NREL
NATIONAL RENEWABLE ENERGY LABORATORY

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15:00
Building 101, Portrait Room

DC Voltage Breakdown of Relied Upon Insulator Materials: Test Method Development and Results From Artificially Weathered Specimens

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Overview

- **Background**
  - -PV backsheets.
  - -Related & developing PV standards.
  - -Hydro- and UV- degradation of PET.

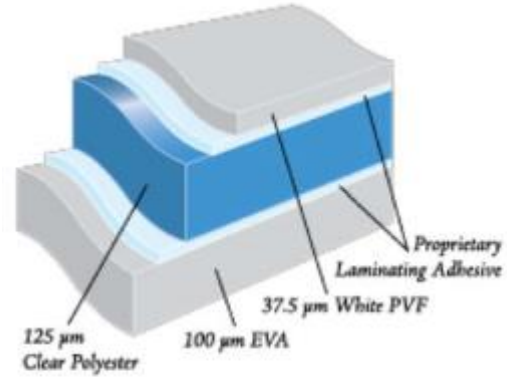
- **Interlaboratory precision study**
  - -Goal: verify repeatability & reproducibility
  - -What critical factors in the experiment were affecting precision?

- **Artificial weathering screen test**
  - -35 out of 55 materials.
  - -Focus on 7 BS's of interest.

- **Summary**
  - -Per study

PV backsheets

- PV backsheets typically consist of laminated polymer sheets.



Example of a TPE backsheet product.
Madico PORTEKT product literature.

- Traditional (benchmark) BS: "TPE".

MATERIAL	EXAMPLE THICKNESS {μm}	LOCATION	PURPOSE
TVF	17	air	UV protection
PET	250	core	electrical insulation
EVA	125	cell	adhesion to encapsulation

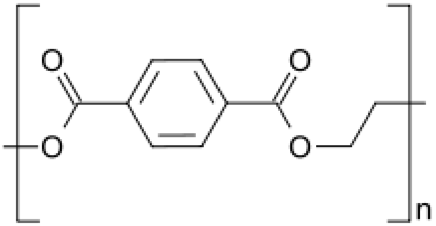
Geretschläger et. al., SOLMAT, 2016.

- Recent BS polymer materials:
  - poly(ethylene terephthalate) (**PET**, UV-stabilized)
  - high density PET (**PPE**, UV-stabilized)
  - polyvinylidene difluoride (**PVDF**)
  - tetrafluoroethylene (TFE) and vinyl (**PTFE**)
  - tetrafluoroethylene, hexafluoropropylene, and vinylidene fluoride laminate (**THV**)
  - polyamide (**PA**)

- PET:

Semicrystalline, oreinted, condensation-cured polymer.
(machine- and traverse- directional anisotropy.)

T_g ~ 75 °C; T_m ~ 260 °C.



Molecular structure of polymer repeat unit for PET.
https://en.wikipedia.org/wiki/Polyethylene_terephthalate

Standards Related Motivation for Breakdown Voltage Test

IEC 61730-1 ed. 2

- Electrical insulation is a key safety requirements for RUI's (backsheets & edge seals).
- IEC 61730-1 ed. 2 (2016) now specifies the strength of insulating materials: $\geq$2 kV + 4·$V_{sys.}$ Example: 8 kV V_{BD} required for a Class II 1.5 kV system.

Breakdown Voltage Test (IEC TS 62788-2, Annex C)

- A DC breakdown voltage (V_{BD}) test is part of IEC TS 62788-2 (frontsheets & backsheets).
- V_{BD} test replaces the unpopular AC "Partial Discharge" test.
- Interlaboratory study was conducted to develop and quantify the precision of the V_{BD} test.

-Miller et. al., Proc IEEE PVSC, 2017.

IEC 61730 ed. 2 Amendment 1

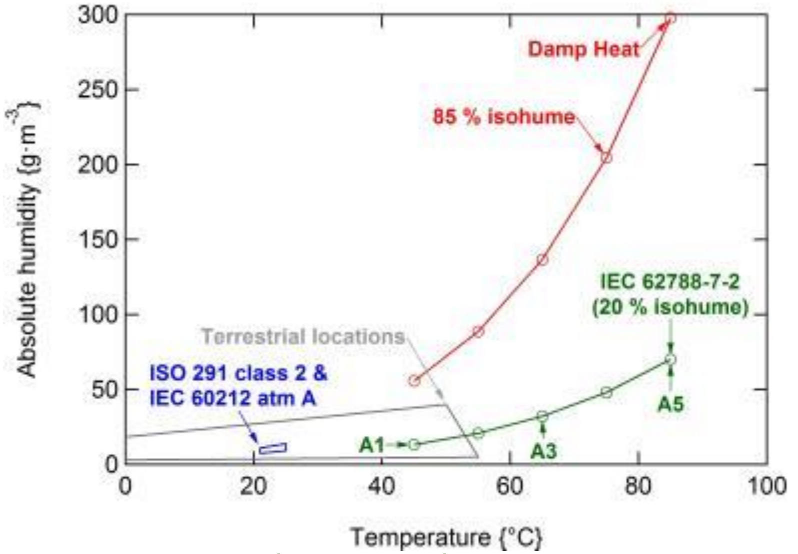
- Amendment to 61730-1 is presently considering adding a UV weathering requirement for Relied Upon Insulators.
- The Amendment project team is considering the EtB characteristic, with a pass/fail criteria.
  - The pass/fail criteria was recently debated.
    - Example: Δ of 50% from RTI/RTE. Starting point, but no have a strong technical basis.
    - Mechanical tensile test to identify cracking, not electrical insulation.
  - The weatherability of V_{BD} for backsheets is unexplored.
  - The V_{BD} of veteran materials is unexplored.

Hydrolysis of PET

- Mechanism: de-esterfication (random scission) of main chain.
- Arrhenius model valid for at least 65°C<T<95°C (module T_{max}).
- Second order dependence on %RH.
- $\ln[c_1] = 39.3$; $E_a = 129$ kJ·mol^{-1}; n=2.

$$\frac{dP}{dt} = c_1 \exp\left[\frac{-E_a}{RT}\right](RH)^n$$

- H_2O concentration in Damp Heat (85°C/85 %RH) greatly exceeds terrestrial environment & recent artificial weathering method(s).
- Rate analysis: significantly less hydrolysis is expected in artificial weathering (IEC TS 62788-7-2) relative to Damp Heat.



