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FY 1992 Measurements and Characterization Branch Annual Report

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P. C. Dippo, Editor

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TABLE OF CONTENTS

INTRODUCTION

The Measurements and Characterization Branch actively supports the advancement of DOE/NREL goals for the development and implementation of the solar photovoltaic (PV) 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 PV 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 PV technologies. Members of the branch disseminate research findings to the technical community in publications and presentations.

The Measurements and Characterization Branch encompasses seven coordinated research groups, providing integrated research and development that covers all aspects of photovoltaic materials/devices characterization.

Surface and Interface Analysis

A.J. Nelson, Senior Scientist and Group Leader
S.E. Asher, Senior Scientist
L.L. Kazmerski, Principal Scientist
H. Moutinho, Research Associate
D.W. Niles, Staff Scientist
R. Reedy, Associate Scientist
A.B. Swartlander-Franz, Associate Scientist

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 atomic force microscopy.

Materials Characterization

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

This group employs advanced microscopy and microanalytical techniques in the determination of crystallographic, defect topographic, compositional, and microelectrical properties of PV 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; microcathodoluminescence; cooperative high-resolution interface analysis using high-voltage electron microscopy; and complete sample preparation facilities, including ion etching with integral SIMS analysis.

Electro-Optical Characterization

R.K. Ahrenkiel, Principal Scientist and Group Leader
D.J. Dunlavy, Associate Scientist
B.M. Keyes, Associate Scientist
D.H. Levi, Staff Scientist
C.R. Osterwald, Staff Scientist
J. Zhang, Research Associate

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 minority-carrier properties. Capabilities and techniques include capacitance-voltage, conductance-voltage, and temperature dependencies; 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 and Module Performance

K.A. Emery, Senior Scientist and Group Leader
E.E. Beck, Technician
H.R. Field, Staff Scientist
S. Rummel, Staff Scientist (PV Engineering and Applications Branch)

The work of this group includes calibrating and maintaining primary PV reference cells; performing efficiency measurements under standard reporting conditions for single and multi-junction cells and modules; calibrating secondary reference cells for the PV community; and evaluating PV device parameters. Capabilities and techniques include; (1) Current-*versus*-voltage measurements as a function of temperature, spectral irradiance, and concentration; (2) Spectral response as a function of temperature, voltage bias, spectral irradiance, and concentration; (3) Laser photoresponse mapping; and (4) detection of measurement-related artifacts present in other current-*versus*-voltage measurement systems.

Materials Durability and Component Reliability

A.W. Czanderna, Research Fellow
G.C. Herdt, Research Associate
F.J. Pern, Senior Scientist
D.E. King, Staff Scientist

The work of this group involves the testing and reliability research of advanced PV submodules and modules, with primary emphasis on PV flat-plate modules. Specific project research activities include testing and investigation of submodule and module performance, stability, energy output, and reliability lifetime characteristics, both under simulated and actual sunlight conditions. These efforts are conducted in conjunction with PV research activities originating both within and outside NREL. The group also coordinates PV thin-film module testing and reliability research for the US 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.

Surface Interactions, Modification, and Stability

A.W. Czanderna, Research Fellow and Group Leader
G.C. Herdt, Research Associate
C.L. Fields, Visiting Professional
D.R. Jung, Research Associate
J.R. Pitts, Senior Scientist

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 (FTIR); and contact-angle measurements, ultramicrogravity, 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 (OMAs).

FTIR Spectroscopic and Research Center

J.D. Webb, Senior Scientist and Group Leader
D.E. King, Staff Scientist
E.J. Johnson, Research Associate

This is a Laboratory-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 impurities or dopants in samples. Techniques include: high-resolution, mid-infrared FTIR spectroscopy in transmittance, specular reflectance, diffuse reflectance, and attenuated total reflectance (ATR) sampling modes using 5 cm^2, 0.5 cm^2, and fiber optic ATR probes, along with environmental control for in-situ analysis and measurement of reaction kinetics for most sampling modes; FT-Raman and FTIR-photoluminescence spectroscopy in the reflective mode; and FTIR microscopy for obtaining mid-infrared transmittance or reflectance spectra of sample areas as small as 10x10 μm, and far-infrared spectroscopy (650-50 cm^{-1}) with transmittance and diffuse reflectance sample modes.

