Degradation in PV Encapsulation Strength of Attachment: An Interlaboratory Study Towards a Climate-Specific Test

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Goal and Activities for PVQAT TG5 (UV, T, RH)

- IEC 61215-2 qualification test prescribes 54 $MJ \cdot m^{-2}$ (~40 days equivalent) IEC 60904-3 AM 1.5G UV radiation dose. IEC 61730-2 safety test: 4x.
  This is << 25 year warranty!

⇒International PV Quality Assurance Task Force (PVQAT)

- **TG5 Goal**: develop UV- and temperature-facilitated test protocol(s) that may be used to compare PV materials, components, and modules relative to long-term field service.

Applications:

- IEC 62892- (module comparative test).
  - -3: UV weathering of encapsulants
  - -4: Start of "Leg 2" test series to query delamination.
    (UV→CML (formerly DML)→HF10→DH500)

- IEC 62788-7-2 (UV weathering of PV materials and components).
  Accelerated aging test(s) for encapsulation, backsheet, adhesives...

Goals for the TG5 Experiment

- Unlike the degradation of encapsulant transmittance, the accelerated conditions and duration to examine delamination are not established.

Goals for the interlaboratory experiment:

1. Quantify the relative significance of factors including UV, T, RH, and time.
   Which factors most reduce strength of attachment during weathering?
   What range of factor values should be applied for aging?

2. Determine if there is significant coupling between UV, T and RH.
   What factors should be applied in a weathering test for attachment?

3. Investigate the spectral requirements for UV light sources,
   e.g., compare specimens aged by Xe-arc and UVA-340 lamps.
   Does visible light affect aging?

Encapsulant Used in the TG5 Experiment

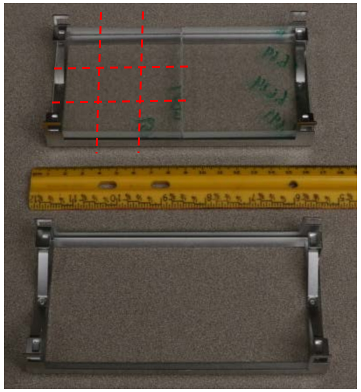
- 1 material examined in interlaboratory study.
- "EVA-B" formulation virtually identical to STR 15295P/UF "fast cure" EVA.
- 15295P/UF commonly used through 1990's, *i.e.*, many veteran installations.

INGREDIENT	DESCRIPTION	MAKER	MASS {g}
Elvax PV1400	EVA resin, 33 wt% Vac	E. I. du Pont	100
Z6030	silane primer, gamma-methacryloxy propyl trimethoxysilane	Dow-Corning Corp.	0.25
TBEC	curing agent, OO-Tertbutyl-O-(2-ethyl-hexyl)-peroxycarbonate	Arkema Inc.	1.5
Lupersol 101	curing agent, 2,5-Bis(tert-butylperoxy)-2,5-dimethylhexane	Arkema Inc.	N/A
Tinuvin 329	UV absorber, benzotriazole type	BASF Corp.	N/A
Cyasorb UV-531	UV absorber, benzophenone type	Cytec Industries Inc.	0.3
Tinuvin 770	hindered amine light stabilizer (HALS)	BASF Corp.	0.1
Tinuvin 123	non-basic aminoether-hindered amine light stabilizer (NOR-HALS)	BASF Corp.	N/A
Naugard P	anti-oxidant (AO), phosphite containing	Chemtura Corp.	0.2
		Designation (Note)	EVA-B (improved, "fast cure")

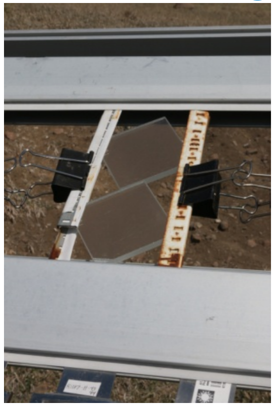
Encapsulant material being examined in the interlaboratory attachment strength experiment.

Details of the TG5 Methods and Experiment: Encapsulant CST Attachment Test

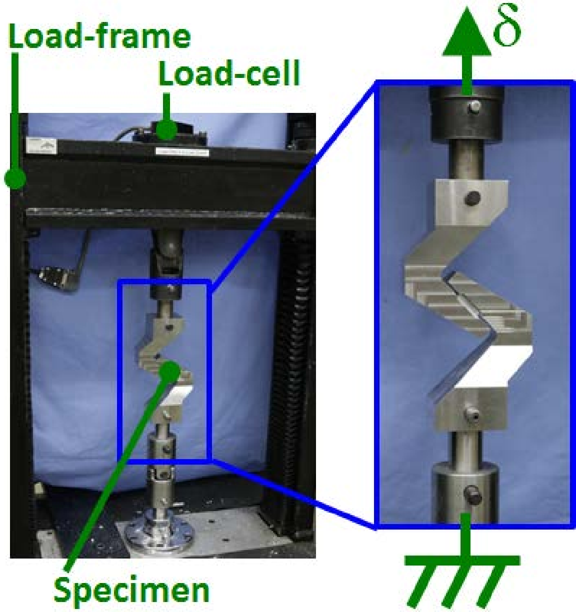
- The Compressive Shear Test (CST) was chosen to quickly survey the UV weathering parameters for attachment of EVA encapsulation.
- Share TG5 weathering between 12 participants. CST performed by NREL.
- 25 mm square specimens (diced, after aging) examined using load-frame.
- Pristine edge quality is critical. Dice using abrasive water jet cutter.



Sample holder configuration for indoor aging at NREL. Samples are diced after aging.



Specimens on outdoor rack, aging in Golden, CO at NREL.



The CST will be used to examine the attachment of EVA. Method from: Chapuis *et. al.*, PIP, 22 (4), 2014, pp.405–41. (EPFL)

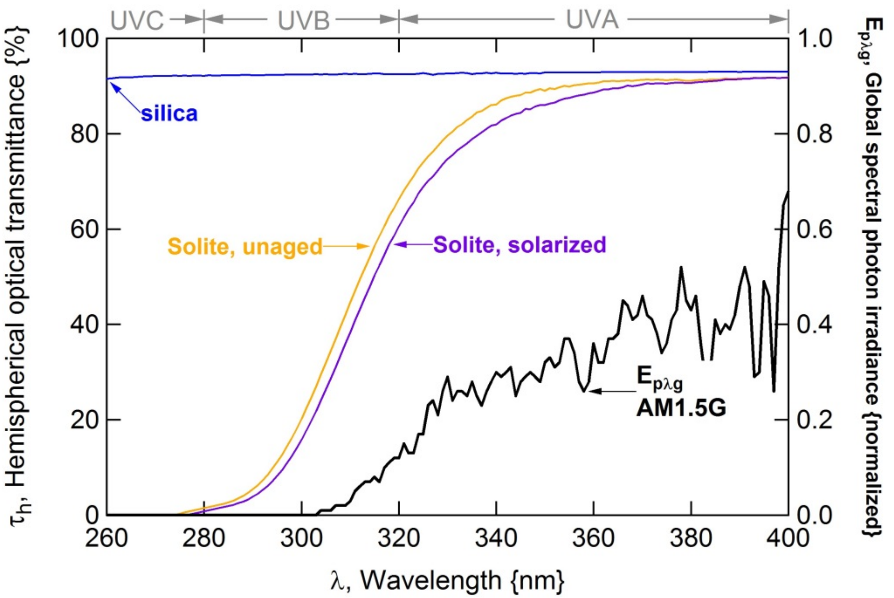
Movie of test: https://pfs.nrel.gov/main.html?download&weblink=ff9d3efc4a89183e8283e5ca01edbf8a&realfilename=IMG_0529.MOV