H_2O concentration: the terrestrial environment is compared to some present IEC PV artificial accelerated aging tests.

T {°C}	%RH {%}	RELATIVE RATE OF HYDROLYSIS {%}	IEC TEST STANDARD AND (CONDITION)
45	20	0.02	62788-7-2 (A1)
55	20	0.1	62788-7-2 (A2)
65	20	0.4	62788-7-2 (A3)
75	20	1.6	62788-7-2 (A4)
85	20	5.5	62788-7-2 (A5)
85	85	100	61730-2 (MST 53) 61215-2 (MQT 13)
37	66	0.08	Bangkok, hot day

Relative rate analysis for Arrhenius model for some present IEC PV artificial accelerated aging tests.

UV Degradation of PET

- Mechanism: de-esterfication (random scission) of main chain.
- O_2 inhibits cross-linking, effects products species via hydroperoxide chemistry.

With O_2: fluorescence (λ_x=340 nm, λ_m=460 nm).
With O_2: "masked" Δm (O_2 consumed with volatile & non volatile products).
With O_2: majority of damage for λ<315 nm.

No O_2 (well behind cell or in thick sample): greatest discoloration (ΔYI).

UV, always: formation carboxyl "acid" (end groups).

Hydrolysis: chemicrystallization. $\Delta\rho \Rightarrow \Delta\varepsilon$. Increased optical haze.

PET degradation (UV & hydrolysis):
- Manifest as mechanical damage (embrittlement: cracks; spalling; voids).
- Catalyzed by:
    - metal ions (from residual catalyst or soil)
    - formulation additives or residuals from manufacture
    - acid/base chemistry (hydrolysis)
    - self-catalyzed (carboxyl end groups, e.g., for hydrolysis)

About the Interlaboratory V_{BD} Precision Study

Backsheet material **specimens** (5 cm x 5 cm x thickness size).
- -polyethylene terephthalate (PET, 2 thicknesses),
- -polyvinyl fluoride (PVF),
- -PVF/PET/PVF backsheet product ("TPT").

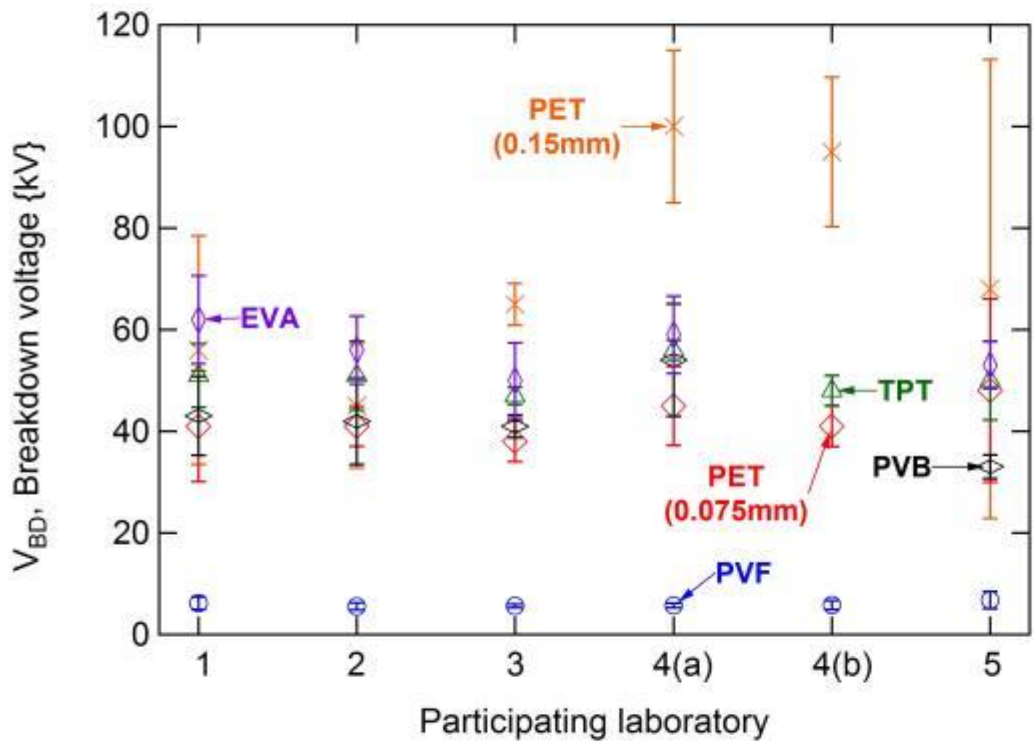
Encapsulant material **specimens** (unlaminated → no microvoids; no surface texture):
- -poly(ethylene-co-vinyl acetate) (EVA), unformulated (no residuals or additives)
- -polyvinyl butyral (PVB)

Test is **performed** in a dielectric medium (e.g., transformer oil) to prevent flashover and limit corona discharge.
- -IEC 60296 is more stringent than ASTM D3487.
- -Different medium products (IEC or ASTM certified) are available in different regions.

Analysis/result: median of the five replicate specimens; in cases where any of the results varied by more than 15% of the average, five additional replicates were tested. The dielectric strength was then determined from the median of the 10 replicates.

-Miller et. al., Proc IEEE PVSC, 2017.

Results of the IEC 62788-2 Interlaboratory Experiment



Results of V_{BD} R-R: The median VBD is given ±2S.D for each participant.

- r~10 kV; R~15 kV.
- V_{BD} PET (0.15 mm) >100 kV (lab 4); some results > 100 kV (lab 5); 56 kV (avg, all other labs).

