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### MILESTONE REPORT # 1

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|---|----------------------------|--------------------------------|
| <b>Project Title:</b> Cost-effective Pilot Line for Flexible PV Modules |                            |                                |
| <b>Contract Number:</b> RD3-53  | <b>Milestone Number:</b> 1 | <b>Report Date:</b> 4/22/2009  |
| <b>Reporting Period:</b> October 23, 2009 to April 22, 2009             |                            |                                |
| <b>Milestone Description:</b> Scaled CIS Absorber                       |                            |                                |
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| <b>Congressional District:</b> 24                                       |                            |                                |

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# MILESTONE 1 SUMMARY REPORT

## Executive Summary

The project leverages the results of InterPhases' RD-78 project to realize a commercially viable solar cell technology for large-scale renewable electricity generation. On the path to achieving a cost-effective manufacturing-oriented design for pilot production of PV modules, the new project seamlessly integrates the results of the completed RD2 project.

The R&D is currently progressing along a dual path of process scale-up and simultaneous optimization, aimed at building a strong base for the pilot production. The research continues to simplify the fabrication, increase reliability and further reduce costs of large-scale solar cell production. Several tasks have been initiated, with the current effort primarily focused on developing scaled copper indium selenide (CIS) absorber technology. Along with process scale-up and pilot line development, we have been developing ways to improve device performance. Fruitful relationships have been established with equipment vendors to develop roll-to-roll (R2R) deposition system and other necessary processing and characterization tools. A detailed characterization task has been launched through various collaborations in order to better correlate the material and electro-optical properties with the PV performance. This milestone report summarizes the results of the last 6 months, pertaining to:

- Uniformity of composition, thickness, structural and electrical properties for scaled CIS films
- Process scale-up and development of R2R deposition system components for pilot line
- Alternate treatments to increase crystallinity and grain size for the CIS absorbers.
- Alternate device components and structures to simplify manufacturing, and
- New methods to evaluate structural and opto-electronic properties of films and devices.

Other activities in this period include: (a) new NSF SBIR Phase II award to develop a TCO window, (b) relocation to a large facility, (c) investor/partner interactions, and (d) recruitment of new technical staff.

## Technical Progress:

### 1. Scaled CIS Absorber Deposition and Analysis

Development of the conceptual pilot line continues to advance as we scale CIS deposition to 4" x 6" foils, using high-speed flow cell. In preparation for the R2R processing, different contacting schemes were investigated to determine their effect on the CIS film uniformity over large areas, focusing on the effects of: (a) the location and number of the electrical contacts during deposition on the potential drop, and (b) the agitation and direction of solution flow on the concentration of the depositing species. The films were analyzed using XRF (X-ray fluorescence), Scanning electron microscopy (SEM) and a 4-point probe.



Figure 1. (a) Photograph of CIS film

The electrodeposited samples appeared uniform, smooth and shiny, Fig. 1. Composition and thickness were measured across the sample surface. Fig. 2 is an example of typical composition variability on the large sample. The results confirm that a potential drop across 4–10" distance from the contact produces films with (a) uniform composition (b) small scatter in thickness, due to edge effects and turbulence in the small volume, (c) minor variations, independent of contact location, and (d) low and uniform sheet resistance for samples with uniform grain structure across the surface. The results attest to the high manufacturability of our special process to produce uniform CIS absorbers across large areas.

## 2. Designs for Roll-to-roll deposition

After careful analysis of the systems and extended discussions with suppliers, a new R2R system has been selected for manufacturing low-cost thin-film PV cells. Based on our specific requirements, alternate blue prints for modular turnkey R2R systems were developed by varying the number and duration of pretreatment and processing steps. The system comprises master drive with pay-off and take-up coiler, pumps and rectifiers electroplating cells, spray rinse and blow-off cell systems. System specifications include line speed, substrate thickness & width and contacts.

