

Invited Paper

Manufacturing of 100mm diameter GaSb substrates for advanced space based applications

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Abstract

Engineered substrates such as large diameter (100mm) GaSb wafers need to be ready years in advance of any major shift in DoD and commercial technology, and typically before much of the rest of the materials and equipment for fabricating next generation devices. Antimony based III-V semiconductors are of significant interest for advanced applications in optoelectronics, high speed transistors, microwave devices, and photovoltaics. GaSb demand is increasing due to its lattice parameter matching of various ternary and quaternary III-V compounds, as their bandgaps can be engineered to cover a wide spectral range. For these stealth and spaced based applications, larger format IRFPAs benefit clearly from next generation starting substrates. In this study, we have manufactured and tested 100mm GaSb substrates. This paper describes the characterization process that provides the best possible GaSb material for advanced IRFPA and SLS epi growth. The analysis of substrate by AFM surface roughness, particles, haze, GaSb oxide character and desorption using XPS, flatness measurements, and SLS based epitaxy quality are shown. By implementing subtle changes in our substrate processing, we show that a Sb-oxide rich surface is routinely provided for rapid desorption. Post-MBE CBIRD structures on the 100mm ULD GaSb were examined and reveals a high intensity, 6.6nm periodicity, low (15.48 arcsec) FWHM peak distribution that suggests low surface strain and excellent lattice matching. The Ra for GaSb is a consistent ~0.2-4nm, with average batch wafer warp of ~4 μm to provide a clean, flat GaSb template critical for next generation epi growth.

KEYWORD LIST

IRFPA, MWIR, antimonides, CBIRD, LWIR, GaSb, surface analysis, MBE

1. INTRODUCTION

Engineered substrates such as large diameter (100mm) GaSb wafers need to be ready years in advance of any major shift in DoD and commercial technology, and typically before much of the rest of the materials and equipment for fabricating next generation devices. The starting GaSb (or InSb) substrate is the first tangible design component to ensure the smooth testing and development of the next generation array product. Volume ramping of the desired substrate is also essential for smooth device fabrication transition.

Gallium antimonide (GaSb) is a semiconductor substrate material whose demand is increasing due to its lattice parameter matching of various ternary and quaternary III-V compounds and its bandgap of which can be engineered to cover a wide spectral range from 0.3 to 1.58 eV (0.8-4.3 μm)¹⁻³. For next generation IR detector fabrication, stringent GaSb specifications have been imposed. Advanced SLS based IRFPAs require atomically smooth surfaces with rapidly desorbed thin oxides for high vacuum MBE device fabrication⁴⁻⁶. The GaSb specifics are often manufacturer dependent with respect to GaSb orientation and

dopant levels. Large diameter haze and flatness must also be low and consistent. With substrate backside removal as part of the IRFPA fabrication process, an extended wavelength transparency substrate may be preferred⁷. With worldwide production partnership, Galaxy Compound Semiconductors, Inc. and Wafer Technology are currently gearing for volume ramp up of large diameter GaSb material in-sync with industry demand for next generation infrared detectors. This paper examines the state of the art materials processing and MBE characterization for large diameter GaSb which verifies next generation device fabrication capability.

2. GaSb REQUIREMENTS

GaSb substrates from a large diameter crucible of an Sb rich Czochralski melt are cut to within $\pm 0.1^\circ$ of the customer preferred crystal orientation for 100mm substrate processing. The requirements for large diameter GaSb substrates are based upon critical industry requirements for controlling the MBE growth layers of SLS based IRFPA detectors. As individual monolayers of GaSb or InAs ($\leq 1\text{nm}$) are deposited directly upon the GaSb substrate, stringent attributes of average surface roughness ($R_a \sim 0.1\text{-}0.3\text{nm}$), oxide layer thickness ($\sim 2\text{nm}$), and easily desorbable composition (Sb-oxide dominant) must be provided to the end user. A low particle count ($< 5/\text{cm}^2$), low etch pit density ($< 500/\text{any given cm}^2$), and flat substrates ($< 5\mu\text{m}$ warp) must also be delivered.