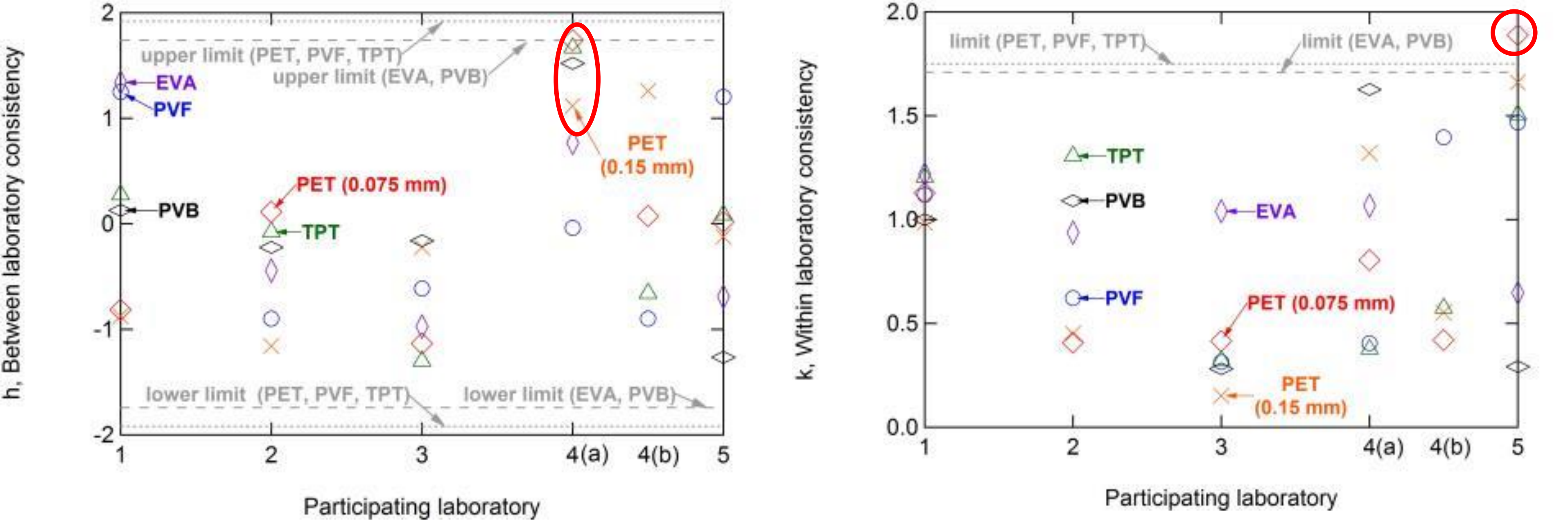
- NREL wanted to confirm the validity of test results prior to subsequent experiments
- Possible factors:

specimen thickness, multiple defect populations, # replicate specimens, sample conditioning, dielectric medium (oil), electrode roughness, unequal electrodes.

- Additional factors (not explored this presentation):

maximum current limit, rate of voltage rise, test polarity, ambient test temperature.

Results of the Interlaboratory Experiment (Continued)



h & k analysis of V_{BD} R-R

- Lab 0 was censored because it approached/exceeded the lower *h* limit for all materials.
- Low V_{BD} for lab 0 attributed to fluorinert fluid as dielectric medium ($\varepsilon_{dielectric} < \varepsilon_{specimen}$).

- Results lab 4 at boundary for *between-lab* variability for several materials.
- Contributing factors: multiple defect populations; small sample size.

- Results lab 5 exceeded *within lab* variability for 1 material.
- Multiple defect populations observed for PET.

Conclusions From the Interlaboratory Study

- **Intralaboratory repeatability (*r*)** was on the order of 10 kV (or 25%).
- **Interlaboratory reproducibility (*R*)** was on the order of 15 kV (or 30%) .
- Precision should improve for the final test method because at least 10 replicates will be required, rather the five replicates as examined in the round-robin.

- Factors including:
  - -**number** of **replicate specimens** (affecting on the order of $\pm$ 3%)
  - -**specimen conditioning** (~25 kV out of 55 kV for TPT)
  - -**dielectric medium** (~5 kV out of 55 kV for TPT)

  were found to readily effect test results… effect on *h*.
- **Multiple defect populations** were only found to be present in PET materials. 100 vs. 60 kV?
- Specimen thickness and electrode surface roughness did not significantly affect this study.

- Some **refinement of the published 62788-2 test method** resulted from this study, including:
  - -10 replicate specimens shall be used
  - -only transformer oil or mineral oil is allowed as a dielectric medium;
  - -use of oil qualified to ASTM D3487 may be used in addition to IEC 60296
  - -use of unequal diameter electrodes is not allowed.

-Miller et. al., Proc IEEE PVSC, 2017.

About the Artificial Weathering Screen Test

Backsheet specimens (5 cm x 5 cm x thickness size).

-35 white, black, or transparent products.

-Products with layers including: Al, EVA, PA, PET, PPE, PVDF, PVF, PTFE, thin film coating.

-Composition of outer layers verified using FTIR.

-$\geq$10 replicates each of:
Unaged and artificially weathered (IEC TS 62788-7-2, A3 for 2000 hours) specimens.

-Specimens conditioning per ISO 527 using a saturated $Mg(NO_3)_2$ solution.

Test performed per IEC TS 62788-2 Annex C.
-Mobil Univolt N61B transformer oil (ASTM D3487).

Analysis/results: Weibull analysis of 10 replicates per IEC 62539.
-90% confidence intervals for α and β
Asses variability of each specimen set.
Asses degradation from weathering.
-Good guidance on identification & treatment of outliers.

Failure analysis: focus on 7 representative results. Correlate with other characteristics.
TPE-1, TPE-3, TAPE, PET-1, PA, TPT-1, TPT-3

V_{BD} Results Will Be Compared To:

Mechanical (mandrel bend) test.

- Does cracking follow from just weathering or weathering + mechanical stress?
- 1.5 cm x 8 cm sheet specimens.
- Weathered in 250 h increments up to 4000 h cumulative.
- 6.35 mm ∅ stainless steel rod ⇒ 5% mechanical strain for 300 μm thick BS.

Kempe et. al., Proc. IEEE PVSC, 2017.

- **FTIR**.
  - -Look for overt change in chemistry.
  - -ZnSe ATR crystal.
  - -Data normalized to maximum intensity from 4000 cm^{-1} to 600 cm^{-1}.

- **Optical microscopy.**
  - -Look for cracks, roughness, delamination.
  - -Air- and sun-surfaces, sides, cross-sections.

