SAND2022-9475C

Observation of epitaxial growth of quantum dots on GaSb substrate in TEM samples induced by focused ion beam preparation

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Abstract

We report an unusual artifact observed in transmission electron microscopy (TEM) samples on the surface of GaSb substrate induced by Ga^+ focused ion beam (FIB) preparation. The Ga^+ FIB-ed TEM samples with a quantum structure made of Al/AlSb/GaSb/InAs/$Al_{0.33}Ga_{0.67}Sb$ multilayers on a GaSb substrate can be easily modified by varying FIB conditions such as Ga^+ beam energy. Dependent on the conditions, the Al islands initially on top of the multilayer stack are gradually replaced by Ga, leading to epitaxial formation of $Al_xGa_{1-x}Sb$ (AlGaSb) quantum dots (QDs) whose shapes are conformed to the initial Al islands. A similar effect is also observed even when the top Al islands are capped by an amorphous As layer. The artifacts can be eliminated either by extensive thinning at 5 kV followed by 2kV polishing, or using a cryogenic condition. The mechanism for forming the artifacts has been investigated, and it involves the replacement of Al atoms in the islands by Ga atoms, formation of Al_xGa liquids on the AlSb surface, and the subsequent nucleation of $Al_xGa_{1-x}Sb$ at the liquid-Al_xGa/AlSb interface. The local heating by the ion-beam is likely an important factor.

Materials and Experiments

The FIB induced artifacts in this study were observed in cross-section TEM samples of GaSb (100) substrates with a quantum structure shown schematically in Fig.1a. The multilayer quantum structure was grown by molecular beam epitaxy (MBE). The top Al layer on the AlSb is not flat and forms individual Al islands, with sizes largely dependent on thickness of the Al layer laid down during the MBE growth. Samples with Al layer thicknesses of 10 nm and 30 nm were chosen in this study. Additionally, samples having the 10 nm Al layer capped with a 20 nm amorphous As film were studied.

The cross-section TEM samples were made using a Thermo Fisher Scientific (FEI) Helios 600 or Helios G3. The Ga^+ FIB TEM preparation involved three basic steps: (1) a rough slab cut at energy of 30 keV and beam current of 6-10nA with +/- 1° tilts; (2) a cleaning step at energy of 5 keV and beam current of 15-40 pA with +/-5° tilts; and (3) final polishing step at energy of 2 keV and beam current of 5-10 pA with +/-5° tilts. A FEI Helios G-5 plasma FIB (PFIB) with Xe^+ ions was also used to understand the effect of different ion beam sources. The FEI Helios G-5 is also capable of cooling the specimen for cryo-FIB experiments at a temperature of -100°C.