3. LARGE DIAMETER GaSb CHARACTERIZATION

As the diameter of the substrate increases, each material parameter becomes more challenging to maintain or improve upon. To provide 100mm GaSb to satisfy or exceed critical MBE growth specification, the GaSb boule undergoes a complete processing and characterization as shown in Figure 1. Control documents at every process step are used to ensure a consistent and reproducible product. Multiple inspection steps are used to collect the data, evaluate the flow process capability and generate the wafers for advanced detectors.

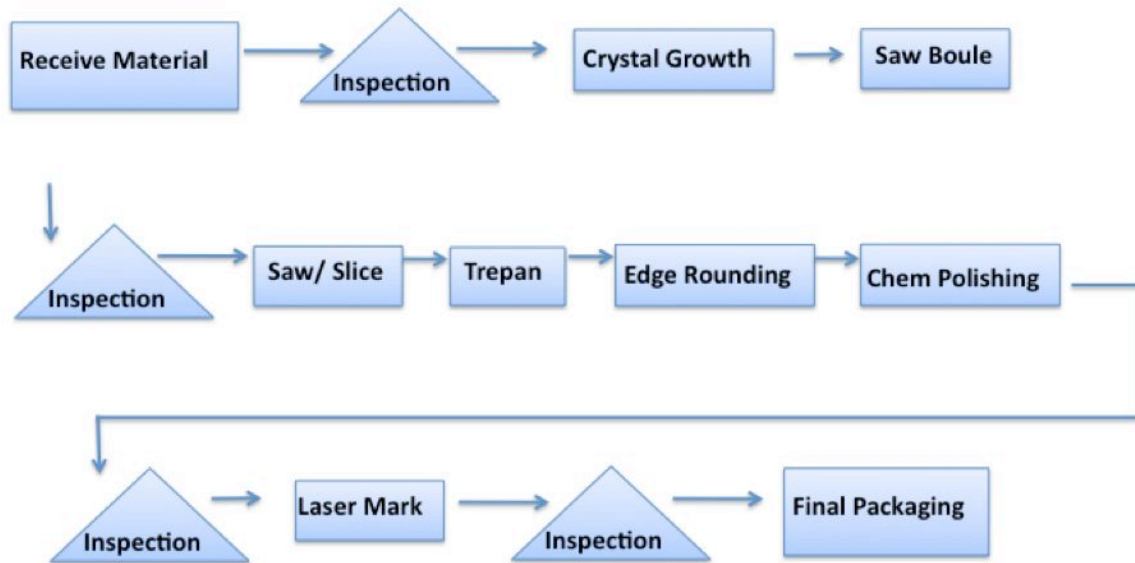


Figure 1. Large diameter 100mm GaSb growth, processing, and inspection flow.

3.1. Surface Morphology

The sawed and polished GaSb substrates are examined for surface roughness by Nomarski optical microscopy as well as periodic sampling by a Digital Instruments Nanoscope III AFM with Si tips used in the tapping mode. A key GaSb surface improvement has recently been implemented as a result of the partnership with IQE and Wafer Technology. The wider access to IQE's testing equipment has provided end users with the best surface polishing and cleaning practices of both companies. The marked improvement of the surface polishing process is indicated by the AFM images of the 100mm GaSb surface as shown in Figure 2, comparing the surface roughness from year 2010 to 2011. Of note, obvious scratch elimination has led to the significant peak-to-valley reduction from ~31nm to ~6-7nm and an average surface roughness reduction from 0.7-0.8nm to 0.2-0.4nm.

