

Degradation in PV Encapsulation Adhesion: An Interlaboratory Study Towards a Climate-Specific Test

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2015 Atlas/NIST Workshop on Photovoltaic Materials Durability

Session 3: Adhesion of PV Components

09:15-09:45 Weds, 2015/12/09, Green room (Administrative Bldg.)

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

- PV safety, qualification tests (61215, 61646, 61730-2) presently prescribe up to 137 days equivalent IEC 60904-3 AM 1.5G UV-B radiation dose.
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).
 - 1: Start of “Leg 2” test series to query delamination.
(UV→CML (formerly DML)→HF10→DH500)
 - 3: Encapsulation UV weathering test
- IEC 62788-7-2 (UV weathering of PV materials and components).
Accelerated aging test(s) for backsheet, encapsulation, adhesives...

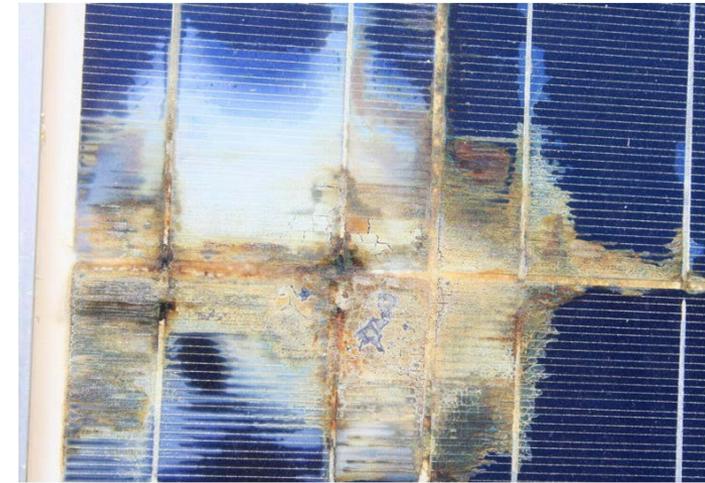
Adhesion: Comparison to Historic and Outdoor Data

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

Anecdotally...

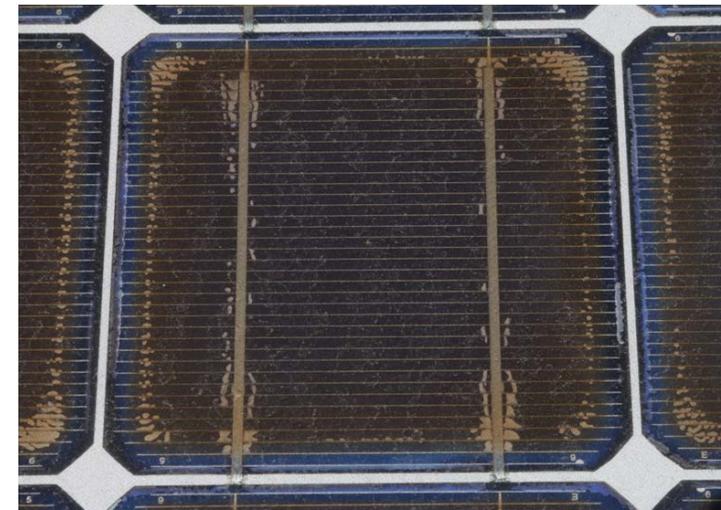
- Encapsulant/cell interface often reported as weaker than encapsulant/glass.
(Delamination observed at both interfaces in veteran installations).
- Importance of primer and degree of cure in establishing good adhesion.
- Delamination often precedes corrosion.

- Unknown if delamination correlates with encapsulation discoloration.



_____ 9.5 cm

Delamination 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.

Wohlgemuth et. al., "Evaluation of PV Module Field Performance",
Proc IEEE PVSC 2015.

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 importance of factors including UV, T, %RH, and time.
What parameter “space” should be applied for aging?
2. Determine if there is significant coupling between relevant aging parameters.
What factors should be applied in a weathering test for adhesion?
3. Investigate the spectral requirements for UV light sources,
i.e., by comparing specimens aged by Xe-arc, UVA-340, metal-halide.
Does visible light affect aging?

Encapsulant Used in the TG5 Experiment

- 1 material examined in interlaboratory study.
- "EVA-B" formulation similar 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")

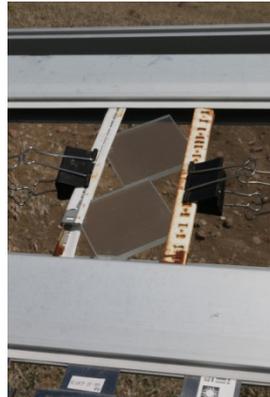
Encapsulant material being examined in the interlaboratory attachment strength experiment.

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

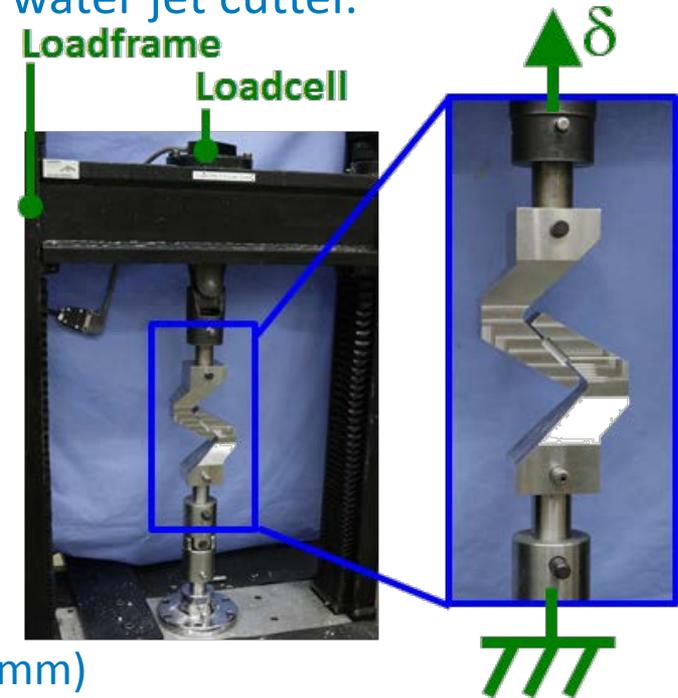
- Share TG5 weathering between 12 participants. CST performed by NREL.
- 25 mm square specimens (diced, after aging) examined using loadframe.
- Pristine edge quality is critical. Dice using abrasive water jet cutter.



