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Fracture Mechanics and Testing of Interface Adhesion Strength in Multilayered Structures – Application in Advanced Solar PV Materials and Technology

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Abstract

Quantitative characterization techniques based on fracture mechanics were proposed and used to examine the interface strength in multilayered structures. Novel *in situ* micro-fracture testing for nanolayers inside a Scanning Electron Microscope (SEM) is developed to observe fracture evolution during bending deformation of nanolayered films. Other quantitative technique on interface fracture mechanics has been the 4-point bending or double cantilever beam bending methods. In this paper we will discuss about these two techniques and their applications. In particular, the Double Cantilever Beam bending test method using Delaminator v8.2 Adhesion testing system has been used to quantify the adhesion strength between the Solar PV backsheet and encapsulant. The environmental conditions in tropical countries makes the photovoltaics components vulnerable to salt mist and water vapour as well as acid penetration. Under moisture condition, the hydrolysis reaction of water vapour with backsheet materials release acetic acid, causing delamination and further corrosion of the encapsulant and inter-metallic connectors on solar cells by the salt mist leading to electrical shorts, heat accumulation and fire. Understanding the interface strength between these two materials and its degradation with typical environments in tropical and near-ocean regions is instrumental to enable robust and reliable solar PV technology for such regions.

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Keywords: In Situ micro-fracture; Delamination; Corrosion; Encapsulant; Interface adhesion;

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1. INTRODUCTION

The Photovoltaic (PV) modules in the tropical regions are exposed to extreme environments such as high humidity and temperature, high concentration of salt, minerals, and acidic rains. This poses a significant threat to the reliability of the modules, thus leading to failure. The traditional PV module designed for four-season region is being used worldwide irrespective of the different environmental conditions at the places they are installed. This design is in a way overdesigned as well as under designed for tropical regions on certain aspects such as high strength (to withstand heavy snow, gusty wind and hail impacts) and vulnerability to extreme environmental conditions in the tropical regions respectively. Apart from maintaining the performance of the module, the packaging materials such as backsheet and encapsulant are also significant to ensure the long-term reliability in a harsh environment [1-3].

Polymer backsheet layer serves to provide insulation barrier to prevent electrical current leak from solar cells during field exposure. In the presence of moisture, water vapour will undergo hydrolysis reaction with the backsheet materials discharging acetic acid and causing delamination. Moreover backsheet delamination might result in penetration of a large amount of moisture as well as salt leading to cracking and a wide spread corrosion. This can cause further corrosion of the polymer encapsulant as well as inter-metallic connectors on the solar cells and in-turn electrical short circuit, heat accumulation and possibly fire. In addition to this polymer corrosion and encapsulant discolouration will also be induced as a result of exposure to salt mist from the oceans and ultraviolet (UV) radiation along with alternating day-night temperature cycles, reducing sunlight absorption as well as output efficiency [4-6].

Thus suitable packaging materials are to be chosen. Also the module design has to be tweaked in a way so as to improve its reliability and performance in the intended region. This can be done only by studying the effect of the environment on the module and their failure mechanism. The fracture mechanics and characterization of the interface between various layers is thus essential. We plan to study the reliability of the multilayered structures (for example solar panel modules in this case) by two techniques. One is to study the interface adhesion between the layers and the other technique is to study the fracture of materials at nanoscale level both described in the coming section.

The study of fracture of materials at nanoscales requires adopting microscale testing techniques to obtain the sensitivity needed for response from nanostructures. In addition to problems with sample preparation and loading at these small length scales, there is also a lack of ASTM standards for testing at such dimensions. This makes it difficult to compare results obtained from different studies. Applicability of linear elastic fracture mechanics in fracture toughness studies also rests on the assumption of small scale yielding criterion, which is difficult to maintain with decreasing sample dimensions. There is thus certainly a need for research of fracture mechanics of materials at the smaller scales (micron and nanometer scales). Focused ion beam (FIB) machining or photolithographic techniques are the most common tools of choice to prepare samples at such length scales. The technique has been used in many instances [7, 8] for the making of single cantilevers, micropillars, microbeams and tensile specimens which are eventually loaded using nanoindenters, microtensile testers or *in situ* testing facilities [8, 9].

