

OFFICE OF NAVAL RESEARCH
END-OF-THE-YEAR REPORT
PUBLICATIONS/PATENTS/PRESENTATIONS/HONORS/STUDENTS REPORT

for

GRANT: N00014-97-1-0951

PR Number 97PR07098-00

Colin E. Wood, Program Officer

Interfacial Bonding Research for Compliant Substrates

David L. Miller and Theresa S. Mayer, Principal Investigators

The Pennsylvania State University
College of Engineering
Department of Electrical Engineering
University Park, PA 16802

Office of Sponsored Programs
110 Technology Center Building
University Park, PA 16802-7000

July 24, 1998

Reproduction in whole, or in part, is permitted for any purpose of the United States Government.

This document has been approved for public release and sale, its distribution is unlimited.

19980805 145

Aims of the project

The goals of this research project as stated in the original proposal are “the improvement of epitaxially grown lattice-mismatched III-V compound semiconductors by development of a practical compliant substrate technology, and the use of this technology for the integration of optoelectronic emitters and detectors with silicon circuits.” “A key to our project is investigation of the roles of interfacial material composition, surface chemistry, and interfacial mass transport during bonding and subsequent annealing on the performance of wafer-bonded compliant substrates for the subsequent growth of lattice-mismatched III-V compounds, including GaN. We will use our capabilities for epitaxial growth and *in situ* surface modification to prepare GaAs and Si wafer surfaces for direct wafer-to-wafer bonding. We will then bond those substrates in the same vacuum where their surfaces were prepared, assuring control over the surface composition and chemistry, and determine how the compliant substrates’ properties depend on interfacial composition and bonding conditions. We will apply our understanding of compliant substrates to the development of a practical process for the growth of III-V photonic devices on GaAs compliant substrate pads located at multiple sites on a Si wafer.”

Our overall goals are essentially unchanged over the past year, although the experiments we are using to understand and improve compliant substrate technology and science have changed as we have learned from our experiments and other groups’ results. Originally we proposed to investigate the atomic details of wafer bonding in vacuum in order to understand how wafer bonding, and specifically twist bonding, results in a compliant interface. Since preparing the proposal, however, we have become increasingly skeptical of the ability of direct wafer bonding to provide compliance over a large area. We have therefore focused on two areas: relaxed epitaxial growth of InGaAs on a twist-bonded GaAs compliant substrates¹ and GaAs compliant substrates bonded with intermediate SiO₂ or indium oxide layers, and relaxed epitaxial growth of InGaAs on a compliant substrate fabricated by epitaxial growth of an AlAs/GaAs structure with the subsequent oxidation of the AlAs. The latter approach is in many ways similar to one being investigated at the University of California, Santa Barbara.²

Results

Wafer bonding approach

We have successfully bonded GaAs wafers over large areas using techniques widely reported in the literature and implemented in our laboratory prior to the initiation of

this project. Briefly, native oxide is removed from GaAs wafers using $\text{NH}_4\text{OH}:\text{H}_2\text{O}$ and then placed in contact at room temperature. A bond is quickly formed which is sufficiently strong to allow handling of the wafers. This bond is strengthened by annealing in N_2 at 450°C for 30 minutes.

One of the wafers has an epitaxial structure grown on the bonded face comprising 100 nm of $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ followed by a thin (20nm) layer of GaAs. After the two wafers are bonded, the substrate on which the epitaxial layers were grown is removed by grinding and selective etching, with the $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ acting as an etch stop. The $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ is removed selectively by HF or, preferably, HCl, leaving behind a thin GaAs layer bonded to a GaAs substrate. If the wafers are rotated before bonding so that the [110] directions in the surface plane form an acute angle, "twist bonding" is achieved.

We have successfully reproduced the twist bonding of GaAs layers as thin as 20 nm over areas as large as 1 cm x 1 cm. In many cases these layers are defect-free when observed optically under magnification. However, the nucleation of epitaxial growth reveals that the surface is atomically much rougher than a bulk GaAs substrate as the result of the $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ removal. This roughness has been confirmed by AFM. Nucleation is difficult and begins as 3D growth, which would be particularly troublesome in the nucleation of InGaAs on GaAs. The properties of material grown on this surface are degraded, as seen from measurements of GaAs p-n junctions grown on our twist-bonded substrates.