HIGHLIGHTS

General

- **R&D 100 Award** for the development of the ***Atomic Processing Microscope*** [APM].

- Three Branch members were honored with NREL Awards:
  Brian Keyes, Outstanding Performance for "the operation of a facility which produces world class measurements characterizing semiconductor materials."
  John Pern, Outstanding Achievement for "identifying the mechanisms of degradation in the ethylene vinyl acetate (EVA) copolymer used to encapsulate PV modules."
  Roland Pitts, Outstanding Performance for "his work on solar induced surface transformation of materials using the solar furnace."

- More than 13,000 samples/devices/components were characterized during this past year. This included more than 46,000 measurements and tests in collaboration with some 100 organizations.

Surface and Interface Analysis

- Soft X-ray photoemission results on the H_2S gas and plasma-exposed surface of p-InP were used to correlate changes in the electronic structure to changes in the surface chemistry. These measurements indicated that the H_2S gas exposure and plasma exposure at glancing angles type-converts the p-type InP surface to an n-type surface and that the magnitude of the band bending is 0.6 eV, resulting in a homojunction interface. Additionally, for the initial H_2S exposures, the S bonds separately to both the In and the P, while at higher exposures a stable polymeric sulfur (S_n) overlayer forms. This indicates that the formation of the P-S and In-S binary compounds is diffusion limited.

- An investigation on the effects of H_2 plasma exposure of CdS using synchrotron radiation soft X-ray photoemission and optical emission spectroscopy was completed. Plasma species were identified with optical emission spectroscopy, and revealed the presence of Cd_2 and S_2 species for the respective substrates. Photoemission results indicated that exposure to the H_2 plasma preferentially removes the chalcogen component, thus creating chalcogen vacancies in the surface region. Additionally, the surface type converts and proceeds from a flat-band condition to a surface exhibiting band bending. The observed presence of the chalcogen vacancies and the band bending at the surface show the usefulness of plasma processing for tailoring surface electronic properties of semiconductors.

- The formation and stability of the CdS/SnO_2 interface was investigated using X-ray photoemission. In-situ photoemission measurements were acquired after each CdS deposition on SnO_2/glass to observe the development of the interface. Results show that there is minimal chemical interaction or Sn outdiffusion between the two materials. Also, the valence band offset is estimated at $1.2\ eV < \Delta E_v < 1.6\ eV$.

- A synchrotron radiation soft X-ray photoemission investigation on $ZnSe/CuInSe_2$ heterojunction formation was completed at the Alladin source, Synchrotron Radiation Center (SRC), University of Wisconsin-Madison. First-principles band structure calculations (S.-H. Wei and A. Zunger) show that $ZnSe/CuInSe_2$ has a significant valence band offset (VBO): 0.70 ± 0.05 eV for the relaxed interface and 0.60 ± 0.05 eV for the coherent interface. These large values demonstrate the failure of the "common anion rule." This is traced to a stronger Cu,d–Se, p-level repulsion in $CuInSe_2$ than the Zn,d–Se,p repulsion in ZnSe. ZnSe overlayers were sequentially grown in steps on *p*- and *n*–type $CuInSe_2$(112) crystals. RHEED analysis before and during growth indicated that the surfaces were ordered and that the ZnSe grows in registry with the substrate. In-situ 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 Zn 3d core lines. Results of these measurements reveal that the VBO is $\Delta E_v = 0.70 \pm 0.15$ eV, in good agreement with the first-principles prediction. This work marked the fifth year of the collaboration between researchers at SRC and A.J. Nelson , Group Leader of the Surface & Interface Analysis Laboratory.

- A photoemission study of the ZnTe/CdTe heterojunction was performed with an emphasis on valence band offsets as a function of strain relaxation. A valence band offset of zero for this heterojunction would yield an ohmic back contact for PV devices. Analysis of core-level spectra during interface formation demonstrated that the ZnTe/CdTe interface was abrupt and non-reactive. The valence band offset is strain dependent and measure-

ments show that ΔE_v = -0.01 eV for an unstrained interface and ΔE_v = 0.13 eV for a fully strained interface.