Results

Figure 1 – quantum structure as deposited without the FIB induced artifacts

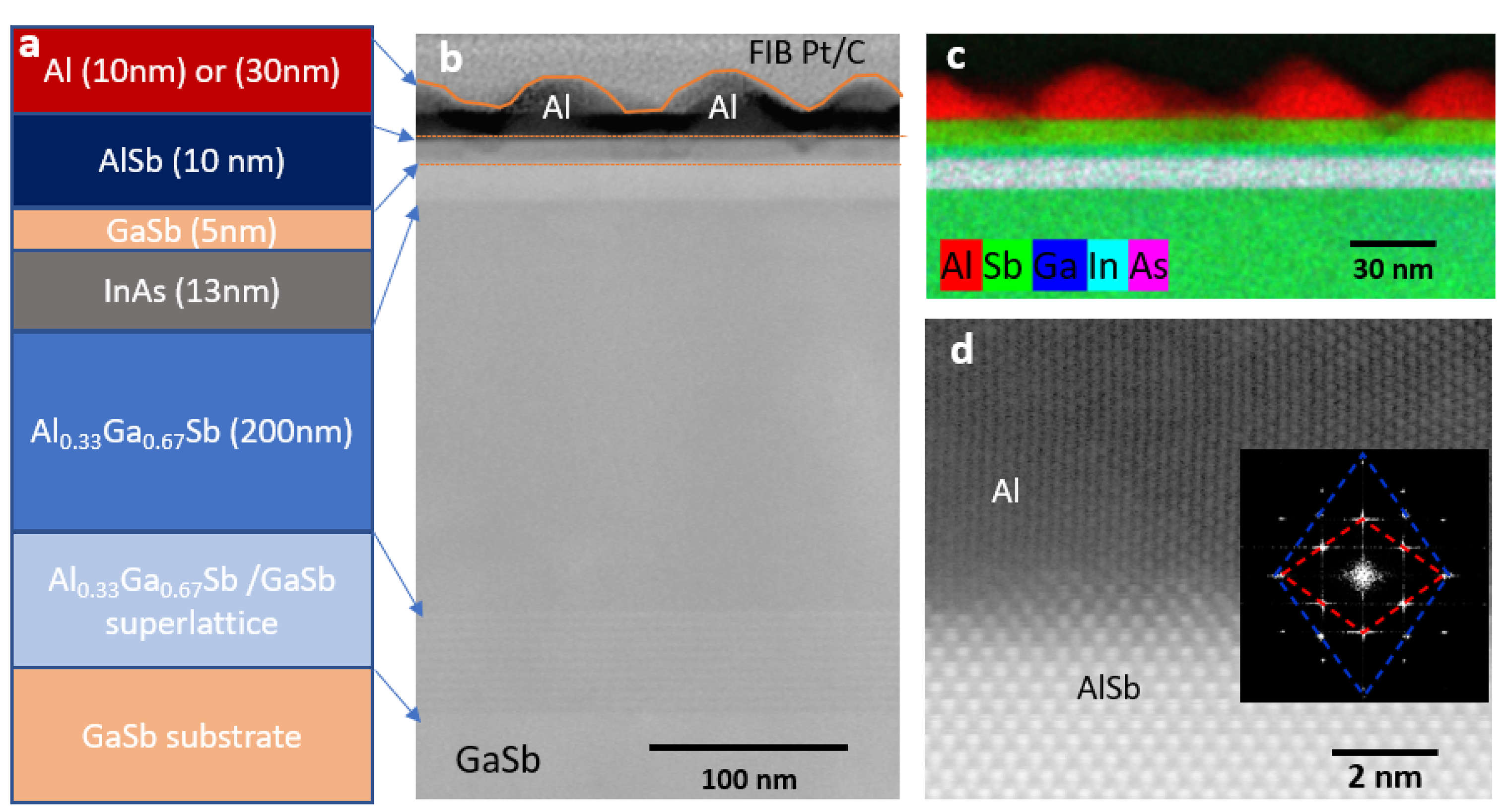


Fig.1. (a) schematic of the quantum structure in cross-section. (b) HAADF STEM image of the quantum structure. (c) EDS maps from top surface showing the Al islands without the FIB induced artifacts. (d) high-resolution HAADF STEM image of Al/AlSb interface along with its FFT pattern in inset. The dashed diamonds in the inset in (d) indicate respective patterns of Al (blue) and AlSb (red) in [110] projections.