In order to provide good surfaces for epitaxial layer nucleation, we have developed a "digital" etching procedure comprising alternate exposure to hydrogen peroxide (oxidizer) and ammonium hydroxide (oxide dissolving) solutions. Not only does this procedure lead to a very well controlled GaAs removal rate of 1.5 nm per cycle, but we have also found that it greatly smoothes the surface which exists following $\text{Al}_{0.3}\text{Ga}_{0.7}\text{As}$ removal. Even so, the surface is still not as smooth as the surface of commercial GaAs wafers.

Growth of $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ on twist-bonded GaAs layers as thin as 20 nm has failed to reveal compliant substrate behavior over macroscopic (mm^2) areas, as witnessed by surface corrugations resulting from strain, by x-ray diffraction linewidths, and by p-n junction leakage currents. These results are summarized in our publication, "The effect of $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ etch stop removal on the preparation of wafer-bonded compliant substrates," by Zhang *et al.* ³

Oxidized AIAs approach

Our second approach to a compliant substrate technology is to use a layer of oxidized AIAs as the weak bonding layer between a very thin GaAs layer and a GaAs

substrate. As with the twist bonding approach, the very thin GaAs, weakly bonded to its supporting wafer, in principle can deform to absorb the strain of lattice-mismatched material grown over it. In this case, the oxidized AlAs plays the role of the twisted bonds in the twist-bonding approach to provide the weak bonding to the substrate. A variation to this scheme, reported at the 1998 Electronic Materials Conference (Ref. 2) is to grow a pseudomorphic layer atop the thin GaAs before oxidation of the AlAs, allowing the oxidation process to relieve strain in that layer.

No wafer bonding is needed in this process, but we must have an intact thin GaAs layer on top of the oxidized AlAs with a surface which is suitable for epitaxial growth. Once again, as with the twist bonding approach, surface preparation is a key to successful nucleation of lattice-mismatched materials.

We have successfully produced intact GaAs layers as thin as 30 nm on top of oxidized AlAs in stripes 100 μ m wide. The oxidation process removes about 10 nm of the originally 40 nm GaAs layer. Thinner GaAs layers have so far resulted in non-uniform oxidation across the stripes. We have also developed an *in situ* etch using iodine vapor in our molecular beam epitaxy system to thin the GaAs layer. Unfortunately, the surface of the GaAs layer after oxidation (with or without iodine etching) is atomically rough, and growth of lattice-mismatched material (InGaAs) on it results in 3D nucleation. We have not observed any indication of compliant behavior in this system over macroscopic areas. We are continuing to work on improving the GaAs surface properties to eliminate nucleation as a complicating factor in lattice-mismatched epitaxial growth on these substrates.

Plans for the coming year

In the coming year we plan to continue to investigate both the twist bonding and the oxide layer methods of forming thin compliant GaAs substrates. Our focus is on strain relaxation without threading dislocation generation in the overlying epitaxial layers in macroscopic areas. We will continue to use x-ray diffraction to measure lattice relaxation related to dislocation formation and p-n junction reverse leakage current as a measure of the electrical quality of the overlying layers. We will supplement these measurements with AFM images of surface roughness, which can reveal important details of the initial surface roughness and of surface corrugations due to dislocation generation in InGaAs.

Reproducibility of oxidation has been an annoying problem during the first year, so we will be improving our oxidation capability this Fall.

So far, we have tested substrate compliance using InGaAs. Dislocations appear to move easily in this material and the resultant inhomogeneous strain leads to surface roughening. Nucleation is very sensitive to substrate preparation because of the high

mobility of Ga and In atoms on the growing surface. We will begin to investigate other, possibly more dislocation-resistant, materials such as InAlAs.

Other groups have reported that GaAs layers adjacent to oxidized AlAs can be damaged by oxygen or arsenic-related point defect diffusion. We will investigate this effect on our thin substrates by burying thin diffusion-blocking AlGaAs layers or superlattices within the GaAs compliant layers. We also plan to measure the electrical properties of the thin GaAs layers directly, before and following AlAs oxidation, to determine the contribution of the oxidation process to surface roughness or point defects which may diffuse into epitaxial layers grown subsequently.

One of the most important parameters which must be measured to evaluate the suitability of these structures for compliant substrates is the actual degree of compliance obtainable from twist bonded interfaces and from oxidized AlAs layers. A determination of the compliance of twist-bonded interfaces and oxide layers which is independent of the nucleation and growth of lattice mismatched materials would be an important milestone. We are thinking about methods to extract this information.

