*Subcontractor Report*

# Development of New Low-Cost, High-Performance, PV Module Encapsulant/Packaging Materials

## Annual Technical Report, Phase 1
## 22 October 2002–30 September 2003

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*Enfield, Connecticut*

**NREL**

**National Renewable Energy Laboratory**

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**NOTICE**

# PREFACE

The primary objectives of this subcontract are for STR to work with US-based PV module manufacturers representing crystalline silicon, polycrystalline silicon, amorphous silicon, copper indium diselenide (CIS), and other state of the art thin film technologies to develop formulations, production processes, prototype and qualify new low-cost, high-performance photovoltaic module encapsulants/packaging materials. The manufacturers will assist in the identification of each materials' deficiencies while undergoing development, then ultimately in the qualification of the final optimized materials designed to specifically meet their requirements. Upon completion of this program, new low-cost, high-performance, PV module encapsulant/packaging materials will be qualified, by one or more end-users, for their specific application. This document reports on progress toward these objectives and goals through approximately the first year of this three-year subcontract October 2002 through September 2005.

# SUMMARY

Information gathering on topics related to thin film module technology including device performance/failure analysis, glass stability, and device encapsulation have been completed. This information has provided concepts and considerations for module failure analysis, accelerated testing design, and encapsulation formulation strategy for thin film modules.

Interfacial characterization of thin film modules is still in process at the completion of the first year of the contract. Analytical methods have been developed for the individual interfacial components for thin film type modules both "as manufactured" and after field exposure or laboratory aging. The baseline analysis on the "as manufactured" modules is nearing completion and analysis of aged failed fielded modules is scheduled during the first several months of Phase II.

The pre-commercialization activities under this Phase I first year effort have included the formulation and extrusion optimization for the Super Fast Cure and Flame Retardant EVA based encapsulants designed for the crystalline type photovoltaic module market. Commercial module lamination trials, IEC qualification testing, and initiation of long term fielding at the STAR facility will not be completed until early in the second year of funding.

## INTRODUCTION

The work summarized herein focuses on three main objectives. The first of these objectives was to gather technical information on topics relevant to the scheduled Phase II encapsulant development work to be initiated under the second year of funding. The second was to gain an understanding of changes in interfacial chemistries within failed thin-film module technologies to hopefully gain insight into the surface chemistry of the interfacial regions of certain commercial modules. The third objective was to optimize both formulation and process of super fast-cure and the flame retardant formulations as a means of improving their processing performance during manufacture and the overall module performance.

The majority of the first year effort involved optimizing two high performance encapsulant materials that were previously formulated by Specialized Technology Resources Inc. under BP Solarex subcontract ZAX-8-17647-05 awarded by DOE/NREL (1998-2001). The first encapsulant was a faster-curing (super fast-cure) EVA based encapsulant and the second a flame-retardant (FR) formulation. Photovoltaic modules manufactured with the super fast-cure formulation could be laminated using a six (6) minute process[1,2]. The flame-retardant formulation allowed modules to meet the Class B flammability rating under UL 1703[1]. The Task 3 Effort under this Phase I contract addresses the optimizing effort of "faster-curing" and "flame-retardant" EVA based encapsulant systems with a focus on overcoming its experienced process shortcomings for manufacturing scale-up.

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**TASK 1: INFORMATION SEARCH FOR DEVELOPMENT OF THIN-FILM ENCAPSULANTS**

The objective of the Task 1 information search was to identify research or technology pertinent to encapsulation. The following summary will identify issues and practices appearing in the literature that may prove instrumental to the development of new and improved encapsulant/packaging materials for thin film devices. The information search was implemented by the following two surveys.

- Module Manufacturers Survey
- Literature Survey

Significant findings from each of the two surveys are organized by survey type, then information category.

**Module Manufacturer Survey**

STR made individual site visits and conducted interviews of the module manufacturer technical staff to understand the current reliability issues associated with the specific thin film technologies. BP Solar, Shell Solar, and Energy Photovoltaics, Inc. were participating team members. The visits contained an exchange in some cases of proprietary information. Following is a summary of the major unclassified findings of all of the team members on the following topics: Module Attributes, Encapsulant Attributes, Edge Deletion, Process, and Testing.

− Module Attributes

- Most thin film devices are sensitive to moisture exposure
- Most manufacturing processes avoid acid and water exposure
- Module efficiencies are reduced after exposure to 85°C/85%RH
- Special edge seals sometimes help to maintain device efficiency over the duration of qualification testing

− Encapsulant Attributes

- Encapsulant adhesion has not been characterized
- Desire low moisture vapor permeability
- Ideally would like low adhesion to device, high adhesion to glass
- Good electrical insulation
- Optical clarity required for some, not for all
- Thermoplastic OK – must have creep resistance at use temperature
- Upper process temperature is 200°C
- Process friendly – eliminate vacuum laminator or cure times

− Edge Deletion

- Required to isolate device from ground
- Sandblast and laser methods used
- Edge deleted surfaces air cleaned prior to laminating

- Avoid moisture or acidic substances during process

− Process

- Process highly proprietary to each manufacturer
- Device packaging represents approximately one half of module production costs
- Desire lower cost packaging
- Desire faster process through put
- Need to keep process temperatures below 200°C

− Testing

- High humidity produces reduction in module efficiency
- Failure mechanisms not well understood
- Glass corrosion appears to be a factor
- 85°C/85%RH testing sometime produces erroneous pass/fail information
- Better aging tests required but not yet defined

**Literature Survey**

A literature review was conducted to identify technical research related to thin film module reliability, conceptual ideas that may be used as a platform for new encapsulation technology, and materials that have potential as new encapsulants or additives of interest in the development of new encapsulants. The search strategy involved a series of approaches that are summarized and organized by Photovoltaic Industry, Patent, and General Literature search strategies.

Photovoltaic Search Strategy

The NREL, Sandia, IEEE, and JPL databases were searched for journal and conference proceeding articles describing research pertinent to the reliability and failure analysis of thin film modules. This search identified papers and presentations with respect to thin film module reliability and provided contacts for NREL and other government employed scientists involved in the study of thin film module reliability. Topics of interest included failure analysis of thin film modules, accelerated testing methods, and reduction of moisture ingression.

The topics covered by NREL publications indicate researchers Joe del Cueto, Gary Jorgensen, Tom McMahon, Carl Osterwald, and Joel Pankow as having conducted substantial research involving the reliability of thin film modules. They have been targeted as having special insight into the reliability issues pertaining to thin film modules, and will be utilized as resources going forward for the encapsulant development.

