### FY 1991 Measurements and Characterization Branch Annual Report

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### 1.0 INTRODUCTION

### 1.1 Introduction

The Measurements and Characterization Branch actively supports the advancement of DOE/NREL goals for the development and implementation of the solar photovoltaic technology. The primary focus of the laboratories is to provide state-of-the-art analytical capabilities for materials and device characterization and fabrication. The branch houses a comprehensive facility that is capable of providing information on the full range of photovoltaic components. A major objective of the branch is to aggressively pursue collaborative research with other government laboratories, universities, and industrial firms for the advancement of photovoltaic technologies. Members of the branch disseminate research findings to the technical community in publications and presentations. Along with detailed accounts of the individual group's accomplishments over the past year, this report contains five appendices that detail the following information: the capabilities and technical qualifications of the staff (Appendix A); the outside laboratories with which members of the branch have collaborated in the past year (Appendix B); technical publications that have emanated from the branch in the past fiscal year (Appendix C); major equipment and capabilities of the various groups (Appendix D); and a partial list of acronyms used in this report (Appendix E).

### 1.2 Organization

- The Branch has undergone one organizational change since the previous reporting period. The Device Development Group and the MBE facility were moved into a new branch that was created in the new Photovoltaics Division. The new branch is entitled the PV Device Development Branch and it includes the MBE facility. This group is included in this report as part of the Measurements and Characterization Branch..
- The Measurements and Characterization Branch encompasses nine coordinated research groups, providing integrated research and development that covers all aspects of photovoltaic materials/devices characterization, fabrication, and modeling. A summary of the capabilities of each group is given in Appendix D. The support research and services available are explained briefly in the following paragraphs.

### **Surface and Interface Analysis**

 The work of this group entails determinations of the chemical, compositional, and microelectrical characteristics of materials, surfaces, and device interfaces with extremely high depth and spatial resolution. Techniques include secondary ion mass spectrometry (SIMS); Auger electron spectroscopy (AES); scanning Auger microscopy (SAM); X-ray photoelectron spectroscopy (XPS); electron-energy loss spectroscopy (EELS); electron-stimulated desorption spectroscopy (ESD); electron-beam-induced current and voltage (EBIC and EBIV); Auger voltage contrast (AVC); high-resolution elemental and ionic mapping, including volume-indexing; cooperative soft x-ray synchrotron-source analysis; scanning tunneling microscopy (STM); and molecular beam epitaxy (MBE).

### **Materials Characterization**

This group employs advanced microscopy and microanalytical techniques in the determination of crystallographic, defect topographic, compositional, and microelectrical properties of photovoltaic materials and devices. These include electron probe microanalysis (EPMA) with energy dispersive and wavelength dispersive spectroscopy (EDS and WDS); scanning electron microscopy (SEM) to liquid helium temperatures; scanning transmission electron microscopy (TEM and STEM); voltage contrast; electron-beam-induced current and voltage characterization; electron diffraction; electron channeling; image analysis; X-ray diffraction; X-ray fluorescence; micro-cathodoluminescence; cooperative high-resolution interface analysis using high-voltage electron microscopy; and complete sample preparation facilities, including ion etching with integral SIMS analysis.

### **Device Development**

 This group provides modeling and fabrication support for the development of cell structures, including diagnostic device preparation, optical conducting film fabrication and studies, thin-film and space cells, and computer analysis and design. Capabilities include precision sputtering equipment for deposition of optical films, antireflection coatings, metallizations, transparent conductors, etc.; research clean room facilities; III-V cell-processing capabilities; photolithography and mask design and fabrication; InP-alloy metal-organic chemical vapor deposition (MOCVD); optical and electrical characterization for films and coatings; plasma deposition; photoconductivity and ellipsometry; contact resistance determinations; and computer modeling programs for optical coatings and solar-cell design.

### **Electro-Optical Characterization**

 The work of this group involves determination of the electrical and optical properties of materials and solar cells and laser spectroscopies, including photoluminescence and specialized determinations of minoritycarrier properties. Capabilities and techniques include capacitance-voltage, conductance-voltage, and temperature dependences; Hall effect/van der Pauw measurements; laser scanning; deep-level transient spectroscopy (DLTS); photoluminescence (PL); minority-carrier lifetime (to the picosecond range) and diffusion-length measurements; determination of minority-carrier mobility by time-of-flight (TOF) measurements; picosecond laser spectroscopies; optical spectrophotometry; and ellipsometry. In addition, new facilities have been developed for the electrical characterization of high temperature superconducting materials. This includes measurements of resistance versus temperature (R vs. T), Meissner mutual inductance (MMI), and critical current.

### **Cell Performance**

The work of this group includes calibrating and maintaining primary photovoltaic reference cells; performing efficiency measurements under standard reporting conditions; calibrating secondary reference cells for the photovoltaic community; evaluating photovoltaic device parameters. Capabilities and techniques include current-versus-voltage measurements as functions of temperature, spectral and total irradiance; spectral response as functions of temperature, voltage bias, and spectral and total irradiance; laser photo response mapping; and detection of measurement-related artifacts present in other current-versus-voltage measurement systems.

### Surface Interactions, Modification, and Stability

• The work of this group involves the fabrication, modification, and characterization of surfaces and interfaces to better understand their properties and structures. Capabilities include surface spectroscopies (ion scattering spectrometry (ISS), XPS, SIMS, fast atom bombardment (FAB), AES, and SAM); Fourier transform infrared spectroscopy (FT-IR); and contact-angle measurements, ultramicrogravimetry, and quartz crystal microbalance measurements. The group is able to perform reactions in the high-pressure chamber on the LHS-10 system, or in a solar furnace. In addition, the group has experience in the preparation and characterization of polymer films and organized molecular assemblies (OMA).

### FTIR Spectroscopic and Research Center

This is an Institute-wide service center providing Fourier-transform infrared (FTIR) spectroscopic analysis and experimental design for determination of the covalent or lattice-bonding structure of materials, the effects of chemical reactions on materials, as well as the chemical composition and sub-ppm levels of impuri-

ties or dopants in samples. Techniques include: high-resolution, mid-infrared FTIR spectroscopy in transmittance, specular reflectance, diffuse reclectance, and attenuated total reflectance (ATR) sampling modes using 5 cm², 0.5 cm², and fiber optic ATR probes, along with environmental control for in-situ analysis and measurement of reaction kinetics for most sampling modes; FT-Raman spectroscopy in both reflective and refractive modes; and FTIR microscopy for obtaining midinfrared transmittance or reflectance spectra of sample areas as small as 10 X 10  $\mu m$ , and farinfrared spectroscopy (650-50 cm²-1) with transmittance and diffuse reflectance sampling modes.

### 1.3 Summary

These laboratories have been established to support the U.S. Photovoltaic Program. Researchers involved in the Program, as well as the photovoltaic industry and international research groups, are invited to utilize and interact with the Measurements and Characterization Branch for the advancement of their technologies.

### 2.0 SURFACE AND INTERFACE ANALYSIS

### 2.1 Research Staff

A.J. Nelson, Senior Scientist and Group Leader P. Sheldon, Senior Scientist S.E. Asher, Senior Scientist A.B. Swartzlander-Franz, Associate Scientist D.W. Niles, Staff Scientist L.L. Kazmerski, Principal Scientist

### 2.2 Introduction

The chemical, compositional, and microelectrical properties of photovoltaic materials and devices are analyzed in these laboratories. Staff researchers support both internal and external researchers and provide timely analysis on fundamental compositional problems that limit the performance and operating lifetime of specific material systems and photovoltaic cells and modules.

The Surface and Interface Analysis Laboratories specialize in analyzing individual layers of a photovoltaic material, device, or module. Examining materials a monolayer at a time, researchers determine the types of impurities present, their chemical or electrical makeup, and/or their relative or absolute concentration and location. The staff correlates the presence of impurities with the electrical properties and performance of the cell or module.

Using sophisticated surface microprobe and epitaxial-growth systems, the staff can conduct complementary compositional and electrical analyses, with multiple techniques applied concurrently or consecutively to the same area, as well as grow diagnostic device structures. Additionally, research scientists have extensive experience in photovoltaic device physics, epitaxial growth, and surface analysis, providing unique capabilities in the area of device characterization.

Recent activities in this research area include continued development of Auger line-shape analysis for determining chemical bonding at surfaces and grain boundaries in photovoltaic materials. In addition to the in-house-photoemission activities, several experiments were completed at the Synchrotron Radiation Center. Specifically, (i) the band diagram for the Cd(In)S/CuInSe<sub>2</sub> heterojunction and (ii) contact formation on CuInSe<sub>2</sub> were directly determined from these photoemission measurements. Additionally, photoemission investigations of ECR plasma-modified surfaces of semiconductors was initiated. SIMS was used to characterize the light-induced diffusion of H in a-Si:H films. Atomic-level scanning tunneling microscopy (STM) was

used as well to study semiconductor surfaces and interfaces, imaging and analyzing grain boundaries, and characterizing defects in photovoltaic materials.

The molecular beam epitaxy (MBE) staff utilizes the controlled-growth environment and in-situ diagnostics available with MBE to study problems relevant to photovoltaics. This work, performed in collaboration with both internal and external researchers, falls into two categories. The first of these is to support the Measurements and Characterization Branch, by designing and growing electrical and structural diagnostic devices for instrument qualification. For example: (i) to provide electrically characterized standards necessary for quantitative AES and SIMS analysis; (ii) to provide precisely grown layered structures for sputter rate determination and depth-resolution qualification for AES and SIMS analysis; and (iii) to provide specialized structures for instrument development (DLTS, CV, and minority carrier lifetime measurements). The second category involves internal research, which over the past year has focused on RHEED intensity oscillation instrument development and using novel growth techniques to optimize minority carrier lifetimes in AlGaAs/GaAs DH structures.

### 2.3 Equipment/Analysis Development

### (ECR) Plasma Modification with in-situ Analytical Capabilities

- A large preparation chamber has been assembled and attached to the PHI 550 XPS system. This chamber houses two effusion cell sources on a 15.24 cm conflat flange, an ECR plasma source, and a small metal evaporator. The substrates can be heated up to 1,000°C during growth and/or plasma processing. The chamber was designed to allow the grown/processed samples to be transfered in vacuum for in-situ analysis (XPS, UPS, AES, EELS).
- Excitation of the Compact ECR source is by 2.45 GHz microwave energy (10-250 watts) coupled into the source through a vacuum window. There are no filaments, cathodes, or grids to contaminate the system or process. A strong axial magnetic field (875 gauss) is used to create electron cyclotron resonance, to promote efficient coupling of the microwaves to the plasma, and to minimize plasma contact with the interior stainless-steel walls of the source. Plasma densities of 10<sup>11</sup> to 10<sup>12</sup> cm<sup>-3</sup> are obtained, giving high ion currents and good gas efficiency. The ions produced by an ECR plasma have energies of 10 to 25 eV, at pressures in the 10<sup>-3</sup> to 10<sup>-4</sup> torr range, which is much less than the ion energies produced by an RF plasma. Further reduction of these ion energies is possible through substrate biasing.

 The primary application of this preparation chamber will be for the growth of ordered/disordered II-VI semiconductor materials (CdTe, CdS, ZnTe, CdZnTe) and interfaces (e.g. heterojunctions, contacts) and for fundamental studies on plasma processed surfaces of III-V, II-VI and I-III-VI<sub>2</sub> semiconductors for improving device performance.

### RHEED Intensity Oscillation Frequency Domain Flux Transient Analysis

- Significant progress has been made in the development of NREL's reflection high energy electron diffraction (RHEED) intensity oscillation hardware and RHEEDfrequency domain analysis software (initially described in the FY1990 annual report). This system is used to measure growth rate and alloy composition in-situ with extreme accuracy on a near-real-time basis. Earlier versions of this system were only capable of monitoring flux rates for the first 100 Å of grown material. The most recent generation of the design has successfully measured the flux rate for as much as 7,500 Å of grown material. This represents a significant improvement in the technique and makes it useful for monitoring heterojunction formation as well as the growth of ultrashort period superlattices. This work has resulted in a record of invention and has generated significant industrial interest. A cooperative research and development agreemtn (CRADA) is presently being negotiated to commercialize this system.
- Hardware and software modifications to the MBE/MEE control system will now allow for simultaneous shutter control and RHEED-intensity oscillation data acquisition. The addition of this feature is significant, because it will allow for in-situ observation and recording of surface and interface characteristics. By monitoring the intensity of the specularly reflected diffraction beam, during heterointerface growth, both qualitative and quantitative information on surface/interface roughness can be obtained. This information can then be used to tune growth parameters (i.e., growth temperature, growth interrupts, alloy composition, etc.) to optimize interface quality. The design for the MBE/ MEE-source flange and control system has been distributed to several research groups that have expressed interest.

#### Quadrapole Mass Spectrometer Data Acquistion System

 Hardware and software development is complete for a computer controlled quadrupole mass spectrometer data acquisition system for the UTI 100C. With this system, the quadrupole can operate in a mass scan, peak scan, or peak monitor mode. In the mass scan mode, the system acquires a full atomic mass unit (AMU) scan, collecting up to eight decades of information in 1/10 the time previously required. In the peak scan mode, the system can identify the intensity of preselected peaks and display them in a histogram plot with tabular data. Finally, in the peak-monitor mode, the system can monitor the peak intensities of up to 8 AMUs as a function of time. In all modes, the data can be acquired with the electron multiplier or Faraday cup, smoothed, displayed in a log or linear format, and rescaled. Prior to the development of this system, the UTI 100C could only operate in the mass scan mode with a fixed range. The addition of this software greatly enhances the capabilities of the UTI 100C and will be an invaluable addition to the MBE. This hardware/ software package has resulted in a record of invention and is being pursued by a commercial company interested in licensing this technology.

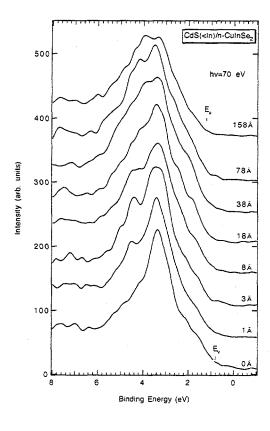
### 2.4 Major Accomplishments: Photovoltaics

### Synchrotron Radiation Photoemission Study on the Effect of In Doping on Cd(In)S/CuInSe<sub>2</sub> Heterojunction Formation [46,47]

Synchrotron radiation soft X-ray photoemission spectroscopy was used to investigate the development of the electronic structure at the CdS(In)/CuInSe<sub>2</sub> heterojunc-tion interface. In-doped CdS overlayers were deposited in steps on single-crystal n-type CuInSe<sub>2</sub> at 250°C. Photoemission measurements were acquired after each growth in order to observe changes in the valence band electronic structure as well as changes in the In 4d, Se 3d, Cd 4d and S 2p core lines. The valence band discontinuity for this heterojunction was experimentally determined as a function of In-dopant concentration and a heterojunction-band diagram for CdS(In) on single-crystal CuInSe<sub>2</sub> was constructed, thus explaining numerous factors affecting the ultimate performance of these single-crystal devices (Figure 2.1). The results again show that the Katnani-Margaritondo method is unreliable in determining offsets for heterojunctions where significant Fermi level pinning may occur and where the local structure and chemistry of the interface depends strongly on the specific heterojunction.

### Photoemission Study on the Formation of Mo Contacts to CulnSe, [44]

Synchrotron radiation soft X-ray photoemission spectroscopy was used to investigate the development of the electronic structure at the Mo/CuInSe<sub>2</sub> interface.



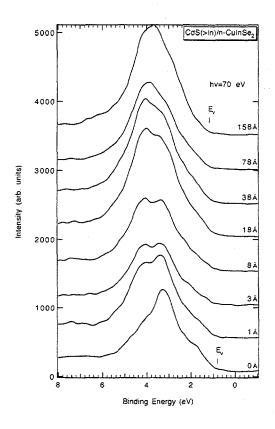


Figure 2.1

Mo overlayers were e-beam deposited in steps on single-crystal *n-type* CulnSe<sub>2</sub> at ambient temperature. Photoemission measurements were acquired after each growth in order to observe changes in the valence-band electronic structure as well as changes in the In 4d, Se 3d, and Mo 4d core lines. Photoemission measurements on the valence band and core lines were also obtained after annealing. Results indicate that the Mo reacts with the CuInSe, during deposition resulting in the formation of In islands and a Mo-Se interfacial phase (MoSe<sub>2</sub>,  $E_g$ =0.2 eV). Results also show that the Mo/n-CulnSe<sub>2</sub> interface is essentially ohmic with a maximum possible Schottky barrier height  $j_b$  of  $\leq 0.2$ eV. This investigation implies that the Mo/CulnSe<sub>2</sub> interface does not consist of a sharp boundary between the metal and semiconductor as historically portrayed, but rather consists of an extended region (≈100 Å) of reacted and/or interdiffused species.