Publications, Presentations, and Student Support

Publications and presentations

C. Zhang, D. Lubyshev, T. N. Jackson, D. L. Miller, T. S. Mayer, "The effect of $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ etch stop removal on the preparation of wafer-bonded compliant substrates," submitted to the Journal of the Electrochemical Society.

C. Zhang, D. I. Lubyshev, W. Cai, J. E. Neal, D. L. Miller, T. S. Mayer, "Demonstration of a GaAs -Based Compliant Substrate Using Wafer Bonding and Substrate Removal Techniques," Proceedings of the 24th International Symposium on Compound Semiconductors, Sept. 11-14, 1997, San Diego, CA, USA, IEEE Press, p. 25.

D. Lubyshev, T. S. Mayer, W-Z. Cai, D. L. Miller, "Lattice Mismatched MBE on Compliant Substrates Produced by Lateral AlGaAs Oxidation," to be presented at the Tenth International Conference on Molecular Beam Epitaxy, Cannes, France, August 31-Sept. 4 1998. Manuscript in preparation.

C. Zhang, D. Lubyshev, D. L. Miller, T. N. Jackson, T. S. Mayer, "Improvements in the Surface Preparation of GaAs-Based Compliant Substrates," presented at the 40th Electronic Materials Conference, Charlottesville, VA, June 24-26, 1998. Unpublished.

Students and others supported

Postdoctoral research scientist Dmitri Lubyshev, 50% FTE for the year.

¹ F. E. Ejeckam, Y. H. Lo, S. Subramanian, H. Q. Hou, B. E. Hammonds, "Lattice engineered compliant substrate for defect-free heteroepitaxial growth," *Appl. Phys. Lett.* **70** (13), 1685 (1997).

² P. Chavarkar, L-J. Zhao, S. Keller, S. Mathis, A. Black, E. Hu, S. Speck, U. Mishra, "Lattice Engineering Using Lateral Oxidation of AlAs: An Approach to Generate Substrates with New Lattice Constants," Abstract Booklet of the 40th Electronic Materials Conference, University of Virginia, Charlottesville, VA, June 24-26, 1998, unpublished.

³ C. Zhang, D. Lubyshev, T. N. Jackson, D. L. Miller, T. S. Mayer, "The effect of $\text{Al}_{0.7}\text{Ga}_{0.3}\text{As}$ etch stop removal on the preparation of wafer-bonded compliant substrates," submitted to the *Journal of the Electrochemical Society*.

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE 7-24-98	3. REPORT TYPE AND DATES COVERED End-Of-Year Report/7-1-98 - 7-30-98		
4. TITLE AND SUBTITLE Interfacial Bonding Research for Compliant Substrates		5. FUNDING NUMBERS Grant: N00014-97-1-0951 PR #: 97PR07098-00		
6. AUTHOR(S) David L. Miller and Theresa S. Mayer, Principal Investigators				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) The Pennsylvania State University College of Engineering/Office of Sponsored Programs 110 Technology Center Building University Park, PA 16802-7000		8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Office of Naval Research ONR 251/Ballston Centre Tower One 800 N. Quincy Street Arlington, VA 22217-5660		10. SPONSORING/MONITORING AGENCY REPORT NUMBER		
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for Public Release		12b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) The goal of this project is the improvement of epitaxially grown lattice-mismatched III-V compound semiconductors by development of a practical compliant substrate technology. We have focused on two areas: relaxed epitaxial growth of InGaAs on a twist-bonded GaAs compliant substrates and GaAs compliant substrates bonded with intermediate SiO ₂ or indium oxide layers; and relaxed epitaxial growth of InGaAs on a compliant substrate fabricated by epitaxial growth of an AlAs/GaAs structure with the subsequent oxidation of the AlAs. We have developed methods to improve the cleanliness and smoothness of the surfaces of both types of compliant substrates for subsequent epitaxial growth and have successfully produced intact GaAs layers as thin as 30 nm on top of oxidized AlAs in stripes 100µm wide. We have successfully grown GaAs epitaxially over thin layers. So far, however, we have not been able to produce large areas of dislocation-free epitaxial material grown on our compliant sub-(over)				
14. SUBJECT TERMS Compliant substrate, epitaxy, GaAs, InGaAs, Molecular beam epitaxy		15. NUMBER OF PAGES 6		
		16. PRICE CODE		
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT	

strates, in part because lattice-mismatched epitaxy still nucleates poorly on these surfaces.