FINAL TECHNICAL REPORT

Project Title: **Ultra Barrier Topsheet (UBT) Film**

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I.) Executive Summary

The lack of availability of low cost, highly reliable solar ultra-barrier top sheets (UBT) has hindered the progress of flexible solar module manufacturers. There is significant unmet demand by both module manufacturers and end-users that stand to benefit from the availability of high efficiency and low cost flexible solar modules. No company has been successful in commercializing such technology.

Compared with glass-on-glass modules, flexible modules, constructed with the 3M UBT, have the following advantages:

- Lower Balance of System costs → less labor and no racking needed for installations as modules can be attached or adhered directly to the roof.
- Light weight →1/10th compared with glass-on-glass
- Higher packing density → 5X the kW per ocean or trucking container
- Higher energy output → better transmission and off-angle performance
- Large area modules → 2 meter wide with no limit on length
- Lower manufacturing cost → Fully automated roll to roll processing instead of batch lamination and assembly

Today's best commercially available barrier films have WVTR (water vapor transmission rates) of approximately 10^{-2} g/m^2 day @ 23C. To achieve 25 year lifetimes, however, the requirement is estimated to be 10^{-4} to 10^{-5} g/m^2 day @ 23C for CIGS and CdTe and 10^{-4} to 10^{-6} g/m^2 day @ 23C for OPV and DSSC [1]. There is, therefore, an obvious need that current barrier films do not meet. Addressing this gap with 3M's UBT will therefore achieve a significant near-term impact on the photovoltaic industry.

In this DOE sponsored program, 3M attacked and achieved the critical UBT features to enable durable flexible high efficiency modules to be produced by a range of customers who have now certified the 3M UBT and are actively developing said flexible modules.

The specific objectives and accomplishments of the work under this proposal were:

1. Scale-up the current Generation-1 UBT from 12" width, as made on 3M's pilot line, to 1+meter width full-scale manufacturing, while maintaining baseline performance metrics (see table below).
   - This objective was fully met.

2. Validate service life of Generation-1 UBT for the 25+ year lifetime demanded by the photovoltaic market.
   - Aggressive testing revealed potential failure modes in the Gen 1 UBT. Deficiencies were identified and corrective action taken in the Gen 2 UBT.

3. Develop a Generation-2 UBT on the pilot line, targeting improved performance relative to baseline, including higher %T (percent transmission), lower water vapor transmission rate (WVTR) with targets based on what the technology needs for 25 year lifetime, proven lifetime of 25 years in solar module construction in the field, and lower cost.
   - Testing of UBT Gen 2 under a wide range of conditions presented in this report failed to reveal any failure mode. Therefore UBT Gen 2 is known to be highly durable. 3M will continue to test towards statistically validating a 25 year lifetime under 3M funding.

4. Transfer Generation-2 UBT from the pilot line to the full-scale manufacturing line within three years.
    - This objective was fully met.

II.) Results and Discussion

The pre-program status at 3M and the program goals achieved are presented below. In this final report we will discuss how the goals were addressed.

Top-Sheet Property	Current	Final
WVTR(g/m^2day)	10^{-3} – 10^{-4}	As low as 10^{-6}
Transmission	~90%	As high as 94%
Production Scale	Pilot-line at 12inch wide	Manufacturing line > 1 m wide
Customer Feedback	Initial testing positive	Certified Component
Product Lifetime	Uknown	Validate 25 year
Cost	~$80/m^2	<$15/m^2

At program initiation, 3M was able to produce Gen. 1 UBT on a narrow web pilot line. Success required that the property and cost targets be met at wide width and high volumes on a production line Production line. A full scale line had been designed, funded and was under construction in 3M's Columbia, Missouri facility.

The UBT fabrication process consists of vacuum deposition of a sequence of polymer and oxide layers on a polyester film substrate in a roll to roll process. Following this attachment to an ETFE top film is made using an adhesive layer. Ultra low defect and pin-hole levels and precise layer thickness and composition were required to achieve the required WVTR levels and optical transmission. High line rates and yields were essential to achieve cost targets. The production line was designed to achieve these requirements based on experience from our pilot coaters and drawing from the best technologies industry had to offer. Process migration from narrow web to high speed wide web coating required excellent engineering as well as invention in several areas. In addition, developing a long life UBT required an aggressive accelerated testing protocol and UBT modifications to correct deficiencies. Results will be presented by initial program goals. In the following sections, 3M's activities and results relative to each program goal in the above table will be presented.

