PVMaT Improvements in the Solarex Photovoltaic Module Manufacturing Technology

Annual Subcontract Report
May 5, 1998 — April 30, 1999

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Solarex, A Business Unit of Amoco/Enron Solar
Frederick Maryland
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NREL Technical Monitor: R.L. Mitchell

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PREFACE

This Annual Technical Progress Report covers the work performed by Solarex for the period May 4, 1998 to April 30, 1999 under DOE/NREL Subcontract # ZAX-8-17647-05 entitled "PVMaT Improvements in the Solarex Photovoltaic Module Manufacturing Technology". This is the first Annual Technical Report for this subcontract. The subcontract is scheduled to run from May 4, 1998 to May 3, 2001.

The following personnel at Solarex have contributed to the technical efforts covered in this report.

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Solarex has been supported by subcontracts at the Automation and Robotics Research Institute at the University of Texas at Arlington (ARRI), at Specialized Technology Resources, Inc., SiNaF Products, Inc., MV Systems, Inc. and the Physics Department at North Carolina State University.

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EXECUTIVE SUMMARY

The objective of this three year PVMaT program is to continue the advancement of Solarex PV manufacturing technologies in order to design and implement a process which produces polycrystalline silicon PV modules that can be sold profitably for $2.00 per peak watt or less and which increases the production capacity of the Frederick plant to at least 25 megawatts per year. Achieving these major objectives is based on meeting the following specific task goals.

Develop a process to produce silicon feedstock from Na$_2$SiF$_6$ that can be sold profitably for less than $15$/kilogram in large quantities. Demonstrate the process in a pilot facility.

Optimize and improve control of the casting process to increase the process yield (kilograms of silicon out divided by kilograms of silicon in) by 7% and to improve material quality such that average cell efficiency increases by 4%.

Reduce the center-to-center cut distance on the wire saw to less than 450 $\mu$m in production, and develop a wire saw process that reduces the consumable costs by at least $0.05$/wafer, that does not require organic cleaners nor result in generation of hazardous waste material.

Develop, demonstrate and implement a cost-effective cell process that produces a minimum average cell efficiency of 15% and improves the cell line electrical yield by 5% when applied to Solarex cast polycrystalline silicon wafers.

Develop and qualify an encapsulation system that meets all technical and reliability requirements and can be laminated and cured in less than 6 minutes.

Improve Solarex's product and materials handling (including efforts in at least 3 separate areas) to increase line yield by 3% and reduce handling labor to save $0.05$/watt.

Improve process measurement and control in the production line (including efforts in at least 3 separate areas) to improve yield by 3% and reduce rework by 50%.

ACCOMPLISHMENTS

Accomplishments during the first year of the program include:

- Verification of the process to produce SiF$_4$, the precursor to silicon feedstock.
- Design of a silicon feedstock pilot facility using the SiNaF process.
- Development of and transfer to manufacturing of a process to use thinner wire in the wire saw.
- Completion of a production trial with recycled SiC.
- Laboratory development of a selective emitter process using rapid thermal processing.
- Fabrication of high efficiency polycrystalline cells using silicon nitride from three different sources.
- Development of a new encapsulation formulation and laboratory demonstration of a six minute lamination cycle.
- Implementation of an automated laminator.
- Laboratory demonstration of automated handling in ceramics.
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1.0 INTRODUCTION

The goal of Solarex's Crystalline PVMaT program is to improve the present Polycrystalline Silicon manufacturing facility to reduce cost, improve efficiency and increase production capacity. Key components of the program are:

- Developing a solar-grade silicon feedstock.
- Optimizing and improving process control in casting.
- Reducing wire diameter and wafer thickness to reduce silicon feedstock requirements.
- Reducing wire saw operating costs.
- Developing a faster cure encapsulant to reduce labor and equipment requirements.
- Increasing cell efficiency and using more robust processes.
- Improving handling and eliminating non-value added handling steps.
- Improving measurement and control throughout the production line.

The results of these efforts will be to reduce PV module production costs so that they can be sold profitably for $2.00 per peak watt or less, to increase the production capacity of the Frederick plant to at least 25 megawatts per year and to improve average cell efficiency to 15%, a secondary effect of which is to reduce the customer’s balance of systems (BOS) costs. These goals are to be achieved while improving the already high reliability of today’s crystalline silicon modules.

Solarex is being supported in this effort by five subcontractors:

1. SiNaF Products, Inc. to work on developing a process and the equipment necessary to produce a solar-grade silicon feedstock.
2. Specialized Technology Resources, Inc. (STR) to develop, qualify and manufacture a faster cure encapsulation system.
3. MV Systems, Inc. to develop production furnaces for the deposition of silicon nitride AR coatings on solar cells.
4. Automation and Robotics Research Institute of the University of Texas at Arlington to assist Solarex in developing equipment for automated handling and improved measurement and control on the production line.
5. North Carolina State University to support Solarex in characterizing silicon nitride films and determining how these films can be used to passivate Solarex cast polycrystalline silicon.

The baseline for this PVMaT program is the polycrystalline process and production line as it existed at the conclusion of Solarex’s previous PVMaT Contract NREL # ZAI-4-11294-01 entitled “Cast Polycrystalline Silicon Photovoltaic Manufacturing Technology Improvements” 1, 2. This baseline is described in more detail in Section 2.0.

The rationale behind the development efforts in this program was to identify specific areas in the baseline process where improvements in handling, process control or the process itself could significantly reduce cost, increase efficiency and/or improve capacity. In addition to these process issues, a materials availability analysis indicated that silicon feedstock must be addressed for the long term success of crystalline silicon PV.

Most of Solarex's (and the crystalline Si PV industry’s) silicon feedstock is scrap purchased from the semiconductor industry. Until 1995 most of this material consisted of tops and tails purchased at low cost. In 1995 a shortage of semiconductor grade silicon lead the crystal growers...
to follow the PV industry lead and reuse their own tops and tails. As the supply of tops and tails
dried up, Solarex and the rest of the PV industry were forced to find new sources of silicon
feedstock. While this shortage has alleviated due to the down turn in semiconductor sales, the
cost has never returned to pre-shortage levels, nor will the available supply of scrap be adequate
to meet the needs of an expanding PV industry. Therefore, Solarex is working with SiNaF
Products, Inc. to develop a solar-grade silicon process to produce the large quantities of silicon
feedstock necessary to support growth in the PV industry.

The present casting stations are limited in the amount of data that they can collect and in the
number and variety of parameters they can control. Production data shows that average cell
efficiency and yields vary considerably from station to station and even from run to run in the
same station. Effort are underway to provide better control and optimization of the casting
process to improve both yields (casting and cell line) and material quality, that should translate
into higher average cell efficiencies.

The wire saw technology is still evolving and improving. A number of potential changes have
been identified that can reduce cost and increase even further the number of wafers/cm of brick
that can be cut, thereby reducing the amount of silicon necessary per watt of modules produced.

Work on cell processing is designed to increase the average cell efficiency to 15% (at Standard
Test Conditions) while improving process control and reducing the overall module manufacturing
cost. This is being accomplished by utilizing new process steps  (PECVD silicon nitride AR
coating and selective emitter diffusion) and by modifying some of the present process steps
(doping, edge isolation and screen printing) to make then more robust, that is easier to control for
higher yields and better efficiency.

The present “Fast Cure” EVA encapsulation system is a significant improvement over the old
“Standard Cure” system. However, even with this system, increasing production volume still
requires a significant investment in lamination equipment, plant floor space and handling
equipment. STR is developing a new encapsulation system, with the goal of reducing the
lamination time to less than 6 minutes.

Although most cell and wafer handling functions have been automated or are in the process of
being automated, there are still a number of areas in the plant where handling improvements can
significantly reduce labor cost and/or improve yield. ARRI assisted Solarex  in identifying those
area in which improvements in handling can have the greatest impact on reducing cost and
increasing capacity. Several handling projects were begun during the first year of the program.

Improved measurement and control during processing should lead to improved yields and higher
average cell efficiency. Efforts during the first year include an assessment of the data collection
and measurement system itself, development of a brick identification system and identification of
one specific area in the plant where additional inline measurements are expected to significantly
improve control of the process.

First year efforts in each these technical areas are discussed in Section 3.0.
2.0 PRESENT PROCESS

Solarex's Crystalline Silicon Technology is based on use of cast polycrystalline silicon wafers. The process flow at the end of NREL Contract # ZAI-4-11294-01 is shown in Table 1.

Table 1
Cast Polycrystalline Si Process Sequence

<table>
<thead>
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<th>Casting of 9 Brick Ingots</th>
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<tr>
<td>Wire Saw Wafering</td>
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<tr>
<td>Cell Process (All Print with Al BSF)</td>
</tr>
<tr>
<td>Module Assembly (XY Positioners)</td>
</tr>
<tr>
<td>Lamination (Fast Cure EVA)</td>
</tr>
<tr>
<td>Finishing</td>
</tr>
</tbody>
</table>

The various segments of Solarex's module manufacturing process as practiced at the beginning of this PVMaT program are described below.

Casting

Solarex has developed and patented a directional solidification casting process specifically designed for photovoltaics. In this process, silicon feedstock is melted in a ceramic crucible and solidified into a large grained semicrystalline silicon ingot. During Solarex’s earlier PVMaT program the size of the cast ingot was increased so that each ingot now yields 9 - 11.4 cm by 11.4 cm bricks. This change was implemented in production resulting in a significant reduction in operating costs and in a doubling of the casting capacity at approximately 20% of the cost that would have been required if the same amount of capacity was added by purchasing new casting stations.

Wafering

During the previous PVMaT Program Solarex developed wire saw technology for cutting large area polycrystalline wafers. The major advantages of wire saws are the reduced kerf and increased production volume. During the course of the PVMaT program we demonstrated:

- That each wire saw cuts as many wafers as 22 to 24 ID saws.
- Wire saws can cut wafers on 475 μm centers versus 600 μm centers for the ID saws, resulting in at least 20% more wafers from each cast ingot.
- Wire saws can cut larger wafers (in our case 11.4 cm by 15.2 cm and 15.2 cm by 15.2 cm) while the ID saws are limited by the blade diameter.

Today all of Solarex’s wafering is performed using wire saws.
Cell Process

The cell process sequence is based on the use of Thick Film Paste (TFP) metallization, where a commercially available screen printed silver paste is applied as the current carrying grid on the front of the solar cell. This process has been designed to be as cost effective as possible. The high temperature process steps including diffusion, firing of the front and back print pastes and Chemical Vapor Deposition (CVD) of a TiO₂ antireflective (AR) coating are all performed in belt furnaces.

During the previous PVMaT Program, Solarex developed a cost effective aluminum paste back surface field (BSF) process. This process was implemented on the Solarex production line using a fully automated screen printing system provided by Baccini. The system is fully operational and has provided a 6.5% increase in average cell efficiency.

Module Assembly

The first part of the module assembly sequence is to solder two solder plated copper tabs onto the front of the solar cells. Solarex procured new automated machines to perform the tabbing operation. Tabbed cells are then laid up into a matrix and the tabs are then soldered to the backs of the solar cells using XY positioners developed in the previous PVMaT program.

Module Lamination

The module construction consists of a low iron, tempered glass superstrate, EVA encapsulant and a Tedlar backsheet. The lamination process, including the cure, is performed in a vacuum lamination system. Then the modules are trimmed and the leads are attached. Finally, every module is flash tested to determine its STC power output.
3.0 PVMAT PROGRAM EFFORTS

The following sections detail the progress made during the first year of the program.

3.1 TASK 1 – Silicon Feedstock Development

In the task 1 effort Solarex worked with subcontractor, SiNaF Products, Inc. to develop a process to produce silicon feedstock from commercial grade H$_2$SiF$_6$ that can be sold profitably for less than $15/kilogram in large quantities. In Year 1 this effort included design of a pilot line for the production of silicon feedstock material using the H$_2$SiF$_6$/Na process.

There are two distinct production processes that comprise the method under development by SiNaF for the production of solar grade silicon. The two processes are the generation of a high purity gas, and its reduction to form the silicon (Si) and co-produced sodium fluoride (NaF).

Gas Generation

The first step in the gas generation was an extensive review of the literature that led SiNaF away from technology proposed by SRI International (SRI). Literature references were adapted resulting in the generation of a high purity intermediate gas, silicon tetrafluoride (SiF$_4$), that has lowered by a factor of ten (10) the capital costs associated with its generation. Equipment was developed and purchased that led to laboratory gas generation using this new technology. A sample of commercial SiF$_4$ was obtained from MEMC, and a comparative analysis was accomplished with the assistance of Sandia National Laboratories. The comparative estimates, provided in Table 2, are believed to be accurate within 25%.

