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RESEARCH ARTICLE

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Using a butt joint test to evaluate photovoltaic edge seal adhesion

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Abstract

Many photovoltaic (PV) technologies have been found to be sensitive to moisture that diffuses into a PV package. Even with the use of impermeable frontsheets and backsheets, moisture can penetrate from the edges of a module. To limit this moisture ingress pathway from occurring, manufacturers often use a low permeability polyisobutylene (PIB)-based edge seal filled with desiccant to further restrict moisture ingress. Moisture ingress studies have shown that these materials are capable of blocking moisture for the 25-year life of a module; but to do so, they must remain well-adhered and free of cracks. This work investigates the potential use of a butt joint test for evaluating the long-term durability of adhesion by looking for significant changes in the failure mode or quantitative value of a butt joint test. A round robin experiment was conducted using six different materials and two sample constructions, with and without effort to control edge pinch. Tests were evaluated looking at the strength of the bond, and the type of failure observed in a round robin test involving five laboratories. It was found that both the measured values, and the observed failure modes were repeatable and reproducible within at 95% confidence interval.

KEYWORDS

adhesion, butt joint, edge seal, hermetic, moisture ingress, permeation

1 | INTRODUCTION

Many thin-film photovoltaic (PV) materials are sensitive to moisture, most importantly those made of CdTe and Cu(In,Ga)S_2.[1] To prevent moisture ingress, impermeable front and backsheets are used, but this still requires the use of an edge seal to prevent ingress from the sides. To function, an edge seal must have a long moisture breakthrough time and/or very low permeation rate, remain adhered to surfaces; and not fracture to create moisture ingress pathways.[2] The current PV industry general practice for evaluating the mechanical durability of an edge seal is to use a lap shear test as specified in IEC 61730-2 MST 36.[3,4] IEC 61730-2 considers a material to have passed if it retains 50% of its initial strength after exposure to the following five conditions in series: 200 hours damp heat MST 53, 60 KWh/m² UV MST 54, 10 humidity freeze cycles MST 52, another 60 KWh/m² UV MST 54, and another 10 humidity freeze cycles MST 52.

The lap shear test raises several concerns. Typically, edge seal materials fail cohesively and have a high dependence of the maximum shear stress on the pull rate. The testing pull rates used in these tests are significantly higher than the shear stress in a PV module, thus the values are not relevant to field use. Also, for most PV applications, the failure mode is dominated by tensile forces, not shear, and it is characterized by the propagation of a crack. In prior work,[5] a Boeing Wedge type of test [6–9] was evaluated because it caused delamination at a rate and in a method consistent with field failure. However, this method required the use of engineering samples, suffered from significant repeatability and reproducibility issues, and demonstrated that the fracture energy of adhesion of these materials was extremely low (between $1\ \text{J/m}^2$ and $10\ \text{J/m}^2$). The polyisobutylene (PIB)-based edge seals used in the PV industry are typically noncrosslinked and filled with desiccant. PIBs have a glass transition temperature of around −75°C [10,11] and no melt transition. Consequently, they have very low cohesive strength resulting in extremely low fracture energy for delamination making them inconsequential for providing any mechanical support to a module and making evaluation of the actual value for the fracture energy, or butt joint strength in this case, irrelevant with respect to module integrity. To really evaluate the durability of an edge seal in a given application the nuances of the mechanical attributes of module construction must be accounted for. The edge seal is not providing mechanical strength but simply needs to maintain its position in the module after the application of thermal and mechanical stresses. The aim of adhesion testing of coupons was to verify that properties are not changing and that an aged module could be reasonably expected to perform similar to an unaged one. Thus, even though the wedge test gives quantitative and more relevant values, a simpler more reproducible method can evaluate whether major mechanical changes are happening or if the failure mode has changed, in line with the philosophy behind MST 36.