### Photoemission Characterization of the H<sub>2</sub> Plasma-Etched Surface of InP [45]

Synchrotron radiation soft X-ray photoemission spectroscopy was used to characterize the surface chemistry of InP before and after exposure to a H<sub>2</sub> plasma. The low power H<sub>2</sub> plasma was generated with a commercial electron cyclotron resonance (ECR) plasma source using a mixture of H2 and Ar, with the plasma exposure being performed at ambient temperature. Plasma species were idenified with quadrupole-based mass spectrometry and optical emission spectroscopy. Photoemission measurements were acquired after each plasma exposure in order to observe changes in the valenceband electronic structure, as well as changes in the In 4d and P 2p core lines. Quadrapole mass spectrometry (QMS) results showed the presence of  $H^+$ ,  $H_2^+$ , and  $H_3^+$ species during ECR plasma exposure. The fluorescence spectra showed features due to emission from atomic H and from molecular hydrogen dissociation. Photoemission results indicate that the surface of this p-type InP is actually n-type and that the H<sub>2</sub> ECR plasma, in its present configuration, totally reduces the InP surface to metallic In (Figure 2.2).

#### **Molecular Beam Epitaxy**

 Real-time RHEED analysis has been used as an in-situ probe, to investigate the quality of AlGaAs/GaAs interfaces grown by MBE. Double heterostructure (DH) devices have been evaluated using RHEED and time resolved PL measurements. The DH structures were grown at low temperatures (≈ 600°C), a regime which generally yields poor-quality AlGaAs/GaAs interfaces but is convenient for conventional GaAs growth. We find that thick AlGaAs barrier layers, grown without

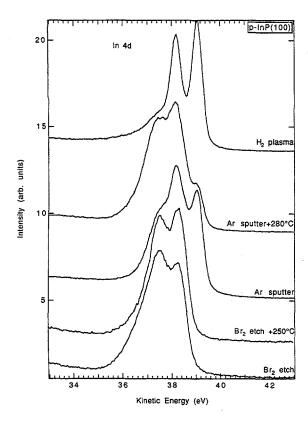


Figure 2.2

interruption, yield poor-quality surfaces that are unable to support RHEED oscillations. DH structures grown under these conditions have short lifetimes, on the order of 7 ns, with correspondingly high interface recombination velocities (see, for example, Figure 2.3, curve a). Conversely, thin barriers (~ 100 Å), employing short-growth interrupts, show strong RHEED oscillations throughout both AlGaAs/GaAs interfaces, indicating superior surface quality. Time-resolved PL measurements support this finding, indicating that thinbarrier DH structures have improved interfaces, with significantly longer lifetimes (~ 90 ns), and reduced interface recombination velocities (see, for example, Figure 2.3, curve b). These initial results indicate that RIO analysis will serve as a valuable in-situ tool for optimizing AlGaAs/GaAs interface quality.

Time-resolved PL measurements of MBE-grown AlGaAs/GaAs DH devices have revealed that the minority carrier lifetime in this material is limited by the GaAs bulk lifetime. Exceptionally low interface recombination velocities (as low as 50 cm/s) have been measured for DH structures grown at low substrate temperatures (≈600°C). However, these structures exhibit poor bulk lifetimes with values two orders of magnitude lower than the theoretical limit. SIMS measurements revealed that the GaAs active layer is unintentionally doped with Al (≈1x10<sup>18</sup> cm<sup>-3</sup>), even though the Al oven

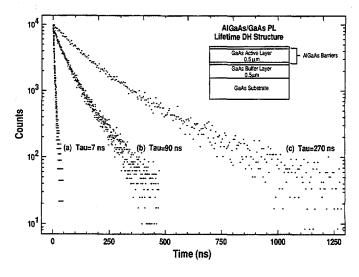


Figure 2.3

does not have a line of sight to the substrate when the shutter is closed. In order to determine whether the Al doping was adversely affecting the minority carrier lifetime, structures were grown in which the Al oven temperature was ramped down during the growth of the GaAs active layer. This resulted in a three-fold increase in the minority carrier lifetime (see Figure 2.3, curve c). These results indicate that the Al doping severely degrades the bulk lifetime of the DH structure and should pave the way for additional improvements.

### **Secondary Ion Mass Spectrometry**

- Secondary ion mass spectrometry (SIMS) was used to study Cr diffusion in c-Si, in collaboration with J.P. Kalejs from the Mobil Solar Energy Corporation. During the production of photovoltaic devices, wafers are subjected to solid source-diffusion of phosphorus (P). This process may have the ability to getter some impurities from the Si during the in-diffusion of the P-rich nlayer. Silicon wafers were implanted with 52Cr at a dose of 10<sup>13</sup> at/cm<sup>2</sup>. Pieces of the original implanted material were then subjected to solid-source in-diffusion of phosphorus (P) during a 30 min anneal at 900°C. In one of the samples, the resulting phosphorosilicate glass (PSG) was etched away to reveal the crystalline Si surface. Depth profiles were obtained for Cr and P. The profiles revealed that up to 95% of the implanted Cr was gettered by the PSG layer and then removed when this layer was etched away. Modelling of the Cr diffusion process is to be performed at Mobil, and other elements are being studied by SIMS.
- Secondary ion mass spectrometry was used to examine amorphous silicon for thermal and light-induced deuterium (D) diffusion, in collaboration with H. Branz of

the Materials Science Branch. This study used D sandwich layers (a-Si:H/a-Si:H:D/a-Si:H/c-Si) that had been subjected to thermal annealing or a combination of thermal annealing and light soaking with red or white light. Diffusion measurement was initially hindered by inaccuracies in the measurement of sputtered crater depths, which lead to large errors (sometimes > 20 nm) when the depth profiles were superimposed. It was found, however, that by using the oxygen signal from the D layer as an internal marker for profile rescaling, the precision could be improved to < 2 nm for multiple profiles from the same sample and < 3 nm for profiles from different samples. This extraordinary reproducibility was obtained by using two simple fitting parameters -- an offset and a depth scale adjustment. In most cases the values of the parameters were < 0.5 nm, well below the error of the measured crater depths of  $\pm 7$ nm. The ability to exactly overlay profiles from different samples allowed quantitative determination differences as small as 14 nm between the diffusion profiles. It will be possible to extend this method to other SIMS analyses of diffusion.

### 2.5 Future Work

- Collaborative research will continue with staff at the Synchrotron Radiation Center, University of Wisconsin-Madison, utilizing high-resolution photoemission techniques to characterize the ZnSe/CulnSe<sub>2</sub> ZnSe/ CdTe heterojunction interfaces.
- Research will continue on characterizing the effects of plasma processing for the purpose of modifying the electronic properties of II-VI and III-V surfaces.
- Research will begin on the growth and in-situ characterization of CdTe, ZnTe, and CdS in an effort to understand order/disorder phenomena and to develop processes necessary for optimum II-VI device performance.

### 3.0 MATERIALS CHARACTERIZATION

### 3.1 Research Staff

M.M. Al-Jassim, Senior Scientist and Group Leader M.H. Bode, Research Associate K.M. Jones, Staff Scientist A.R. Mason, Master Technician R.J. Matson, Staff Scientist J. Zhu, Research Associate

### 3.2 Introduction

Using advanced electron microscopy and microanalytical techniques, the Materials Characterization Group investigates the compositional, crystallographic, structural, defect, luminescent, and topographic properties of photovoltaic and semiconducting materials and devices. The group collaborates extensively with internal and DOE subcontracted researchers and provides analyses crucial to understanding and developing state-of-the-art photovoltaic devices. The work can be divided as follows. (1) Routine support service provided to in-house and subcontracted researchers (e.g., measurement of a sample's chemical composition, measuring the density of defects in a material and determining the crystallinity). (2) Long-term studies of particular problems pertaining to PV devices. This is done in close collaboration with researchers in a great variety of photovoltaic technologies, and the results are often correlated with the crystal-growth conditions and/or with the electrical properties and cell performance. (3) Characterization support in areas other than PV, but in concert with NREL's mission (e.g., the study and determination of the composition and crystal structure of high-temperature superconductors and our business development effort).

### 3.3 Analysis Capabilities

### **Electron Probe X-ray Microanalysis (EPMA)**

 This technique is used primarily to determine the chemical composition of the top 0.5-2 µm of a specimen surface. This is accomplished by both energydispersive spectroscopy (EDS) and wavelength-dispersive spectroscopy (WDS). The instrument used is a fully automated Cameca MBX Electron Microprobe with a Tracor Northern TN-2000 data acquisition system. EDS is normally used for qualitative evaluation, while WDS is used to quantify elemental concentrations to ± 0.5 at. % accuracy. This system can analyze all elements heavier than boron, and in concentrations > 0.5 at. %. Computer automation allows compositional mapping of predetermined regions of interest in a sample.

### **Transmission Electron Microscopy (TEM)**

- This instrument and associated techniques are used primarily for structural and analytical studies involving the examination of thin (200-2000 Å) plan-view and cross-sectional samples. Electron diffraction is used to study the crystal structure of the materials examined, and to reveal grain orientation, crystallographic relationship between grains, and any preferred orientation of polycrystalline films. Bright-field and dark-field imaging are used to investigate the type, density, origin, and three-dimensional distribution of defects, such as dislocations, stacking faults, and grain boundaries. In addition, the instrument can perform X-ray EDS analysis, which is used to evaluate local chemical compositions and compositional inhomogeneities, and electron energy loss spectroscopy (EELS), which yields qualitative information on elements of low atomic number. High resolution (lattice imaging) is often utilized to study the structure and abruptness of interfaces in multilayer structures and to investigate atomic ordering in various compound semiconductors.
- The current system consists of a Philips CM-30 300-keV TEM with a scanning transmission electron microscope (STEM) attachment, a Kevex Delta Class EDS analyzer, and a Gatan 807 EELS spectrometer. The point-to-point resolution of the system is 2.1 Å in the TEM mode and ~15 Å in the STEM mode. Furthermore, the double-tilt goniometer specimen holder allows a ±45° tilt.

### Scanning Electron Microscopy (SEM)

The group supports and applies two extensively developed scanning electron microscope-based systems. The first SEM is a JEOL JSM-35c with the following capabilities: electron channeling (EC), EDS/X-ray analysis augmented by a TN-2000 image automation and X-ray analysis system, backscattered electron imaging (BEI) and cathodolumi-nescence (CL) imaging, in addition to the usual secondary electron imaging (SEI, resolution: 5.0 nm). The system was further developed to include temperature dependent (80K-700K), light-and voltage- biased, planar and cross-sectiona EBIC, and EBIV analysis capabilities. The selected-area electron channeling allows the instrument to determine the crystalline type, orientation, and atomic lattice spacing

of areas down to 20 microns in diameter. The superposition of SEI and X-ray maps allows compositional and topographical features to be correlated. EBIC, or charge collection microscopy (CCM), constitutes the primary use of this system. Junction, or cross-sectional, EBIC is used routinely to determine the position of the electrical junction with respect to the device surface and the intended layers of the device structure. The electrical behavior of the device junction(s) can be monitored as a function of voltage and/or light biasing over a wide range of temperatures. Planar EBIC is used to determine both the density and electrical recombination behavior of crystalline defects, such as dislocations in mismatched heterostructures. This, in turn, can be correlated with device performance parameters. Finally, either junction or planar EBIC can be used for determining the local minority carrier diffusion lengths in a sample material or device. The synergetic power of the system is considerable in its ability to characterize and correlate compositional, structural, electrical, luminescent, and topographical features and properties of a sample at one sitting.

The second SEM system is a JEOL Model JSM-840, which is dedicated to temperature dependent, panchromatic, and spectral cathodoluminescence. CL is used to study the recombination behavior of defects and to measure the relative impurity concentrations, the density and distribution of defects, and minority carrier diffusion lengths and lifetimes. The special temperature-controlled, liquid-helium CL stage allows these properties to be studied at temperatures ranging from room temperature down to 10 K, with the lower temperatures being useful for greater luminescent yield and increased spectral resolution. The panchromatic (or total light) CL imaging is generally used for mapping nonuniformities in the recombination behavior of a material without having to form a device, which would be necessary for EBIC analysis.

### 3.4 Accomplishments

• The electron microprobe continued to have the largest sample-analysis throughput of the branch's analytical capabilities (on average 200-250 samples per month), supporting almost all internal and subcontract groups with accurate and quantitative compositional analysis. Samples analyzed were in both thin-film and bulk form. A very wide variety of photovoltaic materials have been examined, including CuInSe<sub>2</sub>, CuGaSe<sub>2</sub>, ZnCdS, CdTe, CdSe, CuGaInSe<sub>2</sub>, GaAlAs, GaInAs, GaAsP, GaInP, ITO, and amorphous Si, as well as high T<sub>c</sub> superconductors.

• The SEMs and TEM were used extensively to study surface morphology, crystallinity, and defect properties; more than 650 samples were logged during this period. For these studies, secondary electron and EBIC imaging, selected-area electron channeling patterns, and TEM plan-view examination have been employed. In addition to these routine investigations, many collaborative research projects involving internal and external research groups and photovoltaic industry researchers have focused on specific and critical problems encountered in photovoltaic materials and devices. Selected examples demonstrating the usefulness and range of the available analysis techniques follow.

### In Support of the Thin Film CuInSe<sub>2</sub> Project

- TEM was used to investigate the microstructure of CuInSe<sub>2</sub> (CIS) polycrystalline thin films grown by either vacuum coevaporation or evaporation followed by selenization. The grain size was found to depend on the Cu concentration; the higher the Cu concentration, the larger the grains. The grains, in general, columnar with an equiaxed cross section, except in very Cu -poor films. Furthermore, the grains were highly faulted, containing dislocations, stacking faults, and a high density of twins. EDS examination of single grains of CIS indicate that the chemical composition is not constant across the grains. The Se/Cu and Se/In ratios were observed to decrease by a factor of two towards the grain edge, while the Cu/In ratio remained constant.
- The characterization of precursor films of CIS grown by a two-stage process showed that the grains are, in general, larger (> 2 μm) than those in films grown by coevaporation. SEM/EDS analysis of thin TEM planview samples showed the presence of compositionally different grains, such as CIS, Cu<sub>2</sub>Se, and CuSe. SEM/EDS linescans across grain boundaries sometimes displayed a drop in Se concentration at the boundary. Furthermore, transmission electron diffraction (TED) investigations showed the presence of both the chalcopyrite- and the sphalerite- forms of CIS within a single grain.
- The topographical effects of the nature of the precursor layer on the quality of Internation Solar Electric Technologies (ISET) CuInSe<sub>2</sub> films (two stage technique) were examined. It was determined that morphological and stoichiometric nonuniformities present in the precursor layers generally manifest themselves as gross defects in the reacted films. In response, ISET now deposits Te monolayers to successfully effect the nucleation characteristics of the In precursor.

- A range of back-contacting schemes were investigated in collaboration with the NREL Thin Film Group for the effect of the morphology on the depth and lateral uniformity of the electrical activity, as determined by EBIC techniques. That efficient devices can be fabricated on thin films of AI, for example, presages device deposition on AI sheets.
- An SEM/SAM study of the effect of selenization on different metal layers was conducted with ISET. The results were for selenized structures of glass/Mo/(Al, or Ni, or Cu)/Mo. In the Al and Ni cases, the Se did not effect the topography of the outer-Mo layer, and the Se only penetrated into the Mo ~ 600-1000 Å. However, in the Cu case, the topography was quite rough, and unevenly so, and as the Cu out-diffused all the way through the top Mo layer, so the Se penetrated much further into the Mo layer (to ~ 1 μm). Selenizing just Mo and Ti layers resulted in Se diffusion into Mo ~ 1500 Å, and into Ti ~ 3000 Å. The pervasive presence of oxygen through many of these structures was also noted.
- Devices made from large-grain NREL CIS were investigated by EBIC. The material employed Cu-rich precursors, a silicon substrate, and a thin Mo contact. Grains, 2 µm and larger, were examined both in cross section and in plan view for evidence of recombination at the CIS grain boundaries (GB). This was an important opportunity because the geometry and small size of the grains in the conventional CIS material, in comparison to the dimensions of the electron-beam-generation volume, always precluded unambiguous determination of the role of the grain boundaries in device current losses. Counterintuitive, yet consistent with information suggested by earlier EBIC investigations of CIS GBs, the GBs did not show any evidence of GB recombination. Apparently the existing defects are essentially completely passivated in the fabrication process.
- Electroplated, chemically dipped, and evaporated ISET CdS films of differing thicknesses were investigated for topography and electrical uniformity, using the SEM and EBIC. Aside from holes in the evaporated CdS, the overall large-scale electrical uniformity of the evaporated and dipped CdS was good. The electroplated CdS film was quite rough, especially for a thin film of 800 Å. Individual crystallites, approximately 10 µm in diameter, were also observed to be growing out of the 100 nm layers of dipped CdS, under certain growth conditions. The study identified the growth conditions responsible for the large grains. The junctions within the CdS/CIS devices were both relatively uniform and beam insensitive, with a junction depth averaging about 800 nm. The insensitivity to the electron beam

suggests that the incorporated oxygen and, therefore, the CIS material as a whole, should be stable as a material system.

