CHARACTERIZATION TOOLS AND ACCELERATED AGING

SolarTwins 2nd Summer School – Next Generation CST Technologies

Standardized reflector testing and advanced optical characterization tools

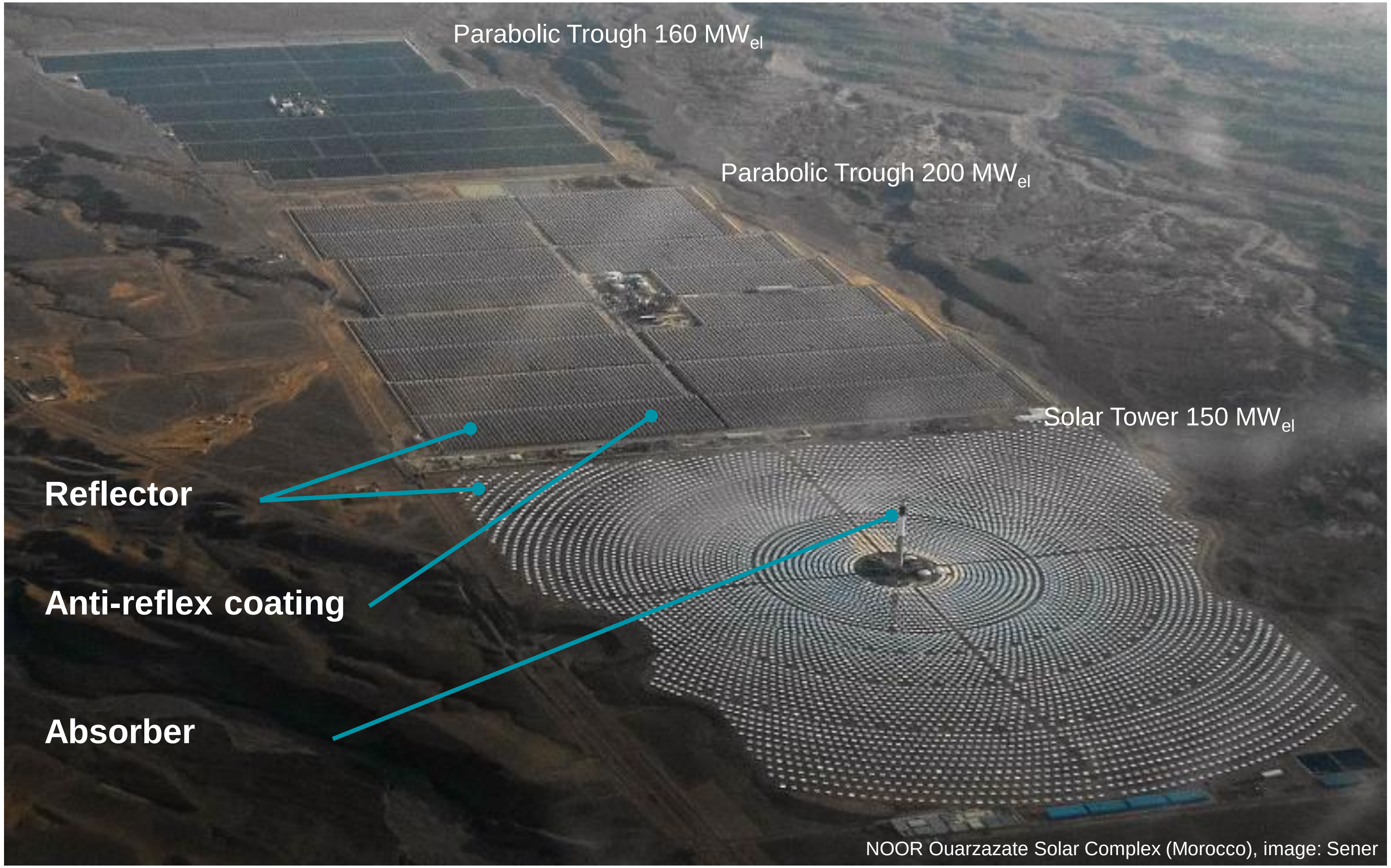
Dr. Florian Wiesinger, Institute of Solar Research, 2. September 2022

- Optical components in CSP
- Quality assessment and instrumentation
- Aging effects and simulation
- Novel characterization tools

OVERVIEW

PSA: owned by CIEMAT [1]

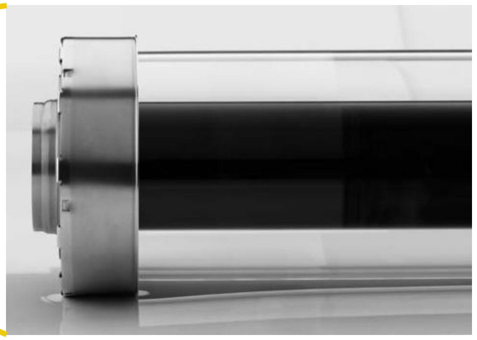
Optical components - overview



Optical components - overview

Parabolic trough collector

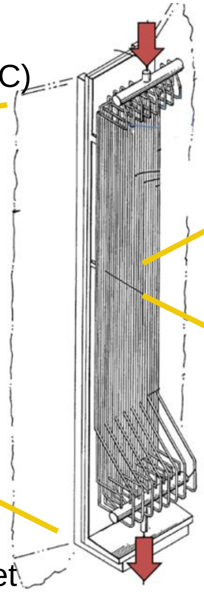
Sputtered absorber layer
α=96%, ε=7.3% (400ºC)



3mm Borosilikat-glass
with anti-reflexcoating
(τ_s= 96.8%)

Solar tower field with central receiver

Solar Salt
inlet (290ºC)



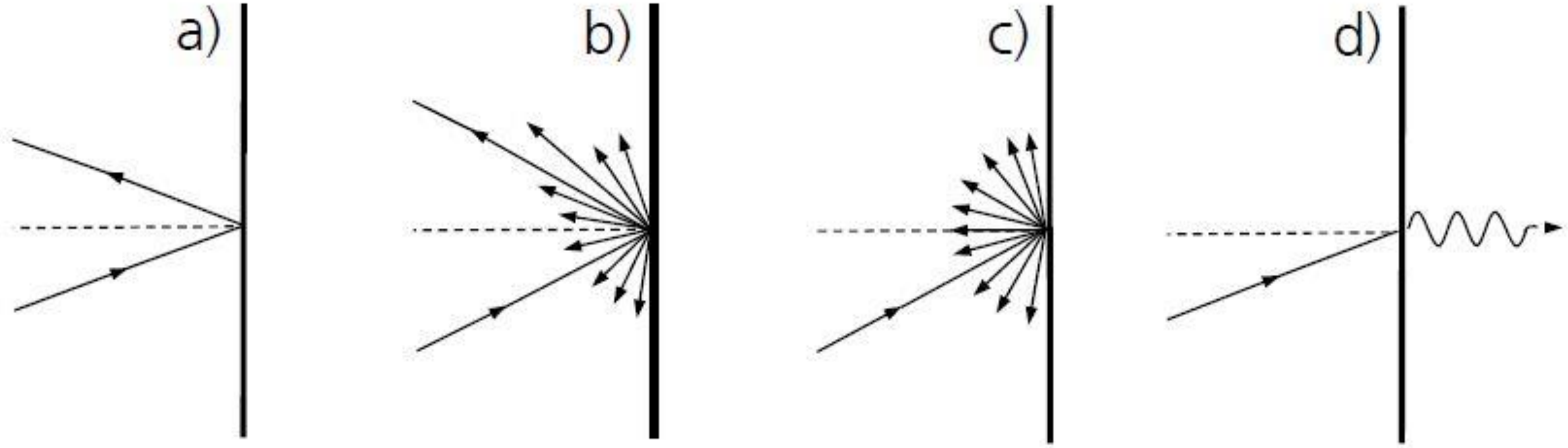
Solar Salt oulet
(565ºC)

Pyromark 2500
α_s = 97 %
$\varepsilon_{750ºC}$= 82%
$\eta_{250kW/m^2,750ºC}$= 77%
$\Delta\alpha_s$ = ~2 %-p/a
T_{skin} ≈ 750ºC

Second surface
silvered-glass
reflectors
$\rho_{15°,660nm,12,5mrad}$=95%

[2]

Optical components – reflectors – theoretical background

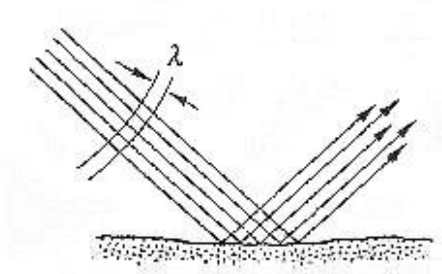


pure specular

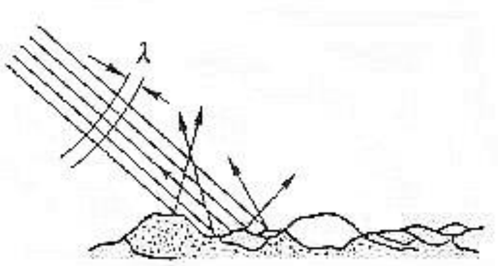
general reflectance,
mixture from a) and c)

pure diffuse

absorptance/
transmittance



highly specular surface



diffuse surface

ρ = reflectance
τ = transmittance
α = absorptance

Optical components – reflectors – theoretical background

Examples for **diffuse** reflecting surfaces (room temperature)

High ρ [%]:

- Titaniumdioxid 99
- Magnesiumoxid 96
  (vapor deposited)
- Gypsum 80
- White Paper 70

Low ρ [%]:

- Black platinum 0.1
- Carbon black 0.8
- Black varnish 1-1.5
- Black paper 5

Examples for **specular** reflecting surfaces

High ρ [%]:

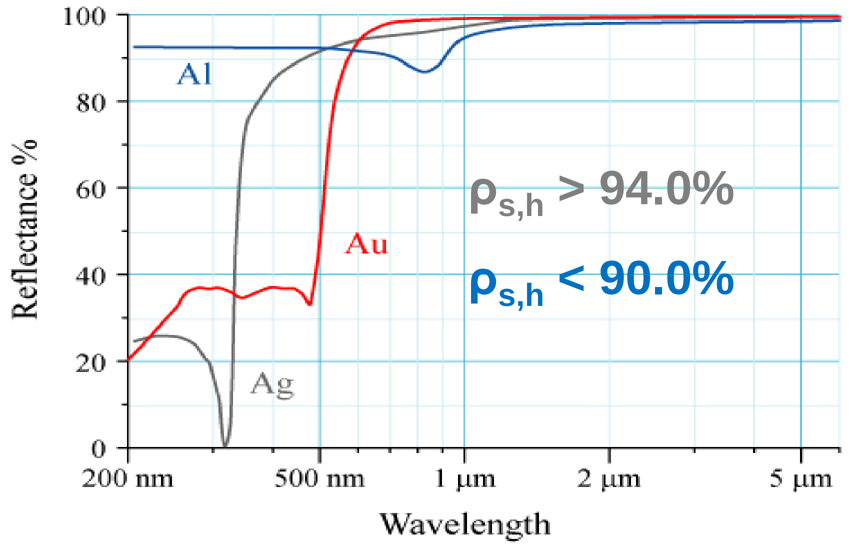
- Aluminum (polished) 87-92
- Silver (polished) 98-80
  (λ-range 0.37-1µm)

