Solar Energy Technologies Program

Office of Energy Efficiency and Renewable Energy

United States Department of Energy

SunShot Solarmat-2 Program

Project Title	Integrated Glass Coating Manufacturing Line
Award Number	DE-EE0006810
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Project Objectives

This project aims to enable US module manufacturers to coat glass with Enki's state of the art tunable functionalized AR coatings at the lowest possible cost and highest possible performance by encapsulating Enki's coating process in an integrated tool that facilitates effective process improvement through metrology and data analysis for greater quality and performance while reducing footprint, operating and capital costs.

The Phase 1 objective was a fully designed manufacturing line, including fully specified equipment ready for issue of purchase requisitions; a detailed economic justification based on market prices at the end of Phase 1 and projected manufacturing costs and a detailed deployment plan for the equipment.

Background

In recent years, the solar PV industry has undergone dramatic expansion driven by rapidly declining costs of module manufacture and installation. Suppliers across the solar value chain face relentless pressure to reduce costs and improve the energy harvest of their products. To compete effectively, manufacturers must enable superior product quality and energy capture at reduced cost. Enki furthers this mission by its highly differentiated, drop-in platform for applying anti-reflective and anti-soiling coatings for PV modules that are optimized for individual customers and end use environments. Current state-of-the-art anti-reflective (AR) coatings for PV offer only AR benefits and must be applied during glass manufacture and cured at very high temperatures. Furthermore, current state-of-the-art AR coatings vary dramatically in durability, and glass companies themselves range widely in their ability to apply the coatings consistently and correctly. As a consequence, module manufacturers and end module buyers may pay more for coatings that do not deliver on their projected performance increase, or that do not exhibit long-term durability in the field. Enki solves these problems by offering the first solution that disassociates glass manufacture from coating. Unlike conventional coatings that must be cured at the high temperatures used for glass tempering, Enki's CleanARC® material is curable at low temperatures directly before, or after module assembly. In addition, the CleanARC chemistry platform is tunable to achieve a wide range of physical properties including extreme durability, best-in-class optical gain, and range of surface energy. By enabling end module manufacturers to apply highly functionalized coatings, and to apply them directly, CleanARC changes the supply chain equation: offering a superior product while at the same time reducing costs and improving quality control.

Based on a several patent pending chemistry and process technologies, Enki's coating utilizes a low cost, wet chemistry process in which precursor organosilane molecules are used to make low molecular-weight silsesquioxane co-polymers. Selection of the precursors, the process conditions, and the catalysts used to create the silica-based polymers enables creation of a solution that forms a dense, highly engineered thin-film when applied to a glass surface. This enables the coating to exceed common durability standards with curing as low as 120 °C. This is in sharp contrast to the other available AR films which require a sintering step in excess of 600 °C as the glass is tempered in initial production.

As the solar industry continues to mature, module buyers and solar plant operators are becoming increasingly sophisticated on the lifetime operating cost and power generation implications of the solar components they procure. The coating on outside cover glass of PV modules affords a unique opportunity to optimize the interaction of the module with its specific end use environment, and optimizing the energy capture and long term durability of this coating is a very high value added activity. Today's state-of-art coatings waste this opportunity. Enki's CleanARC technology platform was conceived to reduce the lifetime levelized cost of produced electricity from solar PV plants by reducing soiling rates, providing high optical gain and high durability. The technology platform is innovative in material structure, material tunability/functionalization and processability attributes. Taken together, these innovations offer a significantly superior and more durable product that also changes the supply chain equation by allowing module manufacturers to cost effectively coat incoming glass (or completed modules) themselves. This is an important innovation, because module companies (and in particular vertically integrated module companies who also develop and manage solar assets) are best positioned to articulate and capture the value in a superior coating product, and they are most incentivized to ensure high product quality and energy production. Today, module manufacturers go to great lengths to qualify both the coating materials and process controls used by their glass suppliers. They find themselves locked into a limited number of supply choices, yet often find they are receiving coatings that do not deliver on their promised gain. Enki's coating platform solves this problem and greatly simplifies glass supply chain logistics by allowing module manufacturers to source bare glass from the broadest pool of potential suppliers, directly control the quality of applied coatings to maximize energy gain at the module level, minimize cosmetic variations in modules at end of line and tailor the applied coating to specific customer's needs and environments without increasing their overall inventory burden. Enki's platform also fits well with a contract module assembly business model, where solar technology companies leverage the lean and highly efficient production expertise of companies such as Flextronics or Foxconn. Such contract assemblers are eager to provide additional value and simplify their own supply chains.

Task 1 – Equipment Specification & Procurement

Objective

This task encompassed all equipment development and design and all interactions with equipment vendors to develop final drawings and specifications to the point where budgetary quotations can be issued by the vendors. The first objective was to fully design the line to the point where a detailed economic analysis could be performed and if the final design is economically viable then the equipment designs are ready to order. The second objective was to develop new technology that elevates a typical glass coating manufacturing line to state of the art.

Technical Accomplishments

Plasma System Proof of Principle

The major objective of this sub-task was to identify an advanced method of surface treatment that can potentially eliminate the need for a large and expensive glass washer.

Experience with coating large quantities of glass and modules has helped us greatly understand the issues related to surface preparation. In the case of crystalline solar module glass the glass as received at the module manufacturer is now typically pre-washed. If it is not washed, or has been stored for more than a month or so, then a very basic washing cycle is all that is required to re-activate the surface for coating. However, surface contamination, from glass handling equipment such as suction cups and conveyor rollers can affect the surface and result in visible marks after coating. This surface contamination is very difficult to remove. Our focus therefore shifted to investigate if plasma surface treatment could solve this issue rather than simply activate the surface for coating as envisioned in the original plan.