<table>
<thead>
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<th>Origin of Gas Sample</th>
<th>Hydrogen Fluoride (HF)</th>
<th>Hydrogen Chloride (HCl)</th>
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<td>SiNaF Products</td>
<td>25 ppm</td>
<td>15 ppm</td>
</tr>
<tr>
<td>MEMC</td>
<td>40 ppm</td>
<td>500 ppm</td>
</tr>
</tbody>
</table>

The results of this comparative study are very encouraging because it shows that SiNaF can generate the SiF$_4$ gas with a purity equal to or better than that which is commercially available. These results have allowed SiNaF to optimize the process for easy scaling to pilot production plant volumes.

The primary feed material for gas generation, also identified by SRI, is generated by the phosphate fertilizer industry. This acid is over produced by the industry. However, the over production is insufficient to meet the current as well as the future needs of the photovoltaic (PV) industry. This has led SiNaF to identify other inexpensive starting materials that can supplement its need for a raw material supply to be used in its technology.

SiNaF has identified two additional silicon-containing materials for the production of the gaseous intermediate. They are a spent catalyst from the petroleum industry and mine tailings, generated from hard rock mining activities and accumulated over the last 150 years in the American west.
Both materials not only produce the needed gas intermediate but allow the further production of metals associated with the starting materials. These technologies developed by SiNaF for added gas production have established an unlimited raw material base for the production of PV grade silicon.

Silicon Production

Working in conjunction with Sandia National Laboratories, SiNaF conducted a study of the interaction of a graphite crucible, sodium fluoride and granular silicon “fines” obtained from MEMC. These silicon “fines” were mixed with reagent grade sodium fluoride and heated to 1450° C. After quenching, the crucible and its contents were stabilized (capped) with epoxy and sectioned. These sectioned pieces were studied using a scanning electron microscope (SEM) at the Department of Earth and Planetary Science on the campus of the University of New Mexico.

Figure 1 is a micrograph showing micrometer sized silicon intermingled with the NaF slag. The picture also shows some of the graphite crucible in the lower left hand corner. Because of the gray scale back-scatter electron images, the Si appears brighter than the NaF, which is in turn brighter than the graphite of the crucible and epoxy cap. In Figure 2 the Si is white, NaF is gray and graphite is back.

Some general observations and conclusions:

- All the micrographs show the distinct phase separation between the silicon (Si) and sodium fluoride (NaF). This phase separation is very significant because of the apparent ease of separation of the two phases. This will be very important when actual products are produced and separated commercially.
- The NaF is observed penetrating the crucible material. This is significant because of the nature of the Si expansion upon cooling. The lack of Si penetration of the crucible material should avoid attack of the container walls during the actual manufacturing processes.

SiNaF is continuing to work with Sandia National Laboratories to obtain further data on materials to assist in design and construction of the reduction reactor.

Business Plan and Development

SiNaF has been working with Technology Ventures Corporation (TVC), a Division of Lockheed Martin Corporation, to develop a business plan that can be presented to interested financial institutions. TVC provides business development services to start-up technologies. SiNaF has received an invitation to participate in the 1999 Equity Capital Symposium sponsored by TVC to be held in Albuquerque, New Mexico. At this symposium SiNaF will be provided exposure to venture capitalist funding sources.

Engineering and Design

SiNaF has been working with Viridian Technology Incorporated, a Michigan engineering firm, to develop and implement design criteria for laboratory and pilot plant facilities. This cooperative effort has led to a new and innovative design for reduction reactors including both batch and continuous configurations. This work has developed to a point where the cost of a continuous reactor has been estimated. With the completion of the investigation of materials of construction by Sandia National Laboratories, the final reactor costs and configuration will be prepared.
Figure 1
Micrograph of Si mixed with NaF slag

Figure 2
Micrograph of Si (white), NaF (gray) and graphite crucible (black)
This cooperative effort has also led to laboratory experiments for the optimization of the gas generation process from the engineering scale-up perspective. The equipment list for the gas generation side of the pilot plant/mini-production facility is almost complete.

In the past, Viridian has worked closely with DuPont Sodium in the design of sodium metal storage, transfer and metering systems. These systems have been constructed and are currently being used by a number of industries. Viridian will be able to use these “off-the-shelf” designs in the plant configurations for SiNaF.

SiNaF and Viridian have finished design work of the laboratory scale equipment that contains the sodium melt, transfer and metering system and the reduction reactor. Adding this sodium system and reduction reactor to the gas supply equipment already available at SiNaF will allow the production of silicon in batches of approximately 1.5 kilograms during the second year of the program. This will conclusively demonstrate SiNaF’s ability to generate solar grade silicon feedstock for the PV industry.

3.2 Task 2: Casting Optimization and Control

In the task 2 effort Solarex is improving control of and optimizing the casting process to increase the process yield (kilograms of silicon out divided by kilograms of silicon in) by 3% and improve the material quality, such that average cell efficiency increases by 1%. This is being achieved by improved definition of the salient casting parameters and implementation of new automated casting control systems.

Solarex casting stations have operated with microprocessor controls for a number of years. These units were state of the art when first implemented, but are now obsolete. They are impossible to replace, hard to repair and do not provide sufficient memory for the additional monitoring and control we would like to add to the system. Therefore, the first step in the casting optimization was to replace these controllers with new PC based systems. A new generation, PC based controller was configured and programmed to run exactly the same process as the old controllers. This was installed on a new group of four stations (a quad). This quad was made operational using the new PV based controller.

To improve process control computer controlled vacuum valves were added to every casting station and mass flow controllers were added to two quads of casting station. The PC based controllers were then modified to automatically switch the vacuum and gas flows valves.

With the success of the PC based controller on one quad, we decided to install these controllers on all of the casting stations. A PC based controller each quad will be accessible from four touch-screen operator interfaces. Communications between the controller, touch-screens and the Solarex network will be through an Ethernet LAN. All of the hardware has been procured and approximately 50% of the stations are now running on the PC controllers.

One of the PC based controllers was then selected for development of additional instrumentation. A number of temperature, power and fluid flow sensors are being installed on the stations in this quad to collect data to determine what additional parameters should be measured and/or controlled during the process.

The next step in the process was to provide the operations data to the Solarex manufacturing data base. This means uploading the individual run data into the Solarex network. This has now been
accomplished so Solarex engineers can now access the data through the local network and even via the internet from remote sites.

The old control system could display data on the present run, but could not provide any comparison to previous runs. So super-trend charts were installed in place of the real-time trend charts on one of the PC controlled production quads. This allows the operator to scroll back to previous casting runs on the same trend chart on which current data is displayed. This is a valuable tool for the operators to determine if a particular run is having a problem or is proceeding correctly.

The next step will be implementation of alarm acknowledgement, alarm logging, alarm specific pop-up messages, and station diagnostics on the PC controllers. These will help the operator control the process and improve the yield of quality material.

3.3 Task 3: Wire Saw Cost

In the task 3 effort Solarex is working to reduce the center-to-center cut distance on the wire saws in production to less than 450 $\mu$m, and is evaluating glycol-based slurry and several slurry and silicon carbide recycle approaches.

Thinner wire

Thinner wire has several advantages. First, it allows for the reduction in the spacing of the grooves on the wire guides to yield more wafers per centimeter. Second, it reduces the swarf that enters and ultimately degrades the slurry. Our initial plan was to switch to the thinner wire on all saws and then slowly implement a pitch change as the wire guides required replacement. However, Operations decided to transition to the thinner wire without changing the center to center spacing to minimize the wafer thickness variation on the production line. Once the transition to thinner wire is complete a set of wafer thickness experiments are to be conducted to determine what effect thickness has on line yield.

One of the wire saws was set-up to use thinner wire in conjunction with grooved wire guides with a center to center pitch of 450 $\mu$m. Initial testing was done with machine parameters nearly identical to those used on the other production machines with the exception of the table feed, which was set 100 $\mu$m per minute less than normal. This was done to establish a baseline and to minimize the amount of stress that the wire receives. During the initial testing period, there were no problems related to wire breakage.

The cost saving that was achieved by the better utilization of material, that is increasing the wafer yield from 21 to 22 wafers per centimeter, was basically offset by the added cost of the wire. More wire was used and it cost more per kilometer. To reduce the operating costs, a speed-up of the table feed was implemented. Every week, for five weeks, the table speed was increased by 10$\mu$m per minute. During this time there were no wire breakage problems or degrading yields reported. This test allowed Solarex to order the proper lengths of thinner wire, thereby reducing the cost.

As part of the program to switch to thinner wafers a set of experiments were planned. The first two lots were cut on the wire saws with control of the pitch at 450um and 500um respectively, resulting in two different wafer thicknesses being generated. Figure 3 shows histograms of the
wafer thicknesses for these two lots. The average thickness varies from 253 \( \mu \text{m} \) in lot 1 to 309 \( \mu \text{m} \) in lot 2.

**Figure 3**
Wafer Thickness Experiment – Thickness Distributions

The distributions of the two lots are shown in Figure 4. There is very little overlap between the two thickness distributions.

These two groups of wafers were then processed through the cell line. The overall yields were virtually identical, with the thinner wafers actually experiencing 0.2% less breakage.

An experiment was conducted to see if we could reuse the wire. Once the initial run was complete, an additional cut was made using the same wire, but run in reverse. The feed-spool was replaced with a steel-core, thick aluminum flanged spool. The cut went well but the spool deformed due to the force of the wire on it. We plan to get a shaft and take-up spool identical to the current Winder from HCT. Reuse of the wire could save us approximately $0.0375 per watt.
SiC Reclamation

Solarex leased a reclamation system from GTI. It is a decanter type centrifuge system that extracts the larger particles (SiC) out of used slurry. The solids that are discharged are in a thick sludge or cake form. Fresh oil is added to this cake and is mixed to the desired specific gravity before being used again in the wire saw process.

Initial testing was done to see if any noticeable changes would occur in the process due to any breakdown of the silicon carbide. Four used batches of slurry were utilized in the start-up of the system. These batches had already been used for 16 total wire saw runs. They were then sent through the system, remixed and used in one of the wire saws. This cycle was repeated numerous times until there was not enough feed or used slurry to make a new batch. A total of 18 additional runs were made off of the original slurry batch. There was no measurable change in the machine output, power consumption or slurry temperature. Wafer yields were also normal and fairly constant.

A production recycle unit has been ordered and should be in operation in mid-1999.

Glycol Based Slurries

Several cuts were made with HCT’s HS-24 glycol based slurry during the start-up of a new machine. Several runs of 11.4 cm by 11.4 cm wafers turned out exceptionally well. Several runs of 15.2 cm x 15.2 cm bricks were also cut with this material. The machine had several problems that required debugging so it was decided to halt testing until the problems were corrected.

One observation made during the testing was that when a run was removed from the machine, it had to go to the cleaning process ASAP. Otherwise, if the wafers were allowed to soak in water,
or sit in the open environment, pre-cleaning and cassetting was very difficult, resulting in poor yields.

An alternate to use of glycol may be to recycle the oil. This will be evaluated during the second year of the program.

3.4 Task 4: Cell Process Development

In the task 4 effort Solarex is developing, demonstrating and implementing a cost-effective, robust cell process that produces a minimum average cell efficiency of 15% and improves the cell line electrical yield. In the first year, process areas being evaluated and optimized include the aluminum paste BSF, fine-line screen-printing, the selective emitter process, and PECVD silicon nitride antireflective coating (with the assistance of MV Systems and North Carolina State University).

Aluminum Paste BSF

Incorporating a BSF is an important method of increasing cell efficiency, but the process now being utilized has two problems:

- The material cost for the aluminum paste is high.
- The aluminum paste causes wafers to bow. The thinner the wafer the more severe the bowing.

It has been reported in the literature by ECN7, that adding boron dopant to the aluminum paste reduces the amount of paste required to achieve the efficiency gain. Our aluminum paste supplier, Ferro, modified our standard BSF Al paste by adding boron dopant via an inorganic precursor. Using this paste we have demonstrated a 25% reduction in the amount of paste required, while matching the electrical performance of the straight Al paste. We also demonstrated almost equivalent electrical performance using 51% boron doped Al paste. At this thickness the boron bearing paste did not result in beading or blistering. However, in all cases when using this family of boron-aluminum pastes, the wafers bowed more than they normally would with just Al paste, so there was nothing to be gained by using this paste.

