Technology Transfer of the CIGS Technology from the Laboratory to the PVMC

Cooperative Research and Development Final Report

CRADA Number: CRD-12-496

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NOTICE

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Cooperative Research and Development Final Report

In accordance with requirements set forth in the terms of the CRADA agreement, this document is the final CRADA report, including a list of subject inventions, to be forwarded to the DOE Office of Science and Technical Information as part of the commitment to the public to demonstrate results of federally-funded research.

Parties to the Agreement: U.S. Photovoltaic Manufacturing Consortium, Inc.

CRADA Number: CRD-12-496

CRADA Title: Technology Transfer of the CIGS Technology from the Laboratory to the PVMC

Joint Work Statement Funding Table showing DOE commitment:

Estimated Costs	NREL Shared Resources a/k/a Government In-Kind
Year 1	$100,000.00
Year 2	$100,000.00
Year 3	$100,000.00
Modification #1	$867,157.00
Modification #2	$12,167.00
Modification #3	$91,000.00
TOTALS	$1,270,324.00

Abstract of CRADA Work:

PVMC, Inc. (PVMC) and Contractor (NREL) will work together under a CRADA, through a distinct statement of work (SOW) for each project. Each SOW will be negotiated as a modification of the CRADA between PVMC and NREL with a principal investigator (PI) identified by the contractor so as to define specific tasks and funding that reflect the scope and objectives of that project. Projects will focus on extended collaborations with well-defined, long-term goals that are mutually beneficial to PVMC and NREL. Projects under the CRADA may fall within the areas listed below in the summary of research results, calling upon the NREL capabilities and expertise.

Modification 1: The first year of the CRADA will involve training PVMC personnel in the deposition process of the layers in the CIGS device based on the evaporation process, characterization methods used to track the properties of the films, device fabrication and integration of the layers into device compatible with the PVMC approach, and use standard ex-situ characterization tools, and standard and novel in-situ metrology tools for process characterization to insure that the unit films in the MDF facility have the properties defined

above in the unit step evaluation. The scope of the CRADA statement of work is based on knowledge already in the literature and/or in background published IP.

Modification 2: NREL loaned Ocean Optics spectrometer to PVMC. This was returned on 2/15/2018.

Modification 3: The work under this CRADA is aimed at understanding several process effects on silicon wafers and/or solar cells. These include: (i) effects of wafering by diamond wire sawing on the surface damage and the strength of wafers, (ii) electronic characteristics of dielectric-Si interfaces, and (iii) characteristics of Si-Al interfaces formed during firing of silicon solar cells.

Summary of Research Results:

Please note the original PI on this project is no longer at NREL and neither is his predecesor. A stop work order was issued for this project in December 2013, so not all of the items were completed. Notes are made about some of the previously documented work.

1. **CIGS Cell and Module Structure Process and Equipment Optimization, towards PV Manufacturing** – Optimize manufacturing processes and equipment to close the efficiency gap between lab prototyping and commercial production of high efficiency CIGS cells. Assist in implementing productivity approaches towards developing factory systems for next generation PV fabrication.

   PVMC staff members visited NREL and observed CIGS device processing. This included all layers from cleaning glass through top grid contacts. Personnel were trained to implement the NREL 3-stage process for depositing high-efficiency CIGS absorbers. NREL absorber samples were sent to PVMC for device finishing. Current-voltage measurements of these devices were compared to devices completed at NREL. NREL also provided SIMS depth profile data for comparison with CIGS absorbers fabricated at PVMC.

2. **Accelerated Testing and Failure Analysis – Methodology Development for CIGS Reliability Enhancement** – Characterize CIGS materials and process integration interactions to develop specific models around failure mechanisms, performance, and yield to provide direction for process and equipment improvements.

   This task was not fully implemented.

3. **Metrology Development –** Develop metrology techniques for CIGS materials and PV modules.

   Development was started on the use of optical ellipsometery for CIGS characterization. This report describes the preliminary analysis of a real sample made by the NREL CIGS group. The structure is CIGS/Mo/glass. The CIGS layer was deposited with in industry-like process: sputtering Cu+Ga/sputtering In/selenization. The average Ga/III ratio measured by XRF is 0.31. The cross-sectional SEM image and the SIMS depth profile are shown in Figure 1. The experimental reflectance data and the fit are shown in figure 2. The fit returned the following parameters:

1. Total CIGS thickness of 1470 nm.
2. On top of Mo, there is a 57 nm thick interface layer that contains 23% Mo and 77% CIGS.
3. On top of layer-2, there is a porous CIGS layer 494 nm thick with 20% void and 80% CIGS. The Ga/III ratio in the CIGS in layers 2 and 3 are both 0.5.
4. On top of layer-3 is the dense CIGS layer. Its total thickness is 896 nm, with a bottom Ga/III ratio of 0.5 and a top ratio of 0.1.
5. On top of layer-4 is the surface roughness layer 24 nm thick.