− Failure Analysis

Module reliability issues have been documented by studying thin film modules in system grids by following performance changes over time versus the Begin of Life (BOL) performance[3, 4, 5]. The mechanisms and environmental influences that cause module

performance degradation are poorly understood[6], but sodium migration from the soda lime glass, water vapor, and internal electric fields have all been implicated in the loss of module performance[7].

The NREL and Sandia literature review identified issues of glass instability in the thin film modules relative to the adhesion of the glass TCO layer and its contribution to the failure or efficiency of the device.[8] There is an overall emphasis on the role of moisture and adhesion difficulties between the glass and the encapsulant [9,10,11] as well as various module interfaces after damp heat exposure[12].

Leakage currents between the device and the glass and at the interface of the glass and the encapsulant appear to be the most significant factors with respect to the corrosion of the thin film modules[13]. Additionally, there are electrochemical and galvanic corrosion properties inherent in the devices themselves because of the dissimilar material conductors in contact with each other[14]. Contributors to electrochemical corrosion include sodium migration from the soda-lime glass, water vapor, and internal electric fields[15]. Sodium migration can be induced at 85°C when 100 volts are applied across $500cm^2$ of glass[16]. Water vapor greatly accelerates the damage rate, but its absence or near-absence does not completely eliminate the corrosion effect[17]. A high voltage test bed at Florida Solar Energy Center demonstrated that electrical leakage currents vary with ambient humidity[18].

A paper from Florida Solar Energy Center indicated that corrosion of a-Si:H PV modules has occurred in an array operating at 300 V DC in Orlando Florida as well as accelerated testing under high voltage bias in damp heat at NREL[19]. This FSEC work presented a general XPS analysis of EVA on the backing glass indicating the presence of tin, oxygen, silicon, carbon and fluorine at the glass encapsulant surface without mention of sodium. First Solar has indicated that their thin film array performance has been stable over a 5- year period with little incidence of delamination, edge corrosion, or arching problems[20].

Overall, the industry recognizes that damp heat should be a component of qualification testing since this type of testing exposure appears to create a failure response that is similar to that found in fielded modules[21]. However, there is indication in the literature that modules that have demonstrated field stability at NREL do not pass standard accelerated environmental tests[22]. An analysis performed by NREL has suggested that the 85°C/85%RH is unrealistic from the perspective of both temperature and humidity of fielded modules[23].

Other experiments indicate that when permeable back sheets were part of the module construction, peel strengths between glass and EVA were not appreciably affected until exposures of 800 hours where the moisture ingress occurs primarily through the back sheet when tested at 85°C/85% RH [24].

− Module Moisture Ingression

The literature talks about two main routes of moisture ingression into the module through the polymer back sheet or through the edge of the module[25]. For glass/glass type constructions, an edge seal has been used in addition to the pottant as a means of creating a moisture barrier for the module[26]. Using high moisture barrier encapsulants is one methodology of interest to module manufacturers for reducing moisture ingression and improving the reliability of thin film modules. Global Solar believes that the encapsulant's moisture vapor transmission rate needs to be $5 \times 10^{-3}$ $g/m^2/day$ at 25°C or less for the module to pass the current 1000-hour 85°C/85%RH qualification testing[27].

There has been much analysis of the moisture vapor permeability of EVA and other potential encapsulants[28,29]. The current commercial encapsulants do not adequately protect the module from moisture ingression and the encapsulation parameters appear to be unrelated to the water vapor transmission properties of the cured EVA[30].

With respect to potential moisture barrier encapsulation strategies, films are favored over coatings because of their inherent physical attributes including continuity, mechanical strength, adhesion, and cost[31]. A composite design for the encapsulant is desirable[32]. One view on moisture barrier technology is that it is improbable that industry is going to find a perfect polymer-based moisture barrier, and, therefore, cells, components, and the adhesion of the system must be made more tolerant to moisture[33].

Patent Search Strategy

The US patent literature was searched for patented technology and/or ideas on the following topics:

− Encapsulation and Glass Passivation

The patent search indicated technology for packaging LED's that may potentially cross-over to photovoltaic technology[34, 35]. Materials including vapor deposited silicon oxide, silicon nitride, spin-on glass, or spin-on polyimide are noted as being used to protect devices from moisture or other contaminants in addition to electron beam deposited glass[36, 37, 38, 39]. Many of the spin-on glass inventions appear to require fusion temperatures about 400°C.

− Moisture Vapor Barriers

Much of the patented innovations identified deal with synthetic routes for preparing higher moisture barrier resin[40, 41, 42, 43]. One patent described a composite material that may have utility in improving the moisture resistance of encapsulants not requiring optical properties[44]. There were no patents identified that addressed the strict moisture barrier properties that were indicated by the module manufacturers. However, it may be possible to use additives to improve barrier properties. For example, wax has been patented as a corrosion inhibitor in epoxy thermosetting compositions[45].

– Packaging, Food Packaging

The prevalent innovation with respect to food packaging appears to be with composite layered structures[46] and alloys[47, 48]. No significant patents were found pertaining to single polymeric material systems or filled composite structures.

– Coupling Agents

The patent literature discusses the utility of silicone oligomers as coupling agents and improving moisture resistance in coatings, curable adhesives and coatings[49]. Patent literature revealing chemistries that may be more hydrolytically stable than the current state of the art was not located.

General Literature Search Strategy

A general literature search was implemented via internet search engines, Infotrieve.com and Dialog.com for information relating to the following general topics:

– Organic Light Emitting Diodes (OLED), Light Emitting Diodes (LED)

The OLED industry is currently using glass ceramic films as conformal barriers on the electrical components of the devices to protect them from moisture[50]. One such invention that has been developed to prevent moisture exposure is to employ the Barix[TM] process of depositing metal or ceramic oxide layers to provide moisture barrier properties[51]. Another feature of some OLED constructions are desiccant layers that are isolated from the OLED device by a membrane[52].

– Glass

Literature was sought pertaining to soda lime glass chemistry and water transport of ions within the glass. One reference indicated that the XPS analysis can change the surface chemistry of soda lime glass and the amount of sodium detected increases continuously with time[53]. Borosilicate glasses appear to be much better suited with respect to their chemistry and electrical resistance than soda lime glass[54]. AFG Industries has improved the moisture sensitivity of its TCO glass product by reducing the sodium content of the glass and adding a thicker dielectric silica barrier layer [55]. AFG indicates that trapped moisture causes glass corrosion and recommends the following production parameters: glass- to- glass module structure, dry EVA, avoid lamination pinch out or other lamination stress, use acidic detergents to prepare glass for bonding, and to use an edge seal to prevent moisture ingression[56].