Ferro felt that it might be the inorganic form of the boron additive rather than the boron itself that caused the bowing. Therefore, Ferro developed an organometallic boron paste for our evaluation. The organometallic boron paste resulted in cell electrical results that were identical to the standard aluminum paste. Detailed measurements indicated that for cells with boron additive the center displacement was twice as large as for the standard aluminum paste group.

While a number for other experiments were conducted, they all had approximately the same results, namely matched electrical performance, ability to reduce the amount of paste used by approximately 25%, and increased bowing. One of our main goals was to reduce bowing so we could use thinner wafers. Since boron actually makes bowing worse, we have abandoned this work as originally proposed. Since the bowing occurs during the second firing step, we plan to investigate cofiring of BSF and front metallization in a new rapid thermal firing furnace for use with thin cells.
Selective Emitter

One approach to increased cell efficiency is the use of a selective emitter with a deeper junction under the metallization and a shallower junction in the emitter field. In this way the current collection in the emitter can be decoupled from the requirement of the screen printed metallization to have a high surface concentration of phosphorus. This approach has been evaluated at Solarex in the past using masking and etch back techniques to produce the shallower junction in the emitter field. Efficiency gains in excess of 4% were demonstrated. However, cost analysis indicated that this process is not cost effective. Another approach to achieve a selective emitter is to use a screen printable dopant paste for the deeper diffusion under the metallization area. This system may be cost effective, if rapid thermal processing can be used for the light emitter diffusion. For the emitter diffusion, a process has been developed that matches the cell efficiency while producing 2.5 times the throughput of diffused cells in the same equipment footprint as the baseline process now in use at Solarex.

The selective emitter process requires a diffusion under the grid lines that is more highly doped than a normal field diffusion and a diffusion in the emitter field that is lighter than the normal field diffusion. Efforts during the first year of the program included developing processes for both diffusions in an RTC furnace with rapid throughput. Table 3 shows the results from a trial where all of the grid diffusions were performed using the same procedure. Field diffusions processes were established using either faster belt speed or lower temperature to result in specific sheet resistance values.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Emitter (Ω/sq)</th>
<th>Efficiency (%)</th>
<th>Isc (A)</th>
<th>Voc (mV)</th>
<th>FF (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control 50</td>
<td></td>
<td>12.83</td>
<td>3.877</td>
<td>577.9</td>
<td>74.4</td>
</tr>
<tr>
<td>SE 45 Via Belt Speed</td>
<td></td>
<td>12.87</td>
<td>3.875</td>
<td>579.2</td>
<td>74.5</td>
</tr>
<tr>
<td>SE 55 Via Belt Speed</td>
<td></td>
<td>12.88</td>
<td>3.902</td>
<td>578.3</td>
<td>74.2</td>
</tr>
<tr>
<td>SE 75 Via Belt Speed</td>
<td></td>
<td>12.57</td>
<td>3.879</td>
<td>574.8</td>
<td>73.3</td>
</tr>
<tr>
<td>SE 95 Via Belt Speed</td>
<td></td>
<td>12.14</td>
<td>3.864</td>
<td>572.4</td>
<td>71.3</td>
</tr>
<tr>
<td>SE 55 Via Lower Temp</td>
<td></td>
<td>12.98</td>
<td>3.923</td>
<td>579.2</td>
<td>74.2</td>
</tr>
<tr>
<td>SE 75 Via Lower Temp</td>
<td></td>
<td>12.94</td>
<td>3.943</td>
<td>578.2</td>
<td>73.8</td>
</tr>
<tr>
<td>SE 95 Via Lower Temp</td>
<td></td>
<td>12.75</td>
<td>3.937</td>
<td>577.4</td>
<td>72.9</td>
</tr>
</tbody>
</table>

Several important conclusions from this experiment are:

- It is very important how you achieve a higher sheet resistance. Reduced temperature is much more effective than increased belt speed. We still have to optimize this process in conjunction with the grid diffusion and the front metallization firing.
• While higher sheet resistance does significantly increase the current collection, the greater series resistance loss in the emitter leads to a resulting reduction in fill factor. The decreases in fill factor seen in this experiment (for example 74.2% at 55 Ω/square down to 72.9% at 95 Ω/square) is slightly larger but of a similar order of magnitude as our modeling predicts.
• We are not seeing the expected current gains at the highest sheet resistant. At these doping levels surface recombination is probably dominating, requiring a passivating oxide to take advantage of shallow emitters.
• Our best selective emitter cells are 1.2% better in efficiency than the controls.

Efforts are now underway to develop a low cost, but effective oxide passivation step as well as continued optimization of the interaction between the two diffusion processes.

PECVD Silicon Nitride

One approach to increased cell efficiency is the use of hydrogen passivation on both bulk and surface defects in order to increase the minority carrier diffusion length. Kyocera reported on the passivation effects of silicon nitride films deposited by plasma enhanced chemical vapor deposition (PECVD) of SiH₄ and NH₃.⁹ Kyocera has been using this process in commercial production, producing 14.5% efficient polycrystalline solar cells.

PECVD deposition of silicon nitride appears ideally suited for use on solar cells since it:
1. Serves as the antireflective coating, since its index of refraction is appropriate for use between the EVA encapsulant and the silicon surface.
2. Provides a source of hydrogen for passivation of the underlying bulk silicon.
3. Provides a source of hydrogen to passivate the silicon emitter-AR coating interface.

Work at the Interuniversity Micro-Electronics Center (IMEC) in Belgium again verified that PECVD silicon nitride could passivate bulk defects in polycrystalline silicon and therefore increase cell efficiency.¹⁰ Since this work was of such interest to Solarex, we collaborated with IMEC on a set of experiments to evaluate the performance of their PECVD process on Solarex cast polycrystalline silicon. In the experiment, we processed 10 cm by 10 cm cells at Solarex and at IMEC. The results are given in Table 4.

<table>
<thead>
<tr>
<th>IMEC PECVD Silicon Nitride Experiment</th>
<th>Processing</th>
<th>Average Cell Efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solarex Baseline</td>
<td>12.7%</td>
<td></td>
</tr>
<tr>
<td>Solarex BSF</td>
<td>14%</td>
<td></td>
</tr>
<tr>
<td>IMEC TiO₂ with BSF</td>
<td>14%</td>
<td></td>
</tr>
<tr>
<td>IMEC Silicon Nitride with BSF</td>
<td>16.3%</td>
<td></td>
</tr>
</tbody>
</table>

The PECVD process has dramatically improved cell efficiency by more than 15%. So PECVD silicon nitride can passivate Solarex cast polycrystalline, if we can develop the correct process.

A set of modules was fabricated using cells produced by IMEC on Solarex polycrystalline silicon. These modules were successfully subjected to the Module Qualification Test Sequence as defined in IEC 1215.

Sister wafers were sent to IMEC and MV Systems for deposition of SiNₓ. MV Systems deposited SiNₓ at three different temperatures - 400 C, 500 C and 600 C on wafers that had already been
diffused at Solarex. These wafers were then sent to IMEC for front Metallization. The following observations were made:

1. The MV Systems SiN layers were non-uniform over a fairly large area near the edges of the cell. SiN layers deposited at higher temperatures resulted in more uniform thickness of ARC covering.

2. Nitrides deposited at 400 C and 500 C could not withstand the IMEC contact firing process and peeled off.

3. The substrates with SiN deposited at 600 C were put through front metallization at IMEC and resulted in reasonably high cell efficiencies and enhanced fill factor with efficiencies of around 14.5% as shown in Table 5.

The MV Systems SiN<sub>x</sub> appears to make a very good surface passivated coating, as indicated by the high voltage and fill factor, but does not appear to do any bulk passivation, as indicated by the low short circuit current density. A review of the results and literature indicated that MV Systems was working in a feed-gas regime that should result in Si-NH bonded material rather than material with Si-H bonds. The MV Systems process has been optimized for thin film transistors (TFT’s) where a dense stoichiometric silicon nitride film is required. This explains why the MV Systems process resulted in a good dense AR coating film. Since there was no hydrogen released by the front firing step, it did not improve the bulk lifetime. MV Systems then adjusted the ammonia to silane ratio to a regime that increases the fraction of hydrogen in Si-H bonds so that hydrogen will be released from the silicon nitride and diffuse into the bulk silicon during the front firing step. MV Systems wafers processed at IMEC have yielded 15% efficient cells, but the MV Systems cells continue to have lower short circuit current than the cells processed with the IMEC silicon nitride.

### Table 5
MV Systems SiN<sub>x</sub> Cells

<table>
<thead>
<tr>
<th>Sample</th>
<th>Efficiency (%)</th>
<th>Jsc (mA/cm²)</th>
<th>Voc (mV)</th>
<th>FF (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Backspray – TiO&lt;sub&gt;2&lt;/sub&gt;</td>
<td>12.77</td>
<td>29.84</td>
<td>578</td>
<td>74.0</td>
</tr>
<tr>
<td>BSF - TiO&lt;sub&gt;2&lt;/sub&gt;</td>
<td>13.62</td>
<td>31.89</td>
<td>590</td>
<td>72.3</td>
</tr>
<tr>
<td>BSF - IMEC SiN&lt;sub&gt;x&lt;/sub&gt;</td>
<td>14.80</td>
<td>32.36</td>
<td>604</td>
<td>75.7</td>
</tr>
<tr>
<td>BSF – MV Sys SiN&lt;sub&gt;x&lt;/sub&gt;</td>
<td>14.46</td>
<td>30.84</td>
<td>600</td>
<td>78.2</td>
</tr>
</tbody>
</table>

MV Systems processed polished single crystal wafers at varying ammonia to silane ratios (from 2 to 17). These wafers were sent to North Carolina State University for evaluation of film properties. The films produced with lower ratio of ammonia to silane had higher index of refraction, higher concentrations of Si-H bonds and no measurable N-H bonds. Films produced with higher ammonia to silane ratios had lower index of refraction, lower concentrations of Si-H bonds and more N-H bonds confirming our hypothesis.

Solarex also identified a Japanese vendor that manufactures PECVD silicon nitride equipment. A number of Solarex diffused wafers were processed through the Japanese equipment. Some of
these cells were then sent to IMEC to complete the cells using the fire through process. The results were equivalent to the results achieved using IMEC silicon nitride. Some of these cells were also processed through front fire at Solarex. Virtually all of these cells had very good short circuit current (~10% higher than if processed with a TiO2 AR coating) and good open circuit voltages in excess of 600 mV. The most sensitive parameter was fill factor. While the best cells demonstrated efficiency gains in excess of 10% over the standard TiO2 process, the scatter in series resistance meant that many of the cells were not particularly good. Efforts are underway to understand and improve the fire through process, as the last step before implementation of a PECVD silicon nitride process.

3.5 Task 5: Faster Cure Encapsulant

In the task 5 effort Solarex and Specialized Technology Resources, Inc., formerly Springborn Testing and Research (STR), are developing and qualifying an encapsulation system that meets the technical and reliability requirements and can be laminated and cured in less than 6 minutes in the present Solarex laminators.

Problem Definition / Literature Review

STR’s efforts were initiated with an extensive literature search. That search cited nearly 2,000 references that were reviewed via the internet, other computerized database searches, patent searches, and review of the proceedings from the various national and international PV conferences, meetings and workshops. A pertinent Japanese patent JP 93041577, entitled “High transparency laminates production by laminating sheets with thermosetting resin composition containing EVA copolymer and organic peroxide, heat-curing and UV irradiating” was uncovered as being awarded to Bridgestone Tire KK.

Other noteworthy information gathered included:

- BMC Solar in Germany is using Dyneron THV200 in their premium roof modules. Dyneron THV200 is a fluoroterpolymer that is substantially more expensive than the EVA-based encapsulants. When its properties were compared to PhotoCap™ 15295, it was found to offer no significant advantages over EVA-based encapsulants other than its inherent flammability resistance.

- Surlyn™ resins (ionomers) were identified as of interest as a PV encapsulant. A review of DuPont's literature discloses that free and bound methacrylic acid is contained within the ionomer resins. The presence of the acid requires melt processing using stainless steel tooling. A further concern is the ionomer’s high rate of moisture absorption, which necessitates long drying cycles prior to processing. Combined with pricing at approximately two times that of EVA eliminated the ionomers from serious consideration.

- Encapsulant systems based on ethylene butyl and methyl acrylates were also identified as possible candidates for PV. However, liquid encapsulants were eliminated as being too cost prohibitive, difficult to handle and required long curing times.