One of the concerns with MST 36 is that it frequently requires the use of engineered test samples as opposed to actual production modules. Furthermore, if either the frontsheet or backsheet is flexible then a peel test structure must be used which as implemented does not delaminate in a representative manner to field use and is highly dependent on the mechanical properties of the adherend. To overcome these concerns, we are proposing the use of a butt joint test. This allows the use of the edge seal from any module type, even one with tempered glass, and applies the stress in the same primary direction (normal to the surface) as it is in service. Here, we perform a round robin test to determine if this method is reasonably repeatable and reproducible to be used as a standard methodology.

2 | EXPERIMENTAL

2.1 | Sample construction

All edge seal materials used in this work used a PIB matrix with a desiccant in them. Some desiccants used reactive chemistry and others used molecular sieve-based methods.[12–14] Edge seals were obtained from several different manufacturers. Four of the materials were supposed to be "good" and two of which were supposed to be known "bad" materials with respect to their ability to pass IEC 61730 [3,4] and IEC 61215 [15] and with respect to their ability to perform in the field.

Edge seal samples were laminated between two pieces of annealed glass with dimensions of at least 30 cm on each side. Edge seal was just located around the perimeter with an EVA encapsulant in the center portion. Each of the eight different material formulations were labeled as A through H. Most of the samples were laminated using a frame around the perimeter which reduced the amount of edge pinch to a < 0.05 mm difference in thickness between the edge area and the center area of the samples, Figure 1. Two of the "good" materials (A and F) were laminated poorly, intentionally giving them significant edge pinch with between 0.1 mm and 0.05 mm difference between the edge and center area thickness which is significant for a gap between the glass pieces of only about 0.46 mm, Figure 1. Thus, a total of eight different material/construction samples were utilized in the round robin experiment labeled as material "A" through "H". Materials D and E are PIB-based sealants used in constructing insulating glass units. D and E are called known "bad" materials having failed PV module durability tests. Materials A, B, C, F, G, H are PV edge seals where A and F were laminated with significant edge pinch.

2.2 | Removal of test specimens

Test samples were removed from the perimeter of the glass laminates. A water jet cutter would be ideal for this work, but a tile saw was utilized because of its convenient availability in our lab. To do this, two parallel cuts about 1 cm apart and about 7 cm deep were made from the sides and the 1 cm × 7 cm glass piece was broken off. Then, a second cut was used to remove an ~ 1 cm × 1 cm part consisting of glass/PIB/glass suitable for testing. All this cutting and sample preparation were conducted at one lab with the actual

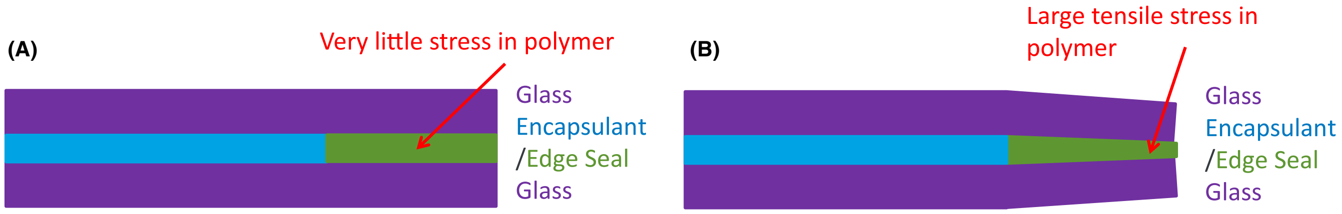

FIGURE 1 A, Schematic of properly laminated edge seal and; B, A laminated edge with severe edge pinch

assembly of test structures and delamination measurements performed as part of the round robin testing.[16] While this round robin experiment was performed using annealed glass, other work conducted in this group successfully used tempered glass which shattered upon cutting. The bolts in the test sample construction are adequate to hold together the fragments of tempered glass (Figure 2).