Production Scale - Manufacturing line > 1m wide

Process scale up work was transferred from 3M's ultra barrier pilot coater to 3M's ultra barrier production coater at the end of Q4, 2011. After installing the production coater in Columbia, Missouri, preliminary experiments combining the polymer deposition and oxide deposition were carried out successfully. A 1.2 m wide x 1000 m long roll was coated and initial test results for WVTR, thickness, %T transmission met initial coater and UBT specifications. UBT qualification trials were run using Generation 1 UBT made on the pilot line and the production coater and the key metrics, e.g. WVTR and %T were monitored as a function of time in controlled environmental chambers. Initial data showed equivalency or better performance for the production coater relative to the pilot line.

Production line material met or exceeded 3M's ultra barrier pilot line coater performance when evaluated via light transmission and layer thickness (Figure 1), and WVTR (Figure 2). Process capability data was collected with respect to critical process parameters such as layer thickness and %T with all resulting Cpk's and Ppk's greater than 1. All effort transferring Gen 1 UBT from the pilot coater to the Production coater concluded successfully under this contract.

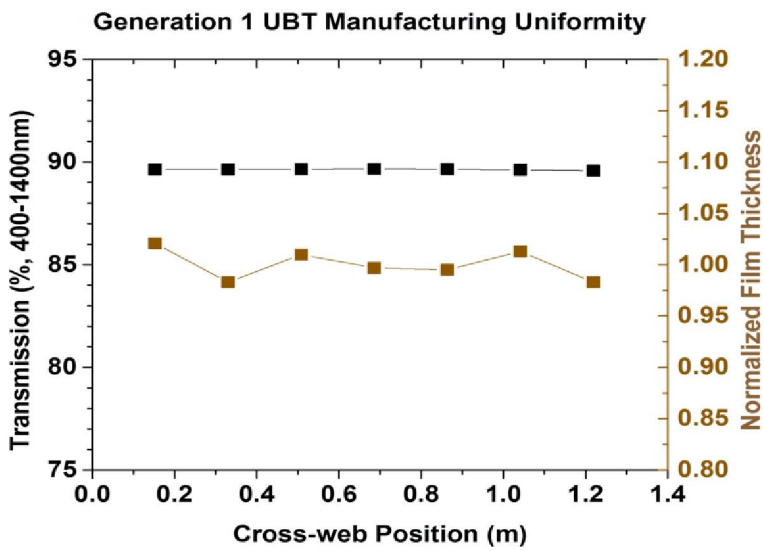


Figure 1.) Wide-web coater cross web UBT optical transmission and film thickness

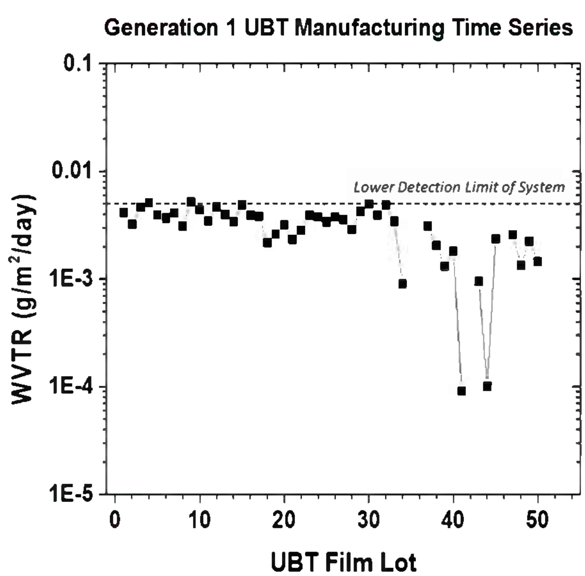


Figure 2.) Wide-web coater WVTR measured at 50°C

WVTR (g/m^2/day): As low as 10^{-6} (g/m^2/day)

3M's Generation 1 UBT and Generation 2 UBT have both demonstrated a Water Vapor Transmission Rate (WVTR) of less than 5 x 10^{-4} g/m^2/day at 23°C as measured by using a MOCON® Permatran® 700 at 50°C and 100% RH. However, validating WVTR measurements below 10^{-3} g/m^2/day is difficult. The best determinant to ensure that the WVTR performance is adequate is to test the actual film in a solar module construction via 85oC/85% RH Damp Heat testing. Multiple solar modules utilizing 3M's Generation 1 and Generation 2 UBT have far exceeded the 1,000 hours testing requirement outlined in IEC 61646. Typically, ultra barrier topsheets are screened for WVTR using a MOCON® Permatran® 700 at 50°C and 100% RH as previously mentioned. The test is allowed to run until the standard deviation of the last 5 readings is less than 0.0025. The samples are evaluated for a pass/fail at the machine detection limit of 0.007 g/m^2/day. 3M's ultra barrier topsheets show an Arrhenius relationship with regard to WVTR performance. Thus, WVTR approximately doubles for every 10 °C increase in temperature. Applying such an Arrenius relation, 0.007 g/m^2/day at 50°C is approximately .0011 g/m^2/day at 23°C.