Initially we proposed to test a prototype plasma system called DCSBD (Direct Current Surface Barrier Discharge) developed by a group at the R&D Center for Low-Cost Plasma and Nanotechnology Surface Modifications (CEPLANT) in the Czech Republic. This system looked very promising, but was still at a relatively immature development level. After additional market research we identified a Korean manufacturer called PSM (Plasma Systems and Materials), that have a USA sales office in San Jose, California. Given the lower technology risk of the PSM system and its equivalence to the DCSBD system we decided to proceed with proof of principle testing with PSM instead of the CEPLANT group.

We also learned that direct flame impingement could also potentially be a low-cost viable alternative to plasma-treatment. We decided to include a proof of principle test of this technique into the plan. We identified a manufacturer called Enercon located in Wisconsin that supplied a flame treatment system for activating the surfaces of plastics prior to coating or painting.

Unfortunately to test both techniques we had to send prepared samples to the manufacturer location for exposure rather than being able to test them locally. This introduces a time and shipping variable into the experiment, wherein the treated glass surface may change after treatment but before we have the opportunity to test it at Enki's location. For the proof of principle experiment we prepared pre-contaminated samples that were shipped to PSM and Enercon. The samples were TEC™ 15 float glass from NSG/Pilkington. Each sample was intentionally contaminated with a rubber suction cup, a silicone suction cup, a strip of grease (Blue Hawk) and a stripe of silicone lubricant. The figure 1 below shows a photo a schematic of a sample. 5 samples were prepared and shipped to PSM in Korea and 12 samples were prepared and shipped to Enercon in Wisconsin. The plan was for each sample to be half exposed (down one side) to the treatment and then returned to Enki for testing.

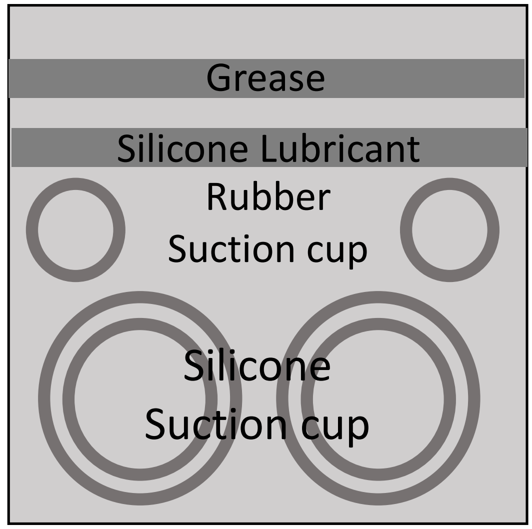
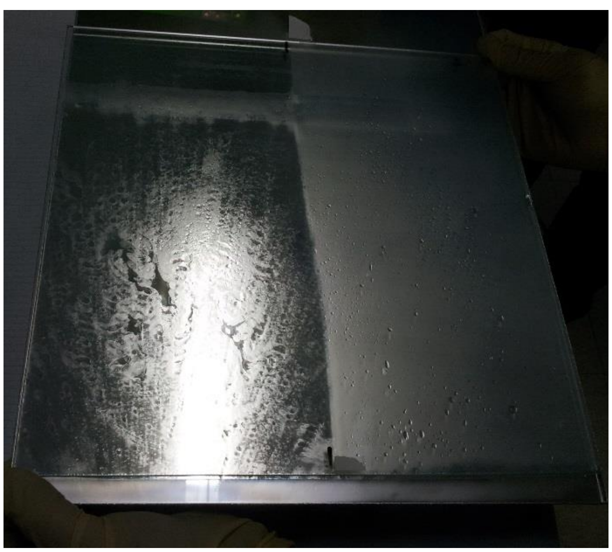
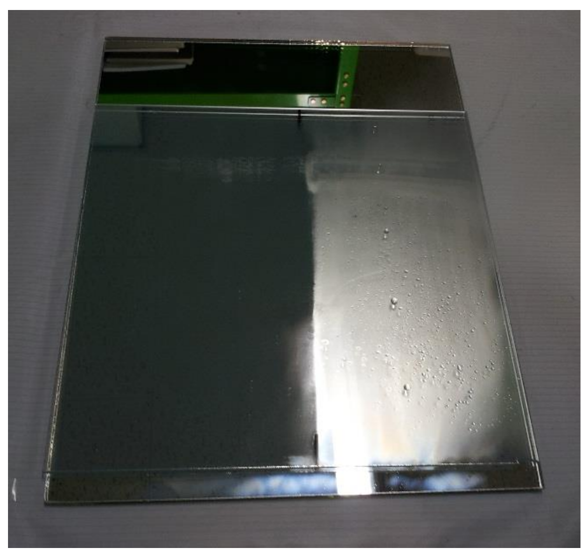


Fig 1. Surface Treatment Sample Preparation

Figure 2 below shows the results of the test at PSM on two of the samples. The left side of each sample was treated with atmospheric plasma. The surface is then sprayed with steam to condense a thin film of water on the surface. The treated side is very hydrophilic which causes the water to form a thin transparent film. The untreated side to much less hydrophilic and forms the familiar fog. This technique is also very effective at highlighting visible defects caused by surface contamination. In the left hand picture (single treatment) it is possible to see a remnant of the silicone suction cup mark in the bottom left corner of the sample.