From the literature review and dialog with industry experts, it was determined that the rate of encapsulant cure might be accelerated by multiple means. Higher lamination temperatures would dramatically reduce the curative’s half-life, thereby reducing the time required to achieve an acceptable level of crosslinking. The employment of acetic acid scavengers and/or peroxide co-agents could lead to faster curing of EVA-based encapsulants at current or higher processing
temperatures. Formulation of the encapsulant with a more active peroxide, that has a shorter half-
life versus the present 00-t-Butyl 0-(2-Ethylhexyl) Monoperoxycarbonate (TBEC) curative, could
speed the process. It is also postulated that the encapsulant’s curative system could be formulated
to be responsive to UV through incorporation of UV Photo-activator, that on pre-treatment with
UV, the curative initiation temperature may be reduced. Aztec Peroxides Inc., claims that
reaction between UV/photoactivator and the TBEC has been reported.

One approach to reducing the encapsulant system’s overall cost is to replace the encapsulant layer
behind the solar cells with a less expensive material that does not have the same critical optical
clarity requirement as the top encapsulant layer. Some of the concerns in doing so are lack of
adhesion between the encapsulant layers and to the backsheet, and mismatches of thermal
expansion and rigidity that could cause serious deformation.

Preliminary Materials Qualification

As a result of the literature search, a number of polymeric materials were selected for
consideration as candidate base resins for the faster curing encapsulant system. For screening
purposes, they were evaluated versus STR 15295 EVA-based encapsulant system. Those
considered include: a number of EVA's ranging from 10-32% vinyl acetate, supplied by 4
different suppliers; a series of ethylene butylacrylate copolymers, ranging from 7-35% butyl
acrylate; ethylene methyl acrylate copolymers, ranging from 10-30% methyl acrylate; and
ethylene octene copolymers ranging from 20-28% octene.

Compression molded sheets were prepared for each material. Each 18 mil thick sample was then
evaluated for its average percent light transmission characteristics over the wavelengths of 360 –
900 nanometers. Note that samples made by compression molding of the Elvax 3185,
PhotoCap™ 15295P and A9918P had measured light transmission values that were ~3% lower
than measured on extruded specimens. (Probably due to surface phenomena induced in the
compression molding process). Therefore, the expected light transmission values were
extrapolated for the extruded specimens.

In conclusion, DuPont’s 32% vinyl acetate containing EVA was found to offer the most
advantage in optical properties (percent light transmission over the wavelengths of 360 – 900 nm)
and is generally less expensive than other alternatives identified. The evaluation results are
presented in Table 6.

Based on these measured results, it appears that only the highest VA content EVA's have the
desired level of optical transparency for use as a front surface encapsulant. Of the other ethylene
copolymers evaluated, only those of the highest copolymer contents were determined to have
average light transmission ranging from 89-91%. All of which are more costly than the EVA
copolymers.

As part of the qualification of alternative lower cost encapsulants for deployment behind the solar
cells, a preliminary study was undertaken. It was noted that polyethylenes of all types gave poor
adhesion to 32% VA type EVA. In some cases there is a mismatch of linear coefficient of
thermal expansion, that resulted in wrinkling of the laminate. Based on the preliminary
investigations, the EVA copolymers having approximately 10% vinyl acetate content appear most
viable for use as a back encapsulation in terms of cost, coefficient of thermal expansion, and
toughness.
### Table 6.
Candidate Encapsulant Resin Percent Light Transmission Values

<table>
<thead>
<tr>
<th>Commercial Name</th>
<th>Generic Description</th>
<th>% T (Measured)</th>
<th>Molded Specimens</th>
<th>Extruded Specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>PhotoCap™ 15295P</td>
<td>Fast Cure EVA</td>
<td>90%</td>
<td></td>
<td>93.5% (1)</td>
</tr>
<tr>
<td>PhotoCap™ A9918P</td>
<td>Standard. Cure EVA</td>
<td>91%</td>
<td></td>
<td>94% (1)</td>
</tr>
<tr>
<td>Elvax 3185</td>
<td>EVA - 32% VA</td>
<td>90%</td>
<td></td>
<td>93% (1)</td>
</tr>
<tr>
<td>Elvax 265</td>
<td>EVA - 28% VA</td>
<td>88%</td>
<td></td>
<td>91% (1)</td>
</tr>
<tr>
<td>Escorene LD-781.36</td>
<td>EVA - 33% VA</td>
<td>90%</td>
<td></td>
<td>93% (2)</td>
</tr>
<tr>
<td>Escorene LD-755.36</td>
<td>EVA - 29% VA</td>
<td>90%</td>
<td></td>
<td>93% (2)</td>
</tr>
<tr>
<td>Escorene LD-723.62</td>
<td>EVA - 19% VA</td>
<td>82%</td>
<td></td>
<td>85% (2)</td>
</tr>
<tr>
<td>Escorene LD-706.62</td>
<td>EVA - 15% VA</td>
<td>80%</td>
<td></td>
<td>83% (2)</td>
</tr>
<tr>
<td>Escorene LD-409.09</td>
<td>EVA - 10% VA</td>
<td>76%</td>
<td></td>
<td>79% (2)</td>
</tr>
<tr>
<td>Ateva 2825</td>
<td>EVA - 28% VA</td>
<td>86%</td>
<td></td>
<td>89% (2)</td>
</tr>
<tr>
<td>Ateva 1841</td>
<td>EVA - 18% VA</td>
<td>80%</td>
<td></td>
<td>83% (2)</td>
</tr>
<tr>
<td>Ateva 1645</td>
<td>EVA - 16% VA</td>
<td>79%</td>
<td></td>
<td>82% (2)</td>
</tr>
<tr>
<td>Evatane 33-45</td>
<td>EVA - 33% VA</td>
<td>84%</td>
<td></td>
<td>87% (2)</td>
</tr>
<tr>
<td>Lotryl 35BA40</td>
<td>EBA - 35% BA</td>
<td>87%</td>
<td></td>
<td>90% (2)</td>
</tr>
<tr>
<td>Lotryl 30BA02</td>
<td>EBA - 30% BA</td>
<td>86%</td>
<td></td>
<td>89% (2)</td>
</tr>
<tr>
<td>Lotryl 28BA175</td>
<td>EBA - 28% BA</td>
<td>80%</td>
<td></td>
<td>83% (2)</td>
</tr>
<tr>
<td>Lotryl 17BA04</td>
<td>EBA - 17% BA</td>
<td>74%</td>
<td></td>
<td>77% (2)</td>
</tr>
<tr>
<td>Lotryl 7BA01</td>
<td>EBA - 7% BA</td>
<td>70%</td>
<td></td>
<td>73% (2)</td>
</tr>
<tr>
<td>Lotryl 35MA05</td>
<td>EMA - 35% MA</td>
<td>87%</td>
<td></td>
<td>90% (2)</td>
</tr>
<tr>
<td>Lotryl 24MA07</td>
<td>EMA - 24% MA</td>
<td>84%</td>
<td></td>
<td>87% (2)</td>
</tr>
<tr>
<td>Engage 8180</td>
<td>Ethylene Octene 28%</td>
<td>84%</td>
<td></td>
<td>87% (2)</td>
</tr>
<tr>
<td>Engage 8400</td>
<td>Ethylene Octene 24%</td>
<td>80%</td>
<td></td>
<td>83% (2)</td>
</tr>
<tr>
<td>Engage 8411</td>
<td>Ethylene Octene 20%</td>
<td>78%</td>
<td></td>
<td>81% (2)</td>
</tr>
<tr>
<td>Vamac E90448-28A</td>
<td>EMA - 31% MA</td>
<td>84%</td>
<td></td>
<td>87% (2)</td>
</tr>
<tr>
<td>Chevron 6335</td>
<td>Ethylene Butene</td>
<td>72%</td>
<td></td>
<td>75% (2)</td>
</tr>
<tr>
<td>Elite 5200</td>
<td>Polyethylene</td>
<td>68%</td>
<td></td>
<td>71% (2)</td>
</tr>
<tr>
<td>Equistar NA345-013</td>
<td>Polyethylene</td>
<td>64%</td>
<td></td>
<td>67% (2)</td>
</tr>
<tr>
<td>Equistar NA214-000</td>
<td>Polyethylene</td>
<td>65%</td>
<td></td>
<td>68% (2)</td>
</tr>
</tbody>
</table>

(1) – As measured  
(2) – Extrapolated

### A Qualitative Cure Test

To reduce the time necessary to qualify the achieved cure level, a simple qualitative test was conceived and its results verified. This test method consists of suspending cured EVA strips with a weight (approximately 3 times the encapsulant weight), in an oven at 95°C with a standard distance marked between two points (A & B). The weighted strips are oven exposed for a minimum of 10 minutes. Cured EVA strips maintained their original measured distance between points A & B. Those un cured, demonstrate elongation caused by melting or softening of the EVA. Correlation between the qualitative oven test and the quantitative gel test indicates that a minimum cure level of 70% has been achieved when the sample passes the 95°C/10-min. oven test.
Feasibility Study To Achieve a Faster Curing Encapsulant

A series of additives were evaluated to determine their effects on acceleration of cure of the EVA-based encapsulants. Unfortunately, only modest gains in curing rates resulted. The single parameter that has the greatest effect on the EVA-based encapsulant’s cure rate is temperature. TBEC peroxide is reported as having a half-life of 3.8 minutes at 150°C. At 171°C the half-life is less than 30 seconds (about an 8-fold increase in rate). However, prior industry experience suggests that higher lamination temperatures are unfeasible as bubble formation occurs in the EVA during lamination at temperatures approaching 155°C. At 171°C, the situation worsens.

Moisture’s Role In 1986 JPL published the IR analysis of the thermal effluents from EVA encapsulants\textsuperscript{11}. The analysis concluded that water evolves at 105°C, carbon dioxide at 120°C, acetone and t-butanol at 177°C and acetic acid at 205°C. It was further determined that at least 1.5 wt. % water can be present in a 33% vinyl acetate containing EVA following short exposure to air.

Based on that information, a series of experiments were conducted to determine what affect drying has on bubble formation during lamination. The experiments were conducted on EVA as normally packaged, partially dried and completely dried. The completely dried specimens were exposed to vacuum for 18 hours at 50°C. The partially dried 18 mil samples were vacuum dried for 1-2 hours at 50°C. Some additional samples received partial drying through storage in a desiccator at room temperature over night. The non-dried specimens had at least one week's exposure to ambient air.

Lamination was accomplished using a compression molding press in the presence of atmospheric air. The following results were noted:

- No bubbles formed during lamination of the dried EVA encapsulants even though it was processed without vacuum.
- All of the partially dried EVA samples produced bubbles.
- All of the non-dried EVA samples produced bubbles as expected.
- Additional samples of the previously dried encapsulants were exposed to ambient air for a week then laminated without vacuum. Bubbles resulted, suggesting that formulation ingredients may not be the sole cause of bubble formation. Moisture may also play a role.

Dimethyl phthalate (DMP) was evaluated in conjunction with EVA to determine if it would inhibit bubble formation. By simply coating DMP onto the 15295P EVA formulation, it appeared to reduce bubble formation. (The DMP functions by forming an azeotrope, 98% water / 2% DMP).

Based on speculation that moisture may contribute to bubble formation during lamination, we investigated the amount of time EVA remains sufficiently dry to avoid bubble generation during lamination. Bubbles were noted following 66 hours exposure to ambient air, while none were noted after 48 hours. Based on these results, a series of experiments were conducted using a laboratory vacuum laminator. The purpose was to verify that drying the EVA-based 15295 encapsulant prior to lamination would reduce bubble formation at high lamination temperatures. Unfortunately, the findings in the vacuum laminator did not agree with the findings from the hot press experiments. In the vacuum laminator experiments bubble formation occurred as the EVA-based encapsulant temperature exceeded 155°C, independent of any pre-drying of the EVA.
Determination of Volatile Content in EVA Resin and Encapsulant: A series of EVA and EVA-based encapsulant samples were submitted to DuPont to ascertain the moisture content of the 32% EVA resin as received by STR prior to processing, versus the moisture content of STR’s fully formulated 15295P encapsulant. DuPont performed Karl Fischer titrations in accordance with ASTM D-789.

In addition to the DuPont moisture content determinations, STR determined the 32% EVA resin’s total volatile content under isothermal conditions at 155°C for 15 minutes under nitrogen, by Thermal Gravimetric Analysis. The moisture content and percent volatiles data are presented in Table 7.