### In Support of the Thin-Film CdTe Project

- The investigation of CdTe thin films by TEM showed that the internal structure of the grains is very diverse. While some grains are virtually defect free, adjacent grains have defect densities at the 109 cm<sup>-2</sup> level. Similar conclusions can be drawn from CL measurements. While some grains do not show any luminescence yield, others have a high luminescence yield. This is due to a varying number of deep levels that act as nonradiative recombination sites for the excess charge carriers. Grain boundaries in the material were found to act as sites of enhanced nonradiative recombination.
- In concert with Stanford University, a study of ion- and photon-assisted doping of thin-film CdTe began this year. Employing EBIC and wavelength dispersive cathodoluminescence (WDCL) for the microanalysis of defects and, in particular, subgrain boundaries, both In Schottky barriers/single crystal and thin film/single crystal devices were investigated. Essentially a preliminary study, identifying processing problems with the diagnostic devices and establishing characterization requirements and restraints, was conducted. Etch pits and their electrical and luminescence properties were correlated. Plans have been made to study the structural properties through the superpositioning of EBIC and/or WDCL images with electron-channeling patterns.
- Junction electron beam-induced-current (JEBIC) scanning electron microscopy was also used to investigate the junction location and uniformity of the recent record 14% efficient CdS/CdTe thin-film device from the University of South Florida (T. and S. Chu). Unlike many of its predecessors in the CdTe family of thin film materials, the junction was a heterojunction rather than a homojunction, and the junction itself was uncommonly uniform, both in its lateral and depth distributions. Parallel studies into the effect of post-deposition heat treatments on this type of device are being conducted as well.

### In Support of the Si Industry

 Spheral silicon structures, grown at Texas Instruments, were characterized by TEM. The bulk of the spheres was defect free. The near-surface region (within 1.0 µm from the surface) has a high density of dislocation loops and precipitates (Figure 3.1). The loops are typically

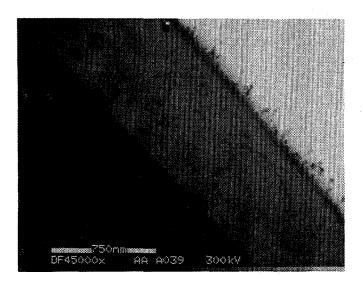


Figure 3.1

0.1 µm in size. This region is continuous around the shell of the spheres. Additionally, there are long lateral dislocation segments around the circumference of the spheres. Some of the dislocations extend as deep as 2.0 µm from the surface.

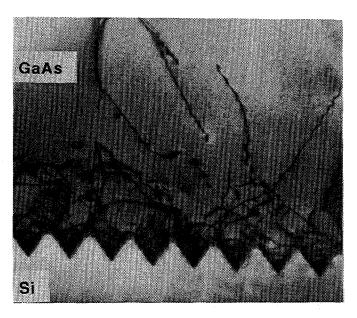


Figure 3.2

### In Support of the High Efficiency Project

 Spire Corporation's GaAs-on-patterned-Si structures were examined by TEM and SEM. TEM studies revealed a high density of misfit dislocations on the {111} facets of the corrugated substrates. Figure 3.2 is a crosssectional micrograph of such a structure, grown by a two-step method without any subsequent heat treatment. Clearly, some threading dislocations propagate

- from the interface region. The use of in-situ annealing affected a drastic reduction in the density of such dislocations and a better dislocation confinement to the Si/GaAs interface region.
- GaAs grown on flat-Si substrates (also from Spire) have been studied by cross-sectional TEM. The nucleation layer of GaAs is grown by atomic layer epitaxy and followed by thermal cycle growth. Most of the dislocations are confined to the interface region. However, some threading dislocations propagate through the active layer, which could severly degrade the device performance.

### 4.0 DEVICE DEVELOPMENT

### 4.1 Research Staff

T.J. Coutts, Group Leader and Principal Scientist
F. Abou El-Fotouh, Senior Scientist
M.W. Wanlass, Senior Scientist
K. Ramanathan, Senior Scientist
T.A. Gessert, Staff Scientist
R.G. Dhere, Staff Scientist
J.S. Ward, Staff Scientist
X. Wu, Visiting Scientist
X. Li, Post Doctoral Researcher

S. Ahrunmwunde, Graduate Student

Y. Qu, Graduate Student

### 4.2 Introduction

The objectives of the Device Development Group are to provide a service-oriented activity to advance the fabrication and development of devices fabricated at NREL, or elsewhere within the DOE National Photovoltaic Program. This includes maintenance of the device fabrication cleanroom; provision of a grid-design facility; maintenance of equipment within the clean room (e.g. the mask aligner); provision of an AR coating design and fabrication service; development of equipment used to advance measurements needed for the above services (e.g. the spectroscopic ellipsometer and other equipment); diagnostics of devices, and feedback to other fabrication groups; development of novel or improved techniques for the fabrication of devices; and study of materials of current or prospective interest to the program. During the last year, the group has strengthened its involvement in the polycrystalline thin film device area and has initiated collaborations with several of NREL's principal sub-contractors and with in-house researchers. Although this process was actually begun last year, the pace has accelerated during the current year. In addition, the group has become involved in the fabrication of diagnostic devices for evaluation of the in-house projects concerned with the growth of high-quality silicon. Work for others in the high-efficiency III-V device area has also increased, and this has led to the usual identifiable benefits to the DOE program.

### 4.3 Accomplishments

### **Projects Related to the DOE Research Tasks**

### Polycrystalline CdTe and CIS cells

 Initiation of an in-house effort for the development of CdTe thin film cells

- Fabricated sputter-deposited CdTe devices with efficiencies of 6.8% (Fig. 4.1) [Ref. 4]
- Established solution growth of thin CdS films for CdTe and CIS projects
- Began fundamental study of stability and nature of CdTe contacts
- Began fundamental study of heat treatment mechanisms in CdTe cell fabrication
- Collaborations established with several subcontractors working on CdTe devices
- Established computer-modelling routine for resistive loss analysis and optimization for series-connected thin-film cells and modules
- Member of group presented invited paper at AVS National Vacuum Symposium on 'Metallization issued in solar cell contacts' [Ref. 27]

#### CdS/CdTe/Au, Global, 1 kW/m2

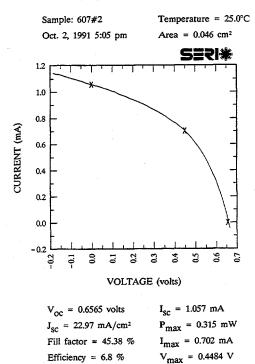
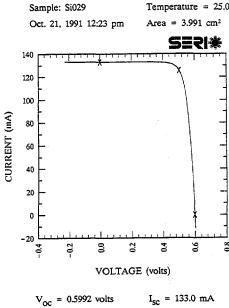


Figure 4.1

- Interaction with ISET on optical analysis of CIS devices led to increased efficiency
- Collaboration begun with Thin Films Group for fabrication/characterization and optimization of ZnO window layers for CIS cells

### NREL ZnS/MgF2 Global 1KW/m<sup>2</sup>

Temperature = 25.0°C



 $J_{SC} = 33.33 \text{ mA/cm}^2$  $P_{max} = 63.3 \text{ mW}$  $I_{max} = 125.6 \text{ mA}$ Fill factor = 79.43 %  $V_{max} = 0.5041 \text{ V}$ Efficiency = 15.9 %

Figure 4.2

#### GaInAsP 1.15 eV. Concentrator 170.95 Sun

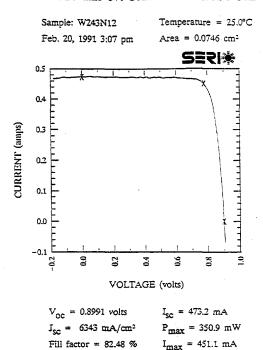


Figure 4.3

 $V_{max} = 0.7778 V$ 

Efficiency = 27.5 %

Established optical modelling routines for the analysis of losses in devices [Ref. 22]

### **Crystalline Solar Cells**

- Established technology for the fabrication of diagnostic Si devices to evaluate in-house crystal growth technology, and achieved efficiencies of near 16% (Fig. 4.2)
- Fabricated single-junction concentrator cell from GalnAsP, with optimum energy gap of 1.15 eV, having efficiency of 27.5% at 171 suns, direct spectrum, 25°C. (Fig. 4.3)
- Further improvements to the spectroscopic ellipsometer
- Member of group received Award for Outstanding Achievement in connection with the development of the first solar cells from NREL with efficiencies greater than 30%
- Submitted an invention disclosure entitled 'Single junction solar cells with the optimum band gap for terrestrial concentrator applications'
- A U.S. patent entitled 'Monolithic tandem solar cell,' No. 5,019,177, was issued [Ref. 69]
- Member of group presented a Plenary paper on high efficiency tandem solar cells at the Photovoltaic Specialists Conference in Las Vegas [Ref. 70]
- Member of group presented an invited paper at the Third International Conference on InP and Related Materials, on InP-based solar cells [Ref. 12]
- Provided a substantial input for the design of the new Solar Energy Research Facility (SERF).

### **Projects Related to the DDF Research Tasks**

- Solution growth of CdS taken to successful conclusion, final report prepared and the work is now included in the main DOE program
- Successfully demonstrated solution growth of CdTe films
- Studies of fundamentals of CdTe solution-growth mechanisms commenced

 Project on heteroepitaxy of InP delayed through suspension of toxic gas, but progress made in characterization of devices

### Projects Related to the WFO Research Tasks

#### Naval Research Laboratories

- Successfully obtained increased funding from the Naval Research Laboratories (NRL) for development of radiation-resistant tandem InP/InGaAs space solar cell
- Performed extensive characterization of temperature coefficients of tandems
- Supplied working tandem reference cells to NRL for their experiments
- Optimized grid design for InP top cell of tandem, and operated device at a concentration of 100X and an efficiency of 24.3% under the direct spectrum
- Supplied samples of tandem cells for radiation resistance experiments
- Collaborated with Sandia Laboratories on the measurements of tandem-cell efficiencies under concentration, resulting in excellent agreement with measurements at NREL
- Three progress meetings held, and progress reviewed as excellent

### NASA

- ITO/InP cells supplied to NASA for flight experiments on UoSaT 5 satellite (first devices from NREL to be flown in space)
- Studies of plasma interaction with InP surface further elucidated nature of type conversion
- Performed small-scale production of 4 cm<sup>2</sup> ITO/InP cells for study of production issues, and published details of these [Ref. 26]
- 16x4 cm<sup>2</sup> ITO/InP cells provided to University and Federal Agencies for collaborative studies

#### Varian

 Supplied bottom cell of GaInAsP (0.95 eV) for stacking beneath an AlGaAs/GaAs tandem fabricated at Varian, leading to a new one-sun AMO efficiency of 25.2% at 25°C, details of which were presented at the Photovoltaic Specialists Conference [Ref. 70]

#### **Additional WFO Activities**

- Presentation made to the Natural Gas Institute in Chicago in connection with potential project on thermophotovoltaics
- Presentation made to a representative of Knowles Atomic Power Laboratory, again in connection with thermophotovoltaics

### 4.4 ES&H Related Activities

- Group member participated in the in-house ES&H management self-assessment team
- Participated in the in-house toxic gas work force to determine feasibility of remaining in Building 16
- Participated in audit of toxic gas systems
- The necessary modifications to the metal organic vapor phase epitaxy (MOVPE) facility, per the SAR, have been determined, designs for these have been developed, and the bulk of the required parts have been ordered
- Group members undertook ES&H refresher training courses on subject of toxic gas activities
- Extensive participation in design of safety and other facilities in new SERF building
- Close attention paid to chemical inventory of group
- Group member acted as Building 16 representative for committee that developed the Laboratory Chemical Hygiene Plan
- Group member appointed as Staff Representative on Laboratory Ventilation Safety Committee

### 5.0 ELECTRO-OPTICAL CHARACTERIZATION

### 5.1 Investigators

R.K. Ahrenkiel-Group Leader and Principal Scientist

B.M. Keyes-Associate Scientist

D.J. Dunlavy-Associate Scientist

L. Roybal-Master technician

D. Levi-Post Doctoral Fellow

J. Zhang-Research Associate

L. Wang-Research Participant

#### 5.2 Introduction

This task characterizes both the electrical and optical characteristics of photovoltaic semiconducting materials and devices. These investigations provide data to researchers leading to a faster development of the photovoltaic technology. Our facilities specialize in unique minority-carrier characterization measurements.

### 5.3 Analysis Techniques

### The Laser Characterization Facility

#### **Time-Resolved Photoluminescence:**

• The time-resolved photoluminescence (TRPL) facility has achieved world-wide recognition in the area of minority-carrier characterization. The primary function of the TRPL facility is determining the minoritycarrier properties of photovoltaic materials and devices. The minority-carrier lifetime is the most important parameter relevant to the performance of photovoltaic devices. This facility is unique to the U.S. semiconductor and photovoltaic community. The facility has been adequately described in the literature.<sup>1</sup>

### **Photoluminescence Spectroscopy:**

A state-of-the-art facility has been developed for performing high speed, high resolution PL spectroscopy of materials at temperatures from 10 K to about 300 K. These data are analyzed for the determination of a number of important properties, including the determination of the bandgap in new materials. Also, PL spectroscopy is used for the identification of optically active impurities in new materials. The great advantage of PL is the ability to perform these measurements nondestructively and noninvaisely on finished devices.

### **Diffusion Time-Of-Flight:**

• A time-of-flight (TOF) system technique was invented here for the measurement of minority-carrier diffusivity.<sup>2</sup> In collaboration with workers at Purdue University, the time resolution of experimental setup has greatly increased. Our current apparatus can resolve transit times longer 25 ps. The apparatus has been widely utilized during the year to provide measurements of D in both n- and p-type III-V materials. The technique was the subject of a recent invited paper in Solid State Electronics.<sup>3</sup>

### **Deep Level Transient Spectroscopy (DLTS):**

 DLTS measurements have become widely used for defect characterization for photovoltaic materials. Using the SULA DLTS measurement system, a large number of defect studies in PV materials have been provided.

### 5.4 Accomplishments

- Collaborative work with Purdue measured the minority-carrier lifetime in n-type GaAs grown by MOCVD.
   The largest lifetimes ever reported were a combination of photon recycling and very low Al<sub>0.30</sub>Ga<sub>0.70</sub>As/GaAs interface recombination velocities.
- A continuation of the above work involved etching away the substrate, which enhances the photon recycling effect. Lifetimes of over 1.0 ms were observed<sup>5</sup> in devices doped to 1.3x10<sup>17</sup> cm<sup>-3</sup> (the radiative lifetime at this level is 38 ns).
- Chemical passivation of GaAs by sulfur-compound treatment was analyzed by our TRPL system. The passivated samples were provided by both in-house (A. Nozik) and subcontractor (M. Timmons, Research Triangle Institute [RTI]) researchers.
- In coordination with M. Timmons (RTI), the lifetime "killer" defect associated with aluminum in Al<sub>x</sub>Ga<sub>1-x</sub>As has been identified both by DLTS and TRPL measurements.
- We have continued to use TRPL to characterize minority-carrier lifetime in poly CdTe with four research groups. These are Photon Energy, University of South Florida (T. Chu), Solar Cells Inc., and the NREL Device Developmeng Group. A lifetime of over 5 ns was reported for one solar cell from Photon Energy. An inital model of recombination processes in poly CdTe has been reported.<sup>6</sup>
- We have achieved chemical passivation of singlecrystal CdTe, as shown by TRPL measurements. This work was performed under a DDF grant.

- Work in collaboration with RTI has produced a GaAs thin film that is mechanically bonded to silicon. Our measured lifetimes<sup>7</sup> in these films exceeds 100 ns which is the largest ever reported in such films.
- PL spectroscopy measurements were performed on single crystals of CdTe, provided by A. Fahrenbruch (Stanford University), to assess the ion-assisted doping process.
- DLTS measurements were made on single-crystal silicon cells from Siemens Solar. These studies were aimed at identifying impurities that were contaminating the crystals during the growth process.
- Double heterostructures, grown by a mixture of conventional MBE and Migration Enhanced Epitaxy (MEE) techniques, were analyzed by TRPL in support of this DDF project.
- An invited paper<sup>8</sup> was submitted to Solid State Electronics, which had a special edition on semiconductor characterization.
- More than 35 TRPL measurements were made on GaInP (J. Olson), to help identify some processing problems that had developed.
- Numerous TRPL measurements were made on InP films grown epitaxially on silicon (Spire Corp), in order to characterize the growth process.
- More than 100 zero-field time-of-flight (ZTOF) measurements were made on n- and p-type GaAs devices grown at Purdue University.

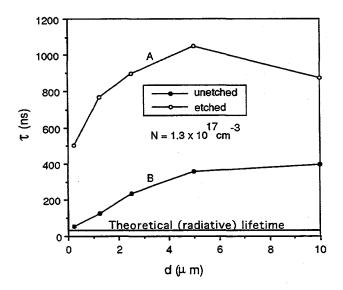


Figure 5.1

- Significant progress has been made in developing contactless lifetime measurement apparatus for silicon wafers. The primary method has been to develop the radio-frequency absorption apparatus. Lifetimes have been measured in silicon with doping levels at about 1x10<sup>17</sup> cm<sup>-3</sup>.
- A pump-probe technique for measuring silicon lifetime is being developed under a DDF grant.
- A new and improved ES&H operating plan for the laser laboratory was developed.
- Effective minority-carrier lifetimes in n-GaAs was measured as a function of the film thickness <sup>5</sup> The lifetime enhancement is caused by photon recycling. The effect may be very important in designing high-efficiency solar cells composed of direct-bandgap semiconductors, such as GaAs, CdTe, etc.

