STUDY OF THE ELECTRONIC SURFACE STATES OF III-V COMPOUNDS AND SILICON

Final Technical Progress Report

1 October, 1978 through 30 September, 1982

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"Studies of the Electronic Surface States of the III-V Compound Semiconductors and Silicon"

Professor W. E. Spicer and Professor I. Lindau, Co-Principal Investigators
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I. Introduction

The work performed over the four year span of this contract comprises a large body of research. Its results are summarized in this document. The volume of work that has been done is significant: it has resulted in 85 publications in the scientific literature, and 6 students at Stanford University receiving their Ph.D. degrees, all under the direction of Professors W. E. Spicer and I. Lindau. The reader is directed to the papers referenced in the "List of Publications" in this report for further detailed information, as well as to the proposals, quarterly reports and semi-annual reports of this contract. The reader is also directed to the proposals and reports of DARPA DAAK-2-74-C-0069 (September 1, 1973 through September 30, 1978); portions of the contract reported on here are, in a scientific sense, a continuation of that older contract.

The technical problem was to study the electronic and atomic structure of the surfaces of several semiconductors. The semiconductors were selected for their technological as well as fundamental importance. The III-V compound semiconductors, especially GaAs, InP, and GaSb, and the column IV semiconductor Si were emphasized, but some work on Ge and HgCdTe was also performed. The predominant methodology used was laboratory experimentation: photoemission spectroscopy excited by synchrotron radiation was utilized heavily, along with angle-resolved photoemission, photoemission excited by conventional ultraviolet and x-ray illumination, low energy electron diffraction, Auger electron spectroscopy, contact potential difference (Kelvin probe) measurements, as well as other techniques which are detailed in the publications. Although our approach was usually fundamental, we were guided by both fundamental and applied questions. In many cases we made important connections between the fundamental and applied. Where possible, we have utilized this knowledge to
advance the development of electronic and opto-electronic devices. Experimental studies were often complemented with theoretical researches, typically by collaboration with leading theoreticians in those fields. Important collaborations which have been stimulated by this research are listed elsewhere in this document.

II. Summary of technical results

- **Schottky barrier formation on the III-V semiconductors.** In contrast to the large amount of theoretical work attempting to explain this in terms of interactions at ideal metal-semiconductor interfaces, we now understand that Schottky barrier formation on the III-V semiconductors is due to Fermi level pinning by defects formed in the semiconductor at the metal-semiconductor interface by the metal deposition. The driving force is the heat of condensation of the metal. In fact, this same mechanism generates interface states at the oxide-GaAs or other insulator-GaAs interface. This Unified Defect Model, which we developed, is now quite widely accepted. It has proven important in III-V semiconductor integrated circuit development. The intrinsic (dangling bond) surface states are moved out of the bandgap of the (110) surfaces of GaAs, InP, and GaSb by atomic rearrangement at the surface. New levels due to atomic defects are created during oxidation or metal deposition. These defect levels influence Fermi level pinning at these metal-semiconductor and insulator-semiconductor interfaces.

- **Attempts to develop GaAs MOS devices on an empirical basis have been expensive and unsuccessful.** Based upon our work we now: (1) can understand why the MOS structure has been more successful on InP and the MES structure has been more successful on GaAs, (2) can correlate the success and failure of MOS and MES technologies on different III-V semiconductor alloys with...
the alloy composition, and (3) can guide future work with scientific engineering rather than empiricism.

- Bonding of column III and V metals on the III-V's. Ga and Al form metallic bonds to the surface, thus these elements form two-dimensional "rafts" which are randomly oriented with respect to the semiconductor surface lattice. Sb forms a directional bond, and an ordered overlayer. This work has implications for understanding epitaxial III-V semiconductor crystal growth. Dynamical LEED intensity calculations were done by A. Kahn of Princeton University. A bonding model was developed based upon the tight-binding calculations of W. Harrison of Stanford University.

Prior to our work it was widely assumed (and predicted by theory) that Al or Ga atoms were bonded to the GaAs (110) surface via As lone pair electrons, or by bridging between As and Ga surface atoms. This sort of bonding, if present, would lead to a large change in surface lattice reconstruction and a distinctive valence band electronic structure. Neither of these effects were observed. Our data shows that there is no major change in the semiconductor surface lattice, and that Al or Ga metallic-like states appear even at submonolayer coverages.