The National Renewable Energy Laboratory (NREL) has continued to evaluate the WVTR performance of Ultra Barrier Film barrier film samples utilizing their e-calcium (e-Ca) test. The e-Ca method developed at NREL measures the change in resistivity of calcium to calculate the permeation rate of water vapor through the barrier film. The method has been developed such that the moisture permeation path is primarily through the test barrier film. In a study to characterize the cross-web and down-web barrier uniformity of the full-scale Generation 1 UBT UBT, sixteen samples were analyzed. The measurements of water vapor transmission rates (WVTR) ranged between 10^{-7} to 10^{-5} g/m2/day with no clear trend seen from sample location. Samples of Generation 2 UBT also underwent e-Ca testing and have a WVTR range between the lower detection limit of the system and 5x10^{-6} g/m2/day (see Figure 3). Generation 1 UBT and Generation 2 UBT have both demonstrated a Water Vapor Transmission Rate (WVTR) as low as 10^{-6} g/m2/day (see Figure 11).

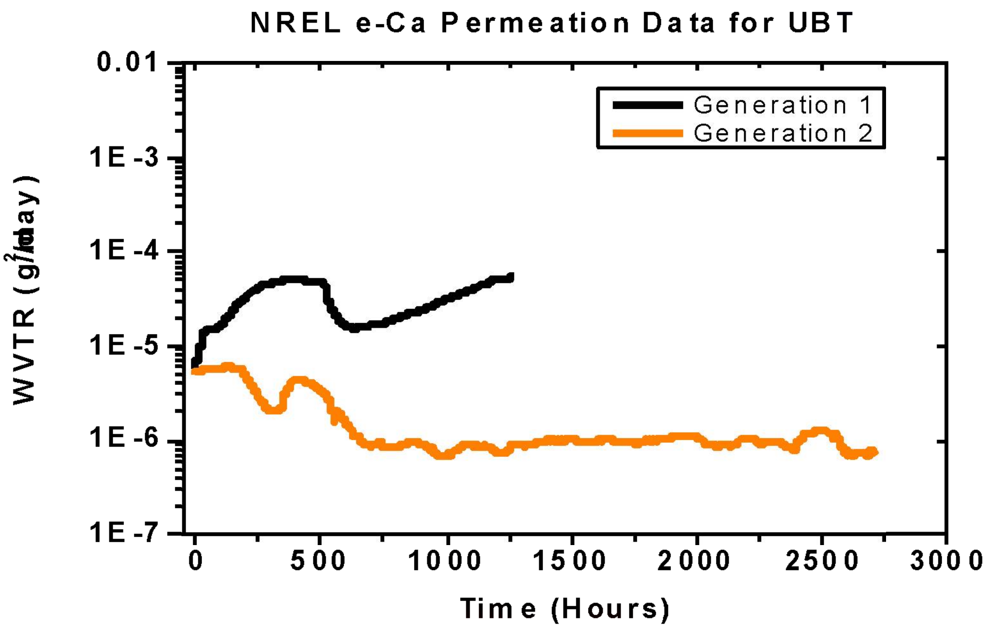


Figure 3.) Generation 2 e-Ca WVTR Testing Results

Optical Transmission: As high as 94%

3M's efforts to improve transmission and transfer this transmission improvement to full scale Production during this contract can be seen by the increase in 3M's Generation 2 UBT vs. 3M's Generation 1 UBT (Figure 4).