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:

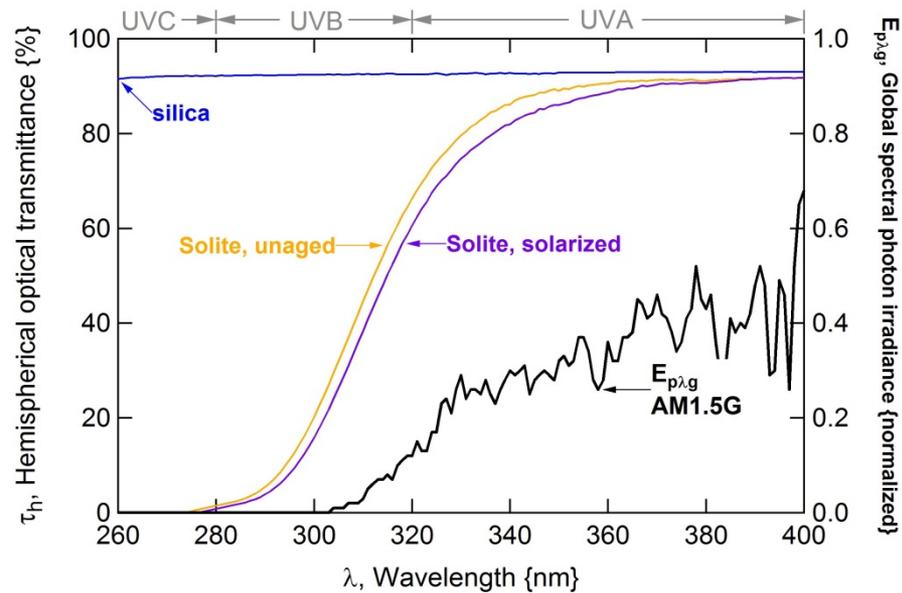
https://pfs.nrel.gov/main.html?download&weblink=ff9d3efc4a89183e8283e5ca01e dbf8a&realfilename=IMG_0529.MOV

User summary:

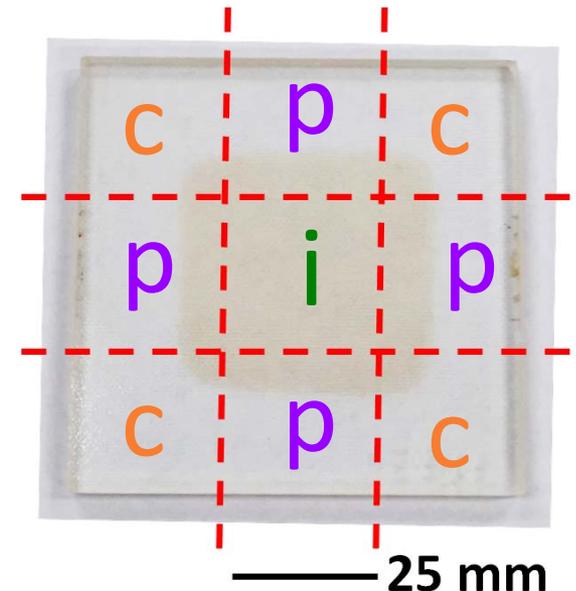
- Geometry: glass/polymer/glass (3.2 mm/0.5 mm/3.2 mm)
- Size: 3" x 3"
- Quantity: 10 replicates of 1 material (pre-conditioned), plus 5 extras (not pre-conditioned)
- Aging: 15, 30, 45, 90, 135, and 180 cumulative days (indoors), or 1, 2, 3, 4, 5 years (outdoors)
- Remove 2 coupons at each increment
- 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 ($\Delta\lambda_{cUV} \sim 5$ nm) through the experiment.



Comparison of silica (for transmittance specimens) and Solite (for CST) glass, shown 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. $\geq(5)$ replicates are used for the periphery measurements.
- Orientation of periphery specimens is alternated through experiment.

Policy for Moisture-Conditioning of Specimens

- Water content may be a critical factor in the experiment.
⇒ maintain stable water content.
- A set of 4 different conditions were applied to render an internal water content (ppm) similar to that in the aging chamber.

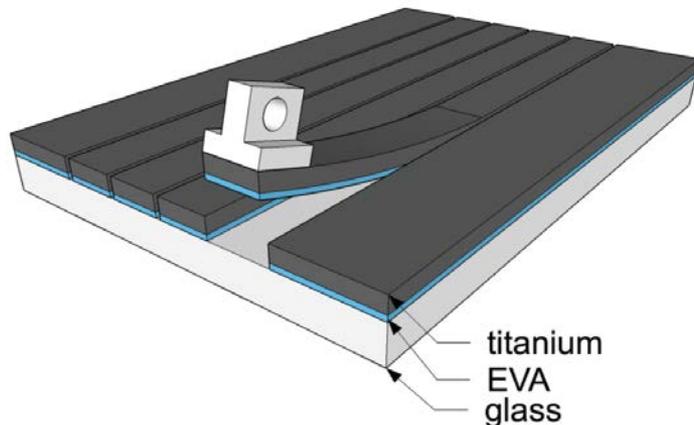
Temperature (°C)	RH (%)	Saturation at RH (g/cm ³)	Concentration Ratio (vs. 40/30%)	Dew Point (°C)	Temperature (°C)	Humidity (%)	Recommendation(s)
40	30	0.000882	1.0	-9.4	30	45.8	Put in Refrigerator
60	30	0.001297	1.5	4.6	30	72.7	Put in Refrigerator
60	50	0.002162	2.5	25.7	30	90.9	
					23	106.3	Put in 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.