- Preliminary angle-resolved photoemission measurements of the column III and column V metals on GaAs (110). These results helped us in specifying an angle-resolved system for purchase.

- Noble metals (Cu,Ag,Au) on GaAs and InP. (This work was supported for a time by DOE, as well as DARPA and ONR. The DOE support has now been terminated.) Significant intermixing has been found between the metal and semiconductor, even at room temperature. Differences have been found between the different metals and the different semiconductors. In general,
the results are not explained by bulk thermodynamics, and we are developing an understanding of surface thermodynamics.

- **Engineered Schottky barriers.** As one result of the Unified Defect Model, we suggested that we might modify the Schottky barrier height by fabricating diodes with heavily doped semiconductor layers at the metal-semiconductor interface. MBE (molecular beam epitaxy) was used for the device fabrication because it provided good control over the thin, heavily doped semiconductor layers, and because it allowed for in situ metallization which minimized interfacial contaminants.

  Al metal was used on n-type GaAs. The unmodified barrier height was 0.80 eV. p+ interfacial layers allowed us to controllably increase the barrier height up to 1.33 eV (compare $E_{\text{gap}} = 1.42$ eV), and n+ interfacial layers allowed us to reduce the barrier height all the way to ohmic behavior. In all cases there was only minimal degradation of the diode electrical characteristics.

- **Oxygen chemisorption on GaAs(110).** This is important for both fundamental and practical reasons. We are attempting to understand the effect of interfacial oxides which are always present in practical devices. We have developed a new valence band measurement which is sensitive to < 0.001 monolayer of oxygen. The chemisorbed state of oxygen on the GaAs (110) surface consists of an oxygen atom bonded to a surface As as well as an oxygen atom bridging between the As and a neighboring Ga. With unexcited oxygen we observe a 2.9 eV shift of the As 3d core level and an asymmetric broadening of the Ga 3d core level with a shift of no more than 0.7 eV. The first of these corresponds to the theoretical predictions of the Goddard group for an oxygen attached to a surface As without breaking any back
bonds (J. Vac. Sci. Technol. 16, 1178 (1979)). In contrast, we have presented definitive evidence that the model of Brundle et al. which assumes As$_2$O$_3$ plus Ga$_2$O$_3$ clusters is incorrect (J. Vac. Sci. Technol. 16, 1186 (1979)). Our work has included a new high sensitivity valence band spectroscopy, studies of As$_2$O$_3$ valence bands, thermal desorption, and oxygen adsorption on sputtered (disordered, Ga rich) GaAs (110).

- **Room temperature oxidation of As and Ga.** Oxygen exposure on amorphous As results in an oxidized phase having an electronic structure similar to both that of isolated (gas phase) As$_4$O$_6$ molecules and that of crystalline As$_2$O$_3$. We have reported an analysis of the bonding in As oxides. The oxidation of Ga resulted in the formation of Ga$_2$O$_3$. We studied the valence band electronic structure, chemical shifts in the Ga 3d, and bonding in the oxide.

- **Oxygen on GaAs(110).** This work is important for understanding III-V semiconductor passivation. We collaborated with P. Mark of Princeton University on LEED and UPS studies of the order and disorder of the GaAs(110) surface during oxygen exposure. A small amount of oxygen (< 0.1 monolayer) pins the surface Fermi level and disorders the valence band electronic structure. The same pinning energy is observed as for the metals.

