

In-situ probing of near and below sputter-threshold ion-induced nanopatterning on GaSb(1 0 0)

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ABSTRACT

This work presents *in-situ* near and below sputter-threshold studies for GaSb(1 0 0) at energies 50, 100 and 200 eV and current densities near $50 \mu\text{Acm}^{-2}$. Variation of incident particle energy probes the energy deposition distribution and its relation to surface composition. *In-situ* analysis is conducted over irradiation modification using Ar singly-charged ions at normal incidence of the surface using complementary techniques including: X-ray photoelectron spectroscopy (XPS) and ion-scattering spectroscopy (LEISS). The former probes 1–3 nm and the latter technique probes the first 1–2 ML or 0.3–0.6 nm. Ex-situ analysis includes HR-SEM to correlated surface morphology with surface composition studied *in-situ* during irradiation. Results indicate ordering of nanodot formation at fluence threshold of about 10^{17}cm^{-2} . Both XPS and LEISS identify Ga_2O_3 islands formation due to GaSb chemical affinity for oxygen followed by an initial enhancement of Ga/Sb = 1.20 ratio and then a sharp drop in Ga relative concentration with LEISS reaching a Sb-dominated terminating 1–2 nm region corresponding to the implantation depth between 50 and 200 eV. XPS shows a slight enrichment of Ga in sub-surface layers that levels to a 1:1 stoichiometry of the crystalline GaSb(1 0 0) surface.

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1. Introduction

Bottom-up, parallel processing techniques are beginning to rival other nanolithography approaches to nanopatterning [1]. However, much work remains in understanding scaling from short-range to long-range ordering as device features continue to decrease beyond sub-20 nm size. Device feature size introduces limits on ion-beam sputtering (IBS) nanopatterning conditions such as incident particle energy given the penetration range 1–4 nm for energies between 0.05 and 2 keV Ar^+ irradiation. Therefore as device functionality require dimensions approaching 1–5 nm, understanding nanopatterning at these scales become more important. Applications of quantum dot confinement also introduce scaling limits and motivate systematic study of dot characteristic size against IBS parameters (e.g. ion energy, angle, ion-target interaction, etc.). Correlating *in-situ* surface composition and sputter erosion evolution to nanostructure synthesis could elucidate on self-organization mechanisms such as the balance between physical sputtering and surface diffusion.

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In this work we present a systematic study of near and below sputter-threshold energies between 50 and 200 eV Ar^+ irradiation at normal incidence of GaSb to examine *in-situ* the role of surface concentration on nanopatterning. In particular, we conduct *in-situ* surface characterization of ion-irradiated surfaces during early stage growth (e.g. 10^{15} – 10^{17}cm^{-2}) of ion-induced nanostructures.

XPS and LEISS studies of GaSb irradiated by energetic ions are sparse. W. Yu et al. studied GaSb, InSb and CdSe surfaces with LEISS and XPS; however these were irradiated with Ar^+ at energies above 3 keV [2]. Another study of GaSb by Möller et al. [3] assessed the role of surface oxides, which for this particular paper it is relevant since the incident particle energy is below 0.2 keV and the implantation is ultra-shallow (<1.0 nm) where oxide coverage needs to be assessed when investigating early stage (low fluence) growth of ion-induced nanostructures on GaSb. The work by LeRoy et al. indicated two possible mechanisms for growth of ion-induced nanostructured pillar features from flat surfaces of Ga–Sb. In particular for GaSb, the segregation of Ga during sputtering as a shield in Ga–Sb erosion. However, Ga and Sb erode near identically due to similar heats of sublimation. In fact for an energy of about 1 keV Ar^+ on GaSb, the corresponding sputter yields are about: $Y_{\text{Ga}} = 1.28$ and $Y_{\text{Sb}} = 1.25$ according to SRIM. Our work elucidates on the important early stages of growth with the strong segregation of Ga to the surface primarily due to the reduction of

surface oxide followed by a sharp decrease in Ga at the ion-induced amorphous layer with a steady-state Sb-dominant region.

2. Experimental setup

All *in-situ* modification and characterization was performed at the Particle and Radiation Interaction with Hard and Soft Matter (PRIHSM) facility at Purdue University. PRIHSM is an ultra-high vacuum (UHV) surface science facility with *in-situ* ion-beam modification and characterization capabilities. Modification is carried out with a gridded broad-beam non-reactive ion source with current densities of up to $40 \mu\text{A}/\text{cm}^2$ and energies from 10 to 200 eV. *In-situ* characterization techniques are carried out using a VG Scienta R3000 charged particle analyzer allowing ultraviolet photoelectron spectroscopy (UPS), angle resolved photoelectron spectroscopy (ARPES), X-ray photoelectron spectroscopy (XPS), and low energy ion-scattering spectroscopy (LEISS). The sample temperature is controllable via a combination of electron-beam heating and liquid nitrogen cooling to achieve sample temperature control up to a maximum of 1150°C . Surface morphology can be investigated ex-situ using scanning electron microscopy (SEM) and atomic force microscopy (AFM).

Undoped (1 0 0) GaSb samples were cleaned in chemical baths of methanol, distilled water, methanol followed by nitrogen gas drying. Due to high chemical reactivity of GaSb surfaces a thin oxide layer ($\sim 4\text{--}5 \text{ nm}$) is found with *in-situ* diagnosis using XPS and LEISS. Surface modification was driven by a normal incidence broad-beam Ar^+ source from 50 to 200 eV at current densities from 10 to $40 \mu\text{A}/\text{cm}^2$.