Figure 2 – same quantum structure with the FIB artifacts

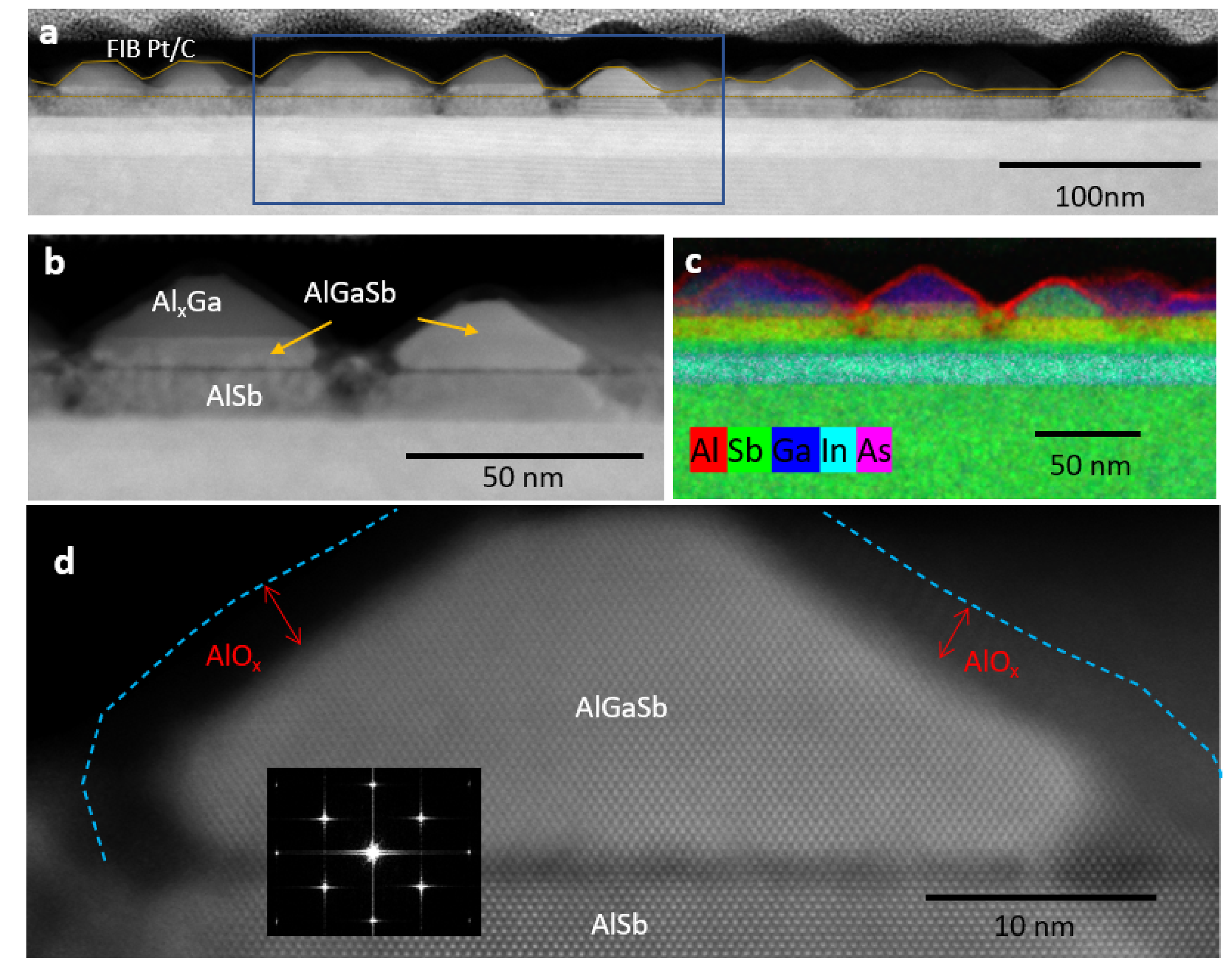


Fig.2. (a, b) HAADF STEM images and (c) EDS maps from the rectangular region marked in (a), showing top layer Al islands have been converted to Al_xGa, Al_xGa/AlGaSb bilayer, and AlGaSb islands during the FIB cutting. (d) high-resolution HAADF STEM image along with the FFT pattern from the region in inset, showing the FIB induced AlGaSb QD forming epitaxy with the AlSb layer underneath.