- Steel(polished) 93
- Stainless Steel 89

→ ρ is highly depending on λ

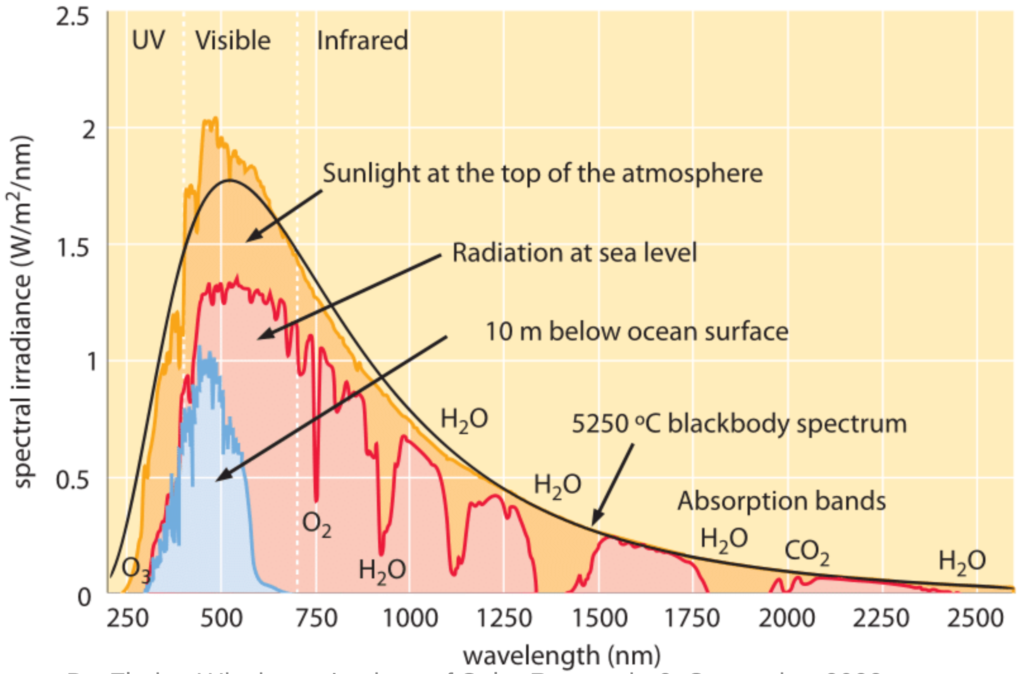
Handbook of Chemistry and Physics, 75th Edition
Manufacturer datasheet: Electro Optical Industries Inc.

Optical components – reflectors – theoretical background

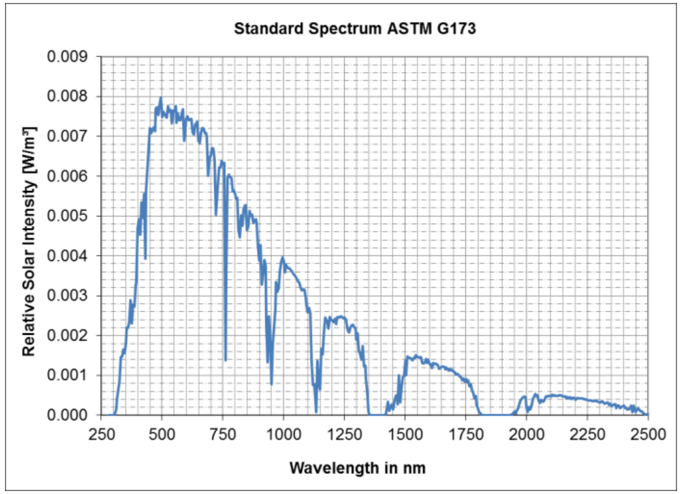


A suitable “mean value” of all relevant solar wavelengths is the solar weighted reflectance

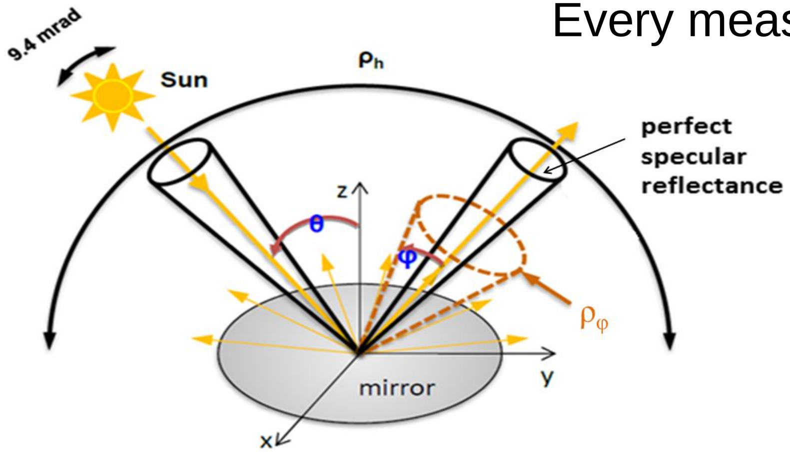
$$\rho_{s,\varphi}([\lambda_a,\lambda_b],\theta_i,\varphi,T_s)=\frac{\sum_{i=0}^{i_{\max}}\rho_{s,\varphi}(\lambda_i,\theta_i,\varphi,T_s)\cdot G_b(\lambda_i)}{\sum_{i=0}^{i_{\max}}G_b(\lambda_i)}$$



The spectral solar irradiance $G_b(\lambda_i)$ can be obtained in 5 nm steps from a reference spectrum, e.g. ASTM G173 with air mass 1.5 and 1000 W/m²



Optical components – reflectors – theoretical background



Every measured reflectance value needs to be declared in the format:

$$\boxed{\rho_{\lambda,\varphi}(\lambda,\ \theta_i,\ \varphi,\ T_s)}$$

λ	wavelength	[nm]
θ_i	incidence angle	[º]
φ	acceptance angle	[mrad]
T_s	surface temperature of the mirror	[ºC]

To indicate solar weighted values use “s“ as index and indicate the wavelength range of the weighting instead of λ

To indicate hemispherical reflectance use “h” instead of φ

Examples: $\rho_{\lambda,\varphi}(660\text{ nm},\ 15º,\ 12.5\text{ mrad},\ 25ºC) = 95.3\%$

$\rho_{s,h}([280,2500nm],\ 8º,\ h,\ 25ºC) = 94.1\%$

[3]

Optical components – reflectors – characterization

Perkin Elmer Lambda 1050 spectrophotometer

Measures hemispherical reflectance, transmittance & absorptance

Multiple Wavelength Portable Specular Reflectometer, Model 15R-RGB

Measures specular reflectance

Optical components – reflectors – characterization

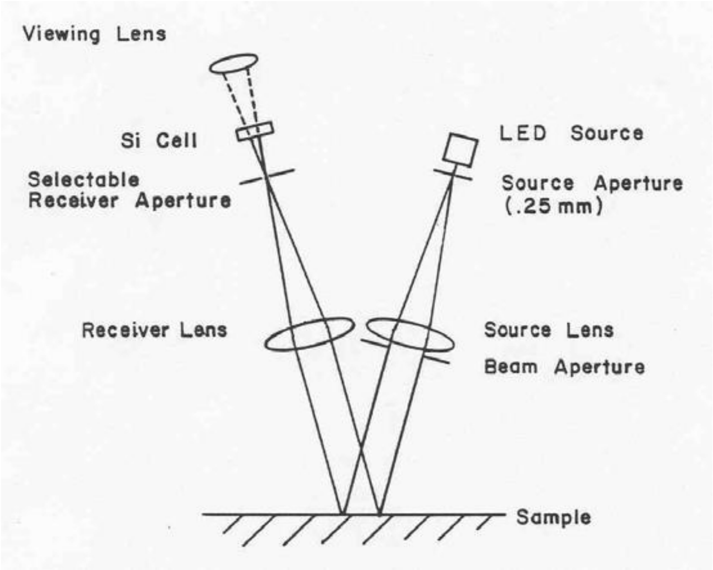
Spectral measurement of specular reflectance is not possible at the state of the art!!

Only monochromatic measurements are possible

Best instrument on the market: D&S reflectometer

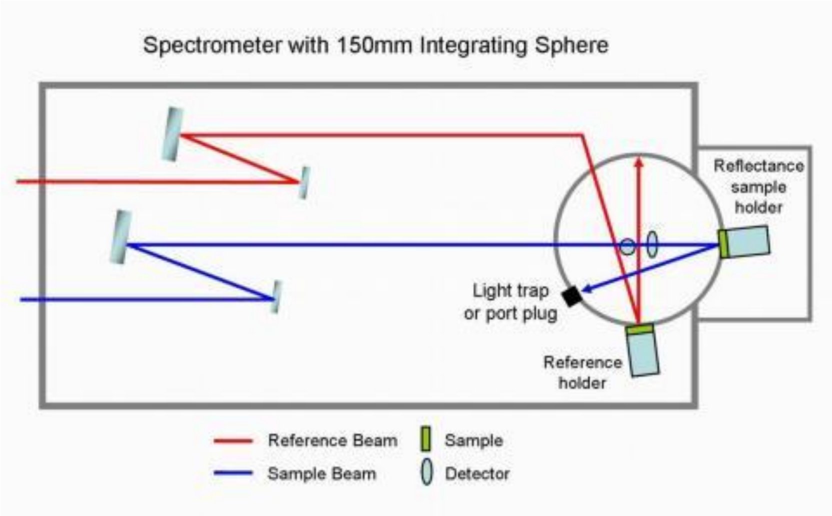
Devices & Services – Multiple Wavelength Portable Specular Reflectometer Model 15R-RGB

- Acceptance angles φ= 2.3, 3.5, 7.5, **12.5**, 23 mrad
- Wavelength of light source: λ= 460, 550, **650**, 720nm
- Incidence angle θ =15°
- Repeatability: +/- 0.002 reflectance units for glass mirrors
- Lower repeatability for flexible mirror samples : +/- 0.004
- Expected uncertainty including reference mirror: +/- 0.007



Optical components – reflectors – characterization

- Measures hemispherical reflectance, transmittance or absorptance
- Wavelength of light source: λ= 250 - 2500 nm
- Incidence angle θ =8°
- High repeatability (<0.2%)



Disadvantages:
• Max. measurement spot 9x17mm²
• No specular measurements

Optical components – reflectors – state of the art

Requirements:

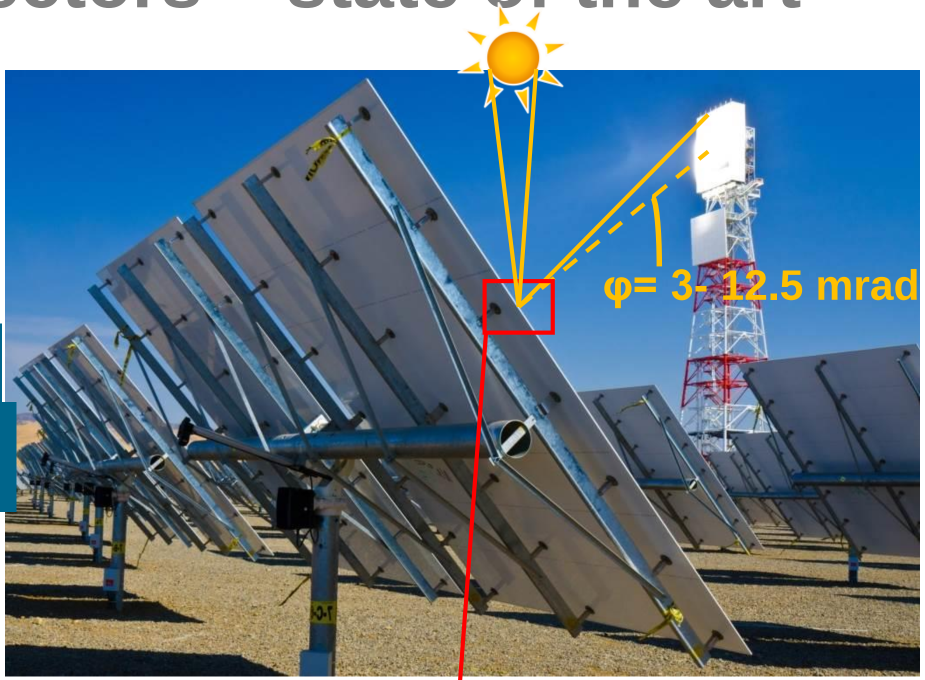
- Low scattering
- High reflectance
- Low cost
- Low degradation rate

