Bayer MaterialScience

Meeting of GRSG - Experts on Plastic Glazing

2010-02-09 BMVBS Bonn, Germany

Polycarbonates

Data Content

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Plastic Glazing – experience and performance tests

- **Experience with polycarbonate as glazing material**
  - Used as windscreen (example)
  - Further action plan agreed between KRD & BMS includes investigation of used polycarbonate windscreens (optical appearance after life time)
  - Used in locations other than windscreens
- Main safety related difference between glass and polycarbonate
  - Impact resistance comparison
- Abrasion resistance
  - Wear resistance according to Taber compared with car wash test
  - Correlation of car wash to Amtec-Kistler (ISO 20566)
  - Test set-up to investigate wiper resistance
  - Comparison of different glazing materials with regard to wiper resistance
- Further properties of polycarbonate
  - Characteristic polycarbonate bulk properties
  - Weather resistance of coated polycarbonate

Forestry machinery drivers´ caps from John Deere

BayVision

- Substrate: 12 mm thick polycarbonate sheet Makrolon® GP 099 from Bayer Sheet Europe
- Reason: extremely demanding requirements for driver safety
  - FOPS (falling-object protective structures) test according ISO 8083
  - OPS (operator protective structures) test according ISO 8084

    can ONLY be fulfilled with polycarbonate

- TIER: KRD Sicherheitstechnik GmbH
- Coating: SHP401 / AS4000 from Momentive Performance Materials GmbH

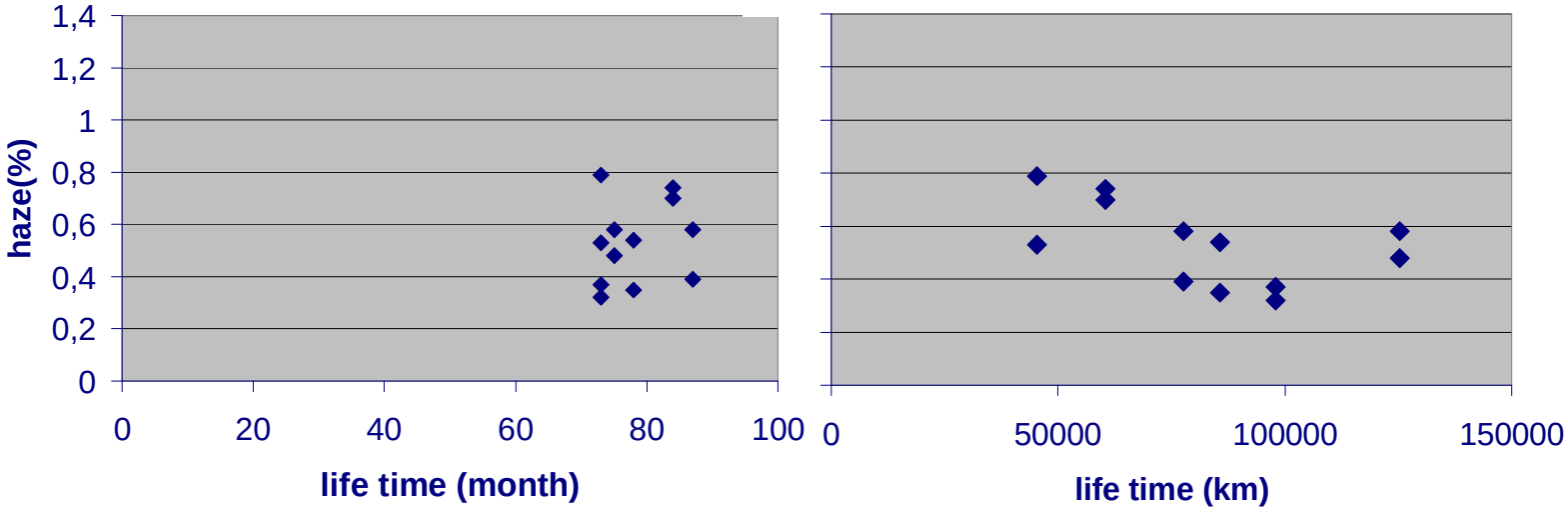
Drivers´ cap of John Deers new E-Series of vehicles for felling timber using polycarbonate glazing solutions from Bayer MaterialScience AG

SMART For Two side window

BayVision

- Substrate: Makrolon 2607 / Makrolon 2405
- TIER: Reiter & Scheffenacker
- Coating: PHC 587 (Eurogard)
- Location: Rom and Neapel, Italy
- Life time info: from 73 to 87 month (1998/1999 to 2005)
- No. of evaluated parts: 12 glazing parts from 6 vehicles

Haze values of the transparent part of the windows



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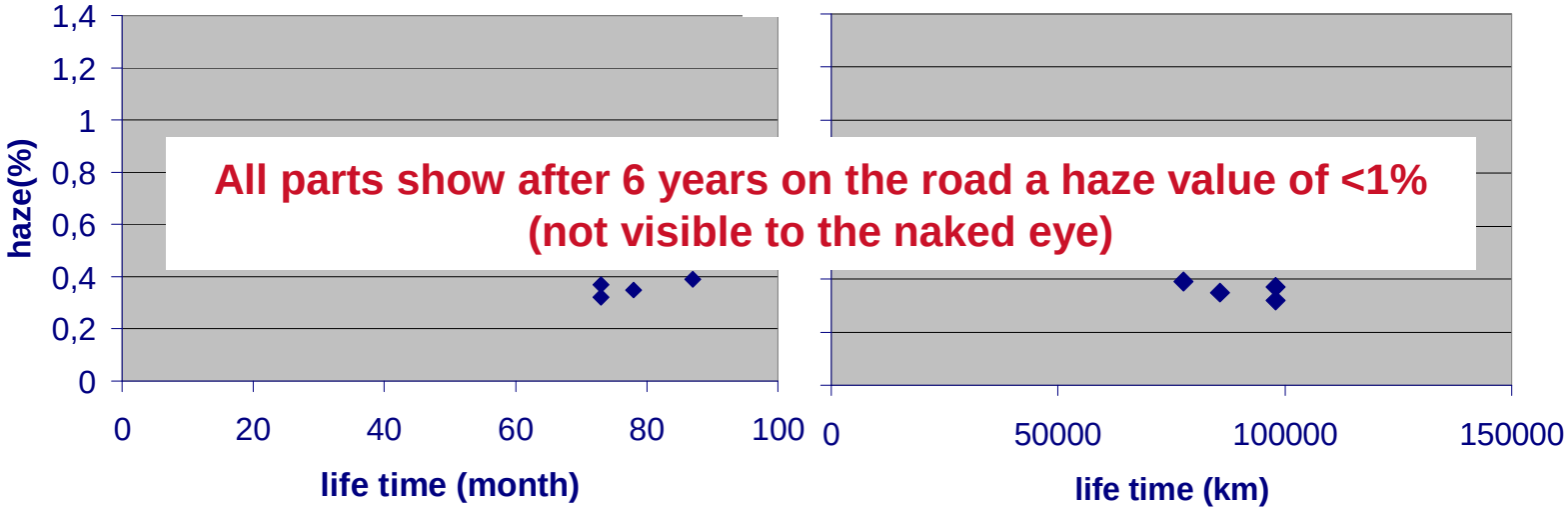

All parts show after 6 years on the road a haze value of <1% (not visible to the naked eye)

Ford Cougar head lamp lenses (driver side)

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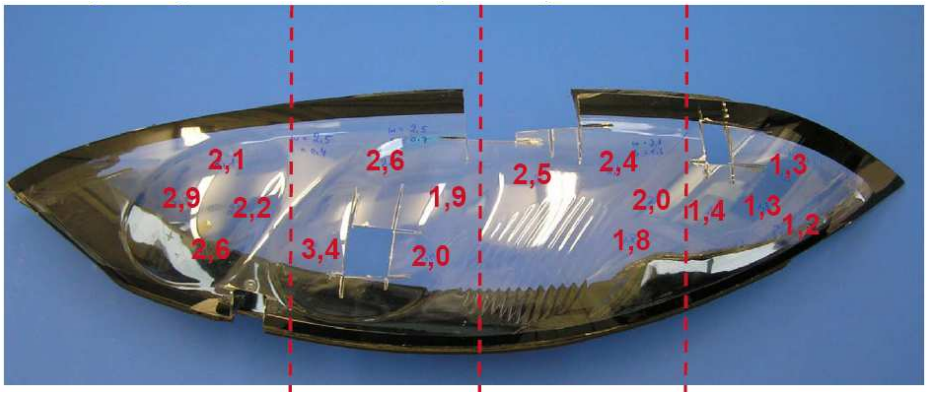
- **Wet Coat:** **SHP 401/ AS4000**
- **Location:** **NRW, Germany**
- **Life time info:** **82 month (01.2000 to 11.2006) & 84.000 km**

- **No. of evaluated parts: 1 part from 1 vehicles**



Haze values of the transparent part of the windows

front: haze 2,4 % (stone chipping)

side: haze 1,3 %

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Safety is the motivation

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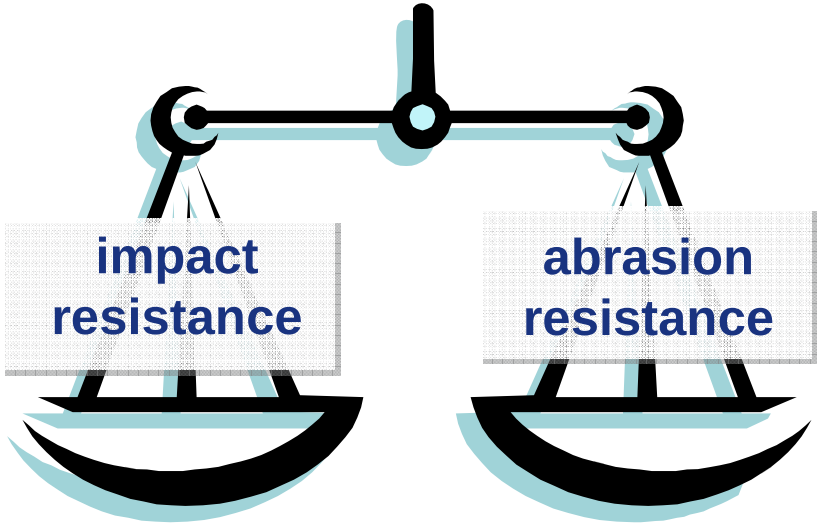
„All glazing materials, …, shall be such that, in the event of shattering, the danger of bodily injury is reduced as far as possible. The glazing material shall be sufficiently resistant to the incidents likely to occur in normal traffic, … .“

- … event of shattering …
- injury is reduced …
- … sufficiently resistant …
- in normal traffic …

(ECE R43 from 2000 in chapter 6. General Requirements)

PC vs. glass



impact penetration test

Taber abrasion test

Safety is the motivation

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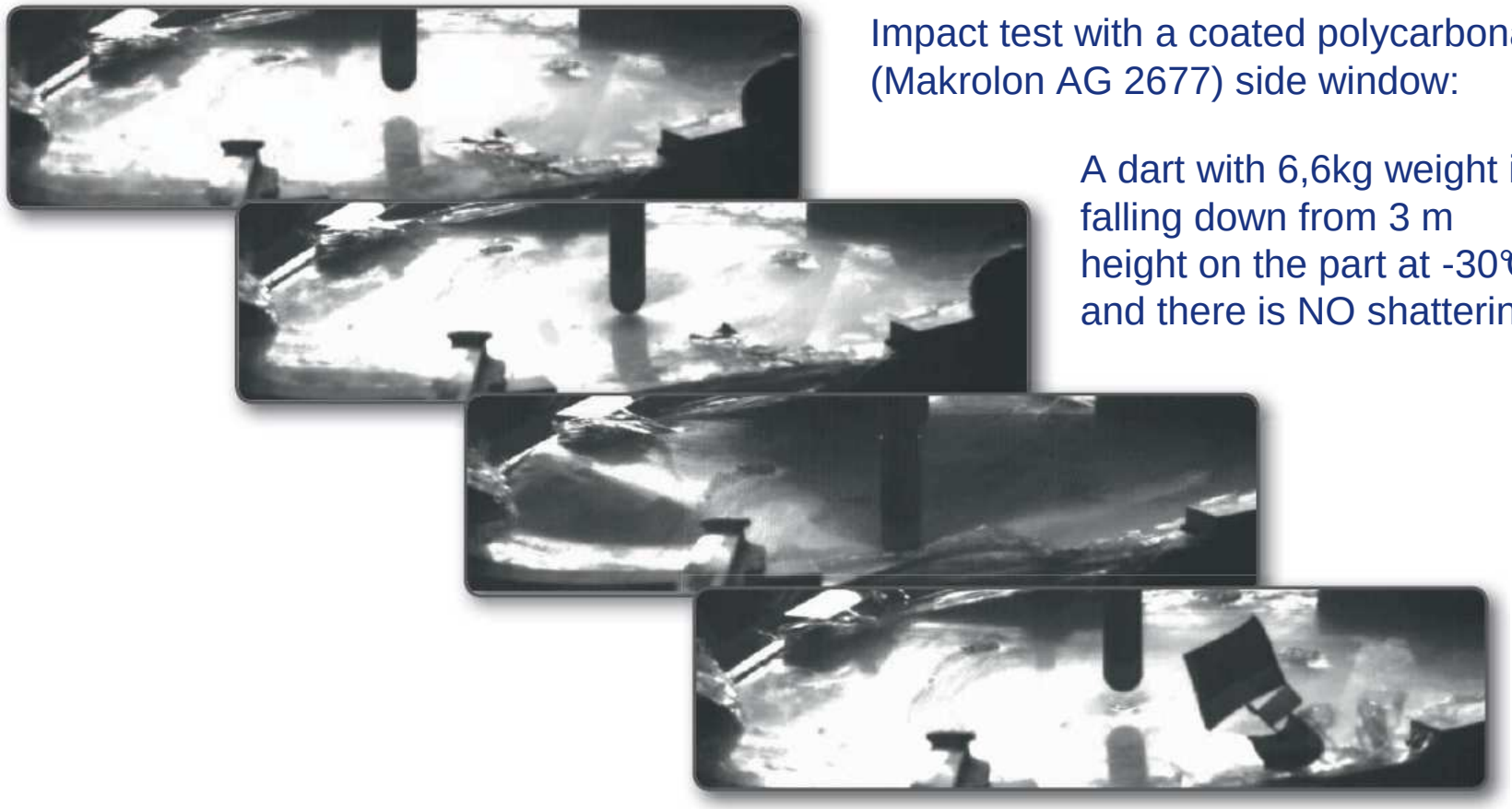

Impact test with a coated polycarbonate (Makrolon AG 2677) side window:

A dart with 6,6kg weight is falling down from 3 m height on the part at -30℃ and there is NO shattering.