### 5.5 Future Plans

- Expand DLTS capability by adding an additional spectrometer
- Develop a second rf -absorption-measurement system for the industrial silicon program
- Develop a new, contactless technique for measuring lifetime in weak-light emitters, such as silicon
- Identify a new funding source for supporting PL lifetime work

### References

- 1. R. K. Ahrenkiel, Solid State Electronics 35, 239 (1992)
- 2. R. K. Ahrenkiel, Appl. Phys. Lett.
- 3. M. Lovejoy et al, Solid State Electronics 35, (1992).
- 4. G. B. Lúsh, H. F. MacMillan, B. M. Keyes, D. H. Levi, M. R. Melloch, R. K. Ahrenkiel, and M. S. Lundstrom, J. Appl. Physics (in press).
- 5. G. B. Lush, H. F. MacMillan, B. M. Keyes, D. H. Levi, M. R. Melloch, R. K. Ahrenkiel, and M. S. Lundstrom, Appl. Phys. Lett. (in press).
- 6. R. K. Ahrenkiel, B. M. Keyes, L. Wang and S. Albright, "Twenty-Second IEEE Photovoltaic Specialists Conference-1991", p. 940
- Specialists Conference-1991", p. 940.
  7. R. Venkatasubramanian, M. L. Timmons, T. P. Humphreys, B. M. Keyes, and R. K. Ahrenkiel, Appl. Phys. Lett. 60, 886, (1992).
- 8. R. K. Ahrenkiel, Solid State Electronics 35, 239 (1992).

## 6.0 MATERIALS DURABILITY AND COMPONENT RELIABILITY

### 6.1 Research Staff

A. W. Czanderna, Research Fellow F.J. Pern, Senior Scientist L. Barr, Associate Scientist B.J. Bachman, Research Associate G. Herdt, Research Associate

### 6.2 Introduction

NREL'S Advanced Photovoltaic Module Testing and Performance Project has two major roles within the National Photovoltaic Program. These roles involve the performance testing and reliability research of advanced photovoltaic submodules and modules, with primary emphasis on photovoltaic flatplate modules. These efforts are conducted in conjunction with PV research activities originating both within and outside NREL. Specific project research activities include testing and investigations of submodule and module performance, stability, energy output, and reliability lifetime characteristics, both under simulated and actual sunlight conditions. The group also coordinates PV thinfilm module testing and reliability research for the U.S. Department of Energy (DOE) Photovoltaic program and develops simulated and outdoor testing methods and, diagnostic techniques to understand and determine module performance and potential failure mechanisms. The project task elements are carried out in two different branches at NRFI.

The title of Section 6.0 is a major task of the project carried out entirely in Measurements and Characterization Branch and has focused specifically on the problem of ethylene vinyl acetate copolymer (EVA) degradation during 1991. The objectives addressed by the subtask elements are given in the first paragraph of each subsection of Section 6.5. Extensive background about the project and EVA degradation is available in a prior report<sup>1</sup> and an initiative.<sup>2</sup> Additional background and extensive references on the EVA yellowing problem are also available in a recently published paper.<sup>3</sup> The group conducts its research in the Module Encapsulation Research Laboratory, Room 268, Building 16 and the Microbalance Laboratory, Room 388, Building 16.

### 6.3 Analytical and Test Capabilities

UV-visible absorption/transmission measurements, conventional wet chemical analysis (e.g., pH measurement, solvent extraction), accelerated (photochemical and thermochemical) durability tests are used with various light sources and ovens (see equipment list, Appendix D) for UV absorbers, stabilizers, polymer materials, and minimodules. In addition, all the surface- and materials-chracterization capabilities of the branch are available for work on the task. Those of special importance are UV-Vis-NIR, FTIR, microbalance, EPMA, XPS, Auger, and SIMS (Appendix D).

### 6.4 Analytical Techniques

Destructive analytical methods and procedures for module encapsulant materials (primarily EVA) have been described in detail.<sup>4</sup> Nondestructive and destructive analytical methods and procedures (prior to the EVA analysis) for determining I-V characteristics for individual solar cells, and analysis of circuit components in a weathered PV module, are currently under development.<sup>5</sup>

### 6.5 Accomplishments

In the past year, we focused our efforts on (1) investigation of EVA degradation mechanisms via carefully designed, simulated degradation experiments; (2) diagnosis of weathering-degraded PV modules; (3) analysis of the degraded and discolored EVA encapsulants; and (4) search for new materials and stabilizers that may be used to modify the current EVA formulation or to develop new encapsulants. The following summarizes our accomplishments.

### EVA degradation mechanisms derived from simulated-degradation experiments

A large number of simulated degradation experiments and various analyses have been performed on various EVA films, to verify or identify the mechanisms that are responsible for the EVA degradation and discoloration. The results show three primary factors are involved: (1) UV exposure, (2) heating, and (3) acetic-acid-catalyzed reactions. The secondary factors contributing to the EVA degradation are (1) metal ion-catalyzed degradation and (2) deactivation of antioxidants by the acetic acid (per literature) produced from the primary degrading reactions.

### Results from simulated degradation experiments, as illustrated in Figure 6.1:

- UV exposure of EVA at low temperatures (< 50°C) will produce acetic acid.
- Heating in the dark at high temperatures (110°-130°C) also produces acetic acid.
- The presence of UV absorber (i.e., Cyasorb UV 531) and antioxidants will reduce, but not prevent, the generation of acetic acid.
- EVA yellowing is enhanced in the presence of acetic acid in a hermetic condition.
- An open environment (that allows escape of the generated acetic acid) will retard the rate of EVA yellowing.
- Light exposure can result in EVA yellowing, as well as color bleaching. Increasing in the light intensity at 85°-90°C may result in a lesser degree of yellowing, due to offset by photobleaching.
- The rate of EVA yellowing by heating is greater without light illumination and increases as the temperature increases.
- Both UV exposure and heating of EVA, with or without acetic acid, produce polyenic chromophores that are similar to those found in the field-degraded, yellowbrown EVA.
- Acetic acid can thermally catalyze the EVA yellowing, which increases as the heating temperature (between 85°C and 130°C) and time (5 to 11 days) increase.
- The Cyasorb UV 531 undergoes photodecomposition at temperatures of 40°C or more.
- Both UV exposure and heating of EVA will result in a loss of Cyasorb UV 531 and a gain of cross linking.
- All EVA formulations (available from Springborn Laboratories) show the effect of acetic-acid-catalyzed yellowing.
- Heating at 85°C in the presence of metal ions, Pb²+ and Sn⁴+, does not result in EVA yellowing, as a preliminary result; under identical heat treatment, Cu²+ causes a greenish color and Ag⁺ a yellow-brown color. Thus, the EVA yellowing may not be strongly related to metal ions generated from corroded buslines (Pb-Sn alloy, coated Cu-foil ribbon) but further detailed work is clearly needed.

### Diagnosis of weathering-degraded PV modules, and analysis of degraded and discolored EVA

In this part, we continued our efforts (1) to characterize the structural changes in degraded and discolored EVA encapsulant samples from PV modules, (2) to establish the

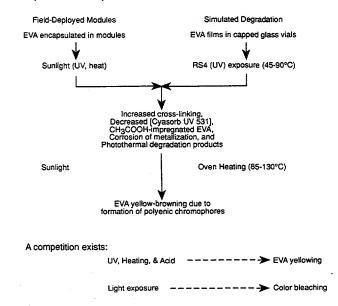


Figure 6.1: Flow diagram showing how results of EVA degradation from field-degraded PV modules compare with those from laboratory simulated experiments.

relationship between the EVA discoloration and PV-module performance, (3) to investigate the effects of EVA degradation on solar cells and circuit components, (4) to explore the feasibility of employing the I-V measurements in the dark and in the light, to determine the PV module failure mechanisms, and (5) to determine the correlation between the performance degradation of a module and the performance of individual solar cells in that module.

### The important results obtained to date are given below:

- The yellow-browning of the weathering-degraded EVA is due to formation of polyenic chromophores of various lengths. The discolored EVA will absorb a large portion of the sunlight below 1100 nm, thus reducing the light available to the solar cells and hence the generation of photocurrent.
- The weathering-degraded EVA in modules, whether discolored or not, shows a large increase in gel content (degree of cross-linking), and a large, to complete, loss of Cyasorb UV 531. Acetic acid and other organic components are also detected.

- Degradation of EVA also causes oxidation and corrosion of the buslines, possibly due to the presence of acetic acid, oxygen, and moisture. A great deal of metal interdiffusion on the buslines is also observed. The results suggest that an increased contact resistance between the oxidized busline and the Ag grids, and an increased series resistance, may be partly responsible for the efficiency loses of PV modules.
- The efficiency of an EVA-discolored PV module is an "average" of all solar-cell units in it. Present results show that an individual solar cell isolated from its neighbor cells may have an efficiency better or worse than the average value. The results suggest the likelihood of localized, severe degradation on the circuit component as the limiting factor dictating the overall module performance.

### EVA degradation mechanisms in fielddegraded PV modules: A summary

From the similarity observed between the results obtained from simulated degradation experiments (Figure 6.1) and those from the analysis of natural weathering-degraded EVA and PV modules, the degradation and discoloration mechanisms of the EVA encapsulant can be summarized as shown in Figure 6.2. A description is provided here.

(A) Simulated Degradation: \_сн₃соон Oven - 85-130°C - Yellow EVA A9918 EVA 15295 EVA 15295P EVA 16718A + CH3COOH (B) Color Bleaching: RS4 (3 lamps, 57°C) 150-W Xe white light 150-W Xe visible light Faded (lighter) colo (light beam focused) (C) Experimentally-verified Results: Elvax 150 & RS4 (3 lamps) OH-cleaned Acetic acid & other organics 46°C EVA increased cross-linking RS4 (3 lamps) 46-87°C Increased cross-linking **FVA** with Loss of Cyasorb UV 531 Stabilizers Acetic acid & other organics

Figure 6.2 Flow diagrams showing conditions and results of laboratory experiments used to elucidate EVA degradation mechanisms in PV modules.

As a result of various photothermal reactions upon exposure to the sunlight (UV and heating), the EVA becomes more cross-linked and loses Cyasorb UV 531 concentration. As a result, acetic acid from multiple deacetylation reactions, and other organic products are produced, and polyenes (yellowing chromophores) are formed. The increased concentration of acetic acid causes a thermal autocatalytic effect that further enhances the yellowing, and may result in oxidation and corrosion of the metallization. Acetic acid can also deactivate the antioxidants (Tinuvin 770 and Naugard P). A competition exists between the EVA yellowing (due to formation of polyenic chromophores) and color photobleaching (due to breaking or shortening of polyenic chromophores). Beyond a certain critical point, when loss of Cyasorb UV 531 and accumulation of acetic acid in the PV modules become significant, the effect of UV-induced and thermally acetic acid-catalyzed yellowing exceeds the rate of photobleaching and causes the EVA to turn yellowbrown. A higher operating temperature of PV modules (e.g., produced with two reflecting mirrors) will favor an earlier and faster EVA yellowing in PV modules than a lower operating temperature. Good examples are the EVA browning of Carrisa Plains PV modules, operating with mirrors and PV modules (without mirrors) deployed at the Southwest Residential (SWRES), in Las Crucas, NM (maximum T =

### New materials for encapsulation (polymers, UV absorbers, stabilizers, and cover glasses):

We have acquired a number of polymer materials, UV absorbers, and stabilizers (antioxidants), either from manufacturers or as free samples from people we have met or contacted. The absorption spectra of these UV absorbers have been studied and documented. Two UV absorbers are considered particularly useful because of their capability of absorbing UV light below 370 nm and 390 nm, respectively. Preliminary work on the photothermal degradation studies has been performed for solvent-cast EVA thin films containing these two new UV absorbers. The work is ongoing. Similar studies were performed for two cover-glass samples provided by OCLI. Transmission measurements indicate both cover glasses may be useful as superstrates because the UV cutoff is below 350-360 nm, although transmission for the glasses laminated with a cured EVA A9918 is from 65% to 90%, between 400 nm and 580 nm.

### Other accomplishments

- Preparing an initiative for DOE to emphasize the need for more resources<sup>2</sup>
- Conducting a literature search on the metal cationcatalyzed, thermal, oxidative degradation of Elvax 150 and EVA, where Elvax 150 is the principal (>99%) component of EVA

- Conducting a literature search on the metal cationcatalyzed, thermal, oxidative degradation of Elvax 150 and EVA, where Elvax 150 is the principal (>99%) component of EVA
- Identifying important capital-equipment needs, and preparing purchase requisitions for a fluorescence spectrophotometer (see reference 1, in Section 6.6) and a high performance liquid chromatograph (HPLC)
- Presenting eight papers in various forums about our work
- Providing periodic letter reports to several industrial collaborators
- Making site visits to SWRES, Mobil Solar, Photon Energy, Siemen's Solar, UFG Corp., and USSC, to discuss PV-module durability and EVA-degradation issues

### 6.6 References

- 1. Keyes, B. M., and P. Dippo, eds., FY 1990 Applied Sciences Branch Annual Report, NREL/TP-213-4461, November, 1991, Section 6.0.
- 2. Czanderna, A. W., R. DeBlasio, and L. Mrig, "Advanced PV Module Materials and Encapsulation System Initiative, May 15, 1991, NREL; a copy may be requested from the first author."
- 3. Pern, F. J., and A. W. Czanderna, Solar Energy Materials and Solar Cells, 25 (1992) 3.
- 4. Pern, F. J., "Procedure for Analysis of Ethylene-Vinyl Acetate Copolymer Pottant Materials," Internal Document.
- 5. Pern, F. J., "Procedure Designed for Failure Mechanism Analysis of EVA-containing Si-based PV Modules," Internal Document.

### 7.0 CELL AND MODULE PERFORMANCE

### 7.1 Research Staff

K.A. Emery, Senior Scientist and Group Leader E.E. Beck, Technician C.R. Osterwald, Staff Scientist S. Rummel, Staff Scientist

### 7.2 Introduction

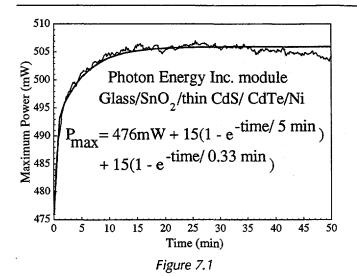
The performance of photovoltaic (PV) devices of all sizes and technologies are evaluated in these laboratories. This group supports the entire PV community by providing (1) Secondary calibrations of photovoltaic cells, (2) efficiency measurements, with respect to a given set of standard reporting conditions, (3) efficiency verification of contract deliverables, (4) current-versus-voltage (I-V) measurements under varying temperature, and (5) spectral irradiance and total irradiance. This group also supports the entire PV community by providing information on PV measurement equipment and systems that are appropriate for the end user, I-V measurement procedures, potential artifacts in the I-V results because of equipment or procedures, and an analysis of the uncertainty in groups performance measurements. This group also performs spectral-irradiance modeling and PV-performance modeling, in support of its ongoing research efforts to reduce the uncertainty in the measured solar spectrum, from 300-4,000 nm, to assist in the optimization of PV devices for a given application, and to explore alternative rating methods.

### FY1991 Accomplishments

\*Editorial note: The laboratory maintained the status-quo in capabilities with insufficient staffing and frequent hardware failures.

- Performed 1,600 cell and 1,200 module-performance measurements and calibrations for researchers internal to NREL, under contract to NREL, the U.S. and international PV industry, and universities
- Gave technical advice on PV-performance-measurement procedures, hardware, and software to researchers internal to NREL, under contract to NREL, the U.S. and international PV industry and universities

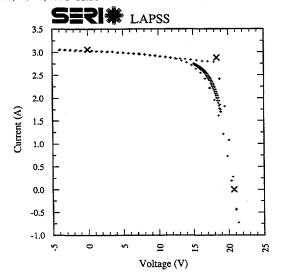
- Developed a data-base-management system for the SPIRE 240 solar simulator and the Spectrolab Large Area Pulsed Solar Simulators (LAPSS), to allow permanent storage and easy retrieval of the tabular or graphical data
- Commenced operation of the LAPSS on a limited basis while performing modifications so that all PV-module and concentrator cell technologies can be evaluated
- Continued documentation of anomalous results in PV-performance measurements arising from measurement artifacts or procedural errors; continued research to understand and reduce the uncertainty in PV-performance measurements at NREL and throughout the PV community
- Established that substantial differences in the measured I-V characteristics of a given device may occur amoung different measurement systems or groups because of premeasurement conditions, bias rate, bias direction, contacting, spatial uniformity of the light source, and algorithms to obtain the maximum power
- Observed reversible improvements in the efficiency of 0.1% to 9%, with forward biasing in the light or dark prior to the I-V measurement, in several InP, CdS/ CuinSe2, and CdS/CdTe, and CdS/Cu2S structures. It appears that this phenomena is related to the quality of the diode properties, with the more ideal diodes having less improvement. For CdS/CuInSe2, this improvement is usually in the voltage, with no observable change in the short-circuit current. For some CdS/CdTe modules and cells, the only way to ensure that the measurement of the efficiency is within ± 3% of the "true" steadystate value at SRC is to track the maximum power as a function of time under continuous illumination, with simulated or natural sunlight (Figure 7.1). The small decrease in the power with illumination time after 30 minutes in Figure 1 was because of heating. The CdTe submodule is a superstrate structure, where light goes through the glass and is collected in the thin film of CdTe. The CdTe submodule was cooled with a box fan under the sample throughout this experiment, to minimize the drop in power with increasing temperature. The increase in PV performance for the CdS/CdTe and CdS/CuInSe<sub>2</sub> devices with time is completely reversible by shorting or reverse-biasing the sample.
- Observed temporal instabilities in crystalline silicon; these artifacts are easily detectable by shorting the sample under illumination and then observing the open-circuit voltage as a function of illumination time. The short-circuit has been observed to change as a function of prior voltage history in some n- and p-type silicon structures and in a InP/GaAs heterostructure. Bias rate or hysteresis effects can be a problem for any



pulsed- or high-speed measurement system in which the bias rate can easily exceed 1000 V/s. In many cases, hysteresis effects are not obvious from the measured I-V characteristics, but are readily apparent if the sample is biased in both directions. Unfortunately, most commercial I-V measurement systems are incapable of biasing in both directions, so the only other choice is to reduce the bias rate near the maximum power voltage and determine if there is any change in the maximum power.