Table 7.
Percent Moisture and Volatile Content of EVA Resin and EVA-Based Encapsulant

<table>
<thead>
<tr>
<th>Item</th>
<th>Sample Description</th>
<th>Karl Fischer Titration Water, PPM (wt.%)</th>
<th>Thermal Gravimetric Analysis Volatiles, PPM (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>32% EVA resin, from unopened container</td>
<td>288 (0.0288)</td>
<td>340 (0.03399)</td>
</tr>
<tr>
<td>2</td>
<td>32% EVA resin, 3 days in open air</td>
<td>286 (0.0286)</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>32% EVA resin, dried 24 hrs. under vacuum</td>
<td>-</td>
<td>351 (0.03510)</td>
</tr>
<tr>
<td>4</td>
<td>32% EVA resin, dried 3 days under vacuum</td>
<td>-</td>
<td>194 (0.01939)</td>
</tr>
<tr>
<td>5</td>
<td>15295P encapsulant, freshly manufactured</td>
<td>65 (0.0065)</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>15295P encapsulant, 3 days in open air</td>
<td>78 (0.0078)</td>
<td>-</td>
</tr>
</tbody>
</table>

Contrary to our hypothesis, there is minimal moisture contained within the EVA-based 15295P encapsulant. This suggests that the volatiles evolving as bubbles in the encapsulant near 155°C are probably linked to the formulation’s constituents not its moisture content.

Based on the moisture content determinations and TGA, the maximum water content of the EVA encapsulant is less than 0.06%. It is likely that nearly all of the volatiles emitted from the EVA during vacuum lamination evolve from the curative. As the half-life of TBEC peroxide at 163°C is less than one minute, approximately 80% of the peroxide should be decomposed in 2.5 minutes at 163°C. Based on experiments conducted using a laboratory vacuum laminator, it was determined that a minimum vacuum evacuation time of 2 minutes, 15 seconds was necessary to remove all volatiles.

Similar experiments were carried out using a candidate alternative “hotter” curative that has a shorter half-life than TBEC peroxide. Similar results were obtained with this curative at 154°C, versus the 163°C TBEC experiments.

Formulation Modifications: Ensuing laboratory efforts identified an experimental formulation that required a lamination cycle of seven minutes to pass the qualitative cure test. The encapsulant configuration was composed of 2 different EVA formulations. The superstrate EVA portion consisted of a modified version of the PhotoCap™ 15295 formulation, to contain one additional ingredient. The substrate EVA formulation was also based on the 15295 formulation, though a 10% EVA copolymer was substituted for the 32% EVA. Both EVA based encapsulants were dried prior to lamination.

The lamination cycle consisted of air evacuation for 3 minutes at 145°C-150°C, followed by a 2 minute press at 150°C-155°C under vacuum, followed by an additional 2 minutes at 155°C-158°C at atmospheric pressure. Temperature fluctuations were observed and believed to be caused by the exothermic reaction of the curative system (increasing dramatically with
temperature) and the evolution of volatiles that actually cools the EVA in the laminator. This endothermic effect accelerates with increased platen temperature.

Increased vacuum dramatically increases the rate of volatilization. However, the source of volatiles apparently becomes depleted in time, as the rate of volatilization decreases with time. There appears to be intense volatilization under vacuum between 145°C and 155°C. To avoid bubble entrapment, it may be necessary to eliminate the vacuum at temperatures at or above 155°C.

Processing Variables Using TBEC Peroxide Vs. An Alternative Curative

STR's fast-curing 15295 and 15295P EVA-based encapsulants employ a TBEC peroxide cure system. Lamination tests at atmospheric pressure indicated that cure can be accomplished in 3.5 minutes total at 170°C with substantial bubble formation. Based on half-life determinations and experimentation, this corresponds to a 6-minute cure at approximately 160°C. Similarly, based on kinetic predictions, an alternative cure system was evaluated, with expectations of behaving similarly using a 6 minute lamination cycle at 152°C. A number of variables were examined for their effect on lamination using both cure systems in the EVA-based encapsulant.

- For both cure systems, the initial vacuum should be applied as quickly as possible.
- One open question is how fast to transition from vacuum to atmospheric pressure in the lower chamber. Evacuation of the laboratory test samples was accomplished in 2-2.5 minutes for platen temperatures ranging from 149-163°C, at which time the top chamber was pressurized to one atmosphere over a 5-second interval. Five to 10 seconds later the lower chamber was also pressurized. Adequate flow of the encapsulants was realized.
- Experiments were conducted employing molecular sieves as a scavenger for volatiles. The sieves were formulated into the back layer of EVA-based encapsulant. These experiments proved unsuccessful, as the sieves were difficult to disperse, and actually nucleate bubble formation.
- Additional experiments were conducted to determine the impact of using vacuum dried EVA versus EVA exposed to air for over two days. The dried encapsulants appear to cure faster.
- Experiments on surface texture were also conducted. The results suggest that textured encapsulant surfaces aid in the evacuation of air from the laminate.
- Experiments with multiple temperature ramping and processing indicated no advantage over use of a single platen temperature.

Based on the experimental results, it was determined that the best lamination process use a single platen temperature that is dependent upon the cure system employed, (160°C for the TBEC peroxide curative and 152°C for the alternative curative). This lamination cycle employs a 5-10 second transition from vacuum to air in the top laminator chamber, and a 5-10 second vacuum to air transition in the bottom chamber.

Faster-Curing Lamination Trials of Experimental Formulations

A series of lamination trials were conducted using three formulations of EVA. The first being the PhotoCap™ 15295P formulation, the second, a modified version of 15295P containing an alternative cure system substituted for the TBEC peroxide, and X15303P, an enhanced UV stabilized “Fast-Cure” EVA formulation, as developed under STR’s PVMaT 3A contract. Two lamination processes were utilized:
1. Complete processing was accomplished using a laboratory vacuum laminator in a total 6-minute cycle. The laminator was maintained at constant temperature for the entire lamination sequence. Air evacuation was accomplished for the first two minutes and 15 seconds by pulling vacuum on both top and bottom chambers. Subsequently, pressure was immediately applied, requiring 5 seconds to return to atmospheric pressure in the top chamber. Immediately following pressing of the laminate, the bottom chamber was also returned to atmospheric pressure, over a 5-second interval. The laminate remained in the laminator for an additional 3 minutes and 35 seconds to impart cure. Platen temperature was maintained at 163°C for the 15295P and X15303P. The modified 15295P with alternative curative was processed at 154°C.

2. Processing was accomplished in two stages. The first stage involving a four-minute vacuum lamination cycle, followed by a 3-minute oven post cure cycle. As in (1) above, the laminator was maintained at constant temperature for the entire lamination sequence. Air evacuation was accomplished for the first two minutes and 15 seconds by pulling vacuum on both top and bottom chambers. Subsequently, pressure was immediately applied, requiring 5 seconds to return to atmospheric pressure in the top chamber. Immediately following pressing of the laminate, the bottom chamber was also returned to atmospheric pressure, over a 5-second interval. The laminate remained in the laminator for an additional 35 seconds at atmospheric pressure. Immediately the laminates were removed and placed in an air circulating oven that was maintained at the laminator’s platen temperature and held for three minutes. The platen and oven temperatures were maintained at 163°C for the 15295P and X15303P. The modified 15295P with alternative curative was processed at 154°C.

The results are presented in Table 8. From the results in Table 8, it was concluded that it is possible to use a six minute lamination cycle to process 15295P, X15303P, and the modified version of 15295P containing the alternative cure system, to produce bubble-free glass/EVA/TPE laminates. However, the 2-stage lamination cycle involving an oven post cure, requires additional time to accomplish complete cure above 80%.

Results of Laboratory Lamination Trials Using Six Minute Cycles

Further experiments were conducted using an alternative “hotter” cure system in place TBEC peroxide in EVA-based encapsulant formulations based on 15295P and 15303P. The alternative cure agent was evaluated for its advantage in curing rate.

The initial experiments indicated that a 135 second initial vacuum treatment was necessary to remove trapped air and volatiles. Subsequent trials proved that complete evacuation could be accomplished in 110 seconds. Of course, this is a function of vacuum efficiency and the surface area of the modules being evacuated. This observation may not have any correlation to actual commercial manufacturing processes or modules.

When replacing TBEC with an alternative cure agent at similar concentration, and using a lamination cycle of 6 minutes at 160°C ± 3°C, a gel content of 74.5% results. Under the same conditions the 15295 TBEC based formulation yielded a gel content of 58.6%. To achieve a gel content in the range of 80% or greater, both peroxides required lamination processing at higher temperatures, 166°C ± 3°C, for 6 minutes. In conclusion, it appears that the crosslinking efficiency of the alternative cure agent is poorer than that of the TBEC peroxide at similar concentrations. At the concentration used, 80% gel content was very difficult to achieve using the alternative cure agent. While 80% cure has been our target, to consume as much cure agent as possible, it may not be necessary to cure to 80% using the alternative...
In this instance, all of the cure agent may well be consumed although a lower gel content results. In the past, prior to discoloration issues arising, EVA curing to a level greater than 65% was deemed adequate to prevent flow of the EVA or failure in the field.

### Table 8.
Results of “Faster-Curing” Lamination Trials

<table>
<thead>
<tr>
<th>Encapsulant System</th>
<th>Sample No.</th>
<th>Total Time</th>
<th>Process</th>
<th>Laminate Composition¹</th>
<th>Bubbles Present</th>
<th>Creep @ 95°C</th>
<th>Glass Adhesion</th>
<th>Gel Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>15295P</td>
<td>5</td>
<td>6 min. 1</td>
<td>Glass/TPE Aluminum/ TPE</td>
<td>None</td>
<td>—</td>
<td>&gt;43.5 lbs./in.</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>6 min. 1</td>
<td>TPE Aluminum/ TPE</td>
<td>None</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>6 min. 1</td>
<td>TPE Aluminum/ TPE</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>83.2%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>D-1</td>
<td>7 min. 2</td>
<td>TPE Aluminum/ FEP</td>
<td>None</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>D-2</td>
<td>7 min. 2</td>
<td>FEP</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>73.8%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-1</td>
<td>6 min. 1</td>
<td>Aluminum/ TPE</td>
<td>None</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-2</td>
<td>6 min. 1</td>
<td>Glass/TPE Aluminum/ TPE</td>
<td>None</td>
<td>—</td>
<td>&gt;31.5 lbs./in.</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-3</td>
<td>7 min. 2</td>
<td>TPE Aluminum/ FEP</td>
<td>None</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-4</td>
<td>6 min. 1</td>
<td>FEP Aluminum/ TPE</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>84.6%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-5</td>
<td>7 min. 2</td>
<td>FEP</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>53.1%</td>
<td></td>
</tr>
<tr>
<td>Mod. 15295P (using Alternative cure system)</td>
<td>H-1</td>
<td>6 min. 1</td>
<td>Aluminum/ TPE</td>
<td>None</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-2</td>
<td>6 min. 1</td>
<td>Glass/TPE Aluminum/ TPE</td>
<td>None</td>
<td>—</td>
<td>&gt;31.5 lbs./in</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-3</td>
<td>7 min. 2</td>
<td>TPE Aluminum/ FEP</td>
<td>None</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-4</td>
<td>6 min. 1</td>
<td>FEP Aluminum/ TPE</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>84.6%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>H-5</td>
<td>7 min. 2</td>
<td>FEP</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>53.1%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>J-1</td>
<td>6 min. 1</td>
<td>Aluminum/ FEP</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>79.5%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B-1</td>
<td>6 min. 1</td>
<td>FEP Aluminum/ TPE</td>
<td>Some</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B-2</td>
<td>7 min. 2</td>
<td>FEP Aluminum/ TPE</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>77.7%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B-3</td>
<td>6 min. 1</td>
<td>FEP Aluminum/ TPE</td>
<td>Some</td>
<td>—</td>
<td>—</td>
<td>85.3%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B-4</td>
<td>7 min. 2</td>
<td>FEP</td>
<td>Some</td>
<td>Passed</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B-5</td>
<td>6 min. 1</td>
<td>Glass/TPE Aluminum/ TPE</td>
<td>None</td>
<td>—</td>
<td>&gt;4.10 lb/in</td>
<td>glass broke</td>
<td></td>
</tr>
</tbody>
</table>

¹ Aluminum/TPE = Teflon coated aluminum plate/TPE, used to separate EVA from superstrate for testing.