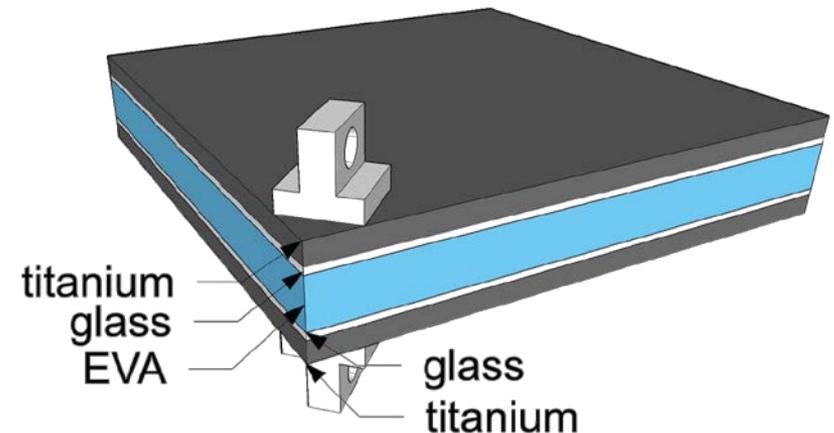
- Specimens were pre-conditioned for 1 month prior to distributing to participants.
- Specimens were conditioned before aging and after dicing.
- Effect of preconditioning will be examined later.

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: $\max[\tau_{xy}]$ {MPa} or U_{\max} {J}.
- **Not G_{IC} {J·m⁻²}! Can't be related 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 traditional SCB implementation for encapsulation coupon specimens. *From Bosco et. al., Proc PVMRW 2015.*



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

Novoa et. al., "Environmental Mechanisms of Debonding in Photovoltaic Backsheets", SOLMAT, 120, 2014, 87-93.

Bosco et. al., "A Fracture Mechanics Based Approach to Adhesion Testing in the PV Module", Proc. PVMRW 2015.

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.

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),

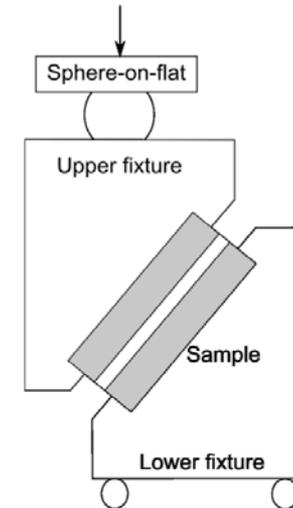
ASTM D3410

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

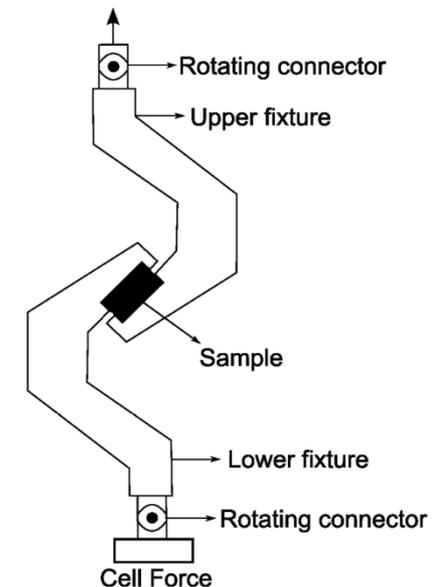
ASTM D3846 (Standard Test Method for In-Plane Shear Strength of Reinforced Plastics),

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

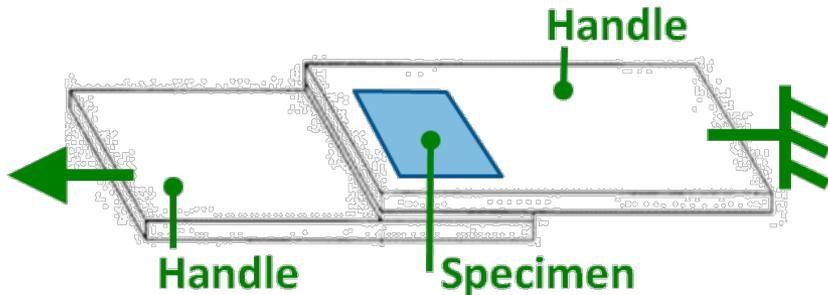


Tensile grip CST implementation.
from Chapuis et. al., DOI: 10.1002/pip.2270

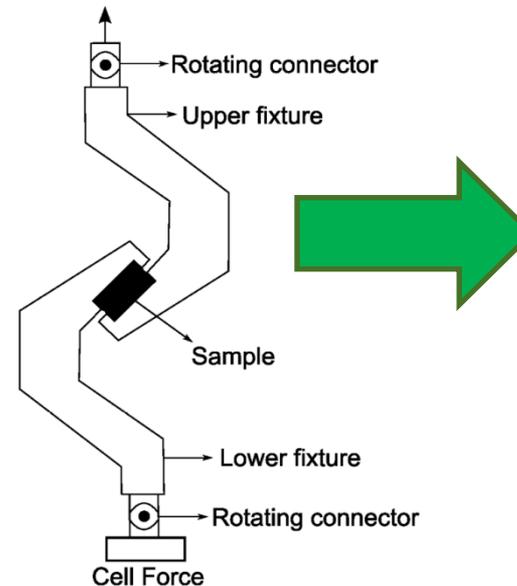
Compressive Shear Test: Limitations & Implementation

- CST removes some limitations of overlap shear test:

- Premature fracture of the substrate handles.
- Cantilever effect (compliance) on the handles.
- Reduced misalignment from sample fabrication
(batch machine diced on computer controlled X- Y- stage).



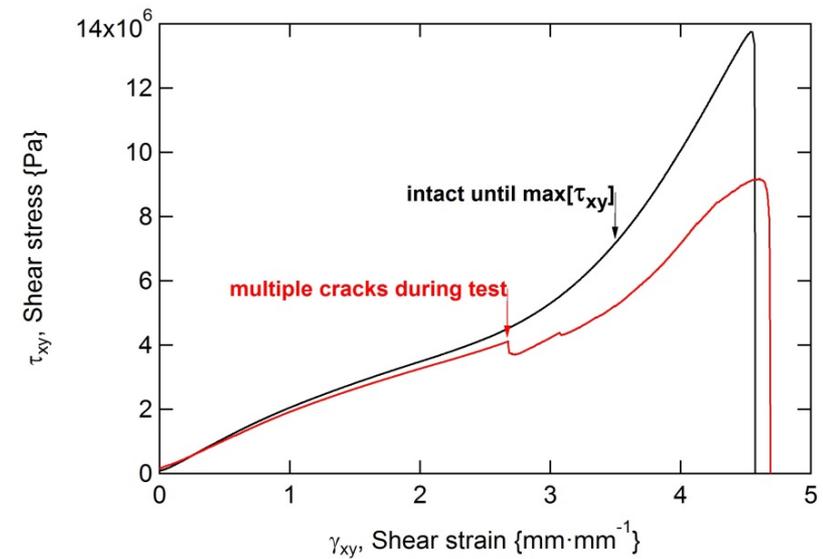
Schematic showing the implementation of the overlap shear test.