Sodium and phosphorus at the interface of the encapsulant and the glass has been found to correlate with a loss of adhesion strength in the encapsulant[57]. Methods of passivating the sodium in the glass were investigated through the literature. It appears that the electrical currents are able to mobilize sodium ions through passivating coatings including oxidized phosphorous and doped silicon dioxide lattices[58, 59] or silicon nitride/ silicon oxide barriers[60].

A laboratory technique has been developed by Energy Photovoltaics, Inc. wherein an electric field is applied to heated glass to test the TCO delamination[61]. According to this

reference, the sodium ions in the glass increased the amount of current that passed through the glass and subsequently was a factor in TCO delamination. The electrical biasing appeared to make the glass more susceptible to TCO delamination. Furthermore, the effects of voltage biasing lie dormant in the absence of moisture. When moisture is later introduced, the TCO begins to delaminate from the glass. The electrical current did not appear to have a similar effect on borosilicate glass having approximately 25% of the sodium of standard soda-lime glass.

– Electronic Devices

General literature on the failure mechanisms of electronic devices indicates ion contamination to be problematic. It has been documented that moisture and other contaminants can enter encapsulants both along the interface between the device leads and the plastic or through the bulk of the plastic [62]. The literature discusses the use of high purity components as well as passivation to protect electronic devices from ion migration. The microelectronic industry has experimented with utilizing inorganic glassy and/or organic polymeric protective insulation layers to provide mechanical and chemical protection of devices. The protective layers often have their own issues with respect to localized structural defects that rendered the metal vulnerable to corrosive attack. Trapped moisture and contaminants in cracks and pin- holes accelerated the corrosion mechanisms [63].

– Moisture Vapor Transmission, Moisture Barrier

A general survey was conducted to locate physical data relative to the moisture barrier properties of polymeric materials to identify candidate materials that might have superior moisture vapor barrier properties compared to EVA, butyl, and silicone rubbers. Literature and information was procured on True Seal PIB insulating glass sealant for comparison with properties of EVA or other alternative encapsulant materials. The PIB is used as a main moisture vapor seal between the glass and metal spacer in insulating glass units[64]. Data was sought to determine the relative moisture vapor transmission rates of candidate polymers for the encapsulant development. A number of sources provided limited data on generic materials[65, 66, 67, 68].

Water vapor barriers are of interest to the pharmaceutical blister packaging industry. The best current moisture-barrier film is PTCFE which has a water vapor transmission rate of 0.03 at 100°F and 90% RH[69].

– Encapsulants

There has been some work in crystalline silicon modules with an alternative encapsulant that does not require vacuum lamination to process[70]. Other European manufacturers are interested in using PVB as thin film module encapsulants[71].

The literature search did not locate documents that define the optimum encapsulant requirements with respect to adhesion strength, dielectric properties, elongation, modulus, or coefficient of linear thermal expansion. However, a calcium test has been used in the OLED industry to accelerate screening of potential encapsulants[72].

**TASK 2: INTERFACIAL CHARACTERIZATION OF ENCAPSULANT BONDING**

Failure mechanisms in current thin film devices are often attributed throughout the industry to moisture penetration into the module through the bulk of the encapsulant. The emphasis of Task 2 was to characterize the interfacial regions of thin film devices, both new and fielded, to develop a better understanding of the interfacial surface chemistry and to look for changes that may impact adhesion of the encapsulant to the various device substrates. The interfacial regions of the edge delete perimeter of thin film modules are of primary interest in this analysis since the commercial grades of EVA encapsulants have exhibited lower adhesion within the edge-deleted regions. One major objective of this research was to determine the role of the edge-deleted region in moisture transport into the thin film module and subsequent corrosion of the thin film device.

The instrumented analysis was performed under subcontract by the University of Connecticut Institute of Materials Science (UCONN-IMS) as a means of better understanding the relationship between the encapsulant and device reliability. Early stages of this effort focused on developing meaningful analytical test procedures to be used in the module characterization. The procedures were established on individual component specimens supplied by the manufacturing team member participants. The procedures were then used in the baseline analysis of unaged thin film devices and will be later used to analyze aged failed thin film modules. Concurrent work was also performed by STR to assist in development of the testing procedures and also to develop and validate potential accelerated aging protocols that will be utilized under the Phase II, "Task 4- Thin-film Encapsulants- Initial Development."

The conclusions based on the UCONN-IMS literature study have mirrored the industry belief that moisture is a large contributor to the premature failure of the thin film devices. UCONN-IMS postulates the following probable failure mechanism:

1. Water migrates through EVA to glass/EVA interface.
2. Water rapidly degrades soda-lime glass surface (both the EVA and glass corrosion product also probably swell) leading to delamination.
3. Water must be prevented from reaching the EVA/glass interface.

The UCONN-IMS analysis of unaged interfaces is nearing completion at this end of the first year funding. Techniques utilized in this evaluation are X-ray photoelectron spectroscopy (XPS), scanning electron microscopy coupled with energy dispersive X-ray analysis (SEM/EDX), and optical profilometry. Major findings and conclusions are still pending at this time (to be addressed in contract deliverable D-1.5 target date October 1, 2003).

Modules available to the UCONN-IMS analysis of failed fielded modules so far include one failed a-Silicon module supplied by BP Solar as well as four purchased fielded modules from Arizona State University. NREL is in the process of securing examples of modules from PowerLight Corporation. The objective of this analysis will be to identify interfacial chemistries at both visible defects and other regions of the fielded modules.

The analysis will attempt to compare the interfacial regions of fielded failed modules with those that have no prior fielding history.

Additional characterization of physical properties is scheduled to develop a better understanding of moisture ingression through the EVA encapsulant and its effect on adhesive strength and interfacial chemistry.  A testing protocol involving Karl Fisher determination of moisture concentration relative to adhesive strength, migration into the module, and interfacial chemistry is in design stage for this purpose for implementation in mid October.  Additional activities involve the design and validation of accelerated aging protocols for candidate encapsulant materials.  One such proposed method for screening encapsulants is to involve applying a voltage bias to test coupons before exposure to high temperature and humidity.  The interfacial chemistry of specimens aged in this manner will be validated by the testing protocol developed by UCONN-IMS based on their findings on aged failed modules.

