ARRAYS TO ATOMS: THE EVOLUTION OF CHARACTERIZATION

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ABSTRACT
Characterization is one important component of the photovoltaics research and development effort. It has supported the evolution of the technology, and measurement techniques have evolved in response to the technology's needs. This paper highlights some important evaluation and verification techniques, covering macroscale through microscale characterization methods. Some emphasis is placed upon the nanoscale regime, in which the characterization and processing of semiconductors is leading to areas of atomic engineering of materials.

INTRODUCTION
We sometimes tend to fragment or compartmentalize our technology into special technical interests. We can mistakenly think of a particular materials system, a particular discipline or a particular approach as the whole of photovoltaics. In fact, the technology owes its scientific advancement to an ensemble of creative contributions from its parts—covering thought, planning, realization, evaluation, and verification. Theory, materials, processing, device development, modeling, testing, and measurement are co-dependent. None of these areas can stand alone; together, they provide the basis, the successes and the future directions of the technology. It is the purpose of this paper to highlight the evaluation and verification aspects of photovoltaics, the area of characterization. The objective is to reinforce how characterization has helped in the evolution of the technology, and how characterization techniques have evolved in response to the technology's needs. This examination will include some historical perspectives, an evaluation of the present, and a look to the future. Table 1 summarizes a selection of characterization techniques commonly used in support of photovoltaic device and component evaluations. It is conceded that all aspects and the entirety of measurements of even this limited set cannot be adequately addressed in this space. Therefore, only some of the more visible and interesting techniques will be examined. Macroscale and microscale methods are discussed, primarily in terms of the state-of-the-art, technical requirements. Of special interest to this paper (and the author) is the area of nanoscale analysis and technology. This includes the introduction of recent results that are at the foundation of not only diagnostic evaluations, but also the engineering of materials at the very atomic level—the not far off future for electronic developments.

<table>
<thead>
<tr>
<th>Cells and Modules</th>
<th>Materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Simulator efficiency (continuous, pulsed)</td>
<td>Scanning electron microscopy (EDS, EBIC, CL, channeling)</td>
</tr>
<tr>
<td>Outdoor performance</td>
<td>Transmission electron microscopy</td>
</tr>
<tr>
<td>Quantum efficiency</td>
<td>Electron microprobe analysis</td>
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<td>Current-voltage</td>
<td>X-ray diffraction</td>
</tr>
<tr>
<td>Reference cell calibration</td>
<td>Quartz and beam microbalance</td>
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<tr>
<td>Spectral measurements</td>
<td>Relative and durability</td>
</tr>
</tbody>
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<tr>
<th>Electro-Optical Measurements</th>
<th>Surface Analysis</th>
</tr>
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<tbody>
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<td>Spectrophotometry</td>
<td>Scanning Auger electron spectroscopy</td>
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<td>Ellipsometry</td>
<td>Electron energy loss spectroscopy</td>
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<td>Photoluminescence</td>
<td>X-ray photoelectron spectroscopy</td>
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<tr>
<td>Photoconductivity</td>
<td>Ultraviolet photoelectron spectroscopy</td>
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<tr>
<td>Fourier transform</td>
<td>Deep-level transient spectroscopy</td>
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<td>Infrared techniques</td>
<td>Synchrotron source measurements</td>
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<tr>
<td>Deep-level transient spectroscopy</td>
<td>Secondary ion mass spectrometry</td>
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<tr>
<td>Capacitance, current-voltage</td>
<td>Neutral ion spectrometry</td>
</tr>
<tr>
<td>Minority-carrier parameters</td>
<td>Ion scattering spectrometry</td>
</tr>
<tr>
<td>Scanning tunneling microscopy</td>
<td>Atomic force microscopy</td>
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<td>Atomic force microscopy</td>
<td>Ballistic electron energy microscopy</td>
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MACROSCALE MEASUREMENTS

The measurements in this regime are those defined from the array through the cell level. These constitute primarily those in the electro-optical area, and concentrate on the evaluation of component performance. Certainly the emphasis of the macroscale measurements have been those relating to modules and cells—the visible reference points for the viability of this technology.