- **Oxygen on GaAs versus InP.** The GaAs oxides are unstable, contributing to problems with GaAs MOSFET's. The InP oxide is stable, as only a surface oxide is formed and the InP is not "torn-up". This result, along with our Unified Defect Model, explains the success on InP MOSFET's and GaAs MESFET's, and the failure of GaAs MOSFET's. This work also allows us to understand dark-line defects and device failure in AlGaAs LED's and lasers, and the longer lifetimes of InGaAsP LED's and lasers.
• **Laser enhanced oxidation of GaAs.** In addition to its importance for understanding the oxidation and passivation of semiconductors, our laser enhanced oxidation work has implications for understanding the lifetimes and failure of practical LED's and semiconductor lasers. Our work was the first demonstration of laser enhanced chemisorption of oxygen on a semiconductor surface (GaAs(110)). We observed an increase of the oxygen sticking coefficient of several orders of magnitude under low intensity laser illumination (3 watts/cm\(^2\), 514.5 nm). Our work indicates that this enhancement is due to an increase in the free electron and hole densities at the surface, due to the recombination of photo generated carriers. Prior work showed that the break-up of the \(O_2\) molecule was the rate limiting step. Electrons and holes excited by the laser in GaAs may facilitate \(O_2\) break-up and chemisorption. We found the same oxygen surface chemistry with or without laser irradiation, however the laser enhanced results have forced a modification of what we had previously interpreted as the saturated oxygen coverage.

• **NEA (negative electron affinity) photocathodes.** Much of this work revolves around understanding the oxidation of Cs on GaAs. This is important fundamentally because of the very ionic bond formed between Cs and oxygen, and the different oxidation states of oxygen (we have observed four). Cs on GaAs is the extreme of a very electropositive element and one which interacts much more atomically than metallically. Understanding interface electrostatics for the Cs-oxygen-GaAs system then, has provided much insight into oxygen-metal and metal-semiconductor interactions in general. We have made key connections between this work and practical NEA and field assisted photocathodes. These contributions include improvements in production, and the opening of possibilities for
making stable cathodes at longer wavelengths and for reducing the threshold of response (using III-V semiconductor alloys). All these topics will be amplified on in the next few paragraphs.

- **Oxygen on Cs-GaAs.** One monolayer of Cs increases the oxygen sticking coefficient by $10^9$. A chemical shift of As to higher binding energy indicates the oxygen is bonding first to surface As atoms and secondly to Ga atoms, not to the Cs as was expected. The lack of downward shifts of Cs core levels confirms that Cs-oxides are not formed, although suboxides may be present. We have identified three of the four oxidation states of the oxygen atoms as $O^{-2}$, $O_2^2$, $O_2^-$. These assignments are based upon comparisons of the multiplet structure in our photoemission spectra with the multiplet structure of free oxygen ions, and comparisons of our measured binding energies with calculated values. We now understand that the difficulty with many narrower gap III-V semiconductors may be the formation of interfacial bulk oxides with the semiconductor.

- **NEA activation process.** We successfully activated p-GaAs to the NEA condition. The GaAs-O-Cs dipole plus a polarization of the Cs$^+$-$O^{-2}$-Cs$^+$ sandwich layer causes the NEA condition. Understanding that the GaAs-O layer lies beneath the Cs-surface oxygen layer (a possibility overlooked in previous models), points out that the processing of practical devices could be modified to minimize this contamination and the harmful heterojunction it creates. Third generation night vision goggles (developed under this contract) have proven superior to second generation goggles using S-20 photocathodes. The identification of the GaAs-O bonding layer explains the better yield obtained with the two step activation process, compared with the single step "yo-yo" process. OH adsorption may be one degradation
mechanism.

- **HgCdTe materials studies.** While this effort has essentially been financed in a joint program with the Santa Barbara Research Corporation, under this DARPA contract we covered some preliminary expenses in early 1980. These included obtaining a visiting professor, training a student, preliminary CdTe investigations, and organizing a HgCdTe conference.

- **Clean Si(111) surface.** We have studied the 2 x 1 structure obtained on cleaving and the 7 x 7 reconstruction which occurs after annealing. Several models for the 7 x 7 surface have been proposed. Our work is in support of the defect-type model.

- **Si(111) and oxygen.** The cleaved 2 x 1 surface transforms to the 7 x 7 after annealing above 200°C. Understanding these reconstructions is important to understanding covalent bonding at the surface. A direct determination of the structure of the 7 x 7 surface has proved difficult. We have studied this by looking at the oxygen adsorption properties. Our experiments on the oxidation of Si 2 x 1 narrowed the range of possible Si-0 bonding configurations. This result has applications for a fundamental understanding of Si surfaces, and also for the initial stages of oxidation which are so important in VLSI applications.