- The surface and bulk compositions of polycrystalline $CuInSe_2$ thin films were determined for non-stoichiometric (non-stoichiometric means $Cu/In \neq 1$) films by XPS. Results revealed that when $Cu/In < 1$, the bulk of the thin film is a mixture of In_2Se_3 and $CuInSe_2$ with a skin of $Cu_1In_3Se_5$. Also, for $Cu/In > 1$, a surface skin of excess Cu mixed with $CuInSe_2$ was observed. The difference in the electronic structure of the surface versus the bulk of these films was further investigated using synchrotron-radiation-induced photoemission. The enhanced grain $CuInSe_2$ films were sputter etched (500 V Ar) and analyzed in-situ to determine core-level binding energies and Fermi level positions for the n-type $CuIn_3Se_5$ surface and the p-type $CuInSe_2$ bulk within ± 0.1 eV. The transition between the n-type surface and the p-type bulk was experimentally observed by noting the change in the position of the valence band maximum (VBM) relative to the Fermi level (E_F). From these measurements, the valence band offset (ΔE_v) between these two layers was determined to be 0.40 eV. Measurement of the work functions (f) was also completed and reveals φ = 4.75 eV for the $CuIn_3Se_5$ surface layer and φ = 4.04 eV for the bulk $CuInSe_2$.

- A novel method for fabricating CdS on CdTe and for passivating surface and grain boundary defects in CdTe was developed using H_2S plasma modification of CdTe at elevated temperatures.

- CdS_xTe_{1-x} and ZnS_xSe_{1-x} alloys were fabricated using molecular beam epitaxial (MBE) growth of CdTe and ZnSe, respectively, in an ECR H_2S plasma. Substrate temperature, gas flow, and plasma power determine the film composition (XPS analysis) and growth rate.

Materials Characterization

- Initiated and maintained collaborative programs with the silicon industry (Texas Instruments (TI), Mobil Solar, Solarex, and Crystal Systems) to characterize defects in Si. Considerable progress has been made in characterizing TI's silicon spheres. This is leading to a CRADA between TI and NREL.

- Collaborated with Solar Cells, Inc., Golden Photon, Martin Marietta, the Institute of Energy Conversion , and the in-house CdTe program on the characterization of state-of-the-art CdTe thin-film solar cells. Transmission electron microscopy (TEM) was used to study the structural defects in these films, while SEM was used to investigate the electrical and luminescent properties of the material.

- Provided TEM characterization of hydrogen-related defects in hydrogenated polycrystalline Si.

- Collaborated with Kopin Corporation and Spire Corporation on the characterization of (GaAs/GaInP) and (GaAs/Si, InP/Si).

- New technique developments (for (a) a new EDX detector with an ultrathin Be window, allowing the detection of C and O; and (b) an on-line image analysis system using a CCD array, which allows the recording, analysis, and storage of TEM images directly from the microscope in electronic form) have been acquired and implemented.

  These new attachments were installed and tested. They have significantly enhanced our characterization capabilities.

- Business development: Collaborated with Ramtron Corporation on the characterization of lead zirconate titanate (PZT) ferroelectric thin films. This collaboration resulted in an outside funding of $86,000 during 1992 and a significant improvement of Ramtron's dynamic memory devices.

Electro-Optical Characterization

- A specially designed low-temperature photoluminescence spectroscopy system was brought on-line. Using a variable temperature cryostat combined with the optical multichannel analyzer (OMA), PL data over sample temperatures from 2 K to 300 K can be provided on a rapid turn-around basis.

- A contactless minority-carrier lifetime measurement facility was developed for industry support of crystalline silicon.

- Time-resolved photoluminescence (TRPL) was successfully used to characterize the minority-carrier lifetime of polycrystalline CdTe provided by internal and external research groups. This NREL-developed technique is being used to provide the first accurate lifetime data for these polycrystalline semiconductors, and is assisting CdTe cell research and development groups for the optimization of their cells.

- An identification of three oxygen complexes in $Al_xGa_{1-x}As$ that are "lifetime-killer" defects was made in collaboration with the Research Triangle Institute.

- Time-resolved photoluminescence analysis of n- and p-type InP wafers supplied by the Naval Research Laboratories (NRL) indicated that the lifetime in n-type wafers is orders of magnitude larger than in comparably doped p-type wafers. These results are typical of the contributions of this technique for the qualification of material for potential cell fabrication.

