

HBr-based gas cluster ion beam smoothing as a final polish for the production of MBE-epi-ready GaSb wafers

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ABSTRACT

For the first time, a gas cluster ion beam (GCIB) process incorporating Br gas was successfully used on GaSb (100) as a surface preparation technique for molecular beam epitaxy (MBE). Thermal X-ray photoelectron spectroscopy (TXPS) revealed that the surface oxide layer contained bonded bromine which was liberated at ~400 °C and Sb-oxides that were liberated at ~500 °C, leaving mostly Ga-oxides. Reflection high energy electron diffraction (RHEED) analysis within the MBE growth chamber showed a standard 1 x 3 reconstruction pattern in a Sb overpressure, confirming that Ga-oxides were desorbed by 530 °C, leaving a smooth single crystal surface for epitaxial growth. In addition, the epifaces of the Br-GCIB finished surfaces showed uniform step-terrace formations as compared to the swirled step terrace epifaces grown on chemical mechanical polishing (CMP) finished surfaces.

INTRODUCTION

GaSb substrates have advantages that make them attractive for implementation of very long wavelength infrared (VLWIR) detectors with higher operating temperatures for space based and stealth applications [1,2]. A significant perception that inhibits widespread commercial application of GaSb substrates regards the inconsistency of their surfaces for epitaxial growth and device fabrication. In particular, producing a scratch free GaSb surface with a readily desorbed oxide for epi-growth is difficult. The material is soft and easily abraded, and standard chemical-mechanical polishing (CMP) techniques may leave undesirable surface oxides that are stubborn to desorb.

Recently, bromine ion beam assisted etching (Br-IBAE) has been shown to be useful in etching CMP surfaces of small GaSb pieces [3,4]. In addition, the surface oxide formed during Br-IBAE is thin and easily desorbed in an MBE vacuum chamber, attributed to the strong Br chemistry. However, as a manufacturing technique, the Br-IBAE method will require appreciable design modification for full wafer or increased size wafer processing. Of significance, low energy gas cluster ion beam processing (GCIB) using O₂, F, and Ar gas sources have been found to be very effective at removing surface and subsurface damage on GaSb and InSb substrates [5,6,7]. GCIB, a nanoscale surface modification process, has provided a uniform, average surface roughness (R_a) of ~1-3 Å across semiconductor substrates ≤ 300 mm in diameter. GCIB has already been accepted in large scale thin film manufacturing. However, the use of GCIB on GaSb and InSb has been shown to leave a thick (~12-15 nm) surface oxide. This has required a high temperature (~530 °C), extended time for surface oxide desorption in the MBE chamber prior to high quality MBE superlattice growth.

Our research and development has focused on the use of Br for GaSb surface preparation using both Br-IBAE and Br-GCIB. The objective was to compare these surfaces with standard GaSb with a CMP finish, and analyze the “epi-readiness” of the three types of surface finishes. For the first time, a Br halogen gas cluster ion beam process on GaSb substrates has resulted in successful direct molecular beam epitaxy (MBE) growth and strong photoluminescence from the GaSb/AlGaSb quantum well structures.

EXPERIMENT

A Digital Instrument Atomic Force Microscope (DI AFM) acquired AFM images using the tapping mode for both $1 \mu\text{m}^2$ scans and $100 \mu\text{m}^2$ scans across the pre- and post- Br-GCIB, Br-IBAE, and as-received CMP GaSb surfaces. Rocking curve X-ray diffraction was used to examine the precision of GaSb (100) orientation and FWHM of the crystals, the orientation consistently found to be within GaSb (100) $\pm 0.1^\circ$.