The accurate and standardized determination and reporting of the performance of photovoltaic components are essential for the advancement of the technology. The validity of these evaluations is required for meaningful comparisons of the cell, module, and array performance, not only between the various PV technologies, but also to evaluate alternative methods of electrical power generation. The assessment of the performance of cells and modules under defined and accepted...
standard, reproducible test conditions is a major requirement for the meaningful appraisal and eventual acceptance of photovoltaics.

Perhaps, there is no more controversial and sensitive area of characterization than this area of performance evaluation. Efficiency has a straightforward definition, but its evaluation is complex [1]. As photovoltaic devices and modules have become more complicated, so have the methods required to accurately, reproducibly, and fairly evaluate their performances. There may be more standards and regulations covering this area of characterization than any other. There are certainly as many innovative methods used to try to circumvent these rules and procedures as the number of rules and procedures themselves.

One foundation of terrestrial cell and module measurements in the U.S. is traced to the publication of the Solar Cell Radiation Handbook by NASA/JPL in 1973 [2]. Although written for space applications, it provided the basis for instrumentation and simulated sunlight measurements of solar cell parameters. Following the direction provided by the NSF/RANN Cherry Hill Workshop for Photovoltaic Conversion of Solar Energy for Terrestrial Applications in October, 1973, two important Terrestrial Photovoltaic Measurement Workshops were held in 1975 and 1976 under the auspices of ERDA and NASA Lewis Research Center. The results of these meetings, which convened representatives from most of the U.S. PV efforts, were published in a set of terrestrial cell and array measurement procedures by NASA Lewis in 1977 [3]. These provided the initial guidance for the U.S. terrestrial program for the evaluation of PV components, and reflected the heavy influence of and experience with extraterrestrial PV. Because of expertise in the space photovoltaic technologies, NASA served as the center for cell evaluation for the U.S. terrestrial programs until these activities were transferred to SERI in 1980. Since then, national and international intercomparisons, technique developments and international cooperation have led to a set of standard measurement and reporting conditions and procedures, and increasing confidence in efficiency measurements worldwide.

The standard reporting conditions are defined by a reference spectral irradiance, total irradiance, temperature and standard definition of area, and are summarized in Table 2 [4]. The discussion of these accepted, consensus conditions are reported in detail elsewhere. However, their examination provides some insight into the variety of sources of error that can enter into the determination of the efficiency of a cell or module. These sources of uncertainty include factors relating to geometry (device area), electronic instrumentation (calibration, stability), the light source (irradiance, spectral distribution, spectral measurement, uniformity), conditions (contacts, temperature), procedures and definitions, and a host of others. Uncertainties in these conditions lead to considerations of accuracies in measuring the photovoltaic efficiencies. A summary of what is considered to be the status of efficiency determination for one sun cells, concentrator cells and modules is provided in Table 3. The technology of measuring cells has received more attention than that for modules, and the limitations have been more thoroughly addressed. This is reflected in comparing the accuracies of cells vs. modules in Table 3. The best current methods are defined as those that provide the best evaluation. For cells, they are simulator based. In some cases, the accuracies in determining the efficiencies of modules in simulators can only be provided within 10%, and outdoor measurements are used to minimize errors. Typical methods are those encountered in typical laboratory environments. These accuracy observations are based upon intercomparisons of measurements between NREL and many other laboratories worldwide. The results of formal, international intercomparisons are also cited, and provide about the same accuracy ranges. In general, the confidence in measuring accurate and believable efficiencies at

<table>
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<th>Application</th>
<th>Irradiance (W/m^2)</th>
<th>Reference Spectrum</th>
<th>Temperature (°C)</th>
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<tr>
<td>Terrestrial</td>
<td>1000</td>
<td>Global</td>
<td>25</td>
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<tr>
<td>Terrestrial</td>
<td>&gt;1000</td>
<td>Direct</td>
<td>28</td>
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<td>Concentration &gt; 1 sun*</td>
<td>&gt;1000</td>
<td>Direct</td>
<td>28</td>
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<td>Method 1</td>
<td>1000</td>
<td>Global</td>
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<td>Method 2</td>
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*Single crystal and multi-crystal Si only
+Based on PEP 85 and PEP 87