Aluminum/FEP = Teflon coated aluminum plate/FEP, used to isolate EVA for testing.
Experiments Using a 10% Vinyl Acetate EVA Copolymer-based Encapsulant Layer For Behind the Solar Cells

A series of experiments was conducted substituting 10% vinyl acetate EVA for the 32% vinyl acetate EVA. The purpose was to evaluate the utilization of a lower cost material for behind the solar cells. The 10% vinyl acetate EVA requires less cure, and can offer cost, performance and processing time advantages. The 10% EVA was formulated to contain less TBEC peroxide. It was noted that when combining this EVA with the normal 32% vinyl acetate EVA front layer, the level of EVA flashing out of the laminate decreased during lamination.

Curing of the composition for six minutes at 166°C±3°C by vacuum lamination resulted in a gel content of 68% for the 10% EVA layer. Qualitative testing suggests that this lower gel content results in performance that is similar to curing the 32% EVA to an 80% gel content. Both materials resist cold flow at 100°C. With the standard loading of TBEC, a 6 minute lamination cycle at 166°C resulted in a gel content of >80% for the 10% EVA.

Further Optimization Experiments to Define Processing Parameters for the EVA Formulations

A series of experiments were conducted using the formulation 15303P based on TBEC peroxide plus that formulation with the alternative cure agent substituted for TBEC. Experiments were performed with and without 10% vinyl acetate EVA in place of the 32% vinyl acetate EVA. Processing was carried out based on two sets of process conditions as outlined below.

Process A consisted of 1 minute 50 seconds under vacuum, then pressing for 5 seconds using 1 atmosphere, followed by rapid repressurization of the bottom chamber, and no compression for the balance of 6 minutes.

Process B consisted of 1 minute 45 seconds under vacuum, then rapidly applying atmospheric pressure to the top chamber and holding the laminate under pressure for the balance of 6 minutes.

The results from these experiments are presented in Table 9. As a key to Table 9, the standard formulation 15303P containing TBEC peroxide is designated as the “J” series. While a similar formulation, substituting the alternative cure agent for TBEC is designated as the “K” series.

Conclusions from this set of experiments are:

1. Process A yielded higher gel content than Process B.
2. Incorporation of an EVA back layer composed of 10% vinyl acetate containing EVA would resist deformation or flow at 100°C, even with the 10% vinyl acetate EVA layer cured to only a 68% gel content.
3. At standard cure agent concentration, both formulations reliably processed in 6 minutes at 166°C±3°C to adequate gel content levels.

Dynamic Stability Testing of Alternative Cure EVA Formulations

The ability to process the alternative cure formulations was assessed. Based on a series of rheological studies at various processing temperatures, it was determined that these formulations are sufficiently stable to process through STR’s standard manufacturing equipment using normal processing parameters.
**Table 9**

EVA Processing Experiments

<table>
<thead>
<tr>
<th>15303P (Alternative Cure Agent)</th>
<th>Temperature °C</th>
<th>Process</th>
<th>Gel Content (%)</th>
<th>Passes 95°C Creep Test</th>
<th>Passes 100°C Creep Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>K-10</td>
<td>166</td>
<td>A</td>
<td>84.0</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>K-30</td>
<td>162</td>
<td>A</td>
<td>84.6</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>K-32</td>
<td>166</td>
<td>A</td>
<td>83.6</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>K-28</td>
<td>162</td>
<td>A</td>
<td>79.5</td>
<td>Yes</td>
<td>Barely</td>
</tr>
<tr>
<td>K-27</td>
<td>161</td>
<td>A</td>
<td>74.4</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>K-1</td>
<td>160</td>
<td>A</td>
<td>74.5</td>
<td>Yes</td>
<td>No</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>15303P (TBEC Peroxide)</th>
<th>Temperature °C</th>
<th>Process</th>
<th>Gel Content (%)</th>
<th>Passes 95°C Creep Test</th>
<th>Passes 100°C Creep Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>J-12</td>
<td>166</td>
<td>A</td>
<td>88.2</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>J-42</td>
<td>166</td>
<td>A</td>
<td>85.9</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>J-3</td>
<td>163</td>
<td>A</td>
<td>84.5</td>
<td>Yes</td>
<td>Yes</td>
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<tr>
<td>J-1</td>
<td>163</td>
<td>A</td>
<td>79.5</td>
<td>Yes</td>
<td>Barely</td>
</tr>
<tr>
<td>J-32</td>
<td>160</td>
<td>A</td>
<td>58.6</td>
<td>Yes</td>
<td>No</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>15303P (TBEC or Alternate Cure Agent) /10% EVA Layer</th>
<th>Temperature °C</th>
<th>Process</th>
<th>Gel Content (%)</th>
<th>Passes 95°C Creep Test</th>
<th>Passes 100°C Creep Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>J-52/G</td>
<td>166</td>
<td>B</td>
<td>71.1</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>J-50/G</td>
<td>163</td>
<td>B</td>
<td>68.1</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>K-39/G</td>
<td>160</td>
<td>B</td>
<td>67.5</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>J-47/G</td>
<td>166</td>
<td>A</td>
<td>85.7</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>J-43/G</td>
<td>166</td>
<td>A</td>
<td>78.3</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>K-3/G</td>
<td>161</td>
<td>A</td>
<td>74.8</td>
<td>Yes</td>
<td>Yes</td>
</tr>
</tbody>
</table>

**Determination of the Effects of Cure Agent Concentration**

Based on a series of qualitative cure efficiency tests and subsequent gel testing, it was concluded that an 80% gel content may be necessary to prevent EVA deformation in a PV module during long term, high temperature operation. These results suggest that EVA gel contents ranging between 75-80% may flow or creep upon long term exposures at 90°C.

To evaluate this a series of experiments were conducted to determine the effect of cure agent concentration versus cure temperature at a constant lamination time of 6 minutes. Each of two cure systems were evaluated, TBEC and the candidate alternative cure agent. The EVA was fully formulated based on the X15303P “fast-curing” grade. The results are presented in Table 10.
Table 10.
Gel Contents vs. Peroxide Type, Cure Agent Concentration, and Platen Temperature

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Cure Agent</th>
<th>Conc.</th>
<th>Platen Temp.</th>
<th>Gel Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>J-32</td>
<td>TBEC</td>
<td>A</td>
<td>160°C</td>
<td>58.6%</td>
</tr>
<tr>
<td>J-3</td>
<td>TBEC</td>
<td>A</td>
<td>163°C</td>
<td>84.5%</td>
</tr>
<tr>
<td>J-1</td>
<td>TBEC</td>
<td>A</td>
<td>163°C</td>
<td>79.5%</td>
</tr>
<tr>
<td>J-42</td>
<td>TBEC</td>
<td>A</td>
<td>166°C</td>
<td>85.9%</td>
</tr>
<tr>
<td>J-12</td>
<td>TBEC</td>
<td>A</td>
<td>166°C</td>
<td>88.3%</td>
</tr>
<tr>
<td>K-53</td>
<td>Alt. Cure</td>
<td>A</td>
<td>149°C</td>
<td>64.8%</td>
</tr>
<tr>
<td>K-1</td>
<td>Alt. Cure</td>
<td>A</td>
<td>160°C</td>
<td>74.5%</td>
</tr>
<tr>
<td>K-27</td>
<td>Alt. Cure</td>
<td>A</td>
<td>161°C</td>
<td>74.5%</td>
</tr>
<tr>
<td>K-28</td>
<td>Alt. Cure</td>
<td>A</td>
<td>162°C</td>
<td>79.5%</td>
</tr>
<tr>
<td>K-10</td>
<td>Alt. Cure</td>
<td>A</td>
<td>166°C</td>
<td>84%</td>
</tr>
<tr>
<td>K-32</td>
<td>Alt. Cure</td>
<td>A</td>
<td>166°C</td>
<td>83.6%</td>
</tr>
<tr>
<td>M-1</td>
<td>TBEC</td>
<td>D</td>
<td>163°C</td>
<td>84.4%</td>
</tr>
<tr>
<td>M-2</td>
<td>TBEC</td>
<td>D</td>
<td>166°C</td>
<td>88.7%</td>
</tr>
<tr>
<td>M-3</td>
<td>TBEC</td>
<td>D</td>
<td>166°C</td>
<td>91.8%</td>
</tr>
</tbody>
</table>

An additional series of gel experiments were conducted, based on the above experimental series of J, K, and M using a 10 minute lamination cycle at 166°C. Those gel content values are presented in Table 11.

Table 11
Gel Contents vs. Cure Agent & Concentration at 10 Minutes at 166°C

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Cure Agent</th>
<th>Conc.</th>
<th>Platen Temp./Time</th>
<th>Gel Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>J-67</td>
<td>TBEC</td>
<td>A</td>
<td>166°C/10 Min.</td>
<td>91.5%</td>
</tr>
<tr>
<td>K-54</td>
<td>Alt. Cure</td>
<td>A</td>
<td>166°C/10 Min.</td>
<td>85.8%</td>
</tr>
<tr>
<td>M-4</td>
<td>TBEC</td>
<td>D</td>
<td>166°C/10 Min.</td>
<td>92.0%</td>
</tr>
</tbody>
</table>

A third series of experiments was conducted using the “fast-curing” 15295P EVA-based encapsulant. Once again a 6 minute lamination cycle was utilized. The results are presented in Table 12.

Table 12
Gel Contents of 15295P EVA vs. Lamination Temperature

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Lamination Time</th>
<th>Platen Temp.</th>
<th>Gel Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>15295P-4</td>
<td>6 Min.</td>
<td>163°C</td>
<td>79.7%</td>
</tr>
<tr>
<td>15295P-5</td>
<td>6 Min.</td>
<td>166°C</td>
<td>87.2%</td>
</tr>
<tr>
<td>15295P-12</td>
<td>6 Min.</td>
<td>166°C</td>
<td>88.0%</td>
</tr>
</tbody>
</table>

Based on the data generated in the above experiments, it appears that each of the compositions will provide adequate flow/creep resistance at 90°C when laminated using a 6 minute cycle at 163°C or higher. The data also suggests the higher concentration formulations adequately crosslink the EVA.
A series of additional lamination trials was conducted using larger laminates. The intent was to explore the affects of increasing the laminate size on the degree of cure. The results are presented in Table 13.

Table 13
Gel Content Determination Using Larger Laminates vs. TBEC Concentration

<table>
<thead>
<tr>
<th>Sample ID.</th>
<th>Laminate Size</th>
<th>TBEC Peroxide</th>
<th>% Gel Content</th>
<th>Lamination Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6” x 6” B</td>
<td>87.0%</td>
<td>6 min.</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>5” x 5” B</td>
<td>88.6%</td>
<td>6 min.</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>6” x 6” B</td>
<td>89.6%</td>
<td>6 min.</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>6” x 6” C</td>
<td>92.6%</td>
<td>6 min.</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>6” x 6” C</td>
<td>92.0%</td>
<td>6 min.</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>6” x 9” C</td>
<td>88.5%</td>
<td>6 min.</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>6” x 6” E</td>
<td>93.9%</td>
<td>6 min.</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>6” x 6” E</td>
<td>91.5%</td>
<td>7 min.</td>
<td></td>
</tr>
</tbody>
</table>

It was concluded from these experiments that these larger laminates performed in a manner similar to the smaller laminates. It was further noted that modifying the cure agent concentration does not appear to modify the level of measurable gel or degree of cure.

An further set of experiments was conducted on even larger laminates. Although tremendous degree of bubbling was encountered initially at 149-150°C, it was determined that by insuring complete removal of all of the air prior to melting of the encapsulant, the bubbles could be avoided. As a result bubble free lamination has been demonstrated at a laminator platen temperature of 166°C.

Formulating for Lower Temperature Lamination

Laboratory efforts have demonstrated that a 6 minute lamination cycle at 163°C - 166°C can yield a cured EVA module. However, with the PV communities difficulties associated with bubble propagation at process temperatures greater that 155°C - 160°C, it is desirable to maintain laminator platen temperatures below this range. Today it is unlikely that any PV module manufacturer is laminating EVA modules at platen temperatures above 160°C.

