

Adhesion Strength Study of EVA Encapsulants on Glass Substrates

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ABSTRACT

An extensive peel-test study was conducted to investigate the various factors that may affect the adhesion strength of photovoltaic module encapsulants, primarily ethylene-vinyl acetate (EVA), on glass substrates of various laminates based on a common configuration of glass/encapsulant/backfoil. The results show that "pure" or "absolute" adhesion strength of EVA-to-glass was very difficult to obtain because of tensile deformation of the soft, semi-elastic EVA layer upon pulling. A mechanically "strong enough" backing foil on the EVA was critical to achieving the "apparent" adhesion strength. Peel test method with a 90-degree-pull yielded similar results to a 180-degree-pull. The 90-degree-pull method better revealed the four stages of delamination failure of the EVA/backfoil layers. The adhesion strength is affected by a number of factors, which include EVA type, formulation, backfoil type and manufacturing source, glass type, and surface priming treatment on the glass surface or on the backfoil. Effects of the glass-cleaning method and surface texture are not obvious. Direct priming treatments used in the work did not improve, or even worsened, the adhesion. Aging of EVA by storage over ~5 years reduced notably the adhesion strength. Lower adhesion strengths were observed for the blank (unformulated) EVA and non-EVA copolymers, such as poly(ethylene-co-methacrylate) (PEMA) or poly(ethylene-co-butylacrylate) (PEBA). Their adhesion strengths increased if the copolymers were cross-linked. Transparent fluoropolymer superstrates such as Tefzel™ and Dureflex™ films used for thin-film PV modules showed low adhesion strengths to the EVA at a level of ~2 N/mm.

1. Introduction

Adhesion strength of polymeric encapsulants such as EVA to the glass substrates on PV modules is an important factor that can affect critically the performance reliability and durability of modules exposed to weathering environments. Delamination of EVA from the glass superstrates on field-deployed crystalline-Si modules has been observed before [1]. Lately, largely reduced adhesion strength of EVA from glass plates was reported after being subjected to the damp-heat test in an environment test chamber under 85% relative humidity at 85°C [2,3]. In previous studies, we observed that moisture ingress and retention occurred in the glass/EVA/glass laminates, and that moisture condensation occurred in the glass/glass assembly

with EVA around the edges only, when the samples were exposed in the dark to high-low temperature and relative humidity cycles in a weatherometer [4]. In the former laminates, the originally clear and transparent EVA layer would become white/turbid, which gradually disappeared over time when the laminates were placed in the air to dry. The milky white turbidity and its subsequent gradual disappearance were also observed on EVA/glass and glass/EVA/glass laminates soaked in a 85°C water bath in later experiments [5]. The EVA layer on the EVA/glass laminates could be peeled off fairly easily when the laminates were still wet and hot, but was largely restored when dried. This adhesion recovery was also observed by another group [2]. There is a lack of systematic research to understand adhesion issues for the EVA used in PV modules, however. To address such adhesion reliability issues, we conducted experiments to understand and determine the factors that may significantly affect the adhesion strength of EVA/substrate measured by conventional peel-test methods, to quantify the water uptake and loss by EVA laminated with various substrates, and to examine the hydrolytic stability of various silanes on glass substrates. This paper focuses primarily on the adhesion strength study.

2. Experimental

2.1. Sample Preparations: Various glasses, EVA encapsulant formulations, and backing foils from different sources were used for the laminations in a custom-made, programmable, double-bag vacuum laminator. Non-EVA encapsulants were also included in the study. All the polymer films were either new or less than 6 months old. Sources of EVA and backfoils are not identified herein. Laminations were performed in either fast or regular-cure cycles, depending on the EVA formulation. The gel content was determined for the cured EVA. The samples were prepared by separate laminations for each set designated for a given specific test objective in order to allow for comparison. The glass plates were thoroughly cleaned by different methods, and primed if needed, 30 m to 1 h before use.