User summary:

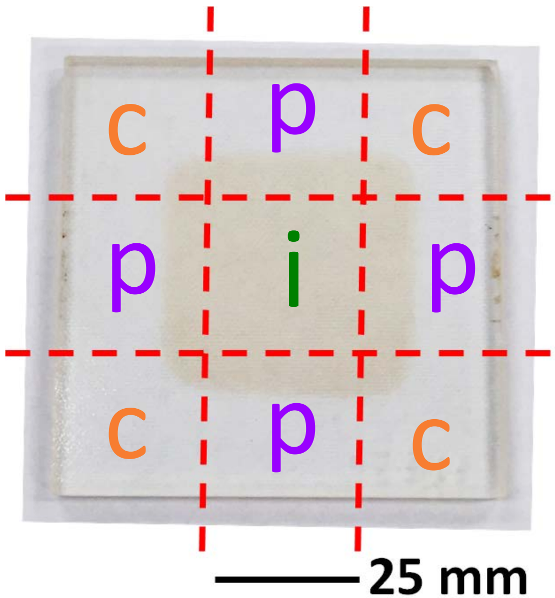
- Geometry: glass/polymer/glass (3.2 mm/0.5 mm/3.2 mm)
- Size: 3” x 3”
- Quantity: 10 replicates of 1 material (H_2O pre-conditioned)
- Aging: 15, 30, 45, 90, 135, and 180 cumulative days (indoors), or 1, 2, 3, 4, 5 years (outdoors)
- Remove 2 coupons at each increment
- Measurements (destructive): age at each laboratory/test site, then sent to NREL for measurement.

Specimens Used in the TG5 Experiment

- Textured Solite (AGC Solar/Asahi Glass Co.) superstrate and substrate.
- Solite PV glass attenuates UV.
- Solite solarizes slightly ($\Delta\lambda_{cUV}$: 295→297 nm) through the experiment.



Comparison of silica (for transmittance specimens) and Solite (for CST) glass, sown relative to the AM1.5G spectrum.



Photograph of discolored aged coupon, showing the approximate locations of interior (i), periphery (p), and corner (c) specimens.

- Specimen coupons are diced to be able to compare periphery and interior.
- 2 coupons aged each duration. ≥(5) replicates are used for the periphery measurements.
- Alternate the orientation of periphery specimens through experiment.

Policy for Moisture-Conditioning of Specimens

- Water content may be a critical factor in the experiment.

⇒ maintain stable water concentration through experiment.

- A set of 4 different conditions were applied to render an internal water content (ppm) similar to that during aging.

Temperature (°C)	RH (%)	Saturation at RH (g/cm^3)	Concentration Ratio (vs. 40/30%)	Dew Point (°C)	Temperature (°C)	Humidity (%)	Recommendation(s)
40	30	0.000882	1.0	-9.4	30	45.8	Put in Refrigerator
60	30	0.001297	1.5	4.6	30	72.7	Put in Refrigerator
60	50	0.002162	2.5	25.7	30	90.9	
					23	106.3	Put in sealed jar above water (not in water), at ambient T.
80	30	0.001826	2.1	18.4	23	89.8	Put in sealed jar above water (not in water), at ambient T.
40	50	0.001470	1.7	9.5	25	69.1	Put in 25C/69.1% chamber
80	50	0.003043	3.5	41.7	40	103.5	Put in a jar at 41.7C and 100% RH
					45	93.6	Put in 45C/85% chamber

Summary of the pre-conditioning/storage conditions for samples for the TG5 experiment. Conditions prescribed based on Kempe et. al., Proc. IEEE PVSC, 2013, 120-125.

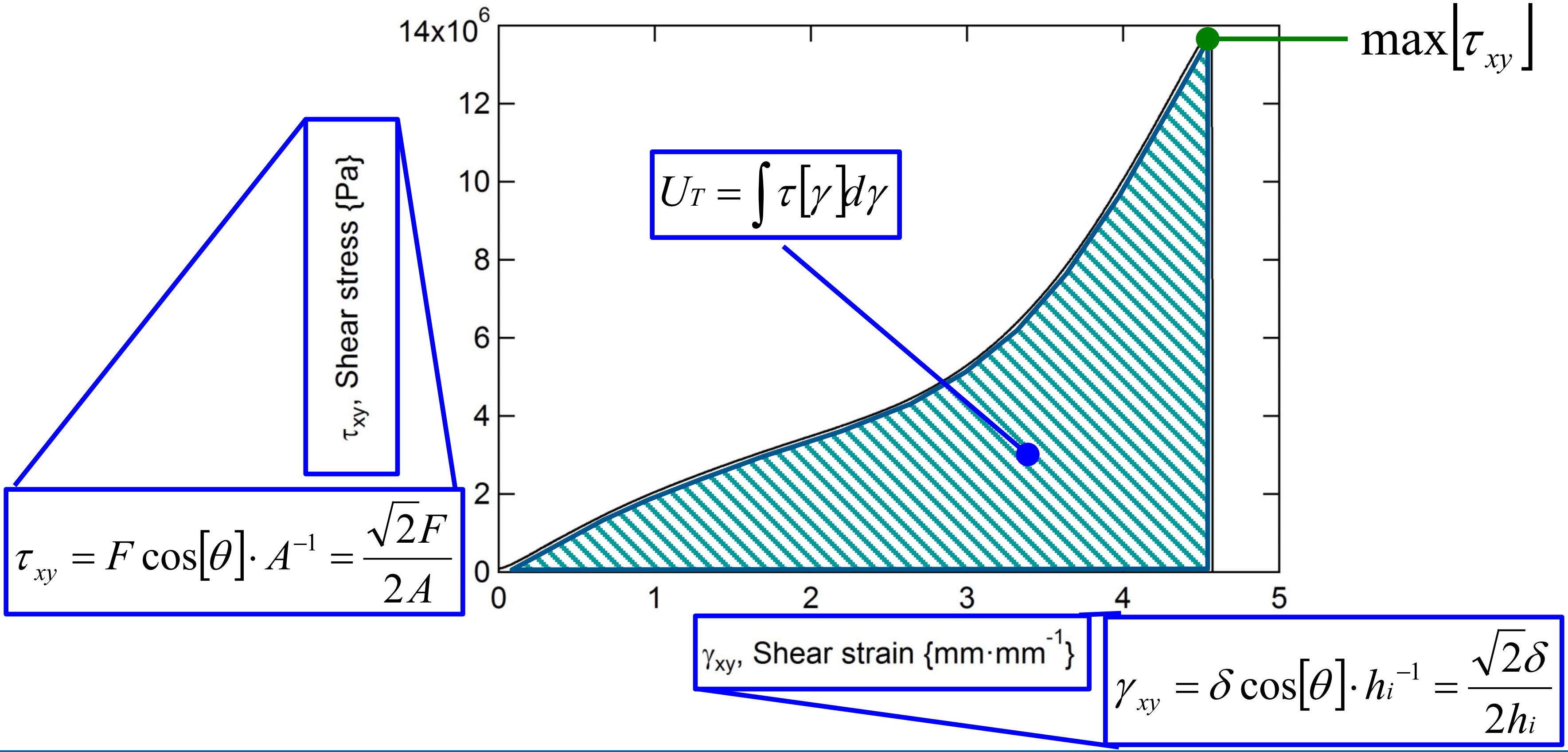
- Specimens were pre-conditioned for 1 month prior to distributing to participants.
- Coupons also conditioned after aging and after dicing specimens.
- Effect of moisture will be examined later.