General Results Screen Test Study

	UNAGED		WEATHERED		CHANGE (FINAL - INITIAL)		
Material (Shorthand)	α, Characteristic V_{BD}{kV}	β, Weibull Modulus {unitless}	α, Characteristic V_{BD}{kV}	β, Weibull Modulus {unitless}	α, Characteristic V_{BD}{kV}	α, Characteristic V_{BD}{%}	β, Weibull Modulus {unitless}
TPE-1	67-**68**-69	24-**36**-60	14-**17**-20	2-**3**-5	-52	-76	-32
TPE-3	83-**86**-88	12-**18**-31	39-**39**-40	21-**31**-51	-46	-54	12
FAE-2	95-**97**-99	19-**29**-48	42-**53**-65	2-**3**-4	-44	-45	-26
TAPE	63-**64**-84	48-**72**-121	37-**40**-44	4-**7**-11	-24	-37	-65
PET-1	87-**96**-105	4-**6**-10	62-**67**-71	6-**8**-14	-29	-30	2
PET-3	79-**84**-88	7-**11**-18	54-**59**-64	4-**7**-11	-25	-30	-4
PET-2	95-**97**-99	16-**24**-41	60-**105**-170 74-**77**-79	1-**2**-4 13-**22**-47	-20	-21	-2
PA	73-**78**-82	6-**9**-16	64-**68**-71	7-**11**-18	-10	-13	1
FPE-4	80-**84**-88	8-**13**-21	74-**75**-76	40-**59**-99	-9	-11	47
FPE-4	80-**84**-88	8-**13**-21	74-**75**-76	40-**59**-99	-9	-11	47
PET-4	76-**79**-83	8-**12**-20	73-**74**-76	19-**20**-48	-5	-6	17
PE	66-**67**-68	19-**28**-48	64-**65**-66	17-**25**-43	-2	-3	-3
TPT-3	50-**51**-52	19-**29**-49	49-**50**-51	19-**28**-48	-1	-3	-1
FAE-1	62-**66**-71	6-**10**-21	63-**65**-67	16-**28**-67	-1	-2	18
PVDF-4	64-**67**-70	9-**13**-22	64-**67**-71	8-**11**-19	0	0	-2
FPF-13	64-**64**-65	42-**62**-105	34-**50**-85	17-**25**-43	0	0	-12
PAE	47-**48**-50	12-**17**-29	51-**54**-57	8-**11**-19	6	12	-6
PPP-2	>100	N/A	73-**76**-78	12-**18**-31	?	?	?
TPT-1	>100	N/A	>100	N/A			

*V_{BD} results for materials tested to date. The Weibull scale and shape parameters are given for **90% confidence interval**.*

- 24 materials shown here. 11 remain to be tested.
- V_{BD} reduced >50% after 2000h IEC TS 62788-7-2 A3 (red backsheets).
- V_{BD} reduced >25% (orange backsheets).

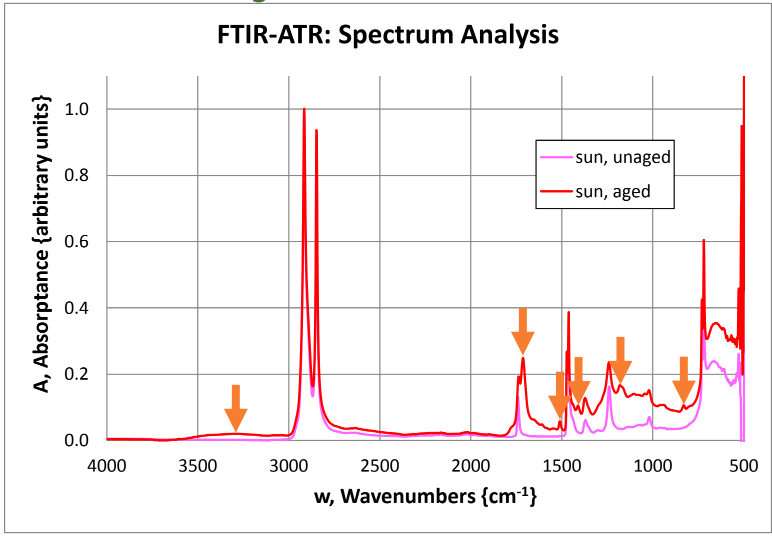
TPE-1 & TPE-3: Greatly Reduced Voltage With Cracking of the E-Layer

- ΔV_{BD} both materials exceeds 50%.
(Unaged & weathered BS's statistically distinct).

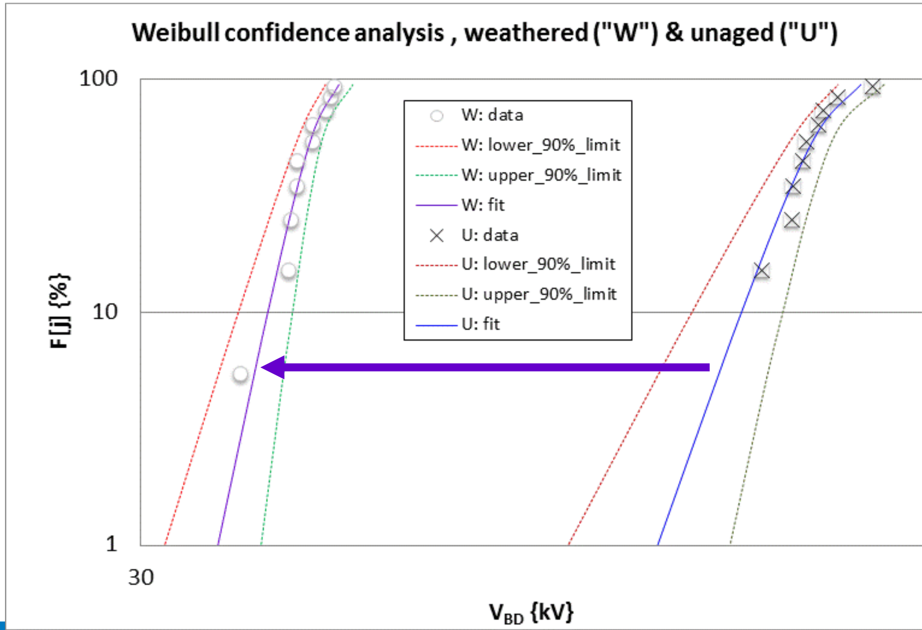
- Major (>10%) new peaks as well as peak broadening observed in FTIR for sun side (EVA).

- Cracking of sun side observed from weathering.
- Cracking may largely follow from the -film only- specimen geometry.

TPE-3: Overlay of FTIR spectrum for sun side (EVA). Notable changes are identified with an arrow.