Indications

The major indications at this point in the Task 2 analysis is that soda lime glass that has been utilized in the fabrication of thin film devices appears poorly suited to fabrication of photovoltaic modules from the perspective that the it is not especially stable when exposed to voltage and moisture.  The literature indicated a migration of sodium ions to the TCO surface of the modules and, inevitably, delamination of the TCO layer from the glass.  This problem appears to be delayed if moisture can be eliminated at the surface of the glass.

Given the instability in the glass, it appears that enhancing EVA adhesion to the glass likely will not play a substantial role in improving the reliability of the photovoltaic modules.  It appears that the moisture vapor transmission through the bulk encapsulant must be minimized in order to extend the service life of the modules.

**TASK 3: MANUFACTURING SCALE-UP OF EVA BASED ENCAPSLANT SYSTEMS**

Super Fast Cure (SFC) and Flame Retardant (FR EVA) encapsulants were optimized as a means of improving observable process shortcomings noted from their initial extrusion trials.  The SFC encapsulant required additional formulation optimization to correct a cumbersome, time intensive compounding procedure, screw slippage, premature crosslinking during extrusion, and failure of the IEC 61215 qualification test for wet hi-potential.  The FR EVA required optimization to correct its unsatisfactory level of crosslinking and to substitute a non halogen-based additive for its current halogen-based additive.  Halogenated chemicals are considered environmental risks, particularly in Europe where a non-halogenated FR formulation may prove to be more acceptable and practical.

**Experimental Design**

Based on the nature and make-up of the curative package in the SFC formulation, it appears that one of the curative's constituents was responsible for the observed screw slippage.  It was surmised further that this same constituent was part of the mechanism

that reduced the encapsulant's ability to prevent current leakage during the wet hi-pot evaluation. Therefore, reducing and/or minimizing this specific curative additive was the goal, while maintaining the fast and efficient curing properties of the overall curative package.

Moving Die Rheometry (MDR)

MDR, a standard test method that analyzes crosslinking efficiency in rubber and plastic materials, was used to measure curing kinetics to access the impact of modifications to curing packages or evaluate new curing systems. The rheometer measures the shear modulus (i.e. torque) as a function of time at a set temperature during the crosslinking of a polymer. Torque development is a function of crosslink density; as the polymer crosslinks, the resistance to shear (measured as torque) increases. All MDR measurements were carried out in accordance with ASTM D 6204-97.

Mooney Scorch Analysis

The Mooney viscometer is an instrument that compares the processability and stability characteristics of polymer materials. The viscosity is measured as a function of time at constant temperature. Like MDR, the viscosity is related to the curing activity of the polymer. A material that has a strong tendency to crosslink prematurely (i.e. scorch) during processing will have a higher Mooney viscosity per unit of time compared to a material that is not subject to scorching. Mooney Scorch analysis was performed at 104°C.

Mixing Bowl

The mixing bowl measures crosslinking level at constant temperature like the Mooney viscometer, but a shear component is introduced that simulates more accurately the conditions within an extruder. Evaluations with the mixing bowl were accomplished using a Brabender Plasticorder/Rheometer fitted with an oil-heated mixing bowl and high-shear blade mixers. The bowl was maintained at three temperatures, 99°C. 103°C, and 107°C, and formulations were sheared at 100 rpm.

Strand-die Extrusion

A single-screw pilot extruder with 1/16- inch diameter strand-die was used to simulate production processing of each of the experimental compositions. The extrusions were run at a 107°C melt temperature and 3000-PSI backpressure so that the effect of flow restrictions on stability of the experimental compositions could be evaluated.

Flame Impingement

To evaluate the flame-retarding and flammability performance of candidate FR EVA formulations, a test method was modeled after 16CFR Part 1610. Experimental materials were exposed directly to a butane flame at 1,895°C for 15 minutes. During flame impingement, the material was observed for burning, charring, and/or dripping characteristics, as well as the ability of the flame to penetrate through the sample material.

## Results and Discussion

Super Fast-Cure Formulation:  Formulation Development

To address the issues with this formulation, the overall liquid content in the curative package was systematically reduced and the curing kinetics measured.  Figures 1 and 2 show MDR curves for various SFC formulations as a function of liquid content at 150°C, 155°C, and 160°C.  The maximum torque achieved (S'$_{Max}$), which is directly proportional to the crosslinking efficiency, decreased with the reduction of liquid content.



**Figure 1.  MDR Curves for SFC Formulations and 15295P at A) 150°C and B) 155°C**
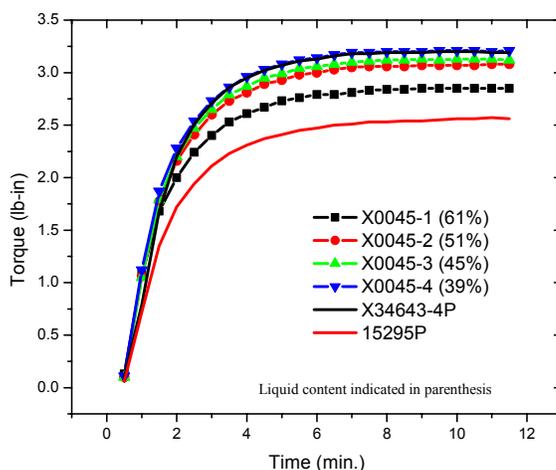


**Figure 2. MDR Curves for SFC Formulations and 15295P at 160°C**

Table 1 compares the peak curing rates of the various SFC formulations to the original SFC encapsulant, X34643-4P, and 15295P.  The SFC reformulations had slightly

greater curing rates than the original SFC material.  Out of the four SFC reformulations, X0045-4 (39% liquid content) had the most similar curing kinetics to X34643-4P.