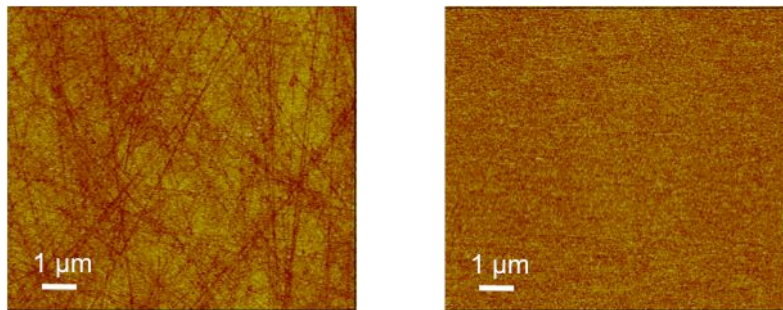


Figure 2. AFM images ($10 \times 10 \mu\text{m}^2$) showing January 2010 (left image) and January 2011 (right image) surface morphology metrics of 100mm GaSb confirming a significantly improved polishing process.

3.2. Surface Cleanliness

Each GaSb substrate is inspected for surface cleanliness by a KLA-Tencor Surfscan 6220. Particle counts (per cm^2) and haze (ppm) are measured and analyzed for 50mm to 100mm GaSb substrates. With a 5mm edge exclusion, particles are binned into size ranges, counted, color coded, and mapped. Figure 3 shows a

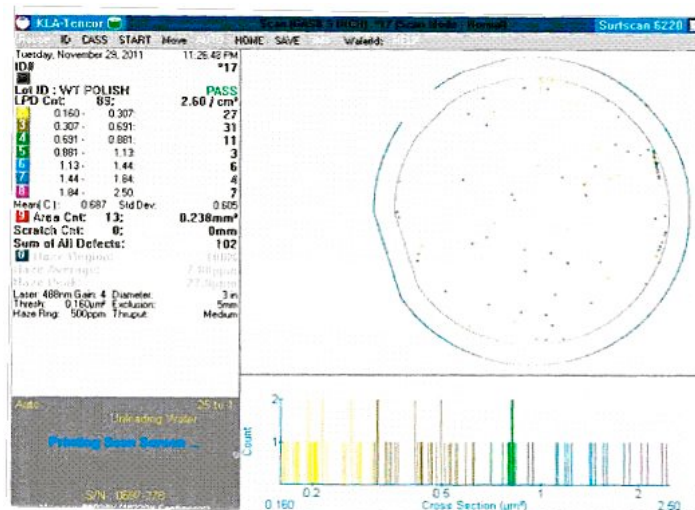


Figure 3. Surfscan particle map showing average count of $2.60/\text{cm}^2$ and average haze 7.8ppm for 75mm GaSb.

75mm GaSb cleaned wafer surface with an average particle count of $2.60/\text{cm}^2$ and 7.8 ppm haze. To reduce the haze to values of <5 ppm, additional spin, rinse, dry may be required. The haze is calculated with 100% of the wafer area analyzed. The particle measurement may be provided at every step of the substrate finishing process to determine which, if any, areas of processing require reduced particulate efforts. Careful tracking of particles and haze for each wafer may be maintained for pre- and post-epi growth correlation of defects or subsequent IRFPA pixel viability.

3.3. Wafer Flatness

Critical parameters for state-of-the-art epi growth for advanced detectors include the 100mm GaSb wafer warp, bow, and TTV. The wafer flatness can either be measured by a 4D Technology Wyko 600 interferometer or newly implemented Tropel Flatmaster. A vacuum chuck is not employed for the measurement, giving a free standing bow and warp analysis. Figure 4 shows the Wyko interferometry analysis of a 100mm n:GaSb(100), indicating the flatness measured as peak-to-valley at $0.75\mu\text{m}$ for free standing wafer warp. Flatness values are reported for the center 90% area of the 100mm wafers with a 5mm exclusion. The Wyko reveals the general warp shape of the GaSb wafer (convex, concave, saddle) through X-Y graphing.