Employing a new technique for fracture toughness testing in thin films and nanostructured materials/films using FIB machined microbeams (Fig. 1), K_{IC} (critical stress intensity factor in mode I crack growth) values will be determined quantitatively and the evolution of crack growth and modes in nanolayered materials can be investigated. Pairing the high sensitivity of nanomechanical testing with the high spatial resolution of electron microscopy creates a powerful tool for direct observation mechanical characterization of nanostructures and nanomaterials. This is only recently enabled by the picoscale depth sensing indenter capability that can be interfaced with a scanning electron microscope (SEM). With this system, it is now possible to perform quantitative nanomechanical testing while simultaneously imaging with the SEM. One example of such capability is the PI 85 SEM PicoIndenter tool (Fig. 2). This tool is simply an indenter that has picoscale sensitivity that can be used as a standard fracture testing capability for the machined microbeam structures that we envisioned with our nanolayers on the substrate (Fig. 1).

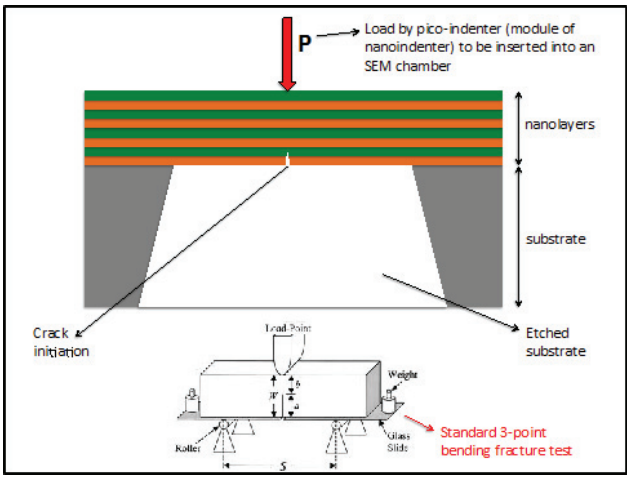
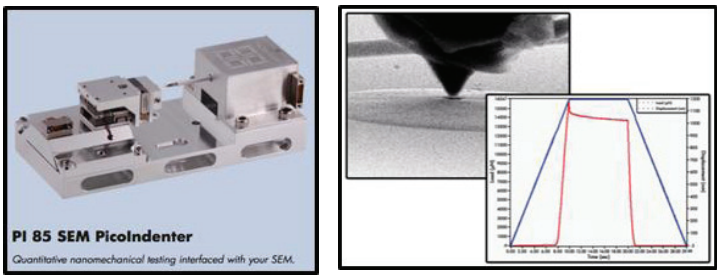


Fig. 1. A proposed novel in situ micro-fracture testing for nanostructured materials/films enabled only by recent advances in nanomaterials characterization and manipulation tools such as FIB (Focused Ion Beam) and a Pico-Indenter (recent advances in Nano indentation tooling) as well as MEMS techniques such as etching of substrates and building of cantilever structures



(a) (b)

Fig. 2. . Nanomechanical tester that can be interfaced within an SEM environment (a) would allow mechanical data to be fully synchronized (b) with the SEM video (in situ observation) revealing unique insights that could lead to complete understanding of materials behaviours at the nanoscales (Courtesy of Hysitron Inc.)

While this technique is currently still a work in progress in our research group and will certainly provide a monitoring capability of interface adhesion degradation during an accelerated environmental testing for instance, this paper will focus on the study using the Delaminator v8.2 to quantify interface adhesion strength between the backsheet and the encapsulant materials, albeit without the monitoring capability of how the fracture initiates and propagates, etc. This is performed by testing the adhesion energy of two different encapsulant materials (EVA and Non-EVA encapsulant) against the same typical composite backsheet in solar PV industry. A valid fracture mechanics based testing method was carried out using the Delamination v8.2 Adhesion testing system. The tests are indeed still rather preliminary as they were carried out at this stage only at normal conditions i.e. without the effect of salt or damp heat. The delamination energy was quantified and the most common fracture locations for the two different encapsulants were observed.