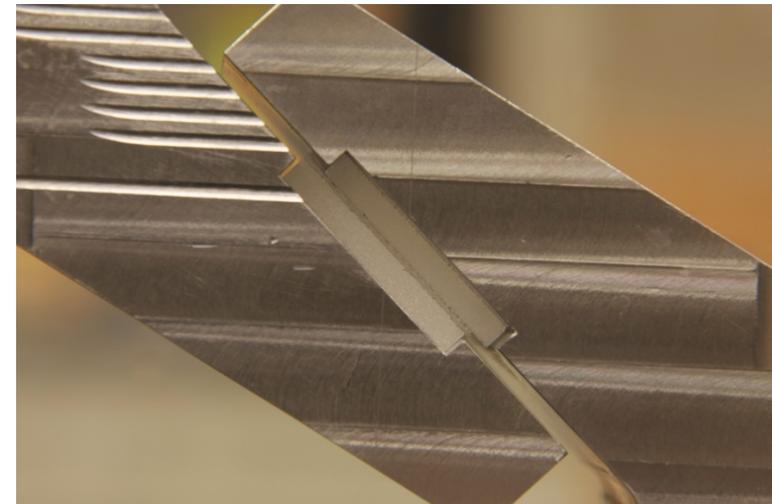
- TG5: two universal joints aid mechanical alignment.

Compressive Shear Test: Apparatus and Implementation

- Data excluded for samples with substrate cracking during test. 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.)
- Substantial deformation observed for unaged PV encapsulation materials (typically elastomers).



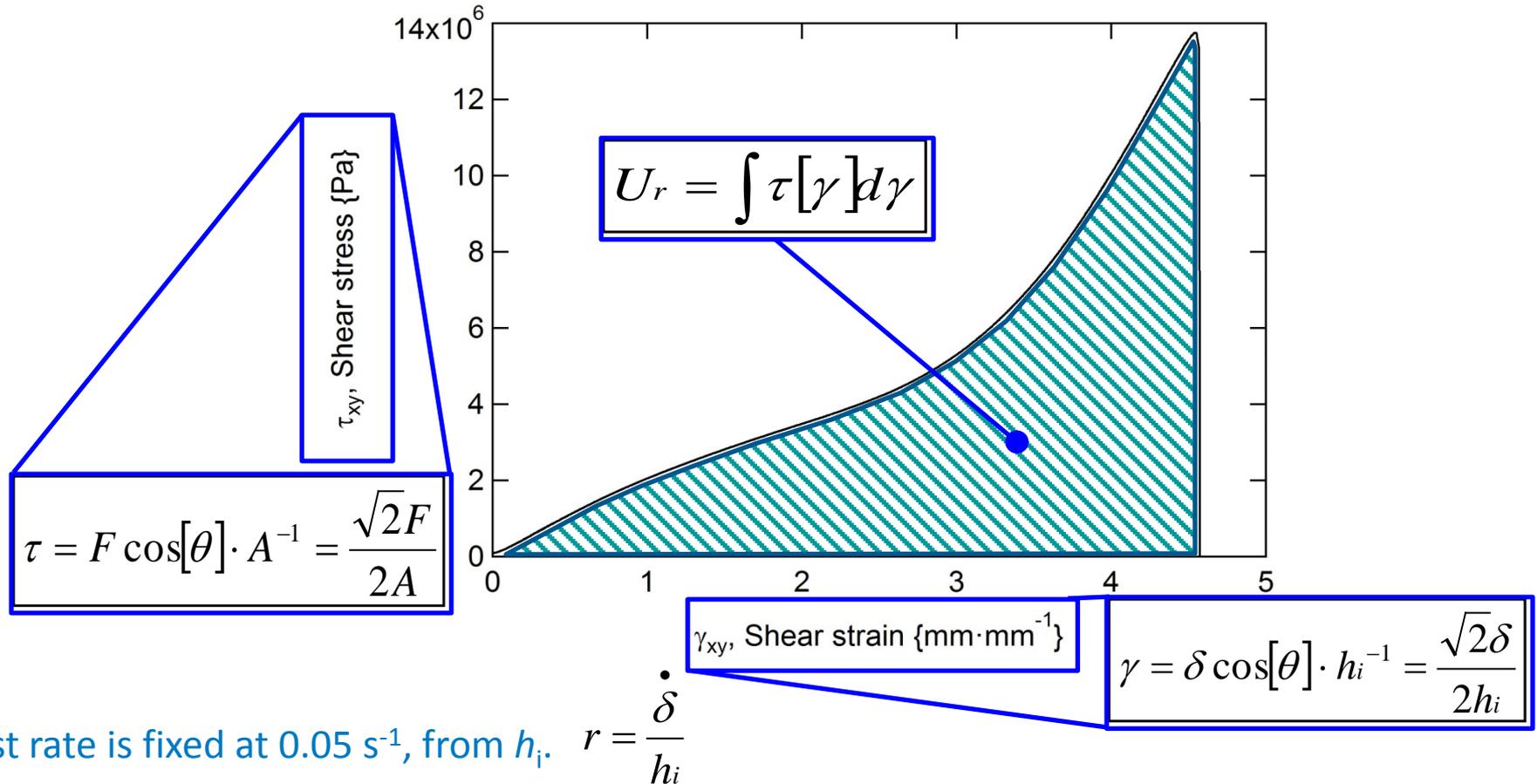
Comparison of data profiles for intact and cracked specimens.