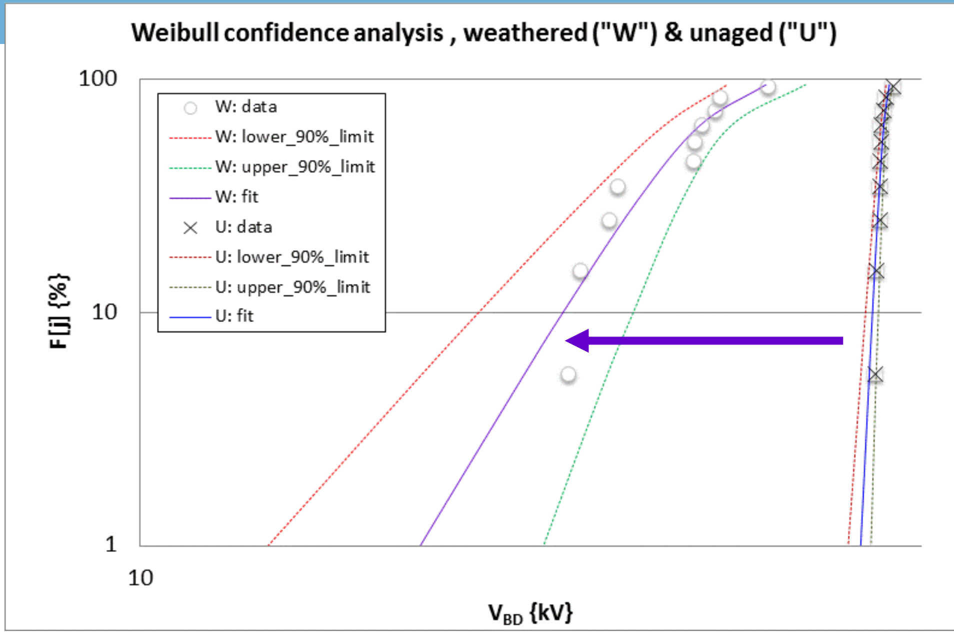
TPE-1: Failure was observed to interact with cracks on the sun side. Similar interaction may not have occurred for TPE-3.



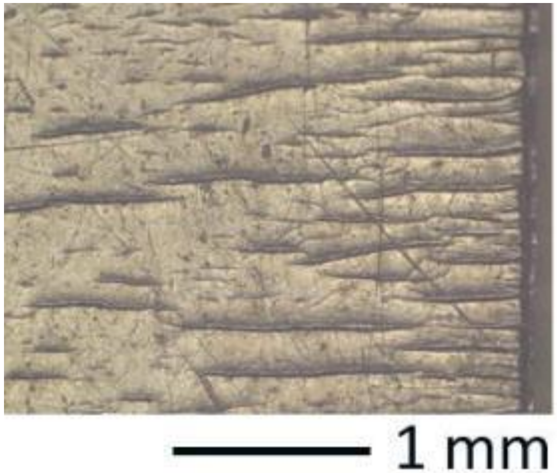
TPE-3: Overlay of test results; the 90% confidence intervals are shown for the unaged (U) and weathered (W) specimens.

TAPE: Different Electrical (V_{BD}) and Mechanical (Bend) Performance

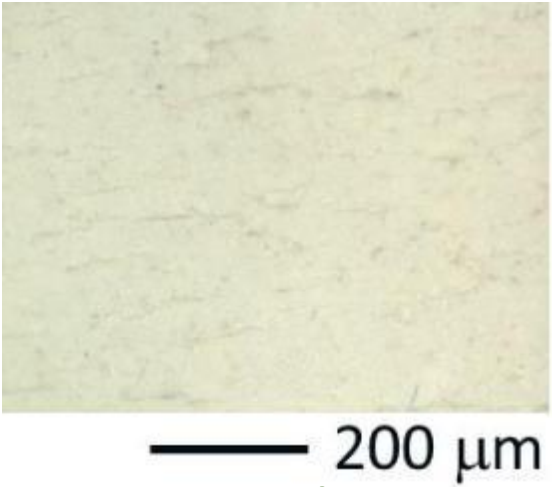
- ΔV_{BD} ~ 37%, with significant variability for weathered specimens.
- Major new peaks as well as peak broadening observed in FTIR for sun side (EVA). Similar to TPE's.
- Macroscopic cracking of sun side observed, with microscopic cracking of air side… both *only after bend test*.



TAPE: Overlay of test results; the 90% confidence intervals are shown for the unaged (U) and weathered (W) specimens.



TAPE: Cracking observed for the sun side (EVA) is shown in a microscopy image of the surface.



TAPE: Cracking observed for the air side (PVF) is shown in a microscopy image of the surface.

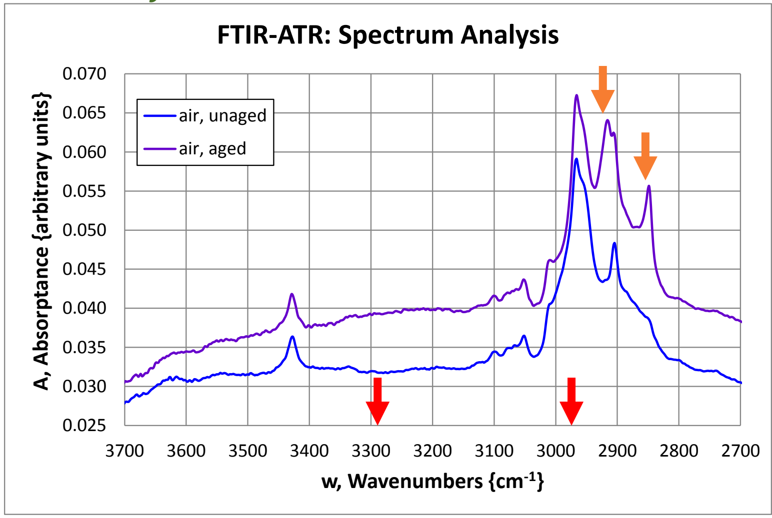
PET-1: Researched Core Material

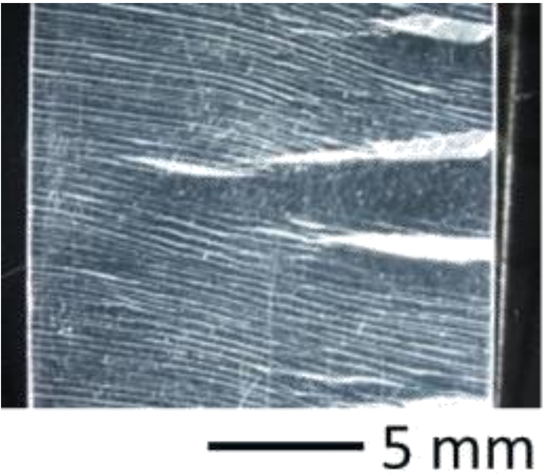
- ΔV_{BD} ~ 30% for weathered specimens.