Sample ID: S 100 sn 103 Reference cell ID: S02 Fri, Nov 1, 1991 12:28 PM Temperature: 20.3°C Area: 8115 cm<sup>2</sup>

Irradiance: 1000 Wm-2 ASTM Global



 $P_{max}$ : 52.840 W, s = 2.76%

 $V_{oc}$ : 20.776 V, s = 0.04%

V<sub>max</sub>: 18.3719 V

 $I_{sc}$ : 3.0581 A, s = 0.17%

I max: 2.8761 A

 $J_{sc}$ : 376.84  $\mu A \text{ cm}^{-2}$ 

Efficiency: 6.5 %

Fill Factor: 0.8317

Figure 7.2

Developed a custom data acquisition for the Spectrolab LAPSS, to identify and attempt to minimize temporal instabilities: The system is capable of varying the initial voltage, final voltage, bias rate, and bias direction. An example of hysteresis observed in a commercial Si module is shown in Figure 7.2. The outer curve was a single sweep from forward bias to reverse bias during the ~2 ms pulse. The inner curve was generated from a separate single flash with the voltage from reverse towards forward bias. The cluster of data points near P<sub>max</sub> was generated by another flash while limiting the voltage bias range to points near P<sub>max</sub>, indicating that the "true" steady-state characteristics are represented by the inner, lower-power curve. The V<sub>oc</sub> and I<sub>sc</sub> points were also measured during separate flashes.

# 8.0 SURFACE INTERACTIONS, MODIFICATIONS, AND STABILITY GROUP

### 8.1 Research Staff

A. W. Czanderna, Research Fellow and Group Leader J. R. Pitts, Senior Physicist D.E. King, Postdoctoral Research Associate E. Tracy, Staff Scientist G.C. Herdt, Research Associate Y. Shinton, Master Technician C.L. Fields, Visiting Professional R.L. Vojdani, Undergraduate Intern

### 8.2 Introduction

The Surface Interactions, Modification, and Stability Group (SIMSG) has been part of the Measurements and Characterization Branch since October 1989. Most personnel and projects are funded from the Solar Industrial and Solar Buildings programs, in addition to the internally funded programs. Since our funding is secured from several sources outside the Photovoltaics Division, the purpose and/or objectives, background, and accomplishments of each task are addressed by the references in Section 8.7 and Appendix C.

The purposes of the SIMSG are (1) to correlate the composition, bonding, and/or structure and other properties at interfaces with the performance properties of solar energy conversion, opto-, and electronic devices; (2) to understand the mechanisms and processes occurring at interfaces that limit device durability; and (3) to modify the interfaces to improve their properties and/or stability. Specific types of solar-relevant problems are highlighted in a previous publication, workshop, and recent review chapter.

Topical areas for study in surface science include radiation-induced surface transformations, organized molecular assemblies, <sup>4</sup> polymer/metal (oxide) interfaces, interdiffusion, metallization corrosion, radiation-induced surface and near-surface reactions, and interface stability in multilayer thin-film devices. The personnel of the SIMSG were involved in FY 1991 tasks as follows: (1) solar induced surface transformations and modifications (SISTM); (2) X-ray photoelectron spectroscopy (XPS) and ion scattering spectroscopy (ISS) of organized molecular assembly (OMA)/copper interfaces; (3) advanced desiccant materials research; (4) criteria, test methods, and durability of electrochromic windows; and (5) organizing a surface-processing workshop. Publications

or major NREL reports have been completed. In addition, review chapters on ion spectroscopies for surface analysis and on the most widely used methods of surface analysis were published during the year. In subsections of 8.3 and 8.4, the analytical capabilities and techniques related to tasks 1 through 5 are summarized.

### 8.3 Analytical Capabilities

The analysis capabilities include ISS, XPS, secondary ion mass spectroscopy (SIMS), Auger electron spectroscopy (AES), scanning Augermicroscopy (SAM), Fourier transform infrared (FTIR) spectrometery, residual gas analysis (RGA), contact angle, ultramicrogravimetry, neutral and ion beams, <sup>5, 6</sup> reaction chambers, solar simulators, excimer dye laser, a high-intensity (2,400 suns) solar furnace, thin-film deposit and control monitors, thin-film deposition, adhesion testing, profilometers, metallographs, X-ray apparatus, and access to capabilities of the surface and optical characterization groups, as well as the FTIR Spectroscopic Research Center. Appendix D provides a listing of the major instrumentation and test equipment, their features, and their specifications.

### 8.4 Analytical Techniques

Descriptions of the surface analytical techniques in the Leybold LHS-10 (XPS, AES, ISS, SIMS) and Physical Electronics 545 C (AES) systems are available, in general,<sup>3</sup> and in considerable detail.<sup>8</sup> The FTIR capability, including using the solar simulator, excimer dye laser, and reaction-chamber techniques, is treated adequately in Section 9.0 (Webb et al.). The use of RGA, ion beams for analysis and depth profiling, profilometers, and adhesion testing (Sebastian) equipment has also been described.<sup>4</sup>

### 8.5 Accomplishments: Photovoltaics

As stated in the introduction, the projects conducted by the SIMSG in FY 1991 were not generally carried out in support of the Photovoltaic (PV) Technology Program. The contributions made by SIMSG personnel to PV projects are included in Section 6.3.4 of this report.

### 8.6 Accomplishments: Related Research Areas

### **Solar Induced Surface Transformations and Modifications (SISTM)**

Filed two records of invention

- Carried out pyrolysis experiments at the flux limit available at the NREL solar furnace, where the measured average flux is 2 kW/cm<sup>2</sup> and the calculated peak flux is 2.5 kW/cm<sup>2</sup>
- Prepared diamond-like films, graphitic films, and SiC films up to 1 µm thick, and established process conditions where each film type will form
- Formed YBCO-type, high-temperature superconductors on MgO substrates by metalorganic deposition in the solar furnace, with critical current densities of 30,000 A/cm<sup>2</sup>
- Supported solar-furnace related DOE initiatives in synthesis and processing, surface modification, and lunar-materials processing
- Supported three different study groups from the National Academy of Sciences, SRI International, and MIT Lincoln Laboratories, each of which advocates supporting the current research efforts, calls for increased funding of the work, and recommends delaying industrial development
- Contacted three major industrial clients; an reaching the final negotiating stages for forming CRADAs

### Organized Molecular Assembly (OMA)/ Copper Interfaces

- Demonstrated that copper, deposited onto COOH endgroups of an OMA, is oxidized to the +1 state, interacts principally, if not exclusively, by replacing the H of the hydroxyl group, and completes it interactions at 0.3 to 0.6 nm of coverage [20,38]
- Improved the LHS-10 computerized data acquisition capabilities with a state-of-the-art minicomputer
- Prepared a patent disclosure where OMAs may protect surfaces from dirt accumulation for a renewable energy device
- Explored further interactions between copper and CN (cyanide) end groups on an OMA; preparing a draft of a manuscript on charging effects when OMAs are attached to an insulating surface (Al<sub>2</sub>O<sub>3</sub>)

#### **Advanced Desiccant Materials Research**

 Confirmed that both cationic and anionic polymers (supplied by Eastman Kodak) have excellent potential as desiccants for cooling systems [18] and are comparable to the potential of the sodium and lithium salts of polystyrene sulfonic acid [21]

- Demonstrated that quartz-crystal microbalance (QCM) adsorption measurements on sample masses of 0.3 to 0.5 mg yield more precise isotherms than a sorption test facility that uses 3 to 5 g of sample [32]
- Used the QCM for water sorption studies on a-WO<sub>3</sub> and ethylene vinyl acetate (EVA copolymers) at temperatures from 14° to 60°C
- Improved the software for data acquisition and manipulation with the QCM

### **Electrochromic Windows (ECW)**

- Completed site visitations to nine major organizations with active research, development, production, or interest in ECW; summarizing the results in a report [14]
- Reaffirmed that durability studies of multilayer devices require final configuration, accelerated and abbreviated life testing, diagnostic evaluation, and established degradation mechanisms to be most effective

### **Surface Processing Workshop**

- Planned, organized, and held the workshop on December 10, 11, and 12, 1991, in Dearborn, MI [15]
- Secured 22 authors to prepare plenary papers about surface characterization or surface modification
- Secured 14 chairmen for 10 working groups to prepare 10 summaries of needs and opportunities in some aspect of surface processing [Ref. 15]
- Secured another 40 individuals from industry, academia, and government laboratories to participate in the workshop
- Prepared a draft initiative, for DOE, for funding a multiyear effort in surface processing as applied to conservation and renewable technologies

### 8.7 References

1. Czanderna, A. W., Solar Energy Materials, Vol. 5, 1981, p. 349.

2. Czanderna, A. W., and R. J. Gottschall, eds., "Basic Research Needs and Opportunities on Interfaces in Solar Energy Materials," DOE/OER, CONF. 8006156, Apr. 1981; Special Issue Mat. Sci. Engr. Vol. 53, 1982, pp. 1-168.

3. Czanderna, A. W., Materials Characterization Using Ion, Electron, and Photon Probes, in Industrial Materials Science and Engineering, L. E. Murr, ed.,

Dekker, NY, 1984, pp. 471-517. (SERI/TR-255-2217, July 1984).

- 4. Swalen, J. D., et al., Molecular Monolayers and Films, Panel Report, Division of Materials Sciences, OER/DMS, Langmuir, Vol. 3, No. 9, 1987, p. 932.
- 5. Czanderna, A. W., and S. P. Wolsky, eds., Microweighing in Vacuum and Controlled Environments, Amsterdam: Elsevier, 1980.
- 6. Lu, C., and A. W. Czanderna (eds), Applications of Piezolelectric Quartz Crystal Microbalances, Amsterdam: Elsevier, 1984.
- 7. Czanderna, A. W., and T. M. Thomas, J. Vac. Sci. Technol. A, Vol. 5, No. 9, 1987, p. 2412.
- 8. Pitts, J.R., Silver-Silicon Bonding on Silica Surfaces, SERI/TR-255-2410, Apr. 1985, Ph.D. Thesis, University of Denver.

# 9.0 FTIR SPECTROSCOPIC RESEARCH AND SERVICE CENTER

### 9.1 Research Staff

J.D. Webb (Senior Chemist), Center Manager D.E. King (Staff Chemist)
E.J. Johnson, DOE/AWU Student Fellow

### 9.2 Introduction

The FTIR Spectroscopic Research Center began operations in the NREL Measurements and Characterization Branch at the beginning of FY1991. The center was established to satisfy capital equipment requests from a number of NREL researchers for Fourier Transform Infrared (FTIR) analytical capabilities. These requests originated in the former Materials Science and Engineering Division, the Mechanical and Industrial Technology Division, and the Fuels and Chemicals Research and Engineering Division. The diverse sources of these requests gave acquisition of FTIR equipment a high priority. The \$325K in GPE funds used to purchase and install the center's equipment in 16/387 will be augmented by \$25K in capital equipment funds from the FY1992 DOE National Photovoltaics Program, and by \$15K in equipment provided by WFO sponsors. The equipment and its capabilities were described in the FY1990 Branch Annual Report.

The center operates as a recharge center at an FY1992 rate of \$85/hr. The Center provides vibrational absorbance and Raman spectroscopic analysis to a number of customers, both inside and outside NREL. The Center also provides surface analysis services (primarily X-ray photoelectron spectroscopy, or XPS), using equipment in 16/384 generously made available by the Surface Interactions, Modifications, and Stability Group. The latter group's equipment was described in a previous chapter of this report. Rates for similar services charged by outside laboratories offering FTIR spectroscopy range from \$95/hr to \$200/hr. The laboratories at the lower end of this range have equipment inferior in many respects to that available in the center, and no private, commercial, or university laboratory in the Rocky Mountain region currently has as complete an array of FTIR spectroscopic equipment as NREL's FTIR center. This versatility was mandated by the diverse range of analytical needs and research interests of NREL scientists, and makes this laboratory a true center of excellence in FTIR spectroscopy. The primary components of the recharge cost are staff salaries, 16/387 floor space, and equipment maintenance. The recharge mode of operation is equitable in the sense that it reflects the real costs of analyses performed or supervised by experienced staff, minus the very significant costs of the equipment itself. The recharge mode also guarantees access to the center's capabilities on an equal basis to any NREL researcher.

The center's recharges and level of service to customers will roughly double in FY1992 relative to FY1991. Our abilities to offer this increased level of service will be greatly enhanced by the addition of David King to our staff. King is a graduate of University of South Florida in analytical chemistry, and will join the center staff in FY1992 following completion of his postdoctoral work in our Surface Interactions, Modifications, and Stability Group.

The center's principal customers in FY1991 were Science Applications International Corporation (SAIC), the amorphous silicon and module reliability research subtasks of the NREL PV Program, the NREL Solar Detoxification Research Program, and the NREL Materials Processing Subtask. We expect our activities in support of the PV Program to increase substantially in FY1992, especially with regard to support of the PV industry. The WFO contract with SAIC, now in its second year, allows us to provide analytical support to the Propellant Bondlines Subactivity of the NASA Solid Rocket Motor (SRM) Program. Our work on this contract included FTIR analysis of contaminants in the bondlines that secure the solid propellant to the case of SRM's, such as the NASA space shuttle and Titan boosters, as well as in-situ FTIR and microwave monitoring of curing reactions in the polymeric components of the bondlines.

#### Accomplishments

 We performed quantitative analyses of contaminants on various surfaces of an SRM bondline, using FTIRattenuated total reflection (ATR) and diffuse reflection (DR) spectroscopy. The data in Figure 9.1 show that a

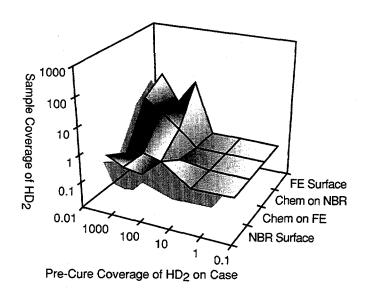


Figure 9.1

grease used as a preservative on the steel SRM cases has migrated through the bondline case primer to the adjacent insulation surface during cure of the insulation at elevated temperatures, and that extent of migration is a function of the level of case contamination by the grease. The sensitivity of the FTIR analytical methods is less than 1  $\mu g/cm^2$  for several bondline contaminants, or approximately an order of magnitude higher than other techniques, e.g., surface analysis, used by the SRM industry.

 We performed quantitative analyses of III-V impurities and dopants in crystalline silicon in support of the PV industry. The far-infrared spectrum of one such sample, recorded at a temperature of 8 K using our cryostatic sample mount, is shown in Figure 9.2. This spectrum reveals the presence of boron and aluminum in the sample at approximately the 10<sup>14</sup>/cm<sup>3</sup> level.

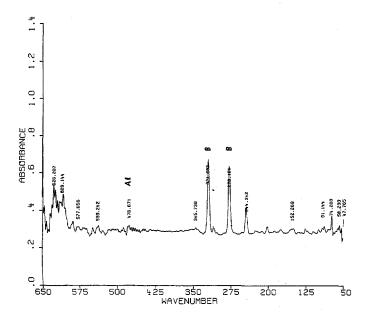


Figure 9.2

### APPENDIX A: STAFF BIOGRAPHIES

Fouad Abou-Elfotouh (NREL 1986): Senior Scientist. Research and support interests and responsibilities: electro-optical properties (including defect states and their behavior) of semiconductor (thin films and single crystals); fabrication, characterization, and modeling of heterojunction devices, solar cell back contacts; and development of spectroscopic ellipsometer.

Richard K. Ahrenkiel (NREL 1981): Principal Scientist and Group Leader. Research and support interests and responsibilities: materials characterization by photoluminescence and diffusion measurements; deep-level-transient-spectroscopy; C-V measurements; and superconductor characterization.

Mowafak M. Al-Jassim (NREL 1983): Senior Scientist and Group Leader, Materials Science. Research and support interests and responsibilities: transmission electron microscopy (TEM); scanning electron microscopy (SEM); defect analysis in semiconductors, and epitaxial growth by MOCVD and MBE.

Sarah (Sally) Asher (NREL 1985): Senior Scientist, Analytical Chemistry. Research and support interests and responsibilities: surface science; operation and maintenance of SIMS facility.