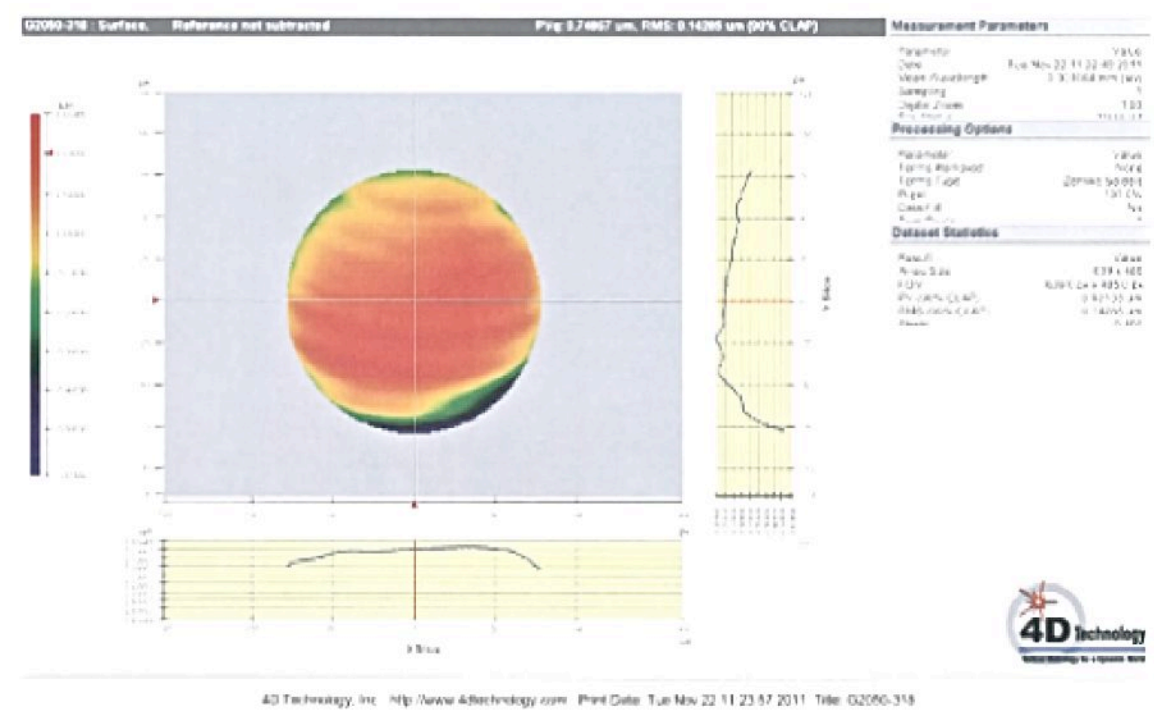


Figure 4. Wyko Interferometry of (100)GaSb wafer flatness, lot G2036, 100mm diameter.

Figure 5 shows a chart of 73 n:GaSb (100) substrates of 75mm diameter that were manufactured and measured for bow, warp, and TTV by a Tropel flatmaster. The wafer warp ranges from 2-8 μm , with an

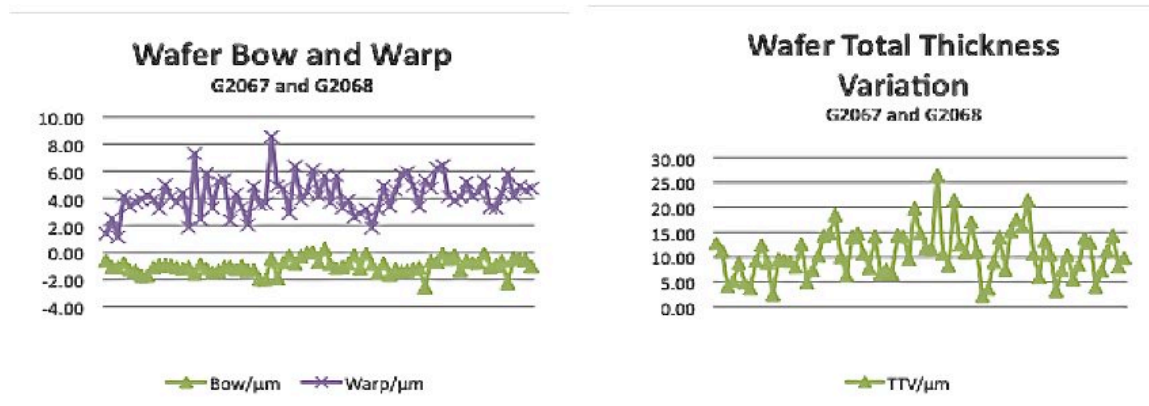
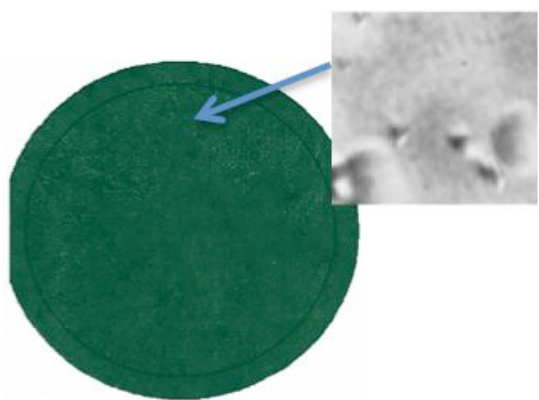