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

Compressive Shear Test: Data and Analysis

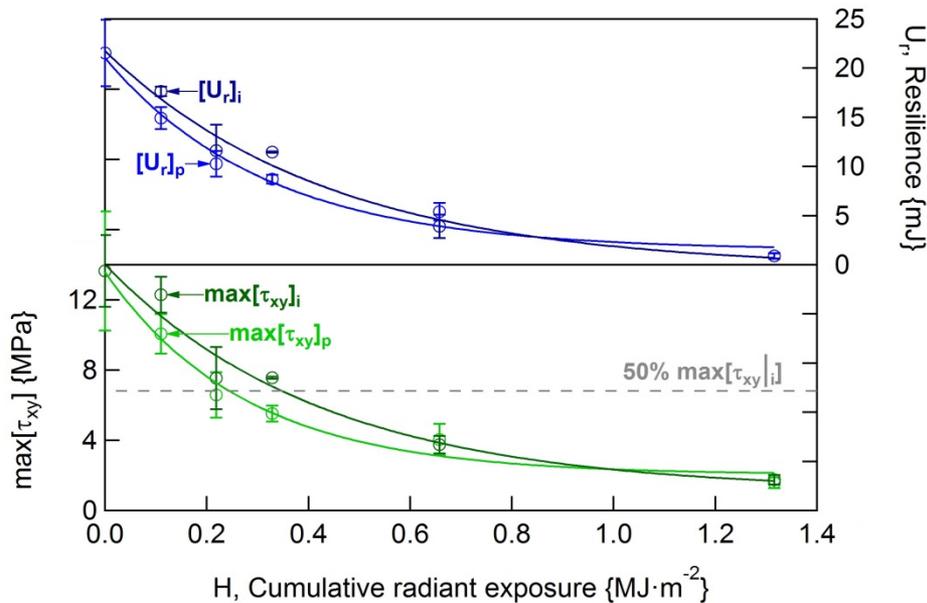
- Strain is calculated from the crosshead displacement, based on the initial specimen geometry (encapsulation thickness, $h_i=0.45$ mm) and known test angle ($\theta=45^\circ$).
- The strain is not compensated for compliance of the instrument and grips.
- Shear stress is calculated from the measured load, based on the initial specimen geometry (length [25 mm] and width [25 mm] of the cut specimens) and known test angle.
- The resilience is automatically calculated from the measured area of stress/strain data profile.



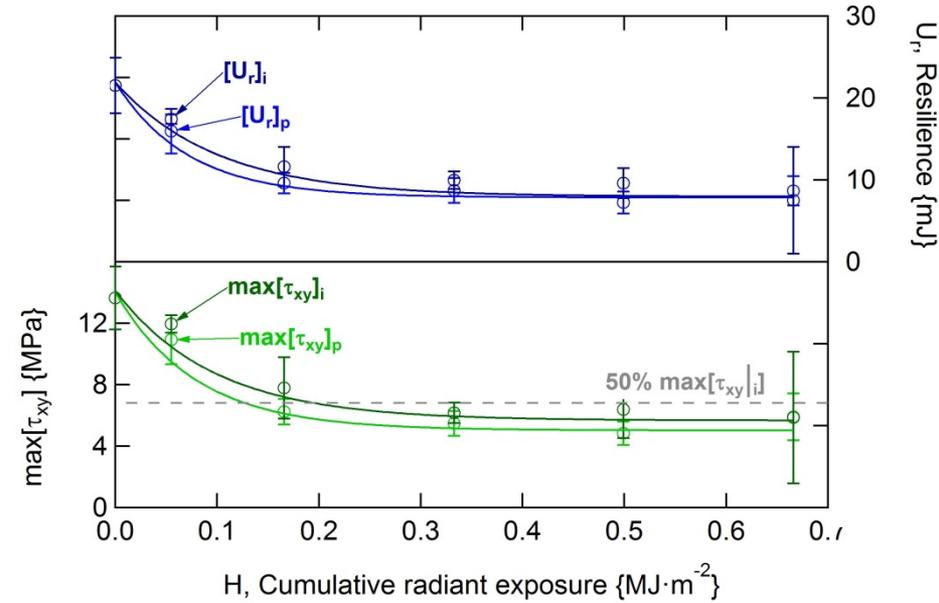
- Test rate is fixed at 0.05 s^{-1} , from h_i .

Profound Reduction in Strength Observed in First Experiments

- Strength and resilience 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)?



Weathering performed in custom UVA-340 fluorescent UV chamber (60°C/50%RH) at Fraunhofer ISE.

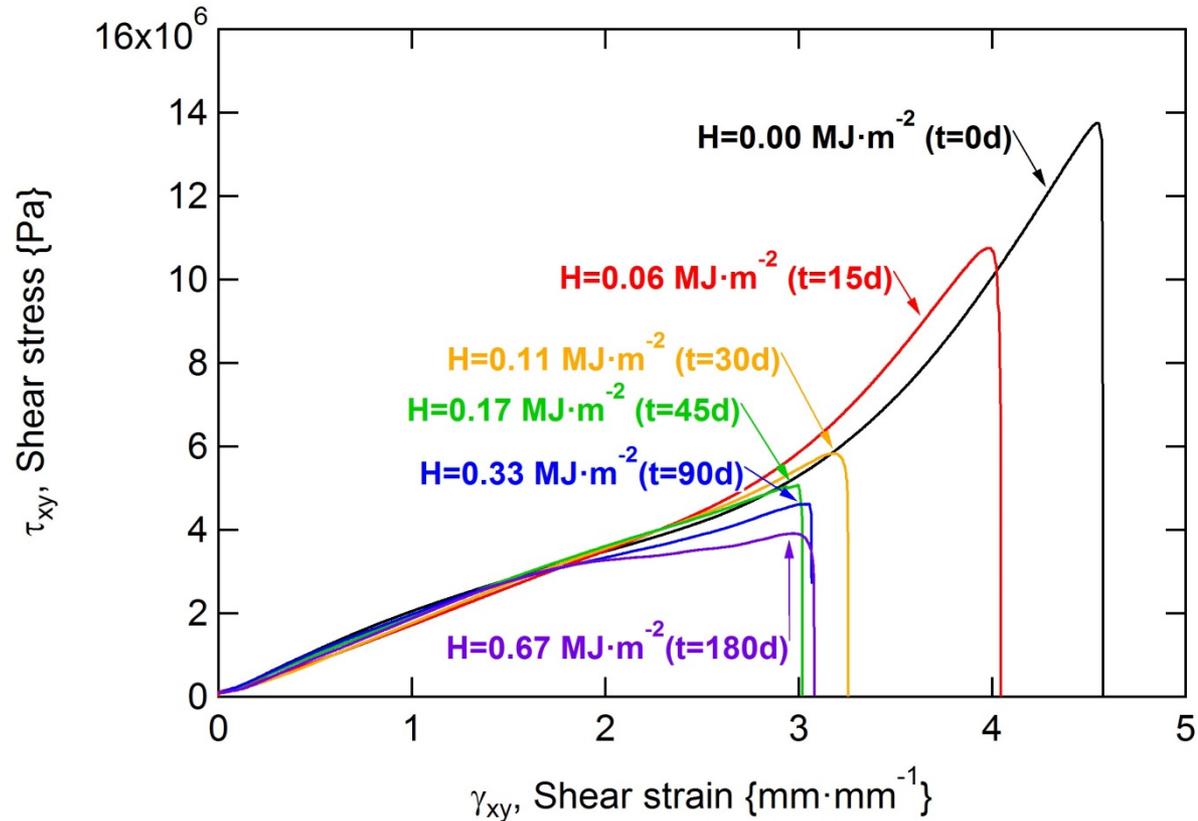


Weathering performed in Xe chamber (60°C/50%RH) at NREL. Trendline fits to guide the eye. i=interior; p=periphery samples.