2.3 | Assembly of butt joint test structures

Glass/PIB/glass test specimens were adhered to zinc plated 5/16”-18 thread size, 1” long elevator bolts using Hardman general purpose two-part epoxy (MFR #: 4005-BG10). Here, care must be taken to ensure the test specimen is centered along the axis of the elevator bolts and that the two elevator bolts are centered and aligned, Figure 3. Placing the epoxy first onto the glass prior to contact with the bolt minimized the movement of the sample as it cured. A sample holder consisting of a metal plate with holes in it fixes the bottom elevator bolt in a vertical position while the test specimens are adhered in the center of the bolt. Then, once the adhesive is sufficiently cured, the second elevator bolt is adhered to the top of the test structure. Here, care must be taken to cover the whole surface but not to have extra epoxy that flows down to span the gap between the bolts and not so much epoxy that the bolts rotate or slide prior to the setting of the epoxy. Once cured, visual inspection is performed, and poorly aligned samples are discarded. This assembly procedure was performed by all round robin participants.

3 | RESULTS AND DISCUSSION

3.1 | Determination of appropriate pull rate

Field failure of PV modules involving delamination or edge seal failure happens over a long time-frame; therefore, to get representative values for adhesion, where viscous flow of the polymer does not affect the measurement, one would want to use extremely slow testing pull rates. To investigate this possibility, we measured all eight materials and constructions at pull rates from 0.01 mm/min to 1000 mm/min, Figure 4. At the very lowest pull rates, the maximum stress continues to decrease with a power law relationship to the pull rate. This indicates that the maximum stress is dominated by the viscoelastic response of the polymer. This happens for all the materials meaning that any reasonable choice of pull speed, which can be utilized in typical equipment, will still be arbitrary in that it will not represent the stresses and strain rates applicable when failure is observed in fielded modules.

At the high end of pull rates, the upward trend of maximum stress (σ_{max}) hits a plateau between 100 and 1000 mm/min. Some materials show a smaller value at 1000 mm/min vs 100 mm/min supporting the idea that this is just an experimental anomaly. Complete failure of these materials happens when the glass plates have been pulled by less than 1 mm, thus the compliance of the fixture and inertial effects begin to be very important at these high pull rates. Assuming this load frame response is typical or at least common, using such high pull rates is not a viable option.

For material A, five replicate measurements were made by one lab at pull rates from 0.01 mm/min to 1000 mm/min to determine if there was an optimal pull speed, Figure 5. For this material there appears to be a trend for more consistent data at lower pull rates with one outlier at 10 mm/min. Considering that the data at 1000 mm/min are highly suspect because of instrumental limitations, caused by compliance and inertial effects, this higher variability is not relevant making the trend reasonably possibly just an experimental anomaly.

A number of materials, A, B, C, and E showed a transition from 100% cohesive failure at low strain rates beginning at pull rates of <0.01 mm/min, 1000 mm/min, 100 mm/min, and 10 mm/min, respectively. At higher pull rates the stress applied to the polymer is increased because of the strain rate dependence of the polymer viscoelastic properties. As the stress increases it eventually becomes large enough for the failure mode to switch from cohesive to adhesive as the material begins to behave more like a solid. For all but one material this transition began at pull rates less than 10 mm/min. It was discussed to run the pull tests at these higher pull rates to be able to probe/evaluate the less desirable adhesive failure but considering that instrumental limitations begin to

FIGURE 2 Photos showing specimen removal using a tile saw

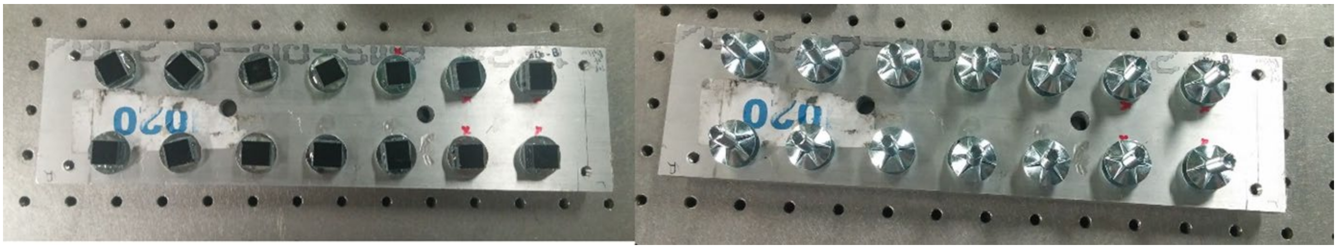

FIGURE 3 Photo of assembly of test structures. Samples were fixed in the load frame using grips designed for holding rods. The threading of the bolts helps to aid in securing the test structures