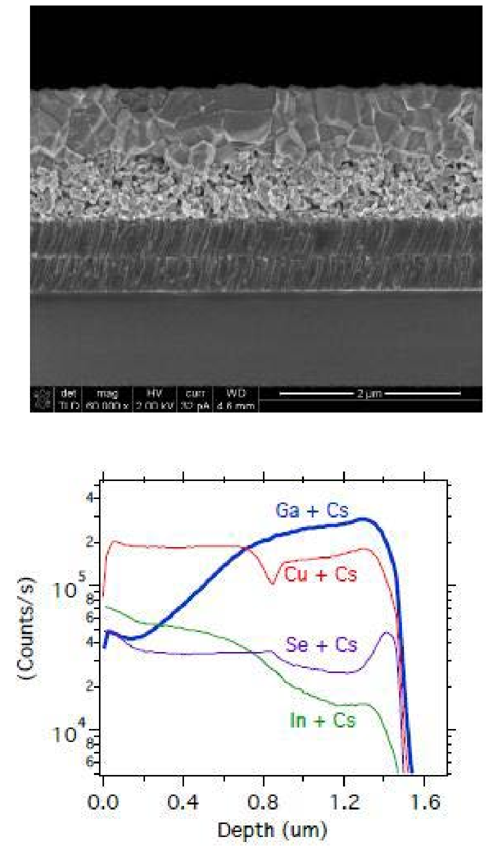

Figure 1. The cross-sectional SEM image and the SIMS depth profile of the measured sample.

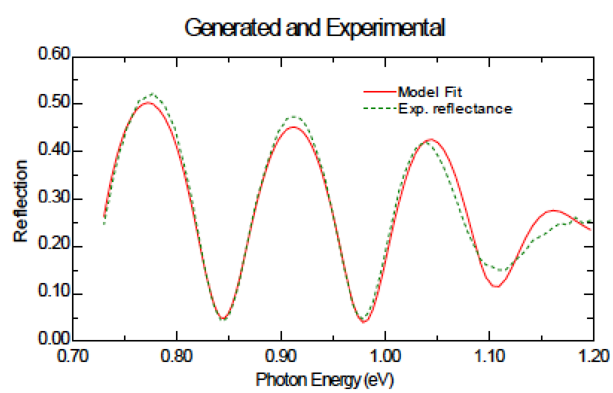

Figure 2. The experimental reflectance data and the model fit.

That the difference between data and fit is similar to or may be even larger than the difference between two previously simulated curves corresponding to different Ga profiles. It seems single-shot optical metrology alone is not a reliable way to deduce the Ga profile. We shall try to highlight the potential for process control, meaning if we have a standard process and its standard optical signal, then certain deviations from it can be detected. More simulations will be done for this purpose.

Both p- and s- polarized reflectance were measured in a wide range of angle of incidence: 15 – 80 degree. Reflectance measurements become less accurate at larger AOIs, indicated by a c-Si sample. The curves in my report is p- polarized reflectance at AOI=15 degree. I also took the ellipsometry data of these samples. I found large discrepancy between the fit results based on SE and R data. Actually I cannot find a good fit to the SE data, possibly because it is too sensitive to the surface conditions.

The SEM and SIMS data of all of the four samples from NREL are in Figure 3. Figure 4 compares their p- reflectance at AOI=15. The significantly lower reflectance for C3462 and C3526 is due to the surface scattering, instead of anything in the bulk of the CIGS film. It is necessary to take this scattering into consideration for reflectometry analysis. This is one lesson learned from measuring NREL samples. The ellipsometry software we are using does not support this capability.