- $\Delta\tau_{xy}$ exceeds the rule of 50% for pass/fail, typically applied in relative thermal index (RTI) tests.
- 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 (greatest τ and γ) following prolonged aging.



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

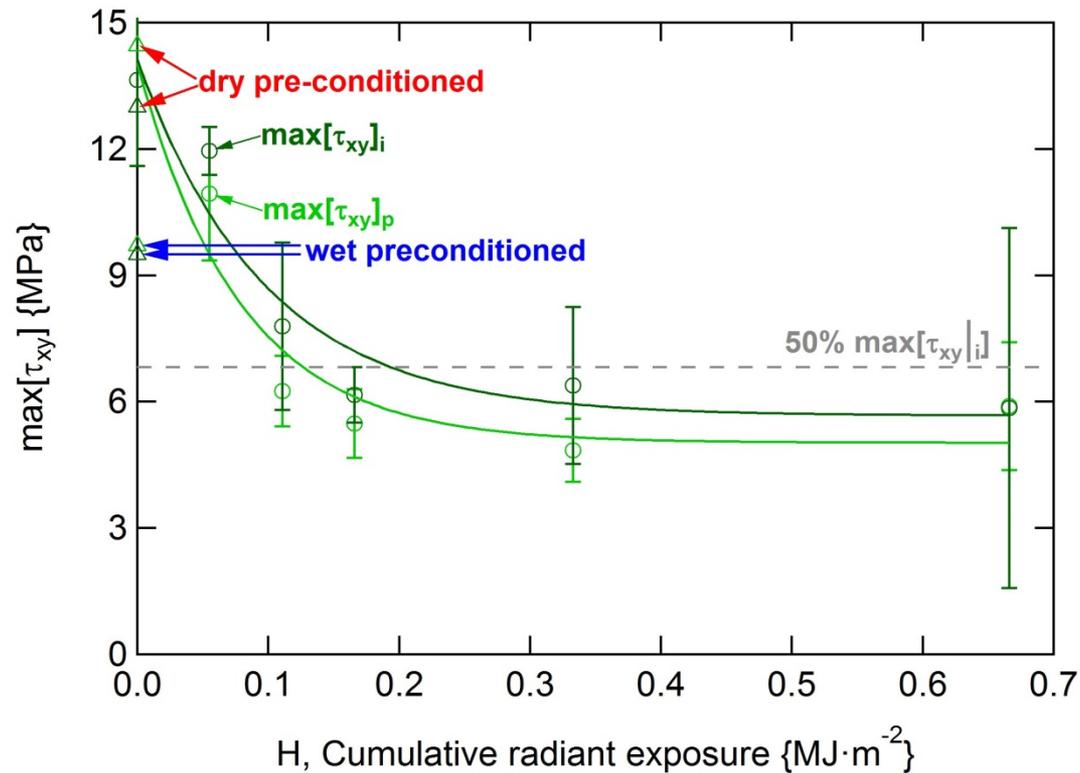
- Slight variability in sharpness of τ_{xy} peak (sudden failure vs. yielding/slip) seen at all ages.

Specimen Preconditioning is Significant

Experiment: compare the effect of preconditioning (1 month) including...

(a) dessicate at 25°C

(b) chamber at 45°C/85%RH



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

- Dessicated samples overlapped the previous data for $H=0$.

Data is repeatable. Previous data shown for unaged –dessicated- specimens.

- 25% reduction in $\max[\tau_{xy}]$ for wet preconditioned specimens.

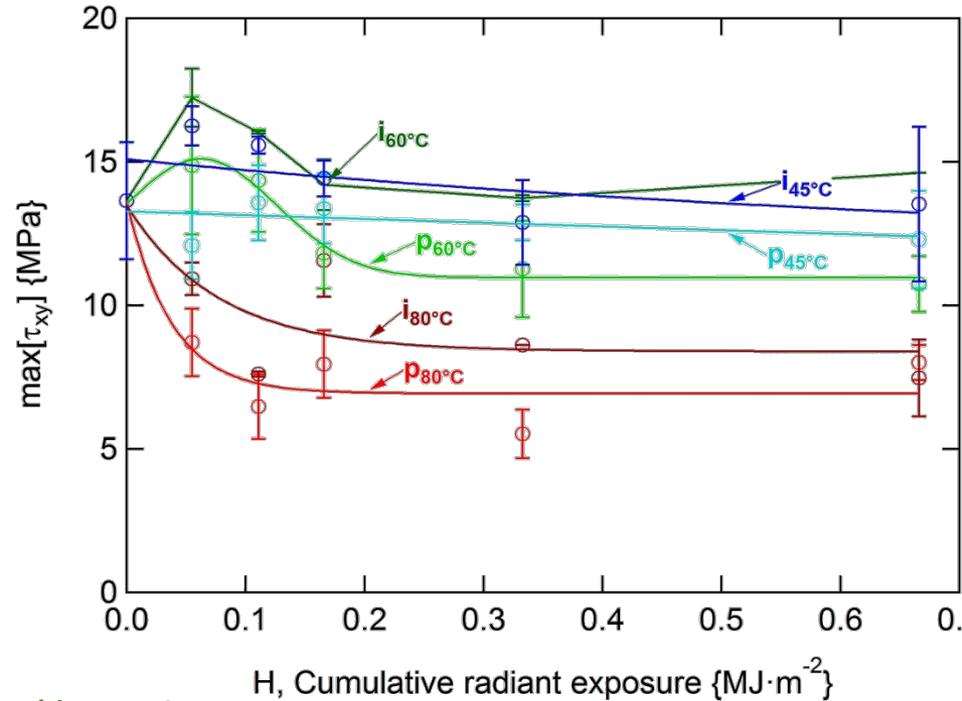
Effect of preconditioning is comparable to the effect of chamber weathering.