≤0.4%-p at φ= 12.5 mrad

$\rho_{s,h}$ = 94.5%

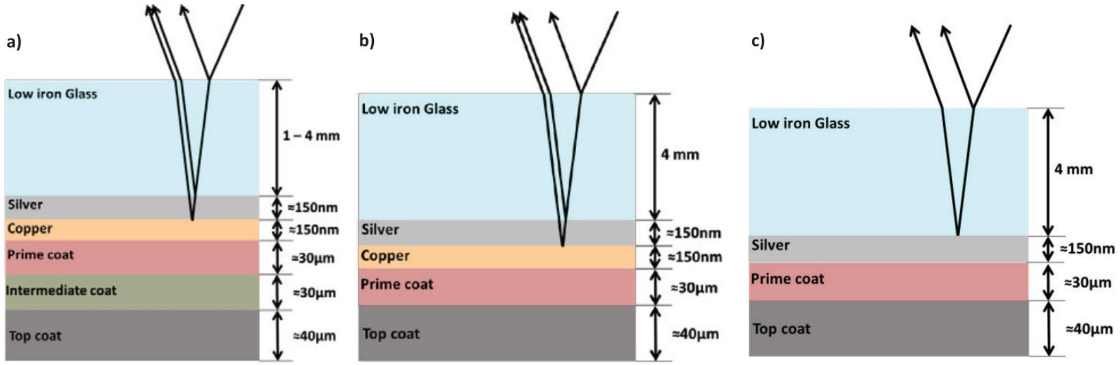
~12€/m² (2005: ~50€/m²)

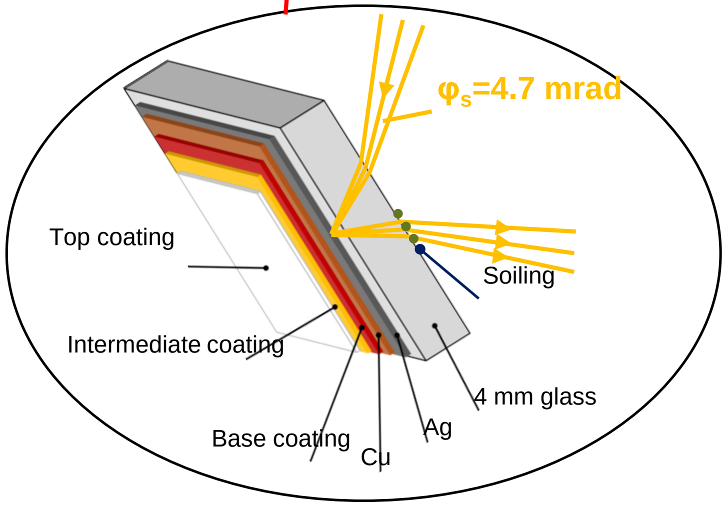
1% reflectance degradation ≈ annual loss of 0.5 Mio.€



Current research activities

- 2-layer protective lacquers to reduce cost





Optical components – reflectors – state of the art

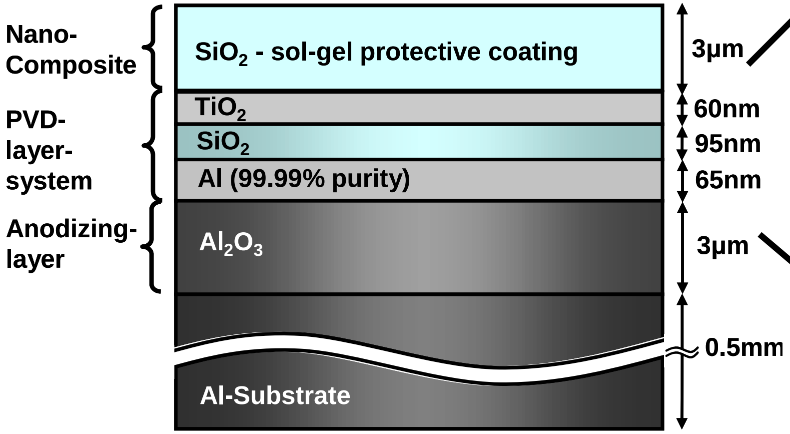
Mirror types – PVD coated aluminum reflectors

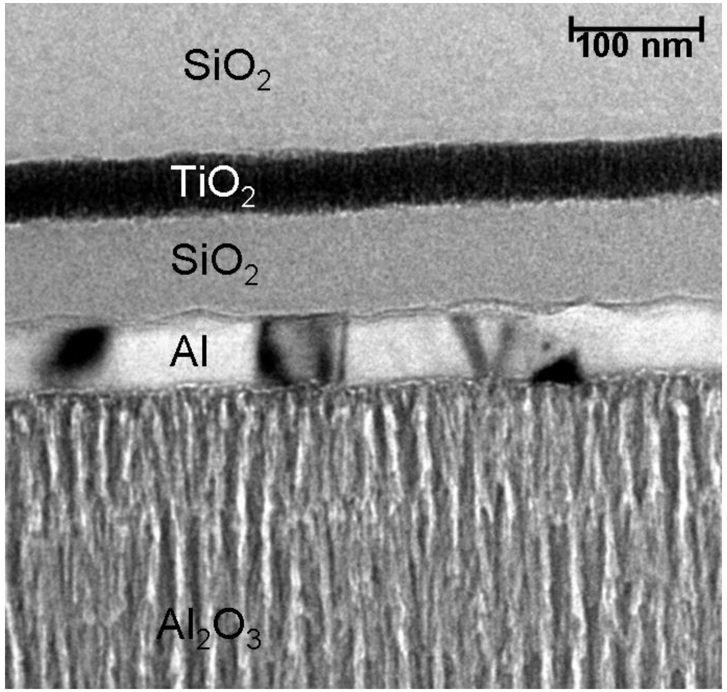
Typical reflectance values:

$\rho_{s,h}([280,2500nm], 8º, h, 25ºC) = 90.0\%$

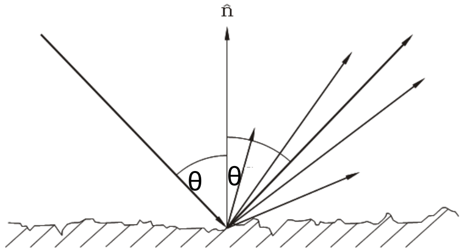
$\rho_{\lambda,\varphi}(660\ nm, 15º, 12.5\ mrad, 25ºC) = 85.5\%$

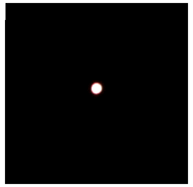
+ cost
+ flexible
- durability
- reflectance

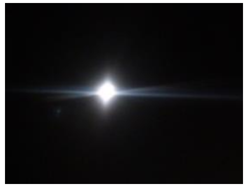


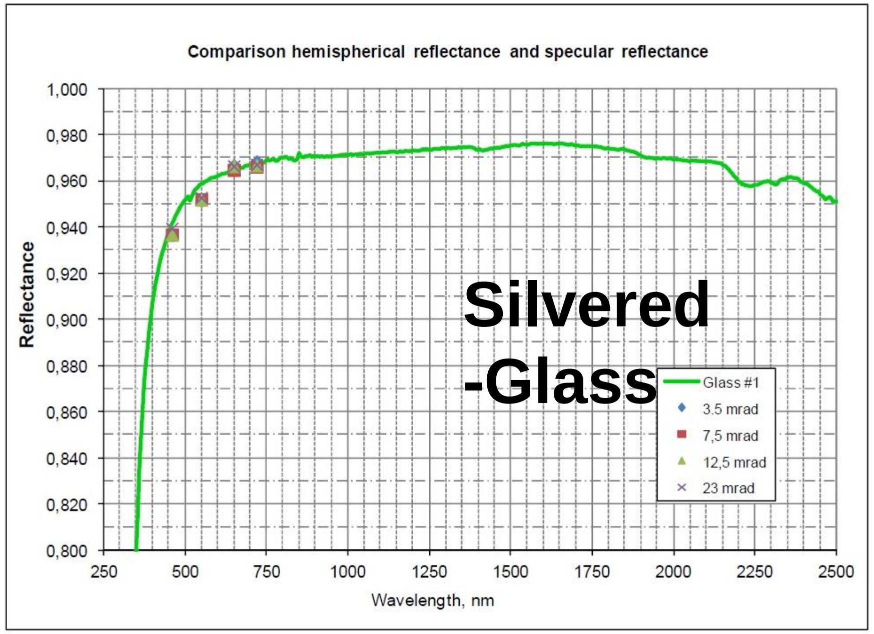


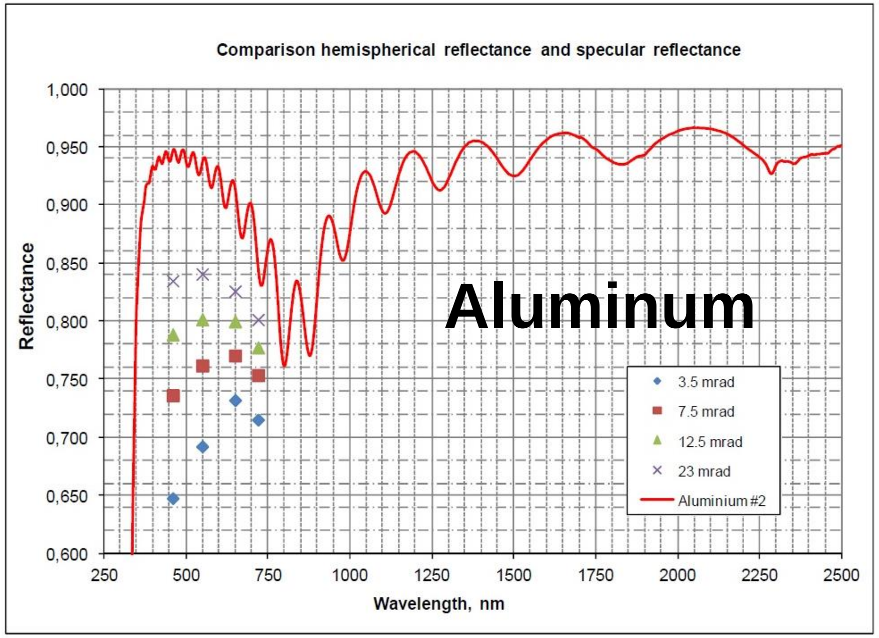
Optical components – reflectors – state of the art



