Evaluation of Solar Grade Silicon Produced by the Institute of Physics and Technology

Cooperative Research and Development Final Report

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In accordance with Requirements set forth in Article XI.A(3) of the CRADA document, this document is the final CRADA report, including a list of Subject Inventions, to be forwarded to the Office of Science and Technical Information as part of the commitment to the public to demonstrate results of federally funded research.

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CRADA Title: Evaluation of Solar Grade Silicon Produced by the Institute of Physics and Technology

Parties to the Agreement: Solar Power Industries

Joint Work Statement Funding Table showing DOE commitment:

Estimated Costs	NREL Shared Resources
Year 1	$425,000.00
Year 2	$575,000.00
Year 3	$00.00
TOTALS	$1,000,000.00

Abstract of CRADA work:

NREL and Solar Power Industries will cooperate to evaluate technology for producing solar grade silicon from industrial waste of the phosphorus industry, as developed by the Institute of Physics and Technology (IPT), Kazakhstan. Evaluation will have a technical component to assess the material quality and a business component to assess the economics of the IPT process. The total amount of silicon produced by IPT is expected to be quite limited (50 kg), so evaluations will need to be done on relatively small quantities (≈ 5 kg/sample).

Department of Energy (DOE) laboratories, including NREL, have been placing subcontracts for research and development services with scientific institutes in the Newly Independent States (NIS) under various DOE programs for many years. Currently, there are approximately 190 subcontracts under a relatively new DOE program known at the Newly Independent States - Industrial Partnering Program (NIS-IPP). The remaining subcontracts with NIS scientific institutes are under various other DOE programs.

The NIS-IPP supports the national security interest of preventing the proliferation of weapons of mass destruction through cooperative projects between the United States and military-related institutes in the NIS. The goal of NIS-IPP projects is to redirect technologies, materials, resources, and personnel in the NIS to non-military scientific and commercial research and development. NIS-IPP projects are being implemented through subcontracts funded by the U.S. Government between NIS military-related

institutes and the following 10 DOE national laboratories: Argonne National Laboratory, Brookhaven National Laboratory, Idaho National Engineering Laboratory, Lawrence Berkeley National Laboratory, Lawrence Livermore National Laboratory, Oak Ridge National Laboratory, Pacific Northwest Laboratory, and Sandia National Laboratories Los Alamos, the National Energy Technology Laboratory (NETL), and NREL.

Summary of Research Results: There were several tasks outlined by the CRADA agreement that defined the roles of NREL and Solar Power Industries (SPI), they are summarized below:

SPI Tasks

Task 1. Qualification run of minicaster with undoped semiconductor grade silicon

Task 2. Minicaster ingot with doped semiconductor grade silicon (month 9)

Task 3. Minicaster ingot with undoped IPT silicon (month 12)

Task 4. Minicaster ingots with doped IPT silicon (month 21)

Task 5. Economic assessment of IPT silicon process (month 24)

Task 6. Reporting and deliverables (month 25)

NREL Tasks

Task 1. Silicon from the IPT process (ongoing, months 9 through 18)

Task 2. Monocrystalline ingot data (month 21)

Task 3. Characterization (ongoing, months 3 through 24)

Solar Power Industries performed Tasks 1 and 2 and reported in one single report since it made sense to combine the cell fabrication, sorting, and testing in one run on their solar cell line. The electronic grade polysilicon cast well in the mini-caster producing 156 mm x 156 mm square cells with a thickness of 220 μm, which allowed them to be processed on a manufacturing line with little more than attention to position in the line. The finished cells demonstrated that the furnace was capable of producing good ingots with average efficiencies for doped and undoped cast wafers of 15% and 13% efficiency respectively. The doped electronic grade wafers were p-type 2 Ω-cm with a before and after processing lifetime of 1-30 μs to 15.8 μs respectively. Lifetime and resistivity data showed that the undoped wafers were lightly doped p-type 10 Ω-cm with a before-processing lifetime range of 1-26 μs, which was improved by cell processing to 15-26 μs.