2.2. Peel Tests: Peel tests were based on ASTM D903-98 and measured by using an Instron tensile-test apparatus of Model 5500 fitted with Load Cell Model 2511-104 with a constant crosshead speed of 1 cm/min at ambient room temperatures. The samples were tested mostly with a 90-degree pull on a custom-modified Parker-Daedal Model

4615 Linear Motion Slide; and 180-degree pull tests were also conducted on some samples for comparison.

3. Results and Discussion

3.1. The Need of A Backing Foil and Use of Apparent Adhesion Strength

When the 90-degree-pull test was performed on glass/EVA laminates *without* a backing foil, tensile deformation of the soft, semi-elastic EVA layer caused the film to "neck" or narrow quickly after the first ~1-2 mm peel from the glass surface, and showed a linear increase in strength as shown in Fig. 1. The reproducible results indicate that it is difficult, if not impossible, to derive a reliable "pure" or "absolute" EVA-to-glass adhesion strength from the pull load-distance curve. When the pull tests were performed on glass/EVA/backfoil laminates, significant differences in the load-distance curve feature were obtained, as illustrated in Fig. 2 for a 90-degree pull and a 180-degree pull. The two methods yielded similarly a maximum of $\sim 12 \pm 0.4$ N/mm for the two central strips on the 4" x 4" borosilicate/EVA/TPT laminate (TPT: Tedlar™/Polyester/Tedlar trilayer). Although the 180-degree-pull curve tends to produce "cleaner" curves, close visual observations revealed four stages were involved in the process of pull-forced-delamination failure, which are better revealed in the 90-degree-pull.

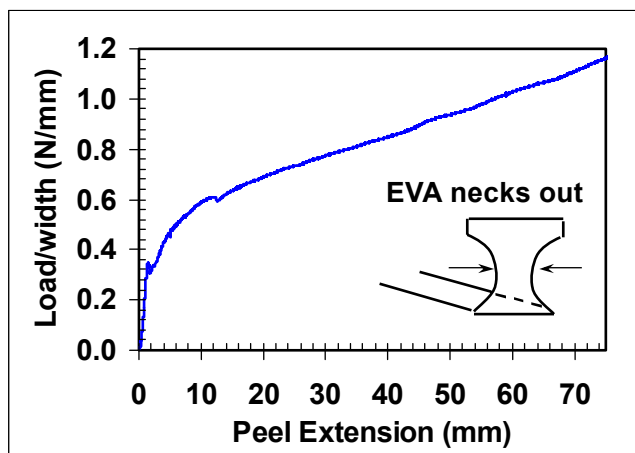


Figure 1. Load-peel distance curve for a borosilicate/EVA laminate *without* a backing foil, showing the necking (narrowing) of the soft, semi-elastic EVA layer.

The four-stage pattern can be generalized as below:

1. An initial phase that includes a short distance EVA pull-off (ca. 1~2 mm) from the glass surface, development of an EVA "flap," and lift-up or bending of the backfoil from EVA.
2. A second phase that includes extending-then-breaking of the EVA flap and more lift-off of the backfoil from EVA.
3. A third phase that involves the peel-off of backfoil from the EVA.
4. A fourth phase that shows delamination of the EVA-sided Tedlar layer from the PT layers.

These four phases or stages may not clearly show on each sample test. The second and third stages may be mixed,

resulting in "unzipping" of the EVA/TPT from the glass and TPT from the EVA, as shown in Fig. 2. Occasionally, the EVA flap lengthened too much and had to be cut with a blade to complete the test.

Because of these factors, the peel tests can only produce an "apparent" adhesion strength as a consequence of the EVA-on-backfoil combination. The results reported herein, therefore, are referred to this apparent adhesion strength. Furthermore, the maximum adhesion strength was derived from the highest load point within the first 1 cm pull distance for most samples, and within 1~2 cm for a few others. Comparison of the results should be more relevant for a given specific set of samples that were prepared together in a given lamination run.

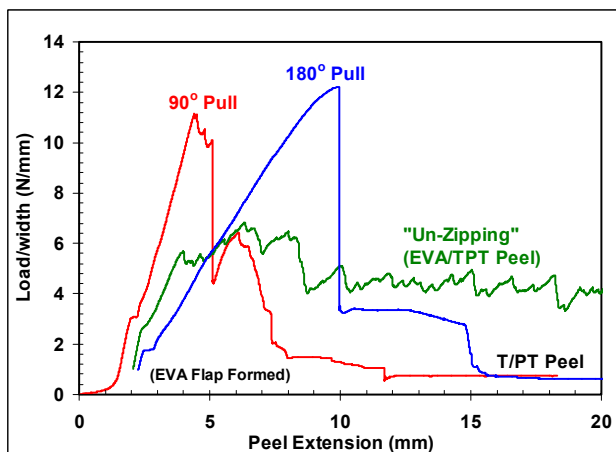


Figure 2. Load-peel distance curves for a borosilicate/15295P EVA/M-TPT laminate tested by a 90-degree-pull method, a 180-degree-pull method, and an "unzipping" of the EVA/TPT layers from the glass substrates upon 90-degree-pull.