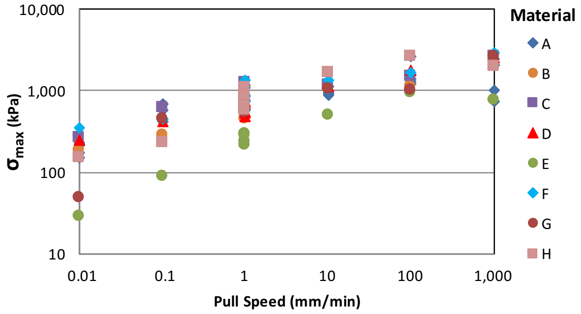

FIGURE 4 Evaluation of pull speed on measured butt joint strength

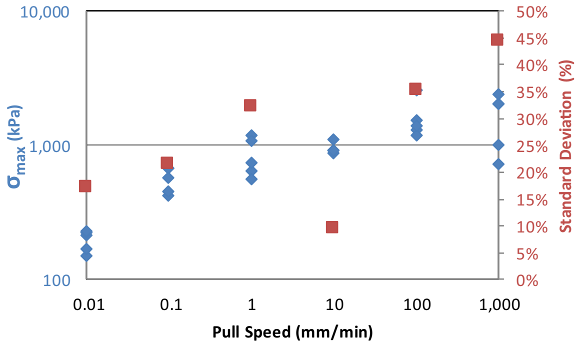

FIGURE 5 Measurement of PIB-A as a function of pull speed with five replicates for each decade alongside the percent standard deviation at each pull speed

be important and that this further deviates from failure rates and modes seen in the field, it was decided to use a lower pull rate commonly favoring cohesive failure modes.

With these considerations, a pull speed of 1 mm/min was chosen for the round robin experiment. This was sufficiently far away from rates where there are instrumental limitations and about as low as practical given the excessive time needed to pull at 0.01 mm/min.

3.2 | Evaluation of failure mode

In addition to measuring the maximum stress, the failure mode is also important for assessing changes in properties. For example, if the failure mode changes from cohesive to adhesive even without changes in the max stress, this is a sign that there may be significant changes in the mechanical properties of a material as it ages.[17] Documentation of the failure mode with photos is highly recommended for clearer meaning of the rating as well as for comparison of the mode change after exposure tests.

Round robin participants were shown images of examples of different failure modes, Figure 6, to aid in interpretation of their results.[18] With adhesive failure, Figure 6A, typically all the material ends up on one side of the test specimen leaving an extremely clean surface. Frequently, the adhesive ends up split between the two surfaces with some clean area on both surfaces, Figure 6B. Here, one must determine the percentage adhesive failure by summing up the total amount of adhesive area on both pieces of glass. For example, in Figure 6B, one side is showing about 10% adhesive, and the other glass side has about 60% for a total of 70% adhesive failure and 30% cohesive failure. In many other cases, the edge seal fails completely cohesively, Figure 6C, leaving material on both glass surfaces. This was the general level of instruction given to the participating laboratories for evaluating the failure mode.

3.3 | Round robin results

Each of the participants were sent six or more pieces of all eight material samples precut to ~1 cm × 1 cm glass/PIB/glass test samples and a set of instruction. Each lab was responsible for assembling at least five test samples onto bolts, testing them in a load frame at 1 mm/min, and evaluating the results. This round robin experiment was evaluating the ability to reproducibly and repeatably assemble samples and to identify/duplicate the failure modes reproducibly and repeatably.