Sample #1 - Single Treatment

Sample #2 - Double Treatment

Fig. 2 Plasma Treated Samples immediately after treatment

The table 1 below summarizes the experiment and results performed by PSM. The grease and silicone stripes were not removed by any of the treatments. Most of the cup marks were removed, but they were quite hard to see even on the untreated side.

Sample No.	Speed (m/min)	Treated		Untreated	
		Silicone	Rubber	Silicone	Rubber
1	1.8	Not Visible	Not Visible	Not Visible	Not Visible
2	0.9*	Not Visible	Visible	Not Visible	Visible
3	0.9	Not Visible	Not Visible	Not Visible	Visible
4	1.2	Not Visible	Not Visible	Visible	Visible
5	1.5	Not Visible	Not Visible	Not Visible	Visible

* Two passes at 1.8 m/min ≈ 0.9 m/min

Table 1. Summary of PSM Plasma Results

Table 2 below shows the process conditions used by Enercon to flame treat the samples. The initial and final Dyne/cm is a direct measure of surface energy, the higher the number then the more hydrophilic (activated) the surface.

System	Gap (mm)	Speed (m/min)	Initial Dyne/cm	Final Dyne/cm	Comments
Dyne-A-Mite IT	6	15.2	30	70	Treated sample, tested then destroyed. Would need multiple heads to cover entire surface.
Dyne-A-Flame	38	15.2	30	66	Treated sample, tested then destroyed. Achieved adequate treatment.
Dyne-A-Flame	25	7.6	30	70	Treated sample, tested then returned for customer evaluation. Achieved excellent treatment but did not affect grease or silicone. Labeled treated half on backside.
Dyne-A-Flame	25	7.6	30	72	Treated sample, tested then destroyed. Attempted to clean grease and silicone with no success.

Table 2. Summary of Test Conditions for Enercon Flame Treatment Experiment

As before in the plasma experiment the grease and silicone lubricant strips were not removed. Unfortunately the flame treatment did not successfully remove the suction cup marks either. Table 3 below summarizes the visual results on the 11 samples that were returned to Enki.

Sample No.	Treated		Untreated	
	Silicone	Rubber	Silicone	Rubber

1	Visible	Visible	Visible	Visible
2	Visible	Visible	Visible	Visible
3	Visible (less)	Visible (less)	Visible	Visible
4	Visible (less)	Visible (less)	Visible	Visible
5	Visible (less)	Visible (less)	Visible	Visible
6	Visible	Visible	Visible	Visible
7	Visible (less)	Visible (less)	Visible	Visible
8	Visible	Visible (less)	Visible	Visible
9	Visible	Visible	Visible	Visible
10	Visible	Visible	Visible	Visible
11	Visible	Visible	Visible	Visible

Table 3. Summary of Enercon Flame Treatment Results

While the major focus of this task was to evaluate how well each surface treatment removed visual defects caused by surface contamination a second goal was to determine that the surface activation was sufficient to achieve a good adhesion to our coating. Direct measurement of adhesion of these types of very thin, very hard coating on glass is not possible. However, abrasion testing is a reasonable proxy. Performance is measured by reduction in optical transmission with abrasion cycles. For this test we used a Taber linear abraser and a test protocol based on ISO-9211-4 which specifies a CS-10F "wearaser" pad and 10N of force. This is a very aggressive test that quickly removes the coating. Given that we believe the flame treatment was not as good as the plasma treatment, we elected to use samples returned from Enercon for the abrasion experiment. 5 30x30cm samples were selected and 4 10x10cm coupons were cut from each, two from the treated side and two from the untreated side. This yielded a total of 10 treated and 10 untreated coupons that were then coated with Enki's material. While there appeared to be a clear benefit for performance of the treated samples (in that they lose slightly less optical transmission) the difference between the two sample sets was not statistically significant.

Our conclusions at the end of the experiment were that plasma works better than flame to remove contamination. Both treatments work well to activate the surface. However we did not see a statistically significant improvement in abrasion resistance with the treatment. Given that the plasma system worked better in our tests and its commercial availability, should a pre-treatment option be included in the integrated manufacturing line, then a plasma system from PSM will be selected.

Pre-Treat Vendor Drawing and Specification

The objective of this task was to obtain a budgetary quotation for the pre-treat system that is sufficiently detailed to perform the subsequent economic analysis and to obtain initial drawings and specifications to give clarity on exactly what is being quoted.

The pre-treat process removes surface contamination and activates the surface prior to coating. We have shown that both surface activation and removal of light contamination can be achieved by simple washing. However heavy contamination such as that from glass handling equipment such as silicone rubber suction cups or conveyor rollers cannot be removed by washing alone. For this type of contamination we have shown that an atmospheric plasma system is effective.

For the purposes of the economic analysis we obtain three different quotes that represent three levels of pre-treat capability. This allows us to model various scenarios of incoming glass quality and cleanliness. For a basic glass washer we obtained a quotation from Billco Manufacturing Inc. Zelienople, PA, for a model 310-48-8 Flat Glass Washing & Drying Machine along with a second quotation for the same washer with the addition of an optional 2x oscillating scrub unit (4-rows of cup brushes that rotate while moving back and forth in the glass surface). For an atmospheric plasma cleaner that could be added subsequent to the glass washer we obtained a quotation from PSM Korea for their DRXP2-1200 Plasma Tool.

Figure 3 below shows plan and elevation views of the glass washer. Figure 4 below show plan and elevation views of the plasma tool.