- Collaborative work with Purdue University found that lifetimes in epitaxial GaAs exceeded 1.0 microsecond by enhancing the photon recycling effect. This lifetime is 27 times greater than the radiative or theoretical lifetime.

Photovoltaic Standards Activities

- The Measurements and Characterization Program was asked to lead the international intercomparison for solar cells. Carl Osterwald was appointed chair of the ***PEP '93 International Intercomparison.*** The first organizational meeting was held in conjunction with the 11th European Photovoltaic Solar Energy Conference in Switzerland. These international PV measurement intercomparisons are conducted about every 5 years under the direction of the Photovoltaic Energy Project (PEP). This meeting, which was attended by representatives from major international PV laboratories, established the objectives, sample set, calibration methods, and schedule for the next intercomparison. The PEP '93 Intercomparison has been scheduled to begin in July, 1993, after a second participants' meeting that will be held during the 23rd IEEE PVSC in Louisville, Kentucky. Two separate sample sets will be circulated. The first will be composed entirely of 2x2-cm single-crystal Si reference cells that each participant will calibrate with their best calibration method. The results of these calibrations will be analyzed and used to establish the World PV Scale (WPVS) of primary reference cells. The second sample set will consist of devices from newer PV technologies that have unique measurement problems, such as narrow band-gap cells, large-area cells, and series-connected tandem cells. Each participant is free to use whatever methods are appropriate to measure the second sample set.

- The first results from laboratories participating in the ***ASTM Cell and Module Intercomparison*** were received and are currently being processed. These results are expected to be reported at the 23rd IEEE PVSC. Carl Osterwald chairs this activity.

- Revisions of ASTM draft PV standards for ASTM E44.09 (subcommittee on photovoltaics) were prepared. In conjunction with Heraeus DSET Labs, a revision of the solar weathering document was produced. This document is important to the U.S. PV program as it deals with determination of degradation of PV modules from UV exposure.

Cell Performance

- A new standardized outdoor I-V system for evaluating the wide variety of PV cells and modules under natural sunlight has been built. The system is designed to cover the current range from ± 10 μA to ± 40 A and a voltage range from ± 0.001 V to ± 100 V. The system can bias in either direction and is designed to identify bias related artifacts. The system also measures the spectral irradiance, air temperature, sample temperature, wind speed, humidity, and direct and diffuse irradiance. In addition, the system is capable of biasing over a user-defined range.

- A new outdoor I-V system for comparing rating methods for crystalline and thin-film technologies has been completed and is currently performing data acquisition at NREL on crystalline Si, CIS, a-Si, and CdTe modules. The system can perform I-V measurements of 16 modules at 1-minute intervals versus air temperature, sample temperature, wind speed, humidity, direct and diffuse irradiance, and spectra. The system was developed to allow the comparison of all the various module rating methods at the same site and time interval with all relevant PV technologies.

- The large-area pulsed solar simulator (LAPSS) data acquisition system has been modified to handle high capacitance modules using a capacitive sweep circuit with varying sweep duration and the ability to measure the current at a fixed power supply voltage. The safety of the LAPSS system has been improved by incorporating a new HV cable with interlocks supplied by the manufacturer as a result of an informal suggestion during the Tiger Team visit. The high intensity lamp housing for the pulsed solar simulator has been installed.

- The Spectrolab X25 continuous solar simulator, used for standardized single and multi-junction cell and submodule measurements and calibration, has been upgraded to accommodate 0.3 m^2 single and multi-junction modules.

- In support of internal research, subcontracted research, PV manufacturers, and the PV community, 1054 spectral response measurements, 1339 cell I-V calibrations, and 1931 module measurements were performed. These measurements/calibrations give the entire PV community a calibration traceability path to NREL. US manufacturers use this calibration traceability for their quality control programs.

Materials Durability and Component Reliability

- The broad-based applications potential has been elucidated for using fluorescence analysis (FA) to monitor or establish (1) the extent of ethylene vinyl acetate (EVA) discoloration and degradation in field-deployed PV modules, (2) the extent of discoloration of other PV encapsulant polymers, (3) the relative number of residual chromophores in EVA at various stages of processing, and (4) an understanding of structural changes occurring within polymers that produce discoloration. The FA was also used to assist other researchers at NREL for solution-grown CdS thin films, porous Si, and electrochromic samples.