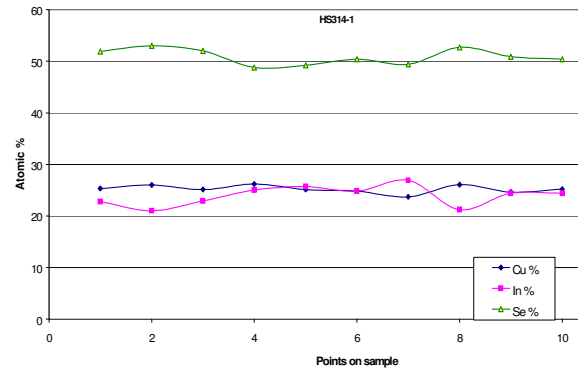


Figure 2. Variation in atomic % of In, Se and Cu for a single contacted sample

## 3. Substrate Treatments

In the context of process scale-up and pilot line development, we have evaluated alternate substrate materials, surface treatments and superstrate configurations that may allow easier deposition and hence are more conducive to the R2R pilot line.

New substrate materials that were investigated provide an alternative low cost deposition method for CIS absorbers. Uniform and compact precursors were deposited and are being further processed into absorbers. This approach allows doping the absorber with elements that are beneficial to the device performance.

Initial research on superstrate device configuration appears to exhibit many promising attributes relative to the current substrate cell; it allows better ohmic contact and easier processing of the window layer prior to absorber deposition. Process parameters are being further optimized to improve the output of PV devices.

Based on the analysis of results of several pretreatments for the current substrates it was determined that an acid etch and rinse were adequate and the most cost-effective step to use with a R2R line. As shown in Fig. 3, the substrate roughness at microscopic levels has no significant effect on the smoothness and adhesion of the absorber layer.

## 4. AFM Characterization of Electrodeposited CIS/steel foil

Characterization studies continue to evaluate the factors that influence the critical PV properties of electrodeposited CIS on foil. The films are 2-3 $\mu\text{m}$  thick and generally comprise In-rich compositions. As-deposited samples show lower resistance, typically in the hundreds  $\Omega/\text{sq}$  or lower. These films are usually degenerate with very low photocurrent.

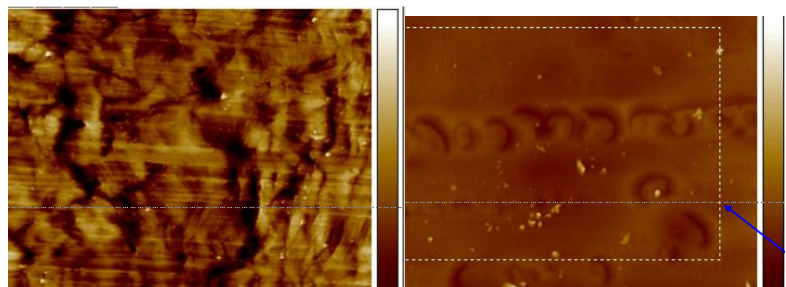


Figure 3. Surface AFM (a) substrate and (b) CIS/substrate

AFM micrographs in Fig. 3 compare the surface roughness for the steel foil and a CIS/foil sample at the same magnification, on a 100 $\mu\text{m}$  x 100 $\mu\text{m}$  area. The result shows that the CIS film is 30 times smoother than the metal substrate. Thus the microscopic defects and imperfections of the substrate do not transmit into the electrodeposited CIS layer. This indicates that the device can use lower quality metal foil with minimal pretreatment to remove macroscopic roughness, further reducing its cost.

Conductive AFM, Fig. 4, uses a secondary imaging mode derived from contact AFM to detect conductivity variations in the CIS semiconducting film. With conductivity probe grounded, the data shows higher conductivity (upto 100mV) between the grains, possibly due to the grain boundaries or substrate defects. Such areas could provide a shunting path that may lower the photocurrent.