PV laboratories worldwide has increased significantly due to the efforts of the standards and testing organizations over the past 10 years, especially in the cell area. However, problems do exist—especially for multiple junction devices and for modules. These two areas are critical to the PV industry, and are currently the main focus for performance evaluation. Multiple-source simulators are currently the best approach to determining the current-voltage characteristics of multi-junctions and tandems. However, area-related problems, spectral considerations, capacitance, and temperature effects are among the limitations to current, large-area pulsed simulator methods employed for modules—especially multi-junctions having high capacity. One approach is to use the methods successfully employed for the cell technologies. This requires the development of large area, continuous simulators, and the measurement of the spectral response of the modules. Attention to increasing the accuracy of module measurement is the current frontier for the performance area, and one that is critical. The evolution of this technology requires, as for previous cell and module standards, a consensus of the industry and users, and depends upon technical input, evaluation and acceptance by many sources within the PV community.
Other areas

Certainly, a variety of other macroscale measurement areas are critical to photovoltaic evaluation. This includes investigations of the materials and methods used to encapsulate modules, and the methods used to establish reliability and durability. With the advancement of the technology, the emphasis has begun to shift more to developments relating to qualification of components for long operating lifetimes [5]. The establishment of performance levels is important; keeping the performance at these levels is critical.

MICROSCALE MEASUREMENTS

The microscale measurements are the workhorses and foundations for the photovoltaic technologies. Emerging and established technologies have critical analytical requirements for the diagnostic investigations of components, especially at the materials level. The measurement needs span the optical, mechanical, electrical, and chemical fields. Their application ranges from "failure analysis" to "directive and preventative analysis" to guide short term paths to technology demonstration. The range of such analytical tools is expansive, and a few are cited in this section.

Microcharacterization

Electron microscopy and related techniques provide important information on the structural, chemical, and physical characteristics of materials and materials interfaces. Electron probe microanalysis gives the first indication of compositional integrities for many PV materials development activities. The quantitative nature of this technique and its high accuracy (±0.1 at.-%) make it an indispensable tool. This and a variety of other compositional techniques are used for PV materials evaluation, and are summarized in Table 4. Scanning electron microscopy and its analytical capabilities for determining electrical properties (e.g., electron beam induced current and voltage measurements), structure (e.g., electron channeling), and composition (e.g., energy dispersive X-ray spectroscopy) have been important in providing research directions for new materials and device structures. Transmission electron microscopy has been prevalent in materials science since the 1950s. It has contributed important information to various photovoltaic technologies, including both single-crystal and polycrystalline structures. Its value has been primarily in structural and chemical investigations, especially of interfaces. The technique has advanced significantly over the past decade, as indicated by the improvement in spatial resolution capabilities shown in Fig. 1. Typical instrument point-to-point resolutions have improved by a factor of two, to the sub 1.5 Å level today with significantly brighter beam sources than used prior to 1980. This improvement has led to the ability to provide atomic-level information on defects and chemistry at device junctions; information that has been used by III-V single-crystal, III-Vs grown on Si, and II-VI polycrystalline technologies for optimizing cell processing leading to cell performance improvements [6,7]. The importance of electron microscopy to photovoltaics will continue to increase, as more attention is given to cell interfacial events. This is especially true with the advent of field emission sources, since the capability of small area analysis is enhanced. For example, 10 Å spot sizes can be routinely used for analytical (microdiffraction and EDS) purposes.