- Degradation mechanisms of EVA encapsulant films have been identified via various acceleration experiments. The key thermal and photothermal EVA discoloration from polyene formation and color reversal by photobleaching are competing processes during the photothermal degradation process, and photobleaching of the discolored EVA can be effected by both the UV and visible light in the presence of air.

- The effects of EVA yellowing/browning on the I-V and efficiency of the solar cells were quantified in a degradation study of various EVA-encapsulated solar cells under accelerated thermal and photothermal conditions. The results further support previously established conclusions that UV exposure, elevated temperature, and hermetic enclosure are three major EVA-browning factors.

- The degradation mechanisms of PVB encapsulants in weathered PV modules are similar to those for EVA, as shown by FA and other techniques.

- Procedures were developed for polymer thin film sample preparation techniques, test methods, and numeric analysis, and experiments were conducted for developing and ranking new formulations and new encapsulation materials with various UV absorbers, anti-oxidants, and UV-absorbing glass superstrates.

- Development of procedures, techniques, and test methods for the diagnostic analysis of weather-degraded PV modules was continued (including non-destructive hot-spot infrared imaging, isolation of individual solar cells, resistances, and dark I-V measurements of the isolated solar cells, and destructive EVA sampling and compositional analysis). The analysis was completed for a large number of degraded EVA films sampled from various modules made by several PV manufacturers. The results show that EVA degradation and discoloration result primarily from the loss of Cyasorb UV 531 and the formation of long conjugated C=C bonds due to loss of acetate pendant groups.

- Studies were initiated on the formation mechanisms of SnO_2/CdS interfaces and their stability in real environments. Modules that were degraded by water vapor have been acquired for analysis by surface analysis techniques.

- The purchase, installation, and testing were completed for three light sources, including a DSET Suntest CPS table-top exposure system, an Oriel 1-kW Xe arc lamp light source, and an Oriel 1-kW Xe arc lamp UV-enhanced solar simulator, resulting in a greatly improved capability and capacity of performing accelerated photodegradation experiments. The purchase, installation, and testing were completed for the hardware, computer system, and software of an HP Model 1090/M high performance liquid chromatograph (HPLC) and a SPEX Model FL112 fluorescence spectrophotometer.

- Practical suggestions were made to staff members of several PV manufacturers for improving the current EVA formulations and processing conditions, on the basis of NREL accumulated experimental results and observations.

Surface Interactions, Modification, and Stability

- A number of experiments in rapid thermal processing at the NREL High Flux Solar Furnace were carried out. These included processing of ceramic super-

conductors, the growth of diamond-like carbon films, metallization and joining of engineered ceramic materials, and cladding of cermet powders to stainless steel.

- NREL CRADA #5 was initiated with Brush Wellman, Inc., in June, 1992. Work has proceeded on schedule for new methods of metallizing and bonding engineered ceramic materials. Negotiations for extending the CRADA into a second phase of development are underway. Contributions were made to the development of the Coors CRADA and planning of experiments to elucidate the growth mechanism of SiC on SiO_2 surfaces in the high-flux solar environment.

- Patent disclosures were filed on three promising technologies. The applications are in various stages of preparation: "Rapid Thermal Metalorganic Deposition of Metal and Oxide Thin Films," NREL IR#92-10, DOE Case No. S-75,895; "Solar-Induced Chemical Vapor Deposition (CVD) of Diamond-Type Carbon Films," NREL IR#92-27; and "Modification of Aerodynamic Surfaces to Minimize Soil Retention," NREL IR#92-22.

- In studies of copper deposited onto the organic functional end group of organized molecular assemblies (OMAs), we used XPS to show that Cu interacts with the OH groups of 11-mercaptoundecanol for coverages below 0.5 nm. At higher coverages, copper penetrates the OMA and reaches the OMA/Au interface. For Cu deposited onto OMAs with a CN end group, weak interactions occur below 0.1 nm; then further deposition of copper resides primarily at the OMA/Au interface.