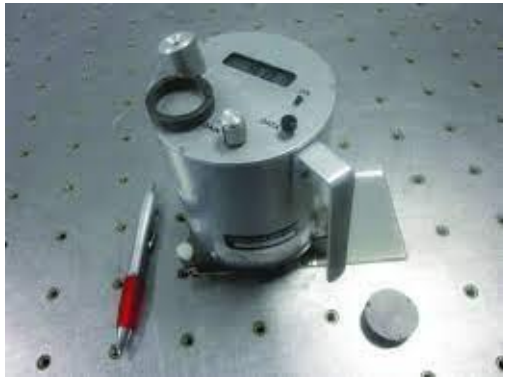








Optical components – alternative reflectometers



D&S (various)

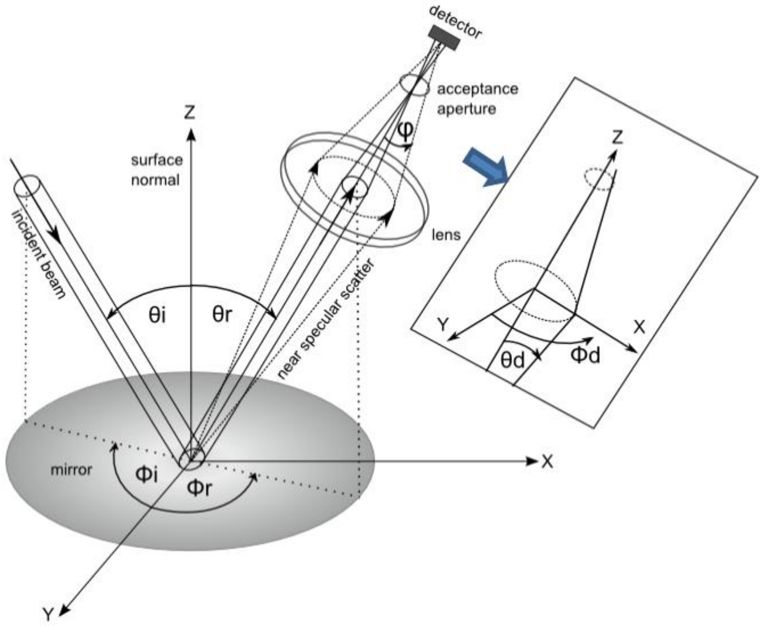
Condor

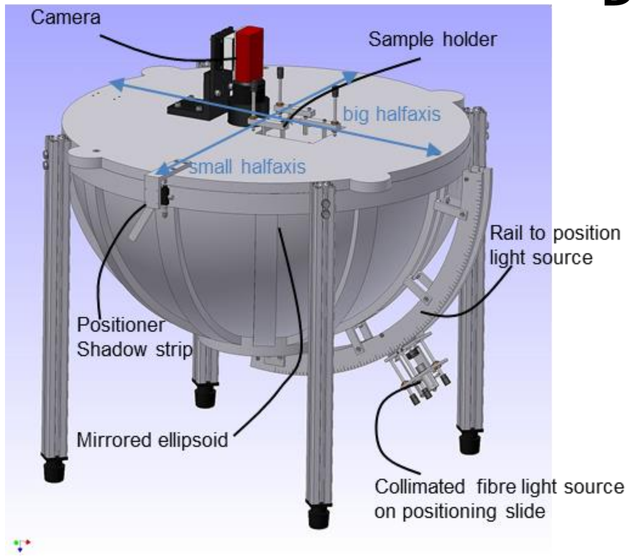
Konica Minolta

SOC 410 Solar

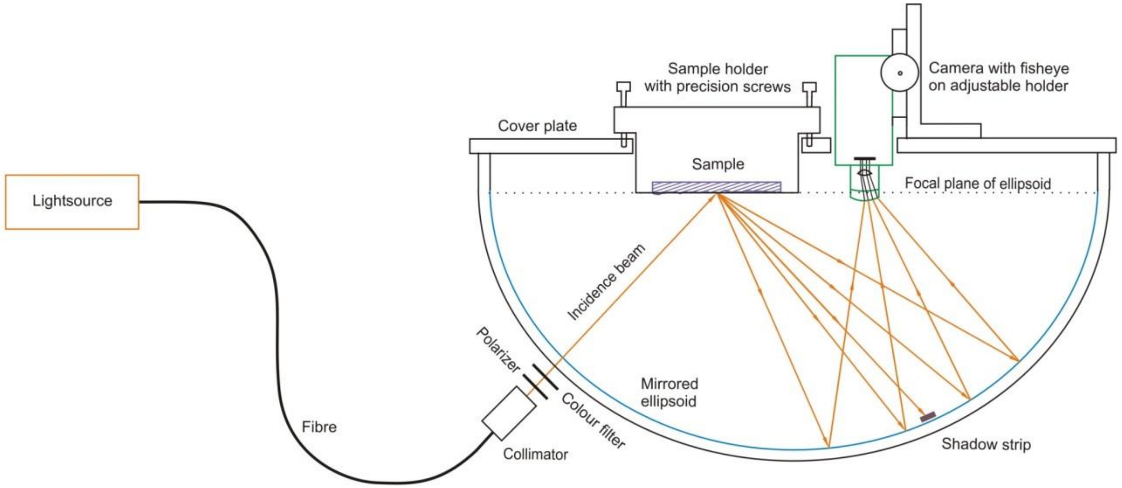
pFlex

Optical components – novel developments - MIRA





- Measurement of complete BRDF
- Allows to compute reflectance as function of φ
- Variable incidence angle θ = 6°- 60°
- White light or λ = 450, 500, 550, 650, 700, 850, 940 nm

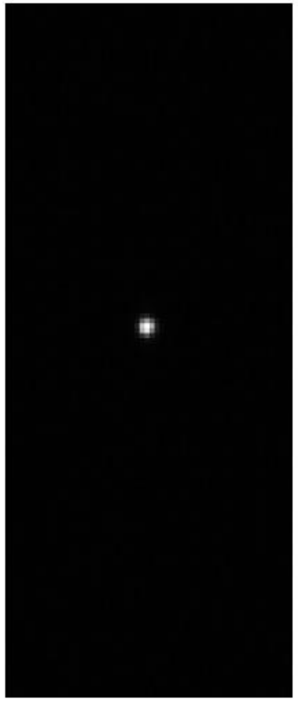


[4]

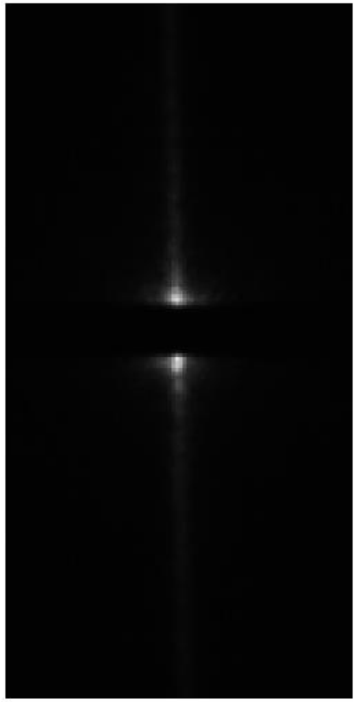
Optical components – novel developments - MIRA

Pictures to be taken for analysis:

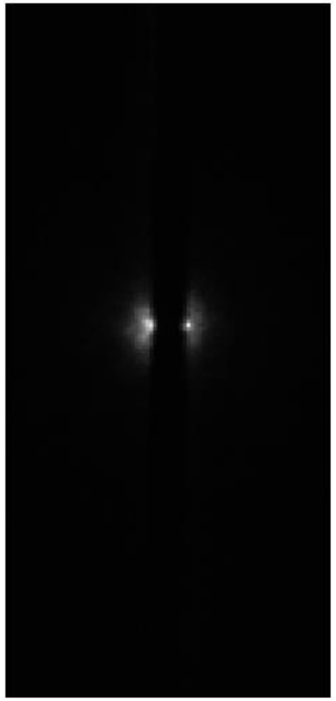
peak image

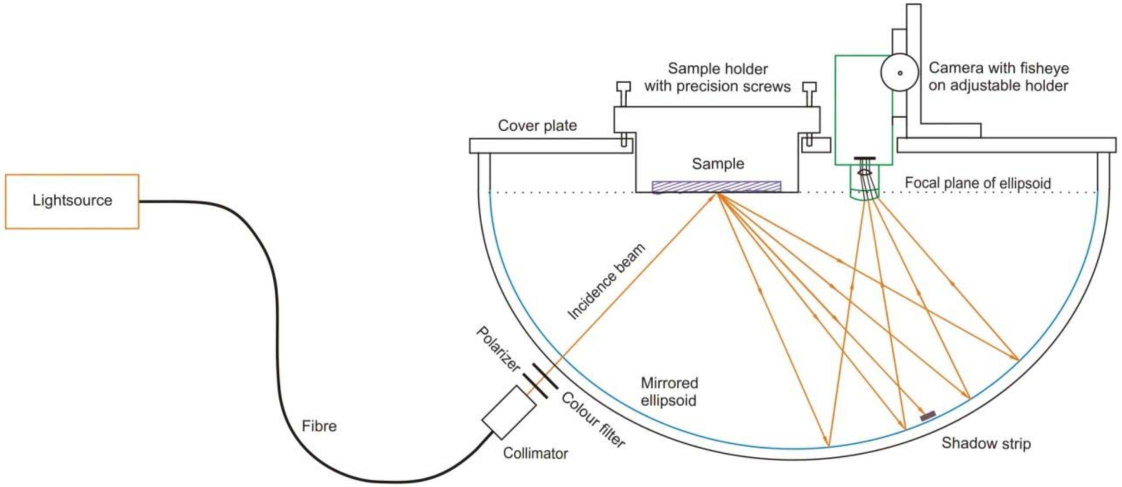


horizontal shadow strip



vertical shadow strip

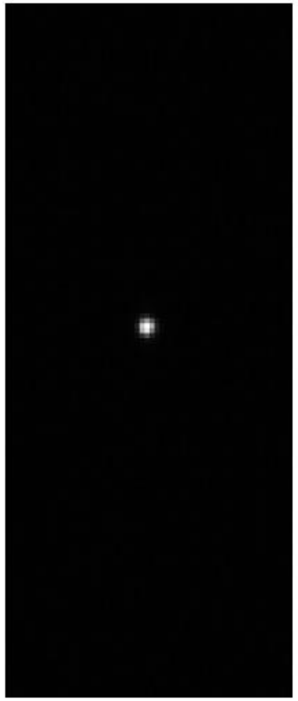




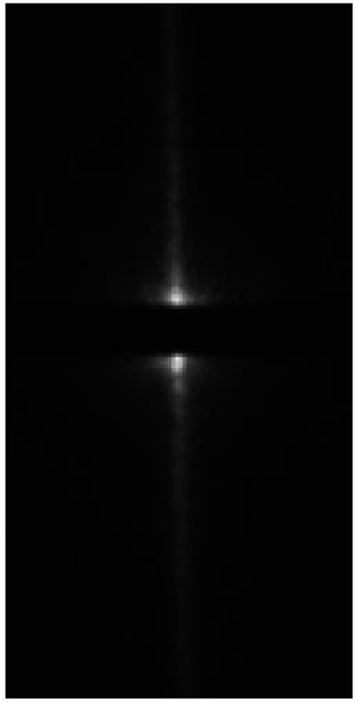
[4]