- Minor new peaks in FTIR for monolithic material, air & sun side.
- Ratio at 3290 cm^{-1}/2970 cm^{-1} (-OH/C-H) may be used to assess carboxyl end group formation.

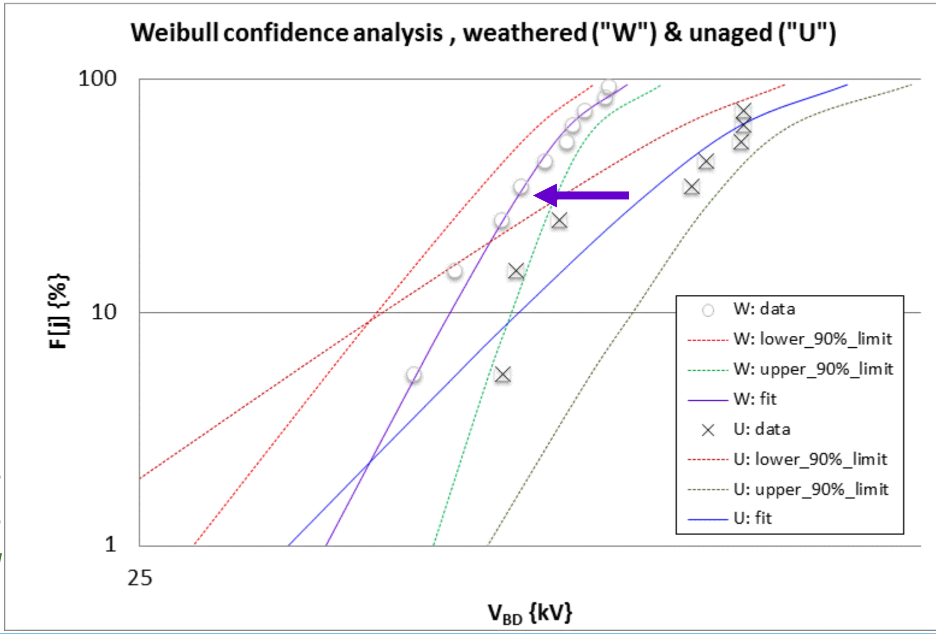
- Cracking observed after weathering.
- Modest discoloration (YI) from weathering; enhanced by dielectric oil.

PET-1: Overlay of FTIR spectrum for sun side (PET). Key features are identified with an arrow.





PET-1: Cracking is shown in a microscopy image of a bend test specimen.



PET-1: Overlay of test results; the 90% confidence intervals are shown for the unaged (U) and weathered (W) specimens.

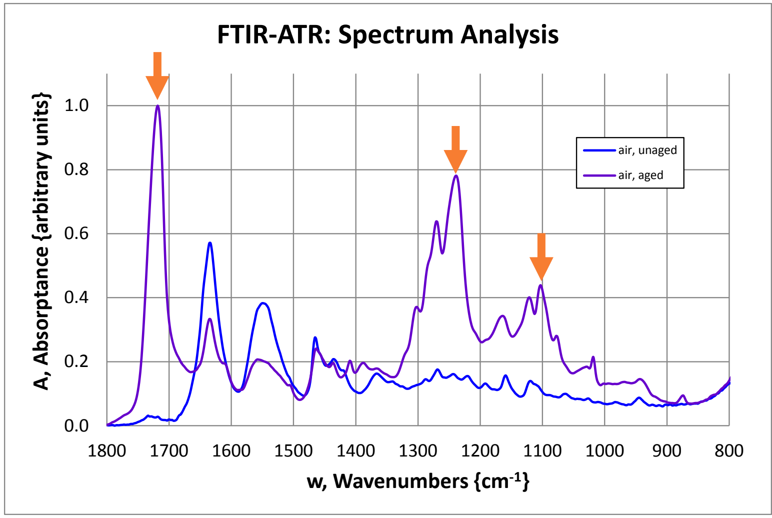
PA: Known Problematic Material

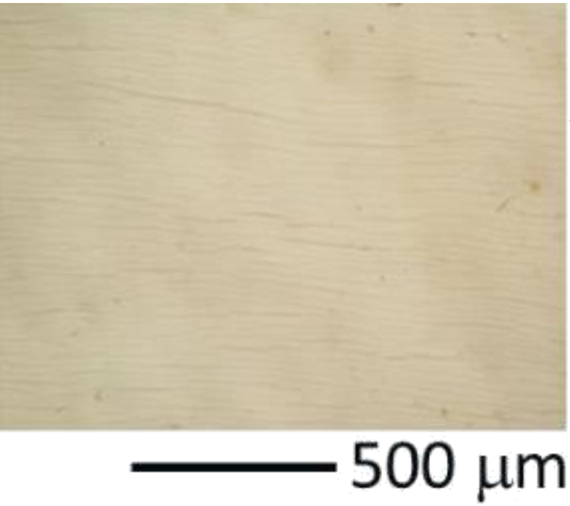
- ΔV_{BD} ~ 13% for weathered specimens.

- Major and minor changes observed, both intensity changes and new peaks.
- Similar changes on air (PA) and sun (PA) surfaces.