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 seen at 45 & 60°C.

•Fraunhofer ISE:
minimal $\Delta\tau_{xy}$ seen at 40°C (1x UV).



Xe weathering performed at 3M (all chambers $E_{340\text{nm}}=1.0 \text{ W}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}/30\%RH$). Data connected or trendline fitted to guide the eye.

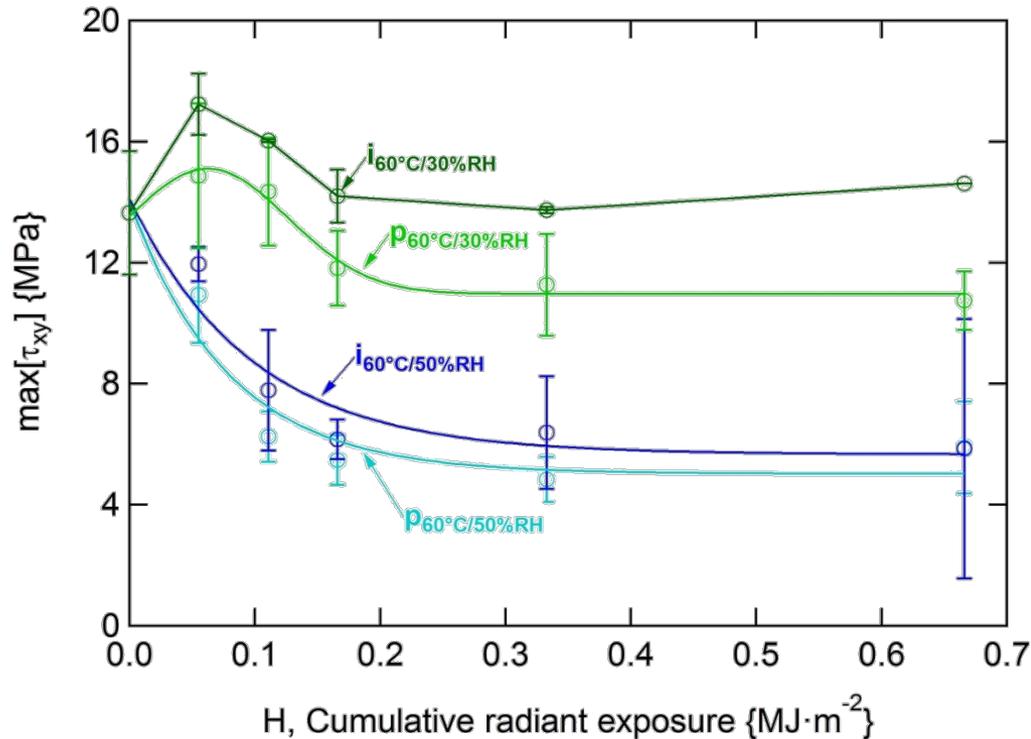
Possible explanations:

- Threshold of UV, T, or RH required to invoke substantial damage?
 - Effect is due only to absorbed moisture (polymer plasticization, not UV)?
 - $T_m \sim 60^\circ\text{C}$ for EVA. Phase change may affect rate of aging?
- ⇒ Additional results (e.g., dark chambers) should elucidate.

Humidity During Aging is Significant

What is effect of %RH? \Rightarrow 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.
- Reminds that %RH (during weathering, CST) is significant!!!



Weathering performed in Xe chamber (60°C/30%RH at 3M) or (60°C/50%RH at NREL).

- 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).

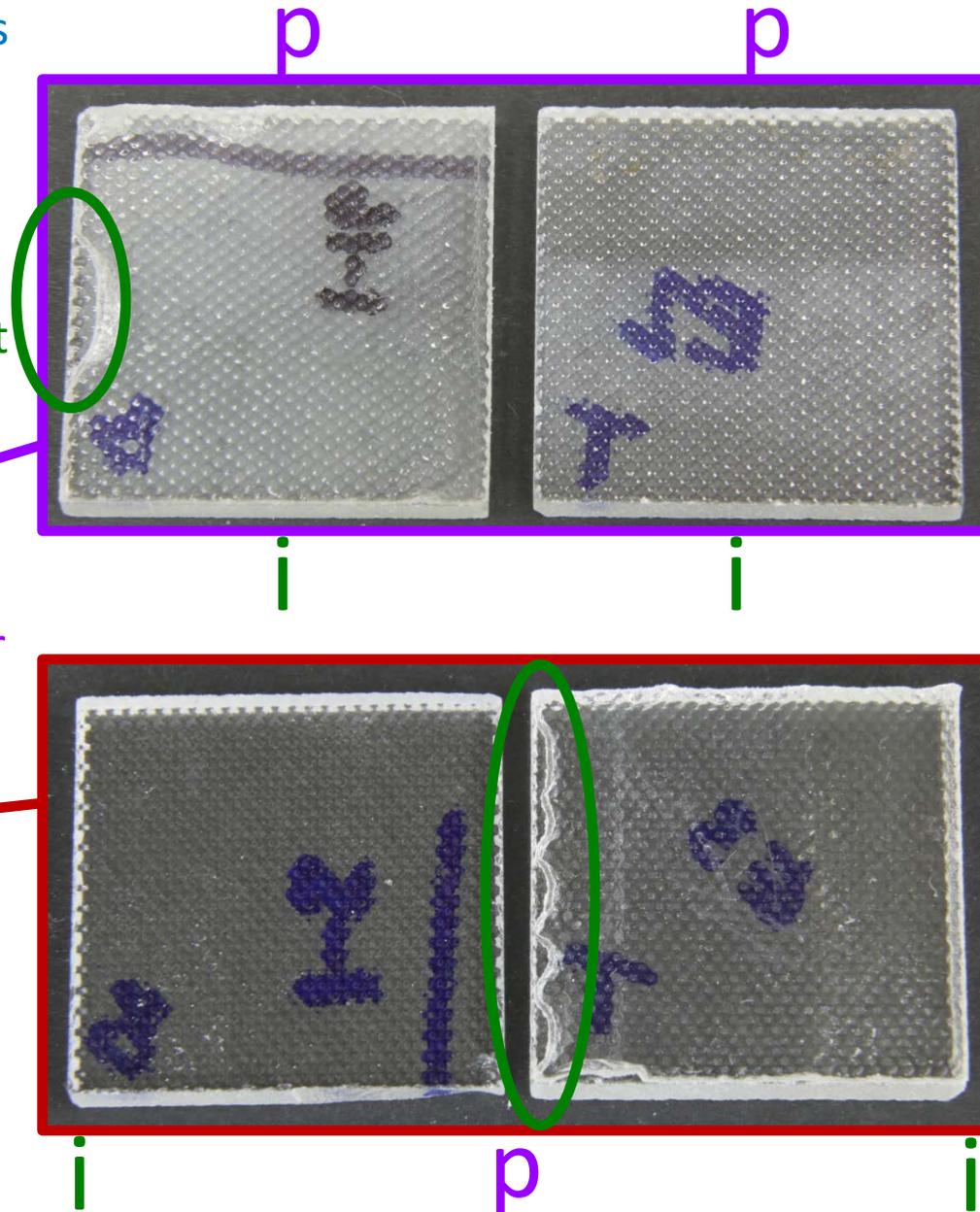
Typical Final Morphology of CST Specimens