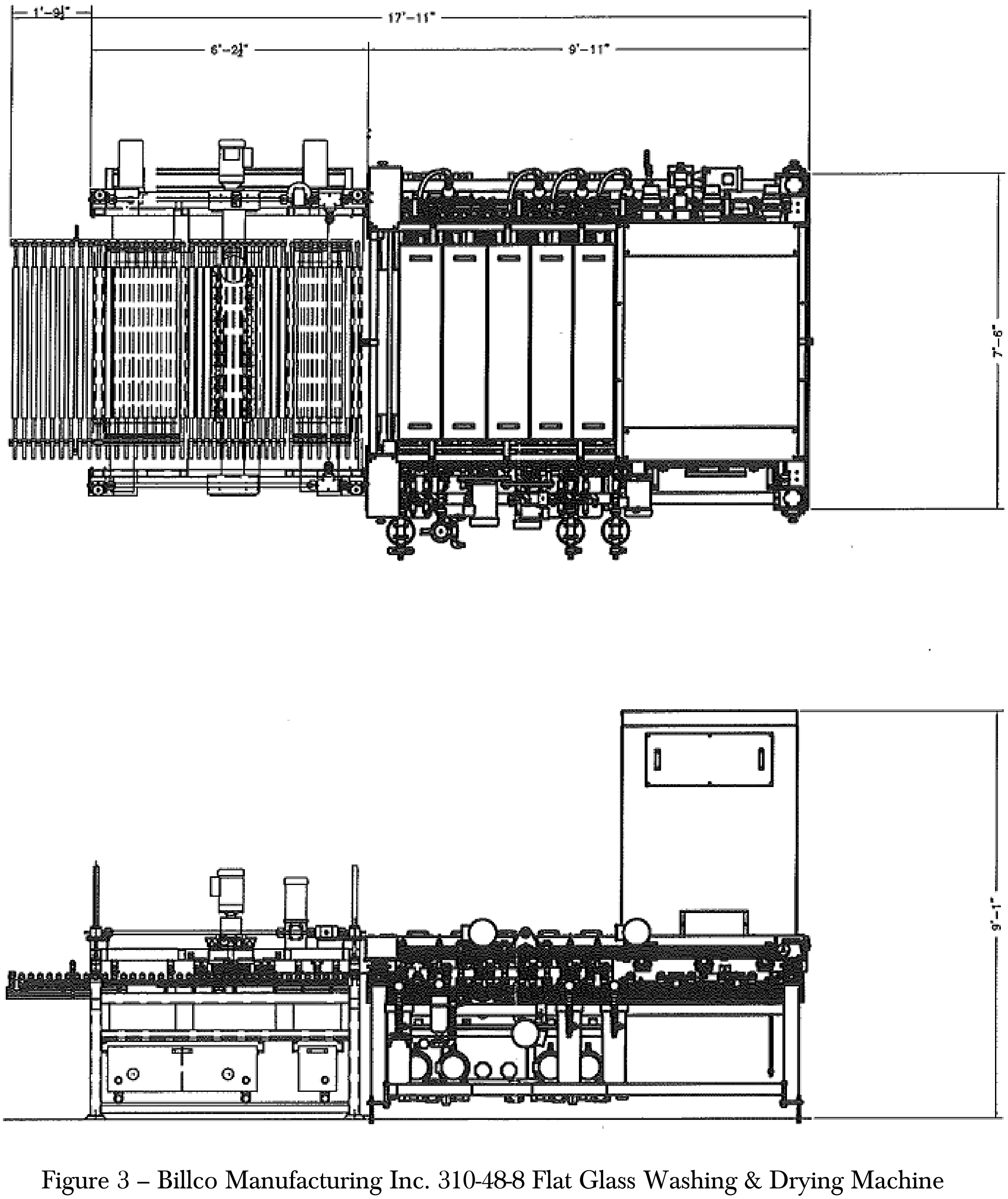


Figure 3 – Billco Manufacturing Inc. 310-48-8 Flat Glass Washing & Drying Machine

