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Depth profiling of degradation of multilayer photovoltaic backsheets after accelerated laboratory weathering: Cross-sectional Raman imaging

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ABSTRACT

Understanding of the durability of each individual layer and their interfaces in a multilayered photovoltaic (PV) backsheet is critical to the design and selection of materials for making reliable and high performance PV modules. In this study, Raman imaging was used to depth profile the chemical degradation of a multilayer commercial backsheet film exposed to ultra-violet (UV) radiation at 85 °C, 5% relative humidity (RH, dry) and 85 °C, 60% RH (humid) on the NIST (National Institute of Standards and Technology) SPHERE (*Simulated Photodegradation via High Energy Radiant Exposure*). The backsheet film was a multipart laminate comprising of a pigmented polyethylene terephthalate (PET)-based outer layer, PET core layer and three ethylene vinyl acetate (EVA) layers having different vinyl acetate (VA) contents, along with two inner adhesive layers between PET outer and PET core layers, and PET core and EVA layers. Cross-sectional samples were prepared by cryo-microtomy for various characterizations. The multilayer structures were examined by laser scanning confocal and atomic force microscopies, while their chemical degradation profiles were obtained by Raman spectroscopic imaging. Non-uniform degradation was observed in the aged backsheet film, and both UV and moisture appeared to significantly affect the degradation profiles of the multilayers. Severe degradation, indicated by high fluorescence, occurred in the outermost region of the pigmented PET outer layer, and the degradation gradient extended to approximately 20 μm to the bulk. It was also found that the inner adhesive layers were severely deteriorated under moist condition, indicating that the long-term adhesion between the layers could be a major area of concern for multilayer backsheets used in a humid environment. The relationship between the sharp (non-uniform) degradation profile, resultant internal stress, and ultimate failures (cracking and delamination) was discussed as well.

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1. Introduction

Polymeric packaging materials are important to the safety, performance, durability, and reliability of photovoltaic (PV) modules during field operation. The backsheet, the outermost packaging material on the back side of a PV module that directly adheres to encapsulant, is the first line of back side defense against environmental aging in the field [1,2]. In general, the backsheet is a polymeric multilayer laminate system engineered so that each layer can address multiple purposes such as weatherability, insulation, adhesion promotion, and light reflection. Fluoropolymers are commonly used as the outermost weathering resistant layer of backsheets due to their chemical and thermal stability. Polyethylene terephthalate (PET) is often used as an insulation layer (usually core layer of the backsheet) because of its low cost, low electrical conductivity and high mechanical strength [1–3]. Moreover, to block ultra-violet (UV) from the back side and redirect reflected sunlight from the front side of a PV module, white inorganic pigments, such as titanium dioxide (TiO_2) and barium sulfate ($BaSO_4$), are widely used in backsheet layers to modify opacity and reflectivity of polymeric layers [2].

To improve long-term stability and prolong service life of backsheets, complex additives such as antioxidants, hydrolysis stabilizers, heat stabilizers, UV absorbers/stabilizers, silane coupling agents, curing agents, and flame retardants are commonly added in the polymeric matrix of each layer for various purposes. The existence of additives results in the polymer degradation processes having multiple variable mechanisms. Usually, the amounts of each additive are as small as 0.1–2.0% mass fraction [4–8]. Consequently, it is difficult to detect the additives using conventional vibrational spectroscopy. However, the formation of conjugated double bonds such as α,β-unsaturated carbonyl groups

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during weathering may contribute significantly to the enhanced fluorescence and/or luminescence emission when degraded samples are illuminated with UV or visible light [4]. Therefore, besides conventional vibrational spectroscopies, fluorescence and luminescence-based analytical techniques can be employed as a synergistic approach to monitor the extent of degradation [9–11].

The current requirement of greater than 25-year service life for PV module is a big challenge to module and backsheet manufacturers. Practical in-field testing results showed that significant backsheet failures such as cracking, yellowing and delamination occurred within a 5-year service [2]. Previous research efforts have investigated backsheet degradation and failure by studying either individual material components or mini-modules [2,3,12–18]. Some studies focused on characterization of backsheet film as a whole as well as its top/bottom surfaces [2,14–16], while others addressed the adhesion behaviors between layers by peeling tests [3,12,13]. However, there are limited data showing a degradation profile across backsheet layers and how different aging stresses impact individual layers [17,18]. For this reason, an in-depth investigation on the degradation profile of polymeric multilayer backsheet is needed for a better understanding of backsheet failure mechanisms.

Cross-sectional analysis can provide robust information for depth profiling of multilayer systems [19,20]. Raman spectroscopy is known to be non-destructive for samples, and can reveal polymer degradation by means of not only the fingerprint of vibrational spectrum but also fluorescence emission [10]. Furthermore, instead of single-point or one-dimensional spectroscopic characterization, two-dimensional spatial spectroscopy such as Raman imaging has been shown to accurately describe complex polymeric components [21,22]. For these reasons, we have employed the combination of cryo-microtomy cross-sectioning and Raman microscopic imaging to investigate degradation profile in multilayer backsheet system.