- A critical review of research needs and opportunities in surface processing was completed and addresses 10 topical areas of applied surface science. Six of the topical areas (i.e., corrosion, polymer/metal (oxide) interfaces, thin-film multilayer solar collectors, accelerated life testing, interfacial microchemical characterization, and organized molecular assemblies) are directly relevant to PV devices. The other topics covered were fuel cells and solid batteries, lubrication and wear surfaces, photoelectrochemistry, and conducting polymers.

- The proceedings of a surface processing workshop were edited, published as an NREL document [24], and accepted for publication in Critical Reviews in Surface Chemistry. The 440-page NREL document contains current status summaries of methods of surface characterization, surface modification, and the recommendations of research needs and opportunities in 10 topical areas of applied surface science (see previous bullet).

- An invention disclosure was made for using OMAs on high surface area materials as selective molecular getters.

- A collaborative study is now in progress with the University of Denver using their 0-Atom Beam Facility to expose surfaces for XPS, FTIR, and ISS analysis at NREL. Initial results have been obtained after exposing Ag films to the ground-state, ca. 5-eV, 0-atom source and for several silicone polymers.

- The adsorption of toluene and trichloroethylene (TCE) and the competitive adsorption of water on several different polymers has been investigated using beam microbalance techniques. Polycarbonate coated onto high-area silica gels is the best polymer of those used for adsorbing TCE.

FTIR Spectroscopic Research Center

- Room-temperature photoluminescence (PL) spectra were obtained from crystalline Si, GeGaAs, $CuInSe_2$, and GaInAsP using the Nicolet 800 FTIR spectrophotometer with an FT-Raman accessory. This new measurement technique enhances our capability to measure PL in the infrared, and will enable sensitive, convenient characterization of these materials.

- We contributed to a detailed study of differences in hydrogen evolution rate from annealed, hydrogenated amorphous silicon samples prepared using different deposition methods. The results contributed to better understanding of structural differences between these materials.

- Under our work-for-others contract with NASA through SAIC, we published a paper describing the interactions between contaminants in the composite materials used to prepare solid rocket motors.

INSTRUMENTATION

SURFACE AND INTERFACE ANALYSIS LABORATORY

Instrument	Range/Capacity	Unique Features and Uses
XPS/Auger System (Perkin Elmer PHI 550)	2-mm spot size detection sensitivity of 0.1 at. % Li to U, 1-cm^2 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^2 sample size	Performs SAM and SIMS analysis with depth profiling, quantitative elemental analysis, and surface compositional maps, up to (5000X micrographs of surface features).
Scanning Auger Microprobe (Perkin-Elmer PHI 600)	0.02-μm spot size, same detection sensitivities as for PHI 590 1-cm^2 sample size	Performs SAMS and SIMS analysis with depth profiling quantitative elemental analysis and surface compositional maps, (up to 50,000X micrographs of surface features). Hot/cold stage.
Ion Microprobes (Cameca IMS-3f)	10-200-μm beam spot: Cs, O, or Ar primary ions; all elements and isotopes, H-U, sensitivities of 1 ppm-1 ppb: 1-cm^2 sample size	Performs high-sensitivity profiling and mass scans with 10-nm depth resolution; mass resolution ($M\Delta/M$) = 10,000 lateral ion imaging resolution of 1 μm.
Scanning Tunneling Microscope (RHK 100 and McAllister)	Atomic-level spatial resolution	Nanometer through atomic level imaging of conducting samples; light bias for higher resistivity semiconductors; ultrahigh vacuum chamber; sample and tip exchange.
Atomic Processing Microscope (APM)	Atomic-level spatial resolution; spectroscopic imaging	STM capabilities for spectroscopic imaging; atomic and nanoscale processing, including single-atom manipulation; nanoscale characterization.
Scanning Tunneling Microscope/ Atomic Force Microscope (Park Scientific)	Atomic-level spatial resolution	Ultrahigh vacuum STM and AFM capabilities.
Scanning Force Microscope (Park Scientific AutoProbe)	Contact and non-contact imaging on nanometer scale	Air-operated atomic force microscope for the nanoscale characterization of non-conducting surfaces, including biological samples.