  The room temperature adsorption of oxygen on the 2 x 1 and 7 x 7 reconstructed Si(111) surfaces was shown by core level shifts and sticking coefficients to proceed differently, indicating the different natures of these surfaces. As indicated above, our results show unambiguous support for the defect-type model of the 7 x 7 surface. The configuration of oxygen on the 2 x 1 surface was studied by examining the effects of annealing on the Si 2p core level shifts.
Silicides: Overview. The ultimate objective of this work is to contribute to IC technology. We are coordinating our efforts with other groups at Stanford. Professor R. Helms has JSEP and DARPA funds to investigate the effects of impurities (O, C, other) on transition metal silicide Schottky barriers. His studies of the (100) surface have included Auger electron spectroscopy with sputter profiling, ultraviolet photoemission spectroscopy, I-V and C-V measurements, heat treatments, and both in situ fabrication as well as measurements on practical devices. He has had important interactions with IBM, E. Fishkill. Professor J. Plummer is studying transition metals on poly Si for high temperature gates and interconnects.

Silicides: Transition and noble metals on Si (and, to a lesser extent, Ge-metal contacts). The focus here is to understand the physics of the silicide formation and the electronic and chemical structure at the interface, including the hybridization of the s, p, and d orbitals of the metal and Si, and intermixing and compound formation. We are in the process of obtaining a sufficiently large body of data for metals on Si to be able to begin seeing systematics and making generalizations. Most (and probably all) of the Si(111) surface states, which are visible near the valence band maximum, are removed by a wide range of metals (transition, group III, Cs) and typical contaminants (oxygen). Therefore, there is no evidence that native Si surface states play a role in Schottky barrier formation. The goals of this silicide work include the development of silicides as Schottky barriers, MOS gates, and interconnects with sufficiently high conductivity and high temperature stability.

We have emphasized Si contacts with Ni, Pd, Pt, Ag, Cu, and Au, and Ge contacts with Ag. We have measured the abruptness of the interfaces, the
nature of the Si-metal bonding, and the effect of oxide layers and impurities. The (often implicit) assumption in prior attempts to model the Si-silicide interface is that of an abrupt interface. We have seen that this is incorrect. One must consider metal atoms which have moved into the Si. The degree of intermixing which we have found, in order of increased intermixing on Si, is: Ag--Cu--Ni--Pd--Au. This has device implications; for example, a thin Ag layer followed by Pd may insure an abrupt interface and stable Pd$_2$Si silicide.

- **Pd on Si(111).** We have formed these contacts in ultrahigh vacuum with metal layers from 1 Å to 100's Å thick. Nearly stoichiometric Pd$_2$Si is formed at room temperature for monolayer coverages. Elevated temperatures are required to form the bulk silicide. Some intermixing occurs even at 77 K. Auger electron spectroscopy depth profiles indicate room temperature Pd-Si intermixing over ~1100 Å. Once the bulk silicide is formed, a graded interface for a few atomic layers is present between the Si and Pd$_2$Si. Pd reduces the native Si oxides, with oxygen appearing on the metal surface. The Cooper Minimum method which we developed (in photoemission spectroscopy utilizing synchrotron radiation) allowed us to separate the valence band contributions. The Pd d bands are filled, and pulled 2 eV towards higher binding energy in Pd silicide with respect to pure Pd metal. Thus, there is very little charge transfer from Si to Pd. This work has benefited from collaborations with I. Abbati and L. Braicovich of Milan, Italy.

The reactions between Pd and Si were followed in ultraviolet photoemission spectroscopy. Changes in the valence band density of states indicated a break-up of the Si tetrahedral bond. We have strong evidence against the existence of Si-Pd$_2$Si interface states suggested by others.
In soft x-ray photoemission spectroscopy, we observed a gradual turn on of asymmetric Doniach-Sunjic broadening of the Si 2p core level due to Si moving into metallic surroundings. This can be correlated with the conductivity of the silicides. Partial funding was provided under this contract for Stanford University Professor S. Doniach for collaborative efforts on the above work.