Bonnie J. Bachman (NREL 1991): Research Associate. Research interests and responsibilities: polymer degradation and processing; metal ion catalyzed degradation of EVA copolymer; thesis project; TG and related methods; DSC and TMA; high temperature polymers for electronics and packaging.

Linda Barr (NREL 1991): Associate Chemist. Research interests and responsibilities: dissection and analysis of PV modules; UV-vis measurements; gel content determination; extraction of fresh and field-degraded polymers.

Elvira Beck (NREL 1987): Master Technician. Research and support interests and responsibilities: photovoltaic cell measurements and characterization by area, spectral response; and quantum efficiency; intensity, current vs. voltage, and reference cell calibrations.

Riyad Ahmad Bitar (NREL 1991): Visiting Scientist Laser Spectroscopy. Research and support interests and responsibilities: electro-optical characterization of semiconductors; materials characterization by photoluminescence; and electrical relaxation in semiconductors and insulators.

Michael H. Bode (NREL 1992): Senior Scientist. Research interests and responsibilities: transmission electron microscopy (TEM); scanning electron microscopy (SEM); structural, electrical and optical characterization of semiconductors; semiconductor interface analysis

Timothy J. Coutts (NREL 1984): Principal Scientist and Group Leader. Research and support interests and responsibilities: materials and devices fabrication and characterization; development of new analytical equipment; modeling of terrestrial and space solar cells.

Alvin W. Czanderna (NREL 1978): Research Fellow and Group Leader. Research and support interests and responsibilities: surface science; reactions at interfaces; stability and durability of multilayer thin film devices; interactions at polymer/metal (oxide) interfaces; surface analysis; ultramicrogravimetry.

Ramesh Dhere (NREL 1985): Staff Scientist. Research and support interests and responsibilities; optical and structural characterization of materials; analysis and modeling of devices.

Patricia C. Dippo (NREL 1980): Administrative Assistant. Research and support interests and responsibilities: supports five of the groups within the branch and prepares branch correspondence; reports; etc.; organizes day-to-day branch operations.

Donald J. Dunlavy (NREL 1981): Staff Scientist. Research and support interests and responsibilities: minority carrier characterization by photoluminescence and diffusion measurements; design, modification; and maintenance of all test equipment.

Keith A. Emery (NREL 1980): Senior Scientist and Group Leader. Research and support interests and responsibilities: photovoltaic performance characterization by current versus voltage, temperature; spectrum, and intensity; PV calibration; quantum efficiency; PV standards.

Clark L. Fields (NREL 1990) Visiting Professional (Summer), Prof. of Chemistry at the University of Northern Colorado, Greeley. Research and support interests and responsibilities: rapid thermal processing of materials; metalorganic chemistry; boron chemistry; chemical vapor deposition; ceramic superconductors.

Amy B. Swartzlander-Franz (NREL 1980): Associate Scientist. Research and support interests and responsibilities: characterization of solar and electronic devices, including superconductors, using SAM; AES, and XPS.

Timothy A. Gessert (NREL 1984): Staff Scientist. Research and support interests and responsibilities: device processing technology; optical and electrical thin film deposition and characterization; metallization science; solar cell grid and antireflection technology modeling and optimization.

John P. Goral (NREL 1986): Staff Scientist. Research and support interests and responsibilities: transmission electron microscopy; electron microprobe analysis; structure and properties of photovoltaic materials; defects and interfaces in semiconductors. Deceased July 1991.

Falah Hasoon (NREL 1990): Visiting Professional. Research and support interests and responsibilities: scanning electron microscopy; luminescent properties, and defect examination in semiconductors.

Greg Herdt (NREL 1991): Research Associate. Research and support interest and responsibilities: applied surface science; sorption phenomena of desiccant materials using microgravimetric and flow techniques; computerized evaluation and modeling of sorption processes.

Kim M. Jones (NREL 1981): Staff Scientist. Research and support interests and responsibilities: transmission electron microscopy; analytical electron microscopy; defect analysis in semiconductors.

Elizabeth (Jo) Johnson (NREL 1990): AWU Student Fellow. Research and support interests and responsibilities: Fourier transform infrared (FTIR) spectroscopy and quantitative analysis.

Lawrence L. Kazmerski (NREL 1977): Principal Scientist and Branch Manager. Research and support interests and responsibilities: scanning tunneling microscopy; surface and interface analysis research.

Brian M. Keyes (NREL 1988): Associate Engineer. Research and support interests and responsibilities: minority carrier characterization by photoluminescence and diffusion measurements; modeling of the above measurements; computer analysis and interfacing.

David King (NREL 1989): Staff Analytical Chemist. Research and support interests and responsibilities: surface analysis; FTIR spectroscopy; thin films and interfaces.

Dean Levi (NREL 1990): Research Associate. Research and support interests and responsibilities: time-resolved photo-luminescence measurements of minority carrier lifetime; photon recycling; surface recombination velocity; chemical passivation of surface defects. Also extending laboratory capabilities through development of new measurement techniques.

Xiaonan Li (NREL 1985): Postdoctoral Associate. Research and support interests and responsibilities: device processing; optical and electrical thin film deposition and characterization; metallization vacuum technology.

Alice Mason (NREL 1984): Master Technician. Research and support interests and responsibilities: sample preparation for microscopy examination.

Richard Matson (NREL 1979): Staff Scientist. Research and support interests and responsibilities: scanning electron microscopy; X-ray microanalysis; defect examination in semiconductors and luminescent properties.

Helio Moutinho (NREL 1991): Research Associate. Research and support interests and responsibilities: materials characterization by scanning tunneling microscopy.

Art J. Nelson (NREL 1985): Senior Scientist and Group Leader. Research and support interests and responsibilities: surface and interface analysis research; surface and grain boundary phenomena in semiconducting and superconducting materials; synchrotron radiation photoemission on crystalline; polycrystalline, and amorphous materials; growth and characterization of II-VI semiconductors; plasma modification of surfaces.

David W. Niles (NREL 1991): Staff Scientist. Research and support interests and responsibilities: characterization of photovoltaic materials; devices and modules; surface and interface phenomena; measurement-system development.

Carl Osterwald (NREL 1982): Staff Scientist. Research and support interests and responsibilities: measurement system design and development; primary photovoltaic reference cell calibration; spectral response; current versus voltage measurements.

F.J. Pern (NREL 1984): Senior Scientist. Research and support interests and responsibilities: material fabrication and characterization; spectral, analytical, and electrochemical characterization of PV encapsulant polymers, metalization, and other module materials; module performance testing and analysis of failure mechanisms.

J. Roland Pitts (NREL 1979) Senior Physicist. Research and support interest and responsibilities: surface modification using particle beams; surface and interface science; stimulated desorption; transient thermal processes; optical and physical properties of thin films.

Kannan Ramanathan (NREL 1989): Senior Scientist. Research and support interests and responsibilities: fabrication and characterization of polycrystalline thin films and devices; transparent conducting oxides; optical and electrical measurements.

Elizabeth (Jo) Rouse (NREL 1990): AWU Student Fellow. Research and support interests and responsibilities: Fourier Transform Infrared (FTIR) spectroscopy and quantitative analysis.

Lorenzo L. Roybal (NREL 1982): Master Technician. Research and support interests and responsibilities: designs, modifies, machines, maintains, and operates characterization systems for superconductivity, DLTS, C-V/G-V and other test stations.

Peter Sheldon (NREL 1979): Senior Scientist. Research and support interests and responsibilities: thin film and semiconductor device research growing a variety of III-V and group IV semiconductors by molecular beam epitaxy (MBE) and electrical characterization of these materials.

Yvonne Shinton (NREL 1984) Master Technician. Research and support interests and responsibilities: metallography; optical microscopy; X-ray diffraction; vacuum microbalance measurements. [Mechanical and Industrial Technology Division].

Mark W. Wanlass (NREL 1980): Staff Scientist. Research and support interests and responsibilities: metalorganic vapor phase epitaxy; device design, fabrication, characterization and modeling.

Scott Ward (NREL 1989): Research Associate. Research and support interests and responsibilities: device processing and design; generation of photolithographic masks and application of Entech prismatic covers; tandem solar cell development.

John D. Webb (NREL 1978): Senior Chemist and Center Manager. Research and support interests and responsibilities: Fourier Transform Infrared (FTIR) spectroscopy; polymer chemistry; and photochemistry; operation, maintenance, and business development for the FTIR Spectroscopic Research and Service Center.

Regina Witherspoon (NREL, 1990): Administrative Assistant. Research and support interests and responsibilities: supports Materials Durability and Component Reliability Group and the Surface Interactions Modifications, and Stability Group by the preparation of correspondence, reports, etc.; day-to-day group operations.

Jane Zhu (NREL, 1991): Research Associate. Research and support interests and responsibilities: Materials science: transmission electron microscopy; structure and properties of materials, defects, and interfaces in heterostructures.

# APPENDIX B: COOPERATIVE LABORATORIES

Advanced Photovoltaic Systems Ain Shams University, Egypt Airtech Corporation

Alabama Power Company

Argonne National Laboratory

Arizona State University

Armco Research and Technology Applied Solar Energy Corporation

Astropower, Inc.

AT&T Bell Laboratories

Ben Gurion University, Israel

Boeing Aerospace Company

Brooklyn Polytechnic Institute

Brookhaven National Laboratories

**Brown University** 

California Institute of Technology

Cargocaire Engineering, Inc.

Carnegie-Mellon University Chronar Corporation Colorado

Colorado School of Mines

Colorado State University

Coors Ceramic

Cornell University

Crystal Systems, Inc.

**Duke University** 

**Eaton Corporation** 

**EIC** Laboratories

Electrotechnical Laboratory, Japan

ENEA, Italy

**Energy Conversion Devices** 

ENTECH, Inc.

Electric Power Research Institute

**EXXON Research** 

Florida Solar Energy Center

Ford Aerospace

Franhofer Institute for Solar Energy Germany

Fuji Electric, Japan

Georgia Institute of Technology

Georgia Tech Research Institute

Glasstech Solar

Grumman Aerospace Corporation

**GTE Laboratories** 

Harvard University

**Howard University** 

Hughes Research Laboratories

**IBM** 

Indian Institute of Technology, Delhi

Institute of Energy Conversion

Institute for Non-Ferrous Metals, Beijing, China

Instituto Militar de Engenharia, Brazil

International Energy Foundation

International Solar Electric Technologies

Iowa State University

Jet Propulsion Laboratory

King Abdul-Aziz University, Saudi Arabia

Kopin Corporation

Lawrence Berkeley Laboratories

Louisiana State University

Mankato State University

Man-Tech Development, Inc.

Martin Marietta

Massachusetts Institute of Technology

Materials Research Group

Matsushita, Japan

MIT Lincoln Laboratory

Mobil Solar Energy Corporation

Montana State University

Motorola

NASA Lewis Research Center

National Institute for Silicon Technology, Pakistan

National Physical Laboratory, New Delhi, India

National Institute for Standards and Technology

Naval Research Laboratories

Newcastle-Upon-Tyne Polytechnic, England

Nippon Telegraph & Telephone, Japan

North Carolina State University

Oak Ridge National Laboratory

Oregon State University

Osaka University, Japan Pacific Gas and Electric

Pennsylvania State University

Photon Energy, Inc.

Princeton University

PTB, Braumschweig, Germany

**Purdue University** 

Rensselaer Polytechnic Institute

Research Triangle Institute

Sandia National Laboratories

Santa Barbara Research Center

Sanyo, Japan

Science Applications International Corporation

Shanghai Institute of Metallurgy, China

Siemens Solar Industries

State University of New York

Solar Cells, Inc.

Solarex

Solec International, Inc.

Spectrolab

SPIRE Corporation

Stanford University

Syracuse University

Texas Instruments, Inc.

Tideland Signal, Inc.

University of Colorado

University of Delaware

University of Florida

University of Illinois

University of North Carolina

University of Oregon

University of Sao Paulo, Brazil

University of Southern California
University of South Florida
University of Texas
University of Toledo
University of Utah
University of Washington
University of Wisconsin
Utility Power Group
United Solar Systems Corporation
Varian Associates
Washington State University
Weizmann Institute of Science, Israel
Westinghouse Electric Corporation
Wuhan University, China
Xerox PARC

# APPENDIX C: BIBLIOGRAPHY

- Abou-Elfotouh, F.A.; Horner, G.S.; Coutts, T.J.; and Wanlass, M.W.; "Broad Band Spectroscopic Ellipsometry for the Characterization of Photovoltaic Materials", Proc. 10th PV AR&D Meeting, Lakewood, Colorado, October 23-25, 1990.
- Abou-Elfotouh, F.; Moutinho, H.; Bakry, A.; and Coutts, T.J.; "Characterization of the Defect Levels in Copper Indium Diselenide", Proc. 10th PV AR&D Meeting, Lakewood, Colorado, October 23-25, 1990.
- Abou-Elfotouh, F.A.; Kazmerski, L.L.; Moutinho, H.R.; Nelson, A.J.; and Bakry, A.M.; "Determination and Observation of Electronic Defect Levels in CulnSe<sub>2</sub> Crystals and Thin Films", Proc. 37th National Symposium of the American Vacuum Society, Toronto, Canada, October 8-12, 1990, J. Vac. Sci. Technol., A9 (3), May/ June 1991.
- Abou-Elfotouh, F.A.; Soliman, M.; Riad, A.E.; Al-Jassim, M.M.; and Coutts, T.J.; "Preparation and Characterization of Polycrystalline rf Sputtered CdTe Thin Films for PV Application", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Ahrenkiel, R.K.; Keyes, B.M.; Wang, L.; and Albright, S.P.; "Minority-Carrier Lifetime of Polycrystalline CdTe in CdS/CdTe Solar Cells", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Ahrenkiel, R.K.; Keyes, B.M.; "Hole Lifetime in n-Type GaAs", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- Al-Jassim, M.M.; Ahrenkiel, R.K.; Wanlass, M.W.; Olson, J.M.; and Vernon S.M.; "Structural and Luminescent Characterization of InP and GaInP on Si", Proc. 10th PV AR&D Meeting, Lakewood, Colorado, October 23-25, 1990.
- Asher, S.E.; Branz, H.M.; and Nelson, B.P.; "SIMS Measurements of Light-Induced Deuterium Diffusion in Amorphous Silicon", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- Asher, S.E.; "SIMS Investigations of Substrate/Film Interdiffusion in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> Films Grown on SrTiO<sub>3</sub> and LaAlO<sub>3</sub>", J. Vac. Sci. Technol., A9(3), May/June 1991.

- Baouchi, W.A.; and Nelson, A.J.; "EELS Analysis of Thin ARC Evaporated Diamond Like Carbon Films", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- Beal, J.A.; Cromar, M.W.; Harvey, T.E.; Johansson, M.E.; Ono, R.H.; Reintsema, C.D.; Rudman, D.A.; Nelson A.J.; Asher, S.E.; and Swartzlander, A.B.; "YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>-d/Insulator Multilayers for Crossover Fabrication, IEEE Trans. Magnetics 27 (2), 1596 (1991).
- Coutts, T.J.; Wanlass, M.W.; Gessert, T.A.; Li, X.; and Ward, J.S.; "Progress in InP-Based Solar Cells", Proc. 3rd International Conference on InP and Related Materials, Cardiff, Wales, April 8 - 11, 1991.
- 13. Czanderna, A.W.; "Aspects of Surface Science for the Physical Chemistry Curriculum", NREL/TP-213-4500, December, 1991.
- 14. Czanderna, A.W.; "Durability Issues: Electrochromic Devices and Windows", Internal Report, Dec. 1991.
- 15. Czanderna, A.W.; and Landgrebe, A.; Proc. Surface Processing Workshop, 1991.
- Czanderna, A.W.; "Aspects of Surface Science for the Physical Chemistry Curriculum", NREL/TP-213-4500, Dec. 1991; for R. Moore and R. Schwenz, eds., Revitalizing Phyiscal Chemistry Lecture and Laboratory Courses, ACS Books, Amer. Chem. Soc., Washington, DC.
- Czanderna, A.W.; "XPS of Organized Molecular Assembly/Copper Interfaces", Proc. 37th National Symposium of the American Vacuum Society in Toronto, October 8-12, 1990, J. Vac. Sci. Technol., A9 (3), May/June 1991.
- 18. Czanderna, A.W.; Herdt, G.C.; and Tillman, N.T.; "Polymers as Advanced Materials for Desiccant Applications", NREL/TP-213-4695, December (1991).
- 19. Czanderna, A.W.; and Hercules, D.; "Ion Spectroscopies for Surface Analysis, " Eds., Plenum, New York, NY, (1991).
- Czanderna, A.W.; King, D.E.; and Spaulding, D.; "Metal Overlayers on Organic Functional Groups of Self Organized Molecular Assemblies. 1. XPS of Interactions of Cu/COOH on 11-Mercaptoundecanoic Acid Self Assembled Monolayers", J. Vac. Sci. Technol., A9 (1991), pp. 2607.