MATERIALS CHARACTERIZATION GROUP

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 resolution back-scattered electronic imaging (BEI) 9-nm resolution	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 sub-bandgap (defect) luminescence with high resolution and correlation with topography.
TEM Philips CM - 30	KeV: 300, resolution: 2.3 Å, Tilt: ± 60°	Performs structural, analytical, and high resolution examination of a wide range of materials.

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	65-300 K, 3.2-22.0 μÅ, 1-10 V rms	T_c 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 AND MODULE PERFORMANCE GROUP

Instrument	Range/Capacity	Unique Features and Uses
Spectrolab X25 (With Multisource Attachment)	100 Wm^{-2} to 20,000 Wm^{-2}, user-controlled spectral and total irradiance 33 x 33 cm^2 beam	Solar simulation under standard or user-defined reporting conditions.
Current vs Voltage (I-V) Measurement System	± 50 to ± 1 μV, ± 8 A to ± 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, dark I-V, I-V measurement artifacts.
Spectral Response Measurement System	300-2,000 nm expandable to 10,000nm, light bias to ~5 suns, voltage bias to ± 40 V, current range 1 pA to 4 A, up to 100 cm diameter monochromatic beam	Quantum efficiency, device parameters. Capable of measuring modules or small cells.
Spectrolab Large Area Pulsed Solar Simulator (LAPSS)	2 x 2-m area at 1000 Wm^{-2}, ± 100 μA to ± 13 A full-scale current, ± 1 V to ± 70 V voltage range, custom data acquisition system	Module measurements, detect and correct I-V artifacts related to a pulsed light source's large bias rate.
Spectrolab Pulsed Concentrator Solar Simulator	10 x 20-cm area at 1 to 2000 suns, ±100 μA to ±13 A full-scale current, ±1 V to ±80 V voltage range, 1-msec pulse	Concentrator I-V measurements, correct bias rate, temperature, and spectral artifacts.
PV Calibration System	Two Newtonian trackers, four samples at a time biased to I_{SC}, temperature control and monitoring 40 samples at a time if I_{SC} 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).
PV Rating Analysis System	I-V of 16 modules at 1-min intervals vs air temperature, sample temperature, wind speed, humidity, direct and diffuse irradiance, and spectra	Compare all of the various module rating methods at the same site and time interval with all relevant PV technologies.
LICOR Spectroradiometer	300-1,100 nm, 4-nm resolution, global light (integrating sphere), direct normal light (5.0° field of view), Teflon dome diffuser, fiber optic probe. Computer model extension of outdoor spectra to 300-4000 nm	Required for efficiency measurements and calibrations, simulator spectral characterization.

GER Spectroradiometer	300 to 1100 nm, 4-nm resolution global light (integrating sphere, diffusing plate), direct normal light (5.0° field of view)	Required for efficiency measurements and calibrations, simulator spectral characterization.
HP1000 Computer and Macintosh Computers		Control of measurement systems; data base management.
Standardized Outdoor I-V System	I-V of cells and modules, ± 10 μA to ± 40 A, ± 0.001 V to ± 100 V with air temperature, sample temperature, wind speed, humidity, direct and diffuse irradiance, spectra, and data base	Flexibility to evaluate any PV cell or module at one sun or under concentration. Designed to evaluate bias related artifacts.
Spire Solar Simulator	I-V measurements on modules and cells 0.1 mA to 20 A, 0-70 V	High throughput module measurements.

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 1 at. % for XPS, AES, and ISS; of ca. 10^{-4} at. % for SIMS	Depth profiling with a 1-mm ≤ 1-cm 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 surface analysis 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 10^6 ng/cm^2; detection sensitivity of 1 ng/cm^2; 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 through solids 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, 250 W/cm, 40 mm FWHM	Long focal length in primary concentrators (7.05 m); capable of inserting secondary concentrator to boost flux to the range of 5000 W/cm^2; 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 ± 1-nm sensitivity; auto-level, microscope X-Y translation stage for sample positioning 12.5-μm and 5-μm diameter tips available.
Oriel Model 6732 Solar Simulator	Unfiltered spectral output from 0.25 to 2.5 μm at about 17 solar constants (collimated 5-cm^2 output beam).	Equipped with filters and dichroic reflectors 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^2 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^{-7} 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^{-1}, resolution 0.1 cm^{-1}, sensitivity 10^{-5} O.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^{-1} Stokes	Collects Raman and photolumi-nescence spectra of solid and liquid samples.