3.2. Factors that Affect the EVA-to-Glass Adhesion

The apparent adhesion strength of EVA to glass substrate was found to depend on several factors. The most important ones are the types of encapsulants and backing foils used and their manufacturing. For EVA, the effects arise from the vinyl acetate (VA) content of the EVA, chemical formulations of EVA, presence and type of adhesion promoter such as silane coupling agent, and extent of EVA cross-linking (i.e., gel content). These are also true for the non-EVA polymers such as PEBA and PEMA studied here. On the backing foils, it was found that the actual manufacturing conditions and method can have a profound effect even for an identical type of backfoil such as TPT (Tedlar/polyester/Tedlar) trilayer laminate. Effects of glass type (for 1/8" thickness), surface cleaning, and texture are not so obvious. Direct priming on the glass surface did not improve, sometimes even worsened, the EVA adhesion to the glass substrates.

3.2.1 Effects of Encapsulant Type and Formulation

Table 1 shows the maximum apparent adhesion strength obtained for a number of glass/EVA (or non-EVA)/TPT laminates using 90-degree-pull test. The EVA formulations typically contain a peroxide curing agent for

cross-linking, to enhance the mechanical strength of the EVA layer to fixate and support the crystalline-Si solar cell strings, and a silane-based coupling agent, to enhance the adhesion between the EVA and substrates including the glass plate and solar cells. Most EVA formulations use a Dow Corning Z-6030TM, gamma-methacryloxy-propyl-trimethoxysilane. Details of the formulation are not readily available here for each of the commercial EVA products, however.

As seen in Table 1 for the fast-cure EVAs, 28%VA-EVA (TW0312A, custom formulated and extruded at NREL) yielded higher adhesion strength (>10 N/mm) than those of 33%VA-EVA (6~8 N/mm). The results for 33%VA-EVA are close to those reported elsewhere [2,3]. However, if the 28%VA-EVA contains an anti-block agent (EVX240W, DuPont's Elvax 240W), substantial reduction resulted (3~4 N/mm). Extent of EVA cross-linking does not appear to make much difference. For the slow-cure EVAs, the 33%VA-EVA and 28%VA-EVA appear to be fairly similar in producing the adhesion strength within the experimental uncertainty (*ca.* ± ~1 N/mm). For slow-cure A9918P EVA aged 5 years in storage, the gel content decreased and the adhesion strength also decreased to 3.4~4.2 N/mm. Blank polymer films unformulated with any ingredients such as 33%VA Elvax 150 (DuPont), PEBA, and PEMA all show very low adhesion strength, apparently a consequence of a absence of a silane coupling agent and curing agent for cross-linking. After a fast-cure formulation with silane and curing agent, adhesion strength of PEMA increased substantially to ~8 N/mm.

Table 1. Adhesion Strength for Various Encapsulant Formulations¹ in Borosilicate/EVA/M-TPT Laminates Measured with 90-Degree-Pull

Plate Size (in.x in.)	EVA Type	Formulation ID	Ad. Strength (N/mm)		Gel (%)
			Test 1	Test 2	
Fast Cure					
2 x 3	33%VA	15295P	8.07	6.02	77~89
2 x 3	33%VA	ZJ-FC	7.42	6.60	80
2 x 3	33%VA?	BP-FC	8.13	7.04	70
2 x 3	28%VA	TW0312A	10.88	10.25	87
2 x 3	28%VA	EVX240W	3.39	4.33	70
2 x 3	28%VA?	HS SC-42 ³	7.67	6.49	89
2 x 3	Non-EVA	PEMA	8.37	7.69	70
Slow Cure					
4 x 4	33%VA	ZJ-SC	8.58	9.93	80
4 x 4	33%VA	A9918P ⁴	4.21	3.42	74~79
4 x 4	28%VA	SC-0515C	6.78	8.21	77~83
2 x 3	28%VA?	HS SC-42 ³	5.89	7.53	89
Unformulated					
2 x 3	33%VA	Elvax 150	4.92	4.98	0
2 x 2	Non-EVA	PEBA	1.79	1.64	0
2 x 2	Non-EVA	PEMA	2.84	2.29	0

¹ FC: Fast cure; SC: Slow cure. The EVA films are made by different sources with varying formulations.