ECE R43 mechanical requirements

BayVision

MECHANICAL STRENGTH TEST
227 g BALL TEST
according to ECE R43 Annex 14, Item 5

A hardened-steel ball with a mass of 227 ± 2 g and a diameter of approximately 38 mm is dropping freely from a height (2 to 5m) depending on the sheet thickness (≤3 bis ≥6mm) onto the test pieces having room temperature or -18℃ ± 2℃.

…The ball test shall be considered to have given a satisfactory result if the following conditions are met: the ball does not penetrate the test piece, the test piece does not break into separate pieces.

☑ Makrolon AG2677 coated on both sides with polysiloxane lacquers

HEADFORM TEST
according to ECE R43, Annex 14, Item 4

For panes like side windows, back windows and sunroofs which have reduced impact possibilities (classification VIII/B) the drop height shall be 1.5 m. The HIC value is also to be measured.

…The test piece or sample is not penetrated nor shall it break into fully separate large pieces. The HIC value is less than 1000.

☑ Makrolon AG2677 coated on both sides with polysiloxane lacquers

Testing mechanical properties

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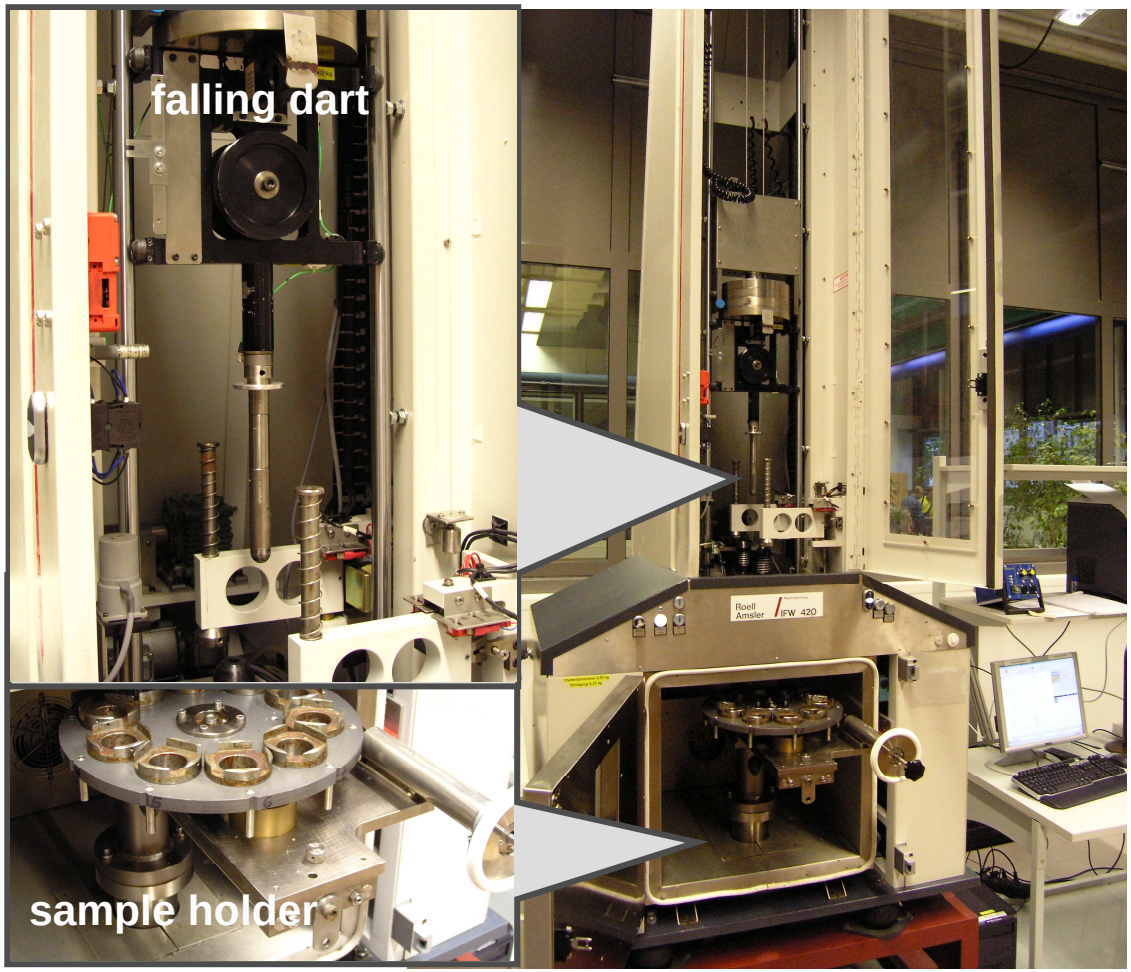
Impact Toughness

using a dart impact test according to DIN 6603

- mass of falling dart: 13kg
- diameter of falling dart: 20mm
- impact velocity from 2m/s to 13m/s
- adjustable temperature

➢ Basic mechanical properties determined on standard specimen test series

➢ Transfer on real part behavior by simulation models and spot tests



Dart impact test - results

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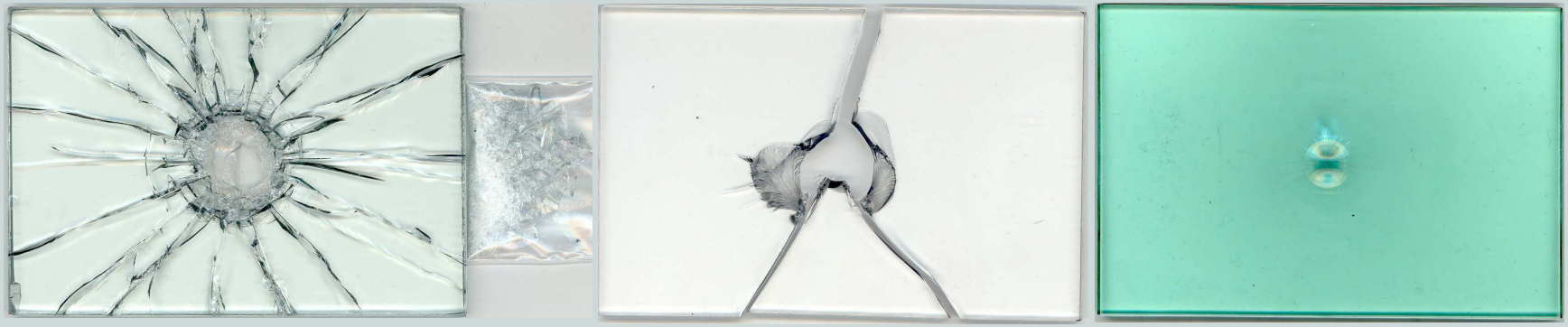
Fracture pattern at room temperature and an impact velocity of 2,3 m/s



laminated glass | polymethylmetacrylate | polycarbonate

Fracture pattern at -30℃ and an impact velocity of 2,3 m/s

Dart impact test - results

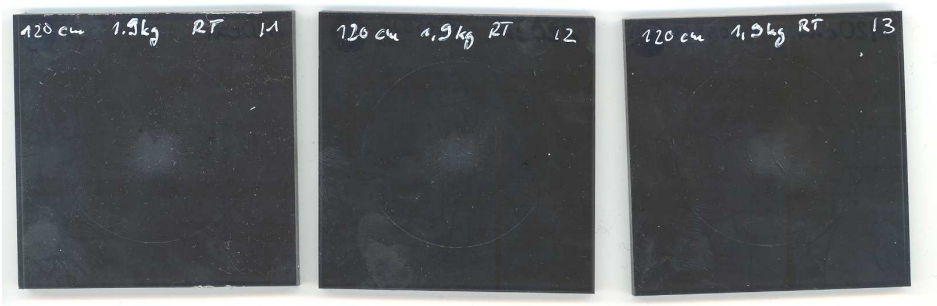
The impact of the so called **head form test** correlates (FEM - simulation based on an existing sunroof) with an impact achieved at a drop height of **15cm** using a small drop impact tester with a mass of falling dart of 1,9kg

laminated automotive glass

breakage at **5cm** drop height

slivered glass at **20cm** drop height

penetration of the film at **100cm** drop height



coated & weathered polycarbonate part no fracture at **120cm** drop height

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ECE R43 abrasion requirements

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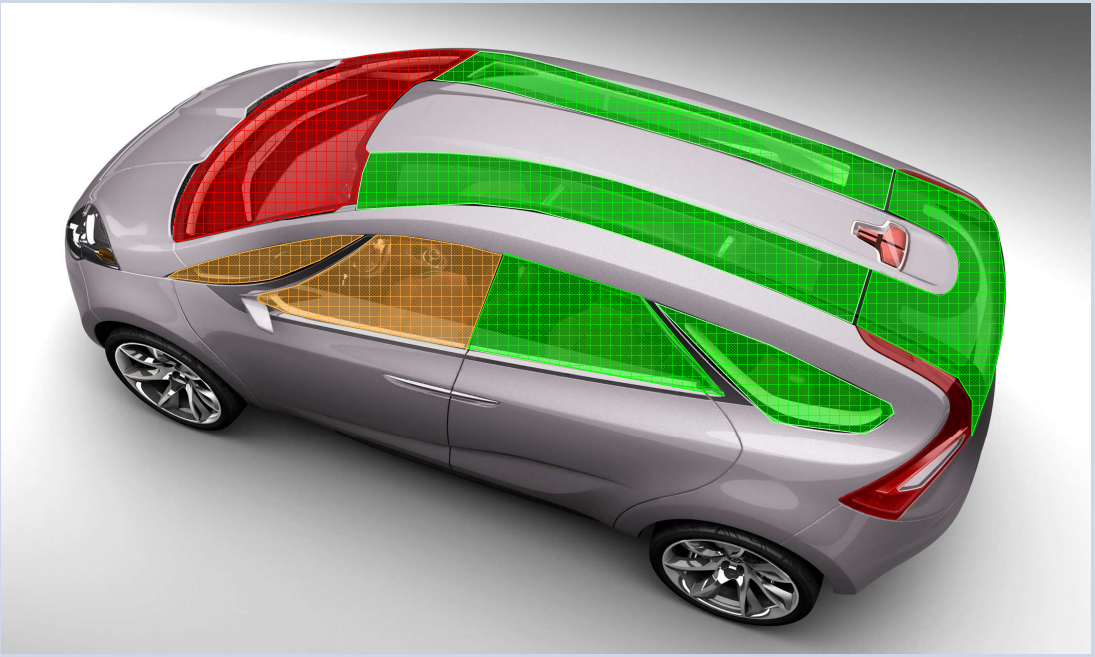
Wear protection depending on window location is defined via

- max. haze increase after
- certain exposure times
- using defined test set up (Taber Abrasion)

Questions:

- acceptable haze - level ?
- real wear during use ?



Windscreen: not allowed, resp. not defined

Diver visibility:

requisite: Δ_{haze} (Taber) < 2% after 1000 cycles

not requisite: Δ_{haze} (Taber) < 10% after 500 cycles

TEST Taber® Abraser Test

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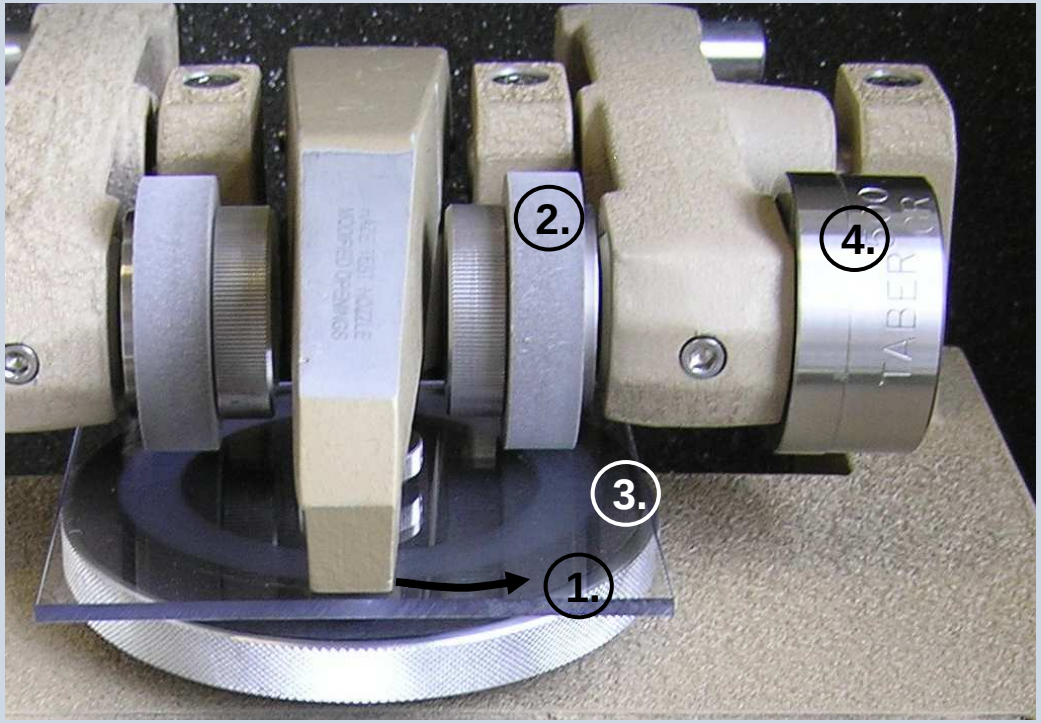
- **Test scope:**

  resistance against a material having abrasive particles in a resilient binder

- **Test advantage / disadvantage**
  - well known & accepted
  - correlation to real use **?**



1. **rotating specimen holder**
2. **abrasive wheel CS 10F**
3. **abraded track on the test plaque**
4. **500g load**

(ASTM D 1044 / DIN 52347)

Taber test - new CS-10F wheels

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Info from Taber Industries (www.cs-10f.com)

Taber's CS-10F Calibrase® abrading wheels manufactured between October 2002 and September 2004 may produce test results that are different than your historic values.