In this study, a commercially available backsheet consisting of PET/PET/ethylene vinyl acetate (EVA), commonly named as PPE, was selected as a model sample due to its low cost compared to fluoropolymer-based backsheets. Unless stated otherwise, the accelerated laboratory weathering of multilayer PPE backsheet was performed for 1600 h at 85 °C/5% RH (dry) and 85 °C/60% RH (humid) conditions on the NIST (National Institute of Standards and Technology) SPHERE (Simulated Photodegradation via High Energy Radiant Exposure) weathering chamber. The accumulated UV irradiance on each specimen is approximately 170 W/m^2 between 300 nm and 400 nm after 1600 h exposure on the SPHERE. Assuming 10% albedo for the reflected light on the backsheet during field exposure [2,23], the UV dose for this exposure (~980 MJ/m^2 between 300 nm and 400 nm) is approximately equivalent to that of a 25-year field exposure on the back side of an on-rack PV module in Phoenix, Arizona.

To analyze the depth profile after degradation, cryo-microtomy was used to obtain cross-sectional PPE specimens with a flat surface parallel to the film thickness. The evolution of the multilayer structure and chemical degradation profiles were examined before and after accelerated laboratory weathering by laser scanning confocal microscopy (LSCM), atomic force microscopy (AFM), and Raman imaging.

2. Experimental[1]

2.1. Backsheet specimens and accelerated laboratory weathering

A commercial, free-standing, multilayer PPE backsheet film was used as received for this study. Circular specimens with 19 mm in diameter were punched from the PPE backsheet film and then placed on a sample holder for accelerated laboratory weathering on the NIST 2-m diameter integrating sphere-based high intensity UV weathering facility, referred to as SPHERE [24] at 85 °C ± 0.5 °C under 5% ± 3% RH (dry) and 60% ± 3% RH (humid) for 1600 h. A few specimens were exposed up to 3280 h to investigate ultimate failure modes including cracking or delamination. The UV irradiance on the PET outer layer side of the samples was approximately 170 W/m^2 between 300 nm and 400 nm.

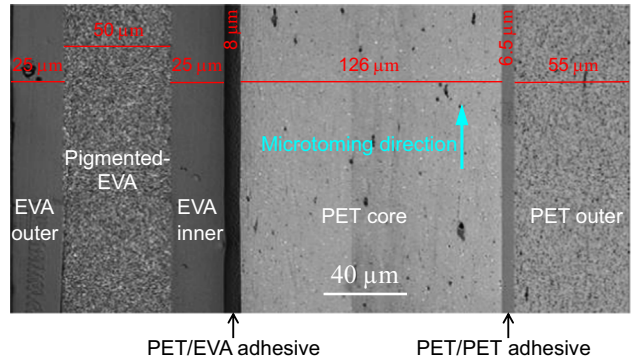

Fig. 1. LSCM image of the cross-section of fresh PPE backsheet with various polymer layers and their corresponding thicknesses. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

To prepare cross-sectional specimens, 3 mm × 4 mm pieces cut from the center of the 19 mm circular sample were embedded in an epoxy molding. Cryo-microtoming was carried out on a Leica EM FC7 (Leica Mikrosysteme GmbH) with glass blade for coarse surfacing and then followed with diamond blade for fine surfacing. The liquid N_2 was used to cool the sample, ensuring a brittle fracture resulted from the microtoming to minimize the surface roughness for microscopic and spectroscopic imaging [25]. The cross-section is parallel to the film thickness. Care was taken to make sure that the microtoming direction did not cross the layers of PPE to avoid smearing between layers as the blue arrow shown in Fig. 1.

2.2. Laser scanning confocal microscopy and atomic force microscopy characterizations

Images of the cross-section and exposed (weathered) surfaces were acquired using a Zeiss LSM510 META (Carl Zeiss, Inc., Oberkochen, Germany) laser scanning confocal microscope in reflection mode with a laser wavelength of 543 nm. A series of single images (optical slices) each having a *z*-direction step size of 100 nm were obtained by adjusting the focal plane in the *z*-direction. Objectives of 10 × and 50 × (Zeiss, Germany) were used to observe two or more samples per sampling interval with sufficient observation locations to check uniformity of sample. The pixel size of the LSCM images is 512 pixels by 512 pixels. The optical slices of each specimen were carefully analyzed and representative images located at proper *z*-position are presented.

AFM topographical images were acquired by using a Dimension Icon® AFM (Bruker) with PeakForce QNM® (Quantitative Nanomechanical Property Mapping) based on peak force tapping mode. An antimony doped silicon probe with reflective aluminum back side coating (RTESPA, Bruker) was used. The probe spring constant is 40 N/m ± 5 N/m with resonant frequency of approximately

[1] Certain commercial equipment, instruments or materials are identified in this paper in order to specify the experimental procedure adequately. Such

(*footnote continued*)
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