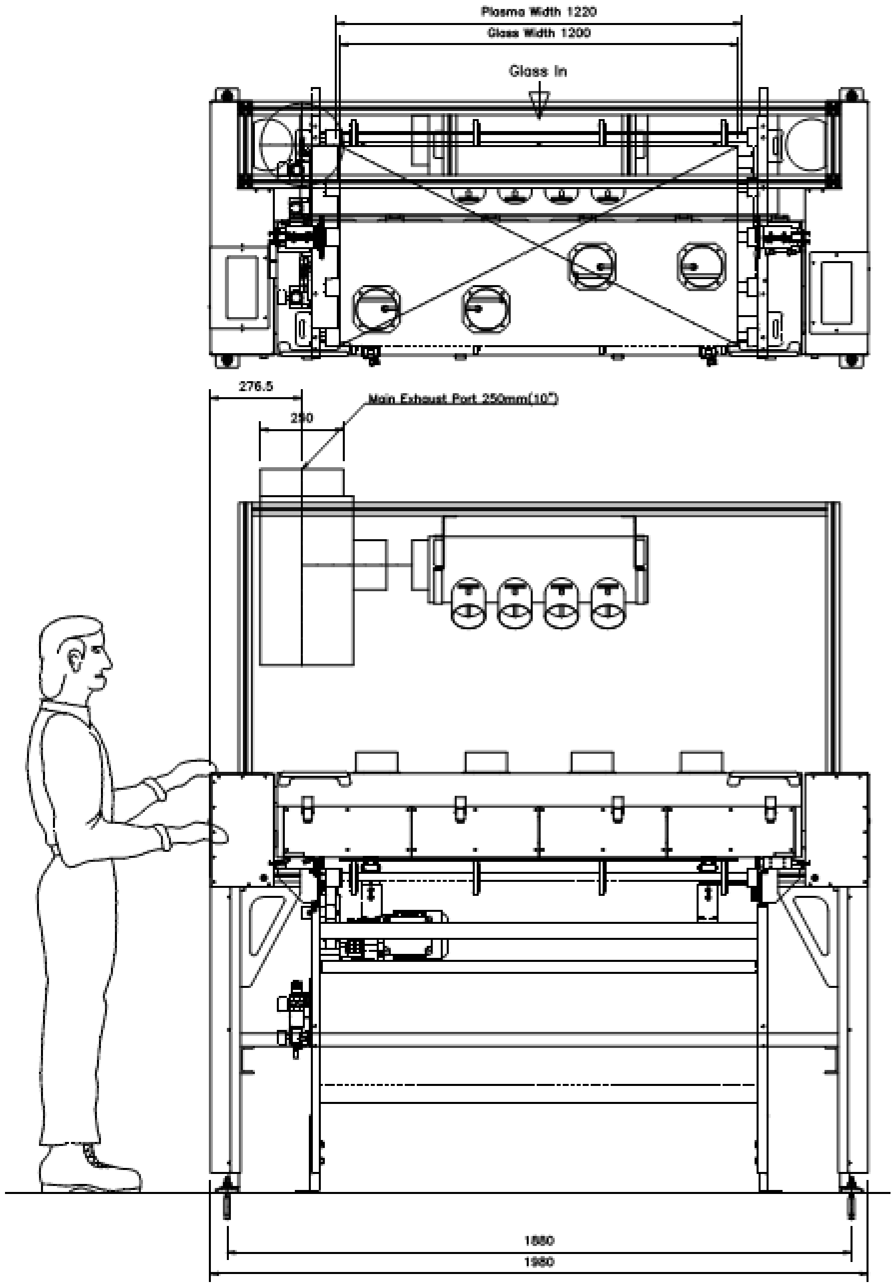


Figure 4 – PSM Korea DRXP2-1200 Plasma Tool

Substrate ID Mark Station Drawing and Specification

The objective of this task was to obtain a budgetary quotation for the laser-mark system that is sufficiently detailed to perform the subsequent economic analysis and to obtain initial drawings and specifications to give clarity on exactly what is being quoted.

Additionally this task included a basic proof of principle test to show that the equipment selected could perform the function needed.

The purpose of the substrate ID mark process is to apply a unique mark that can be used to permanently identify a substrate and enable traceability to the time, equipment and materials used when the substrate was coated. This traceability is a foundational feature of creating robust process improvement in a manufacturing environment.

There was substantial existence proof that a CO_2 laser system can be used to laser mark glass. For example, we know that laser marks can also be found on tempered glass used in the automotive industry. Based on this information we engaged with Synrad Inc., of Mukilteo, WA to perform an experiment on small coupons of both coated and uncoated low-iron patterned glass used in the crystalline solar module industry.

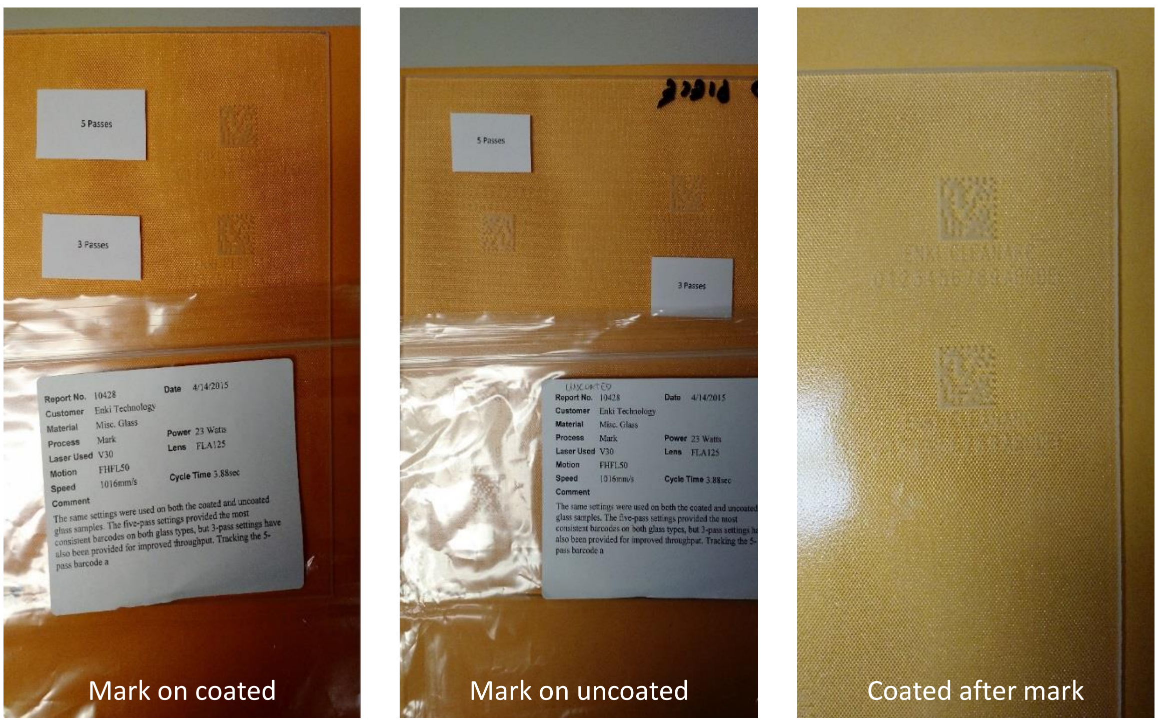


Figure 5 – Laser marked glass coupons

Figure 5 above shows the results of the experiment. We provided 2 coated and 2 uncoated 20 x 20 cm samples of solar glass to Synrad. After some optimization they marked all 4 pieces and returned them to us. We then coated one of the previously uncoated marked pieces to prove that we could both coat over the mark and that the mark would still be readable after coating.