Optical components – novel developments - MIRA

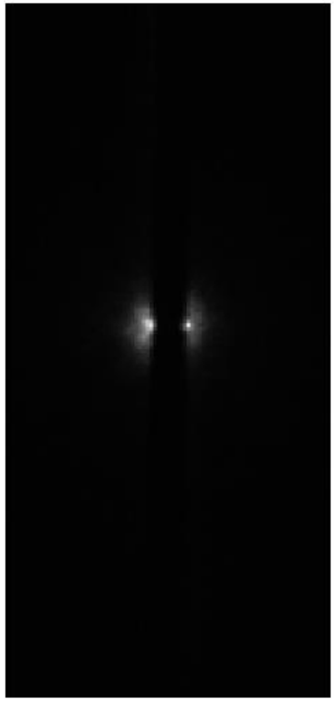
peak image

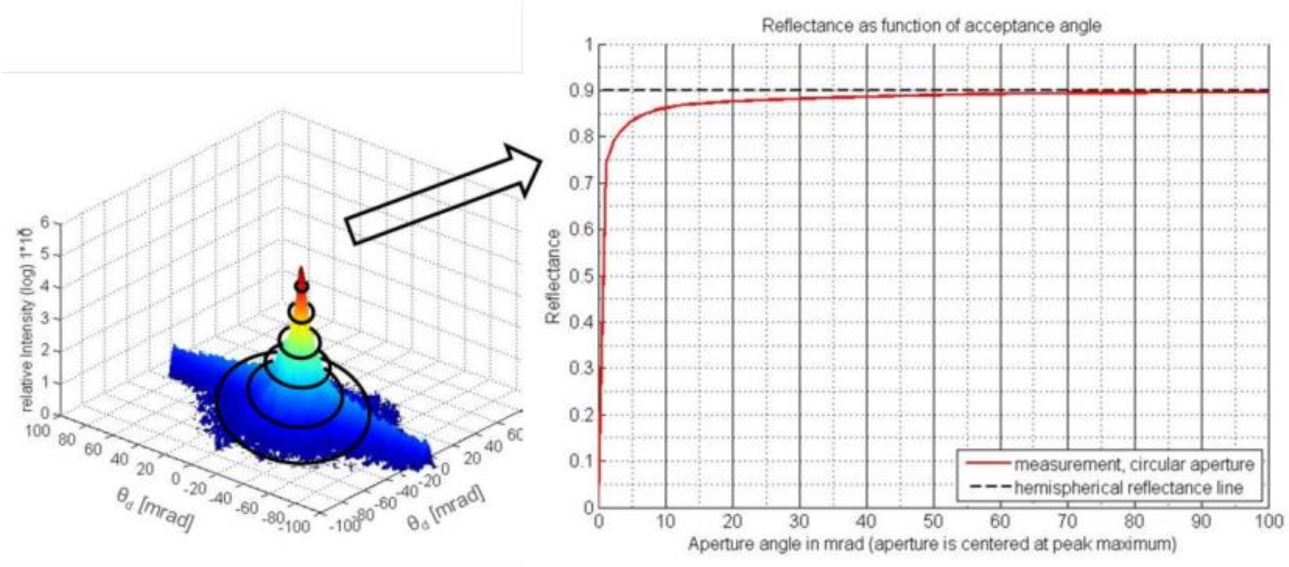


horizontal shadow strip



vertical shadow strip

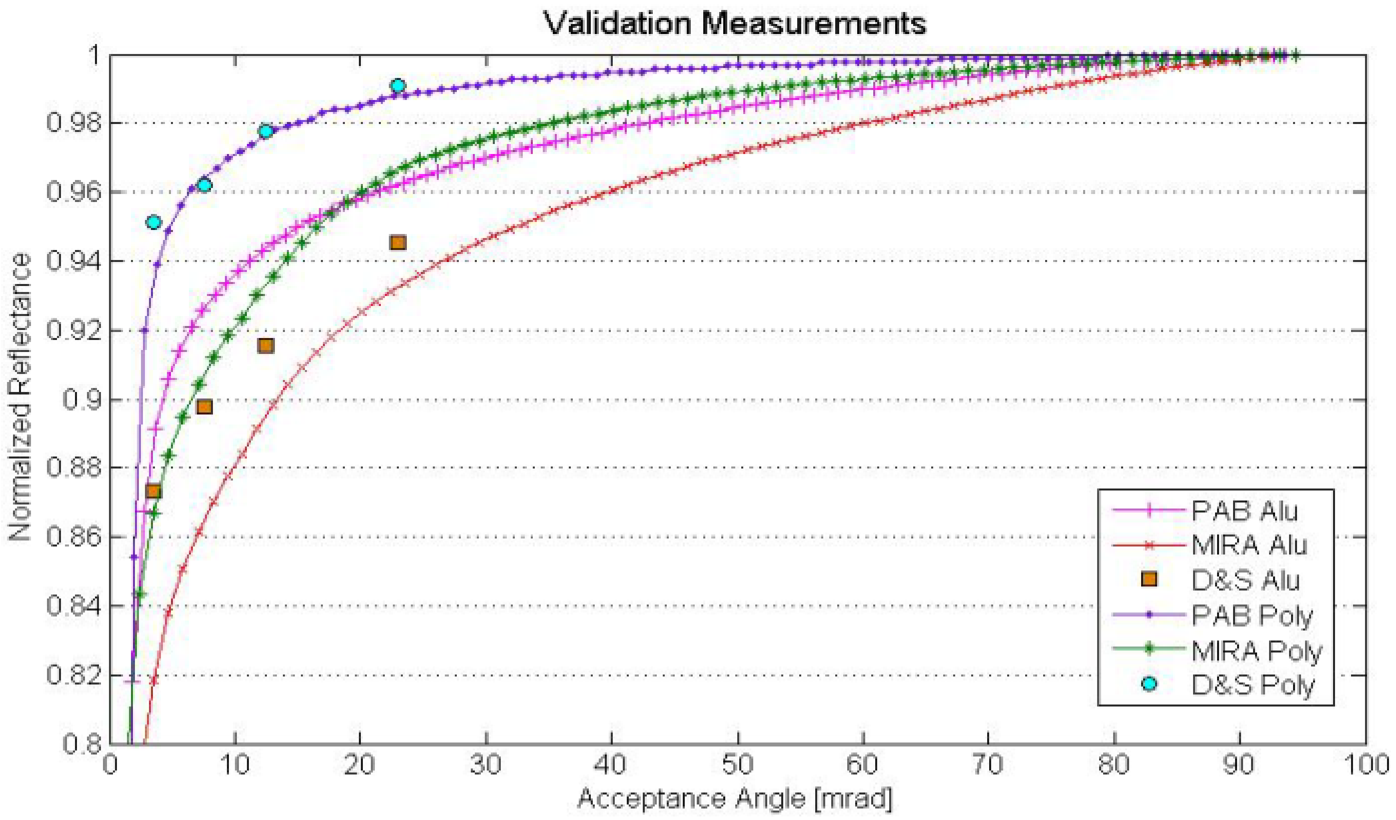




Backscattered Reflectance Distribution Function can be spherically integrated to Reflectance Over Acceptance angle function

[4]

Optical components – novel developments - MIRA



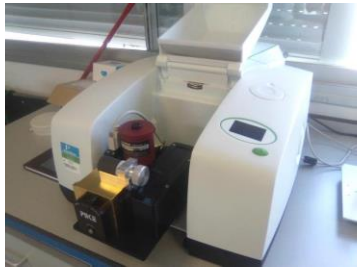
D&S measurements at slightly different wavelength

[4]

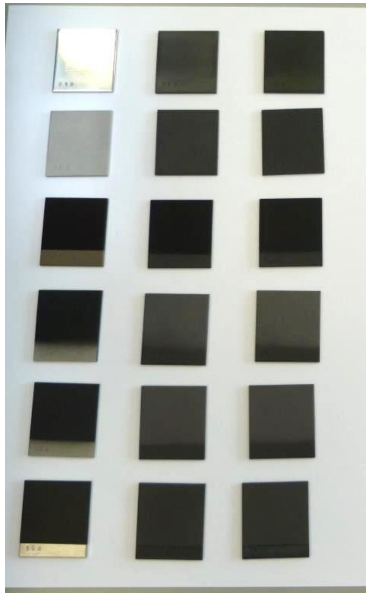
Optical components – absorber coatings – theory

Room Temperature (RT)
Discrete meas. (points)

- **Optical characterization (OPAC, CIEMAT-DLR)**
  - Spectral measurements
    - Measurement Equipment
      - Lambda1050 (0.28 – 2.5 µm)
      - Frontier FTIR (2 … 16 µm)
    - Quantitative analysis
      - α_{sol} (AM1.5, ASTM G173-03)
      - $\varepsilon_{th,calc}$ (Tmax)
  - Visual surface inspection
    - mm scale (Camera)
    - µm scale (Microscope)



Substrate metal in form of tubes or sheets



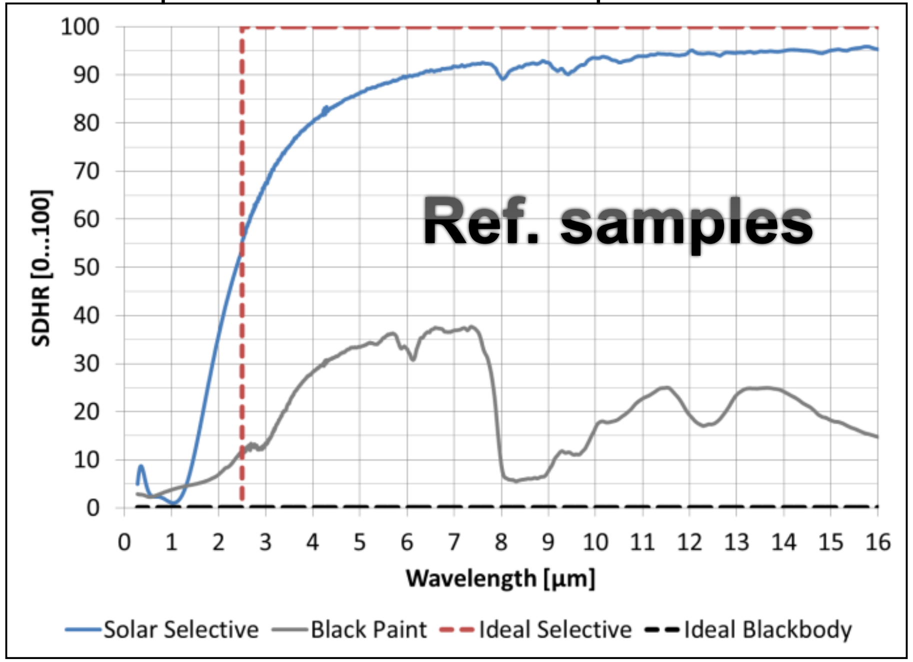
No quantitative adhesion test
No thermal conductivity data
No metallographic analysis