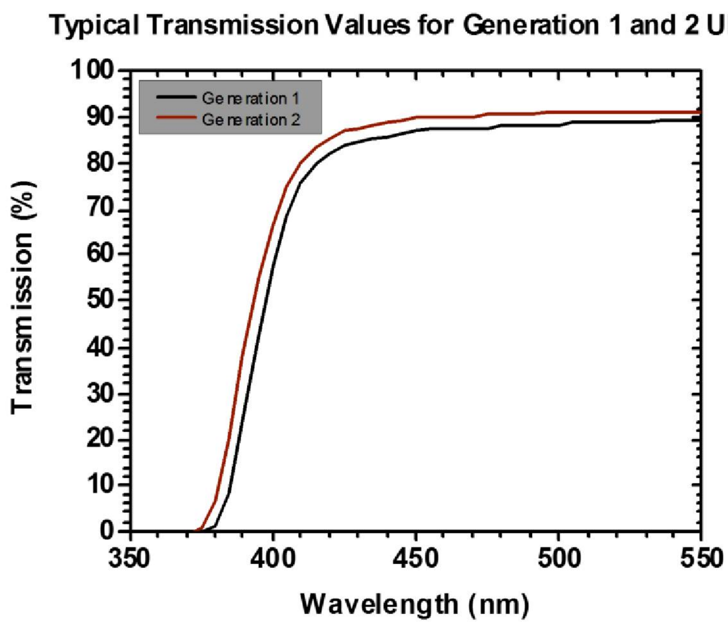


Figure 4.) Optical Transmission versus wavelength for Gen 1 and 2 UBT

The %T measurement results of the ultra barrier topsheets when measured at normal incidence using a Lambda 900 spectrophotometer (avg. 400nm – 1400nm) are reported in the full scan graphs of Figure 5 (Generation 1 UBT) and Figure 6 (Generation 2 UBT). Additionally, Figure 4 and 5 show that the %T is highly stable after numerous environmental exposure conditions, indicating that %T of 3M's Generation 1 UBT and Generation 2 UBT is highly robust. The environmental exposures in Figure 4 and Figure 5 are described as follows:

600 TS = 600 cycles Thermal Shock (-40ºC to 90ºC)

60 HF = 60 cycles Humidity Freeze (-40ºC to to 85ºC @ 85% RH)

3000 85 C = 3000 Hrs at 85ºC

3000 DH = 3000 Hrs Damp Heat (85ºC/85% RH)

804 TUV = 804 MJ/m^2 Total UV 295-385nm (1000 MJ/m^2 ~ 9200 hrs ASTM G155 Cycle 1 Test)

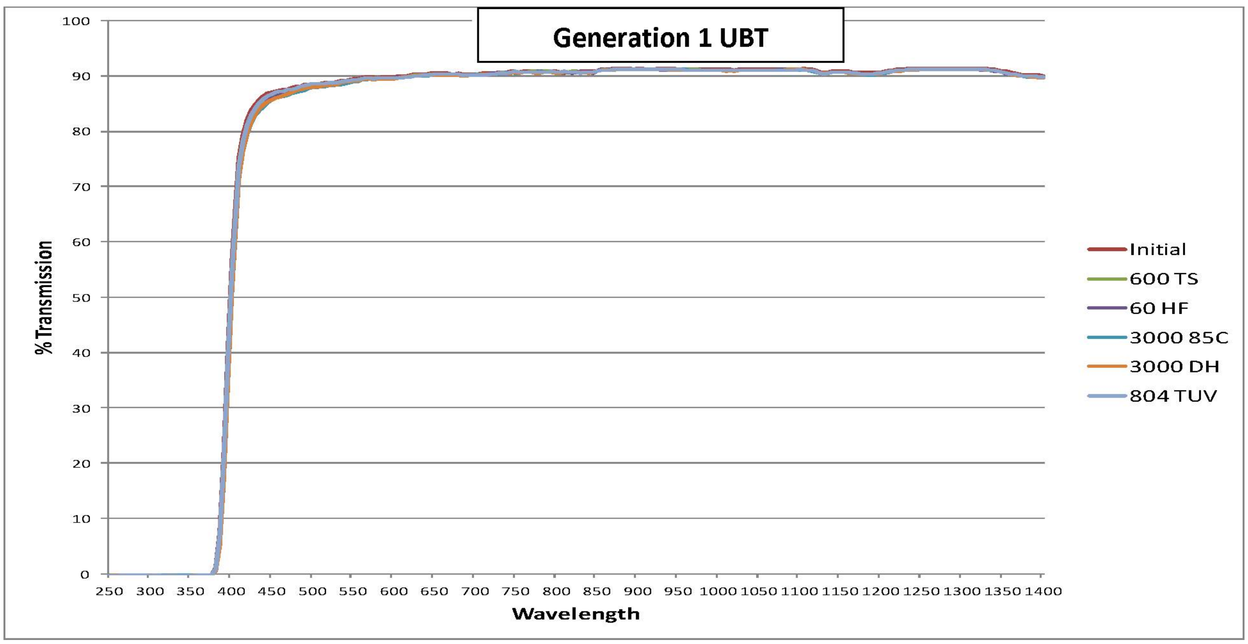


Figure 5.) Generation 1: Lambda (avg. 400nm – 1400nm) = 90.0 %T

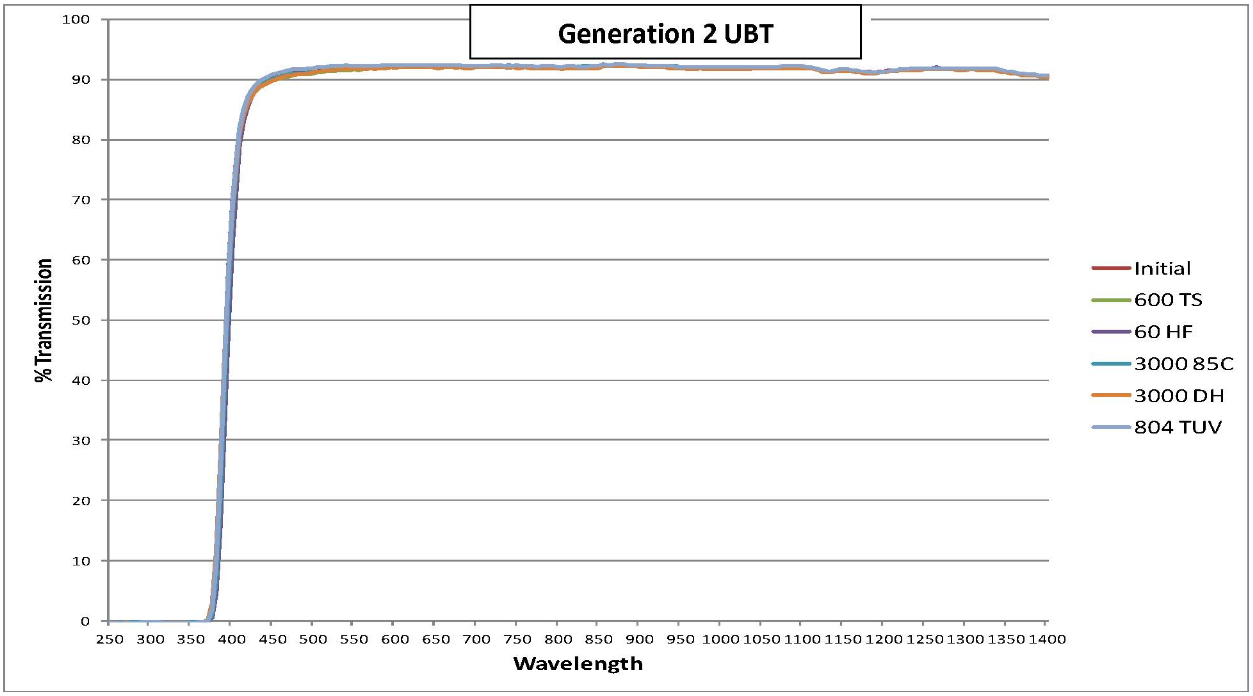
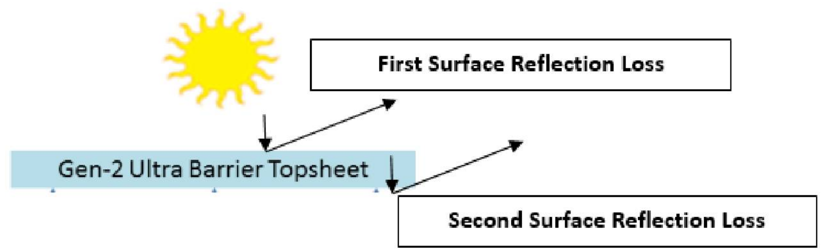
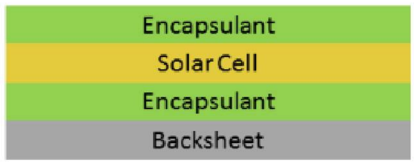


Figure 6.) Generation 2: Lambda (avg. 400nm – 1400nm) = 91.5%T

Transmission (in full module construction) has been shown to be greater than 94%, however, current market performance demands may require even higher light harvesting performance, most likely by addressing the first surface reflection via anti-reflection coatings. When reporting transmission in full module construction (such as to estimate how much light a solar cell will receive) one has to make the following assumptions about the reflection losses as measured by the lambda spectrophotometer. The calculation details below show how the %T was estimated in a full module construction given the First and Second Surface reflection losses as described in Figure 7.





Cross-sectional View of Module with Gen-2 UBT

Figure 7.) Mock-up of Full PV Module with Topsheet as measured using spectrophotometer

Second Surface Reflection Calculation:

Second Surface Reflection (R) can be calculated using a materials index of refraction (n):

$$R = \left(\frac{n1 - n2}{n1 + n2}\right)^2$$

	Air	ETFE	Oriented PET	Common EVA Encapsulant
n	1	1.4	1.65	1.5

Interface	Reflection Loss
Air to ETFE	2.778%
PET to Air	6.020%
PET to Encapsulant	0.230%

Taking into account the second surface reflection losses as measured by the lambda spectrophotometer and the refractive indexes of the materials, one can assume that when using Generation 2 UBT the solar module will receive 97.3% of the light. Additional losses from absorption in the encapsulant or losses to reflection at the encapsulant-to-solar-cell interface may occur.