The summary statistics for the round robin are shown in detail in Table 1 including both maximum stress and failure mode characterization expressed as the percentage cohesive failure. The analysis was performed in accordance with ASTM E691 which determines if the method is repeatable and reproducible. Repeatable means that each laboratory can perform the experiment with a similar standard deviation and that the given results are consistent with the standard deviation within a 95% confidence interval. Reproducible means

FIGURE 6 Examples of different failure modes. A, 100% adhesive failure. B, ~70% adhesive ~30% cohesive failure. C, 100% cohesive failure

Material	Method	$\bar{\bar{X}}$	$S_{\bar{x}}$	Sr	S_R	r	R
A	Stress (kPa)	890	290	250	370	710	1000
	% Cohesive failure	9.2	16	11	19	31	52
B	Stress (kPa)	880	340	320	450	900	1300
	% Cohesive failure	100	0	0	0	0	0
C	Stress (kPa)	880	230	230	310	640	870
	% Cohesive failure	100	0	0	0	0	0
D	Stress (kPa)	800	310	190	350	530	990
	% Cohesive failure	15	9.7	16	17	44	48
E	Stress (kPa)	400	170	91	190	250	520
	% Cohesive failure	100	0	0	0	0	0
F	Stress (kPa)	1000	280	300	390	830	1100
	% Cohesive failure	0	0	0	0	0	0
G	Stress (kPa)	730	180	140	210	380	600
	% Cohesive failure	37	48	4	49	11	140
H	Stress (kPa)	830	110	150	180	420	490
	% Cohesive failure	100	0	0	0	0	0

TABLE 1 Butt joint round robin results from five labs for eight materials summary statistics $\bar{\bar{X}}$ is the standard deviation of all measurements, S_r is the repeatability standard deviation (within a lab), S_R is the reproducibility standard deviation (between labs), r is the 95% repeatability limit, and R is the 95% reproducibility limit[16]

that the differences between measurements at different laboratories are possible within a 95% confidence interval given the inherent testing variability.

Material F, failed adhesively at all strain rates, but had the highest maximum stress value of 1000 kPa. This indicates that F had a high cohesive strength and likely a reasonably high adhesion strength that is higher than materials A, D, and G which had a mixed failure mode. However, it is not possible to know if its adhesion strength is higher than the materials that always failed cohesively, B, C, E, H.

The known bad materials, D and E, had maximum stresses of 800 kPa and 400 kPa, respectively. Material E failed cohesively which means that the low adhesive strength does not necessarily predict failure because it is possible that when used in a module, the applied strains might not be large enough to tear it apart. Material D had an average value for its maximum stress but because of the presence of some adhesive character in the failure mode, these results indicate it is likely to have lower adhesive strength than the rest of the materials. However, this is insufficient to determine conclusively if it is a bad material.

Prior to being cut out of the module, the edge seal may or may not be under stress. PIB-based edge seal can flow in response to strain and one would expect the bulk of the actual curvature, and hence tensile/compressive strain to be accomplished by the well-bonded encapsulant leaving the glass at the perimeter flat but possibly at an angle to produce edge pinch. However, once cut, the glass in the edge

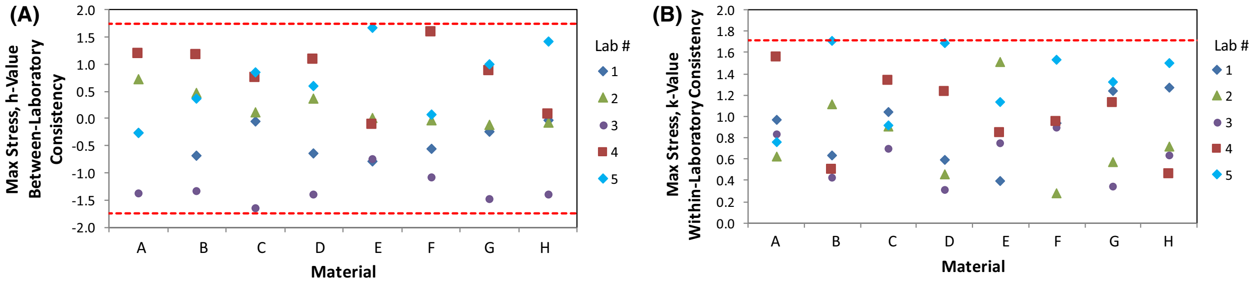

FIGURE 7 Maximum stress results. The dashed red line indicates the limit for which to a 95% confidence limit the given results are expected for the given method variability. A, Reproducibility or between-laboratory consistency. B, Repeatability or within-laboratory consistency