CS-10F (Type I)	CS-10F (Type II)	CS-10F (Type III)	CS-10F (Type IV)
			
Red color Original wheels Expired 2003	Pinkish-red color Low haze reading due to residue	Pink color High haze readings	Brown color New (now the only available) wheels

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CS-10F (Type I)

CS-10F (Type II)

CS-10F (Type III)

CS-10F (Type IV)

Wheel specification

i.) dimension (45 to 50 mm diameter & 12.5 mm thick) and
ii.) hardness (72 ± 5 IRHD)
no further specifications (e.g. amount or kind of particles, binder material, etc.)

fulfilled for all types (besides III)**,** but nevertheless the different wheels generated values for AS4000 coated polycarbonate from < 2 to >10 % (25% for III) after 1000 cycles

Taber test - test method changes

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ASTM D 1044 – ASTM Subcommittee D20.10 has a work item WK4442 that was submitted July 2004 which addresses the proposed changes for ASTM D 1044.

ANSI Z26.1 Test 17 – In September 2004, SAE's Safety Glazing Committee received a replacement document for the abrasion resistance tests.

- To ensure repeatability and reproducibility, it is advised that you verify the test method being used is current and reflects the most up-to-date version. All companies following ASTM D1044 should be using the test method released in 2005 (referenced as ASTM D1044-05).

"Important" Changes:

Wheel Refacing :

- 25 cycles using the fine side of an ST 11 refacing stone (old: ASTM: 25 cycles ST 11 stone; Taber manual: 25 cycles S 11 refacing paper)

Cleaning Procedure:

- Soft bristle, anti-static brush and isopropyl alcohol soaked lint free cloth (old: ASTM: soft camel´s hair brush)

Apparatus:

- Vacuum pick-up nozzle with enlarged opening

Taber test – measurement system and equipment capability

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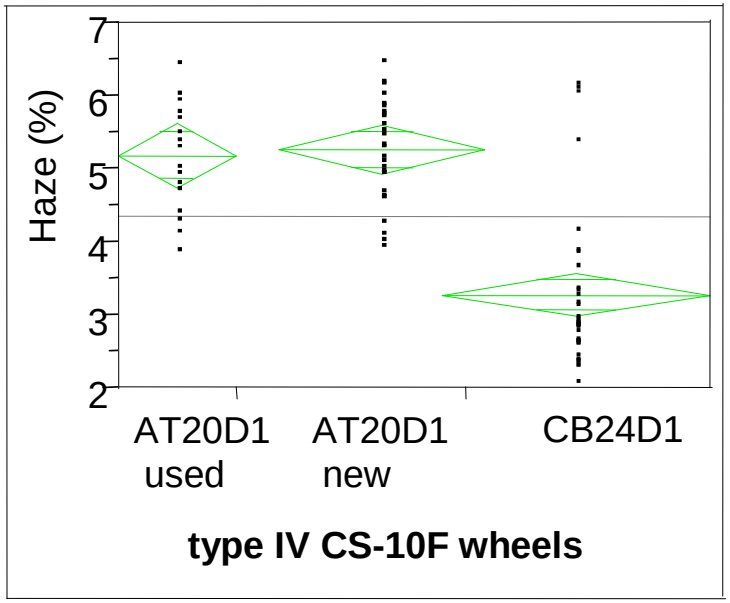

Oneway Anova

Information Taber: all new wheels are comparable; the first type IV wheels released, still had some variations in quality

Email from Taber Industries 2007-11-16:

Our quality records for wheel lot AT20D1 indicate that they are within our internal tolerances. Lot CB24D1 also meets our internal specifications, but the quality requirements that are in place for this wheel lot are more stringent than the earlier type IV wheel lots (of which AT20D1 is). After we released the Type IV generation CS-10F wheels, Taber continued to evaluate our manufacturing processes to ensure consistency of our wheels.

The most significant improvement to be implemented from this effort **was expanding our testing protocol to incorporate additional materials during our final approval process**. Please recognize that you are comparing data from a first release product run with the current wheel. While the wheels may have passed our internal tests, we never went back to confirm if the initial lots would pass the more stringent requirements.

Taber test – measurement system and equipment capability II

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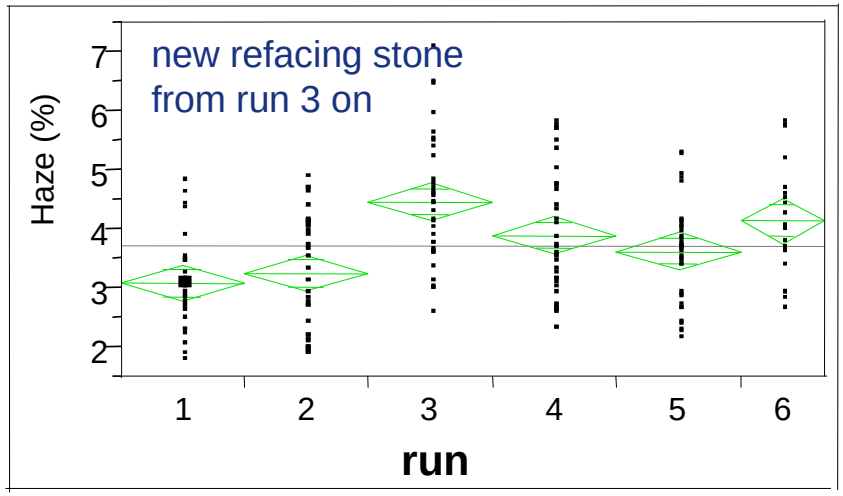
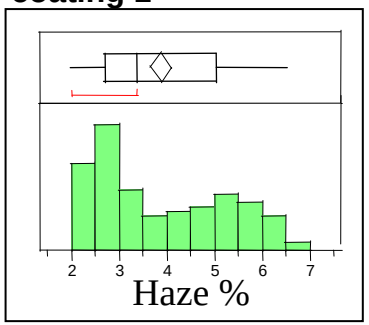
effect of the refacing stone (across all coating systems)



- switching to a new refacing stone increases the measured values
- this effect occurs with all coating systems

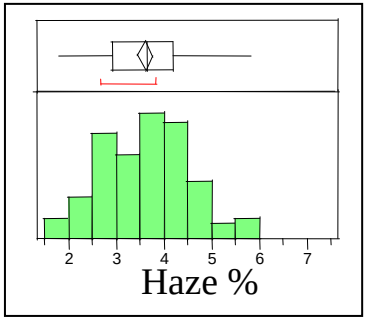
Comparison of different coating systems

coating 1



average value: 3.9 %, standard deviation: 1.3 %

coating 2



average value: 3.6 %, standard deviation: 0.9 %

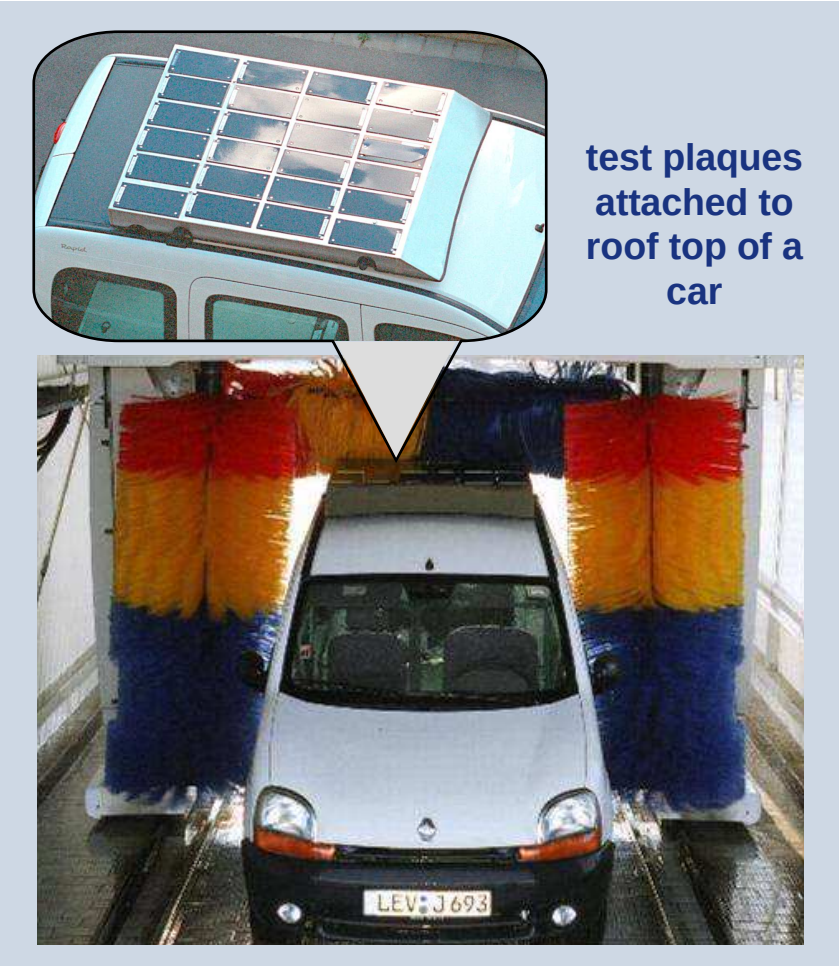
- strong deviation depending on coating system
- in some cases the deviation is as large as the given limit of 2% Δ haze
- some tests pass the <2% Δ haze limit

TEST Real car wash test

- **Test scope:**
  resistance against car wash conditions

- **Test conditions:**
  - test plaques getting dirty during outdoor use in Lev & cleaned once a week in a car wash
  - X-shaped, spliced, PE bristle-containing brushes
  - no wax / cleaning agent

- **Test advantage / disadvantage**
  - realistic
  - not standardized



test plaques attached to roof top of a car

Taber vs. car wash

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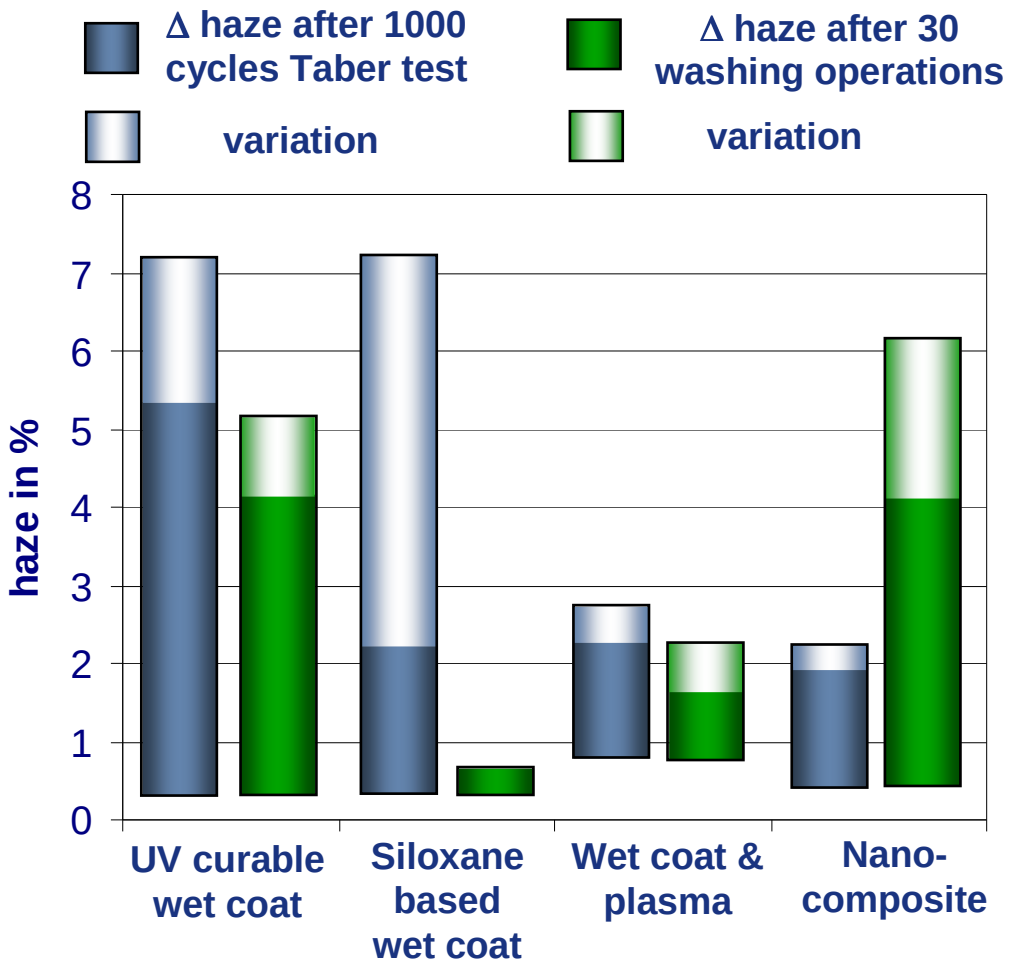
- no correlation
  - ➢ realistic abrasive wear test ?

Car wash ✔ Taber ?