**Table 1.  Peak Curing Rates (S'/min.) as a Function of Temperature for SFC Formulations.**

|        | X34643P | 15295P | X0045-1 |
|--------|---------|--------|---------|
| 150°C  | 0.99    | 0.8    | 1.03    |
| 155°C  | 1.43    | 1.22   | 1.55    |
| 160°C  | 2.09    | 1.77   | 2.25    |

|        | X0045-2 | X0045-3 | X0045-4 |
|--------|---------|---------|---------|
| 150°C  | 1.06    | 1.06    | 1.06    |
| 155°C  | 1.61    | 1.56    | 1.59    |
| 160°C  | 2.38    | 2.37    | 2.52    |

Additional reformulation of the SFC was done following the MDR results on the 0045 formulation series.  Figures 3 and 4 show MDR curves of the 122-61 SFC series as a function of LC at 150°C, 155°C, and 160°C.  Based on the reformulated SFC formulation series (0045 and 122-61), a 39% liquid content appears to be the optimum level for maximizing curing performance and minimizing liquid content.
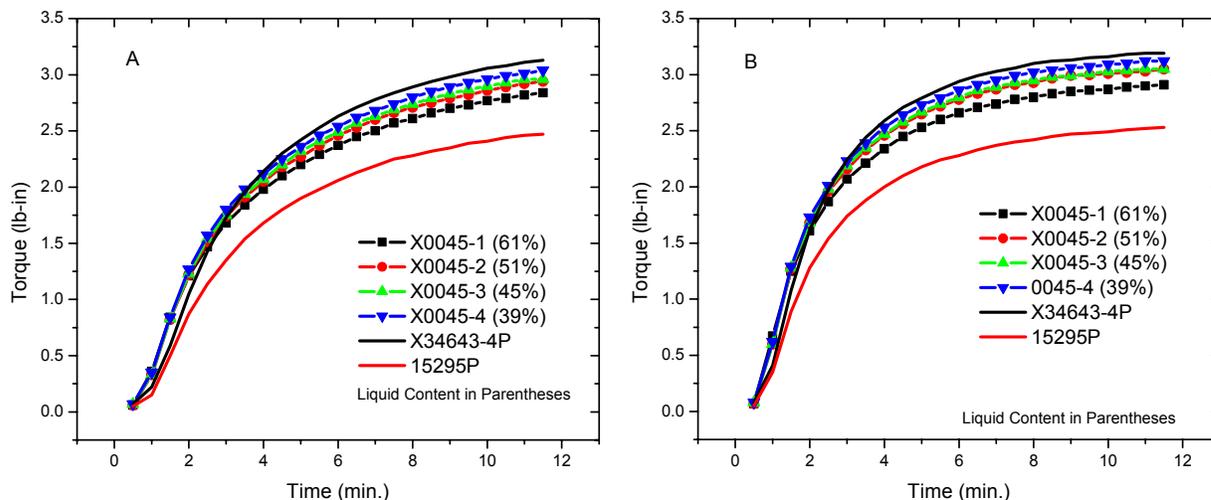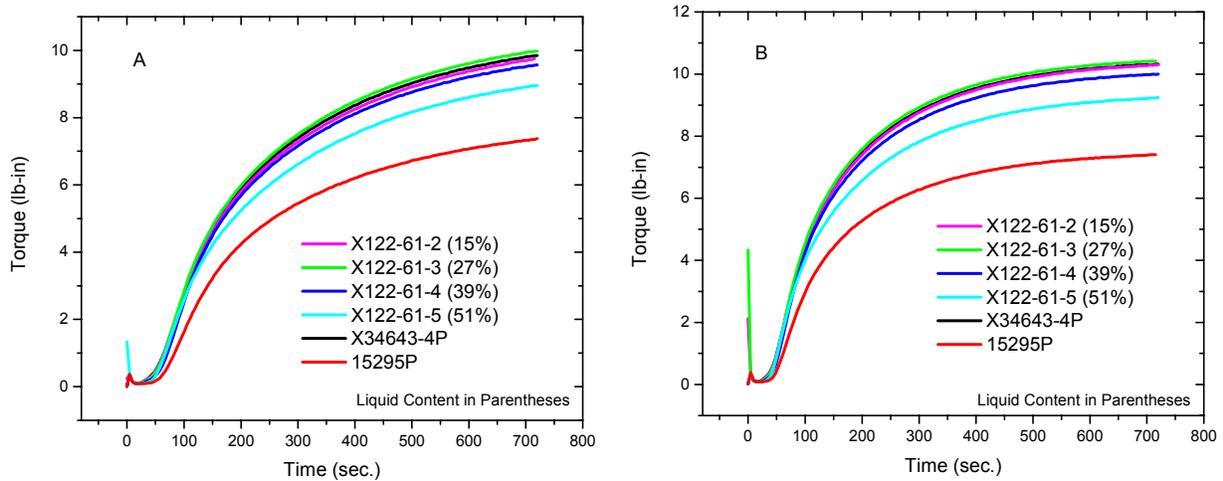


**Figure 3.  MDR Curves for SFC Formulations and 15295P at A) 150°C and B) 155°C**
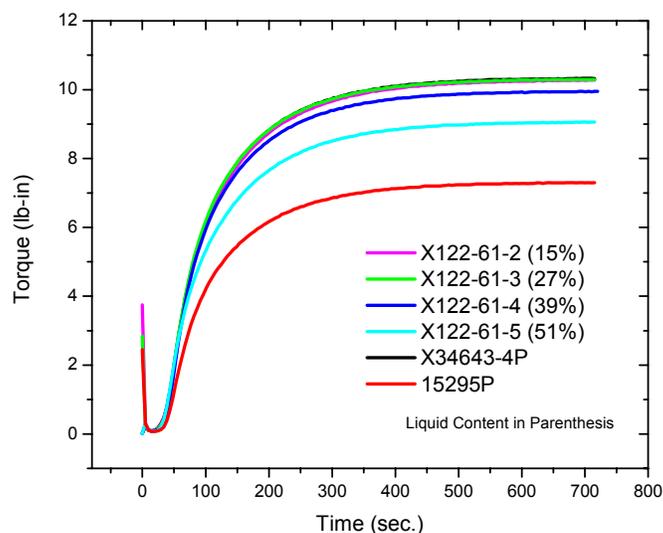
**Figure 4.  MDR curves for SFC formulations and 15295P at 160°C**

X0045-4, X34643-4P, and 15295P were evaluated with the Mooney viscometer in order to compare the processability of each material.  Figure 5 shows the Mooney Scorch data for all three formulations at 104°C.  The data surprisingly showed that both SFC formulations had similar process stability compared to the control 15295P formulation, although scorching was noted to occur with the original SFC formulation during its extrusion trials.
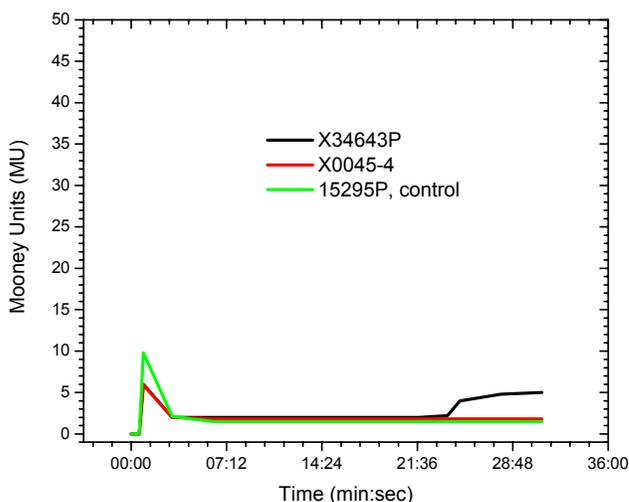


**Figure 5.  Mooney Scorch data for SFC formulations and 15295P**

To follow-up the Mooney Scorch analysis, a mixing bowl experiment was performed on 15295P and both SFC materials.  Because process stability was the focus of this experiment, the critical measurement from the mixing bowl study was the time it takes for the material to initiate crosslinking.