To prove the mark was machine readable, we selected an off-the-shelf barcode scanner camera manufactured by Microscan and distributed by Redline Solutions Inc., of Santa Clara, CA. Figure 6 below shows the output of the camera reading both coated and uncoated marks and also shows the output of the recognition software showing that the mark as successfully read and decoded.

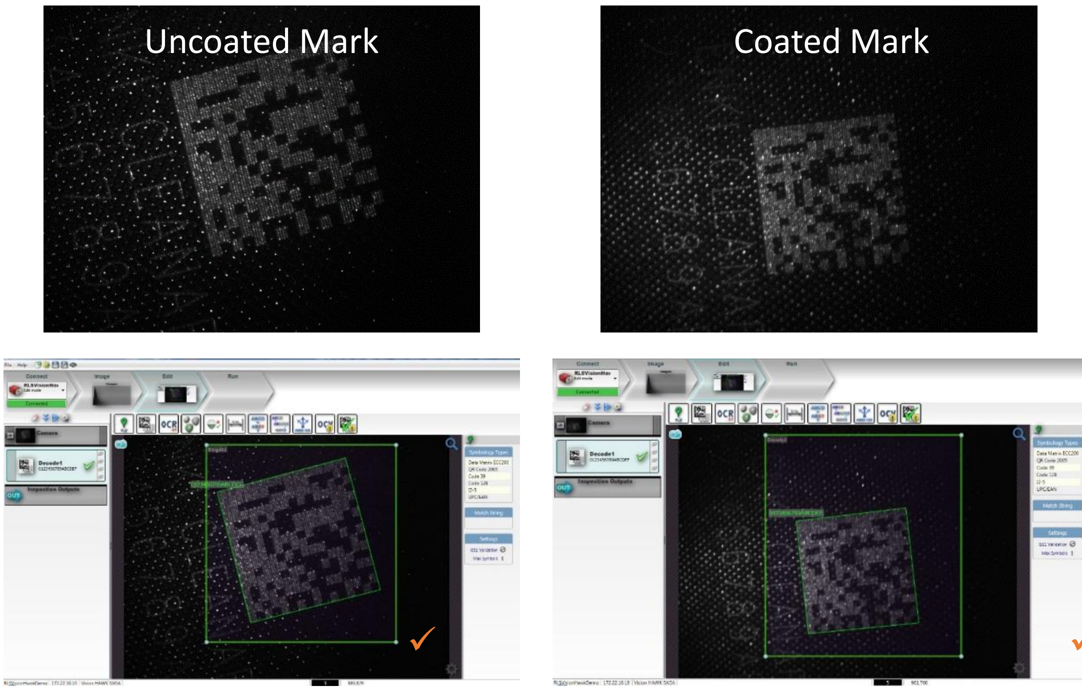


Figure 6 – Results of Barcode Reader Test

Shown in figure 7 below, the selected laser mark system from Synrad is the FSV30SFG Firestar 30W laser with marking head, Focusing Lens and mounting kit. The selected reader was Redline Solutions Vision Hawk Camera with a 30° lens, darkfield light and material sensor. Shown in figure 8.

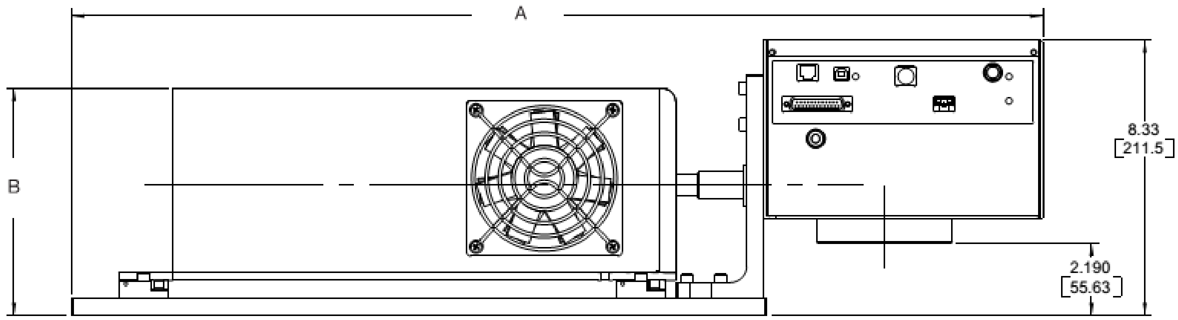


Figure 7 – Drawing of Synrad Laser & Marking Head

Figure 8 – Redline Solutions Vision Hawk Camera

Chemical Dispense Unit (CDU) NVC Proof of Principle

The Enki coating material is composed of mostly volatile solvent and a small percentage of non-volatile content (NVC). When the coating is applied to a substrate the volatile solvent evaporates and the NVC forms the final thin-film coating.

The coating system constantly circulates coating material through the system and during this process solvent is lost to evaporation. Therefore make-up solvent must be added to keep the NVC with its control limits. In commercial Chinese coating lines this process is done manually and control over the NVC is poor. This task was executed to demonstrate proof of principle of controlling NVC automatically using simple metrology and algorithms.