Optical components – absorber coatings – theory

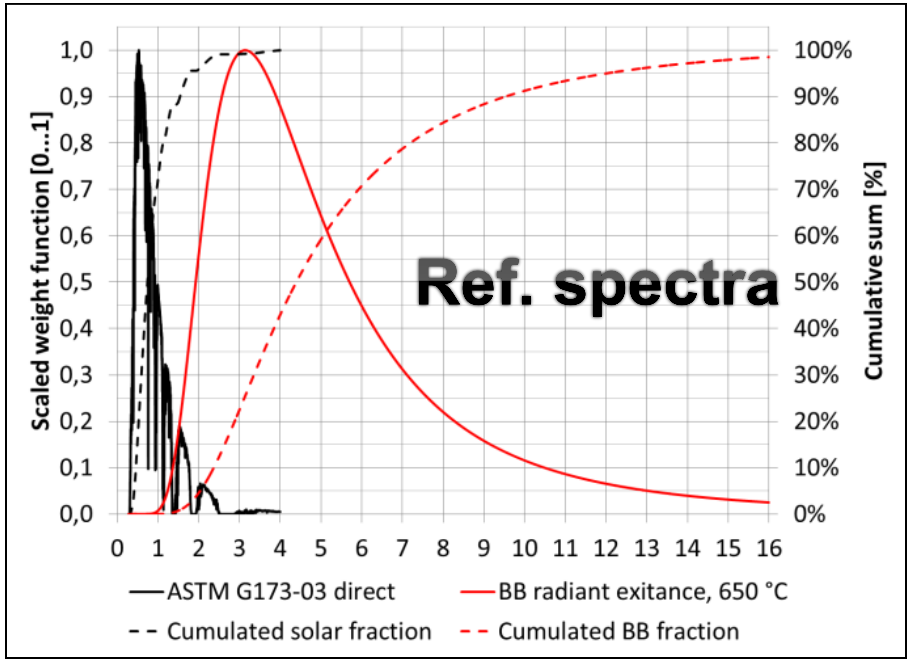
- **Baseline calibration**

$$\rho_{sample}(\lambda) = \frac{R_{sample,meas}(\lambda)}{R_{baseline,meas}(\lambda)} \cdot \rho_{baseline,ref}(\lambda)$$

SDHR: Spectral Directional –Hemispherical Reflectance

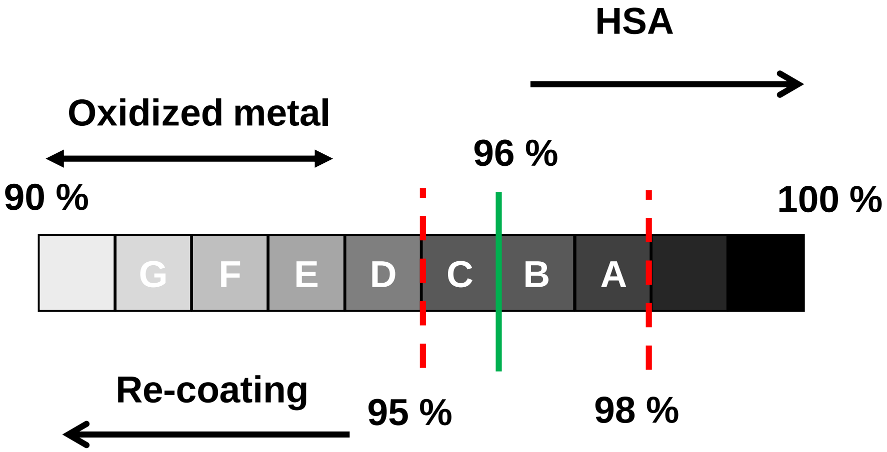


- **Weight functions**:
  - α_{sol}: ASTM G173-03 direct, AM1.5
  - ε_{th}: Blackbody spectrum (650/750 °C)
  - 8-12° incidence angle (Near normal)

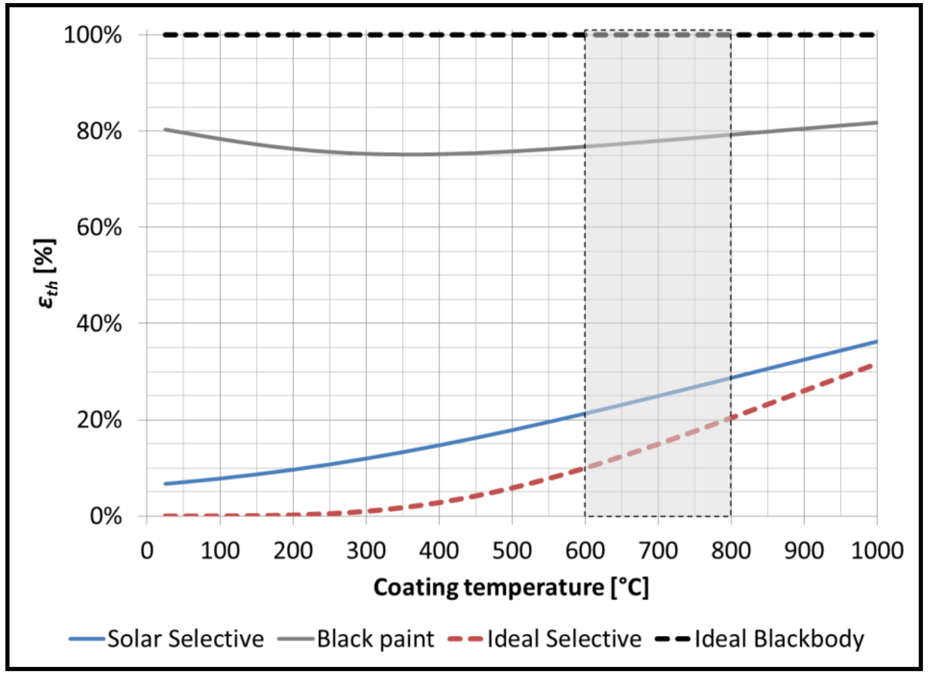


Optical components – absorber coatings – theory

- Solar absorptance α_{sol}
  - HSA: High Solar Absorptance (> 96%)



- Thermal emittance ε_{th} (T)



Optical components – absorber coatings – theory

- Opto-thermal efficiency:

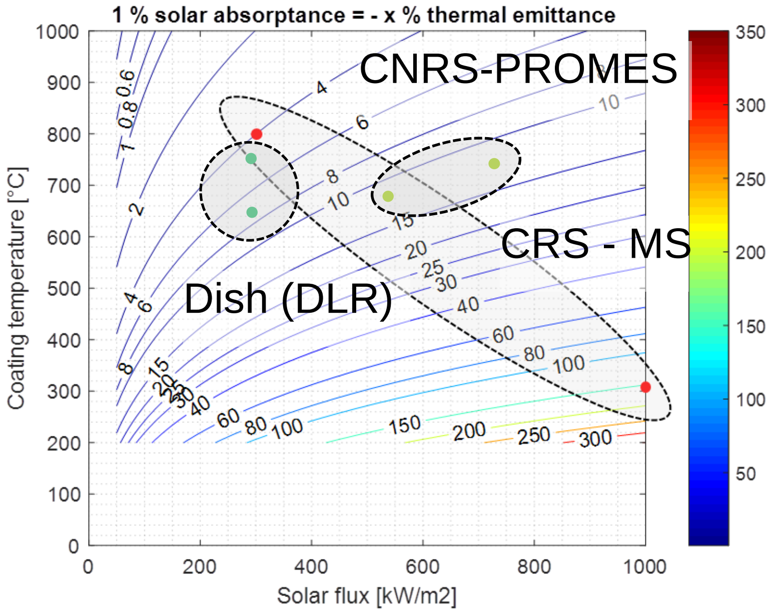
$$\eta_{coating} \approx \frac{\alpha_{sol} \cdot \dot{q}''_{sol} - \varepsilon_{th}(T_{abs}).\sigma.T^4_{abs}}{\dot{q}''_{sol}}$$

- Trade-off factor Z:

$$Z = \frac{\Delta\alpha_{sol}}{\Delta\varepsilon_{th}} = -\frac{\dot{q}''_{sol}}{\sigma.T^4_{abs}}$$

- Solar absorptance predominant for Central Receiver System (CRS)

- Allowable Flux Density (AFD)
  Vant-Hull, *J. Sol. Energy Eng*. 2002, 124(2): 165-169
  - for Molten Salt HTF (Corrosion)
  - ***High* Flux & *Low* Temp,**
    ***Low* Flux & *High* Temp.**



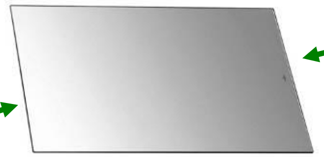
short break & discussion

afterwards: outdoor exposure and aging simulation

Accelerated aging – outdoor exposure

Radiation
- total solar radiation
- especially UV-A, UV-B

Mechanical stresses
- wind loads
- stresses due to mounting



Environmental pollutants
- chemical reactions with dust or sand
- pollutants from surrounding industry (e.g. coal plants)
- proximity to sea