- the car wash performance of siloxane based wet coats on PC is better than systems which fulfill the "Δ haze (Taber) < 2% after 1000 cycles" - requirement



Car wash - long term results

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- long term performance

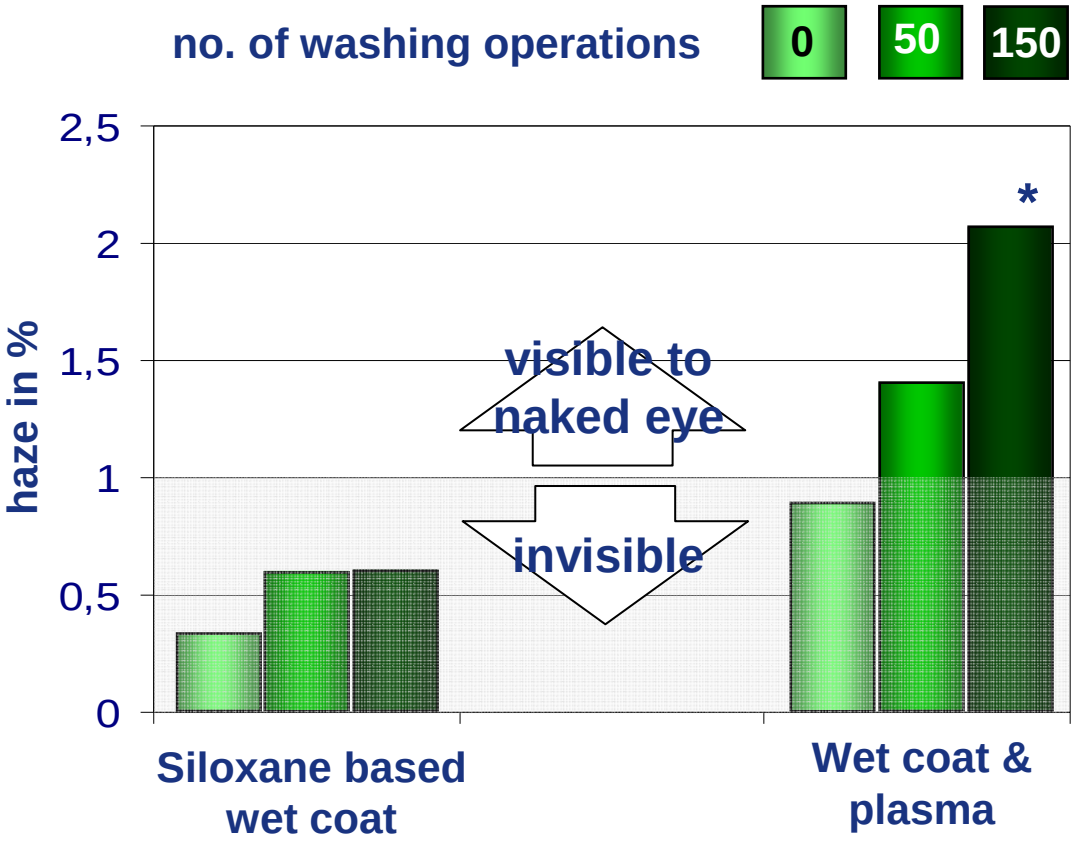
Siloxane based wet coat on PC ► no visible haze even after long term exposure

ECE R43:

"… sufficiently resistant … in normal traffic …"

➢ acceptable haze – level ?

invisible ✔



* > 50 washing operations delamination of the plasma layer from the wet coat occurs

TEST Amtec-Kistler (ISO 20566)

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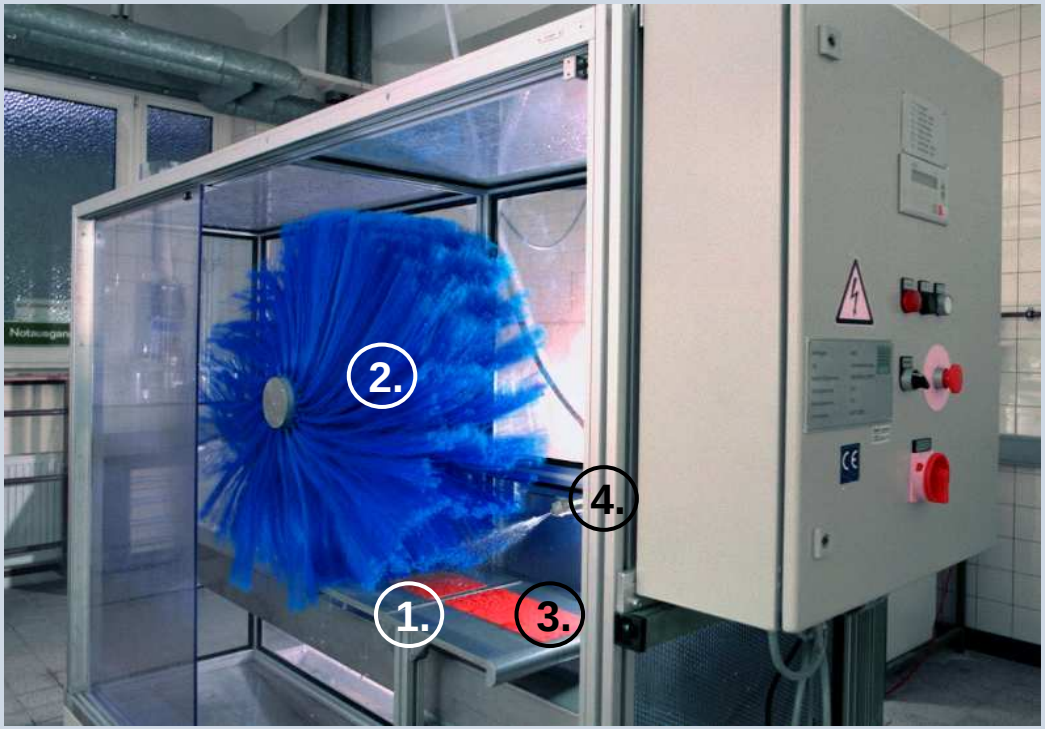
- **Test scope:**

  scratch resistance of coatings against laboratory car wash (used to classify car body paints)

- **Test conditions:**
  - gloss or haze determination after 10 washing operations (double strokes) with a
  - penetration depth of the bristle of 100mm using
  - quartz powder as artificial dirt



1. **sample holder moving back and forth**
2. **rotating brush using x-shaped, spliced PE bristles**
3. **test sample**
4. **“1,5g quartz powder per 1l tap water” sprayed with 2,2 l/min at 300 kPa**

Car wash vs. Amtec-Kistler

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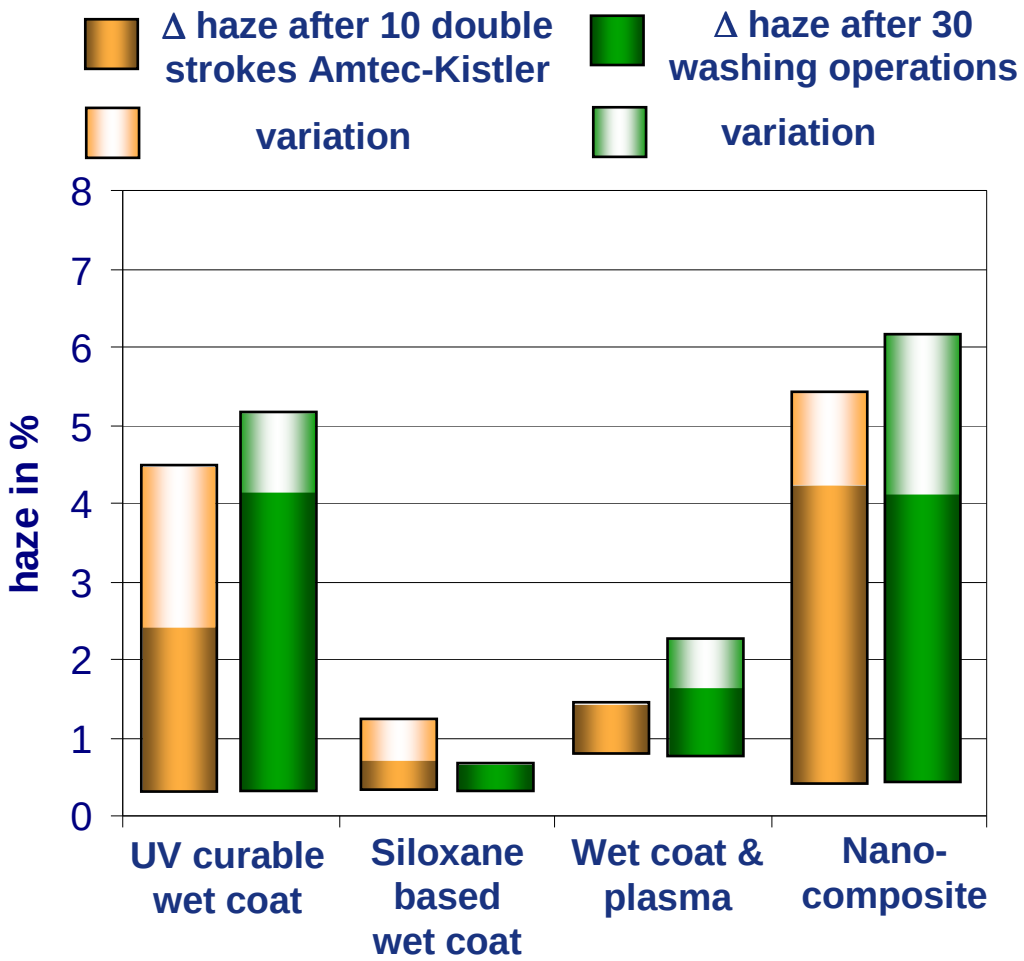
- good correlation between real and laboratory car wash
- in contrast to the comparison with Taber here the same two systems appear equally suitable
  - ➢ Amtec-Kistler would be a suitable test for glazing parts to check wear resistance against car wash conditions



Wiper resistance - real vs. lab

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- realistic wear test ✔
- invisible haze ✔ increase (below the hurdle of an (undefined) acceptable one)

Wiper resistance - real vs. lab

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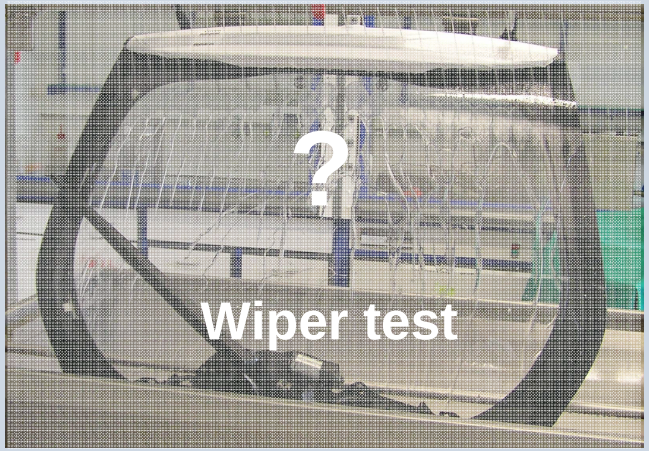

down-size

check correlation

lab test wiper resistance

TEST Wiper lab test

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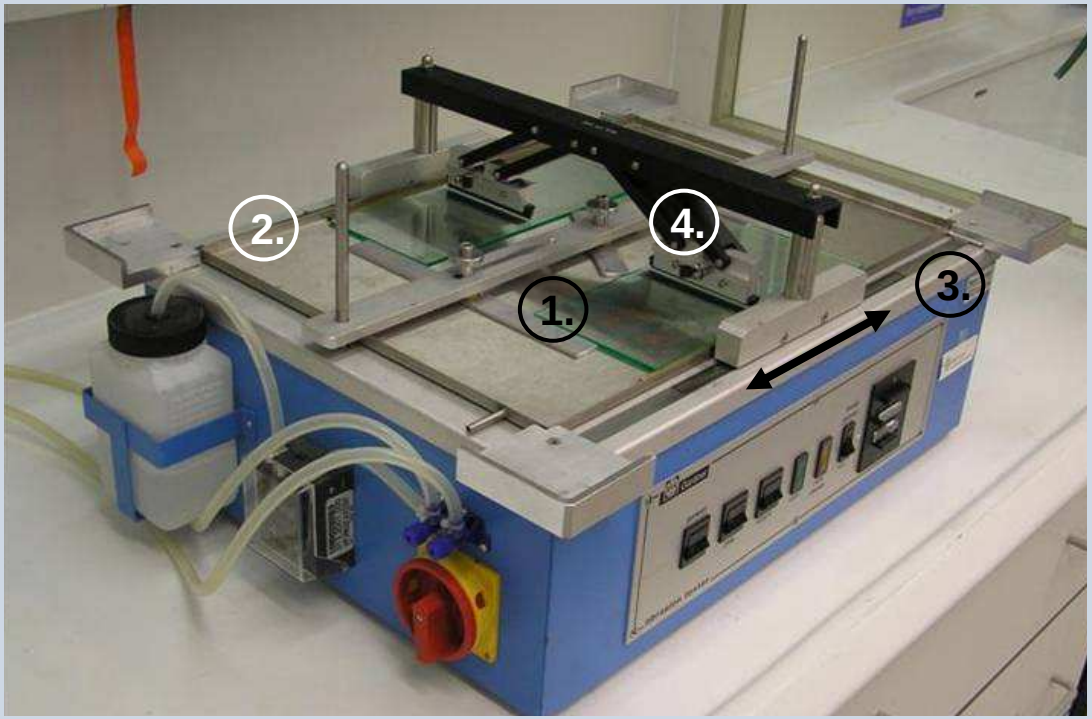
- **Test scope:**
  resistance against rubber based wiper

- **Test conditions:**
  - no. of wipe double strokes
  - dry / wet strokes with or without standardized dirt (ISO 12103)
  - different rubber types for the wiper and different substrates



1. **fixed test plaque**
2. **basin for testing under wet conditions**
3. **wiping direction (back and forth is one double stroke)**
4. **moving rubber wiper**

Wiper lab test results

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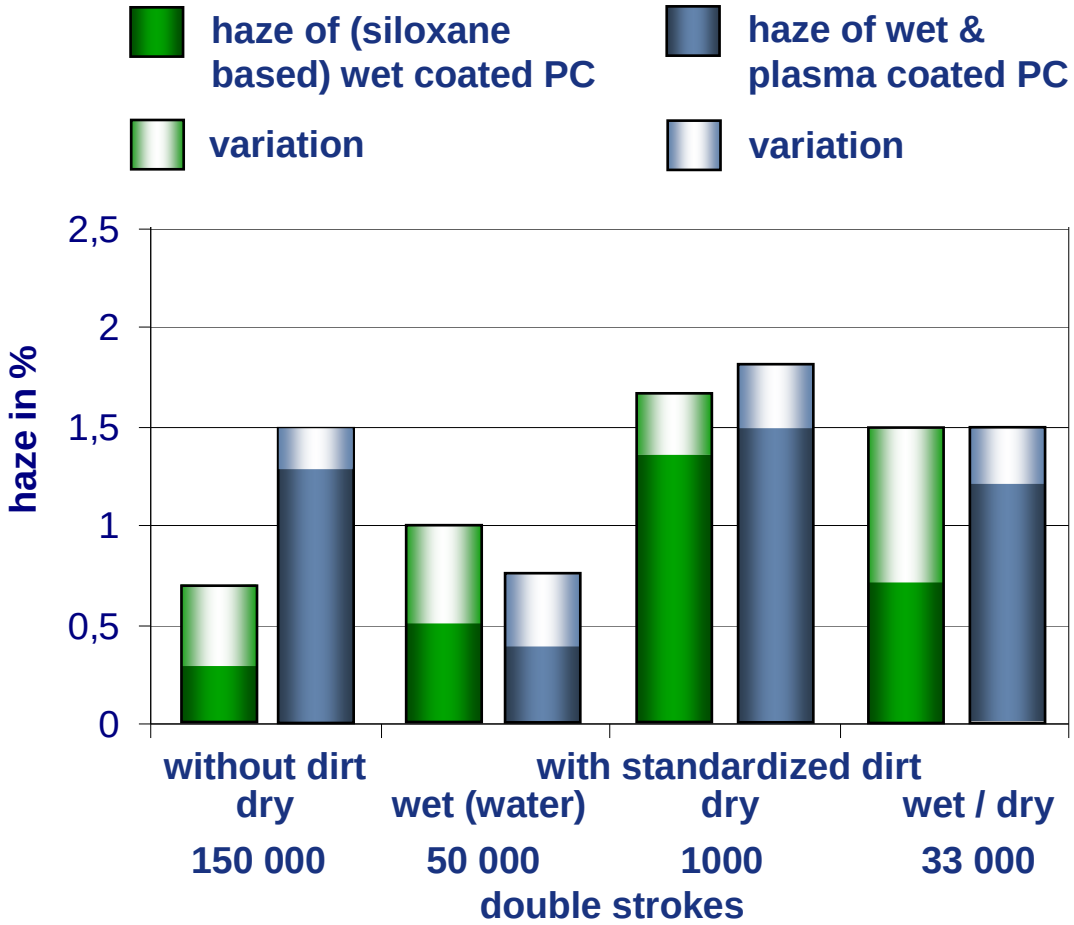
- resistance depending on test conditions
  - without dirt ✓
  - with water ✓
  - dirt & without water