Figure 5. n:GaSb (100) substrate bow, warp, and TTV measured for a 73 wafer batch, 75mm diameter.

average of 4.22 μm (STD=1.34 μm). As verified by flatness measurements, the bow is slightly concave, averaging 0.97 μm (STD=0.54 μm). The measured substrates show the total thickness variation to batch average at 10.68 μm . Clearly, a challenge for manufacturing GaSb substrates is reducing the TTV to <5 μm for every wafer.

3.4. Crystalline Carrier Concentration and Defects

Critical parameters for state-of-the-art epi growth for advanced detectors include the 100mm GaSb wafer carrier concentration (Hall effect), mobility, and wafer etch pit density assessment. Wafers are culled from each batch for characterization that includes chemical etching as a function of substrate orientation. The wafer is artificially gridded after the surface etch and examined with Nomarski optical microscopy. With a 32mm major wafer flat, a 10mm total exclusion edge is implemented for 100mm diameter GaSb substrate maps. Defect density is recorded for each cm^2 . For GaSb, >1000/ cm^2 defect average is specified, but typically the extended wafer center is significantly less than 100/ cm^2 EPD. Figure 6 shows a completed EPD measured wafer for which there is <500 EPD for any given cm^2 .



Defect decoration on n:GaSb (100)

Carriers: $1.39\text{E}17/\text{cm}^3$
 Mobility: $4.98\text{E}3\text{cm}^2/\text{V-sec}$
 EPD <500 for any cm^2

Figure 6. The defect etch map determines average EPD/ cm^2 for 100mm GaSb.

3.5. Surface Oxide Analysis

X-ray photoelectron spectroscopy (XPS) is used to determine the surface oxide composition of occasional processed substrates. The XPS involves irradiating a sample with monochromatic X-rays of a characteristic energy. This causes photo electrons to be ejected with a range of energies depending on the element from

which they are emitted and the chemical state of that element. The emitted electrons are collected by an energy analyzer and sorted according to their energies. For the analysis of GaSb surfaces with a thin surface oxide, the process is ideal, as XPS typically examines the material composition to a depth of 100nm. The analysis is conducted in a Thermofisher ESCALAB 250i electron spectrometer equipped with a hemispherical sector energy analyzer. A monochromatic Al K α X-ray source is used at a source excitation energy of 15 keV and emission current of 6 mA. Analyzer pass energy of 20 eV with step size of 0.1 eV and dwell time of 50 ms is used. Under these conditions, the energy resolution is better than 0.4 eV. The sample is positioned normal to the analyzer input lenses and base pressure for collection is always at better than 5×10^{-10} mbar (to avoid surface contamination).