Nicolet Nic-Plan FTIR Microscope	Minimum sample size 10 x 10 μm, translational resolution 1 μm, includes automated scanning stage and color micrography	Collects mid-infrared spectra of small-area solid samples in reflectance and transmittance modes; used with System 710.
Nicolet System 710 Medium-Resolution Mid-infrared FTIR Spectrophotometer	Frequncy range 7,000 to 400 cm^{-1} (standard samples), 7,800-650 cm^{-1} (FTIR microscope), resolution 1.0 cm^{-1}	Primarily used with FTIR microscope, but has sample compartment for mid-infrared transmittance analysis.
Nicolet System 20F Medium Resolution Far-infrared FTIR Spectrophotometer	Frequency range 650 - 20 cm^{-1}, resolution 0.7 cm^{-1}, evacuable optics	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 Mid-Infrared FTIR Spectrophotometer	Frequency range, 7800-500 cm^{-1}, expandable to 25,000-100 cm^{-1}, resolution 0.06 cm^{-1}, sensitivity 10^{-5} O.D.	Collects high resolution, low-noise, mid-infrared spectra of solid and liquid samples in transmittance, specular reflectance, and attenuated total reflection modes.

MATERIALS DURABILITY AND COMPONENT RELIABILITY RESEARCH

Instrument	**Range/Capacity**	**Unique Features and Uses**
Denver Instrument A200DS Electronic Analytical Balance	0.1-mg and 0.01-mg resolution	Accurate weights measurement.
DSET Suntest CPS System with a Black Panel Thermometer	1500 -W Xe burner, (20-cm x 28-cm) sample plate (removeable), and programmable timer	Tabletop exposure unit; easy operation for small and large samples.
Oriel Model 81281 UV-ehanced Solar Simulator with a Power Supply (Model 68820)	1000-W Xe lamp (reduced IR intensity)	For accelerated tests for UV degradation of polymer materials and mini-modules.
Oriel Photofeedback Controller (Model 68850)		Light intensity auto-feedback controller for solar simulator.
Oriel 100 W Xe Arc Lamp/Lamp Housing (Model 66021)	Can hold a 450 to 1000-W Xe or Xe(Hg) lamp	For photochemistry and other applications that require intense light.
Oriel Power Supply (Model 66820)	400 to 1000 W	Power supply for the 1000-W Xe lamp.
Oriel Ozone Eater (Model 66087)	Activated carbon fibers	Portable unit, useful for filtering ozone produced from 1000 -W Xe arc lamp.

Sunlighter RS/4 Test System (Model 15)	3 GE RS/4 UV lamps with a turntable	Test chamber for accelerated testing of materials.
HP-8452A UV-visible Spectrophotometer	190 nm to 820 nm, 2-nm resolution	Absorption and transmission measurements.
M.-M. Micromanipulator (Model 6000)	Optical microscope, XYZ-trans-portable platform with four needle probes	For microscopic examination and/or resistance measurement of small objects.
Lab-line Duo-Vac ovens (Three units), Two Mini-Vac Pumps	25°C to 260°C	General purpose heating or drying.
Orion pH Meter, Model 720A (With an Electrode Switch)	pH 0 to 14 and ion-selective analysis	pH measurement for aqueous solutions; useful for analysis of metal cations or anions.
Fluke 8050A Digital Multimeter	AC/DC, MA, V, Ohms	For current, voltage, and resistance measurement.
Keithley 485 Autoranging Picoammeter		Nano- to picoamper measurement.
Cole-Parmer 4658 and VWR Scientific 370 Stirrer/Hot Plate		Stirring and heating.
Omega Digital Thermometers (3)	Digital reading (0.1°C)	Temperature measurement.
Branson Model 3200 Ultrasonic Cleaner		Ultrasonic cleaning.
GraLab 900 Programmable Electronic Timer		Timer.

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This report describes work done by the Measurements and Characterization Branch of the National Renewable Energy Laboratory (NREL) during FY 1992 to provide 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 1 February 1992 through 31 January 1993. 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.

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