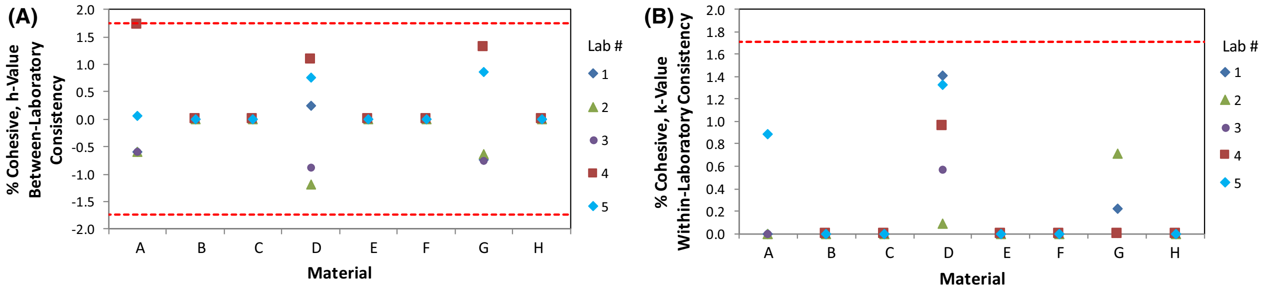

FIGURE 8 Failure mode evaluation results. The dashed red line indicates the limit for which to a 95% confidence limit the given results are expected for the given method variability. A, Reproducibility or between-laboratory consistency. B, Repeatability or within-laboratory consistency

seal test samples should be nearly completely flat. However, it is the nonparallelism of the glass pieces that is of concern potentially creating greater strain in the polymer on one side relative to the other. If significant, this would be expected to decrease the measured value and increase the variability in the measurement.

Materials A and F were poorly laminated to intentionally give them greater edge pinch. The percent uncertainty in their strength was 33% and 28%, respectively, compared to an average of 30 ± 9%. Thus, the edge pinch lamination did not show a statistically significant difference in the measurement uncertainty. Similarly, materials A and F had maximum values of 890 kPa and 1000 kPa, respectively, compared to an average of 870 ± 84 kPa excluding the "bad" samples. This test does not appear to be sensitive to the lamination conditions that produce moderate amounts of edge pinch. Defects in module construction resulting in mild amounts of edge pinch would not be detected by this test. However, this means that if used as a measure for cemented joint evaluation the presence of edge pinch will only be a factor if it is severe enough to spontaneously promote delamination.

Figures 7 and 8 show the results for the different labs for both maximum stress and the failure mode. In few cases, the results are barely repeatable or reproducible within a 95% confidence interval for both the max stress and the failure mode. Laboratory #5 had more concerns than the others but is still not a statistically significant outlier. Laboratory #4 deviated a little more than the others, especially for the mode identification, but again not statistically significant. These results indicate that the butt joint method repeatably and reproducibly can be used to evaluate the adhesion of PIB-based edge seals.

4 | CONCLUSIONS

If an edge seal is considered part of a cemented joint, it must pass an adhesion test as part of IEC 61730. Passing this test enables smaller distances through cemented joints to be used as opposed to creepage and clearance distances. Currently, a lap shear or a peel test is used in the assessment of cemented joints. This work demonstrates that a butt joint may be a good substitute having good reproducibility and repeatability. This is advantageous because it can be done directly on production modules. The method can also be applied on engineered butt coupons, constructed as for this study, for material assessments purposes as well. Alternatively, an engineering sample could be made with small pieces of superstrate and substrate precut prior to lamination.

It should also be noted that this test is not intended to be the final determining factor for whether or not an edge seal is adequate. The adhesion strength of these materials is so small that they should be ignored when considering if the PV package is adhered well enough to stay together, a module level test is needed to determine the overall adequacy of a design which includes an edge seal.[5] The adhesion test is intended to be used to say the adhesion is not changing significantly giving reasonable expectation that it can perform its intended function.

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