Calculation details are as follows:
Lambda measurement of total light transmission = 91.5%
Backside reflectance losses as a result of measuring topsheet only (PET to Air) = 6.0%
Total light transmission without backside reflectance losses = 91.5% + 6.0% = 97.5%
When applied to the solar module and accounting for PET to Encapsulant losses of 0.2% = 97.5%-0.2% = 97.3%

Because of the low reflection loss at the air to ETFE interface, low optical absorbance within the topsheet, and minimal reflection loss at the engineered interfaces; 3M's Generation 2 UBT has met the target of delivering greater than 94% of available light to the solar cell.

Customer Feedback: Certified Component

3M's Generation 1 UBT achieved UL component recognition and official spec-in of 3M's 1.2 meter wide Generation 1 UBT at five manufacturers of flexible CIGS modules. Three of those customers achieved UL and/or IEC certifications of flexible CIGS modules manufactured with 3M's 1.2 meter wide Generation 1 UBT. 3M's Generation 2 UBT achieved UL component recognition and is currently in the UL and/or IEC certification process for flexible CIGS modules at various customers. Certification is expected in 2015.

Cost: < $15/m²

Improving the UV stability of the adhesive enabled the investigation of alternate, lower-cost, PET substrates for 3M's Generation 2 UBT. With the more effective UVA block, both color and mechanical strength of the PET were still maintained while the PET costs were reduced by 28%. Additionally, thinning the ETFE in 3M's Generation 2 UBT reduced ETFE costs by 45%. Thinning of the top fluoropolymer was based on current backsheet constructions. The fluoropolymer is the weatherable material and provides environmental protection as well as flame resistance. 3M currently projects that its Generation-2 UBT will achieve a cost of <$15/sq meter in mass production volumes.

UBT Lifetime

In order to achieve a 25 year lifetime, the ultra barrier top sheet must maintain its low water vapor transmission rate and high optical transmission over extended years in multiple locations worldwide. The aggressive exposure environments include high levels of UV and moisture, standing water, ice, high and low temperatures, and thermal shock. Therefore a very rigorous testing regimen was employed to uncover real and potential failure modes. Once uncovered, UBT improvement efforts were undertaken to improve durability.

Possible failure modes included loss of moisture barrier level due to breaches in the oxide barrier coatings, loss of optical transmission due to UV degradation of the polymer layers, and loss of adhesion and therefore compromise of UBT integrity due to separation between layers in the multilayer UBT and the adjacent customer materials including encapsulants and edge seals.

Photographs of some of the testing environments are shown in Figures 8 to 10. The testing environments included:

- Damp heat (DH) (85C/85%RH) for up to 3000 hours
- Modified ASTM G155 conditions which employee cycles of light, heat, and water spray for up to 1119 MJ/m^2 (equivalent to >9000 hours under ASTM G155)
- Thermal shock (TS) for up to 900 cycles (-40^{o}C to 90 ^{o}C)
- Humidity-freeze (HF) for up to 60 cycles (-40 ^{o}C to 85^{o}C @ 85% RH)
- Dry heat (DH) at 85C for up to 1000 hours
- Outdoor exposure in Arizona, Florida, and Arizona using simulated and actual solar modules both under ambient conditions and standing water for more than 1 year
- Studies at NREL the following conditions
  - 85C, 4 suns
  - 85C, 10 suns
  - 105C, 4 suns
  - 105C, 10 suns

Properties measured included:

- Visual, microscopic, and microchemical inspection to identify and assess any defects.
- Water vapor transmission by Mocon at 50C (Detection limit of 5x10^{-3} gm/m^2/day)
- NREL water vapor transmission using their Ca technique with a detection limit of 10^{-5} gm/m^2/day
- Optical transmission as a function of wavelength
- Peel testing to determine interfacial adhesion levels

Figure 8.) 3M's Accelerated Weathering Chambers (Light, Heat, Water Spray)

Figure 9.) Outdoor Testing of Ultra Barrier Top Sheets on Modules in Florida and Colorado

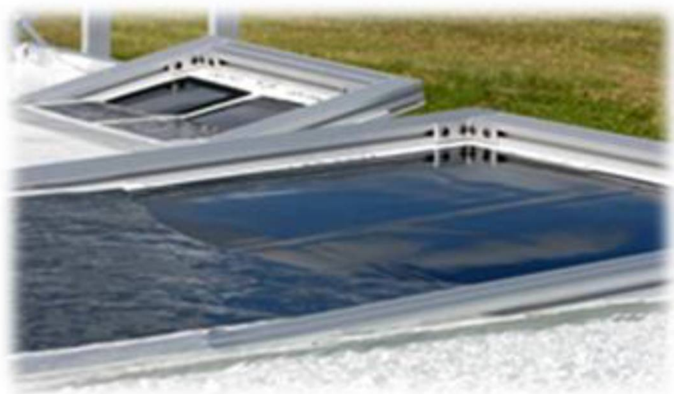


Figure 10.) Outdoor Water Emersion Testing of Ultra Barrier Top Sheets on Modules

UBT Lifetime Testing: Water Vapor Transmission

Most significantly, neither the generation 1 nor generation 2 UBT showed any loss in the ability to limit water ingress to levels consistent with calculations of requirements for a 25 year lifetime of the photovoltaic components being protected. This indicates that the oxide layers in the UBT are extremely robust. As an example, Figure 9 presents NREL results that show that low levels of WVTR were maintained in the extreme conditions of 10 suns and 105C for 2000 hours. Figure 10 shows that low levels of WVTR and good optical transparency were also maintained under damp heat conditions.