- systems reaching the "Δ haze (Taber) < 2% after 1000 cycles" criteria are not performing better



Wiper lab test results

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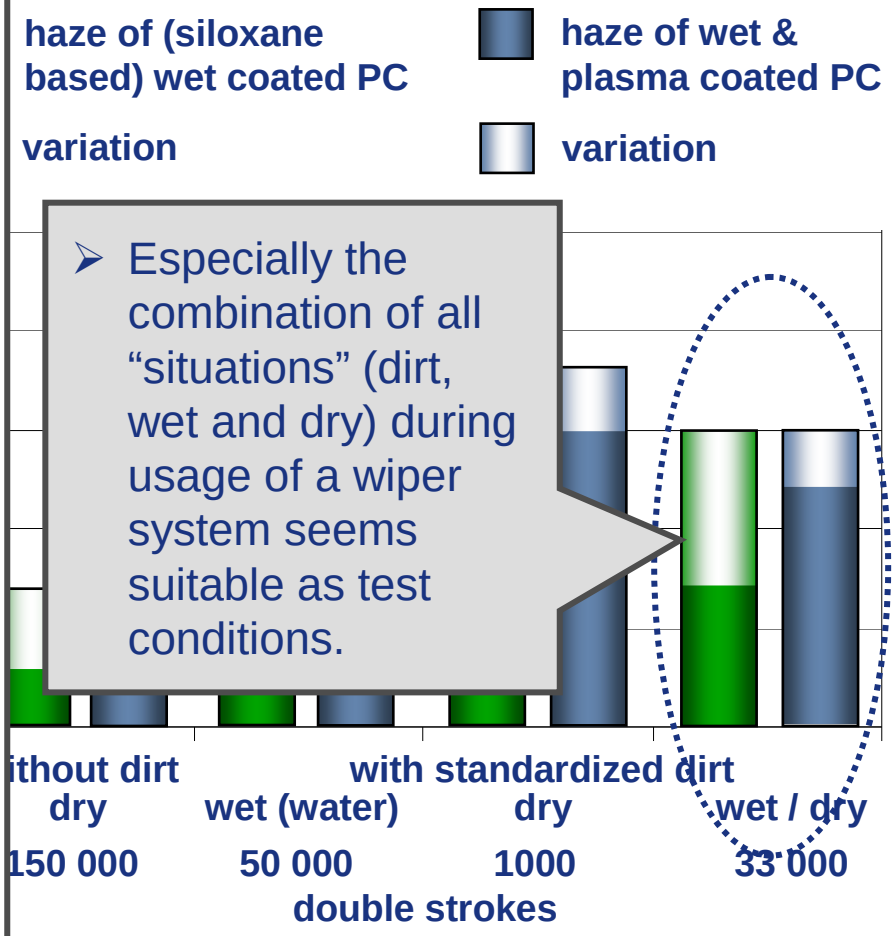
Test conditions:

- wipe speed 14cm/s
- load 20g/cm wiper blade
- wiper blade CR type (polychloro-prene)
- artificial dirt: mixture of salt and oxides in water according to ISO 6255 & ISO 12103 (Arizona test dust)
- test cycle: 2500 double strokes under wet condition, then removal of the water and 500 double strokes during drying up of the dirt
- test cycles is repeated 11 times
- cleaning of the samples via rinsing with water
- measuring haze



➢ Especially the combination of all "situations" (dirt, wet and dry) during usage of a wiper system seems suitable as test conditions.

Comparison of different glazing materials

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	Taber Abrasion Test	Amtec-Kistler Test	Laboratory wiper test	Laboratory wiper test
	CS 10F wheels (Gen IV); 1000 Cycles	10 double strokes; 1.5 g sand per 1l water;	3000 double strokes (2500 wet & 500 dry)) with dirt	33.000 double strokes wet & dry combination with dirt
	Δ haze [%]			
Laminated automotive glass	**1,2**	**0,3**	**0,1**	**0,3**
Siloxane based wet coats on PC	**2,6 (2 - 7)***	**0,8 (0,5 - 1,1)***	**0,2**	**0,6 (0,5 - 1,3)***
UV curable head lamp lense coatings on PC	**7,1 (5 - 7)***	**3,1 (2,2 - 4,3)***	**0,5**	**21**
Uncoated plastics	**30-40**	**10-12**	**9-13**	**--**

*** typical values usually obtained with this kind of coating**

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  - Characteristic polycarbonate bulk properties
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Characteristic polycarbonate bulk properties

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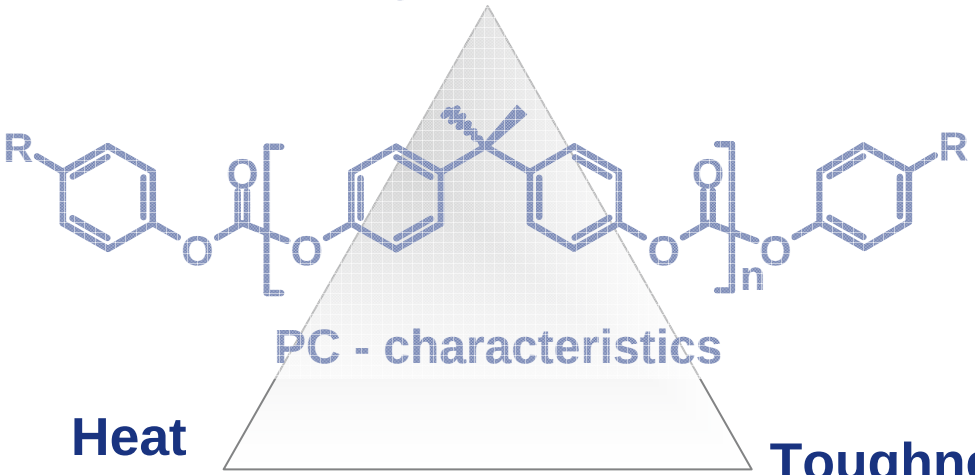
Bisphenol A – Polycarbonate provides an unique combination of

advantage vs. Glas:

- ✓ **weight**
- ✓ **toughness**
- ✓ **design freedom**

Transparency

Clear transparent, like glass
89% @ 2 mm thickness



advantage vs. PMMA:

- ✓ **toughness**
- ✓ **heat resistance**

Heat Resistance

Application range from
– 40℃ up to + 120℃

Toughness

Notched impact strength (acc. ISO 180-A)
60 … 90 kJ / m^2 @ 3.2 mm 23℃

Advantages of Polycarbonate Glazing

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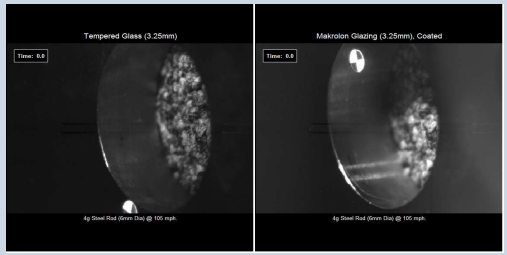
Weight savings up to 50 % vs. glass

- Lower center of gravity
- Reduction of CO_2-emission

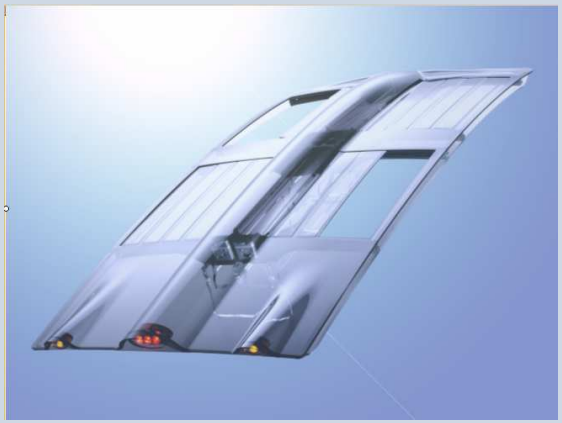
Safety & security

- Passenger retention
- Anti “smash-n-grab”



Styling and design

- Design freedom
- Functional integration



Weight savings potential

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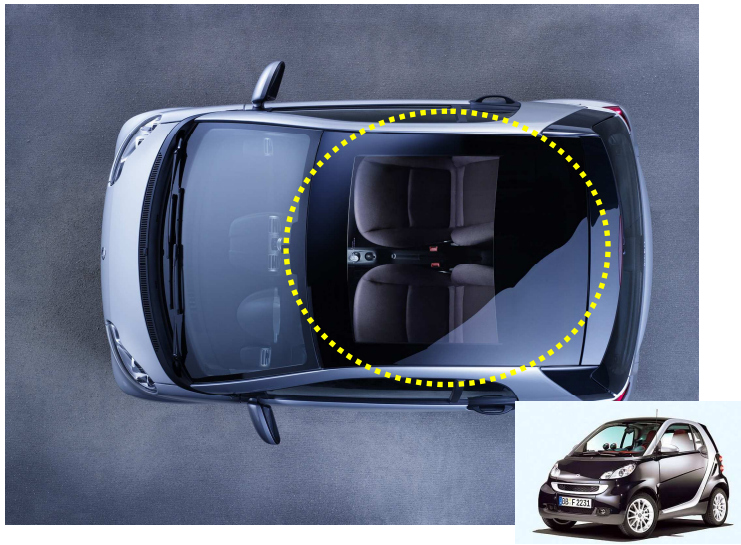
Weight savings potential:

– Density of polycarbonate 1,2 g/cm^3

– Density of glass 2,5 g/cm^3

➔ Theoretical weight savings: ~ 50 %

➔ Achievable in real applications: 30 – 50 %



Realised weight savings in recent application:

Smart fortwo fixed panoramic roof

- 1,2 m^2

➔ Weight savings: ~ 40 % (~ 5,5 kg)

Resource Efficiency

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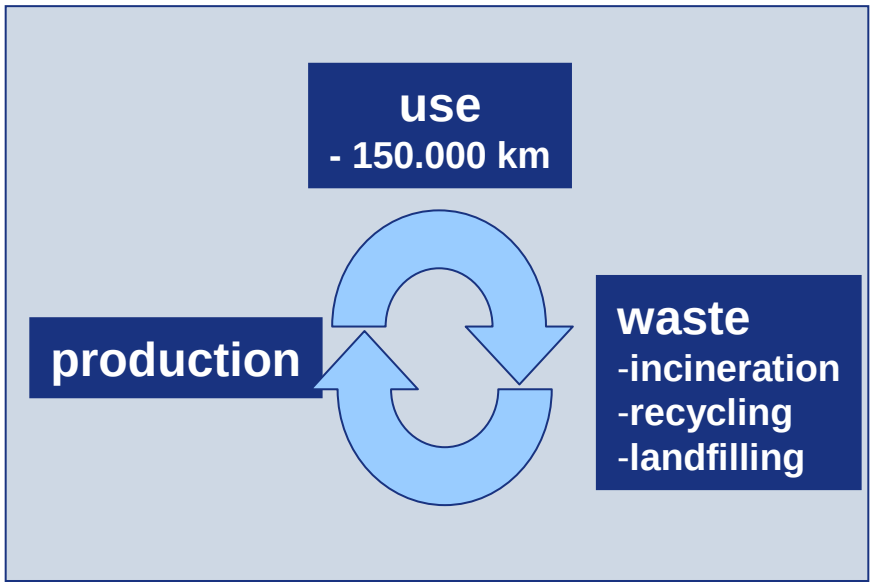
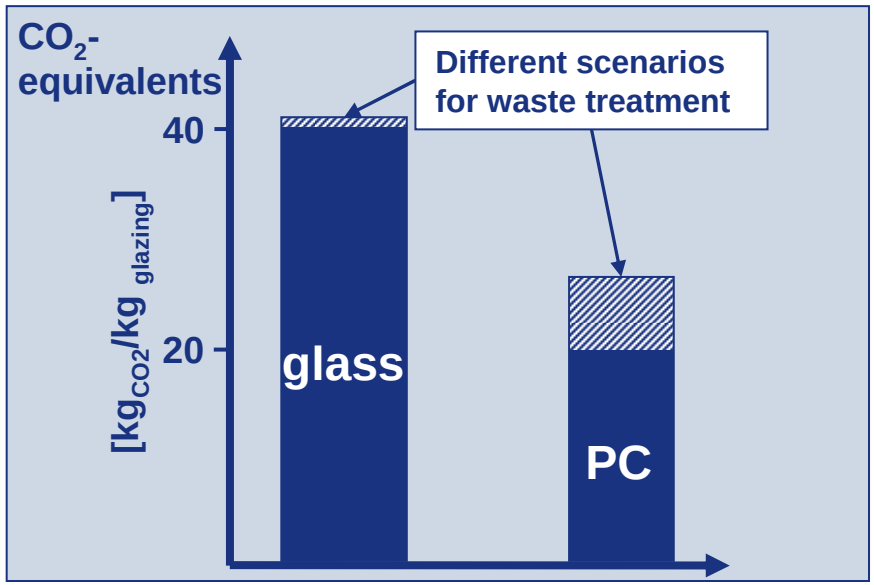
Life cycle analysis on automotive glazing

➔ Polycarbonate vs conventional glass





➔ **1 kg PC saves ~ 14 – 22 kg CO_2-emission along total life cycle, compared to glass.**

Source: GUA GmbH, Corporation for Comprehensive Analyses, study commissioned by Bayer MaterialScience

Data sheet AG 2677

BayVision

Bayer MaterialScience

Makrolon AG2677

Grades for / Automotive glazing	Global grade; MVR (300 °C/1.2 kg) 12.5 cm³/10 min; Medium viscosity; UV stabilized; Easy release; Injection molding - Melt temperature 280 - 320 °C; Available in transparent colors only; Automotive glazing; Roof modules
ISO Shortname	ISO 7391-PC,MLR,(,,)-18-9

	Property	Test Condition	Unit	Standard	Value
	Rheological properties				-
C	Melt volume-flow rate	300 °C; 1.2 kg	cm³/10 min	ISO 1133	12.5
C	Molding shrinkage, parallel	60x60x2; 500 bar	%	ISO 294-4	0.65
C	Molding shrinkage, normal	60x60x2; 500 bar	%	ISO 294-4	0.7
	Molding shrinkage, parallel/normal	Value range based on general practical experience	%	b.o. ISO 2577	0.6 - 0.8
	Melt mass-flow rate	300 °C; 1.2 kg	g/10 min	ISO 1133	13
	Mechanical properties (23 °C/50 % r. h.)				
C	Tensile modulus	1 mm/min	MPa	ISO 527-1,-2	2400
C	Yield stress	50 mm/min	MPa	ISO 527-1,-2	67
C	Yield strain	50 mm/min	%	ISO 527-1,-2	6.1
C	Nominal strain at break	50 mm/min	%	ISO 527-1,-2	> 50
	Stress at break	50 mm/min	MPa	ISO 527-1,-2	70
	Strain at break	50 mm/min	%	b.o. ISO 527-1,-2	120
C	Tensile creep modulus	1 h	MPa	ISO 899-1	2200
C	Tensile creep modulus	1000 h	MPa	ISO 899-1	1900
	Flexural modulus	2 mm/min	MPa	ISO 178	2400

Makrolon_AG2677_ISO.pdf

ECE R43 weathering requirements

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- **TEST RESISTANCE TO THE ENVIRONMENT**

  - Resistance to simulated weathering according to ECE R43 Annex 14 Item 6

  …The total ultraviolet radiant exposure with the long arc xenon lamp shall be 500 MJ/m2. During irradiation the test pieces shall be exposed to water spray in continuous cycles. During a cycle of 120 minutes the test pieces are exposed to light without water spray for 102 minutes, and to light with water spray for 18 minutes.