³ Percent VA in the HS EVA was not clear even from DSC analysis, but likely 25%~28%. Used in both fast and slow cure.

⁴ Films stored in the dark for ~5 years.

3.2.2. Effects of Backing Foil Type and Source

The backing foils were found to have the greatest effect on adhesion strength, as shown in Fig. 3 and Table 2. The type, manufacturing source, and surface priming on the adhering side of the back foils all affect the adhesion strength between glass and EVA/backfoil and between EVA and the backfoil.

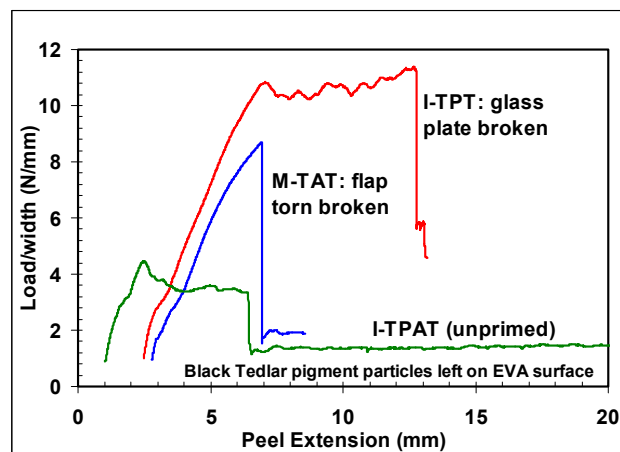


Figure 3. Load-peel distance curves illustrating the effect of backfoil type on the adhesion test of borosilicate/15295P/backfoil laminates using 90-degree-pull method. The backfoil was (a) I-TPT, (b) M-TAT (Tedlar/aluminum foil/Tedlar), and (c) I-TPAT (Tedlar/polyester/aluminum/Tedlar) with one black pigmented, unprimed Tedlar side.

Table 2. Effects of Backing Foil Type and Manufacturing Source

Glass Type (1/8" thick)	Plate Size (in.x in.)	Backfoil Type	Max. Ad. Strength (N/mm)	
			Test 1	Test 2
AFG-KK	2 x 3	M- TPT	4.21	6.83
AFG-KK	2 x 3	I-TPT ¹	10.51	10.83
AFG-KK	2 x 3	M-TAT ²	8.70	8.00
AFG-KK	2 x 3	I-TPAT ³	5.24	4.45
Boro	2 x 3	M-TPE ⁴	11.91	11.73
Boro	2 x 3	Teijin 6429	9.24	10.82

¹ Vertical white strips left on EVA surface; glass plate broke on the second strip test.

² TAT/EVA strip broke from pull by tearing apart from one side.

³ Fine black pigment powder residues left on the EVA layer.

⁴ The glass plate broke on the second strip test.

Mechanically strong backfoils such as I-TPT and M-TPE could result in glass-plate breaking, and weak backfoils such as TAT (Tedlar/aluminum foil/Tedlar) would be torn broken during the tests. The relatively weak bonding between the black-pigmented Tedlar with EVA on I-TPAT (Tedlar/polyester/aluminum foil/Tedlar) resulted in low adhesion because the TPAT backfoil peeled off from EVA fairly easily, leaving the black-pigmented Tedlar particles on the EVA surface. Teijin 6429 film performed similar to a typical TPT laminate [6]. Once the EVA-sided Tedlar layer of a TPT or TPE backing foil was peeled off, the adhesion strength of the PT or PE layers to the Tedlar layer was obtained, which was typically <1 N/mm.

3.2.3. Effects of Glass Type, Surface Cleaning, Treatments, and Textures

Table 3 shows the effects of glass type, surface texture, cleaning method, and priming treatment on EVA adhesion. For borosilicate glass plates, cleaning methods by IPA, sulfuric chromic acid, 2.0 N NaOH soaking, and Liqui-Nox,TM followed by no priming or by 2% Z-6030 priming, did not appear to make a significant difference in the EVA/boro adhesion strengths, which were in the $7 \pm \sim 1$ N/mm range. Similar results were obtained (6~7 N/mm) for 1/16"-thick soda lime microslide plates.