Using our prototype roll-coating line we simulated a real-world coating scenario by running the coater for an extended period of time. We started with a 60 minutes idle period, with no glass being coated and just the material running through the coater; then 60 minutes coating glass; then a final 60 minute idle period. During this 180 minute test we executed the following procedure:

1. Take 3ml sample for NVC
2. Record material weight
3. Enter weight into a spreadsheet to calculate the amount of make-up solvent
4. Add the indicated amount make-up solvent from the spreadsheet
5. Wait until 10min elapsed since 1
6. Go to step 1

Figure 9 below shows a schematic representation of the test. Showing the coating material reservoir and the circulation path through the roll-coater.

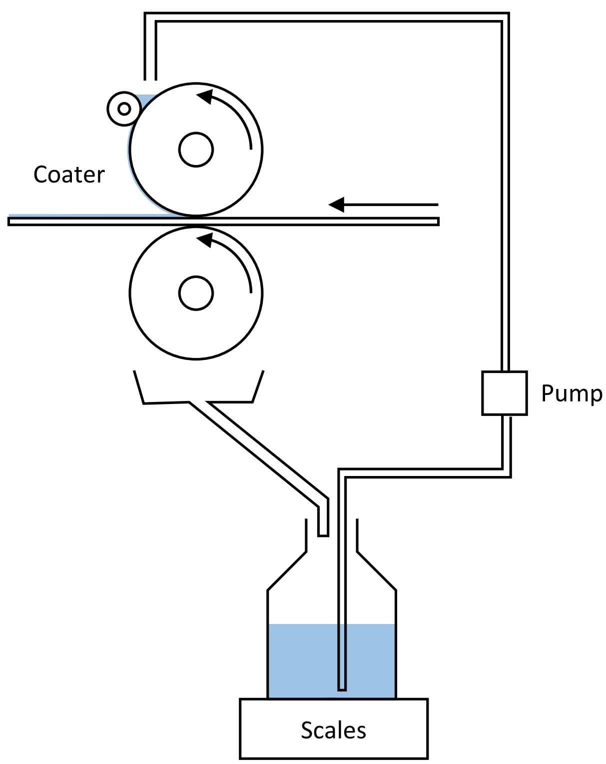


Fig. 9 – Schematic View of Coating Material Loop in Roll-Coater

This experiment was successful in demonstrating that we can control NVC with just a simple algorithm and low-cost sensors to track the weight of the coating material.

Cure Oven Vendor Drawing and Specification

The objective of this task was to obtain a budgetary quotation for the cure-oven that is sufficiently detailed to perform the subsequent economic analysis and to obtain initial drawings and specifications to give clarity on exactly what is being quoted.

As has been mentioned in previous reports, in the context of sol-gel coatings “cure” refers to the condensation of Si-OH groups to an Si-O-Si network and the release of water via the following reaction:

$$\text{Si-OH} + \text{Si-OH} \rightarrow \text{Si-O-Si} + H_2O$$

The initial ‘gelling’ of a coating (that is when it turns from liquid to solid) condenses some of the Si-OH groups. Condensation of the remaining groups is dependent on a cure at higher temperature.

After substantial experimentation with various methods it was determined that radiative heat transfer in the mid-IR range as the most efficient method of heating glass quickly and efficiently.

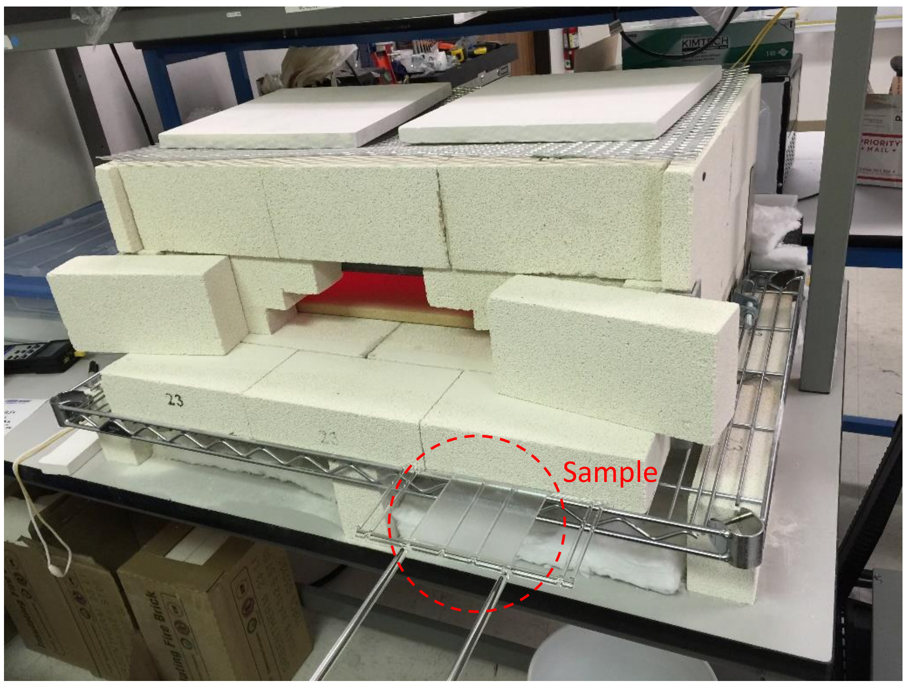


Figure 10 – Proof of Principle IR Cure Oven

Figure 10 above shows the small scale IR oven built to fully characterize the cure process at small scale. This is an electric oven. We tested a comprehensive matrix of times and emitter temperatures with their resulting temperature of the substrate and the degree of cure achieved compared to a baseline process in a conventional convection oven.