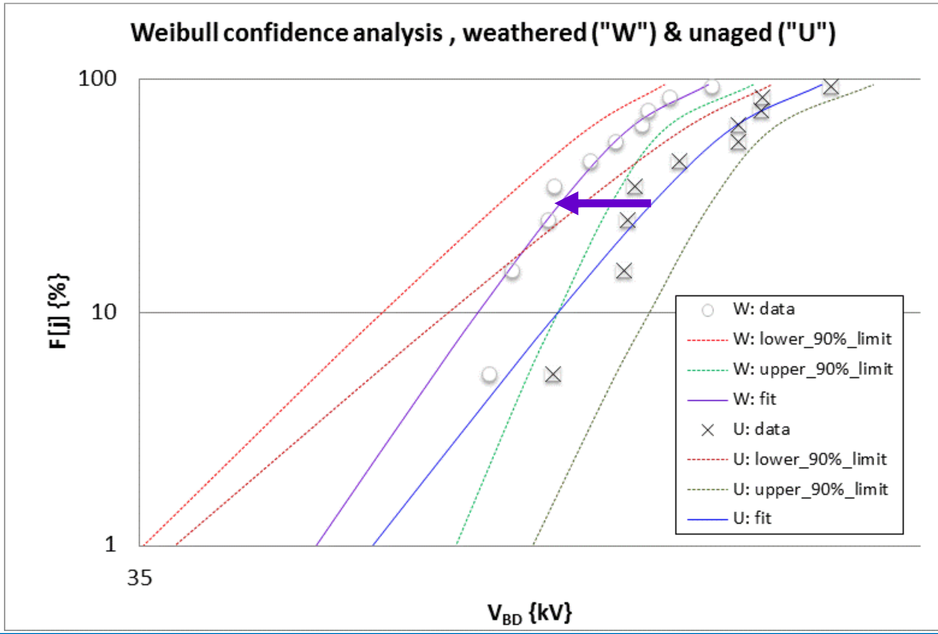
- Micro-scale cracking of air surface observed only after bend test.

PA: Overlay of FTIR spectrum for air side (PA). Some key features are identified with an arrow.





PA: Cracking observed for the air side (PA) is shown in a microscopy image of the surface.

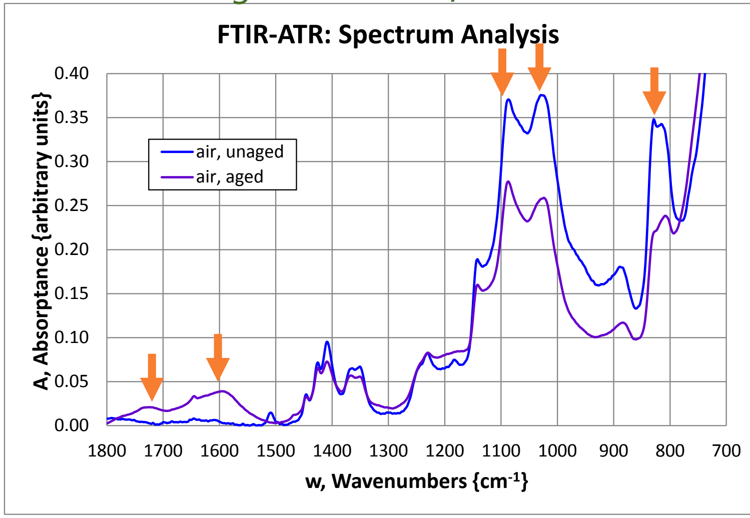


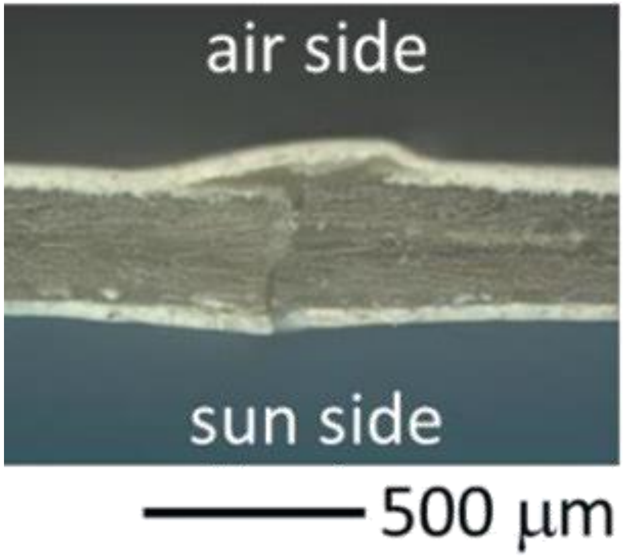
PA: Overlay of test results; the 90% confidence intervals are shown for the unaged (U) and weathered (W) specimens.

TPT-1: Different Electrical (V_{BD}) and Mechanical (Bend) Performance

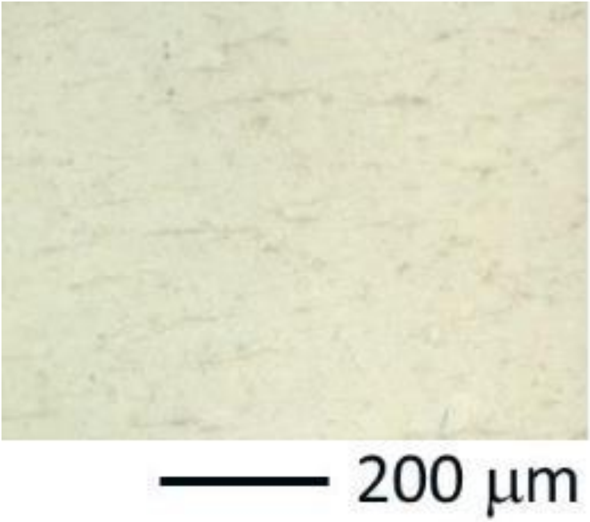
- ΔV_{BD} unknown (all values > 100 kV for unaged & weathered specimens).
- Changes in peak intensity observed in FTIR for air side (PVF).
- Cracking of air side observed only after bend test. Macro-damage: transferred to PVF from PET core. Micro-scale cracking: air side.

TPT-1: Overlay of FTIR spectrum for sun side (EVA). Notable changes are identified with an arrow.





TPT-1: Cracking observed for the air side (PVF) is shown in a side-microscopy image of the surface and edge.