Compressive Shear Test: Data and Analysis

- Strain is calculated from the crosshead displacement, based on the initial thickness, h_i=0.45 mm, and known test angle, θ=45°.
- The strain is not compensated for compliance of the instrument and grips.

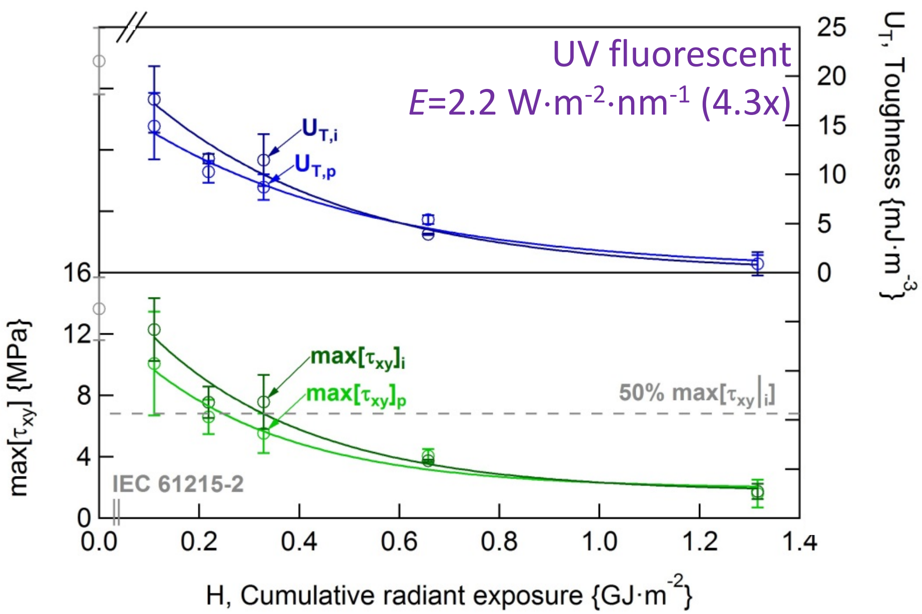
- Shear stress is calculated from the measured load,
`based on the initial specimen length, 25 mm, and width, 25 mm, and known test angle.

- Toughness is automatically calculated from the measured area of the stress/strain data profile.

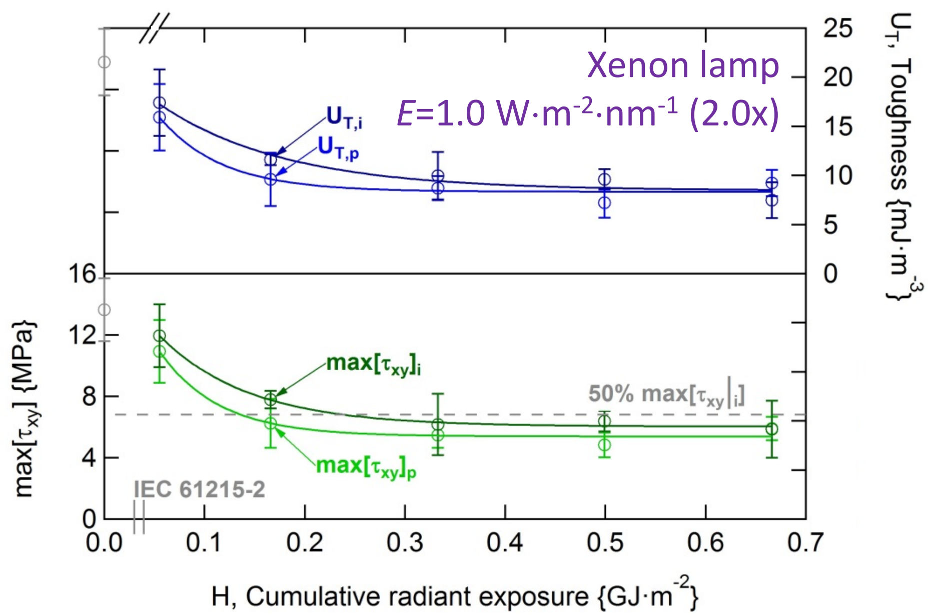


Profound Reduction in Strength Observed in First Experiments

- Strength and toughness were seen to decrease significantly (by 66%) through the first 90 days aging at Fraunhofer ISE.
- Similar magnitude of effect observed for specimens aged at NREL.
- Slow acting mechanism (UV)?



Weathered in custom chamber (60°C/50% RH) at Fraunhofer ISE. Trendline fits strictly to guide the eye. i=interior; p=periphery samples.

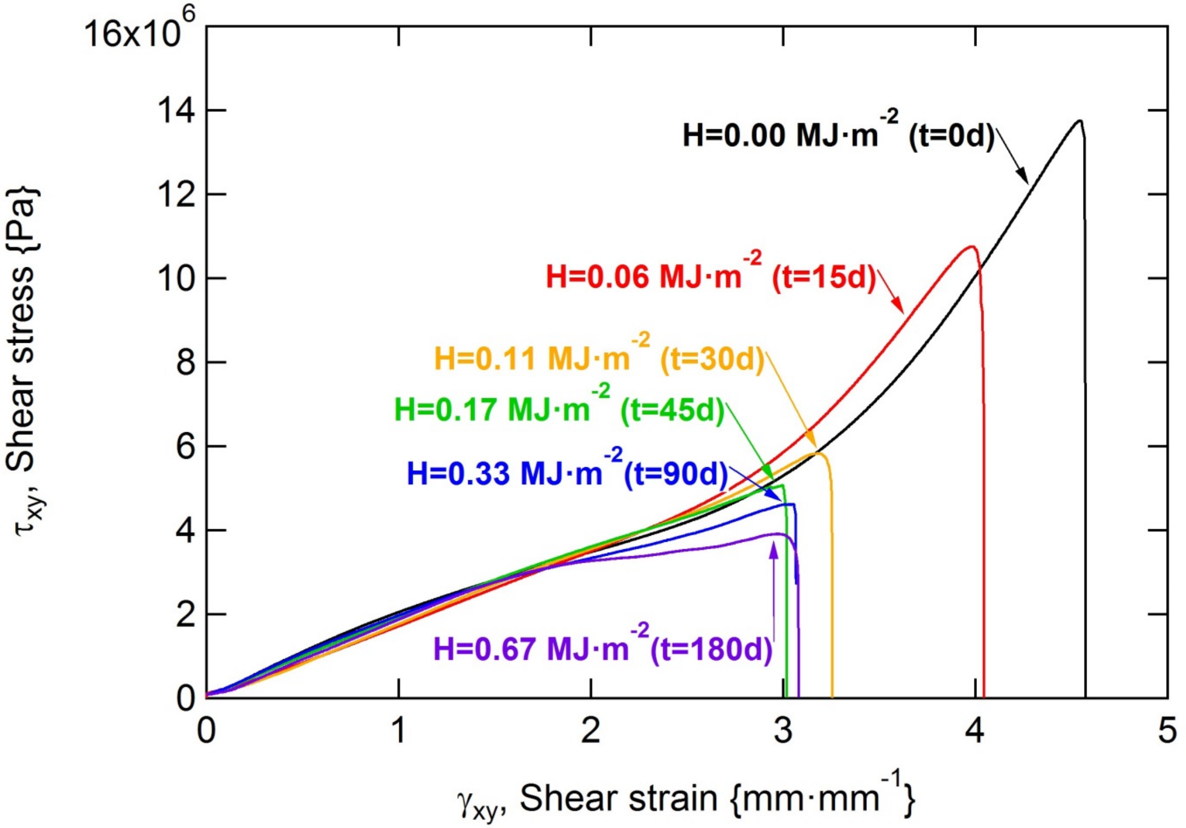


Weathered in Ci5000 chamber (60°C/50% RH) at NREL.