The full-scale oven design uses natural-gas fired pipe burners. These are far more cost efficient than electrical heating elements; especially in the US which enjoys very low natural gas pricing. Figure 11 below shows a picture of one of the gas-fired IR emitters.

Figure 11 – Gas-Fired Pipe Burner

Task 2 – Material Manufacturing Readiness

Objective

The objective of this task is to develop coating materials optimized for automated manufacturing.

Technical Accomplishments

Cure time characterization

In the context of sol-gel coatings "cure" refers to the condensation of Si-OH groups to a Si-O-Si network and the release of water.

In this task our objective was to understand how temperature & time affect the condensation process for our coating and to determine how well cured the coating is for temperature/time regimes that are commercially relevant. The method for this characterization was FTIR (Fourier Transmission Infra-Red Spectroscopy) which provides an absorption spectrum that detects the Si-OH group. The amount of absorption can be used on a relative basis to determine how "cured" the coating is; less absorption = less Si-OH = more cured and no absorption = fully cured.

Samples were prepared by applying an extra thick coating onto silicon wafers. This preparation methods is needed to first have enough material so we can detect a signal and second to have a contrast between the coating and the substrate.

As we gained more experience with short duration cure experiments we realized that there was considerable uncertainty about actual temperatures achieved by the samples for short cure times. Given the fairly gross methods we were using there was a lot of scope for error. We were concerned that the results we were obtaining for different cure times were in fact mostly due to different maximum temperatures achieved in the short duration of the experiment. We hypothesized that if temperature was more important than time then there should be a measurable rate of reaction vs temperature. This suggested to us that we needed a characterization approach that allowed for very precise control of temperature and time. The alternative method we chose was Thermo-Gravimetric Analysis (TGA) which measures mass loss versus temperature. Its main advantage is the very precise temperature/time control possible but its disadvantage is that it's an indirect measure of cure because it assumes that the mass loss measured is because water generated by condensation leaving the film.

Cure time/temp reduction

The objective of this task was to investigate synthesis and formulation modifications to the coating materials that makes the material easier to cure. We have investigated several possible methods to achieve this. As mentioned previously, 'cure' refers to the condensation of silanol (Si-OH) within the coating to form a silica cross-linked network (Si-O-Si). The most direct method of curing is just by heating the coating. This task sought to find ways by which the rate of condensation can be increased at a given temperature.

We tested several different additives and synthesis methods to achieve the objective of this task. At the close of the project we were able to achieve very fast cure time using an improved cure process method rather than through chemical additives. However we did identify some materials with very useful and unexpected effects that improve other properties of the coatings.

Task 3 – Economic Analysis Go/No Go

Objective

The objective of this task was to perform a detailed economic analysis of operating a glass coating line as designed in the US, then to present that analysis as a Go/No-Go review to decide to spend the capital for the project and start Phase 2. In this task we created a detailed total cost of ownership model for the full coating line as operated in a representative US location. We used these cost figures to build a business case for the line based upon current (as of the review) market conditions including domestic market demand and pricing of competing solutions.

Technical Accomplishments

This is based on much of the work completed in task 1 that provides reasonably accurate capital costs for the equipment and operational costs such as labor, energy and consumable usage costs.

Total Cost of Ownership Analysis

Completion of the major capital related sub-tasks under task 1 generated the capital inputs for the total cost of ownership model. Our manufacturing partner provided the other required information; the market price differential for AR coated and uncoated glass delivered from China suppliers to US module manufacturers; the logistics costs of shipping from China to the US and any other costs associated with the purchase of glass from China.

Over 2014-2015 we have observed that within the Chinese glass market the majority of glass is being supplied with AR coatings. This has had the effect of narrowing the price differential between coated and uncoated glass. Customers expect only coated glass, therefore the glass supplier has to supply coated glass or exit the market. This means there is no longer a two tier price for coated and uncoated; it has collapsed to a single price for coated glass that somewhat predictably is only slightly greater than what was charged for uncoated glass previously. However, this has not impacted the suppliers of coating materials to the glass companies (such as Enki Technology). The glass suppliers are not able to pass along the margin reduction to the coating suppliers and so have taken the hit themselves. We believe this is mainly due to the contrast between the high-tech nature of the coating that tends to lock-in module makers that select a specific coating versus the commodity nature of the base glass. The implication of this dynamic is that there is not a significant differential in price that would justify a standalone coating line in the US that is based upon importing base glass from China, assuming that US glass customers are commanding similar pricing from the Chinese glass companies that Chinese module customers enjoy and absent a US based solar glass supplier.

Go/No Go Review to gate Phase II

On September 22nd, 2015 we presented the results of the economic analysis to the Department of Energy. Our conclusion was a recommendation of “No-Go” based on the inability to show economic viability of a US based coating line. The key reason is that Chinese glass manufacturers are offering the AR coating at the cost of just the chemical (i.e. zero labor, facilities or capital costs). Therefore, even with a more productive and lower cost manufacturing line in the US, the costs of US coated glass are higher than similar glass imported from China. Unfortunately there are currently no domestic US glass manufacturers supplying the solar module market.

Patents and IP

Enki has a strong IP position related to sol-gel coating compositions and to processes related to deposition of those coatings on substrates and on solar modules in particular. During the course of the project we filed three patent applications based on work covered by the project.