2. EXPERIMENTAL DETAILS

Backsheet debonding has been previously studied using various specialized techniques such as Peel test, indentation method and the blister test [10]. However, these test methods involves calibration and assumptions leading to qualitative results such as in the case of indentation method where a dilated plastic zone is formed in the film to cause the film to blister [11]. In peel tests, which involve pulling the thin film from the substrate [12], the plastic bending of the film involved makes it difficult to arrive at the exact adhesion energy [13]. Lastly, the blister test where debonding is achieved by creating a cavity in the substrate below the film has various complexities such as chemical reaction between debond and the pressurized environment, the loading system and the etching or machining process involved in producing the cavity [14].

In all the above mentioned techniques, the residual stresses in the thin film relaxes during debonding and in turn adds up to the driving force for debonding which is the ultimate limitation for these techniques. This effect can be large and in some cases it is difficult to measure the residual stress, especially for rigid substrates where curvature techniques cannot be used. To overcome these limitations a quantitative characterization technique such as double cantilever beam test method was used to examine the adhesion strength between the backsheet and the encapsulant.

In double cantilever beam test method, the adhesive failure at the interface is modelled as a crack propagating between two relatively thick beams (Fig. 3). This test method is based on the valid fracture mechanics of the bulk materials and here the interface that has to be studied is sandwiched between two beams and is debonded by applying loads at the ends of the beam. Sufficient beam thickness will be chosen such that the maximum bending stress of the beam is smaller than the yield stress. When the load is applied the beam bends and stores up the elastic strain energy. As the interface debonds, some of the stored energy will be released and this released energy in turn acts as the relaxation energy which provides the necessary driving force for debonding [15].

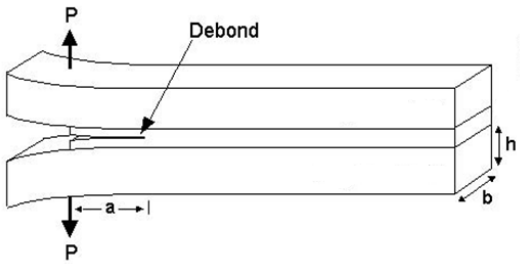


Fig. 3. Double Cantilever Beam test method.

For this study, the single cantilever beam (SCB) testing method based on the above DCB method was employed. This technique has the advantage of constrained stress relaxation of the film during debonding which does not contribute to the debonding driving force. The crack extension force (G) is obtained using Equation 1 as follows:

$$G = \frac{6p^2a^2}{Eb^2h^3} \qquad \text{Equation 1.}$$

Where ‘p’ is the load applied, ‘a’ is the debond length, ‘b’ and ‘h’ are the beam width and height and ‘E’ is the elastic modulus of the beam. The adhesion strength can be characterized as the amount of energy required per unit area to propagate the crack along the interface and is termed as critical crack extension force (G_c) [16].

2.1. Test Specimen

The representative test specimen is fabricated by laminating a layer of encapsulant of thickness 1mm between a tempered glass substrate (3.5 mm) and a photovoltaic composite backsheet (150 – 350 µm) consisting of a layer of polyvinyl fluoride (PVF), a layer of polyethylene terephthalate (PET) and a layer of ethylene-vinyl-acetate (EVA) Seed (Fig. 4). Two samples were prepared with two different encapsulant materials. One with ethylene-vinyl-acetate (EVA) as the encapsulant (Sample 1 - Encap A) and the other with a non-EVA encapsulant (Sample 2 - Encap B). The final layer of EVA seed in the backsheet was to enhance the adhesion between the backsheet and the EVA encapsulant in the case of first sample. The glass substrate was thoroughly cleaned before the lamination and during lamination the layers were fixed at their corresponding position using Kapton tape. The lamination was done at 145º C for 8 minutes under an applied pressure of 1 atm.

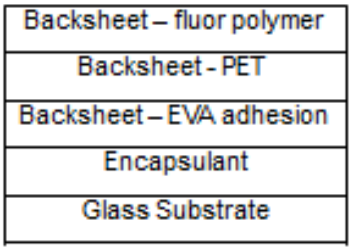
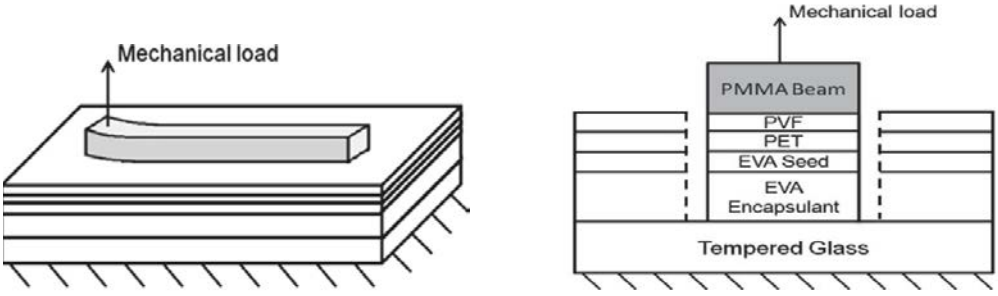


Fig. 4. Test Specimen lay-up consisting of backsheet, encapsulant and glass substrate.