For the Br-GCIB process, we used an Epion experimental gas cluster system with a beam composed of 1% HBr + 99% Ar. A 10 keV GCIB gentle etching step (85 Å GaSb removal) was followed by a 5 keV GCIB smoothing step (65 Å GaSb removal) for a total charge fluence of 1×10^{15} ions/cm². Thermal X-ray photoelectron spectroscopy (TXPS) was implemented to observe the desorption characteristics of the GaSb surfaces. In-situ MBE reflection high energy diffraction (RHEED) was used to analyze the surface prior to and after the Riber 2300 MBE GaSb growth. Cross section transmission electron microscopy (XTEM) was used to examine the quantum well structures and layer interfaces. Low temperature Photoluminescence (PL) was used to measure the quality of the MBE layers.

RESULTS

TXPS revealed that a surface layer containing bonded bromine is formed on the sample and this layer is thermally desorbed by 500 °C (Figure 1). Furthermore,

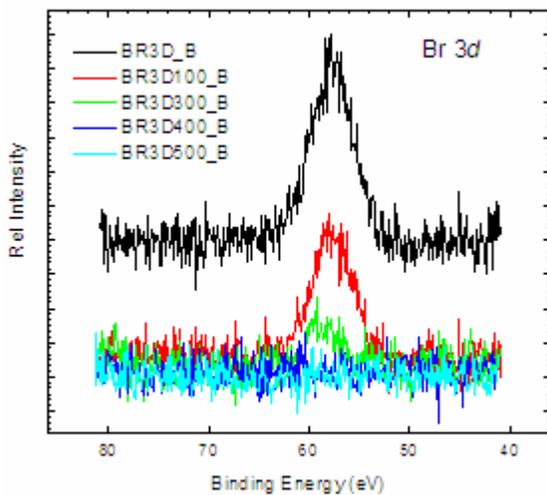


Figure 1. TXPS spectra of surface Br on a Br-GCIB finished substrate as a function of substrate temperature. Complete desorption at 400 °C and above is observed.

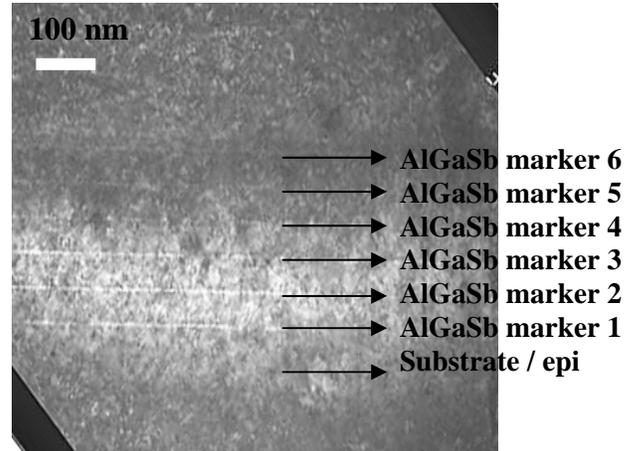


Figure 2. XTEM image of the GaSb (100) substrate to epi interface. AlGaSb marker layers can be observed as bright, straight lines indicating planar epi growth.

TXPS shows that the antimony based oxides have the highest vapor pressure, leaving the surface gallium oxides predominant at 500 °C. RHEED analysis in the MBE growth chamber confirms that the gallium oxides are desorbed by 530 °C, leaving a smooth single crystal surface.