Surface Analysis

Surface elemental compositions, segregated impurity species at interfaces, doping profiles and gradients, interfacial chemical reactions, and quantitative compositional information are fundamental measurements of critical importance to the design
categorized under the generic term "surface analysis" [8]. These characterization tools examine the topmost atomic layers using a variety of input probes (electron, photon, ion) and detect one or a number of output or emitted species. The common techniques include: Auger electron spectroscopy (AES), electron energy loss spectroscopy (EELS), X-ray photoelectron spectroscopy (UPS), ultraviolet photoelectron spectroscopy (UPS), secondary ion mass spectrometry (SIMS), secondary neutral mass spectrometry (SNMS) [9], laser ion mass analyzer (LIMA) or laser microprobe (LMP) or laser microprobe mass analyzer (LAMMA) [10], ion scattering spectroscopy (ISS) or low-energy ion scattering spectroscopy (LEIS) [11], and Rutherford backscattering spectroscopy (RBS) [12]. The method is categorized in terms of the detected species (e.g., AES and XPS are electron spectroscopies, while SIMS is an ion spectroscopy). These methods, the major of which are summarized Table 5, provide qualitative and quantitative information of the chemistry and composition of surface and interface regions in materials.

These techniques provide complementary information. AES has very high-spatial resolution, but is not sensitive to trace level concentrations. SIMS provides this feature, alone with the capability to detect hydrogen directly. However, SIMS has limitations in quantitative areas compared to XPS and AES. Because of the fundamental processes involved and because the X-ray probe in XPS is less destructive than ion or electron beams, it is capable of providing the most dependable information on bonds and chemistry. Each method has its strengths, but the interpretations must be made with an understanding of the limitations. In many cases, an unambiguous solution to a problem may require the use of two or more of these analysis methods. In all cases, interpretation requires experience, knowledge and expertise.

NANOSCALE ANALYSIS AND ATOMIC ENGINEERING

The final and most detailed portion of this paper is devoted to the new nanoscale science and technology area involving scanning probe microscopies. The scanning tunneling microscope (STM) provides not only for the direct imaging of electronic and atomic structure, on nanometer levels, but also the processing and engineering of materials on these same scales. The ability to establish spectroscopic and specific-atom information about the surface of a material is a key element in providing the precision required to perform processing on selected regions with single-atom resolution and accuracy. Previously, it has been shown that material can be deposited on or removed from a surface by applying voltage pulses to the STM tip [13-15]. Removal of adsorbates from semiconductor surfaces and deposition of metal atoms onto such surfaces from the STM probe have been accomplished using the electric field developed between the tip and the surface. The ability to manipulate single atoms on Si surfaces has recently been reported and explained by Lyo and Avouris [16]. Specifically, this group combined the effects of the strong tip-surface electric field with the chemical tip-sample interactions to transfer Si atoms and atom clusters from the surface to the tip, and redeposit these atoms or clusters at predetermined surface sites. In this paper, the ability to perform nanoscale processing and single atom control is demonstrated by utilizing coordinated pulsed electrical fields and photon fields in a new instrument, the Atomic Processing Microscope, a system and method for the atomic engineering and nanocharacterization of material surfaces using proximal probe techniques. The result is the removal of single atoms from a semiconductor surface and the placement of single extrinsic atoms at selected sites. The effective control on the local electro-optical properties has been shown by STM tip-controlled characterization techniques, including nanoscale electron-beam induced current and nanoscale photoluminescence, having spatial resolutions capable of evaluating the effects of the atomic manipulations.

Figure 2 presents the schematic diagram of the basic proximal probe system used for the imaging, processing, and characterization in these experiments [17]. The basic STM is of the inverted tube scanner design, with an automated tip approach. Control, data acquisition, and image processing (through I-V characteristics) are provided by a personal computer [17]. The spectroscopic imaging technique, using specific electronic and photon biasing to distinguish atomic species, has been described previously [17]. The biasing incorporates potential differences between the probe and exact sample surface and wavelength-tunable laser excitation for resonance excitation of bonded atomic species. The photon biasing is provided by a pulsed, tunable dye laser covering the wavelength range 240-1100 nm, and the wavelength-specific light is directed onto the sample surface with a fiber optic element. A similar source is used for the photon-assisted atomic processing in the microscope. A special STM probe has been developed for the atomic-scale processing for extrinsic species. Intrinsic atoms can be removed from the surface to the tip, and be transported to a designated location and repositioned at that site. The control of the electrical properties of defects in semiconductors is important for the improvement of both materials characteristics and device performance. The removal of specific atomic species and the placement of single, extrinsic atoms at specific sites to alter the properties of the defect region is reported herein, with the first illustration of
such a process for GaAs. The effects of these atomic engineering procedures are evaluated with nanoscale resolution using the same proximal probe instrumentation on the same areas.