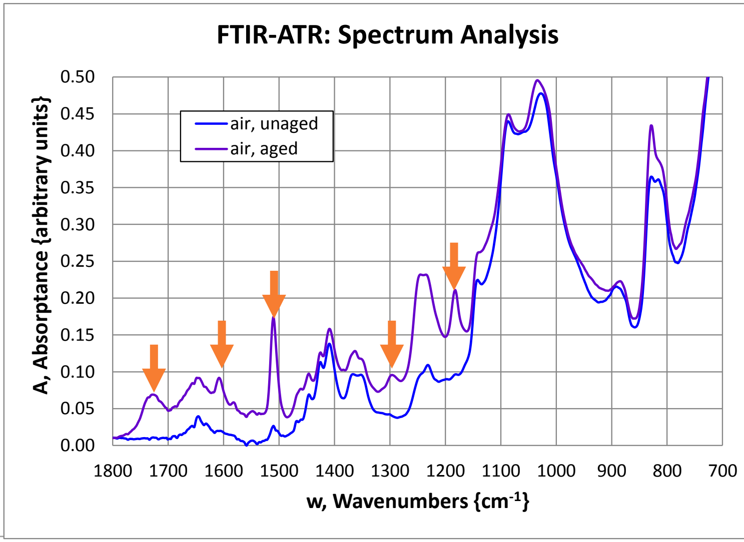


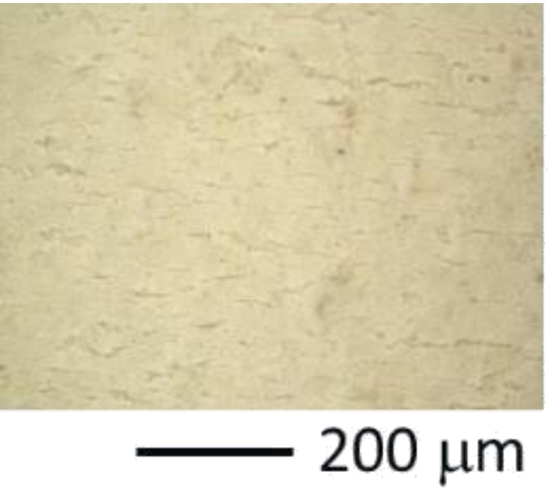
TPT-1: Cracking observed for the air side (PVF) is shown in a microscopy image of the surface.

TPT-3: PV Industry Benchmark Material

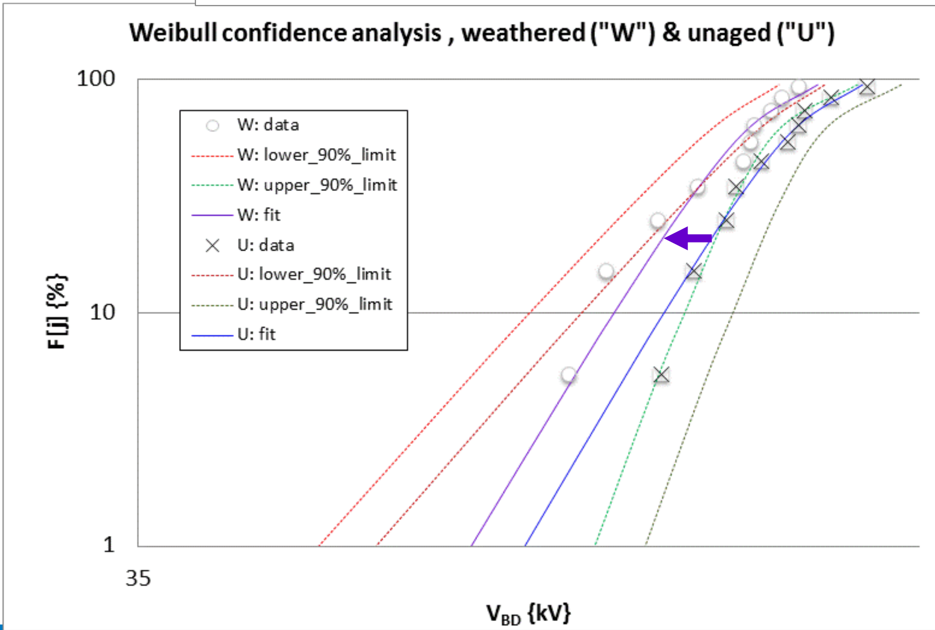
- Minor effect of weathering is suggested. (not outside 90% confidence bounds).
- Changes in peak intensity observed in FTIR for air side (PVF).
- Cracking of air side (PVF) observed in microscope, only after bend test.

TPT-3: Overlay of FTIR spectrum for sun side (EVA). Notable changes are identified with an arrow.





TPT-3: Cracking observed for the air side (PVF) is shown in a microscopy image of the surface.



TPT-3: Overlay of test results; the 90% confidence intervals are shown for the unaged (U) and weathered (W) specimens.

Summary of the Screen Test

- Large ΔV_{BD} sometimes observed. Specimens examined all exceed V_{BD} of 8 kV for a RUI.
  - -May be possible to reduce BS thickness (and module cost).
- Cracking did not always correspond to ΔV_{BD}.
  - -Use test sequence: weathering → mechanical → electrical?
- Improvement of diagnosis and acceptance “limits” for cracking are warranted.
- Measureable decrease in V_{BD} observed for some materials warrants additional study:
  - -Degradation & failure mechanisms.
  - -Specimen geometry used for weathering.
  - -Degradation as function of cumulative radiant exposure.
  - -V_{BD} as a function of ambient temperature.
  - -Artificial-weathering and -abrasion sequence.

INDEX	MATERIAL (Shorthand)	$\Delta\alpha$, Weibull scale parameter V_{BD}{%}	NOTE	EXTERNAL CRACKING (WEATHERING & MANDREL TEST). SCALE: SURFACE (MATERIAL)	EXTERNAL CRACKING (WEATHERING ONLY)	OVERT V_{BD} INTERACTION AT CRACKS?	FITR, AIR SURFACE		FTIR, SUN SURFACE	
							MAJOR CHANGE (>10%)	MINOR CHANGE (<10%)	MAJOR CHANGE (>10%)	MINOR CHANGE (<10%)
1	TPE-1	-76	large V_{BD} drop	macro: sun side (EVA)	Y	Y	N	Y	Y	Y
3	TPE-3	-54	large V_{BD} drop	macro: sun side (EVA)	Y	POSSIBLE	N	Y	Y	Y
6	TAPE	-37	large V_{BD} drop	macro: sun side (EVA) micro: air side (PVF)	N	N/A	Y	Y	Y	Y
22	PET-1	-30	large V_{BD} drop	macro: bulk (monolithic PET)	Y	Y	N	Y	N	Y
56	PA	-13	known bad material	micro: air side (PA)	N	N/A	TBD	TBD	TBD	TBD
16	TPT-1	?	$V_{BD} > 100$ kV cracked in mandrel test	macro: core→air side (PVF) micro: air side (PVF)	N	N/A	Y	Y	N	Y
40	TPT-3	-3	literature benchmark	micro: air side (PVF)	N	N/A	Y	Y	N	Y

Summary of the test results for the 7 representative materials.

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