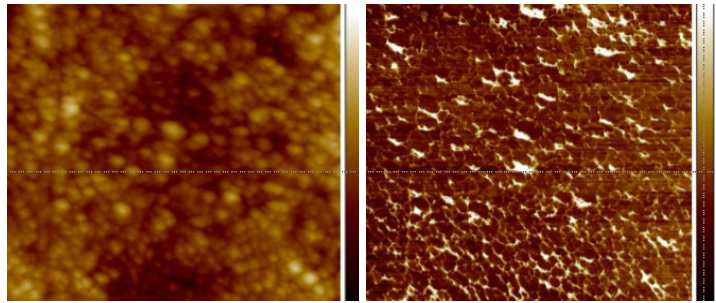


Figure 4. AFM micrograph under 10V bias and  $10^6$  gain

## 5. Anneal/Recrystallization

Low temperature deposition processes require annealing to minimize grain boundary recombination and thus enhance device performance. A number of alternate approaches and post-treatments were devised to induce grain growth in the films. New rapid thermal processing (RTP) equipment was tested by varying the process conditions. Very aggressive annealing conditions were employed to establish an upper tolerance limit for the CIS films. Two sets of annealing procedures were adopted: (RTP-I) low temperature anneal for long periods or (RTP-II) short pulses at high temperature. Their effects were monitored with SEM, AFM, XRF, XRD, sheet resistance and photocurrent measurements.

Annealing treatments increase film resistivity to the  $M\Omega/sq$  range, reflecting decreased degeneracy from reorganization of the electronic structure. The cross sectional SEM shows that the CIS film is  $>4\mu m$  thick, compact and smooth, Fig. 5a. RTP-I enhances the photocurrent without noticeable changes in the film morphology and composition. RTP-II conditions tend to decompose the CIS into volatile Se and InSe compounds. The small grains appear to coalesce into larger columnar grains, Fig. 5b and the resistance increases. Sequential RPT-I and RTP-II steps also resulted in significant loss of In and Se. These conditions provide the limits for annealing CIS/SS samples. Beyond these limits the CIS films tend to peel off the substrate.

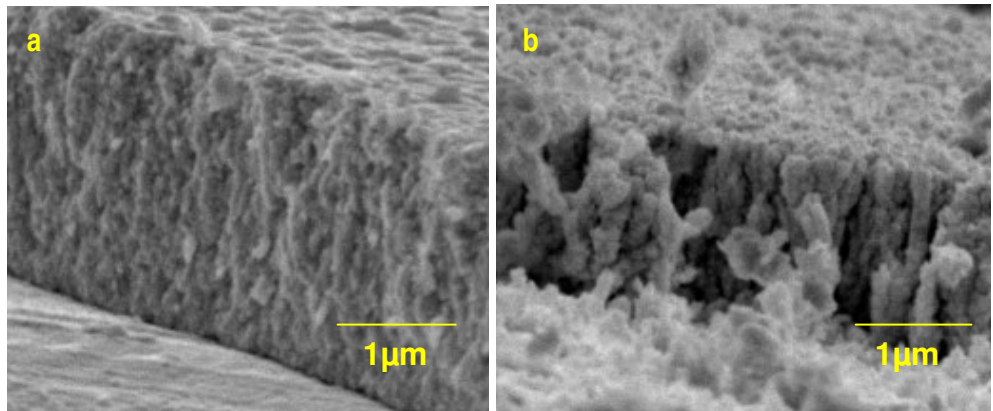


Figure 5. SEM of CIS films after (a) RTP-I and (b) RTP-II

## Other Activities:

InterPhases relocated to a larger facility in Newbury Park on March 27, 2009. Ongoing collaborations with universities and National Labs continue to generate new data. New collaborations have been initiated with university faculty, manufacturers of processing and characterization equipment. In view of the company expansion for pilot development, we have interviewed a number of prospective PhD level candidates in various project-related disciplines. We are in the process of making the selections. The PI participated in a conference on 'Solar Fuels using Electrochemistry' and initiated new academic contacts for potential collaboration in the area of morphology control. Discussions and visits with potential manufacturing partners are in progress to negotiate a co-operation for pilot development.

**Deliverables:**

1. Milestone 1 report document
2. Sample of absorber on 10cm strip
3. Copy of Lease of the new facility
4. Copy of NSF award letter

| <b>Milestone</b> | <b>Name</b>                               | <b>% Completed</b>  |
|------------------|---|---|
| 1                | Scaled electrodeposition for absorber CIS | 100%. Research continues to optimize and scale up the deposition and post thermal treatments CIS films. |
| 2                | Scaled strip cell                         | 5%. Research on alternate materials and device configurations.  |

**Project Status:**

The new tasks for pilot development of CIS solar cells for achieving low cost PV electricity generation are progressing on schedule.