2.2. Procedure

The glass substrate of the backsheet specimen was then fixed to a testing table using a C-clamp. Then a poly-methyl-methacrylate (PMMA) beam of 5mm width, 10mm thickness and 100mm length, was bonded on to the backsheet surface using a superglue (ethyl2-cyanoacrylate) and an accelerant (sodium bicarbonate) as shown in Fig. 5a . An incision was made at the edges of the beam through the thickness of the backsheet and the encapsulant in order to ensure that the debonding is confined to the section directly below the PMMA beam (Fig. 5b).



(a) PMMA beam attached to the backsheet surface; (b) Side view - Incision made at the edges of the beam

Fig. 5. Schematic of the sample and the PMMA beam [16].

Mechanical loading was applied through a loading tab (with a ruby bearing) bonded to the beam at its one end. This loading tab was connected to the high resolution micromechanical (Delaminator v8.2) adhesion testing system operating under displacement control (Fig. 6). The backsheet debonding was initiated by applying a tensile force on the loading tab until the distance between the loading tab and the debond front, that is the debond length was around 15mm. This configuration of the PMMA beam can be considered as that of a single cantilever beam fixed at the debond front. Accurate measurements were possible as the stiffness of the single cantilever beam was much smaller than that of the testing system. The thickness of the PMMA beam was chosen such that there is no significant plastic deformation of the beam due to bending, which can be achieved because the maximum bending stress of the beam will be much smaller than the yield stress.

Fig. 6. The test set up showing the laminate sample fixed to the testing table with C-clamp, the PMMA beam attached to the backsheet surface and the loading tab. The loading tab is connected to the Delaminator v8.2 adhesion testing system.

The test was conducting in accordance with the ASTM E399 fracture standard and in lab air at 45% relative humidity and at a temperature of 25º C. The load was applied through the loading tab and the debond length was monitored using high resolution automated compliance techniques. The debond path location was determined using optical, FTIR and high resolution XPS characterization during the study. This is to ensure that valid cohesion and adhesion values are obtained. The load was applied at a fixed displacement rate of 10 µm/s. The crack begins to propagate at a certain critical load, thus resulting in a slight drop in the load. This is due to the increased compliance. The beam was then stopped from further displacement, keeping the deflection constant. At this point the drop in the load and the crack length are carefully monitored. The specimen is then consecutively unloaded and then loaded. This procedure was repeated several times until the backsheet is completely debonded and the load applied, P, was recorded as a function of the displacement giving us the load-displacement curve. The experimental data such as load, deflection, crack length and the compliance were obtained at various times. From this the debonding driving force (G) was found and the critical crack extension force (G_c) was found as it is the value of G where the load displacement curve starts to deviate from linearity.

3. RESULTS AND DISCUSSION

The debond energy G_c, between the backsheet and the encapsulant was found for both the samples with different encapsulant material. The load vs. displacement curve for one of the sample is shown in Fig. 7. It can be seen that the slope of the unloading curve decreases each time as the crack length increases. From this curve the compliance was calculated and utilized to obtain both the debond length and the delamination energy as shown in Fig. 8(a). The delamination energy vs. the debond length for the sample 2 with non-EVA encapsulant is shown in Fig. 8a.

From the plot it can be seen that the critical crack extension force (G_c) for the sample is 668 J/m^2. Fig. 8b shows the delamination energy for both the samples. It is found that the sample 2 has higher delamination energy than the sample 1.