Sample #	C3394	C3462	C3529	C3526
Type	CoEvap	Metal	Industry-like	Metal
Stack	No, Cu:In:Ga:Se	Cu/Ga/In	CuGa/In	Cu/Ga/In

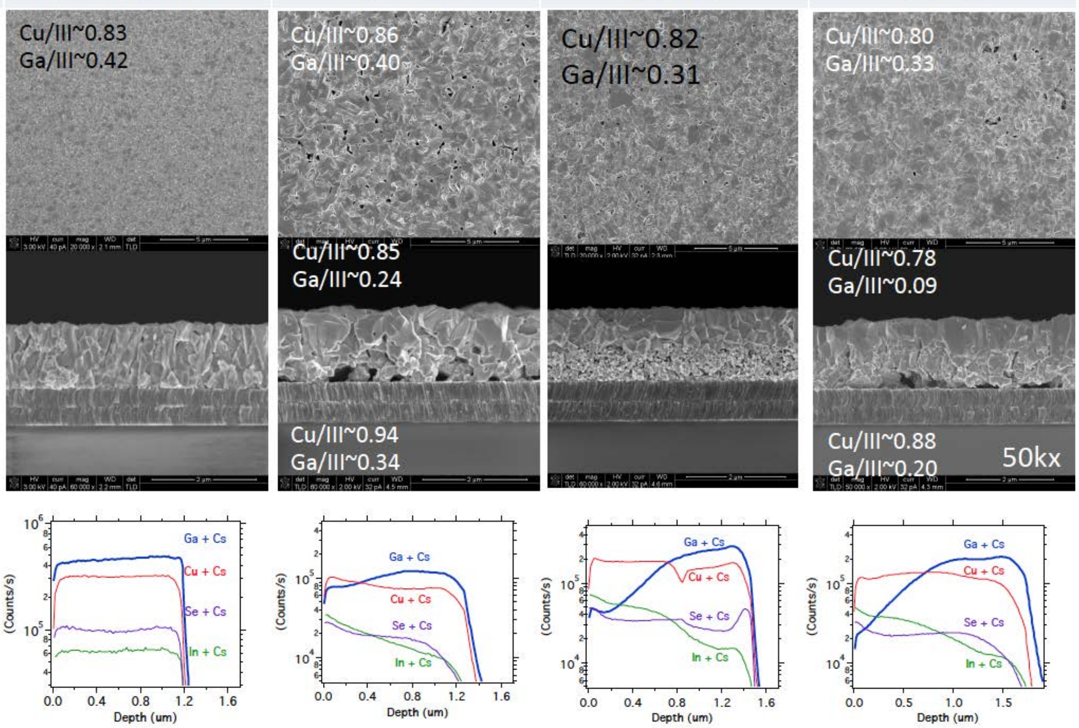

Figure 3. Plan view SEM (top images), cross-sectional SEM (center images), and SIMS depth profiles (bottom graphs) of CIGS absorbers made from different process noted in the rows above.

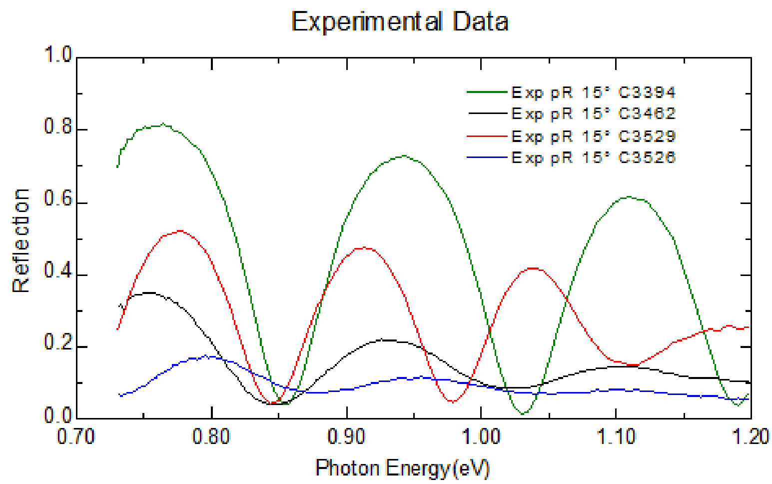

Figure 4. The experimental reflectance data from the four samples in Figure 3.

4. **Cost Modeling and Market Analysis** – Create cost models of CIGS PV modules, broken down by material and process step, to assess future cost targets by category needed in order to reach the SunShot goals.

A presentation was given to PVMC by Alan Goodrich from NREL in March of 2013. It discussed a market summary and analysis in support of R&D strategies on crystalline silicon, CdTe, and CIGS. PVMC published their own cost model at IEEE PVSC in 2013. This task was not fully implemented.

Subject Inventions Listing:

None

ROI #:

None

Report Date:

30 April 2018

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