Figure 3 – growth mechanism of AlGaSb

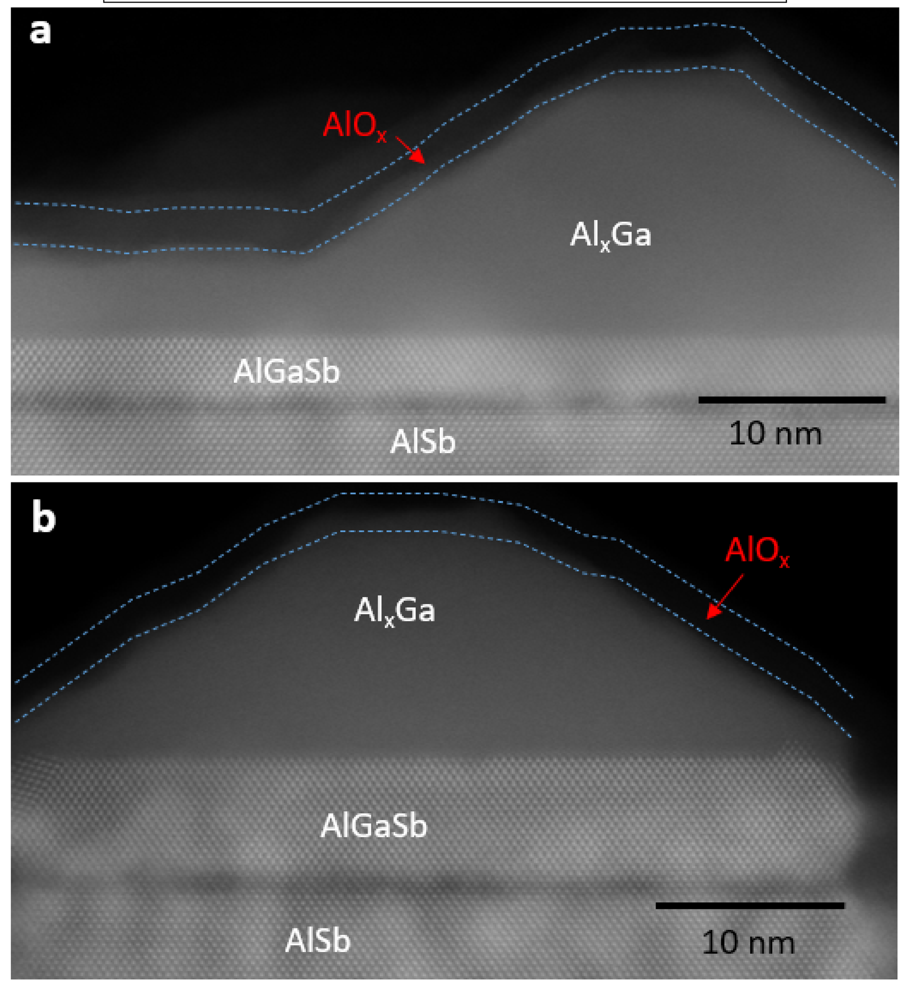


Fig.3. (a, b) HAADF STEM images of the AlGaSb layer sandwiched between Al_xGa and AlSb, showing growing AlGaSb layer at different stages. The islands are covered by a 3nm AlO_x layer. The AlGaSb surface is atomically flat, indicating a layer-by-layer growth mechanism.

Figure 4 – same FIB artifacts observed even when the Al islands were capped by an As layer