- $\Delta\tau_{xy}$ exceeds: 25% - the pass/fail in the RTI test, UL 746C; 50% - in the RTE test, IEC 60216; 50% - for cemented joints in IEC 61730-2.
- Some strength is maintained after prolonged aging, *e.g.*, as in an absolute minimum requirement.

Trends for EVA with Age

- Previous slide suggests strong correlation between τ_{xy} and *U*.
- Elastic region (initial τ and γ) not overtly affected with age.
- Loss of hyperelastic behavior following prolonged aging $\Rightarrow$embrittlement of EVA.



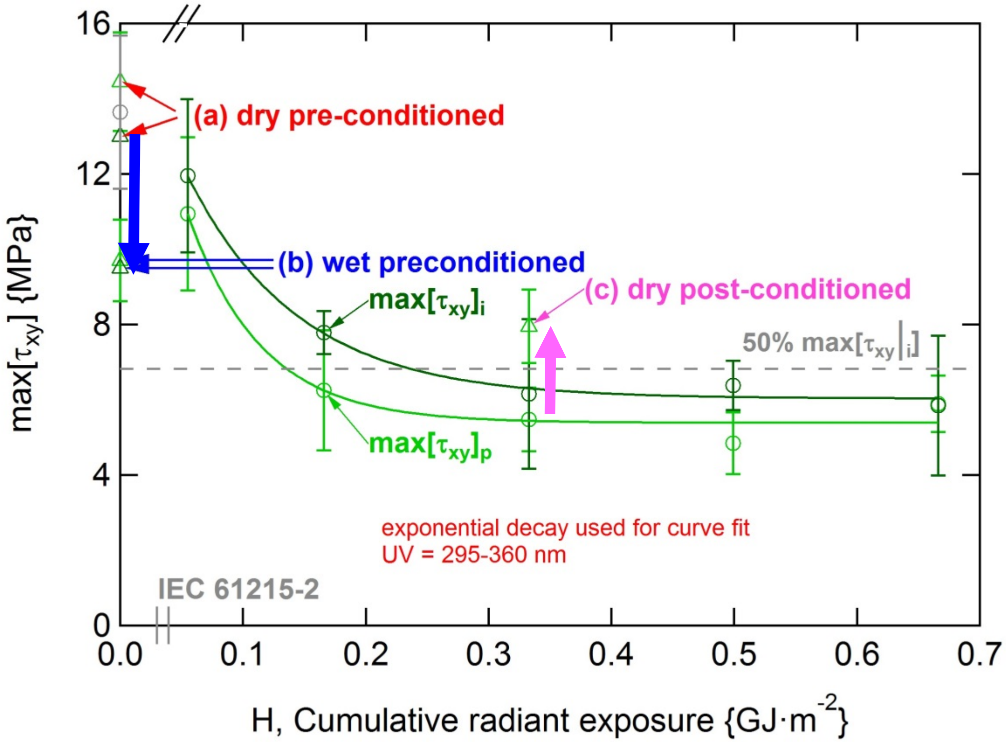
Weathering performed in Xe chamber (60°C/50% RH) at NREL.

- Results dominated by mechanical characteristics of EVA.
- Variability in τ_{xy} peak sharpness (sudden failure vs. yielding/slip) seen at all ages.

Specimen Preconditioning is Significant

Experiment: compare the effect of conditioning (1 month) including…

(a) pre-dessicate at 25°C (b) pre-chamber at 45°C/85% RH (c) post-dessicate at 25°C



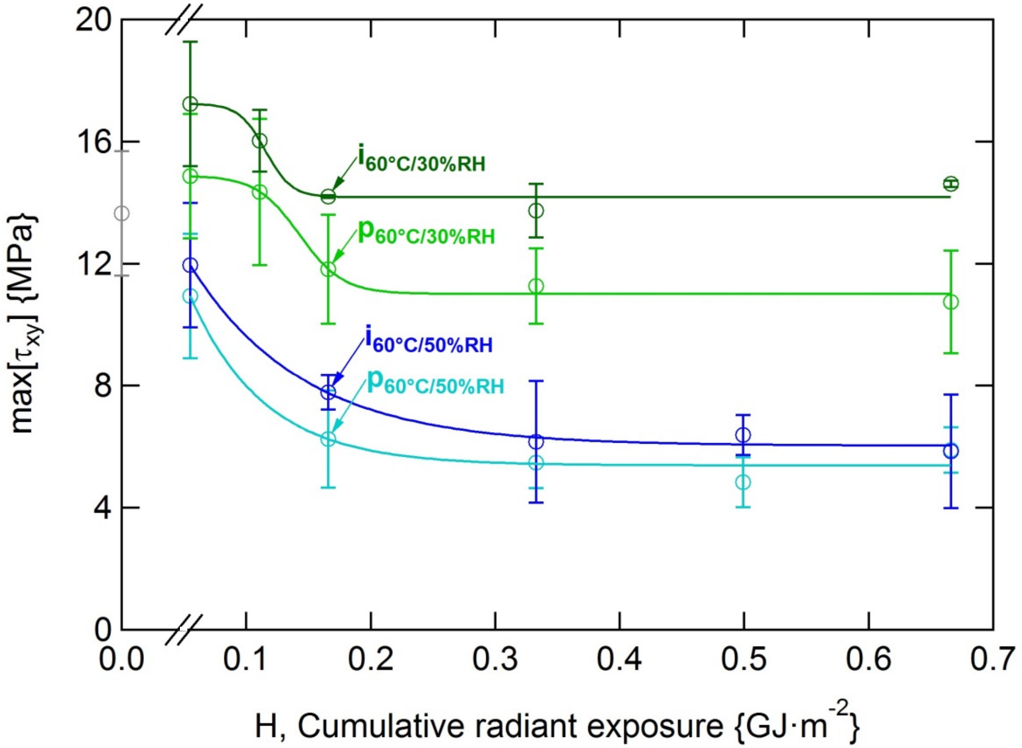
Weathering performed in Xe chamber (60°C/50% RH) at NREL. Trendline fits strictly to guide the eye.

- Dessicated samples overlap (bound) the previous data for *H*=0.
  Data is repeatable. Previous data shown for unaged –dessicated- specimens.
- 3.93 MPa reduction in max[τ_{xy}] for wet preconditioned specimens.
  Effect of preconditioning comparable to chamber weathering, Δmax[τ_{xy}] 4.87 MPa.
- 3.11 MPa increase in max[τ_{xy}] for aged, dessicated specimens.

Humidity During Aging is Significant

What is effect of chamber RH? ⇒ Overlay data at *60°C/30%* and *60°C/50%.*

- τ_{xy} not greatly affected at 30% RH. $\Delta\tau_{xy}$ >50% at 50% RH.
- RH (during weathering) is significant!!! ⇒Hydrothermal degradation?