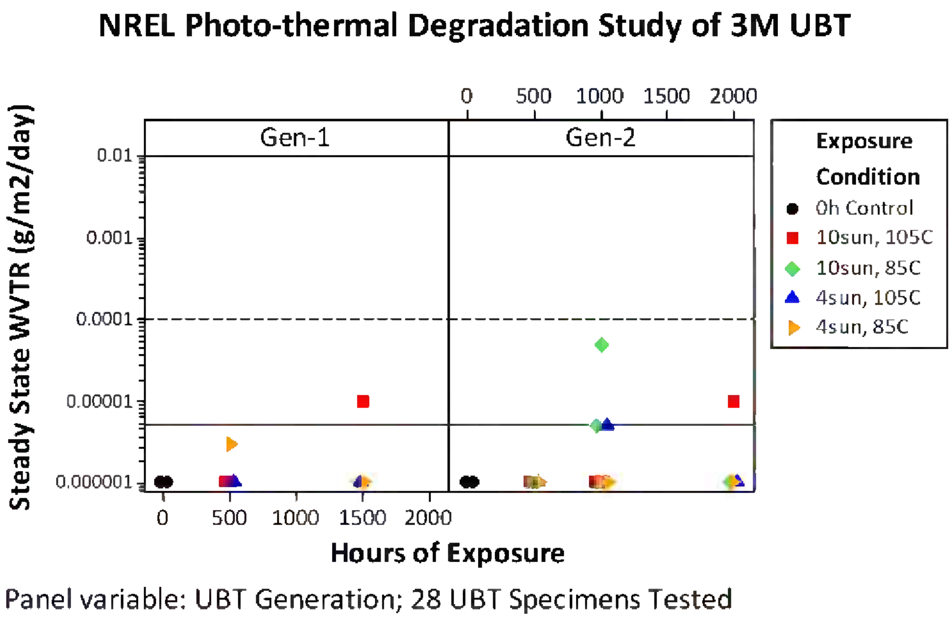


Figure 11.) Water vapor Transmission Rate as a function of light and heat exposure at NREL

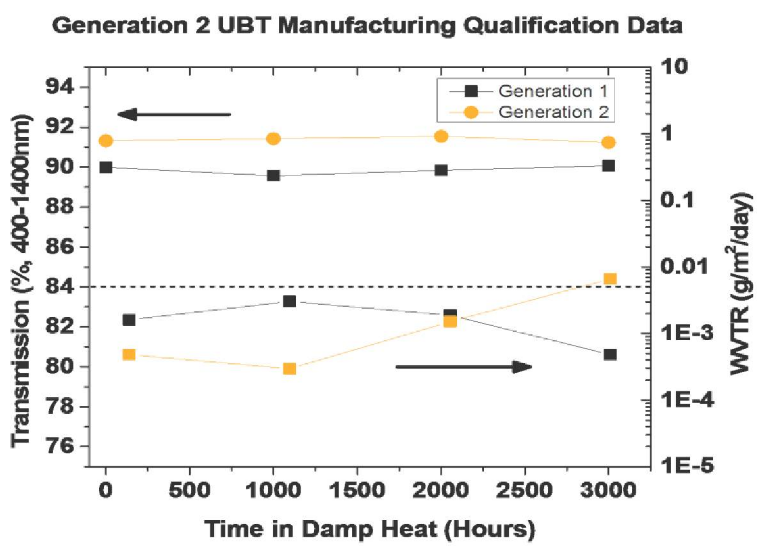


Figure 12.) Optical Transmission and Water vapor Transmission rate as a function of damp heat exposure

UBT Lifetime Testing: Optical Transmission

Results in Figures 5 and 6 show that the optical performance of both Gen 1 and Gen 2 UBT were quite stable under a range of accelerated exposure conditions. However the generation 1 UBT showed significant yellowing and loss of optical transmission as a result of further UV exposure. It was determined that loss of UV blocking ability of the adhesive layer was responsible. Therefore, a successful effort was undertaken to develop a more stable UV blocking additive.