Atomic Processing

The use of the STM tip to manipulate clusters and single atoms at surfaces has been demonstrated on several materials, including Si and CuInSe2. For example, Lyo and Avouris have reported such manipulations for strongly-bound covalent Si atoms at room temperature[16]. Combining the strong electric field between the tip and the surface with the chemical probe-surface interactions, Lyo and Avouris were able to transfers Si clusters and single atoms from the surface to the tip, as well as move and redeposit these at preselected sites. The process is explained by field-evaporation mechanisms that are modified by the mechanical and chemical tip-surface interactions. In the experiments by Lyo and Avouris, the electric field intensity between the tip and the atoms below it is varied by either applying a fixed voltage between the tip and varying the tip-sample separation, or by keeping the distance constant and varying the pulsed voltage applied. The field evaporation is effected by the collapse of the effective tunneling barrier height, defined through the expression for the tunneling current[18]:

\[ I_t = V \exp \left[ -Cz \left( \frac{q\Phi_{\text{eff}}}{z^2} \right) \right] \]

(1)

where, \( V \) is the applied voltage; \( z \), the tip-sample separation; and \( q\Phi_{\text{eff}} \), the effective tunneling barrier height. As the tip-sample distance decreases, \( q\Phi_{\text{eff}} \) is reduced, and within a change of several Å collapses to zero. This collapse in \( q\Phi_{\text{eff}} \) induces the field evaporation process and the control (removal or placement) of atoms or clusters, depending upon the bias polarity and direction of tip movement. Based upon the experience with the spectroscopic imaging, the same reduction in the effective barrier height can be accomplished by application of a suitable photon field. It was previously reported that the expression for the tunneling current of Eq. (1) is modified in the presence of a photon field, to

\[ I_t = V \exp \left[ -Cz \left( \frac{q\Phi_{\text{eff}}}{z^2} - q\Phi' \right) \right] \]

(2)

where \( q\Phi' \) represents the resonance-coupled signal due to the photon field of a specific wavelength. Therefore the reduction of the exponential term to zero can also be accomplished by the application of a suitable photon field for the realization of the required field evaporation process. It is this process, using combined electric and photon biasing to modulate the effective barrier height, that is used in these studies to selectively remove intrinsic atoms from the semiconductor surface, and place intrinsic or extrinsic atoms at designated sites.

The effectiveness of this technique for removal of atoms from the surface is represented in Fig. 3 for GaAs. The power densities in the photon bias used to lower the effective barrier height is 5-10 times that required for the imaging, and are set to be slightly longer than the electric field pulse (i.e., 10-15 ms). Figure 3a presents an initial (comparison) atomic image of a (110) surface that has Ga (false-colored green) and As (red) atoms on the cleavage plane. Figure 3b shows an image of the same region after removing two predesignated atoms from the surface. The conditions are set with a voltage pulse of 2.4 V, and a photon bias set to the wavelength for As, \( \lambda = 886 \text{ nm} \). The conditions are very sensitive to conditions on both bias settings. Figure 3c shows the same procedure on a GaAs surface with a voltage pulse of 4.5 volts, which removes a large cluster of surface atoms. The same procedure can be used for Ga, with different photon and electrical biasing. Since the As is less tightly bound than the Ga, it is easier to manipulate this species.