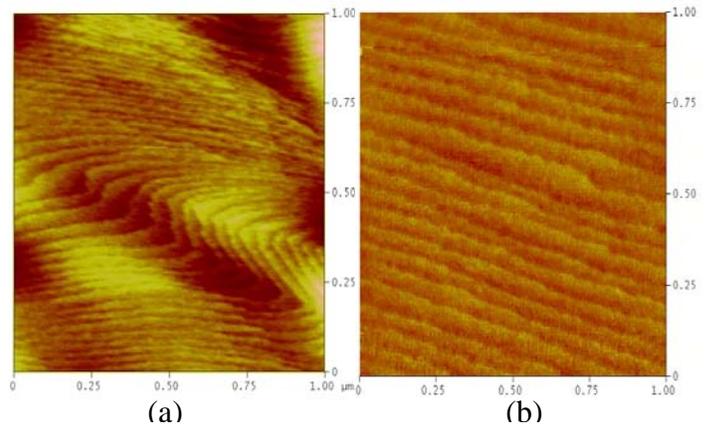


Figure 3: a) AFM ($1 \mu\text{m} \times 1 \mu\text{m}$) of the surface of a typical MBE epitaxial layer applied directly to a CMP polished GaSb wafer. b) AFM ($1 \mu\text{m} \times 1 \mu\text{m}$) of the surface of a MBE epitaxial layer applied directly to a Br-GCIB polished GaSb wafer. The rms roughnesses of the two epi surfaces are a) 0.18 nm and b) 0.07 nm.

The Br-GCIB processed GaSb (100) samples show the standard 1×3 RHEED surface reconstruction pattern under Sb flux at 440 °C. The MBE growth process on the desorbed GaSb surface consists of a $0.50 \mu\text{m}$ -thick GaSb buffer layer followed by a GaSb/AlGaSb quantum well structure followed by a ~ 10 nm GaSb capping layer.

XTEM reveals planar growth and an interface free of voids or dislocations when homoepitaxy is performed on the Br-GCIB surface prepared GaSb substrates. Marker layers of AlGaSb are visible. Figure 2 shows the substrate to epi interface area of MBE GaSb on the Br-GCIB surface with no indication of residual oxide or damage.

AFM images of a typical overgrown CMP surface and Br-GCIB processed surface are shown in Figure 3. The pre-growth rms roughness values for the wafers were 0.51 nm and 0.75 nm respectively while the post growth values were 0.18 nm and 0.07 nm respectively. Strong 77 °K photoluminescence was observed from the quantum well structure of the Br-GCIB processed sample, as shown in Figure 4.

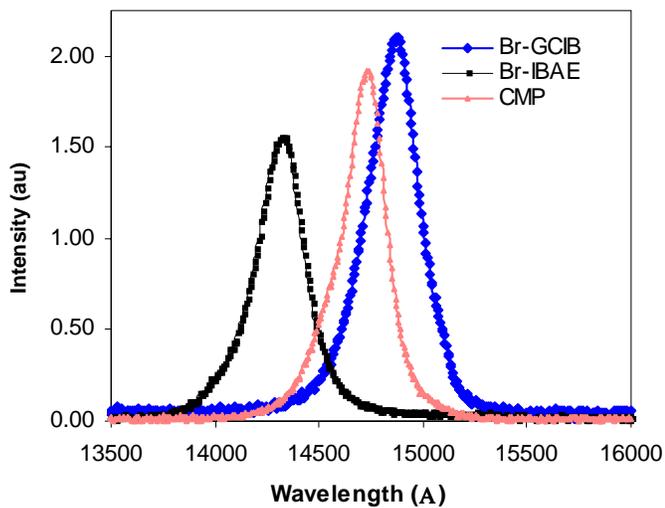


Figure 4. Low temperature photoluminescence spectra for the overgrown structures of CMP finish, Br-IBAE finish, and Br-GCIB finish GaSb (100) substrates.

SUMMARY AND ACKNOWLEDGEMENTS

The Br-GCIB process appears to be significantly more conducive to GaSb surface oxide desorption than the fluorine GCIB process on GaSb developed in earlier work [7]. This study is anticipated to lead into a method for producing “epi-ready” commercial GaSb wafers. The support of the United States Missile Defense Agency under contract number FA8650-04-M-5426 is gratefully acknowledged.

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ACRONYMS

GCIB: Gas Cluster Ion Beam
 MBE: Molecular Beam Epitaxy
 CMP: Chemical Mechanical Polishing
 RHEED: Reflection High Energy Electron Diffraction
 TXPS: Thermal X-ray Photoelectron Spectroscopy