For Task 3 IPT submitted their first batch of polysilicon but were disappointed in its characteristics. While the polysilicon material had acceptable metallic impurity levels for poor solar grade silicon, the material did not satisfy SPI's requirement for dopant impurities. According to Chenlei Wang, Chief Engineer at SPI, "The grain structure demonstrated that 5 kg ingots can be successfully cast using IPT silicon. However, the impurity measurement (B 7.7-24 ppmwt, Al 1.8-22 ppmwt, P 1.3-44 ppmwt) and resistivity mapping (0.1 to 0.3 Ω-cm) show the quality of the first shipment of IPT silicon did not meet SPI's requirement of Upgraded Metallurgical Grade Silicon, which is Boron < 0.5 ppmwt, Phosphorus < 2.0 ppmwt. Although the resistivity and lifetime (unmeasureable) are very low, this ingot will be sliced into 220 μm thick wafers and processed into cells to conclude Tasks 3.2 and 3.3. The next step will be Task 4, which will be to cast a 5 kg ingot with improved silicon from IPT doped to 1.0 Ohm-cm resistivity."

Upon completion of solar cell fabrication on full 156 mm x 156 mm wafers (58), 38 cells survived the processing line with lots of edge chipping and some breakage. All the solar cells fabricated out of the undoped IPT polysilicon material were shunted with no measureable efficiency. SPI works quite a bit with upgraded metallurgical silicon and has a process that can create 14% efficiency solar cells. SPI's opinion of this first batch of material is: "The material (undoped IPT silicon) is not suited for cell production."

While IPT sent 4 allotments of polysilicon from subsequent purification runs, they were not able to secure the necessary input materials to make high purity upgraded metallurgical grade (UMG) polysilicon with suitable impurity content before the fourth allotment. The second and third allotments of polysilicon had impurity contents summarized below:

Allotment (Date In)	Boron (B)	Phosphorus (P)	Aluminum (Al)	Transition Metal
2nd (11/4/2008)	2.2-7.3	15-84	1.9-260	<0.01-0.1wt %
3rd (9/25/2009)	1.5-3.2	7.6-30.2	<1	<1
4th (3/1/2011) Fines				

The third allotment arrived too late and had an impurity content too high for SPI's typical UMG polysilicon specification to perform another cast with dopant added. The fourth allotment was produced at IPT in Almaty, Kazakhstan and arrived well after the close of the CRADA. This material is still stored at NREL, where it awaits solidification or return to Kazakhstan.

Because the purity of IPT silicon was not able to reach expectations, SPI tasks 4 through 6 were never completed and Solar Power Industries laid off the last of its CRADA-linked technical contacts on June 30, 2011.

NREL tasks 1 and 3 were completed as samples were returned to NREL. Our measurements confirmed that this material is unsuitable for crystal growth and will not make good solar cells based on the conventional cell processing that SPI uses. There was some talk about including a collaborator to work with the heavily compensated material that we have. Calisolar was contacted by IPT to perform some cell fabrication, but we never received results from this work. It is unlikely Calisolar was able to achieve any better results because they do not have casting furnaces capable of handling such small amounts silicon. NREL attempted some gettering and solar cell fabrication on IPT-grown CZ multicrystalline ingots but the impurity content was too much for solar cell processing. No attempts were made to grow CZ crystals from IPT feedstock because DOE removed the program from the Silicon Team annual operating plan.

The latest allotment of UMG polysilicon may be suitable for a CZ crystal pull in the future if we can raise funds to do a test pull on 3 kg of UMG polysilicon that IPT delivered March 1, 2011. Civil unrest in the region where IPT was growing CZ crystal to consolidate the UMG polysilicon fines pushed deadlines beyond the scope of this CRADA and the original NIS-IPP project was terminated November 30, 2009 after it had been extended one or two times. We have yet to secure funding to attempt a CZ crystal pull of the fines material, and with the current climate at DOE I don't think we ever will, especially given that the industry is no longer interested in UMG Polysilicon due to the drastic drop in electronic-grade polysilicon commonly used in the solar industry today.

Subject Inventions listing: None

Report Date: 4/25/2012 **Responsible Technical Contact at Alliance/NREL:** Matthew Page

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