Fig. 3. Spectroscopic scanning tunneling microscope image of GaAs (110) surface: (a) initial surface; (b) process surface, As-atom removal. Ga atoms are colored green and As atoms, red.
The ability to manipulate and place atoms is illustrated for CuInSe₂ in Fig. 4a-c which presents an image of the (220) cleavage plane. The conditions for atom manipulation in this system have been previously reported. This spectroscopic image of Fig. 4a shows the positions of Cu (red), In (blue), and Se (magenta) atoms with several existing Se and Cu vacancies present on the pre-processed (220) surface. The first step in this illustrative processing was to selectively remove a single, predetermined Se atom (creating another Se vacancy). This step is shown in Fig. 4b, in which the pre-selected Se-removed site is indicated. The process for placing the extrinsic oxygen atoms is approximately the reverse of the removal process just described. In this case, the atoms (oxygen) are provided through the catheter probe described previously. The oxygen is directed to the surface in the form of a pulse to confine the number and position of the atoms. A negative voltage pulse is applied between the tip and the desired site, synchronized with the pulse of the oxygen atoms. Several conditions were investigated, but a single pulse in the range -2.5 to -3.0 V provided the best control to place the oxygen. Higher fields caused the oxygen to be deposited over larger areas. Smaller fields seem ineffective; multiple negative and any positive pulses contaminate the tip with the oxygen. The preselected placement of oxygen (yellow) at one "native" vacancy and the created Se vacancy is shown in Fig. 5c. The computer controlled procedure is very reproducible for the CuInSe₂, which has minimum problems with oxygen absorption at low temperatures.

Nanocharacterization

The capability to engineer the atomic structure is enhanced by the ability to determine the effects of atomic manipulation with characterization techniques having the same spatial resolution. As previously indicated, macroscale electron beam techniques have been used to evaluate the minority carrier properties of defect regions in semiconductors [19]. The STM and related methods provide this same facility, and have been reported earlier [20]. This nanoscale electron-beam induced current or conductivity (or NEBIC) method is an analog of the conventional EBIC method afforded, for example, by SEM operations. In NEBIC, the transverse electron beam induced current is measured through the sample as the STM probe is scanned across it [20]. The energy of the incoming electron beam is, of course, much lower than that used for conventional EBIC (tens of volts compared to thousands of volts). The generation volume (which dictates the spatial resolution) is not calculated through the normal formulations because of the very low energy of the incoming electron beam. NEBIC has been applied to the evaluation of oxygen place at grain boundaries in CuInSe₂, with the conclusion that the oxygen acts a p-type dopant for the defect region.

Other techniques have also been incorporated into the APM for nanoscale co-characterization. The electron beam source from the STM tip has been used to provide confined-area AES compositional measurements. Nanoscale electro- or cathodoluminescence has been used to provide information on defect levels. This involves using the electric field of the STM probe to generate an optical signal that can be measured by a specially-designed detector shown schematically in Fig. 1. The STM probe can also be used to confine optical excitation from

Fig. 4. Spectroscopic scanning tunneling microscope image of CuInSe₂ (220) surface: (a) initial surface; (b) after removal of Se atoms; (c) after oxygen placement. Cu atoms are colored red, In, blue, Se, magenta and O, yellow.
a laser or other light source through its electric field. This confinement greatly enhances the spatial resolution of the light source, to regimes significantly better than those predicted on the basis of wavelength. This method provides spatial confinement of the light source impinging on the surface to the hundreds of Å level. This source can then be used to generate a photoluminescence signal, as shown in Fig. 5. The data were taken on a grain boundary in CuInSe2, before and after removal of selected Se atoms along its length. The Se-vacancy signal is observed to increase as a result of this atomic processing. Placement of oxygen at these sites, by the process illustrated previously, shows the rise of the oxygen at the Se-site defect, and confirms for the first time on the atomic scale, the actual position of this electronic defect.

![CulnSe2 Nanolevel Photoluminescence](image)

Fig. 5. Nanoscale photoluminescence data from APM for CuInSe2, showing effects of Se removal and O placement.

The use of these nanoscale operations have only begun to have an impact. Their initial application is diagnostic in nature, including the identification of electronic defect processes in photovoltaic semiconductors demonstrated here. The future should bring the ability to produce new materials, to optimize materials, and to grow new structures by atomic manipulations.

**SUMMARY**

Characterization is an important supporting technology for photovoltaics. The range of characterization techniques spans the range from arrays to atoms. The evolution of the characterization technologies has advanced substantially, with many of these techniques evolving in direct response to the requirements of photovoltaics.

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