- Czanderna, A.W., and Neidlinger, H.H.; "Polymers as Advanced Materials for Desiccant Applications. 2. Alkali Salts of Polystyrene Sulfonic Acid", ASHRAE Transactions, 97 (Part 2), (1991) pp. 615-623 (or SERI/ TP-213-4137, January 1991).
- Dhere, R.G.; Ramanathan, K.; and Coutts, T.J.; "Optical Characterization of CulnSe<sub>2</sub> Solar Cells Obtained by the Selenization Method", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Ekin, J.W.; Russek, S.E.; Jeanneret, B.; Nelson, A.J.; and Swartzlander, A.B.; "In-situ Noble Metal/YBCO Thin-Film Contact Interface Studies", Proc. Materials Research Society Spring Meeting, Anaheim, CA, April 29 - May 3 (1991).
- Emery, K.A.; "Efficiency Measurements of Photovoltaic Devices", Chapter 3, Advances in Solar Energy, 1992.
- Frass, L.M.; Avery, J.E.; Gruenbaum, P.E.; Sundaram, V.S.; Emery, K.; and Matson, R.; "Fundamental Characterization Studies of GaSb Solar Cells", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Gessert, T.A.; Li, X.; Coutts, T.J.; and Tzafaras, N.; "Pilot Production of 4cm<sup>2</sup> ITO/InP Photovoltaic Solar Cells", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- 27. Gessert, T.A.; and Coutts, T.J.; "Grid Metallization and Antireflection Coating Optimization for Concentrator and One-Sun Photovoltaic Solar Cells", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- Gessert, T.A.; X. Li; and T.J. Coutts; "Fabrication and Performance Analysis of 4cm<sup>2</sup> ITO/InP Photovoltaic Solar Cells", Proc. 3rd International Conference on InP and Related Materials, Cardiff, Wales, April 8 - 11, 1991.
- Gessert, T.A., Li, X., Phelps, P.W.; Coutts, T.J.; and Tzafaras, N.; "Fabrication and Performance Analysis of 4-cm<sup>2</sup> ITO/InP Photovoltaic Solar Cells", Proc. 11th Space Photovoltaic Research and Technology (SPRAT XI), NASA Lewis Research Center, May 7-9, 1991.
- Gessert, T.A.; Li, X.; and Coutts, T.J.; "Practical Guidelines for Grid Metallization in Photovoltaic Solar Cell Research", Proc. 10th PV AR&D Meeting, Lakewood, Colorado, October 23-25, 1990.

- Goral, J.P.; Al-Jassim, M.M.; and Jones, K.M.; "TEM of CuInSe<sub>2</sub> and CdTe Polycrystalline Thin Films", Proc. 10th PV AR&D Meeting, Lakewood, Colorado, October 23-25, 1990.
- 32. Herdt, G.C.; and Pesaran, A.A.; "Measurement of Sorption Properties of Polymeric Desiccants Using the NREL Desiccant Sorption Test Facility", NREL Progress Report for FY 1991, Dec. 1991.
- 33. Hernandez-Calderon, I.; Melendez-Lira, M.; Niles, D.W.; and Höscht, H.; "Characterization of CdTe/GaAs (100) Heterostructures by Angle Resolved Photoemission, Electron Diffraction and Photoreflectance Spectroscopy", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- 34. Höscht, H.; and Niles, D.W.; "Strain Induced Band-Gap Opening in Epitaxial a-Sn Films", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- Kazmerski, L.L.; "Atomic Imaging, Processing and Nanocharacterization of Semiconductor Surfaces", Proc. International Symposium on Vacuum Science, Thin Film and Surface Science, Wuxi, China, September 16-19, 1991.
- 36. Kazmerski, L.L.; "Photovoltaics: From Atoms to Arrays", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- Kazmerski, L.L.; and Emery, K.E.; "Status and Assesment of the Photovoltaic Technologies," updated version of L.Kazmerski, International Materials Reviews, Vol. 34, p. 185, 1989. Proc. Annual Association for the Advancement of Science Meeting, Washington, DC, on Februry 14, 1991.
- King, D.E.; Czanderna, A.W.; and Spaulding D.; "Interactions of Deposited Copper on COOH of 11-Mercaptoundecanoic Acid: An XPS Study", Proc. Electrochem. Soc. Meeting, Oct. 14-19, 1991 and SERI/TP-213-4376, September (1991).
- 39. King, D.E.; Czanderna, C.W.; and Spaulding D.; "Charging Shifts During XPS Analysis of the CU/Arachidic Acid Interface on Aluminum Oxide Films", J. Vac. Sci. and Technol. 2/92 and NREL TP-3120-4724.
- 40. Molenbroek, E.; Waddington, D.W.; and Emery, K.A.; "Hot Spot Susceptibility and Testing of PV Modules", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.

- 41. Nann, S.; and Emery, K.A.; "A Numerical Analysis of PV-Rating Methods", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Naziripour, A.; Dong, C.; Drexler, J.W.; Swartzlander, A.B.; Nelson, A.J.; and Hermann, A.M.; "Tl-Ba-Ca-Cu-O Superconducting Thin Films with Post-Deposition Processing Using Tl-containing Thin Films as Tl Source", J. Appl. Phys. 70(10), 6495 (1991).
- 43. Nelson, A.J.; Frigo, S.; Mancini, D.; Rosenberg, R.A.; "Photoemission Characterization of the H<sub>2</sub> Plasma Etched Surface of InP", J. Appl. Phys. 70(10), 5619 (1991).
- Nelson, A.J.; Niles, D.W.; and Kazmerski, L.L.; "Photoemission Study on the Formation of Mo Contacts to CuInSe<sub>2</sub>", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- Nelson, A.J., "Soft X-ray Photoemission Investigation of the CdS/CuInSe<sub>2</sub> Heterojunction Interface", 5th International Photovoltaic Science and Engineering Conference (PVSEC-5) in Kyoto, Japan, November, 26–30 (1990).
- Nelson, A.J.; Gebhard, S.; Rockett, A.; Colavita, E.; Englehardt, M.; and Höscht, H.; "Synchrotron Radiation Photoemission Study of CdS/CuInSe<sub>2</sub> Hetero-junction Formation", Phys. Rev. B42 (11), 7518 (1990).
- Nelson, A.J.; Gebhard, S.; Kazmerski, L.L.; Colavita, E.; Englehardt, M.; and Höscht, H.; "Characterization of the Native Oxide of CuInSe<sub>2</sub> Using Synchrotron Radiation Photoemission", Appl. Phys. Lett. 57(14), 1428 (1990).
- 48. Nelson, A.J.; "Formation and Schottky Barrier Height of Au Contacts to CuInSe<sub>2</sub>", Proc. 37th National Symposium of the American Vacuum Society in Toronto, October 8-12, 1990, J. Vac. Sci. Technol., A9 (3), May/ June, 978 (1991)
- Nelson, A.J.; "AES Line-Shape Analysis at Grain Boundaries of Oriented Grained YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>", Proc. 37th National Symposium of the American Vacuum Society in Toronto, Canada, October 8-12, 1990, J. Vac. Sci. Technol., A9 (3), May/June 1991.
- 50. Niles, D.W.; and Höscht, H.; "The Electronic Valence Band Dispersions of Cd<sub>0.8</sub>Zn<sub>0.2</sub>Te Along the (001) direction in K-Space", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.

- Osterwald, C.R; "ASTM Photovltaic Standards Development Status", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- 52. Osterwald, C.R.; Wanlass, M.W.; Ward, J.S.; Keyes, B.M.; Emery, K.A.; and Coutts, T.J.; "Modeled Performance of Monolithic, 3-Terminal InP/Ga<sub>0.47</sub>In<sub>0.53</sub>As Concentrator Solar Cells as a Function of Temperature and Concentration Ratio", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Osterwald, C.R.; and C. Riordan, "Solar Spectral Irradiance: Analysis and Modeling", Proc. 10th PV AR&D Meeting, Lakewood, Colorado, October 23-25, 1990.
- 54. Pern, F.J.; "Luminescence and Absorption Characterization of the Structural Effects of Thermal Processing and Weathering Degradation on Ethylene Vinyl Acetate (EVA) Encapsulant for PV Modules", NREL/TP-412-4605, Nov. 1991.
- 55. Pern, F.J.; and Czanderna, A.W.; "Characterization of Ethylene Vinyl Acetate (EVA) Encapsulant: Effects of Thermal Processing and Weathering Degradation of its Discoloration", Solar Energy Materials and Solar Cells, 25, (1992), pp. 3-23.
- 56. Pern, F.J.; Czanderna, A.W., Emery, K.A.; and Dhere, R.G.; "Weathering Degradation of EVA Encapsulant and the Effect of its Yellowing on Solar Cell Efficiency", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada, pp. 557-561.
- 57. Pern, F.J.; Noufi, R.; Mason, A.M.; and Franz, A.B.; "Characterizations of Electro-Deposited CulnSe<sub>2</sub> Thin Films: Structure, Deposition and Formation Mechanisms", Thin Solid Films, 202, (1991), pp. 299-314.
- 58. Perry, A.J.; Sartwell, B.D.; Valvoda, V.; Rafaja, D.; Williamson, D.L.; and Nelson, A.J.; "Residual Stress and the Effect of Implanted Argon in Films of Zirconium Nitride Made by Physical Vapor Deposition", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- 59. Pitts, J.R.; Fields, C.L.; Tracy, E.; and Shinton, Y.; "Surface Modification Methods: Current Status of Using Highly Concentrated Solar Radiation", Proc. Workshop on Surface Processing and Applications to Transportation and Utilities Technologies, Dec. 10-12, 1991, Dearborn, MI.
- Pitts, J.R.; Stanley, J.T.; Tracy, E.; and Fields, C.L.;
   "Surface Modification Technologies Using Concentrated Solar Radiation", Proc. Workshop on Potential

- Applications of Concentrated Solar Energy, Nov. 7-8, 1990, National Academy Press, (1991), ISBN 0-309-04577-0, pp. 78-83.
- Pitts, J.R.; Tracy, E.; and Shinton, Y.; "Surface Modification Using Concentrated Solar Radiation", Proc. 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- 62. Ramanathan, K.; Dhere, R.G.; and Coutts, T.J.; "A Study of ITO/CdS/ CuInGaSe<sub>2</sub> Thin Film Solar Cells", 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Rohde, S.L.; Sproul, W.D.; Swartzlander, A.B.; Mason, A.; and Nelson, A.J.; "Characterization of TiN Films Deposited Using Multi-cathode Unbalanced Magnetrons", 38th National Symposium of the American Vacuum Society, Seattle, Washington, November 11-15, 1991.
- 64. Roshko, A.; Ono, R.H.; Beal, J.A.; Moreland, J.; Nelson, A.J.; and Asher, S.E.; "Morphology of Silver on YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>-d Thin Films, IEEE Trans. Magnetics 27 (2), 1616 (1991).
- 65. Stanley, J.R.; Fields, C.L.; and Pitts, J.R.; "Solar Induced Surface Transforamtion of Materials", Advanced Materials & Processes, **138** (6), 16 (1990).
- Shen, C.C.; Chang, P.T.; and Emery, K.A.; "Two-Terminal Monolithic InP/InGaAsP Tandem Solar Cells with Tunneling Intercell Ohmic Connections", Proc. 3rd International Conference on InP and Related Materials, Cardiff, Wales, April 8-11, (1991).
- 67. Shen, C.C.; Chang, P.T.; and Emery, K.A.; "Two-Terminal Monolithic InP/InGaAsP Tandem Solar Cells with Tunneling Intercell Ohmic Connections", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Thakur, R.P.S.; Singh, R.; Nelson, A.J.; and Swartzlander, A.B.; "Role of in-situ Rapid Isothermal Processing in Advanced III-V Technology", J. Appl. Phys. 70(7), 3857 (1991).
- 69. Wanlass, M.W.; "Monolithic Tandem Solar Cell", U.S. Patent Number 5,019, 177, May 28, 1991.
- Wanlass, M.W.; Coutts, T.J.; Ward, J.S.; Emery, K.A.; Gessert, T.A.; and Osterwald, C.R.; "Advanced High-Efficiency Concentrator Tandem Solar Cells", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.

- Wanlass, M.W.; Coutts, T.J.; Ward, J.S.; and Emery, K.A.; "High-Efficiency Heteroepitaxial InP Solar Cells", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- Wanlass, M.W.; Ward, J.S.; Coutts, T.J.; Emery, K.A.; Gessert, T.A.; and Osterwald, C.R.; "Monolithic InP/ Ga<sub>0.47</sub>In<sub>0.53</sub>As Tandem Solar Cells for Space", Proc. 11th Space Photovoltaic Research and Technology (SPRAT XI), NASA Lewis Research Center, May 7-9, 1991.
- Wanlass, M.W.; Coutts, T.J.; Ward, J.S;. and Emery, K.A.; "High-Efficiency Heteroepitaxial InP Solar Cells", Proc. 11th Space Photovoltaic Research and Technology (SPRAT XI), NASA Lewis Research Center, May 7-9, 1991.
- Wanlass, M.W.; Coutts, T.J.; Ward, J.S.; Emery, K.A.; and Horner, G.S.; "High-Efficiency, Thin-Film InP Concentrator Solar Cells", Proc. 3rd International Conference on InP and Related Materials, Cardiff, Wales, April 8 - 11, 1991.
- Wanlass, M.W.; Ward, J.S.; Emery, K.A.; Gessert, T.A.; Osterwald, C.R.; and Coutts, T.J.; "High-Performance Concentrator Tandem Solar Cells Based on Infrared-Sensitive Bottom Cells", Proc. 10th PV AR&D meeting, Lakewood, Colorado, October 23-25, 1990.
- 76. Ward, J.S.; Wanlass, M.W.; Coutts, T.J.; Emery, K.A.; and Osterwald, C.R.; "InP Concentrator Solar Cells", Proc. 22nd IEEE Photovoltaic Specialists Conference, October 7-11, 1991, Las Vegas, Nevada.
- 77. Ward, J.S., Wanlass, M.W., Coutts, T.J. and Emery, K.A., SERI, "InP Concentrator Solar Cells for Space Applications", Proc. 11th Space Photovoltaic Research and Technology (SPRAT XI), NASA Lewis Research Center, May 7-9, 1991.

# **APPENDIX D: INSTRUMENTATION**

#### SURFACE AND INTERFACE ANALYSIS LABORATORY

SURFACE AND INTERFACE ANALYSIS LABORATORY		
Instrument	Range/Capacity	Unique Features and Uses
XPS/Auger System (Perkin Elmer PHI 55)	2 mm spot size detection sensitivity of 0.1 at. % Li to U, 1 cm <sup>2</sup> sample size.	Performs AES, XPS, UPS, EELS in conjunction with depth profiling quantitative elemental analysis and chemical bonding information, angle resolved photoemission
Scanning Auger Microprobe (Perkin-Elmer PHI 590)	0.1 µm spot size, SAM detection sensitivity of 0.1 at. % for Li to U, SIMS detection of 1 ppm for H to U, 1 cm <sup>2</sup> sample size	Performs SAM and SIMS analysis with depth profiling, quantitative elemental analysis, and surface compositional maps, up to 5000X micrographs of surface features
	MATERIALS CHARACTERIZATION GROU	P
Instrument	Range/Capacity	Unique Features and Uses
Electron probe X-ray microanalyzer (EPMA) Cameca-MBX	EDS and WDS analysis of an accuracy of ±0.5%	Quantitative compositional analysis of all elements heavier than boron
SEM JEOL JSM-35C	1-49 kV, secondary electron imaging (SEI) 5 nm res. backscattered elect. iomaging (BEI) 9 nm res	EDS: compositional analysis (>Na); EC: Crystalline type orientation and quality; EBIC: Microcharacterization of the electrical activity of electronic materials, junction location, and diffusion length measurement
SEM JEOL JSM-840	0.2-40 kV, liquid helium catholdoluminescence (CL) cold stage. Integrated and spectral CL from 10°C to RT°, from 300 nm to 1.8 μm	Characterize relative impurity concentrations, defect densities and distributions, bandgap (Eg), and subbandgap (defect) luminescence with high resolution and correlation with topography
TEM Philips CM - 30	KeV: 300, resolution: 2.3Å, Tilt: ±60°	Performs structural, analytical, and high resolution examination of a wide range of materials
	DEVICE DEVELOPMENT GROUP	
System	Range/Capacity	Unique Features and Uses
Atmospheric Pressure Metal Organic Vapor Phase Epitaxy		State-of-the-art home-built system using a patented reactor vessel design; highly uniform epilayers and abrupt heterojunctions have been demonstrated. All III-V alloys from the Ga-In-As-P family have

		been synthesized doped n- or p- type. High performance solar cells and customdiagnostic test structures have also been fabricated in these materials
UV/VIS/NIR Spectrophotometer (Beckman Model 5240)	180 nm - 3200 nm	Performs specular transmittance and absorbance measurements; with integrating sphere attachment, also measures total or specular reflection.
IR Spectrophotometer (Perkin Elmer Model 580B)	180 nm - 600 cm <sup>-1</sup>	Measures transmittance in the IR range
Research Ellipsometer (Rudolph Model 43603-200E)	Thickness measurements of 10 Å to 50,000 Å. Measures also N and k	Precision measurements of thickness and refractive index of highly reflective substrates or thin films
Production Ellipsometer (Gaertner Model L116)	Thickness measurements of 10 Å to 50,000 Å. Also measures N and k	Rotating analyzer allows automatic operation for use in trend analysis and uniformity studies
Hall Measurement Apparatus (Alpha Scientific)	Carrier concentration and mobility measurements for resistivities $10^{-4}$ to $1~\Omega$ -cm	Measurement of c.c. and mobility for semicondcutor thin films and bulk materials
Clean Room (Moore and Hanks)	Approximately 11.15 m <sup>2</sup> of Class 100 cleaned environment.	Used to perform photolithography procedure for use in chemical etching, plasma treatment, and metallization.
Mask Aligner (Cobilt Model CA-2020)	XY alignment of 0.125 μm; equally fine rotational alignment; ultra- uniform UV exposure	Alignment and exposure of photo- lithographically treated materials
Vacuum Deposition System (Unifilm Multi-source)	Base pressure = 10 <sup>-8</sup> torr; three DCMS/RFMS sources and ion beam source; computer controlled plantary motion	Deposition of metals and oxides onto semiconductors and dielectrics for fundamental materials studies and solar cell fabrication.