- **Pt-Si(111) interface.** Use of the Cooper Minimum effect allowed us to distinguish changes in Si and Pt valence electronic structure and, hence, to detect changes in Si and Pt bonding during the formation of the contact. The first Pt deposited on the clean Si(111) surface (> 0.07 monolayer) disrupts the Si surface. Moderate Pt coverages (2-10 monolayers) result in a silicide-like surface layer. Thicker Pt coverages (40 monolayers) still show significant Si in the Pt metal, indicating the very strong interactions and intermixing that occurs between Si and Pt. All of the above results are for Pt deposition at room temperature. The Si and Pt core levels shift in opposite directions, because the Si-Pt bond is partially ionic and charge is transferred between the atoms.

- **Ag on Si(111).** In contrast to Pd and Pt which form silicides, Ag forms a eutectic. Of the metals we have studied, only Ag shows no extended intermixing at room temperature. Prior models of this interface were based on the growth of Ag without any Ag-Si bonding. Our data show strong Ag-Si interactions and breaking of the tetrahedral coordination in the Si at room temperature. However, by a comparison of room temperature and liquid nitrogen temperature results, we determined that little or no intermixing takes place at liquid nitrogen temperature.
• **Enhancement of Si oxidation when thin Ag layers are present.** One of the crucial problems in the field of semiconductor d-metal interfaces is to establish if intermixing takes place, or if the interface is atomically sharp so that it can be used as a test case for available theories which assume such a simple geometry. The significant enhancement of Si oxidation when thin Ag layers are present indicated that Ag breaks the sp$^3$ configuration of Si atoms on the Si(111) surface, leaving these atoms more vulnerable to oxygen attack. This result rules out one model that had been proposed for a Ag monolayer on Si, that of Ag adsorbed on top of Si with an atomically abrupt interface.

• **Ge(111)-Ag interface.** A strong chemical interaction takes place in the first two to three monolayers of Ag evaporated on a clean cleaved Ge(111) surface at room temperature. Ag is incorporated into the Ge surface lattice. Further Ag deposition above this coverage results in Ag island formation by the Stransky-Krostanov mechanism. Our work was the first to detect intermixing at the Ge-Ag interface.

The above summary touches on only the highlights of a large and diverse research program. As mentioned earlier, interested readers will find much more information in the publications listed elsewhere in this document, as well as in the proposals and reports of this contract. Only a few of our beneficial collaborations were mentioned above. They are also listed elsewhere in this document.

Many of the experiments described herein were performed at the Stanford Synchrotron Radiation Laboratory which is supported by the National Science Foundation under Grant No. DMR77-27489 in cooperation with the Stanford Linear Accelerator Center and the Department of Energy.
III. Implications for further research

The work performed under this contract raises new questions, and suggests new areas of investigations which are likely to be profitable in both applied and basic fields. Some of these topics are already receiving attention under current funding of our contract no. N00014-83-K-0073 (DARPA and ONR), which is essentially a combination and continuation of contract no. N00014-79-C-0072 (DARPA through ONR) and contract no. N00014-75-C-0289 (ONR).

- The question of the mechanisms by which Schottky barrier heights to the III-V semiconductors are determined has been advanced considerably during the last five years by the introduction, application, and development of our group's Unified Defect Model. This model, far better than any other, explains all the available data on surfaces and practical devices. Very few groups presently take issue with the Unified Defect Model. Most work is now devoted to exploring just how extensive its range of validity is, calculating second-order effects which modify slightly the proposed pinning positions, applying the model to practical devices (either their design or detailed modeling), etc. We are looking into the influences of different metals, different metal deposition conditions, the presence of contaminants, and different semiconductor surfaces and surface treatments. Some of the debate which persists deals with the relative effects of band bending and changes in electron affinity and work function. We intend to use photoemission spectroscopy and contact potential difference measurements to obtain additional information on these subjects, and incorporate what we learn into our theories of Schottky barrier formation.
• Intermixing and interdiffusion of metals on semiconductors, especially the noble metals Cu, Ag, and Au. Included: temperature effects, Fermi level pinning, Schottky barrier and ohmic contact formation.

• Ohmic contacts.

• Studies of (100) and (111) surfaces (surfaces other than the cleavage surface). This will require us to develop new preparation and passivation techniques.