Total fluencies range from 1×10^{15} to $1 \times 10^{18} \text{ cm}^{-2}$. Surface composition was measured *in-situ* pre- and post-irradiation with XPS. For select samples LEISS and XPS were performed at intermediate fluencies throughout the irradiation.

XPS was performed at normal emission of photoelectrons with a source-analyzer angle of 54.7° . A non-monochromatic $\text{Mg K}\alpha$ (1245.3 eV) X-ray source was used with an anode voltage of 13.0 kV and an emission current of 15.0 mA . LEISS employing a 1500 eV He^+ beam was performed at a backscattering angle of 145° . The total probing beam current was 150 nA with a maximum beam flux of $1.4 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$. For both XPS and LEISS an analyzer pass energy of 100 eV was used with a 3.0 mm wide straight slit. All samples were cooled to ensure a temperature from 0 to 25°C throughout the irradiation in order to guard against varying thermal diffusion effects as a function of current density and beam energy. All *in-situ* work was carried out at base pressures of less than $5 \times 10^{-8} \text{ Torr}$.

Surface morphology was probed post-irradiation using an ex-situ H4700 Field-Emission SEM. Quantification of XPS spectra into relative surface concentrations was performed using CasaXPS and Igor Pro v. 6. For LEISS data IGOR Pro software, the background was subtracted from each peak of Ga and Sb and an integral was taken of each resultant, background-subtracted peak. The area from these integrals was used to compare the relative concentration of Ga to Sb as a function of fluence using the formula:

$$y = \frac{A_{\text{Ga}}/\sigma_{\text{Ga}}}{A_{\text{Ga}}/\sigma_{\text{Ga}} + A_{\text{Sb}}/\sigma_{\text{Sb}}} \quad (1)$$

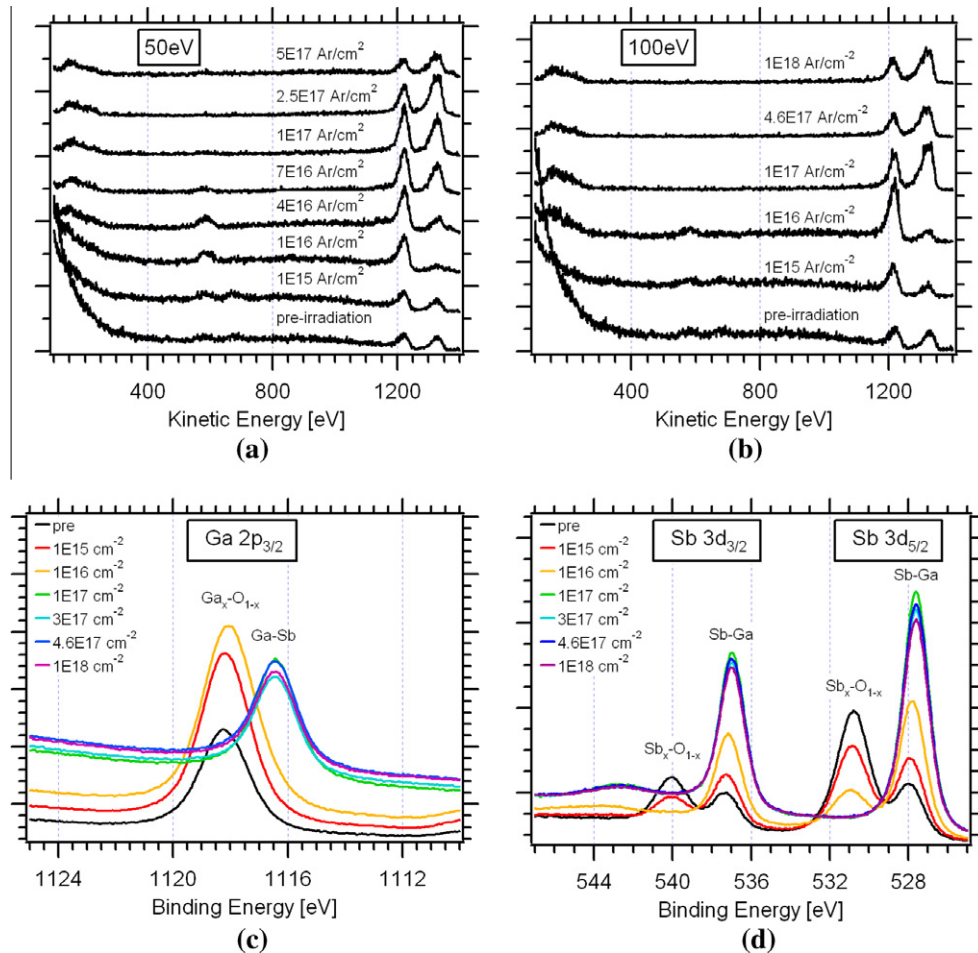


Fig. 1. (a) LEISS spectra shown for 50 eV as a function of Ar^+ fluence and (b) 100 eV compared to (c) XPS spectra of Ga $2p_{3/2}$ and (d) Sb $3d_{3/2}$ and Sb $3d_{5/2}$ spectra for 100 eV Ar^+ irradiation as a function of fluence.

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