Three formulations were evaluated: a control, the original SFC formulation (-4P), and the reformulated SFC formulation (-XP). Table 2 summarizes the results of the mixing bowl experiment. For 15295P, X34643-4P, and X0045-4, crosslink initiation times were 12.5, 7.5, and 8.5 minutes, respectively. Including the shear component demonstrated a more pronounced difference between the formulations. As expected, the reformulated SFC formulation was slightly more stable (i.e. longer time to initiate crosslinking) compared to the original SFC formulation.

**Table 2.  Summary of results from mixing bowl experiment.**

|  | 99 C bowl temp. | | | 103 C bowl temp. | | | 107 C bowl temp. | | |
|---|---|---|---|---|---|---|---|---|---|
| Run | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| Formulation | Control | -4P | -XP | Control | -4P | -XP | Control | -4P | -XP |
| Minimum Torque | 800 | 900 | 900 | 900 | 900 | 900 | 900 | 850 | 850 |
| Time at min. torque (min.) | 11 | 10 | 9 | 7 | 6 | 6 | 5 | 5 | 5 |
| Crosslink Torque | 1500 | 1500 | 1500 | 1200 | 1200 | 1200 | 1200 | 1200 | 1200 |
| Crosslink Temp (C) | 137 | 126 | 128 | 127 | 122 | 124 | 128 | 123 | 126 |
| Crosslink Time (sec.) | 1140 | 810 | 870 | 750 | 450 | 510 | 570 | 360 | 390 |

The maximum material dwell time in an encapsulant production extruder was determined to be five (5) minutes. Both SFC formulations had characteristic times (crosslink initiation times) greater than the residence time, suggesting that the SFC formulations should not crosslink during extrusion. Therefore, it appears that material scorching is dependent more on the flow dynamics and operating conditions of the extruder rather than the material formulation.

Super Fast-Cure Formulation: Extrusion Optimization

Extrusion optimization trials were first conducted on the pilot scale using a pilot extruder and strand die to simulate the flow dynamics of the manufacturing extruder. This pilot test was used to compare the original super fast-cure formulation with the new optimized compositions.

The pilot test proved much less severe than the full- scale production, and was not able to fully reproduce the premature crosslinking that was seen with the same composition on the production equipment. These results obviated the need for the extrusion trials of all viable candidates to be performed on manufacturing scale and strongly indicated that it may be necessary to make modifications to the design of the process equipment.

Further experimentation indicated that the extruder design required modification to successfully process the SFC formulations.  The extruder barrel was optimized, and a proprietary screw was designed based on EVA flow dynamics.  These modifications resulted in significant extrusion performance gains.  STR observed at least a 7% decrease in overall extrusion temperatures and a 30% increase in material output compared to pre-modified process conditions.

Two SFC reformulations were extruded on the modified process: X0045-4/122-61-4 and X122-61-3.  Both materials ran extremely well during the extrusion trial; the sheet form and overall encapsulant quality were excellent.  Product from this trial appeared to laminate and cure effectively.

During extrusion of the original SFC formulation (March 2000 and October 2000), the edges of the encapsulant were wavy; this flow instability was observed within the first 0.375 inches from the edge of the extruded sheet.  This edge waviness was not observed for either of the reformulated SFC formulations on the modified extruder.  Furthermore, sheet thickness was easily kept constant throughout the trial, unlike the run on the previous manufacturing equipment.

There was no observation of screw slippage during the extrusion trial.  Melt pressure remained at expected levels, and when the screw was removed from the barrel for cleaning, there were no pellet deposits and molten resin buildup on the screw.  During the 2000 extrusion trials of the original SFC formulation, STR's production personnel noted deposits on the root and trailing flights of the screw.

Production output (measured in pounds of encapsulant extruded per hour) on the improved SFC formulations was increased by 91% over the previous formulation.  The near doubling of production output with the current process/formulation is about 80% of STR's normal production output with typical EVA-based formulations and is considered satisfactory for production scale.

Faster extrusion rates without affect on the encapsulant were accomplished, in part, by lower temperature profiles.  Die and melt temperatures were 11% and 3% lower, respectively, compared to the previous extrusion conditions with the original SFC formulation.

The temperature profile of the 2003 extrusion trial was also lower, on average, than typical production conditions; die temperatures were approximately 18% lower and melt temperature was 7% lower.  The reduced temperatures still did not affect extruding the SFC formulations into sheet form or the overall encapsulant quality.  Therefore, there appears to be a lot of room to increase the production rate of the SFC formulations with the current extruder setup.

Flame-Retardant Formulation:  Formulation Development

The first generation FR EVA encapsulants did not meet the crosslink density requirements for cured photovoltaic modules.  Central to this reformulation effort was a

new curing system capable of producing an effective crosslink density with similar or better curing performance compared to the STR fast-cure formulation 15295P.

MDR data in Table 3 and Figures 6 and 7 compares 15295P and the original FR formulation (X33579P-FR) to two (2) experimental FR formulations (X122-11-1P and X122-11-2P).  The experimental FR formulations had more robust curing performance (faster cure speed and higher $S'_{Max}$) versus the other controls.  In particular, the curative package in X122-11-2P greatly improved the crosslinking efficiency, resulting in higher $S'_{Max}$ compared to 15295P and the SFC materials.  Although the curing kinetics and efficiencies were improved in both FR encapsulants, these materials appeared to have adequate processing stability under Mooney Scorch (Figure. 8).