The XPS measured the ratios of the Ga-Ga / Sb-Sb and Ga oxide / Sb oxide as derived from the peaks of the spectrum. One important peak distribution is shown in Figure 7. As shown in that figure, Sb3d peak spectra from two different samples from the same GaSb boule are superimposed on the same binding energy range. One sample underwent the 2010 GaSb surface polish and clean and the other has the 2011 GaSb surface polish and clean. The 2010 GaSb polish and clean shows a strong Sb-oxide Sb3d binding peak concentration and a high Sb-Sb surface binding. The 2011 Sb3d spectrum shows a the strong Sb-oxide peak, with the Sb-Sb binding nearly non-existent. The presence of the stronger Sb-oxide peak (rather than a dominant Ga-oxide presence, not shown) is preferred. Overall, XPS studies for the comparison of the surface cleans revealed the 2011 polish and clean consistently produced a Sb-oxide rich surface (~83%), desirable for rapid ultra-high vacuum (MBE) desorption.

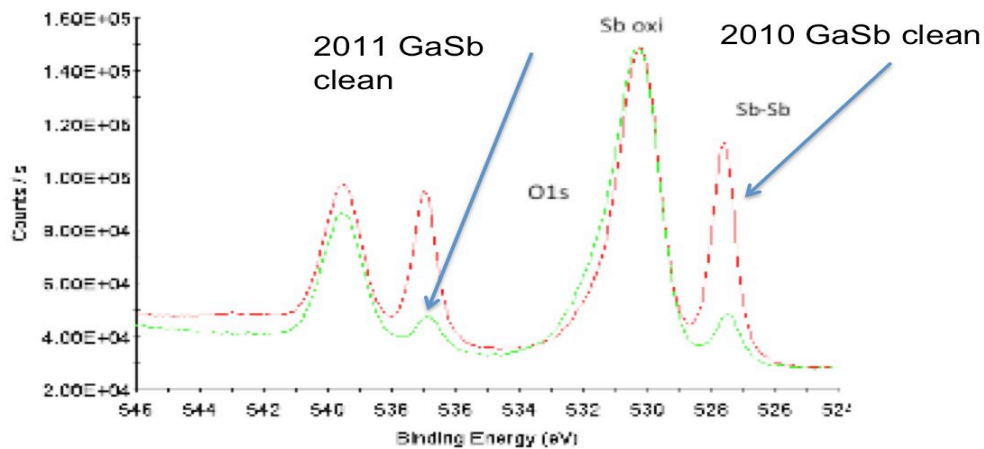


Figure 7. XPS spectra comparing the Sb3d peaks for 100mm GaSb samples from the same boule that compare the 2010 and 2011 polishing and cleaning processes.

3.6. MBE Growth on 100mm GaSb Surface

The ultimate test for state-of-the-art GaSb substrates is the ease and quality of MBE advanced SLS growth on the finished wafer surface. Figure 8 shows the x-ray diffraction (XRD) spectrum of a CBIRD structure from the Jet Propulsion Laboratory (JPL) grown by MBE on a 100mm GaSb substrate⁸⁻⁹. The stringent

layer growth for the CBIRD structure consists of an n-contact, a hole barrier, an absorber, an electron barrier, and a p-contact¹⁰.

The XRD spectrum of Figure 7 for a CBIRD epi structure shows a GaSb substrate peak very close to the zero-order SLS peak, and numerous satellite peaks due to the superlattice that suggests a high quality epitaxy material. The mismatch between the substrate and SLS layers is calculated from the angular difference (θ) between the substrate and SLS peaks. The spectrum shows the initial layer is well-matched to the substrate lattice, with a measured mismatch of $\Delta a/a$ that translates to a strain = -815ppm in tension. The XRD spectrum shows a coincident substrate and zero-order SLS peak within the resolution of the data. The superlattice periodicity is obtained using the XRD spectrum by plotting $2\sin(\theta)/\lambda$ versus the satellite peak number¹⁰. This analysis gives a straight line whose slope is inversely proportional to the superlattice periodicity. The measured periodicity of the sample is $\sim 66.3 \text{ \AA}$ with the FWHM of the $SL_0 = 15.55 \text{ arcsec}$ (0.00432 degrees). The low FWHM implies a high crystalline quality in the superlattice and underlying substrate. The periodicity of the sample implies a lower effective bandgap and hence a longer cutoff wavelength¹¹.