  …The resistance to the simulated weathering shall be considered to have given a satisfactory result if: The light transmittance measured in accordance with annex 3, paragraph 9.1.does not fall below 95% of the pre-weathering value. Additionally, for windows which are required for driver visibility the value shall not fall below 70%. No bubbles or other visible decompositions, discolorations, milkiness or crazing shall occur during weathering.

☑ Makrolon AG2677 coated on both sides with polysiloxane lacquers

Weathering performance of coated polycarbonate

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In contrast to the abrasion resistance where the performance of a protective coating class - defined by the binder material (e.g. siloxane based coating, etc.) - for polycarbonate is all about the same, the weathering performance can vary a lot within the same coating class.

The weathering performance of polycarbonate depends to a large extend on the ability of the coating to filter out the harmful UV radiation. So the combination of the filter effect and it persistency is the key to a longterm weather resistance.

In order to test this long term performance it is necessary to use accelerated weathering methods which allow a to predict the outdoor weathering result with high accuracy.

- **Xenon WOM 0,75** according to ASTM G155 – an accelerated weathering method used by BMS for coated polycarbonate samples
- **Correlation** check of the accelerated weathering Xenon WOM 0,75 with **outdoor weathering**
- **Weathering result** of different coating systems on polycarbonate

Coated PC performance - accelerated weathering

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Xenon WOM 0.75 based on **ASTM G155**

Irradiation W/m^{2}nm	0.75
Light (min)	102
Light/spray (min)	18
Black panel temp. (℃)	70
Inner & outer filter	boro / boro
Temp. in test (℃)	55

- good **correlation** to outdoor weathering (time to failure, failure mode, YI and haze until failure and UV degradation rate)
- high **acceleration factor** (8 times compared to Florida outdoor weathering; 1a FL ≡ 1100h Xenon WOM 0.75 based on radiation energy)

➢ time efficient **comparison** of different coated PC samples under standardized conditions possible

Coated PC performance – predicted outdoor weatherability

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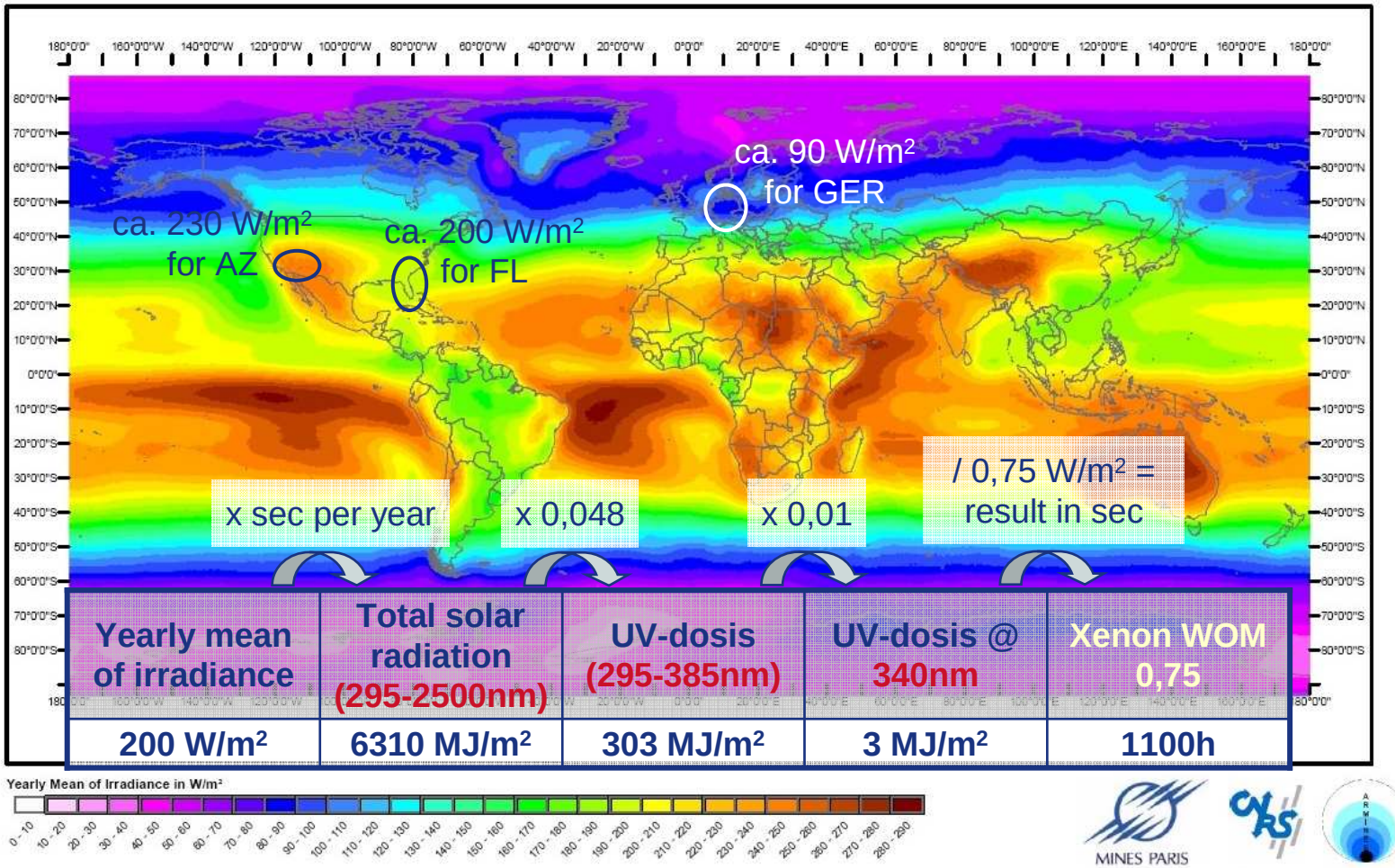

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Coated PC performance – predicted outdoor weatherability

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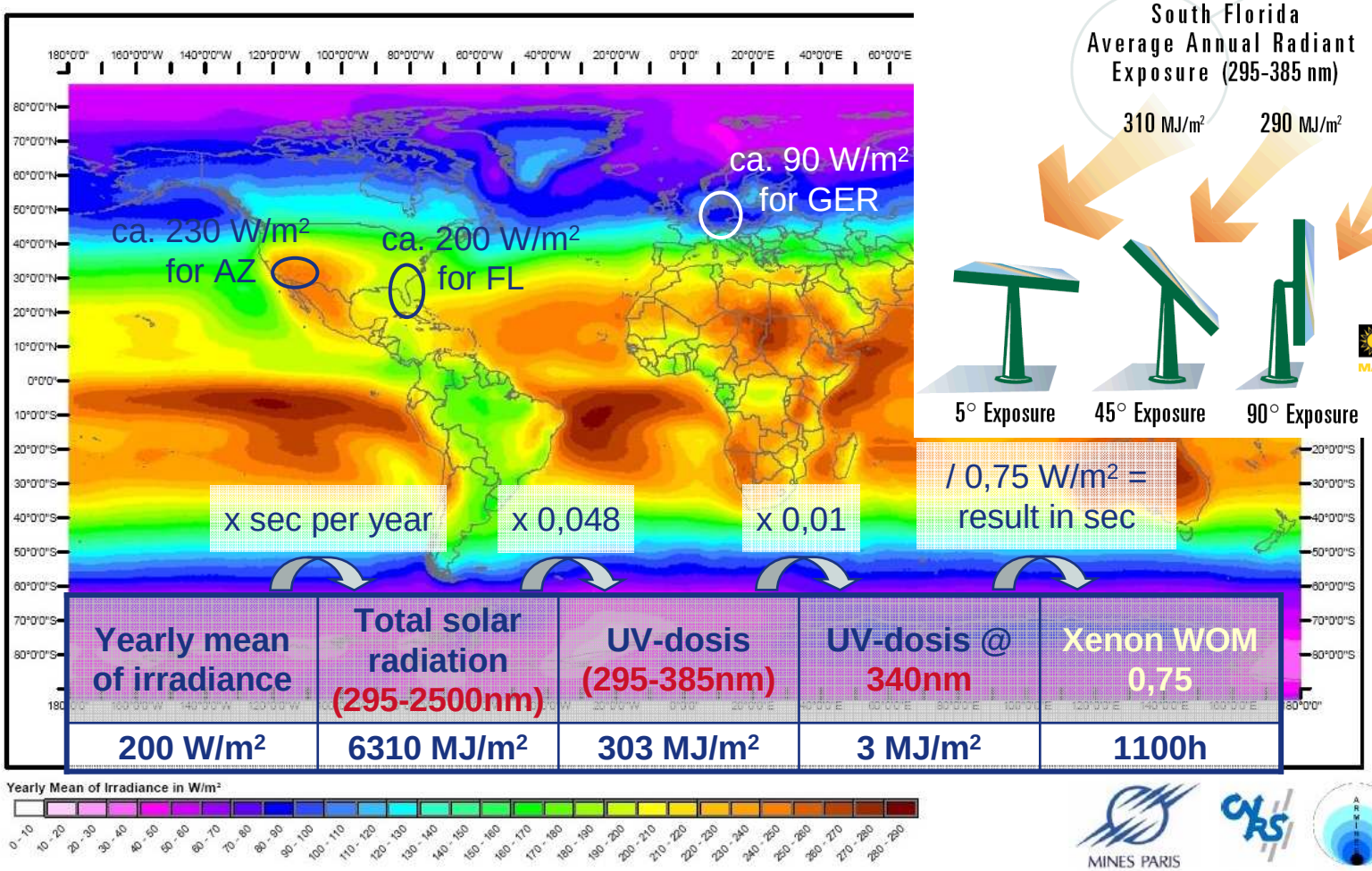

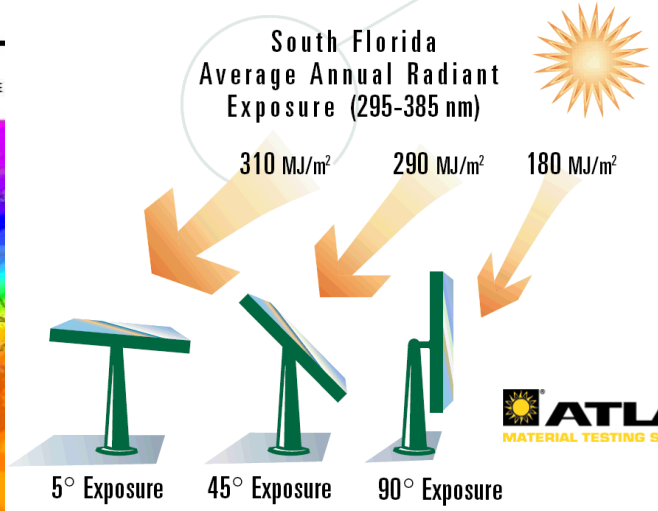

ATLAS MATERIAL TESTING SOLUTIONS

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Why Xenon WOM 0.75 ?

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The used accelerated weathering method is based on **ASTM G155** (Standard Practice for Operating Xenon Arc Light Apparatus for Exposure of Non-Metallic Materials) and called **Xenon WOM 0.75** (formerly

Why using a Xenon arc as light source?

Why using an irradiation of 0.75 W/m²nm at 340nm?