**Table 3.  Peak curing rates (S'/min.) as a function of temperature for FR EVA formulations.**

|        | 15295P | X33579 P-FR | X122-11-1P-FR | X122-11-2P-FR |
|--------|--------|-------------|---------------|---------------|
| 150°C  | 0.8    | 0.53        | 0.65          | 1.11          |
| 155°C  | 1.22   | 1.39        | 0.85          | 1.34          |
| 160°C  | 1.77   | 1.37        | 1.50          | 2.80          |

A non-halogenated FR EVA formulation was evaluated for its flame-retardant effectiveness against the original, halogenated formulation.   Each formulation was laminated to 15295P and then cured prior to flammability testing.  Table 4 summarizes the results of the flame test.   Note that the sample type/orientation describes the laminate and the layer that is first in contact with the flame.  For example, EVA is in contact with the flame, followed by FR-EVA, and scrim for run #1.

When the flame impinged first on the 15295P side, it took less than one (1) minute for the flame to burn through the non-FR layer.  The flame and subsequent combustion appeared to increase in intensity 30 seconds into the start of the test, likely due to cleavage of the highly flammable acetate groups.

Once 15295P was effectively burned off, the flame impingement contacted the FR EVA layer.   However, for both the halogenated and non-halogenated formulations, the penetration of the flame appeared to stop at this layer.  During the rest of the 15-minute exposure period, neither FR formulations burned, nor fueled the butane flame as the 15295P did.   When FR EVA was exposed first to the flame (i.e. impingement first started at the FR EVA layer), the FR layer did not burn, and because the flame could not penetrate this layer, the regular EVA layer underneath only darkened but did not ignite.
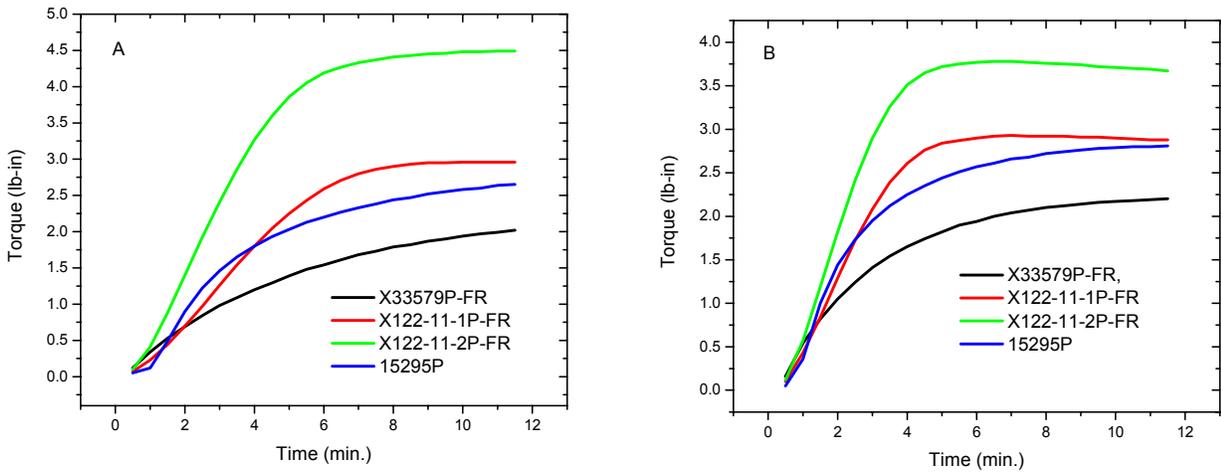
**Figure 6.  MDR Curves for FR EVA Formulations and 15295P at A) 150°C and B) 155°C**
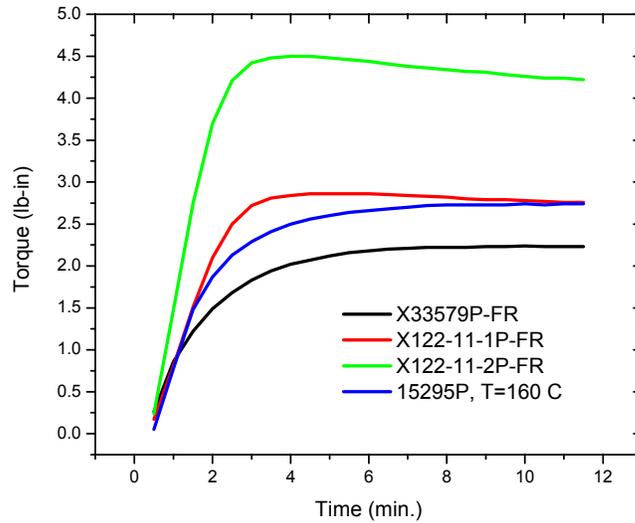


**Figure 7.  MDR Curves for FR EVA Formulations and 15295P at 160°C.**

Depending on the laminate layer exposed to the flame first, the burning patterns on the laminate were nearly identical for both formulations.  Figures 9 and 10 show the burning patterns of each FR formulation.  The similarity in how the material managed the flame impingement illustrated the flame-retardant effectiveness of the non-halogenated formulation.