Previous laboratory trials demonstrated that with a slow enough temperature ramping to 165°C, bubble formation can be prevented. However, it is necessary to allow enough time for all trapped air and released volatiles to be evacuated prior to cure. The encapsulant formulations developed thus far are not capable of curing in a six minute lamination cycle at 149°C. Therefore, further investigations were performed on various cure agent concentrations using different platen temperatures, and a 6-minute lamination cycle. The results of modifying the cure agent concentration of the candidate encapsulant formulations are presented below in Tables 14, 15 and 16.
Table 14
Modified X15303P EVA at TBEC Peroxide Concentration E

<table>
<thead>
<tr>
<th>Experiment No.</th>
<th>Platen Temp. °C</th>
<th>% Gel Content</th>
<th>Qualitative Cure Testing @ 95°C/10 min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>53</td>
<td>149</td>
<td>68</td>
<td>Failed</td>
</tr>
<tr>
<td>87</td>
<td>152</td>
<td>72</td>
<td>Passed</td>
</tr>
<tr>
<td>67</td>
<td>154</td>
<td>73</td>
<td>Passed</td>
</tr>
<tr>
<td>79</td>
<td>154</td>
<td>74</td>
<td>Passed</td>
</tr>
<tr>
<td>70</td>
<td>157</td>
<td>85</td>
<td>Passed</td>
</tr>
<tr>
<td>80</td>
<td>157</td>
<td>83</td>
<td>Passed</td>
</tr>
<tr>
<td>71</td>
<td>160</td>
<td>83</td>
<td>Passed</td>
</tr>
</tbody>
</table>

Table 15
Modified X15303P EVA at TBEC Peroxide Concentration D

<table>
<thead>
<tr>
<th>Experiment No.</th>
<th>Platen Temp. °C</th>
<th>% Gel Content</th>
<th>Qualitative Cure Testing @ 95°C/10 min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>88</td>
<td>152</td>
<td>68</td>
<td>Failed</td>
</tr>
<tr>
<td>66</td>
<td>154</td>
<td>75</td>
<td>Passed</td>
</tr>
<tr>
<td>68</td>
<td>154</td>
<td>84</td>
<td>Passed</td>
</tr>
<tr>
<td>69</td>
<td>157</td>
<td>83</td>
<td>Passed</td>
</tr>
<tr>
<td>82</td>
<td>160</td>
<td>84</td>
<td>Passed</td>
</tr>
<tr>
<td>83</td>
<td>163</td>
<td>85</td>
<td>Passed</td>
</tr>
</tbody>
</table>

Table 16
Modified X15303P EVA at Alternative Curative Concentration E

<table>
<thead>
<tr>
<th>Experiment No.</th>
<th>Platen Temp. °C</th>
<th>% Gel Content</th>
<th>Qualitative Cure Testing @ 95°C/10 min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>75</td>
<td>149</td>
<td>63</td>
<td>Failed</td>
</tr>
<tr>
<td>89</td>
<td>152</td>
<td>67</td>
<td>Failed</td>
</tr>
<tr>
<td>76</td>
<td>154</td>
<td>74</td>
<td>Passed</td>
</tr>
<tr>
<td>81</td>
<td>157</td>
<td>83</td>
<td>Passed</td>
</tr>
<tr>
<td>77</td>
<td>160</td>
<td>86</td>
<td>Passed</td>
</tr>
</tbody>
</table>

A kinetics model was developed to explain the effects of temperature and cure agent concentration on passage of the qualitative cure test. The rate-determining step is the decomposition rate of the cure agent. Comparing the results for standard concentration of TBEC peroxide at 149°C platen temperature with a lamination cycle of 10 - 12 minutes, the following model results (concentration effect is first order, temperature effect is second order). The goal of any other system is to match this level of cure.

This can be written as:

\[ 1.0 = k \text{ (rate constant)} \left( \frac{\text{conc}_2}{\text{conc}_1} \right) \left( \frac{\text{temp}_2}{\text{temp}_1} \right)^2 \]

where \( \text{conc}_2 \) is the final value, \( \text{conc}_1 \) = standard Conc. of TBEC peroxide; \( \text{temp}_1 = 149°C \).

Thus: \( t_2 = \sqrt{(149)^2 \left[ \frac{1}{k} \left( \frac{\text{conc}_2}{\text{conc}_1} \right) \right]} \). For. The 6 min. system:

The standard concentration of TBEC peroxide using a 6 minute lamination cycle at a platen temperature of 168°C was determined to result in an equivalent cure to standard concentration of TBEC peroxide processed for 12 min. at 149°C. Solving for \( k \) in the above equation results in \( k = 0.79 \); this means that utilizing a 6 minutes lamination cycle at 149°C results in a 79% conversion of standard concentration of TBEC into the same product at 10-12 min. at 149°C.
Using this approach, processing temperatures and cure concentrations are theoretically predictable.

Qualification of the Gel Test
The data presented in Table 17 was obtained using EVA formulated with higher concentrations of TBEC peroxide, processed in a laboratory laminator using a 6-minute lamination cycle. Actual platen temperatures are presented in the table.

<table>
<thead>
<tr>
<th>Experiment No.</th>
<th>TBEC Peroxide Conc.</th>
<th>Platen Temperature °C</th>
<th>% Gel Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>51</td>
<td>E</td>
<td>138</td>
<td>2.1</td>
</tr>
<tr>
<td>65</td>
<td>E</td>
<td>143</td>
<td>4.8</td>
</tr>
<tr>
<td>52</td>
<td>D</td>
<td>149</td>
<td>45</td>
</tr>
<tr>
<td>53</td>
<td>D</td>
<td>149</td>
<td>68</td>
</tr>
<tr>
<td>54</td>
<td>E</td>
<td>143</td>
<td>1.8</td>
</tr>
</tbody>
</table>

Based on the dramatic changes in the measured gel contents from one lamination temperature to another, the simple mechanism of crosslinking can not explain the magnitude of the differences. Apparently, there is another mechanism involved, namely chain entanglement, which is a well-known occurrence in rubber curing. At some point, chain length becomes quite long and chains form an entangled super structure. Analysis for gel content involves heating in toluene at 60°C with little or no shearing. In this process the activation energy to convert swollen gels into solution isn’t provided. Therefore various levels of measured gel content may actually be crosslinked to different degrees and actually cured to different levels. Further work is necessary in this area to determine how to accurately measure the cure level of the EVA.

3.6 Task 6: Handling

In the task 6 effort Solarex is developing improve product and materials handling techniques (including efforts in at least 3 separate areas) to increase line yield by 3% and reduce handling labor to save $0.05/watt. Solarex is supported in this task ARRI. Solarex has modeled the production line, identified critical process and resource bottlenecks, and developed alternative models to show how to meet anticipated production increases.

Solarex contracted Wright Williams & Kelly (WWK) to help analyze Frederick’s current capacity, existing bottlenecks, and future capacity. Using Factory Explorer, WWK’s integrated factory analysis tool, the project team first built and validated a model of the production facility. Next, the project team used this model to analyze the factory’s capacity. This project had two primary goals. The first goal was to build a validated Factory Explorer model of the current Frederick facility. The second goal was to use this model to answer the following questions:

- What is the current capacity of the existing facility (measured in both $ and kilowatts), under a given product mix?
- What are the bottlenecks in the existing facility, and how can they be eliminated?
- What is the maximum output (measured in both $ and kilowatts) that can be achieved in the existing facility, under a given product mix?
The initial questions were answered and the validated model of the Frederick Plant is now being used for production planning, new product feasibility, and other analysis. Factory Explorer is also currently being used to model future plant expansion scenarios.

Based on the Factory Explorer analysis and Solarex and ARRI’s familiarity with the Solarex factory the following handling projects was selected for this project:

1. Handling in casting – one of 3 projects in which ARRI will assist Solarex.
2. Lamination automation
3. Ceramic handling(pieces and slip) – one of 3 projects in which ARRI will assist Solarex.
4. Bus bar assembly – one of 3 projects in which ARRI will assist Solarex.

Handling in Casting

This task has been broken down into two parts. The first part involved a Solarex redesign of the casting station to allow for automatic ingot unloading of the station. This task was begun during the first year, with a prototype unit designed and built by an outside vendor. This unit will be installed and tested during year two of the program. The second part of this project will involve ARRI’s efforts to determine options for handling and transporting large ingots from the casting station into the sizing saws. This task will begin in Year 2.

Lamination Automation

After careful review NPC was chosen to provide a laminator with convey-through capability. The order was placed and the equipment has been delivered. NPC has significant experience making convey-through laminators. There system offered a number of important features including the sealing ability of the laminator and the design of the top sheet conveyor.

The NPC laminator has now been delivered to Solarex and installed in conjunction with new stringing and lay-up equipment. This equipment is now being qualified for manufacturing.

Ceramic Handling

As the ceramic pieces get larger and larger, removing them from the molds becomes more difficult. This is particularly an issue for the new single piece crucible. Solarex engineering developed a method of removing the crucibles using an inflatable bladder inside of the piece. ARRI utilized this concept to design a fixture for removing crucibles from their molds and placing them onto a drying table.

Various techniques for removing the ceramic crucibles from the molds were studied. Raising and transporting the crucibles using the interior walls appears to be a feasible approach, and initial calculations of the required pressures have yielded promising results. Based on preliminary experimental testing using a spherically shaped inflatable bladder, Solarex verified that the parts can be successfully removed from the molds with pressures in the range 0.5-1.0 psi. One issue of concern, however, is that only a very slight increase in pressure can cause breakage of the crucibles. Failure of the crucible is most likely due to the localized stress concentration on the interior walls that occurs at the central zone of the sphere. It is expected that by using a bladder with a shape that conforms more closely to that of the crucible, the difference between the “lift pressure” and the “break pressure” will be significantly increased.
Several options for a lifting device were studied to determine which is most suitable for handling the ceramic crucibles. In order to analyze the advantages and drawbacks of each device, the working pressures required for lifting the part were calculated. It is required that the coefficient of friction between the device and the ceramic surface be known before the pressures can be calculated. A commonly used material for inflatable devices is fiber-reinforced neoprene, which seems to be well suited for the current application. For initial analyses, it is assumed that neoprene will be the material of choice. Using a ceramic block and a sample of neoprene material, the coefficient of friction for ceramic on neoprene was experimentally determined to be 0.5775.

The first design approach was to utilize industrial grade inflatable deep sea lifting bags, customized to meet the requirements necessary for lifting/transporting the ceramic crucibles. The bags are vulcanized in a dry heat autoclave to achieve high strength and durability. Such a unit was procured and tested. It was difficult to insert even the smallest unit into the crucible. We will attempt to obtain an even smaller unit and have it modified with a vertical insert so that it only expands radially when inflated.

The second design approach utilized a pneuma-grip device consisting of one or more flexible expanding tubes in a rigid channel-shaped retainer. The retainer will be designed with the tubes located on outside, to allow gripping from interior walls. Independent operating controls guarantee the maintenance of proper pressures in the tubes. When pressurized, the flexible tubes expand to grip the part evenly and securely without causing damage to the surface finish or configuration. The use of multiple tubes offers the advantage of extra security due to the fact that failure of one tube will not affect the standing pressures of others. The object will continue to be held firmly and safely. For parts with weights and sizes similar to the crucibles, the manufacturer recommends a two-tube device with a standard contact width of 1.5 inches. For safety reasons, each tube should be independently capable of supporting the crucible in case one tube fails. A prototype of such a unit will be evaluated during Year 2.

Bus Bar Assembly

The traditional technique used by Solarex to establish an electrical path from the cell matrix to a terminal screw is to lay four solder-plated copper strips (bus bars) at the top of the module which are attached to the top row of cells via similar, thinner strips (interconnects). The inside edges of the four bus bars are then exposed after lamination for soldering to the terminal block lead wires.

A key problem is that the bus bars are made out of thin, flexible material and their placement relative to the matrix is inconsistent. At the same time, Solarex wishes to modify the method of attachment to the terminal block in the near future. Unlike the present free lead wire method, the new method demands more precise positioning of the bus bars.

ARRI provided a number of design concepts to Solarex for review. Several are discussed below:

**Design Concept 1: Flex circuit bus bar**

**Description:** Individual bus bar strips are replaced by a one-piece circuit made out of flexible material.
Advantages: Although the circuit is flexible, once it lays flat on a plane it is sufficiently rigid to ensure stable positioning. A single part is handled, guaranteeing perfect relative positioning between the bus bar and interconnect strips and dramatically expediting assembly. Method is well suited for automated stack feeding and placement.

Disadvantages: It is anticipated that this will be the most expensive of all solutions from a material standpoint. Also, it is unclear whether flex circuit manufacturers can supply material with the required power rating. There are aesthetic concerns as well (color of the backing material).

Design Concept 2: Semi-rigid circuit bus bar

Description: Similar to Design Concept 1, pursued through a manufacturer of lighting track material which laminates metal strips in a plastic sheath (Xenflex). The product is not plyable like a conventional flex circuit.