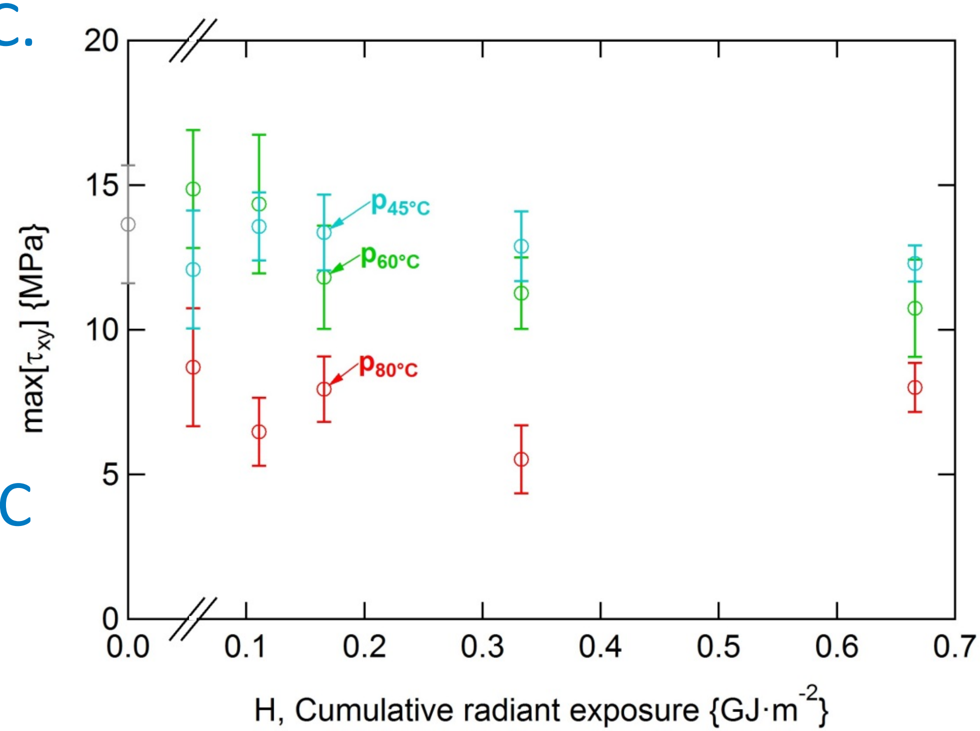
Weathering performed in Xe chamber (60°C/30% RH at 3M) or (60°C/50% RH at NREL). Trendline fits strictly to guide the eye.

- Periphery effected slightly faster than interior.
- Is strength initially –increased- at 60°C/30% RH?
  Improved adhesion seen in PREDICTS I study (PDMS/glass system).

Additional Weathering Data Suggest a Complicated Story

Separately Xe age at 2x AM1.5G and 30% RH, T = 45, 60, or 80 °C.

- 3M: little or no effect at 45 & 60°C.
- Fraunhofer ISE:
minimal $\Delta\tau_{xy}$ at 40°C (1x UV).
- Both labs: Measurable $\Delta\tau_{xy}$ at 80°C



Xe weathering performed at 3M (all chambers E_{340nm}=1.0 W·m^{-2}·nm^{-1}/30%RH).

Possible explanations:

- Threshold of UV, T, or RH required to invoke substantial damage?
- Effects from moisture (polymer plasticization, hydrothermal aging, not UV)?
- T_m~60°C for EVA. Phase change may affect rate of aging?

⇒Additional results (*e.g.*, dark chambers, > 60°C) should elucidate.

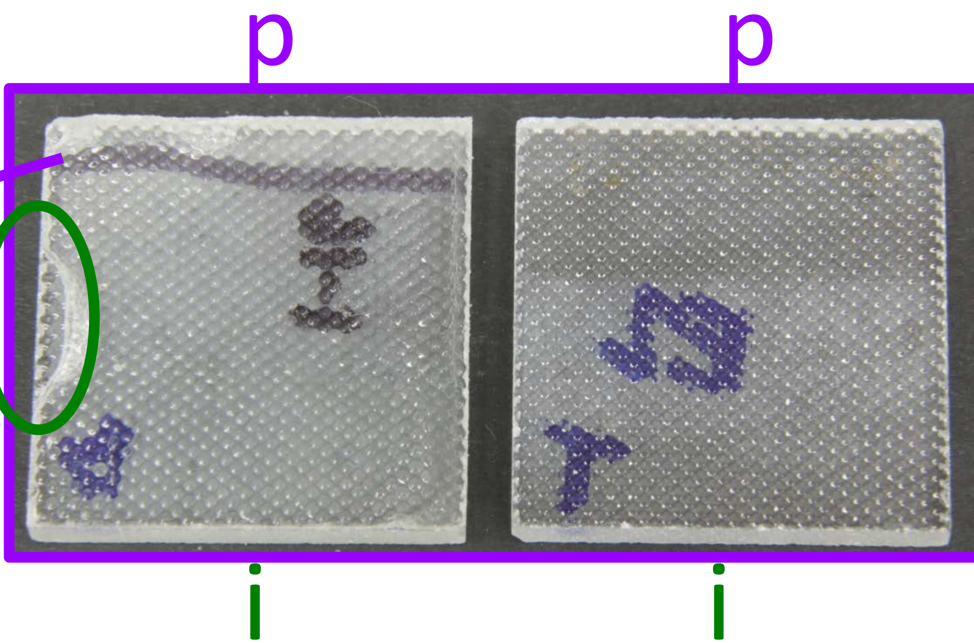
Typical Final Morphology of CST Specimens

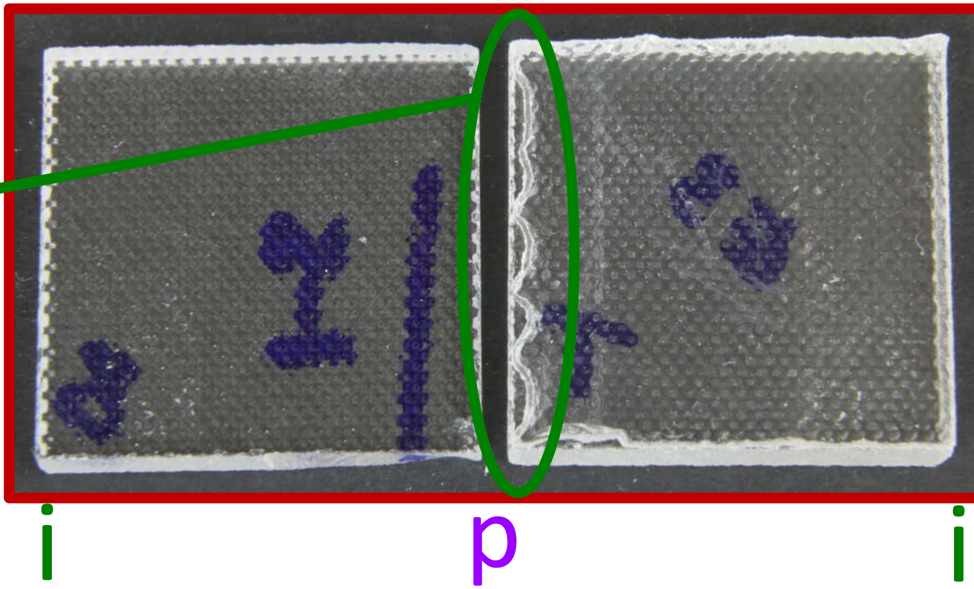
- Majority of encapsulant typically remains on one side of the detached specimens. (Cohesive failure near the interface, or delamination of thick networked layer).

- Encapsulant often remains on the substrate, suggesting UV damage near the incident polymer/glass interface. Corroborated between experiments and specimen F/A prep.

- Often the EVA was not overtly discolored. Solite glass has reduced UV transmittance.

- Localized delamination often observed at specimen edge. (Along loading direction, regardless of specimen orientation).