**Figure 8. Mooney Scorch Data for FR EVA Formulations and 15295P.**



FRONT                    BACK

**Figure 9. Flame-Impinged Samples of Non-Halogenated FR Formulation.**



FRONT                    BACK

**Figure 10. Flame-Impinged Samples of Halogenated FR Formulation.**

**Table 4. Summary of Results from Flame Impingement Experiment.**

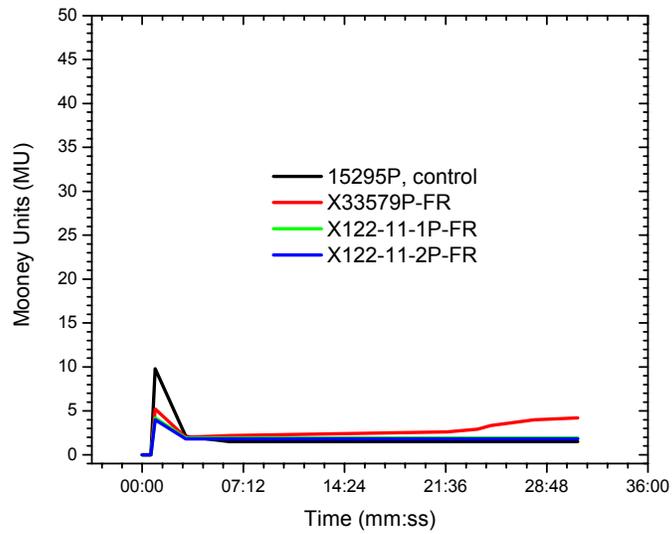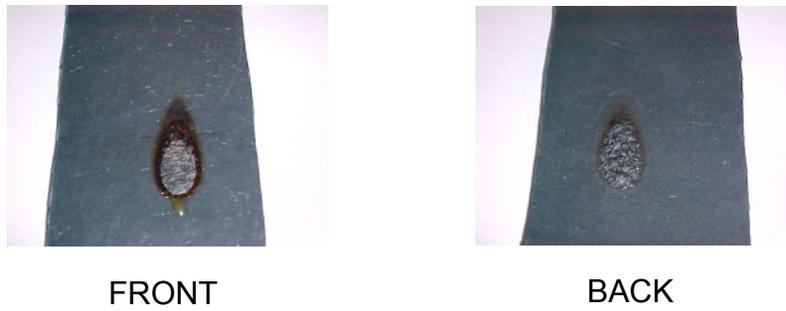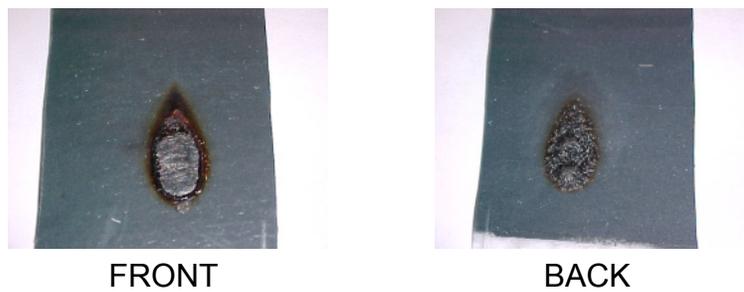| Run | Sample Type/Orientation[1] | Burn-through (yes/no) | Comments |
|---|---|---|---|
| 1 | EVA/FR-EVA/S-layer | N | <ul><li>After 5 min., no burn-through or general combustion.</li><li>Still good after 10 min.</li><li>Looks good after 15 min.</li></ul> |
| 2 | EVA/FR-EVA/S-layer | N | <ul><li>Repeat of run #1.</li><li>Initial burning of top EVA layer – snap, crackle, pop, smoke – then ceases.</li><li>No burn-through after 5, 10, 15 min.</li><li>Flame seems to be hitting a "wall".</li></ul> |
| 3 | EVA/FR-EVA (non-halogenated)/S-layer | N | <ul><li>After 5 min., no burn-through, similar to regular FR-EVA.</li><li>No combustion due to cleavage of acetate groups.</li><li>Same performance as runs 1 and 2.</li></ul> |
| 4 | EVA/FR-EVA (non-halogenated)/S-layer | N | <ul><li>Same performance as run #3.</li></ul> |
| 5 | FR-EVA/EVA | N | <ul><li>Flame stuck at the FR-EVA layer; no burn-through after 3:15.</li><li>Much more puckering behind the laminated compared to previous runs.</li><li>No apparent burn-through after 10 min.</li></ul> |
| 6 | EVA/FR-EVA | Y | <ul><li>FR-EVA layer fell apart that led to burn-through.</li></ul> |
| 7 | FR-EVA/S-layer/EVA | N | <ul><li>After 1.5 min., flame is stalled at the facing encapsulant layer.</li></ul> |
| 8 | EVA/S-layer/FR-EVA | N | <ul><li>Initial bubbling and burning of top EVA layer, then stopped.</li><li>Flame penetration stopped.</li></ul> |
| 9 | EVA/S-layer | Y | <ul><li>Vicious burning – growth of flame.</li><li>EVA is no more; scrim left.</li></ul> |
| 10 | FR-EVA (non-halogenated)/S-layer/EVA | N | <ul><li>Looking good after 10 min,</li><li>Did fine.</li></ul> |
| 11 | EVA/S-layer/FR-EVA (non-halogenated) | N | <ul><li>Top EVA layer burned fast.</li><li>A drop of liquid formed during combustion.</li><li>Localized burning – sizzling and boiling.</li><li>No burn-through.</li></ul> |
| 12 | FR-EVA (non-halogenated)/EVA | N | <ul><li>Flame stuck at the FR-EVA layer.</li><li>No burn-through.</li></ul> |
| 13 | EVA/FR-EVA (non-halogenated) | N | <ul><li>First 5 min., burning of the EVA layer.</li><li>Flame and burning stopped at FR-EVA layer.</li><li>No burn-through.</li></ul> |

## Flame Retardant Formulation:  Extrusion Optimization

The FR EVA formulation X122-11-2P-FR (non-halogenated) was extruded as part of the overall extrusion trial conducted with the optimized production scale extruder setup. The FR EVA formulation ran extremely well during the extrusion trial without complication from the optimized curing performance.  No problems were observed during processing relative to compounding, screw slippage, or premature crosslinking. The reformulated FR encapsulant was produced at typical production process speeds.

STR's next step with the SFC and FR EVA formulations will be module lamination trials with the encapsulant formulations.  Modules made with the SFC materials will be submitted for IEC 61215 qualification testing and fielded on a tracker for continuous monitoring.  Modules with the FR EVA encapsulant will first undergo flammability testing.

## CONCLUSIONS

Excellent progress has been made towards qualifying two new encapsulants for the crystalline type modules and for understanding the current industry findings, and technical issues surrounding the thin film modules technology:

- Obtained insight into the technical issues, concerns, and obstacles faced by each module- manufacturing team member in improving the reliability of their thin film modules

- Evaluated research conducted within the thin film module manufacturing industry and by national team members to better understand where additional analysis or further understanding may be required

- Identified potential formulation strategies for new high performance encapsulants

- Developed analytical procedures for interfacial characterization of failed thin film modules

- Scheduled interfacial characterization of failed thin film modules

- Improved the extrusion performance of Super-Fast Cure and Flame Retardant EVA encapsulants

- Scheduled commercial lamination trials and subsequent IEC qualification testing for the new improved Super-Fast Cure and Flame Retardant EVA

## FUTURE WORK

The Task 2 interfacial analysis work will continue into the second year of funding. The analysis will include aged fielded modules that have experienced substantial loss in efficiency.

The Task 3 manufacturing scale up work will continue into the second year of funding so that commercial module lamination trials may be completed. Modules made with the SFC materials will be submitted for IEC 61215 qualification testing and fielded on a tracker for continuous monitoring. Modules with the FR EVA encapsulant will first undergo large- scale flammability testing before qualification testing, assuming they successfully pass flammability testing.

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