers. Consistency is obtained with complementary secondary ion mass spectrometry (SIMS) and photoluminescence (PL) measurements, strengthening the Auger results. We further investigate the performances of Auger depth profiling to provide reliable compositional information, by discussing the impact of depth resolution, sample orientation and analytical conditions on the quality of the results. Finally, scanning transmission electron microscopy combined with electron dispersive X-ray spectroscopy (STEM-EDX) is carried out to confirm both Auger and SIMS results and to evaluate the performance of Auger depth profiling in terms of spatial resolution.

2. Experimental details

Planar and non-planar heterostructures composed of GaAs/AlAs/InGaAs/AlAs/GaAs stacks are grown by metal organic chemical vapor deposition (MOCVD) using trimethylgallium (TMGa), −aluminium (TMAl), −indium (TMIn) as group-III precursors and tertiarybutylarsine (TBAs) as the group-V precursor. The InGaAs quantum well (QW) of several nanometers in thickness (6 to 19 nm) is encapsulated between two barrier layers of AlAs with thicknesses varying from 7 to 12 nm. The drawings of planar and non-planar heterostructures are shown in Fig. 1, together with the corresponding images measured by scanning transmission electron microscopy (STEM). The non-planar III–V heterostructures are made of selective area growth III–V trenches of widths between 150 and 200 nm, deposited on Si(100) substrates between 60 nm-wide SiO_2 barriers as shown in Fig. 1(b and d). A thick GaAs buffer layer is used to reduce the number of dislocations close to the Si substrate. The SiO_2 walls block the propagation of dislocations gliding on (111) planes.

The Auger experiments are carried out using a PHI 700Xi Auger nanoprobe equipped with a cylindrical mirror analyzer mounted coaxially with the electron column with an energy resolution of 0.5% and high sensitivity (ultimate detection limit ~ 0.1%). Measurements are performed with a primary electron beam at 10 keV (1 nA beam current, probe size ~ 30 nm) at normal incidence. The Auger electrons are collected at an angle of 42° with respect to the primary electron beam. The sampling depth is of 3 nm taking the inelastic mean free path of indium Auger electrons (In(MNN) at 404 eV) in GaAs, estimated to be 1.5 nm [16]. Sputtering is performed with optimized conditions to reduce sputter induced artefacts and improve the depth resolution, i.e. at grazing incidence (28°) with Ar^+ ions accelerated at 500 eV (500 nA) with a raster size of $1 \times 1\ mm^2$. In-depth composition profiles were obtained by combining ion sputtering and Auger electron spectroscopy, acquiring the following Auger lines: In(MNN) at 404 eV, Ga(LMM) at 1070 eV, As(LMM) at 1228 eV, Al(LMM) at 1396 eV, O(KLL) at 510 eV, C(KLL) at 272 eV and Si(KLL) at 1619 eV.

Quantification is performed using the peak-to-peak height obtained after numerical derivation of these Auger lines and using elemental relative sensitivity factors (RSF). In order to estimate these factors for the III–V compounds studied here, Auger analyses are performed on reference bulk heterostructures: i) AlGaAs (240 nm)/GaAs (230 nm)/Si and ii) InGaAs (100 nm)/GaAs (400 nm)/Ge (1.6 μm)/Si, henceforth labelled AGA and IGA respectively. The elemental composition is obtained using Rutherford backscattering spectrometry (RBS). RBS is used here as a reference technique for quantification of the chemical composition. The main advantage of this technique is that quantification is done without the use of any calibration standards, thus yielding to absolute concentration values unlike AES. RBS experiments are performed with an incident He^+ ion beam at 1.6 and 2.2 MeV, an incident current of 3 nA through the target sample with an analysis area of few mm^2, at a backscattering angle of 160°. The atomic concentrations measured for the AGA and IGA reference bulk samples are shown in Table 1. Uncertainties are estimated by combining both statistical and instrumental errors.

Auger pointed analyses are performed on these reference bulk samples with an electron beam at 10 keV (1 nA) in order to estimate the effective RSF values of the III–V elements. These effective RSFs are used to improve quantification by reducing the impact of possible matrix effects contrary to the use of tabulated RSFs. Hydrocarbon surface contamination as well as native oxides are first removed by argon sputtering at 2 keV (1 μA) during 5 min. Then, the effective RSF of arsenic is stated to be the reference one and equal to 0.11, as obtained from the tabulated values provided by PHI in the MultiPak software version 9.1. Finally, the other RSF values are extracted from the Auger intensity ratios, In/As and Ga/As for IGA and Al/As for AGA, and the RBS atomic concentrations by applying the following formula:

$$\mathrm{RSF_X} = \frac{[\mathrm{As}]}{[\mathrm{X}]}\frac{\mathrm{I_{p-pX}}}{\mathrm{I_{p-pAs}}}\mathrm{RSF_{As}} \quad (1)$$

where [X] and I_{p-pX} are respectively the atomic concentration and the peak-to-peak intensity of element X (Ga, Al or In). The evaluated RSF values are given in Table 2 with an uncertainty of ± 6%.

Secondary ion mass spectrometry (SIMS) measurements are performed under O_2^+ sputtering at a low energy of 500 eV and an incident angle of 42° with respect to the sample normal, with an SC-Ultra instrument from CAMECA. The raster size is of 200 μm × 200 μm with a small field aperture of 400 μm in diameter and analyzed area of 33 μm in diameter. Quantification of the SIMS results is done using the same empirical method as for AES, based on measurements on matrix-matched samples. For these references, RBS absolute concentrations are again used in order to estimate the relative sensitivity factors of Al, In, Ga and As. More