Summary & Conclusions

Some evidence of permanent damage from UV Weathering.

- Failure occurs proximate to irradiated surface.
- $\Delta\tau_T$ may be less from UV than from *RH.*
- 6 month test may prove appropriate, from asymptote at <700 $MJ \cdot m^{-2}$.

Very condition sensitive, *i.e.*, factor coupled:

- $\Delta\tau_T$: 80°C>>60°C. $\Delta\tau_{RH}$: 50% RH>>30% RH.
  Suggests little UV degradation occurs without extreme *T* and *RH*.

Cohesive failure –or- delamination of EVA/primer network observed.

Effects of moisture:

- Pre-conditioning is critical! Will extend overall test duration.

Caution:

- UV+mechanical stress not examined here. Permanent UV damage only.
- Glass/encapsulant may not be weakest interface.

Acknowledgements

Put a bird on it! From: https://s-media-cache-ak0.pinimg.com/736x/7c/ce/c9/7ccec951b8615ca99b247ba97a582ea4.jpg

☞There has been fantastic participation in TG5.

Thank you to the many participants for your ongoing support!!!

- If interested in TG5 activities or the experiments, please contact the corresponding regional leader. (See title slide)

This work was supported by the U.S. Department of Energy under Contract No. DE-AC36-08-GO28308 with the National Renewable Energy Laboratory.

NREL STM campus, Dennis Schroeder

🖐Your questions and feedback are much appreciated! Please help me to cover the important details & perspectives.

Extra Slides for Reference…

Questions for the Audience

- Describe your experiences with delamination in PV modules.
- Does manufacturing and process control contribute more significantly to loss of adhesion than weathering?
- What methods should be used to query delamination?

We have proposed a test sequence:

UV→CML (formerly DML)→HF10→DH500

- What is the likelihood of loss of adhesion following thorough (through-thickness) degradation of the encapsulation?

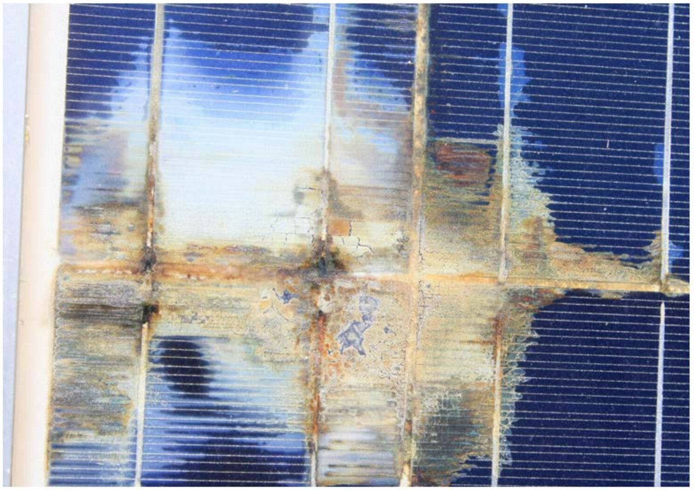
Could a similar concern apply to backsheet?

Adhesion: Comparison to Historic and Outdoor Data

- No history of systematic quantitative study of adhesion exists in the PV literature.

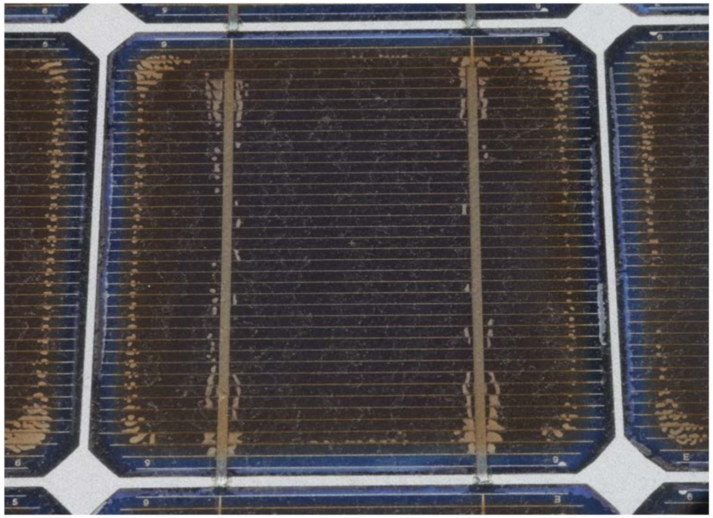
<u>Anecdotally…</u>

- Delamination observed in veteran installations in wide variety of locations.
Wohlgemuth *et. al.*, "Evaluation of PV Module Field Performance", Proc IEEE PVSC 2015.
- Encapsulant/cell interface often reported as weaker than encapsulant/glass. (Delamination observed at both interfaces in veteran installations).
- Importance of primer and degree of cure in establishing good adhesion.
Coulter et. al., DOE/JPL-1012-91, 1983, pp. 1-65.
- Delamination often precedes corrosion.
- Unknown if delamination correlates with encapsulation discoloration.



9.5 cm

Delmaination and subsequent corrosion in EFG-Si cell module at TEP Springerville facility.



5 cm

Delamination at cell-corners and -interconnect ribbons in mono-Si module at SMUD Hedges facility.

Test Matrix for the CST Study

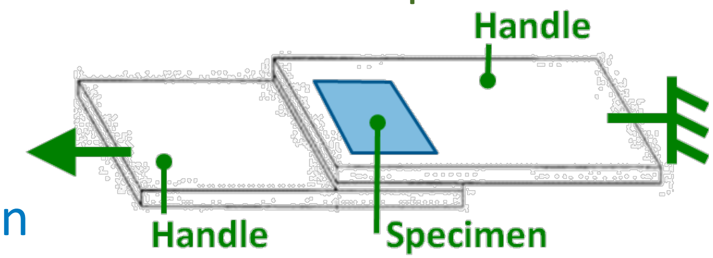
LIGHT SOURCE, FILTER	Xe Arc (right-light/cira filter)				Xe Arc (right-light/cira filter)	Xe Arc (right-light/cira filter)	Xe Arc (right-light/cira filter)	UVA-340 fluorescent (no filter)		UVA-340 fluorescent (no filter)	No light			field deployment (outdoors)
UV LIGHT INTENSITY	2x AM1.5G (102 W•m^{-2} for 300≤λ≤400)				IEC 62788-7-2 standard (0.8 W•m^{-2}@ 340 nm)	IEC 62788-7-2 standard (0.8 W•m^{-2}@ 340 nm)	IEC 62788-7-2 standard (0.8 W•m^{-2}@ 340 nm)	2x AM1.5G (1.0 W•m^{-2}@ 340 nm)		accelerated (~150 W•m^{-2} for 300≤λ≤400)	0 W•m^{-2}			
CHAMBER RELATIVE HUMIDITY {%}	30 ("nominal")			50 ("high")	20 ("low")	20 ("low")	20 ("low")	~7% ("very low")		50 ("high")	30	30	30	ambient
CHAMBER TEMEPRATURE {°C}	40	60	80	60	70	80	90	40	60	60→ 80 → 40	40	60	80	ambient
PARTICIPANT (INSTRUMENT MODEL)	3M (Ci5000)	3M (Ci5000)	3M (Ci5000)	NREL (Ci5000)	CEI (Ci4000)	Fraunhofer CSE (Ci4000)	NREL (Ci5000)	QLAB (QUV)	CWRU (QUV)	Fraunhofer ISE (custom)	NREL	NREL	NREL	ATLAS (EMMA in Phoenix)
		QLAB (QSUN XE3)		QLAB (QSUN XE3)										CWRU (5x in Cleveland)
														ATLAS (rack in Phoenix)
														ATLAS (rack in Miami
														NREL (rack in Golden)
														KACST (rack in Riyadh)
														CEI (Turpan, China)
														CEI (QiongHai, China)