Based on parallel testing, a 10 % increase in UV light transmission was determined to be the threshold for allowing significant UBT yellowing. Figure 13 projects that the level will be well below 10% after 9000 MJ/m^2 which is estimated to be 25 years of exposure in Arizona and similar environments.

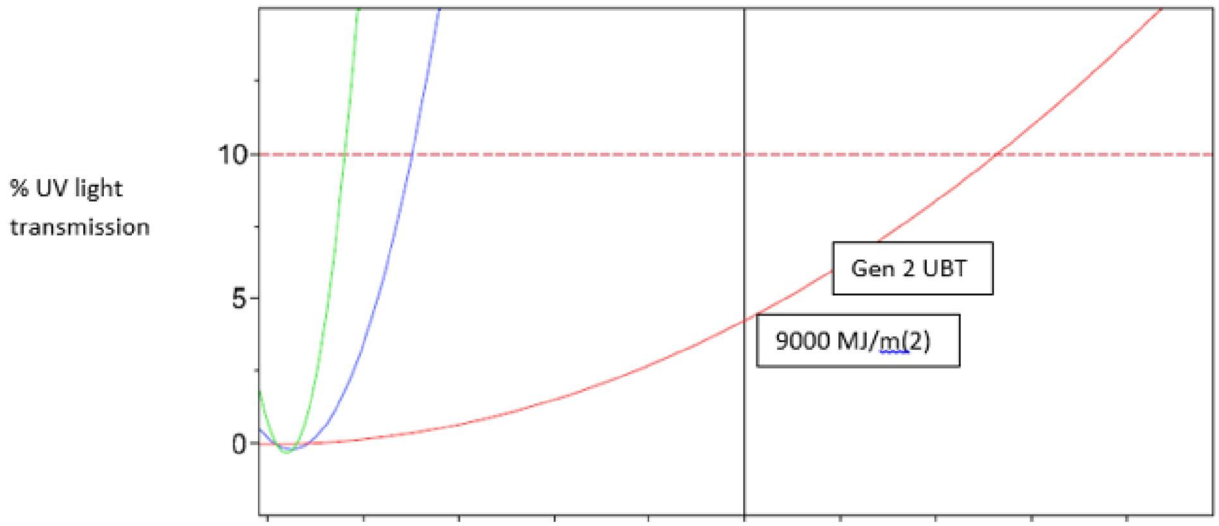


Figure 13.) Quadratic projection of % damaging UV light transmission versus UV exposure in MJ/m^2

UBT Lifetime Testing: Interfacial Adhesion

Although not resulting in delamination, it was observed that interfacial adhesion within the Gen 1 UBT and of Gen 1 UBT to adjacent customer materials like encapsulants and edge seals was reduced by prolonged exposure to damp heat. Therefore modifications to interfacial chemistries were mode to improve performance in the Gen 2 UBT. The Figure 14 presents the dramatic improvements that were made as a result for adhesion within the multilayer UBTs.

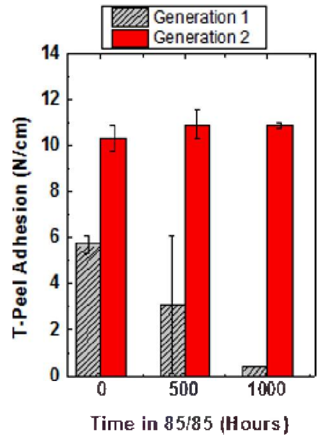


Figure 14.) Interlayer adhesion for Gen 1 and Gen 2 UBT as a function of damp heat exposure

For the Gen 1 UBT, a decrease in adhesion to encapsulants and edges seals was observed after 2000 hours of damp heat. Therefore, improved adhesion stability to customer materials was desired for increased module durability. Modification to the surface of Gen 2 accomplished the required improvement as shown in the table below.

Material type	Adhesion in N/cm			
	Gen1		**Gen2**	
	Initial	Min Post Weathering	Initital	Min Post Weathering
Edge Seal	>5	<5	>40	~25
Thermoplastic Encapsulant	>60	<5	>40	~10
PO Encapsulant	<10	<5	>100	~20

UBT Lifetime Testing: Conclusion

All results indicate that the Gen 2 UBT is extremely durable under all test conditions selected to result in degradation at a more rapid rate than expected in normal outdoor UBT applications. However, verifying a 25 year lifetime with scientific data for all potential exposure conditions proved beyond the capability of the current program due to its complexity and required length of required exposure times. 3M is continuing to test and monitor performance in collaboration with our customers.

Acknowledgements

We wish to acknowledge DOE sponsorship and the active and beneficial participation of the DOE program managers and sponsors throughout the program. Their knowledge and flexibility played key roles in the program's success. We would also like to recognize our partnership under a CRADA with NREL. Their inputs and one-of-a-kind testing capabilities were also essential to program success.