• Closer coupling with MBE (molecular beam epitaxy) and other novel materials synthesis techniques.

• Laser enhanced oxidation, including low temperatures, H2O oxidation, and the oxidation of new semiconductors. Goals include MOS structures, understanding oxygen chemistry on semiconductors, and investigating photon-semiconductor interactions.

• When high fluxes and improved energy resolution are available from the new DOD/Xerox/NSF beam line under construction at SSRL, we will study surface versus bulk chemical shifts, oxidation, and other projects.

• Hydrogen and halides on III-V semiconductor surfaces, with and without oxygen. Applications to practical MIS/MOS devices.

• Further investigations of column III and V elements on the III-V semiconductors, including angle-resolved photoemission, temperature effects, and column III and V elements simultaneously to learn about epitaxial growth mechanisms. Goals are an improved understanding of the fundamental physics and chemistry of III-V crystal growth, temperature dependences,
growth rates, and dopant incorporation.

- Angle-resolved photoemission and LEED (low energy electron diffraction) to investigate domain structures on cleaved surfaces. Many applications.

- SEXAFS (surface extended x-ray adsorption fine structure) on the new DOD/Xerox/NSF SSRL beam line to study the surface geometry of adsorbed species and reconstruction of the clean surface.

- Silicide program expanded to include additional metals V, Nb, Ta, In, Os, and contaminants. We will attempt to develop models and unify the data.

- Device measurements on metal-semiconductor contacts will be incorporated increasingly into our surface physics studies.

- Engineering of interfaces, including techniques to heal semiconductor defects, and the introduction of dopants to modify Fermi level pinning and study interface electrostatics.

- III-V ternary and quaternary semiconductor alloys.

- Interface states at III-V semiconductor heterojunctions.

- Surface chemistry and surface thermodynamics.
IV. Important collaborations fostered by this research

The researches described in this document have benefitted from collaborations with scientists throughout the world. In addition to the formal collaborations, our experimental work and modeling have stimulated much theory and some experiment in labs elsewhere. The following list includes only some of the groups with whom we have collaborated, or whose work has been stimulated (at least in part) by our own.

Professor W. Harrison, Stanford University
Professor L. Braicovich and group, Milan, Italy
Professor G. Ottaviani and group, Italy
IBM, E. Fishkill
Professors P. Mark (deceased) and A. Kahn, Princeton University
Xerox, Palo Alto Research Center
Professors McGill and Goddard, Cal Tech
Professor M. Cohen, U. C. Berkeley
Professor Joannopoulos, M.I.T.
Dr. A. Zunger, SERI
Professor Gatos and coworkers, MIT
Professor Monch and coworkers, University of Duisberg, West Germany
Naval Oceans Systems Center, San Diego
Professor J. Dow, University of Illinois
V. Graduate students who received Ph.D. degrees under this contract

The students listed here were specifically supported by this contract and received Ph.D. degrees during the period of this contract. However, there are several other students in Professors Spicer and Lindau's group at Stanford who were not doing thesis work on semiconductors, but benefitted from working closely with these students. This is most dramatically seen in the many students who did their thesis outside of the semiconductor field, but are nonetheless employed in leadership positions in the semiconductor and electronics industries today.

This is not an inclusive list of all who contributed to this work. Consult the list of publications for a complete list of contributors.

Dr. Chung-Yi Su
Dr. Patrick W. Chye
Dr. C. Michael Garner
Dr. Perry Skeath
Dr. Jeffrey N. Miller
Dr. Se-Jung Oh
VI. Papers Published During the Period: October 1, 1978 through September 30, 1982

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Minimum Al$_0.5$Ga$_0.5$As-GaAs Heterojunction Width determined by Sputter-Auger Techniques, C.M. Garner, C.Y. Su, W.E. Spicer, P.D. Elwood, D. Miller, J.S. Harris, Appl. Phys. Lett. 34(9), 1 May 1979


The Surface Electronic Structure of 3-5 Compounds and the Mechanism of Fermi Level Pinning by Oxygen (Passivation) and Metals (Schottky Barriers), W.E. Spicer, P.W. Chye, C.M. Garner, I. Lindau, and P. Pianetta, Surface Science 86, (1979), 763-788