Advantages: Similar to Design Concept 1, plus current rating is anticipated to be fully adequate.

Disadvantages: Limited ability to create track patterns relative to standard flex circuits (base pattern is a collection of parallel strips with window cutouts in the plastic sheath for connecting). Anticipated to be less expensive than the latter, but still costly overall.

Design Concept 3: Flex circuit stamp

Description: Equivalent to Design Concept 1; instead of using a full-featured flex circuit with embedded bus bars, the current tin-plated strips are retained while a “stamp” of flex circuit material is used to relax the dimensional tolerancing requirements imposed by the surface mount terminal strip.

Advantages: Reduced cost relative to full-featured flex circuit. May be produced on a continuous strip by the manufacturer which is delivered on a tape and cut to length as required (similar to the bus bar and interconnect material).

Disadvantages: Current capacity of the flex circuit unknown. Introduces additional soldering steps (from bus bar to stamp and from stamp to terminal block).

Design Concept 4: Semi-rigid circuit stamp

Description: Equivalent to Design Concepts 3 using the Xenflex material.

Advantages: Equivalent to Design Concept 3, plus current rating is anticipated to be fully adequate.

Disadvantages: Unknown whether the desired stamp pattern can be produced with this material.
Design Concept 5: Jig redesign

Description: Redesign jig to enhance the positional stability of the bus bars and interconnects relative to the cell matrix.

Advantages: Least expensive.

Disadvantages: Bus bars may still be displaced once they come out of the fixture when readied for lamination.

Requests for quote on the stamp concept were followed all the way through for three suppliers — Parlex (flex circuit division), Starfire Lighting, and TycoFlex. Of these, the first two came back with similar and generally favorable quotes. However, the idea was tabled due to cost and assembly concerns.

A second design and round of RFQs was issued for a laminated cable concept that would entirely remove the top bus bars and guarantee the required positional accuracy. The new concept has two basic advantages over the present method:
1. lower variation in bus-bar location, which permits use of a wire-less terminal block, and
2. easier placement on the cell matrix, which is particularly important with the pending implementation of automatic cell stringing machines.

ARRI designed and issued an RFQ for a laminated cable solution to replace the current individually placed bus bars. Of the three companies approached, in the end only the Parlex Laminated Cable Division responded. Based on Parlex’s initial quote, ARRI issued a second design that would reduce the number of different laminate designs from two (mirror images of each other) to one that could be used on both sides of the module. However, although this step would cut supplier costs exactly in half, Solarex would still have to lay two separate parts and align them properly. Further discussion between Parlex, Solarex and ARRI revealed that it would be possible for Parlex to simplify the design even further, developing a single laminated section that would span the entire module, while providing full trace isolation at the center, using the plastic backing to hold all four bus bars in one piece.

At this stage in the development Solarex decided to pursue a different approach for attaching the terminal strips to the bus bars. The new method features slitting and threading the bus bar through the backshe. This makes it possible to solder the terminal strip directly to the bus bars, eliminating the wires. Parlex Laminated Cable division responded with another quote for a single laminated cable that will span the module, yet is folded in the center so it can thread through the back sheet. This concept offers many advantages to the old system. At this time environmental tests are being performed on modules made using this method to determine if adhesion of EVA to the laminated cable is adequate.

3.7 Task 7: Measurement and Control

In the task 7 effort Solarex is developing process measurement and control procedures for use on the production line (including efforts in at least 3 separate areas) to improve yield by 3% and reduce rework by 50%. In order to improve process measurement and control in the production line and reduce rework Solarex, with support from a lower-tier subcontract with ARRI, will implement an improved information system, develop and implement a brick identification system, and perform a factory measurement and control survey to determine what areas require
improvement. Solarex, with support from a lower-tier subcontract with ARRI, will select at least 3 measurement and control projects for implementation in the program.

Data Collection System

Solarex hired Industrial Design Corporation (IDC) to study the current data collection system in use at Solarex. IDC provided a detailed report to Solarex with the following major findings.

1. The flow of information has become fragmented and sometimes incomplete as the factory has grown.
2. The present barcode data collection system is obsolete and has not been updated to contain complete production or process engineering data.
3. Because of the first two, some of the reports generated from the system are incomplete or not available on a timely basis.
4. A new system should be implemented to supervise and report on overall factory conditions as they occur. This should include work-in-progress (WIP), factory efficiencies, yield and throughput.

Solarex has begun the process of upgrading the system based on these recommendations. Key systems requirements have been defined and communicated to potential vendors. A final decision on upgrading the system will be made during Year 2.

Brick ID System

One approach to improve data collection would be to mark and track each wafer. Our plan is to develop a process whereby we mark the face of every silicon brick before wafering. Hopefully, these marks will be readable on the wafers all the way into the finished product and identify the brick from which each wafer came and its position within the brick. This will have the following benefits:

1. Improve tracking so that differences in casting and silicon preparation can be detected at cell test. The best measure of material quality that can be acquired in large quantities is cell performance. Because of the great number of steps between casting and cell test there are many opportunities for lots of wafers to get mixed up making it hard to trust the current lot tracking data when trying to trace variation back to casting. A brick ID system would all but eliminate this problem. The ID codes would be applied after bricks are cut from the ingot. The path a brick and its wafers take from there on will not matter as each wafer would be identified at cell test. Also, the performance of the cell line varies over time, which makes evaluating long term casting experiments difficult. A system could be established where samples from some bricks get released to the cell line over an extended time period. This would allow evaluation of results based on the relative performance of single bricks over time.

2. Provide continuous brick profiling to drive decisions about the usable area in each brick. The material quality varies from top to bottom within each brick. The extreme bottom and top sections are unsuitable for making cells. We remove and recycle sections from the top and bottom of each brick. The performance profile for each brick is different. From careful measurements of a small sample of bricks we have established parameters for cropping the same amount from each brick. We try to set the cropping lines so that we pass some bad material rather than rejecting good material. The brick identification system would provide profile information on every brick allowing us to set the cutoff line more accurately. This information could also help decide when a particular casting stations requires maintenance.
3. Improve control of the cell line by using the brick position data to filter out some of the material related noise in the data. Control of the cell line from cell test feedback is complicated by the variation in material quality within each lot and from lot to lot. Much of this variation is caused by bad material at the tops and bottoms of bricks. This variation could be reduced by only looking at material from the middle of the bricks when querying the cell test database. Brick to brick variations will be easier to detect because of the certainty of identity of each wafer. The random order of cells could help differentiate between temporal changes in the cell line process and differences between bricks.

4. Simplify material flow and tracking with reduced paperwork. Production paperwork and handling can be simplified. No lots would be necessary. Wafers could go from the saw room to wafer cleaning as soon as they were cassetted in any order. The effort taken to keep lots separate would no longer be necessary. Machine cycle counters could track production flow if needed and breakage could be tracked by station and shift if desired with simple hand counters or their electronic equivalent.

5. Allow simpler production of qualified material. Carefully designed controlled cell processing experiments work best if there is little variation in the material. This has been achieved in the past by carefully profiling a brick on a sampling basis and determining if there is a suitable range of wafers within the brick with small variation. The brick ID system would greatly simplify this process. A random sample of a brick could be released to the cell line and the profile could be determined by simply querying the cell test database for that brick.

The technical challenge facing a brick marking system is the development of a way to mark the brick that (1) is retained on the edge of the wafers after sawing, etching, etc. (2) does not cause wafer breakage and (3) can be read easily at cell test. Two methods of marking the silicon bricks prior to the wire saw process were researched. The first method considered is laser etching the brick with a barcode. A barcode scanner or CCD camera can read the laser-etched barcodes. This technology is considered off-the-shelf and vendors are available to provide this equipment. The second method considered is diamond-wheel scribe. This method utilizes a diamond-cutting tool mounted on a device similar to a grinding wheel to cut grooves in the silicon. The grooves cut into the silicon are positioned at different locations along the silicon brick to differentiate the wafer location from the brick from which it was cut.

The amount of information to be encoded on each individual wafer affects the selection of an ID marking method. A nominal set of information storage requirements have been established and justified as follows:
- Ingot serial number – To insure that ingot numbers don’t repeat too often to cause confusion, we have requested an ingot retention capability of 20,000, which can be accommodated with 15 bits of information.
- Brick number – An estimated ceiling for number of bricks per ingot is 32, corresponding to 5 bits.
- Position within the bricks – The projected total number of wafers per brick is 600 to 700. However, it is not strictly necessary to identify each wafer exactly. A resolution of 1 mm in position would be adequate so identification of 250 positions is the maximum required, which can be accommodated by 8 bits.

Based on this analysis the minimum bit pattern required is 28 bits. This can be accommodated with a 4-digit Code 39 barcode using the full ASCII feature.

ARRI recommended laser etching of the barcodes as the preferred approach and provided Solarex with several candidate vendors for both laser systems and bar code readers. Sample bricks were sent to two laser system vendors (AB Laser and Control Micro Systems). Each laser vendor etched one face of each of the silicon bricks with two different types of barcodes. One barcode
type is a 1D code and the other a 2D code. The laser vendors were not able to etch the full length of the brick due to the equipment available in their lab at the time of the testing. Results on the AB Laser sample were not good because of poor contrast. However, the Control Micro Systems bar codes were easy to read on the bricks. These bricks were then wafered and some of the wafers were processed into cells. Neither the wafers nor the cells could be read by a standard bar code reader. However, the laser marks could actually be felt on the edges of both the wafers and cells. We are now trying to determine if an optical identification system can see the marks on the wafers and/or cells.

The second approach uses diamond-wheel scribing, which consists of imparting wide diameter grooves along the brick faces to encode wafer information. The main benefit of this method is that minimal stress concentrating notches would be left on the edge of the wafer, thereby diminishing the chances of cracking during thermal shocks. A second benefit is that the marking technology would be significantly less expensive than laser scribing.

Unfortunately, two considerable drawbacks apply to this technique. The first is that the groove width limits the information density. The best case scenario would be to encode the 20 bits required for the ingot serial number and brick number combined as two 10-bit fields engraved on two of the three free sides of the brick. This would allow for an indentation width of slightly less than 0.5”, which is still adequate. The wafer position within the brick would be encoded in an analog fashion on the remaining face, by scribing a groove along the main diagonal of a second free brick face akin to what’s currently done at Solarex. Finally, an additional fiducial marking would have to be done to identify which side of the wafer is “up.”

The second drawback is that no standard reading method exists. One likely approach would involve a custom-made machine vision system, requiring the placement of the wafer on a backlit fixture for maximum contrast. A mechanical displacement method would require more development than the vision system, and would be more complex and probably no cheaper in the end. In either case, on-line reading of the indentation markings from conveyors, cassettes or stacks would be unfeasible.

Since the laser barcode process has not worked, we will attempt to use this second approach during Year 2.

Doping and Diffusion Measurements

There was consensus of the Solarex engineering staff that diffusion was the most important place to begin improved measurement and control. A prototype measurement and control system for the diffusion area was proposed. ARRI identified the sensors, computer hardware and software necessary for such a unit.

ARRI assembled a prototype unit with sensor interface modules for thermocouples, frequency counters and generic RS-232 interfacing (such as digital scales, bar code readers, digital voltmeters, etc.). A major issue involved the wafer sensor selection and placement for counting. ARRI identified a potential source that eventually resulted in procurement of a sensor that could identify and therefore count silicon wafers.

This prototype system will be deployed in the production line in the first quarter of Year 2.
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This report describes work done by Solarex during the first year of this subcontract. The objective of this three-year PVMaT program is to continue the advancement of Solarex PV manufacturing technologies to design and implement a process that produces polycrystalline silicon PV modules that can be sold profitably for $2.00 per peak watt or less and that will increase the production capacity of the Frederick plant to at least 25 megawatts per year.

Accomplishments during the first year of the program include:
- Verification of the process to produce SiF₄, the precursor to silicon feedstock.
- Design of a silicon feedstock pilot facility using the SiNaF process.
- Development of and transfer to manufacturing of a process to use thinner wire in the wire saw.
- Completion of a production trial with recycled SiC.
- Laboratory development of a selective emitter process using rapid thermal processing.
- Fabrication of high-efficiency polycrystalline cells using silicon nitride from three different sources.
- Development of a new encapsulation formulation and laboratory demonstration of a 6-minute lamination cycle.
- Implementation of an automated laminator.
- Laboratory demonstration of automated handling of ceramics.