Table 3. Effects of Glass Type, Cleaning Method, Surface Treatment, and Texture on the Adhesion Strength of Glass/15295P/M-TPT Laminates Using 90-Degree-Pull

Glass Type (1/8" thick)	Plate Size (in.x in.)	Cleaning Method	Surface Priming	Max. Ad. Strength (N/mm)
Boro	1 x 3	IPA		6.44
Boro	1 x 3	IPA	Z-6030	6.74
Boro	1 x 3	IPA	PECMA	5.14
Boro	1 x 3	IPA/Chrom Acid	Primer C	6.21
Boro	1 x 3	Chromic Acid	Z-6030	6.88
Boro	1 x 3	2.0 N NaOH	Z-6030	6.51
Boro	2 x 3	Liqui-Nox		8.07, 6.02
Boro	2 x 3	Liqui-Nox	Primer-F	5.59, 5.67
Boro	2 x 3	Liqui-Nox	Primer-G	3.74, 5.04
Boro	2 x 3	Liqui-Nox	Primer-H	1.05, 0.88
AFG KK	2 x 3	Liqui-Nox		5.54
AFG KK	2 x 3	Liqui-Nox	Silq. A187	1.69
AFG KK	2 x 3	Liqui-Nox	Primer A	4.64
AFG KK	2 x 3	Liqui-Nox	Z-6030	5.54
AFG KK	1 x 3	Liqui-Nox		4.52
AFG Solatex	1 x 3	Liqui-Nox		4.88
AFG Solite	1 x 3	Liqui-Nox		4.61
Starphire	2 x 3	Liqui-Nox		7.00
Solarphire	2 x 3	Liqui-Nox		7.57
Microslide	1 x 3	IPA	(1/16"-thick)	6.30
Microslide	1 x 3	IPA	Z-6030	6.92
Microslide	1 x 3	IPA	Primer C	5.86

Surface priming of the borosilicate glass surfaces by dipping 2~3 min in custom-formulated solutions (PECMA, and Primers-C, F, G, and H), followed by IPA rinsing and oven heating at 85°C for ~15 min, could affect strongly the EVA adhesion, depending on the primer solution's formulation. Therefore, the surface affinity properties for EVA, which involve siloxane and hydrogen bonding and cross-linking through the Z-6030 silane, appear to be critical. Use of additional molecular methacrylate-type cross-linkers in Primer G and H seems to "block" the typical siloxane and cross-linking between the glass and EVA. This was also observed for the AFG-KK glass that was directly primed with a Silquest A-187TM 3-glycidoxypropyltrimethoxysilane, whereas priming with Z-6030 or Primer A made no or little difference. The test results with AFG's cerium oxide-doped glasses—plain Krystal KlearTM, mildly textured Solatex IITM, and pyramid-textured SoliteTM—show that the EVA adhesion strength appears to be insensitive to the surface texture, which is likely a consequence of the soft, semi-elastic EVA film as discussed above. EVA adhesion to the PPG's

StarphireTM and CeO_x-doped SolarphireTM was similar to the borosilicate glass, but stronger than AFG's soda-lime glasses.

3.3. Weak Adhesion of Fluoropolymer Thin Films

Adhesion of three fluoropolymer thin films to EVA and/or glass surfaces was also studied. TefzelTM films have been used for years as superstrates for thin-film a-Si PV modules, and DureflexTM and Dyneon's THV films (both based on Dyneon's THV220TM fluorinated terpolymer) have been tried recently as super- or substrates or even to replace entirely the EVA/Tefzel combination [6]. The results indicate all three films bonded weakly to the EVA at a level of ~1.3 N/mm for the Tezel and Dureflex, and ~2.6 N/mm for the 3-mil THV film. The adhesion of Dureflex film to a cleaned glass surface was very low at ~0.4 N/mm.

4. Conclusions

The extensive adhesion strength study has clearly demonstrated that the apparent adhesion strength of EVA/backfoil to glass substrates is affected by a number of factors, which include EVA type, formulation, backfoil type and manufacturing source, glass type, and surface priming treatment on the glass surface or on the backfoil. The results indicate that the adhesion test is critically dependent on the use of a mechanically "strong enough" backing foil on the EVA to achieve a meaningful peel test. The peel-test method with a 90-degree-pull yielded similar results to a 180-degree-pull. Effects of the glass-cleaning method and surface texture are not obvious. Fluoropolymer thin films show low adhesion to the EVA or glass. Based on our results and observations, we conclude that the adhesion strengths derived from the peel tests are comparative at best. In addition, it is recommended that all relevant conditions and materials information be clearly specified when an adhesion strength is cited or reported. More details will be presented in the Review Meeting.

5. Acknowledgement

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