- Majority of encapsulant typically remains on one side of the detached specimens. (Cohesive failure near the interface, not delamination).

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

- Most often, the encapsulant remains on the substrate, suggesting UV damage near the incident polymer/glass interface.

- Example: the detached surface was used to identify specimens facing oppositely during weathering. (Scribed # surface towards lamp).



Specimen Temperature Affects Final Morphology

- Failure observed on both incident and dark surfaces of same specimen.

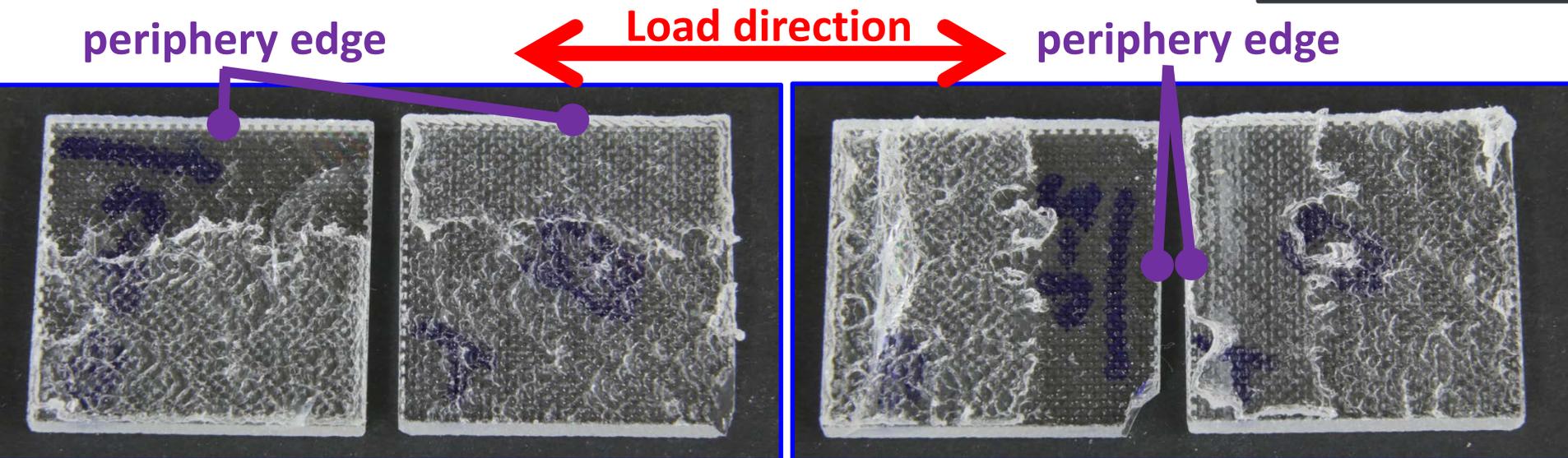
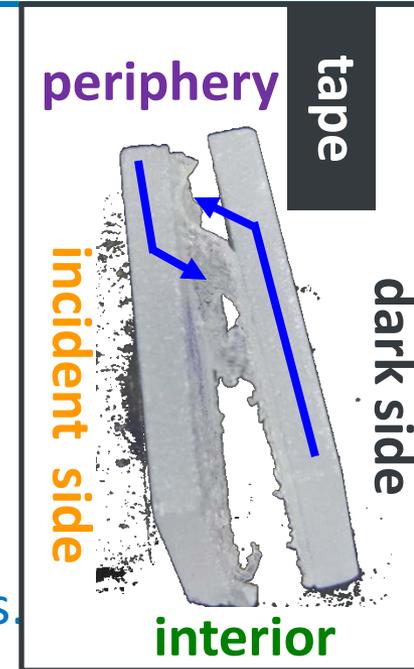
- Difference in morphology at edge and interior (fibrous).

Q: Explain the disparate surfaces?

- Samples fixtured using black double-sided adhesive tape.

- Morphology likely relates to hot edge ($\sim T_{\text{black panel}}$) vs. cool interior (transparent sample).

- $\max[\tau_{xy}]$ comparable for interior- & periphery-located samples.



Comparison, 3M aged samples sheared parallel (left); or perpendicular (right) to the original periphery edge.

Summary & Conclusions

Must treat effect of H₂O:

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

Some evidence of permanent damage from UV Weathering

- The slow acting degradation mechanism?
- Failure occurs proximate to irradiated surface

Very condition sensitive:

- $\Delta\tau_T$: 80°C >> 60°C.
80°C presently proposed in 62788-7-2.
- $\Delta\tau_{RH}$: 50% RH >> 30% RH.
20% RH presently proposed in 62788-7-2.
Need for greater understanding of effect of H₂O.

Periphery vs. interior location:

Minimal effect in EVA. (Greater effect observed other materials).

Caution:

- In-situ effect of UV not examined here. Permanent UV damage only.
- Glass/encapsulant may not be weakest interface.

Acknowledgements

☞ 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)
- Stay tuned for more complete examination of encapsulant attachment strength. IEEE PVSC 2016 conference.

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

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