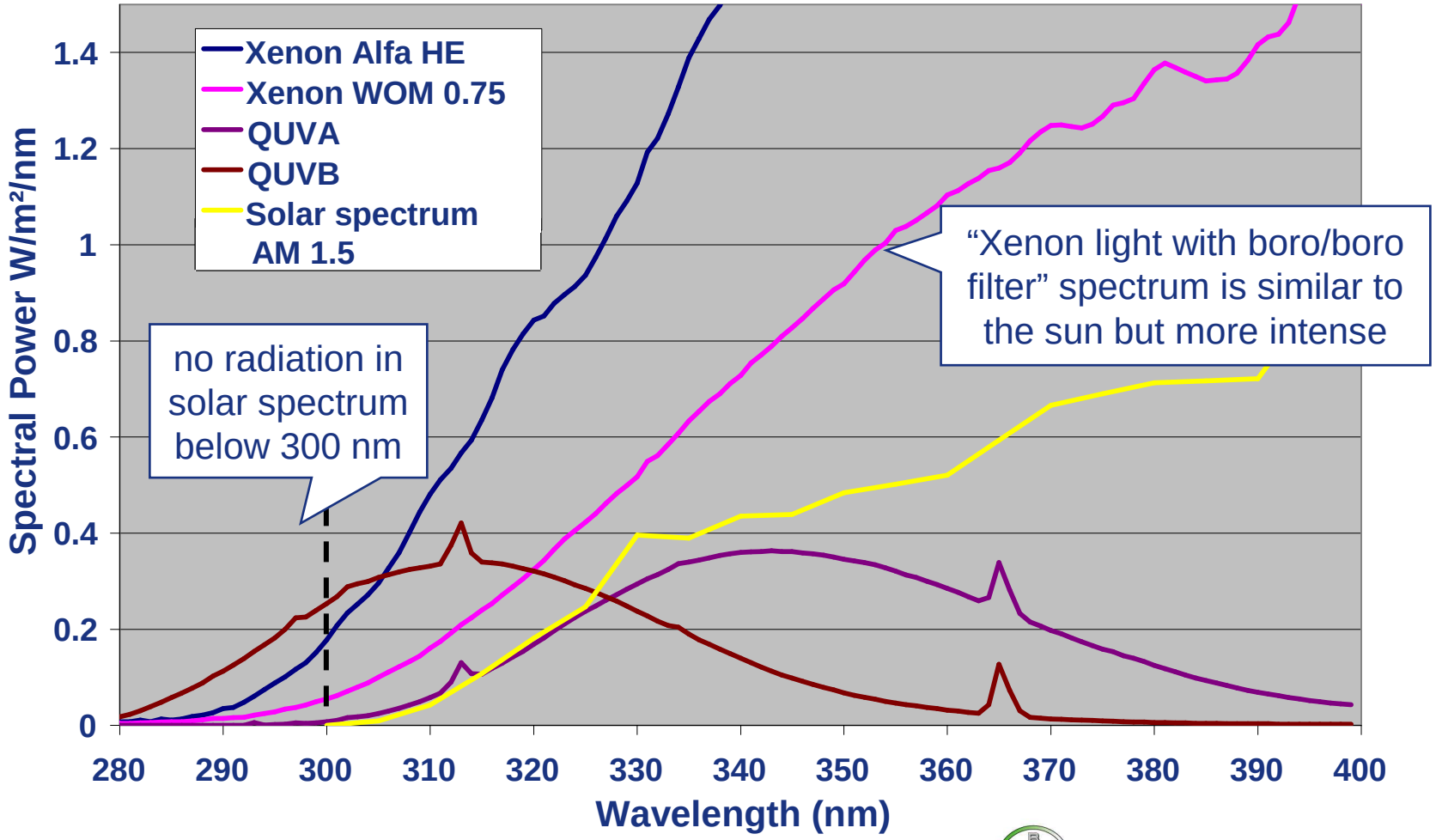
	Xenon
Instrument	[illegible]
Irradi[illegible] W/m²	[illegible]
Light (min)	[illegible]
Li[illegible]	**18**
Bl[illegible] temp. (℃)	**70**
Inner & outer filter	**boro / boro**
Temp. in test (℃)	**55**

Correlation [illegible] radiation

3 MJ/m² @ 340nm is the radiation energy in 1 year FL & the same amount is reached in 1100h Xenon WOM 0.75 so

3 MJ/m2 @ 340nm ≡ **1a FL**
≡ **1100h** Xenon WOM 0.75

Spectra comparison for different light sources



Advantage compared to other accelerated weathering methods

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- Radiation below 300 nm in artificial weathering destroys the coated polycarbonate in a way that will not occur in real life since the solar spectrum has no radiation below 300nm
  - ⇨ QUVB has to much radiation below 300 nm to be considered a realistic test for coated polycarbonate
- The Xenon Alfa HE test has such a high radiation dose, that nonlinear effects take place; i.e. no correlation to real life could be found
- The QUVA is a test with good relevance to outdoor weathering, but the radiation intensity is too low; i.e. weathering tests take too long
- Xenon light with boro / boro filter as the Xenon WOM 0.75 has a spectrum similar to the solar spectrum but more intense
  - ⇨ ideal combination to achieve a high acceleration factor while still having a good correlation to outdoor weathering for coated polycarbonate

Comparison of different Xenon WOM weathering tests

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BMS preferred method

Xenon WOM according to SAE J1960		Xenon WOM 0.75 according to ASTM G155
a.) 40min light b.) 20min light + rain c.) 60min light d.) *60min dark* + rain	Weathering cycle	a.) 108min light b.) 12min light + rain
0,55	Irradiation W/m²nm @340nm	*0,75*
2250h	Equivalent to 1 year outdoor weathering in FL based on radiation energy	1100h
quartz / boro	Inner & outer filter system to adjust bulb spectrum to sunlight	boro / boro
	Correlation to outdoor weathering	proven**

bad fit to spectral power of sun light * (bad outdoor correlation)

Higher acceleration factor (shorter testing time until 1st failure)

* significant amount of for PC harmful radiation (<300nm) which is not present in sun light

** via comparison of 1st failure mode & time, YI & haze values, UV absorber degradation rate

Spectra comparison for different filter systems

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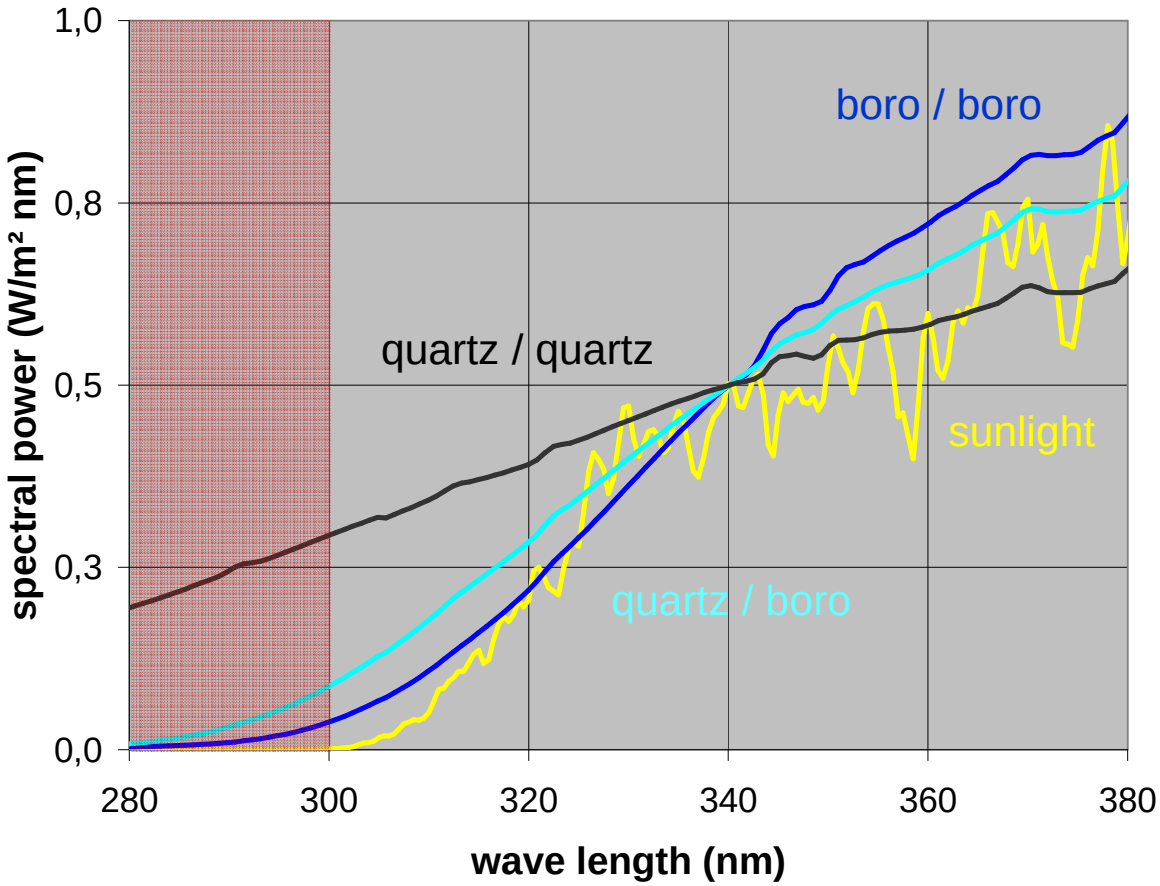

sun light according to ASTM G173-2

Xenon Ci65 boro/boro 0,5W/m2 (DIN ISO)
most common combination for weathering tests (daylight filter system)

Xenon Ci65 quartz/boro 0,5W/m2 (SAE J1960)
weathering tests with somewhat more & shorter UV than sunlight

Xenon Ci65 quartz/quartz 0,5W/m2
testing with considerably more shorter (unrealistic) UV than global solar radiation

Comparison of Xenon WOM 0.75 and outdoor weathering in Florida

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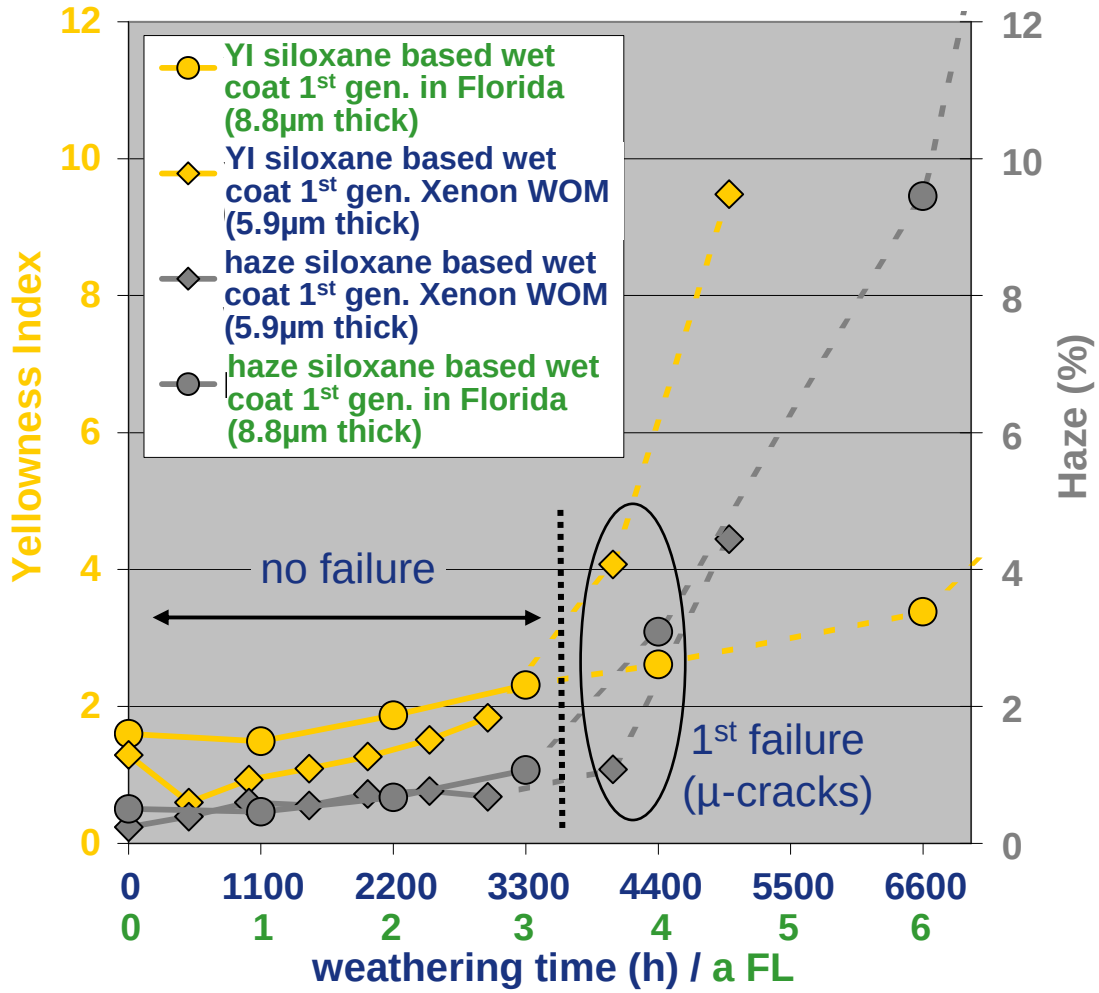

3 MJ/m2 @ 340nm ≡ **1a FL ≡ 1100h** Xenon WOM 0.75

- ⇨ time to failure and failure mode well predicted by Xenon WOM 0.75
- ⇨ correlation of YI and haze is good till the protection layer is damaged

Comparison of UVAD

in Xenon WOM 0.75 and “real” glazing part weathering

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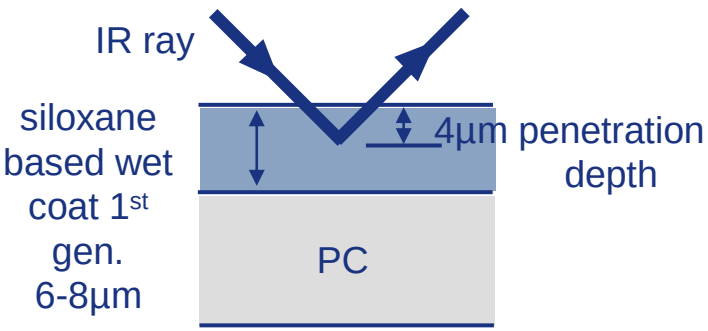
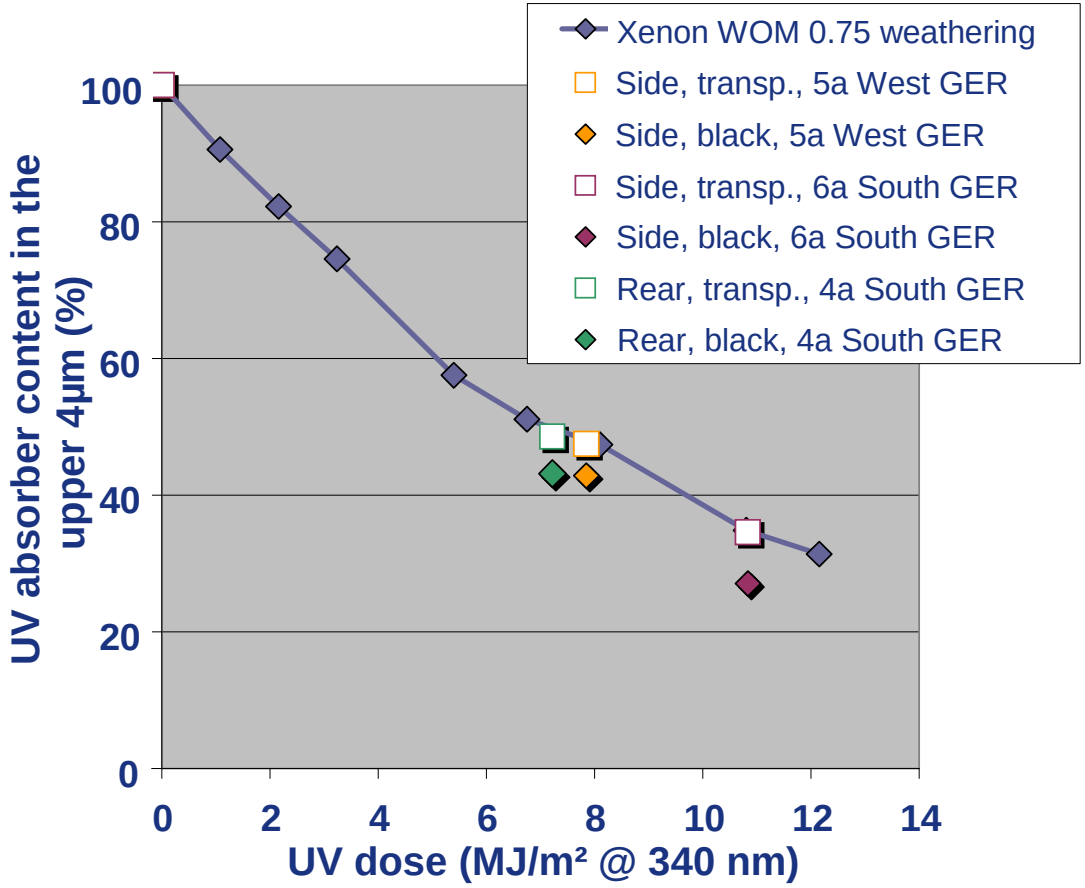
UV absorber degradation (UVAD) measurement in the upper 4µm of the coating using surface IR spectroscopy



