

ABSTRACT

The wide bandgap group III-nitrides including GaN, AlN, and InN are important for visible/UV optoelectronics as well as high power devices. Currently III-nitride devices are grown on non-native substrates, which lead to dislocations and other defects in material. These defects adversely affect the quality and lifetime of the devices. We attempt to grow low defect, single crystal, thick films of InN on sapphire substrates films by plasma-assisted liquid phase epitaxy (PALPE), which is a novel low pressure synthesis technique involving microwave activated nitrogen. Eight growth experiments were performed in which polycrystalline films of InN were grown on liquid Indium pellets. Morphology of the samples was analyzed by optical and scanning electron microscopy. Composition of the samples was determined by energy dispersive spectroscopy (EDS) and by X-ray diffraction (XRD). We also determined an optimal temperature range of ~450-500°C for InN growth. Finally a new sample holder for the PALPE system was designed and constructed.

INTRODUCTION

The group III-Nitrides (GaN, AlN and InN) are of particular technological importance because of the wide range of bandgaps spanned by the nitrides and their alloys. The bandgap of III-Nitride semiconductors ranges from 0.8 to 6.4 eV, which covers the energy spectrum from the infrared to the ultraviolet. The wide range makes the fabrication of LEDs with wavelengths spanning the entire visible spectrum possible. In addition, the high band gaps of III-Nitrides are useful in UV diode lasers and high power devices.

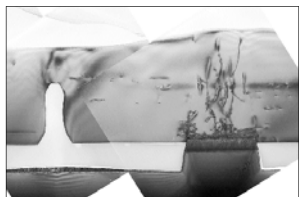


Figure 1: TEM micrograph of $Al_{0.9}Ga_{0.1}N$ film grown via pendeo-epitaxy on unmasked GaN seed posts on a 6H-SiC substrate showing dislocations (dark bands). From review on Pendeo-epitaxy techniques (Davis et al.)

III-Nitride semiconductors also have high thermal, chemical and mechanical stability, as well as other useful physical properties, which makes them an extremely versatile tool for electronic devices.

Currently, commercial devices using III-Nitrides are built on non-native substrates (typically sapphire). However, due to the large mismatch in lattice parameter and the difference in thermal expansion coefficients, the use of non-native substrates leads to the formation of dislocations and residual strains on the semiconductor lattice. These defects affect the quality and performance of the semiconductor.

In order to improve the quality of semiconductor devices, better substrates are needed. We attempt to grow low defect, thick film, single crystals of InN on sapphire substrates by plasma-assisted liquid phase epitaxy (PALPE). These thick films could be used as substrates to fashion higher quality III-Nitride devices. In our experiments growth temperature was varied to investigate optimal growth conditions and processes.

PALPE SYSTEM AND GROWTH METHOD

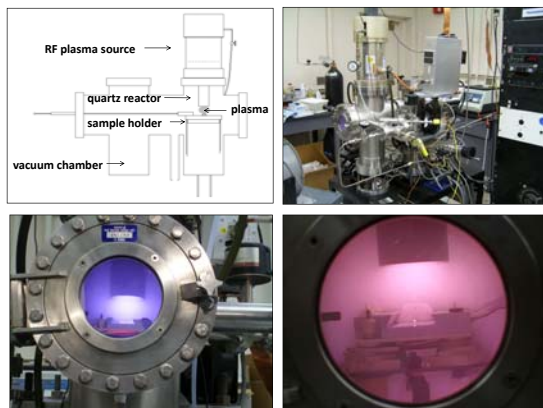


Figure 2: Diagram of inside of PALPE system. Images of exterior showing stainless steel phlanges, plasma source and RF power supply and turbopump (top right). Images of interior of PALPE showing sample holder with sample, resistance heater, quartz reactor and ball plasma during InN growth.

PALPE is a novel low pressure, liquid phase epitaxy technique involving microwave activated nitrogen. The PALPE system uses a Delta Glow™ DG-300 High Energy Plasma Source to deliver activated nitrogen species via a nitrogen plasma to a liquid In sample under high vacuum (high vacuum is achieved first by a roughing pump and then by a turbopump). InN is then grown from the supersaturated melt. Gas flow is controlled by two mass flow controllers (not shown).

RESULTS AND ANALYSIS

Over the course of the year, we performed eight growth experiments on InN. We varied the conditions and made systematic adjustments in growth procedure in attempt to achieve thick film growth. In our experiments we were able to grow very thin polycrystalline crusts over a liquid pellet. The figure below shows a SEM image of the surface of one of the polycrystalline crusts grown.