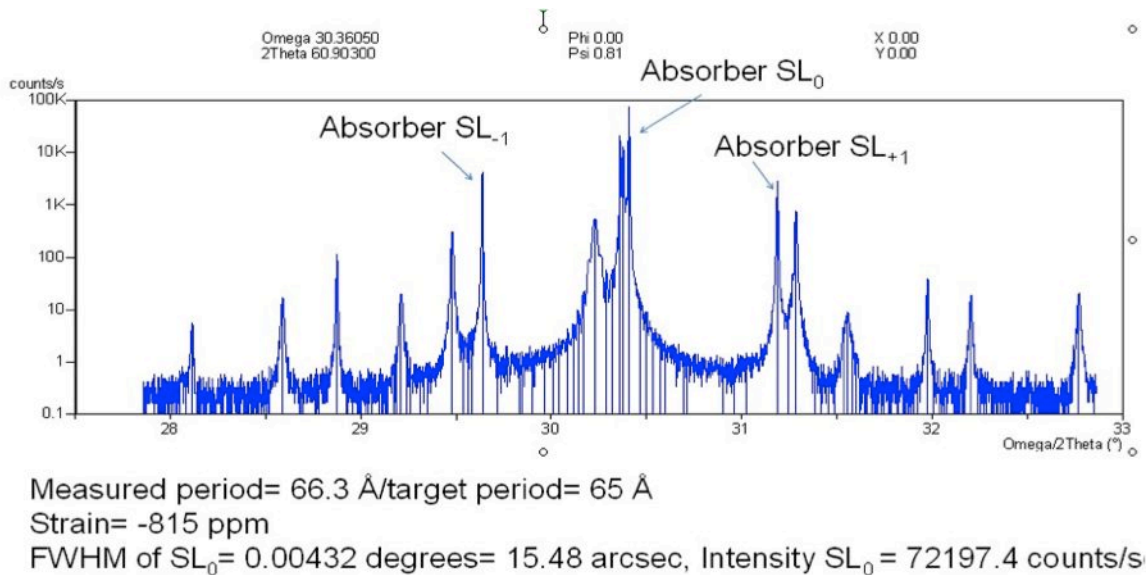


Figure 8. (JPL) XRD measurement of a MBE CBIRD structure grown on a 100mm n:GaSb. The XRD shows a measured period of 66.3 angstroms and a well matched substrate to lattice structure.

4. SUMMARY

In order to provide an ideal starting surface for MBE advanced detector growth and maximum IRFPA pixel viability, the manufacturing of GaSb substrates must continually address risk reduction for scale up of larger diameter megapixel devices. To improve resolution and sensitivity requirements for high performance infrared focal plane array (IRFPA) imaging systems in the 2-25 μm region (77°K), the surface of new larger diameter (100mm) GaSb substrates must meet or surpass stringent demands. This overview has shown that the preparation of next generation wafers requires extensive materials characterization of basic surface properties such as surface smoothness, surface oxide thickness and chemistry, particles, haze, and EPD. Bulk wafer analysis includes parameters of bow, warp and TTV. MBE epi growth also provides an assessment for next generation GaSb substrate readiness. Future challenges include TTV and EPD

reduction as well as pre-epi and post-epi substrate character correlation. Advanced in-house GaSb substrate processing and analysis are on the roadmap for next generation substrate production, the latter of which needs to precede increased megapixel IRFPA fabrication timelines.

5. CONCLUSION

For next generation megapixel GaSb based IR detectors, a low crystallographic defect density, a rapidly desorbing surface oxide, and an ultra-low wafer warp, bow, and TTV remain paramount in the context of substrate production. Increased camera performance ultimately depends upon the quality of the basic material components which must be ready prior to tangible commercial usage. Continuous improvements in substrate fabrication and characterization methods are essential to provide a high yield base for consistent and large scale production of advanced IRFPAs.

6. ACKNOWLEDGEMENT

The authors gratefully acknowledge the support of IQE, Inc. and the XPS data from Drs. Sayah Saied and Baogui Shi of Midlands Surface Analysis.

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