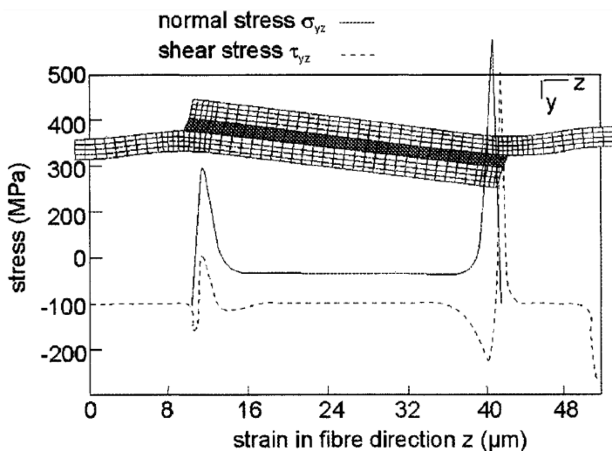
Summary of test conditions applied in this study.

Compressive Shear Test: Limitations & Implementation

- CST removes some limitations of overlap shear test:
  - -Premature fracture of the substrate handles.
  - -Stress concentration at specimen ends.
  - Cantilever effect (compliance) on the handles.
  - Reduced misalignment from sample fabrication (batch machine diced on computer controlled X- Y- stage).

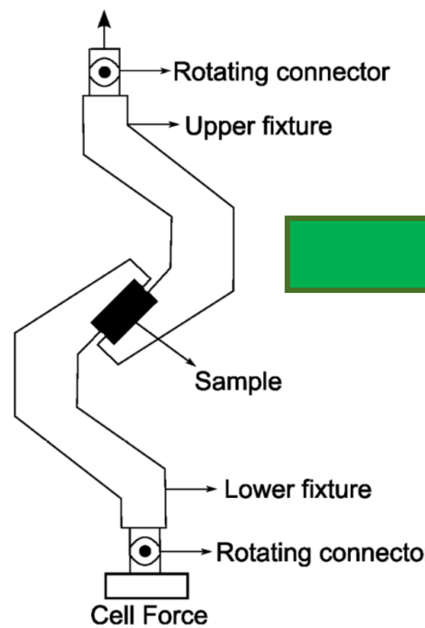
Schematic showing the implementation of the overlap shear test.





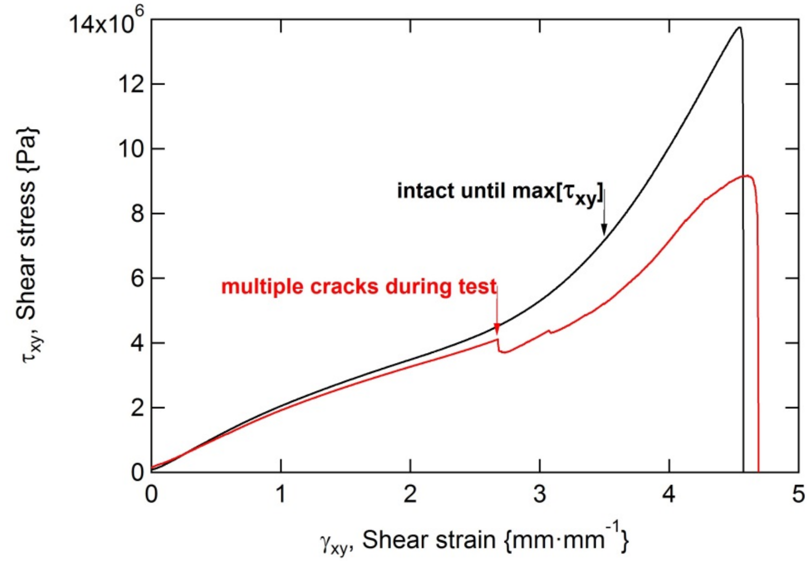
FEA identifies the pitfalls of the overlap shear test. Schneider et. al., Appl. Comp. Mats., 8, 2001, 43-62.

- TG5: two universal joints aid mechanical alignment.



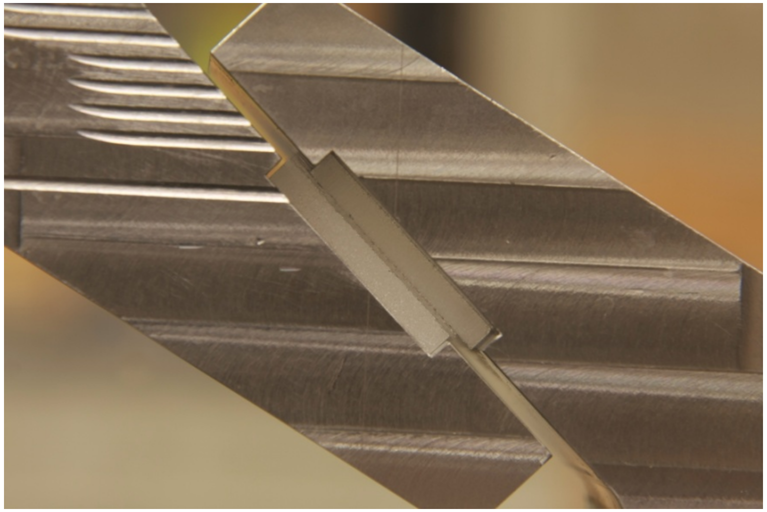
Compressive Shear Test: Apparatus and Implementation

- Data excluded for samples with substrate cracking prior to failure. Visual or audio cracking during test typically corresponded to excursions in data profiles.
- Dicing and specimen handling are critical.
(Maintain good surface quality.
No chips or cracks at edges.)



Comparison of data profiles for intact and cracked specimens.

- Substantial deformation observed for unaged PV encapsulation materials (typically elastomers).



In-situ photo of EVA sample, prior to detachment.

Compressive Shear Test: Method and Apparatus

- CST previously applied in the literature:

A. Jagota et. al., Int. J. Fract., 104, 2000, 105-130.

K. Schneider et. al., Appl. Comp. Mats., 8, 2001, 43-62.

- Elegant implementation (tensile grips) was demonstrated at EPFL/CSEM:

Chapuis et. al., PIP, 22 (4), 2014, pp. 405–414.

- Much less likely to damage test equipment!!!

- Related standards:

(for compressive/in-plane shear, not grip CST implementation)

ASTM D905 (Strength Properties of Adhesive Bonds in Shear by Compression Loading),

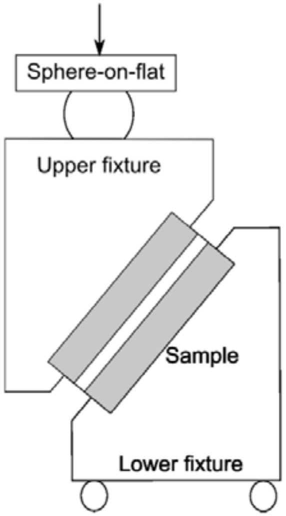
ASTM D3410

(Compressive Properties of Polymer Matrix Composite Materials with Unsupported Gage Section by Shear Loading),

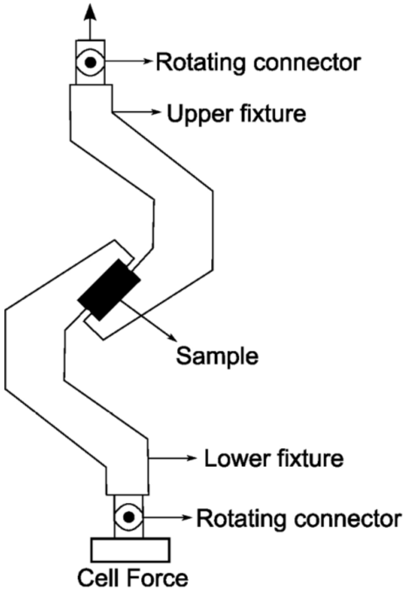
ASTM D3846 (Standard Test Method for In-Plane Shear Strength of Reinforced Plastics),

ASTM D4255 (In-Plane Shear Properties of Polymer Matrix Composite Materials by the Rail Shear Method),

BS 5350 (Determination of bond strength in compressive shear).