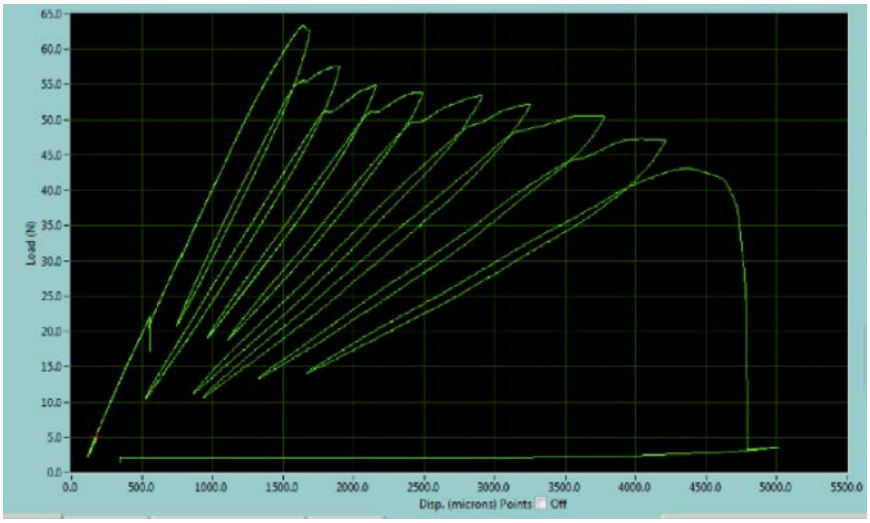
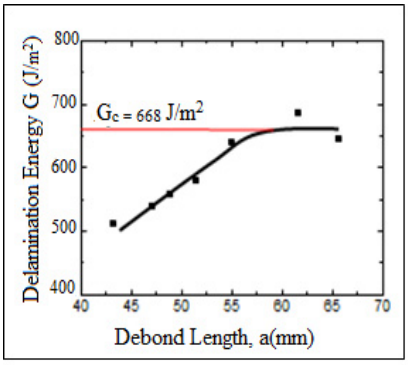
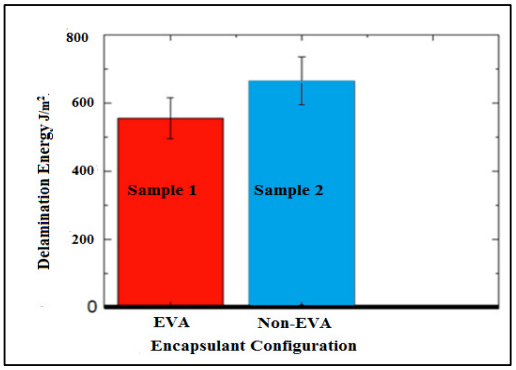


Fig. 7. Load vs Displacement curve





(a) Delamination energy as a function of debond length for sample 2 (b) Critical crack extension force for both the samples

Fig. 8. Crack extension force (G) and critical crack extension force (G_c) obtained from SCB test

The failure mode for the two samples were observed. Seven cases of delamination were observed in both the samples as seen in the Fig. 9.

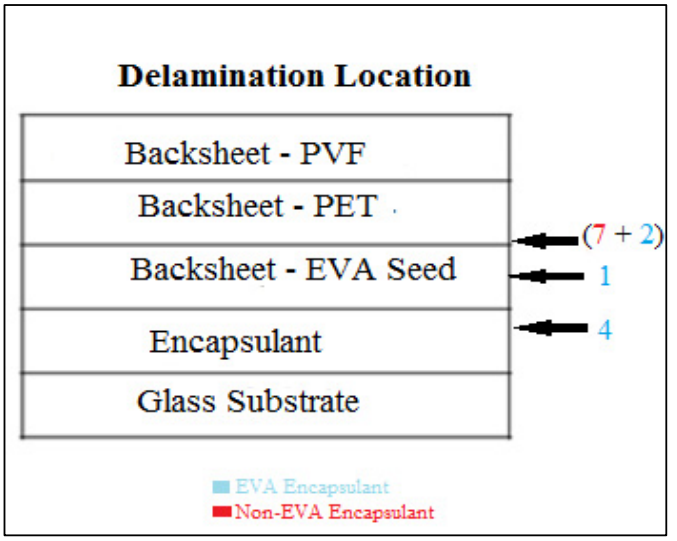


Fig. 9. Delamination location for both the samples. (Red colour indicating the sample 1 with EVA encapsulant and blue colour for sample 2 with non-EVA encapsulant)

All the 7 cases of delamination in sample 1 were found to be adhesive failure between the backsheet layers PET and EVA seed. This is due the backsheet layer EVA seed being the same material as that of encapsulant (EVA) which enhances the adhesion between the two layers. But for sample 2, only 2 cases of delamination were adhesive failure between the backsheet layers PET and EVA seed, while the remaining 5 were cohesive: 4 within the encapsulant layer and 1 within the backsheet layer EVA.