⇨ good correlation between real glazing parts and accelerated weathering regarding the UV absorber degradation



Summary Accelerated Weathering Method

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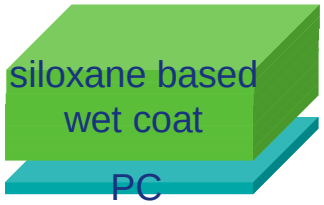
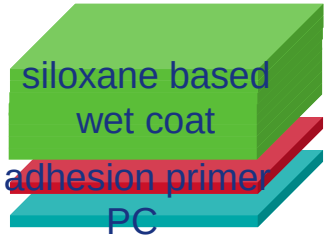
A good correlation exists between Xenon WOM 0.75 (former G26mod) and outdoor as well as "real" glazing part weathering

- The comparison of outdoor and accelerated weathering using siloxane based wet coats 1st gen. on Makrolon® shows that
  - time to failure and failure mode is well predicted in only one eighth of the time
- The UVAD measurement of siloxane based wet coat 1st gen. on Makrolon® used as glazing parts compared with sample plaques after accelerated weathering using surface-IR-spectroscopy shows that
  - acceleration does not induce a markedly different UV absorber degradation and therefore allows a reliable prediction

Coating Systems

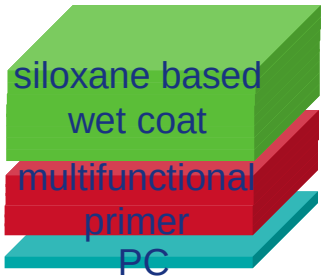




siloxane based wet coat 1st generation (e.g. head lamp lenses)

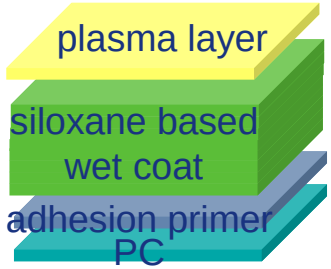
primerless hard coat with one curing step

two layer system (adhesion primer & hard coat) with ether one ("wet on wet") or two curing steps



siloxane based wet coat 2nd generation

two layer system (primer with UV protection & hard coat) with two curing steps ("bake on bake")

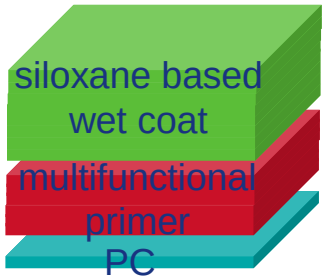


siloxane based wet coat & plasma

three layer system (adhesion primer, hard coat & plasma layer)

New

market introduction phase



siloxane based wet coat 3rd generation

two layer system (primer with **improved** UV protection & hard coat)

Comparison of coating systems
(Xenon WOM 0.75 & transparent samples)

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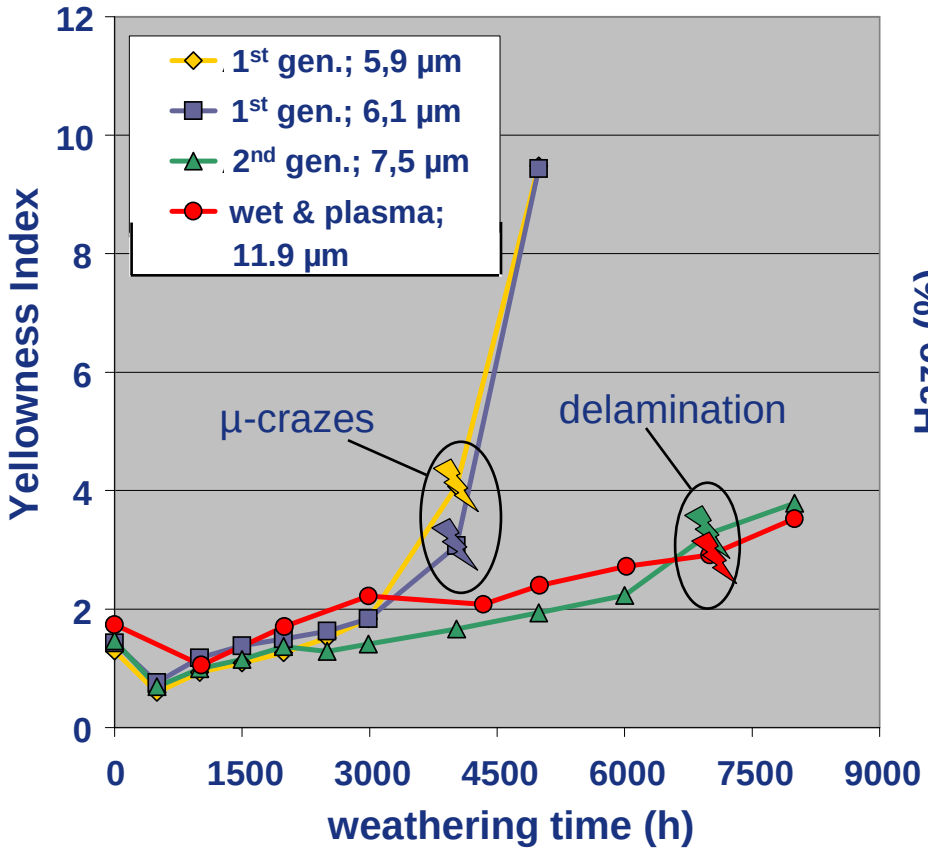
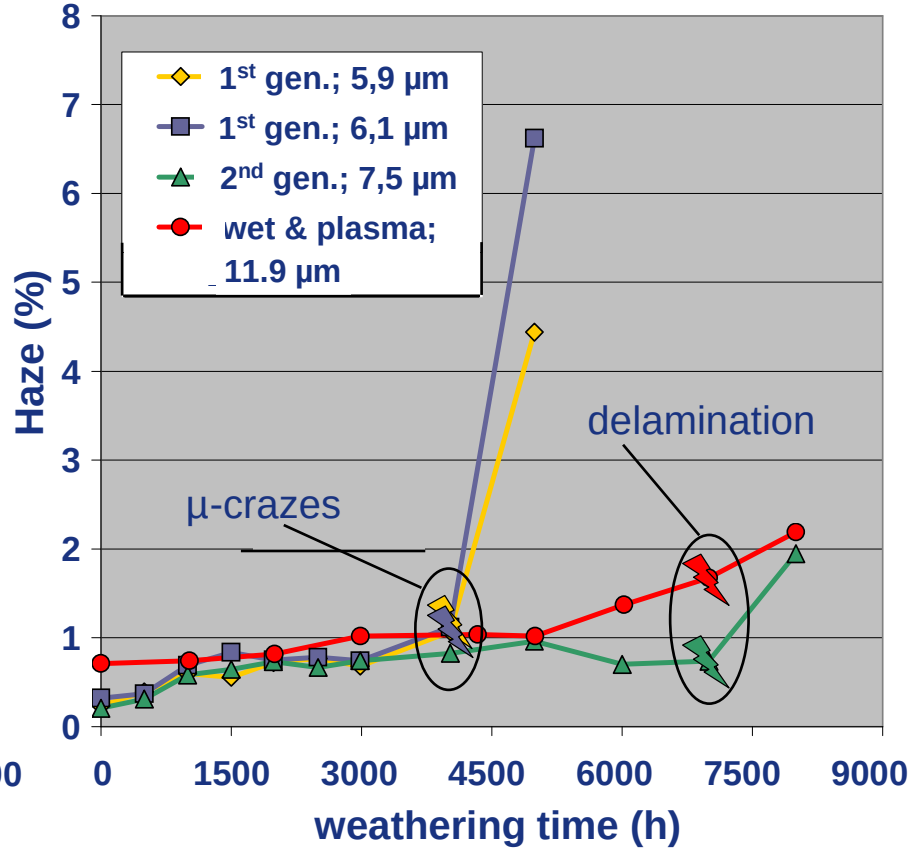
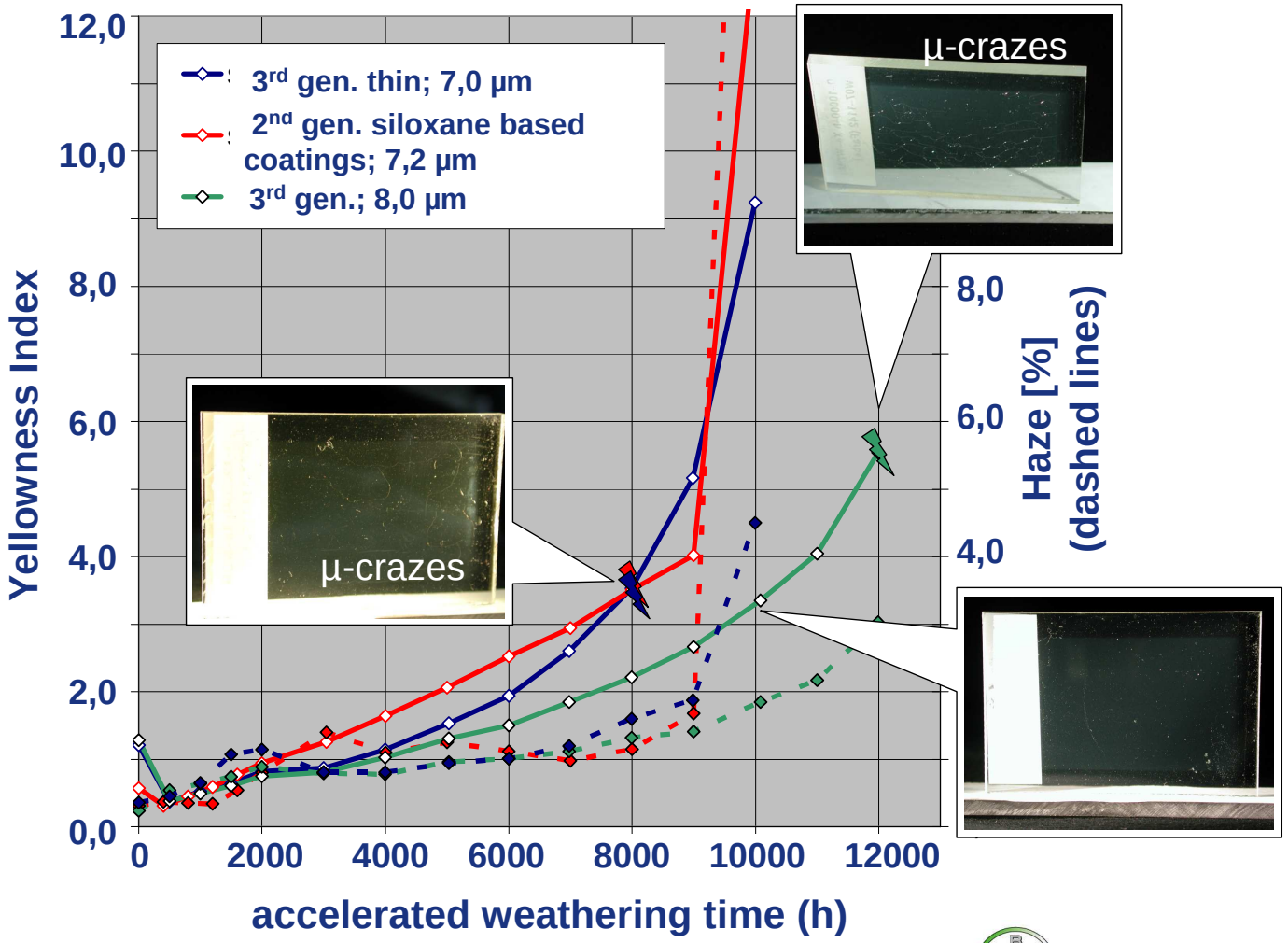
Siloxane based wet coats on AG2677 and wet coat & plasma on clear PC





Comparison of weathering data

2nd vs. 3rd generation on clear Makrolon



Summary of the weathering results

- The weathering performance for different locations can be predicted based on an accelerated (artificial) weathering method and depends mainly on the average irradiation in W/m^2 for this location.
- It is difficult to give a general statements regarding life time (e.g. in hours accelerated weathering time) due to further influencing factors (coating system used, thickness of the coating system and the polycarbonate substrate used (especially the color)).
- The weathering performance of a coating system on black tinted polycarbonate is reduced compared to clear transparent substrates.
- The first visible weathering failure (e.g. µ-crazes) does not mean that it is necessary to replace the glazing similar to a glass based glazing where the first failure is also only a small visible damage probably due to stone-chipping.
- The first weathering failure for a coated polycarbonate glazing part is an optical issue and not a safety issue.

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Thank You For Your Attention

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