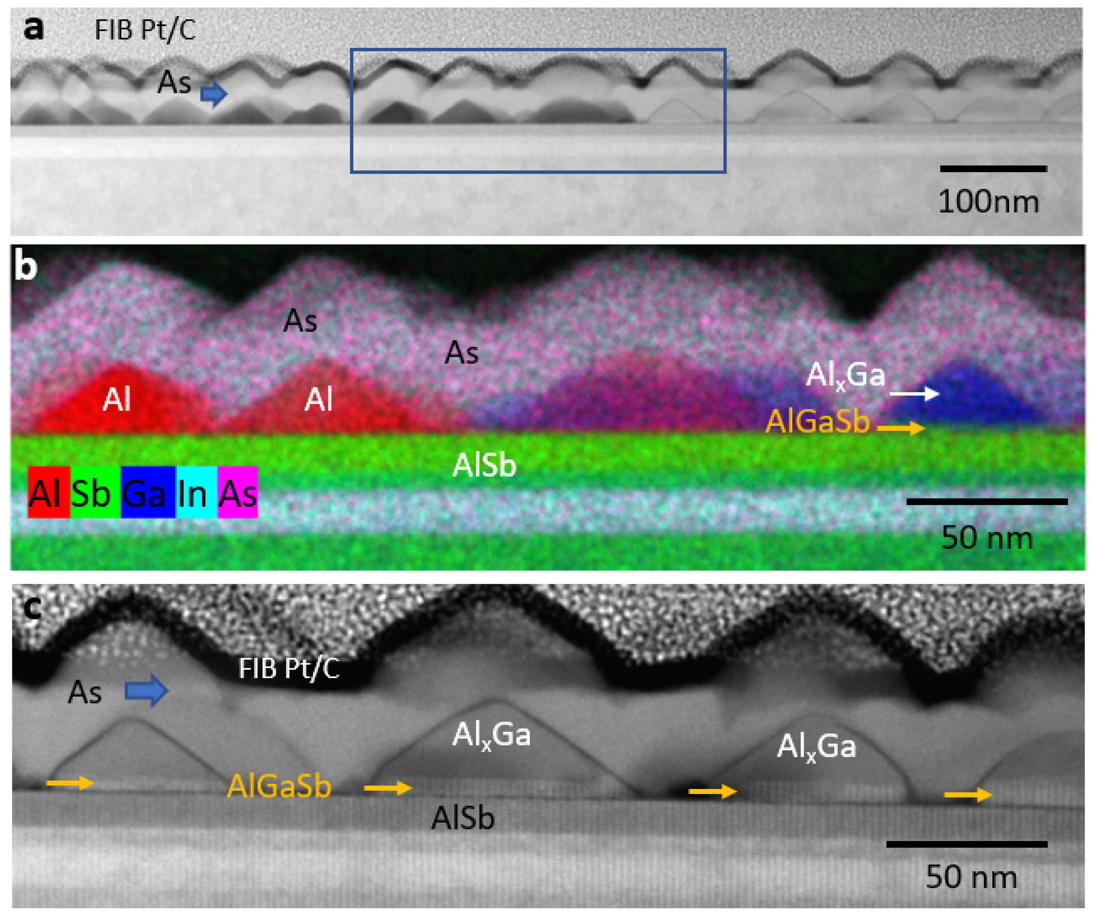
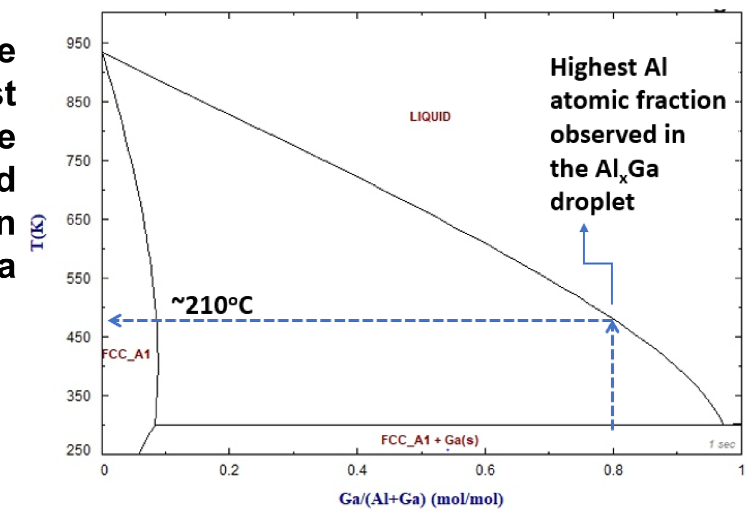


Fig.4. (a) HAADF STEM image and (b) EDS maps from the rectangular region marked in (a), showing top Al islands capped by a 20 nm As layer have been partially converted to Al_xGa or Al_xGa/AlGaSb islands during the FIB cutting. (c) HAADF STEM image from the region at the right side of (a), showing thickening of FIB induced AlGaSb layer at the interface inside the islands from the left to right.

Figure 5 Al-Ga Phase diagram

Fig.5. Al-Ga phase indicates the highest local temperature rise is about 210°C based on Al concentration detected in the Al_xGa droplet



Conclusions

- The FIB induced artifacts in TEM samples with Al islands on GaSb substrate that can lead to the epitaxial growth of AlGaSb QDs on the substrate.
- The artifacts are commonly observed in the TEM samples that have extensive high-energy and high-beam current (e.g., 30 keV, 10 nA) exposure but a limited clean and polishing at low energy (5keV and 2 keV) and low beam current (10pA).
- The effect can be reduced by increasing use of the low energy beam cleaning and polishing, and eliminated by cryo-FIB at a specimen temperature of -100°C.
- The mechanism involves the replacement of Al atoms in the islands by Ga atoms, formation of Al_xGa liquids on the surface, and the subsequent nucleation of $Al_xGa_{1-x}Sb$ at the liquid-Al_xGa/AlSb interface.
- The local heating by the ion-beam is likely an important factor.

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