Kinematic mount CST implementation.
from Chapuis *et. al.*, DOI: 10.1002/pip.2270



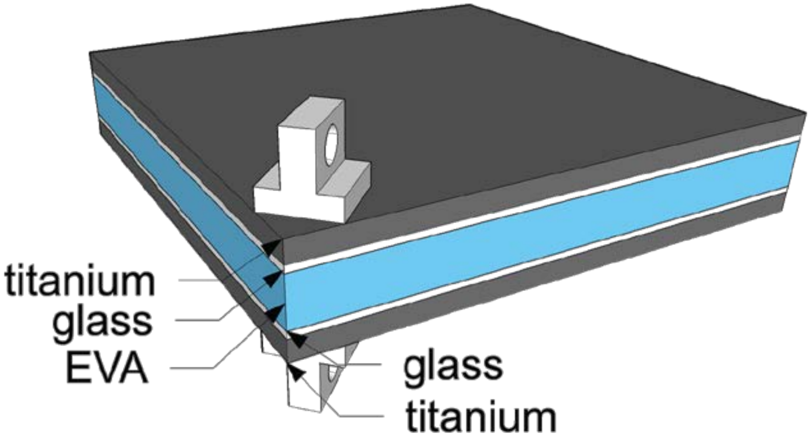
Tensile grip CST implementation.
from Chapuis *et. al.*, DOI: 10.1002/pip.2270

CST is Not a Fracture Mechanics Test!

- Compressive shear test is subject to same limitations as other strength of attachment methods, *e.g.*, defects from sample fabrication.
- CST unit of measure: max[τ_{xy}] {MPa} or max[U_T]{J·m^{-3}}.

Not G_{IC} {J·m^{-2}}! Does not directly relate to the fundamental physics of adhesion.

- CST used here to generally explore UV weathering.
- Fracture mechanics enabled methods presently under development, *e.g.*, IEC 62788-6 standards.



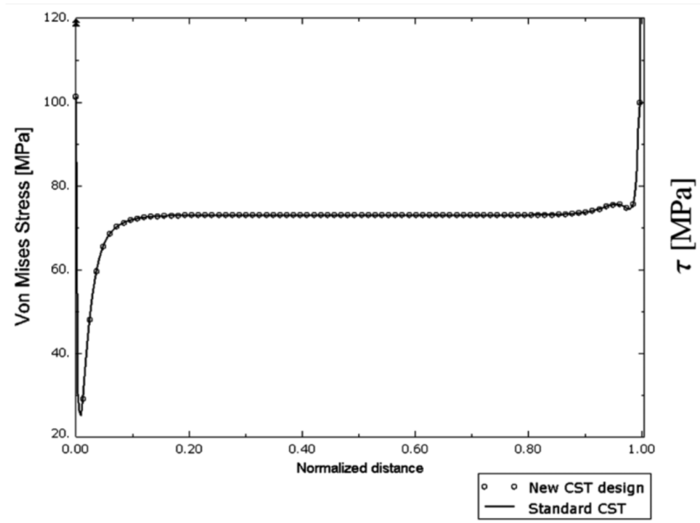
Schematic of "included angle" specimen implementation, shown for encapsulation coupon specimens.
From Bosco et. al., Proc NREL PVMRW 2015.

Novoa *et. al*., "Environmental Mechanisms of Debonding in Photovoltaic Backsheets", SOLMAT, 120, 2014, 87-93.
Novoa *et. al*., "Debonding Kinetics of Photovoltaic Encapsulation in Moist Environments", PIP, 2015. DOI 10.1002/pip.2657.
Bosco *et. al*., "Moving the PV Industry to a Quantitative Adhesion Test Method", Proc. Atlas/NIST Work. PV Mats. Durability, 2015.
Dauskardt *et. al*., "Thermo-Mechanical Degradation Mechanisms Relevant for Field Failures and Solar Lifetimes", Proc. Atlas/NIST Work. PV. Mats. Durability, 2015.
Bosco et. al., "Encapsulation and Backsheet Adhesion Metrology for Photovoltaic Modules", *submitted to PIP*.

Comparing the Mechanical Condition in CST to Fielded PV

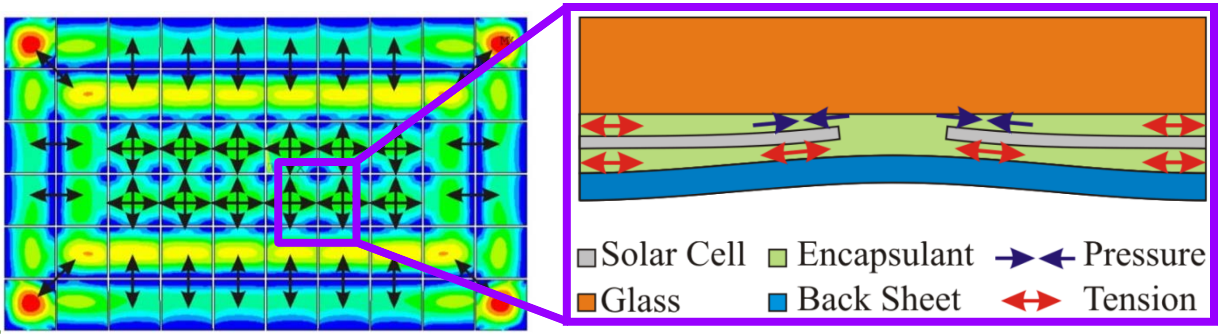
- Elastomers: $\nu \rightarrow 0.5$
  - $\Rightarrow$ approximately incompressible.
- CST is dominated by applied shear.

CHARACTERISTIC	CST	PV
max[strain] {%}	~500	~20
rate {Hz}	$5 \cdot 10^{-2}$	$1 \cdot 10^{1}$ - $1 \cdot 10^{-3}$ - 1×10^{-5}
mode mixity	limited to edges	ubiquitous, *e.g.*, at components, voids
dominant stress	τ_{xy}	more τ_{xy} ($T > T_{m,EVA}$) more σ_{xx} ($T < T_{m,EVA}$)
stress localization	at edges	at components, voids
κ, curvature	minimal, from glass	induced with ΔT, $\sigma_{residual}$
interior glass surface	textured	textured



Analysis of stress distribution in CST specimen.
Chapuis et. al., PIP, 22 (4), 2014, pp. 405–414.

Comparison of mechanical condition invoked by CST and PV modules. Based on:
Dietrich et. al., Proc. SPIE 2013, 8825-4.
xxx



Bending stress in module, with inset of local curvature, deformation, and stress local to cells.
Dietrich *et. al.*, Proc. SPIE 2013, 8825-4.