4. CONCLUSION

Preliminary test was conducted to quantify the interface adhesion between the backsheet and the encapsulant. It is clear that the single cantilever beam test method using the Delaminator v.8.2 adhesion testing system produces very quantitative and repeatable data and its effectiveness can be utilized to study how interface adhesion between various layers especially backsheet and encapsulant in the module degrade with in general moisture but also more specifically with salt and acidic mist which is instrumental to enable robust and reliable design for solar PV technology specifically designed for tropical regions and/or for solar farm on oceans. This can be used to select the correct materials to make a strong, tough and durable adhesive bond.

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References

[1] Czanderna, Encapsulation of PV modules using ethylene vinyl acetate copolymer as a pottant: a critical review, Solar Energy Materials and Solar Cells 43(1996)101–181.

[2] D. L. King, M. a. Quintana, J. a. Kratochvil, D. E. Ellibee, B. R. Hansen, Photovoltaic Module Performance and Durability Following Long- term Field Exposure ,vol. 565, AIP Conference Proceedings (1999)565–571.

[3] A.S. Budiman, G. Illya, V. Handara, A. Caldwell, C. Bonelli, M. Kunz, N. Tamura, and D. Verstraeten. Enabling Thin Silicon Technologies for Next Generation c-Si Solar PV Renewable Energy Systems using Synchrotron X-ray Microdiffraction as Stress and Crack Mechanism Probe. Solar Energy Materials and Solar Cells, 130:303–308, 2014.

[4] S. Murray, C. Hillman, M. Pecht, Environmental aging and deadhesion of siloxane-polyimide-epoxy adhesive, IEEE Transactions on Components and Packaging Technologies 26 (2003) 524–531.

[5] C. Dechthummarong, B. Wiengmoona, D. Chenvidhyaa, C.J ivacatea, K. Kirtikaraa, Physical deterioration of encapsulation and electrical insulation properties of PV modules after long-term operation in Thailand, Solar Energy Materials and Solar Cells 94 (2010) 1437–1440.

[6] H. Li, R. Kikuchi, M. Kumagai, T. Amano, H. Tang, J.-M . Lin, K. Fujiwara, N. Ogawa, Nondestructive estimation of strength deterioration in photovoltaic backsheets using a portable near infrared spectrometer, Solar Energy Materials and Solar Cells 101 (2012) 166–169.

[7] S. Reyntjens, R. Puers: A review of focused ion beam applications in microsystem technology. J. Micromech. Microeng. 11, 287 (2001).

[8] A.A. Tseng: Recent developments in micromilling using focused ion beam technology. J. Micromech. Microeng. 14, R15 (2004).

[9] D. Kiener, C. Motz, T. Schoberl, M. Jenko and G. Dehm: Determination of mechanical properties of copper at the micron scale. Adv. Eng. Mater. 8, 1119 (2006).

[10] R. H. Dauskardt, M. Lane, Q. Ma, N. Krishna, Adhesion and debonding of multi- layer thin film structures, Engineering Fracture Mechanics. 61 (1998) 141–162.

[11] M.D. Drory, J.W. Hutchinson. Proc Royal Soc A 1996;452:2319-41

[12] M.D. Thouless, H.M. Jensen. J Adhesion 1992;38:185-97.

[13] O. Jùrgensen, A. Horsewell, B.F. Sùrensen, 1996. Comment on a new procedure for measuring the decohesion energy for thin ductile films on substrates. A. Bagchi, et al., editors. J Mater Res. 11(8) 2109-11.

[14] H.M. Jensen. The blister test for interface toughness measurement. Eng Fract Mech 1991;40(3):475-86.

[15] J Cognard., J. Adhesion, 1987, 22, 97

[16] R. H. Dauskardt, Fernando D. Novoa, David C. Miller, Environmental mechanisms of debonding in photovoltaic backsheets, Solar Energy Materials and Solar Cells 120 (2014) 87-93.