#### **ELECTRO-OPTICAL CHARACTERIZATION LABORATORY**

Instrument	Range/Capacity	Unique Features and Uses
Photoluminescence Spectroscopy System	CW and cavity dumped (40 kHz to 40 MHz) excitation sources, detection capability from 400 to 1,700 nm sample temperature range of 4 to 310 K	Energy resolved photoluminescence spectroscopy
Photoluminescence Lifetime System	Cavity dumped (40 Khz to 40 MHz) excitation, detection capability from 700 to 1,700 nm, 50 ps time resolution, sample temperature range of 4 to 310 K.	Time resolved photoluminescence spectroscopy for minority carrier lifetime and surface recombination measurements

Diffusion Time-of-Flight System	Cavity dumped (40 kHz to 40 MHz) excitation, detection capability from 700 to 1,700 nm, 45 ps time resolution	Minority carrier diffusion, lifetime, and surface recombination measurements
Superconductivity Test Station	65-300K, 3.2-22.0 μÅ, 1-10 V rms.	T <sub>C</sub> measurement of superconducting materials
Deep Level Transient Spectroscopy (DLTS)	DLTS spectra and transients from 77 to 320 K.	Determination of trap ionization energies, emission rates, and capture cross-sections
Capacitance-Voltage Measurement System	Capacitance as a function of voltage over the frequency range 100 Hz to 100 MHz.	Determination of the effective doping concentration
	CELL PERFORMANCE LABORATORY	
Instrument	Range/Capacity	Unique Features and Uses
Spectrolab X25 (with multisource attachment)	100 Wm <sup>-2</sup> to 20,000 Wm <sup>-2</sup> , user-controlled spectral and total irradiance	Solar simulation under standard or user-defined reporting conditions
Current-vs-Voltage Measurement System	$\pm 50$ to $\pm 1~\mu V$ , $\pm 8~A$ to $\pm 1~pA$ . 0°C to 110°C, voltage bias rates from steady state to 200 V/sec	Efficiency measurements, secondary reference cell calibration, temperature coefficients, diode parameters, I-V measurement artifacts
Spectral Response Measurement System	300-2,000 nm expandable to 10,000 nm, light bias to $^{\circ}5$ suns, voltage biase to $\pm$ 40 V, current range 1 pA to 4 A, up to 15 cm diameter monochromatic beam	Quantum efficiency, device parameters
PV Calibration System	Two Newtonian trackers, four samples at a time biased to I <sub>SC</sub> , temperature control and monitoring 40 samples at a time if I <sub>SC</sub> and total irradiance measured separately and under global sunlight.	Primary calibration of solar cells under direct or global sunlight; or modules under global light (fixed- tilt or normal incidence)
LICOR Spectroradiometer	300-1,100nm, 4 nm resolution, global light (integrating sphere), direct normal light (5.0° field of view), teflon dome diffuser, fiber optic probe	Required for efficiency measurementsand calibrations, simulator spectral characterization
HP1000 Computer and Macintosh Computers		Control of measurement systems, data base management.
Large Area Pulsed Solar Simulator (LAPSS)	2 m by 2 m area at 1000 Wm <sup>2</sup> , ± 100 μA to ±13A full scale current, ±1 V to 70 V voltage range.	Module measurements, defect and I-V artifacts related to the pulsed light source

### SURFACE INTERACTIONS, MODIFICATIONS AND STABILITY GROUP

Instrument	Range/Capacity	Unique Features and Uses
XPS/ISS/SIMS FAB SIMS/AES (Leybold LHS-10 System)	XPS:2 to 10 mm area; ISS; 0.5 to 2 mm spot size; SIMS: 0.5 to 2 mm spot size; AES: 1 µm spot size. Detection sensitivities of 0.1 to 1at. % for XPS, AES, and ISS; of ca. 10-4 at. % for SIMS	Depth profiling with a 1mm ≤ to 1cm < raster range. Thin-film deposition with QC monitor in a preparation chamber; translation of sample on heatable (to 800°C) - coolable (to 196°C) rods from preparation or high pressure chambers (to 10 atm) into the analysis chamber; four different surfaceanalysis probes on one instrument; FAB source for FAB-SIMS
Scanning Auger Microprobe (Physical Electronics 545C)	3.0 µm minimum size for electron beam; detection sensitivity of 0.1 to 1.0 at. % for Li to U	AES or SAM analysis while depth profiling; rapid-load lock and multiple sample carousel permits high throughput for routine analyses
Quartz Crystal Microbalance System (Sycon-NREL)	Range of 106 ng/cm <sup>2</sup> ; detection sensitivity of 1 ng/cm <sup>2</sup> ; practical capacity for hydrophilic polymers is about 1.5 mg; for other solids, 100 mg; T from 15 to 75°C; P from vacuum to 1,500 torr.	Measure adsorption/desorption of gases on solid overlayers adherent to a gold-coated quartz crystal; mass gain or loss during oxidation or reduction; permeation rates of gases throughsolids up to 100 µm thick; especially good for water vapor sorption studies
Beam Microbalance (Sartorius 4300)	Capacity of 3 to 5 g; detection sensibility of 0.1 to 1 µg; measuring range of 400 mg; T from -196 to 1,000°C. P from vacuum to 1,500 torr	Same applications as QCM. Samples (suspended by fibers) can be monitored for outgassing and desorption with an RGA
Deposit Thickness Monitor (Inficon IC-6000)	Measure deposition rates 0.05 nm/min to over 10 nm/s and deposited thicknesses to 10,000 nm to less than 1% accuracy	Monitor vacuum deposited overlayer thicknesses in the LHS - 10 preparation chamber for preparing clean metal deposits and subsequent XPS/ISS/SIMS/AES analysis without exposure to air
Metallograph (LECO Neophot 21)	Mag: 80X to 2,000X polarization quantinet 800 image analysis	Optical microscopy with or without polarized light, Nomarski, prints/slides, and image analysis
Solar Furnace (NREL)	10 kW maximum flux 240 W/cm ≤ 40 mm FWHM	Long focal length in primary concentrators (15.24 m); capable of inserting secondary concentrator to boost flux to the range of 5000 W/cm <sup>2</sup> ; fully automated operation and data acquisition

Profilometers (Tencor Alpha-Step and Sloan Dektak)	Measurement range from 100 nm full scale to 100 μm full scale	Measures step heights and surface topography with $\pm$ 1 nm sensitivity; auto-level, microscope X-Y translation stage for sample positioning 12.5 $\mu$ m and 5 $\mu$ m diameter tips available
Oriel Model 6732 Solar Simulator	Unfiltered spectral output from 0.25 to 2.5 microns at about 17 solar constants (collimated 5 cm" output beam).	Equipped with filters and dichroic reflectos to limit spectral output to a desired range for photochemical studies
Lambda-Physik EMG-50 Excimer Laser and FL-2000 Dye Laser	Monochromatic, coherent output, with wavelengths variable between 197-950 nm (1 cm <sup>2</sup> output beam)	Complements the solar simulator in photochemical studies; excimer is currently set up for 308 nm output, and dye laser is now tuneable between 260 and 320 nm
Polaron Series E6000 Vacuum Coater	10 <sup>-7</sup> torr vacuum capability, sputter or evaporative coating	Used to deposit thin films (usually of metals) onto smooth substrates; typical use is to prepare IR-reflective, metal coated substrates for IR-external-reflection spectroscopy

#### FTIR SPECTROSCOPIC RESEARCH AND SERVICE CENTER

Instrument	Range/Capacity	Unique Features and Uses
Nicolet System 800 High Resolution Mid-Infrared FTIR Spectrophotometer	Frequency range 7,800 to 500 cm <sup>-1</sup> , resolution 0.1 cm <sup>-1</sup> , sensitivity 10-5O D	Collects high-resolution, low-noise, mid-infrared spectra of solid and liquid samples in transmittance, specular and diffuse reflectance, and attenuated total reflection modes; also used with FT-Raman accessory
Nicolet FT-Raman Accessory	Frequency range 3,800-150 cm <sup>-1</sup> Stokes.	Collects Raman spectra of solid and liquid samples in reflective and refractive modes
Nicolet Nic-Plan FTIR Microscope	Minimum sample size 10x10 μm, translational resolution 1 μm.	Collects midinfrared spectra of small-area solid samples in reflectance and transmittance modes; used with System 510
Nicolet System 510 Medium Resolution Midinfrared FTIR Spectrophotometer	Frequncy range 7,000 to 400 cm <sup>-1</sup> (standard samples), 7,800-650 cm <sup>-1</sup> (FTIR microscope), resolution 0.20 cm <sup>-1</sup>	Primarily used with FTIR microscope, but has sample compartment for midinfrared transmittance analysis
Nicolet System 20F Medium Resolution Far-infrared FTIR Spectrophotometer	Frequncy range 650 - 20 cm <sup>-1</sup> , resolution 0.7 cm <sup>-1</sup>	Collects far-infrared spectra of solid samples in transmittance and diffuse reflectance modes
Hansen High-Trancryostatic Sample Mount	Sample temperature control range 8.0 - 450 K	Provides temperature and illumination control for solid samples during infrared transmittance analysis in vacuo; used with Systems 20F and 800.

Nicolet 7199b High-Resolution MidInfrared FTIR Spectrophotometer Frequency range ,-500 cm<sup>-1</sup>, expandable to 2,5000-100 cm<sup>-1</sup>, resolution 0.06 cm<sup>-1</sup>, sensitivity 10<sup>-5</sup>OD

Collects high resolution, low-noise, midinfrared spectra of solid and liquid samples in transmittance, specular reflectance, and attenuated total reflection modes

APPEN	NDIX E: GLOSSARY OF	HREM	High Resolution Electron Microscopy
ACPO	NYMS	HTSC	High Temperature Superconductor
ACKO	71 1 1 1 1 1 S	HTPB	Hydroxyl-Terminated Polybutadiene
		IEC	Institute of Energy Conversion
ADM	Advanced Desiccant Material	IECEC	Intersociety Energy Conversion Engineering
ADP	Advanced Desiccant Polymer		Conference
AES	Auger Electron Spectroscopy	IEEE	Institute of Electrical and Electronic Engineers
AEQE	Absolute External Quantum Efficiency	IR .	Infrared
AM	Air Mass	IS	Metal Oxide Species
APMOVPE	Atmospheric Pressure Metal Organic Vapor	ISET	International Solar Electric Technologies
	Phase Epitaxy	ISS	Ion Scattering Spectrometry
AMPSA	2-Acrylamide-2-Methyl-1-Propane Sulfonic	ITO	Indium Tin Oxide
	Acid	I-V	Current Versus Voltage
AMU	Atomic Mass Unit	J <sub>c</sub>	Critical Current
ARC	Antireflection Coating	J <sub>sc</sub>	Short Circuit Current
ASCII	Decimal computer format	JEBIC	Junction Electron Beam Induced Current
ASTM	American Society for Testing and Materials	JPL	Jet Propulsion Laboratory
ATR	Attenuated Total (internal) Reflection	LPE	Liquid Phase Epitaxy
AVC	Auger Voltage Constant	MBE	Molecular Beam Epitaxy
AWU	Associated Western Universities	MCP	Microchannel Plate
BEI	Backscattered Electron Imaging	MEE	Migration Enhanced Epitaxy
CDCS	Commercial Desiccant Cooling System	MMI	Meissner Mutual Inductance
CEM	Charge Collection Microscopy	MOCVD	Metal Organic Chemical Vapor Deposition
CL	Cathodoluminescence	MOVPE	Metal Organic Vapor Phase Epitaxy
CRADA	Cooperative Research and Development	MQW	Metallization of Multiple Quantum Well
_	Agreement	MRS	Materials Research Society
CV	Current-Voltage or Capacitance-Voltage	MUA	11-Mercaptoundecanoic
DCS	Desiccant Cooling System	NIST	National Institute for Standards and
DDF	Director's Development Fund		Technology
DH	Double Heterostructure	NMAB	National Materials Advisory Board
DLTS	Deep Level Transient Spectroscopy	NOCT	Nominal Operating Cell Temperature
DOE	Department of Energy	NRL	Naval Research Laboratories
DR	Diffuse Reflectance	OFG	Organic Functional Groups
EBIC	Electron Beam Induced Current	OMA	Organized Molecular Assembly
EBIV	Electron Beam Induced Voltage	PEP	Photovoltaic Energy Project
EC	Electrochromic		Photo Chemical Vapor Deposition
EC	Electronchanneling	PMT	Photomultiplier Tube
ECR	Electron Cyclotron Resonance	PSSA PV	Polystyrene Sulfonic Acid
ECW	Electrochromic Windows	PVUSA	Photovoltaic
Eg EDS	Bandgap Energy	PL	Photovoltaics for Utility Scale Application Photoluminescence
EELS	Energy Dispersive Spectroscopy	PZT	Lead Zirconate Titanate
ELLS	Electron Energy Loss Spectroscopy Electroluminescence		Quartz Crystal Microbalance
EMT1	Emerging Technology 1	QCM QC	Quartz Crystal
EOL/BOL	End of Life/Beginning of Life	QE	Quantum Efficiency
EPMA	Electron Probe Microanalysis	QMA	Quadrupole Mass Analysis
EVA	Ethylene Vinyl Acetate	R vs T	Resistance vs Temperature
FA	Fluorescence Analysis	RF	Radio Frequency
FAB	Fast Atom Bombardment	RFP	Request for Proposal
FF	Fill Factor	RGA	Residual Gas Analysis
FFT	Fast Fourier Transform	RH	Relative Humidity
FT-IR	Fourier Transform Infrared	RHEED	Reflection High Energy Electron Diffraction
FWHM	Full Width at Half Maximum	RIO	RHEED Intensity Oscillation
GB	Grain Boundary	RTD	Resistance Temperature Device
GPE	General Purpose Equipment	SA	Self Assembly
HAZ	Heat Affected Zones	SAIC	Science Applications International
HFSF	High Flux Solar Furnace Facility	J/ 110	Corporation
	. Harrian John Farnace Facility		

SAM Scanning Auger Microscopy SEI Secondary Electron Imaging SEIA Solar Energy Industries Association Scanning Electron Microscopy SEM Shallow Homojunction SHI SHS Self-Propogating, High-Temperature **Synthesis** Secondary Ion Mass Spectrometry SIMS SIMSG Surface Interactions, Modifications and Stability Group Solar Induced Surface Transformation of SISTM Materials S/N Signal to Noise Ratio Specular Reflectance SR **SRC** Synchrotron Radiation Center

SRM Solid Rocket Motor

**SSTM** Spectroscopic Scanning Tunneling Microscopy Scanning Transmission Electron Microscope **STEM** 

Scanning Tunneling Microscopy STM

**SWRES** Southwest Residential TAC Time Amplitude Converter TAG Technical Advisory Group Critical Temperature  $T_c$ 

TČO Transparent Conducting Oxide Transmission Electron Diffraction TED Transmission Electron Microscopy TEM

Tetrahydrofuran THF **TMA** Trimethylaluminum Time of Flight TOF

**Technical Review Committee TRC** 

Ultra High Vacuum UHV

**UPS** Ultraviolet Photoelectron Spectroscopy

US1 Utility Scale UV Ultraviolet

V<sub>oc</sub> VB Open Circuit Voltage

Valence Band

**VBM** Valence Band Maximum VHV Very High Vacuum

WDS Wavelength Dispersive Spectroscopy

**WFO** Work for Others

XPS X-ray Photoelectron Spectroscopy

X-ray Diffraction XRD

**ZTOF** Zero Field Time-of-Flight

## **ABSTRACT**

The Measurements and Characterization Branch of the National Renewable Laboratory (NREL) provides comprehensive photovoltaic (PV) materials, devices, characterization, measurement, fabrication, modeling research, and support for the international PV research community, in the context of the U.S. Department of Energy's Photovoltaic Research Program goals. This report summarizes the progress of the Branch from 31 January 1991 through 31 January 1992. The eight technical sections present a succinct overview of the capabilities and accomplishments of each group in the Branch. The Branch is comprised of the following groups: Surface and Interface Analysis; Materials Characterization; Device Development; Electro-optical Characterization; Advanced PV Module Performance and Reliability Research; Cell Performance Characterization; Surface Interactions, Modification, and Stability; and FTIR Spectroscopic Research. The including measurements and tests of PV materials, cells, submodules, and modules. The report contains a comprehensive bibliography of 77 branchoriginated journal and conference publications, which were authored in collaboration with, or in support of, approximately 135 university, industrial, government, and in-house research groups.

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