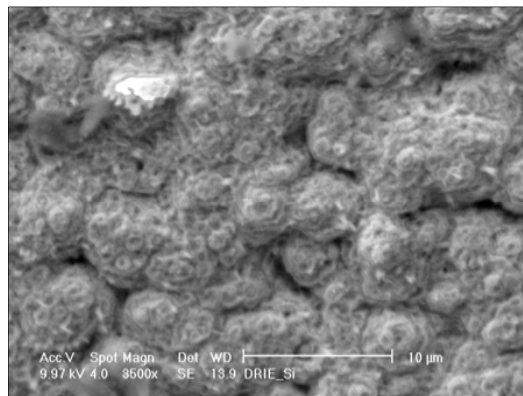


Figure 3: SE micrographs of samples in N-7 grown at 468°C. Sample seems to be made up of colonies of ~micron size InN platelettes.

Growth experiments were analyzed using several methods. First the samples were examined under an Olympus BX-60 microscope. High resolution images of the morphology of the surface of all of the samples was taken on a Phillips XL30 Environmental Scanning Electron Microscope (ESEM). The composition of the crystals was determined by energy dispersive spectroscopy (EDS) on the ESEM and by X-ray diffraction (XRD).

The figure above shows a SEM image of sample InN-7. The image shows what appears to be colonies of InN platelettes. The figures below are the EDS (top) and XRD (bottom) spectra for sample InN-7. Both spectra are checked against the respective chemical databases.

The EDS spectra shows strong peaks for both In and N. In and N are not in stoichiometric ratios (by mass), but this is somewhat expected as EDS does not detect lighter elements as accurately as it does heavier elements. The fact that both In and N peaks are present is significant. The lower figure shows the XRD spectra for the sample. Blue peaks show the actual data and the red and green lines show the known peaks for InN and In metal. The In metal lines are much lower in intensity, which indicates the crust is composed largely of InN with small amounts of free In metal.

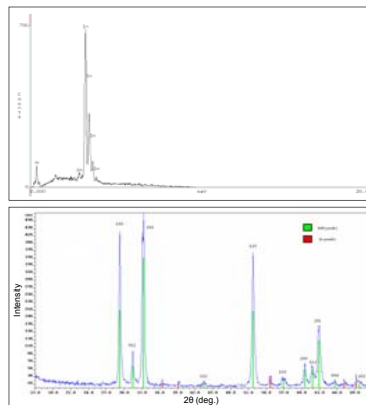


Figure 4: EDS (top) and XRD (lower) spectra for above sample. XRD spectra of wurtzite structure InN and free In for comparison.

REDESIGNED SAMPLE HOLDER

In order to continue with our experiments (particularly those involving the slider), revisions to the system were needed. We redesigned, machined and assembled a new sample holder system that will allow future experiments with the slider. The new holder system is shown below.

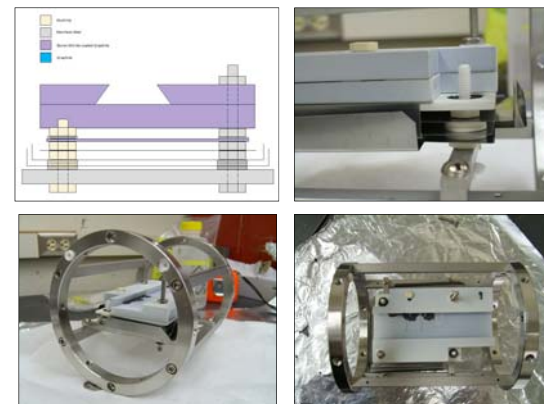


Figure 5: Diagram (top left) and images of the redesigned sample holder showing the heat shields (top right), chassis (lower left), and boat (bottom right).

The new chassis provides more support for the slider and boat and also allows for the boat to be removed for cleaning and maintenance more easily. The most important factor in the new holder design are the thermal properties of the system. Since growth conditions require such high temperatures, thermal expansion and heat loss (due both to radiation and conduction) were given careful consideration.

CONCLUSIONS

- several samples have been grown in the system and determined to be polycrystalline InN
- optimal temperature range of ~450-500°C for InN growth has been determined experimentally
- a new and more effective holder has been designed, constructed and installed into system
- over the course of the year I learned to use PALPE system, scanning electron microscope and X-ray diffractometer with some proficiency

FUTURE WORK

More experiments need to be performed on InN to achieve thick film growth. In addition the system will be used to attempt thick film growth of Aluminum Nitride (AlN) and III-Nitride alloys.

ACKNOWLEDGEMENTS

Many thanks to Prof. Kathleen Kash and Tim Peshek for help with my experiments and to the NSF for funding