Temperature
- cyclic day/night temperature changes
- frost

Humidity
- ambient relative humidity
- dew
- rain

Abrasion/Erosion
- airborne dust or sand
- cleaning mechanism

Accelerated aging – outdoor exposure

Galvanic separation sample-rack

Facing towards equator

45° tilt angle

+ Covers all environmental stresses

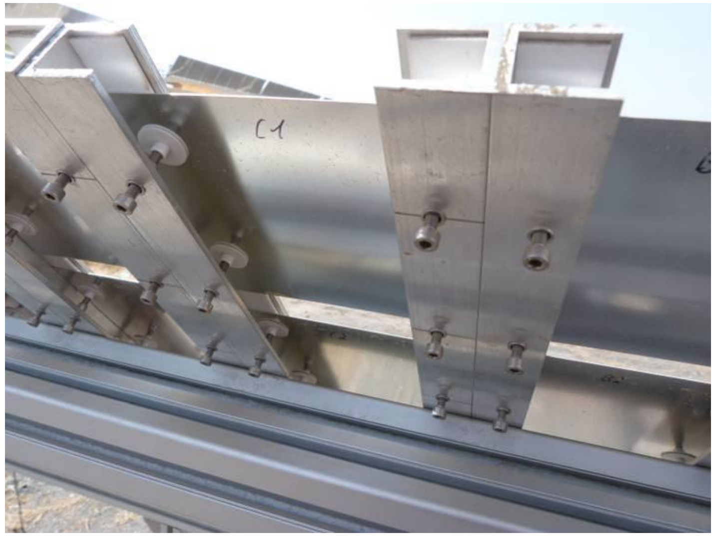
+ Simple

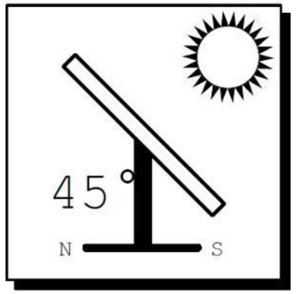
- No acceleration

- Various exposure sites needed

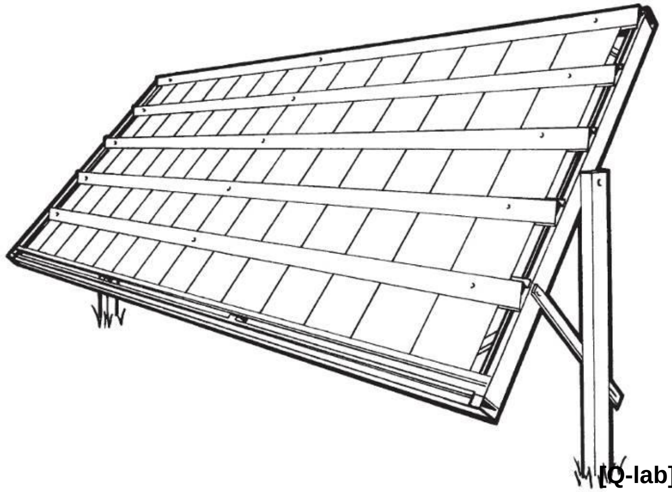
- Requires partners

- Sample degradation during handling / shipping



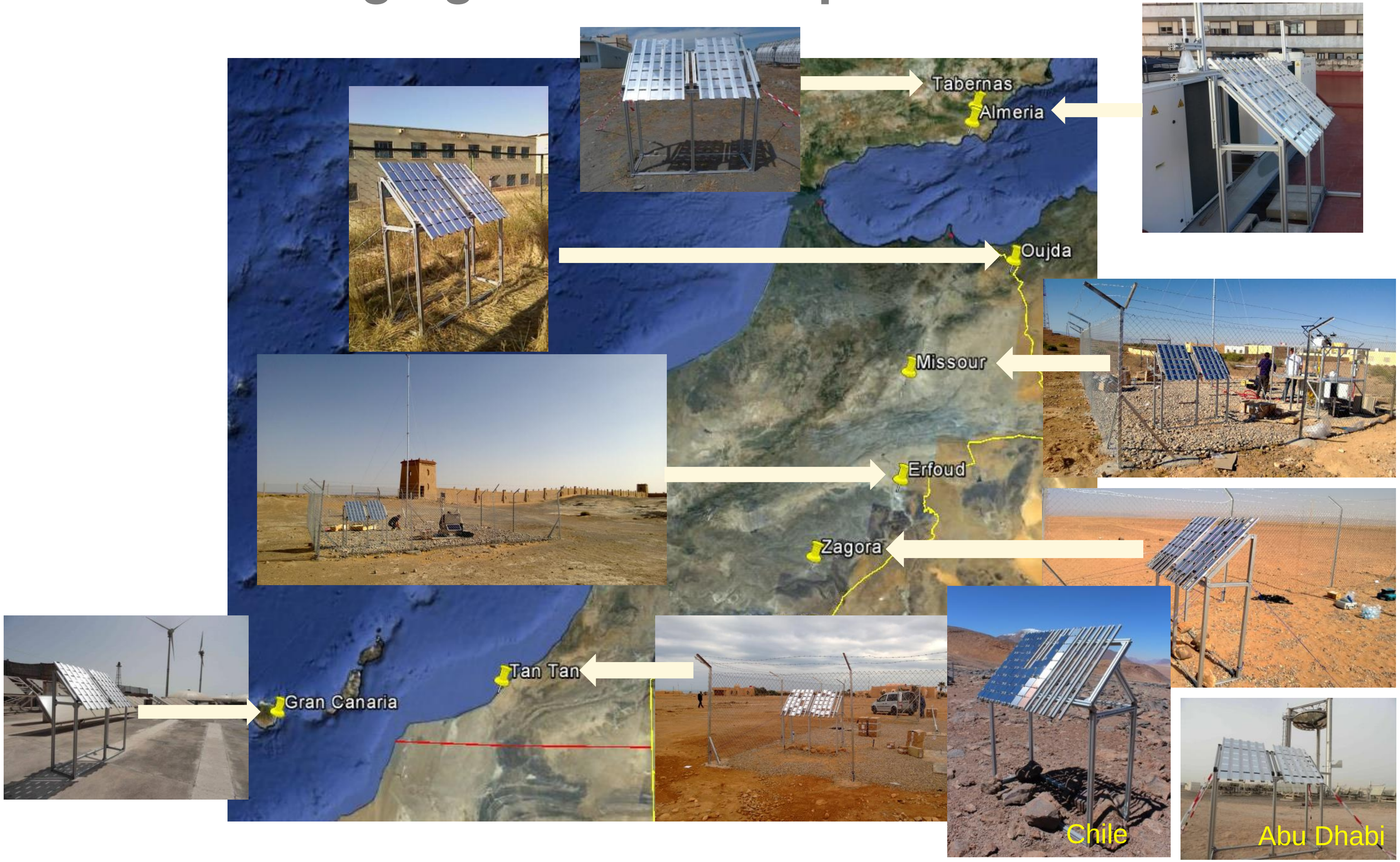


45 degree tilt angle, facing South.



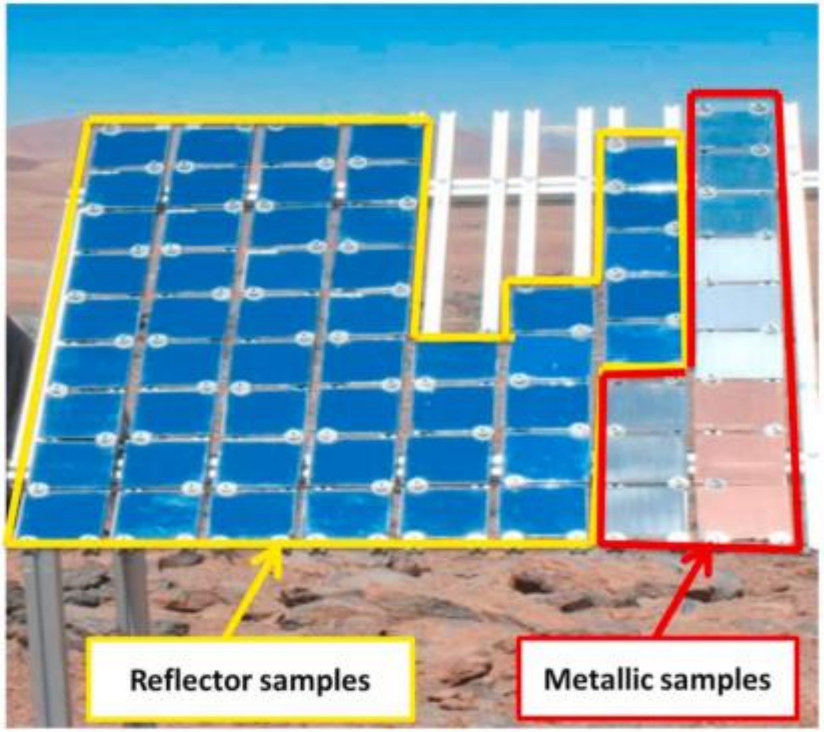
[Q-lab]

Accelerated aging – outdoor exposure



Accelerated aging – outdoor exposure

- In order to assess site corrosivity: ISO 9223:2012 standard:
    - 10x10 cm² coupons of steel, copper, zinc and aluminum exposed for 1 year.
    - Afterwards collect, clean and determine weight loss.
    - Classify in 6 categories (C1,…,C5,CX)



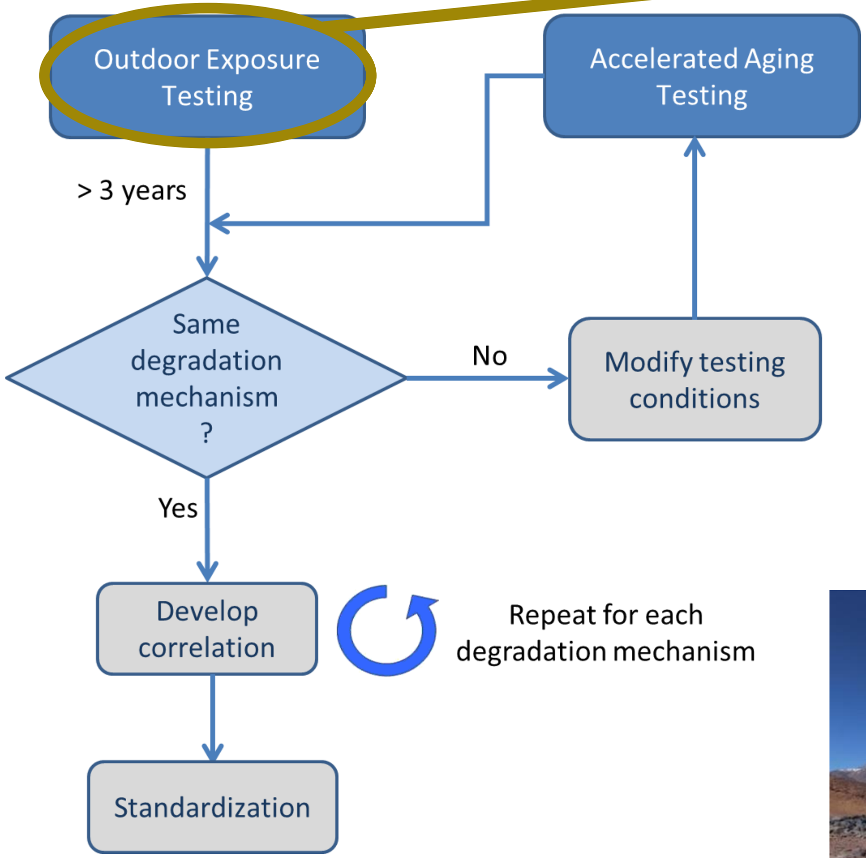
Reflector and standard metal samples in outdoor exposure rack.

Corrosion Testing

Site	Average mass loss (g/m^2)				Average corrosivity class
	Zinc	Cooper	Aluminum	Steel	
Almeria	8.78	43.4	1.19	346	**C4**
Tabernas	3.79	15.3	0.45	132	**C3**
Odeillo	3.30	5.61	0.09	8.59	**C2**
Missour	5.05	7.62	0.14	43.4	**C2**
Erfoud	4.01	19.8	0.30	65.7	**C2**
Zagora	2.44	5.15	0.03	28.3	**C2**
Ouarzazate	3.75	7.15	0.47	19.4	**C2**
Temara	398	190	4.83	7769	**CX**
Atacama Desert	5.41	20.5	0.64	210	**C3**
Chajnantor	3.50	6.38	0.27	103	**C2**

[6]

Accelerated aging – lifetime simulation



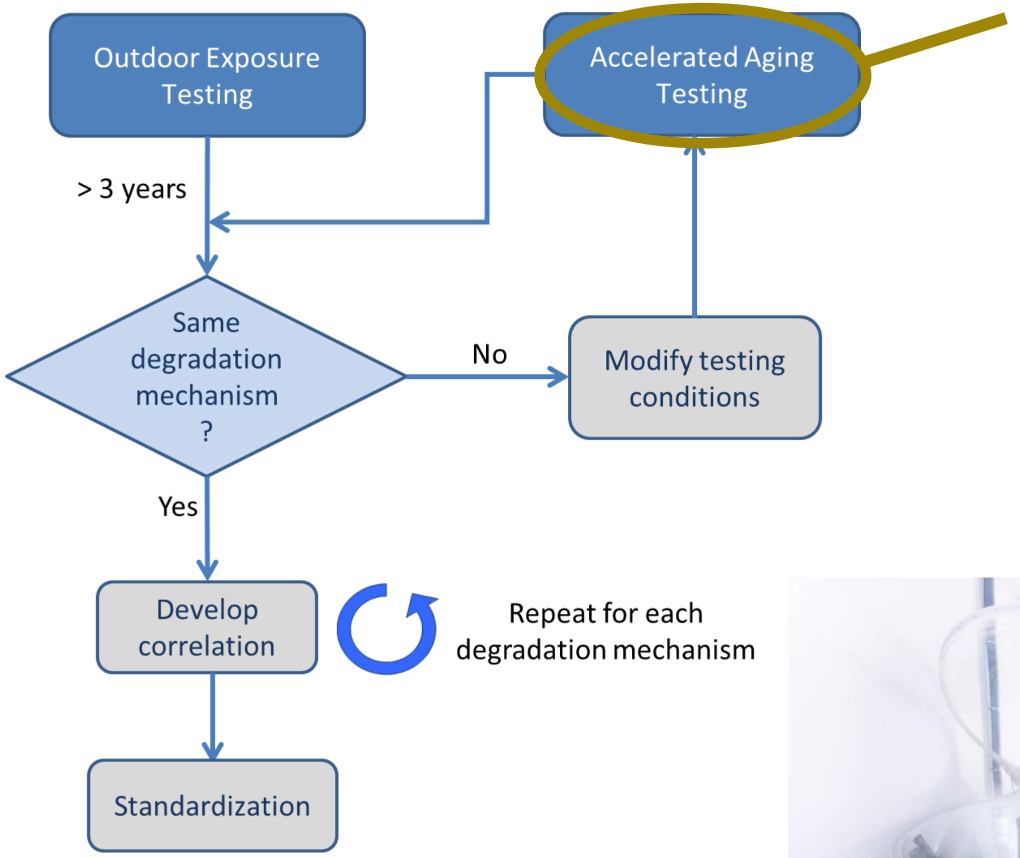
Network of 11 outdoor exposure sites:

	Corrosivity class ISO 9223					*Erosivity class*
Exposure site	**Zn**	**Cu**	**Al**	**Steel**	**Average**	
Temara (MAR)	X	X	4	X	**5.5**	**1**
Almeria (ESP)	3	5	3	3	**3.5**	**1**
Antofagasta (CHL)	3	4	1	3	**2.8**	**1**
Erfoud (MAR)	2	4	2	2	**2.5**	**1**
Tabernas (ESP)	2	4	2	2	**2.5**	**1**
Missour (MAR)	3	3	2	2	**2.5**	**1**
Ouarzazate (MAR)	2	3	2	2	**2.3**	**1**
Chajnantor (CHL)	2	3	2	2	**2.3**	**2**
Zagora (MAR)	2	2	1	2	**1.8**	**3**
Odeillo (FRA)	2	3	1	1	**1.8**	**1**

Chajnantor (Chile) in collaboration with Universidad Santiago de Chile

Erfoud, Morocco in collaboration with IRESEN

Accelerated aging – lifetime simulation

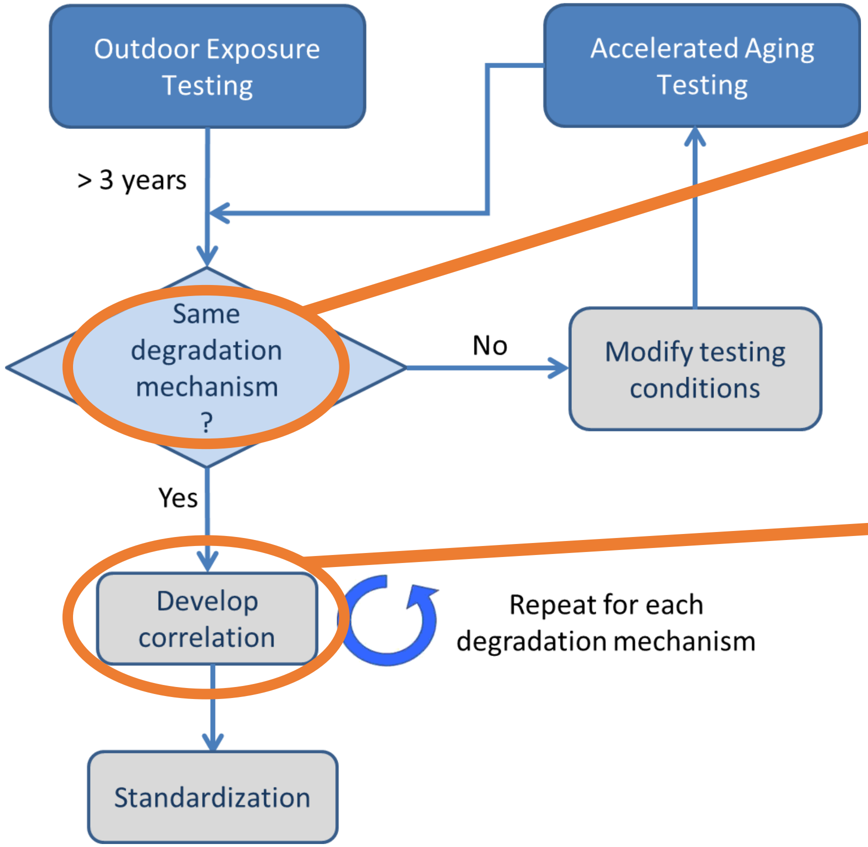


- Neutral Salt Spray (NSS) ISO 9227
- Copper Accelerated Salt Spray (CASS) ISO9227
- Humidity ISO 6270-2
- UV/Humidity ISO 16474-3
- Thermal cycling IEC 62108 (Test 10.6 TCA3)
- Thermal cycling/Humidity (UNE 206016)
- Damp Heat IEC 62108 (Test 10.7b)
- Sand erosion
- …
- Combined testing

Salt Spray testing according to ISO 9227

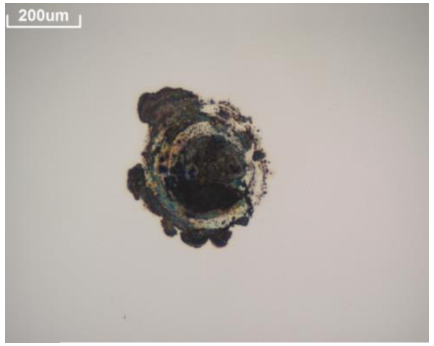
Sand erosion test bench at PSA

Accelerated aging – lifetime simulation

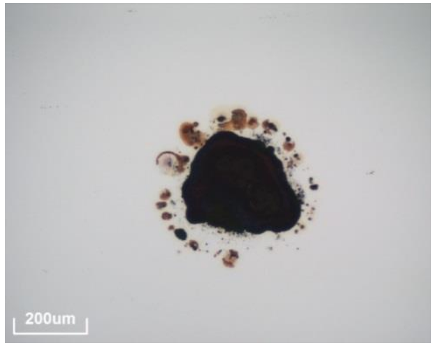


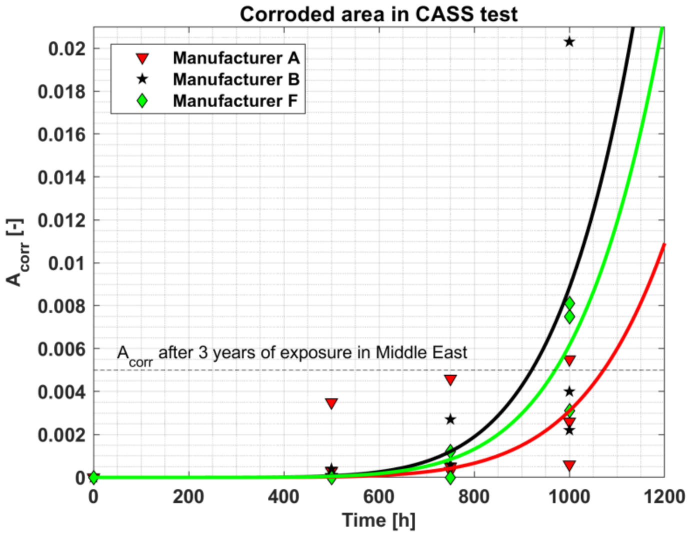
Corrosion of silver layer

After outdoor exposure



After 240h of CASS testing





Rough estimate based on 1 year corrosion data:

Corrosivity class of exposure site	Acceleration factor
X	26
4	41
3	91
2	118

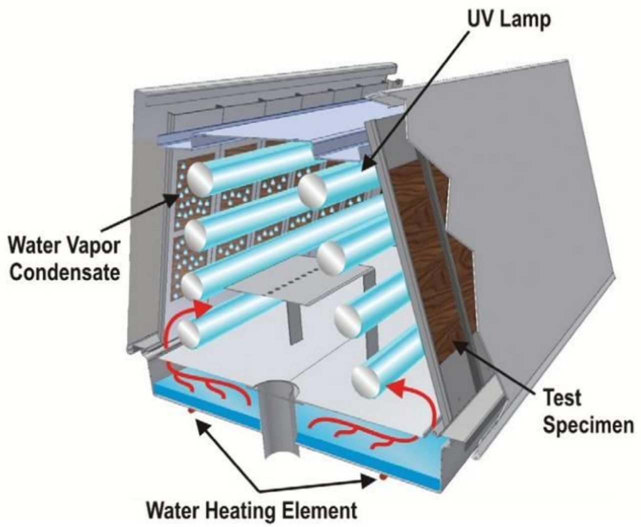
Preliminary data

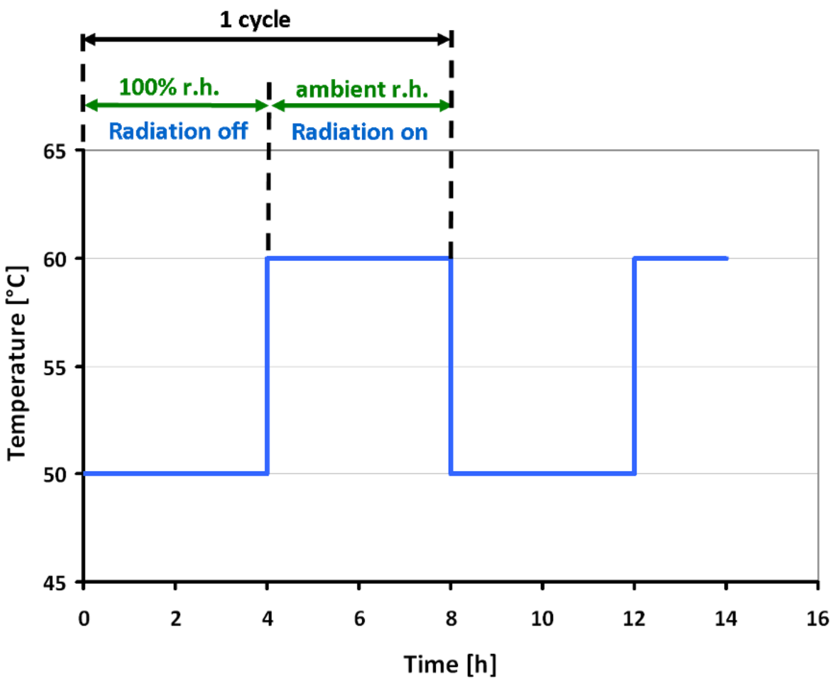
[5]

Accelerated aging – laboratory testing

ISO 11507: UV+Water Test

Chamber temperature:	50 to 60°C
Humidity:	ambient to 100% relative humidity
Radiation:	lamp type II, UVA-340; 290-400 nm; peak emission at 340nm; lamp power matches 1 sun
Cycle time:	8 hours
Testing time:	>1000 hours





Accelerated aging – laboratory testing

ISO 9227: Neutral salt spray test (NSS)

Chamber temperature:	35 ± 2 °C
Humidity:	constant 100% relative humidity
Sprayed solution:	demineralized water + 50 g/l NaCl (pH 6.5 – 7.2)
Condensation rate:	1.5 ± 0.5 ml/h on a surface of 80 cm²
Sample position:	20 ± 5° respect to vertical
Testing time:	480 – 3500 hours

Accelerated aging – laboratory testing

ISO 9227: Copper accelerated salt spray test (CASS)

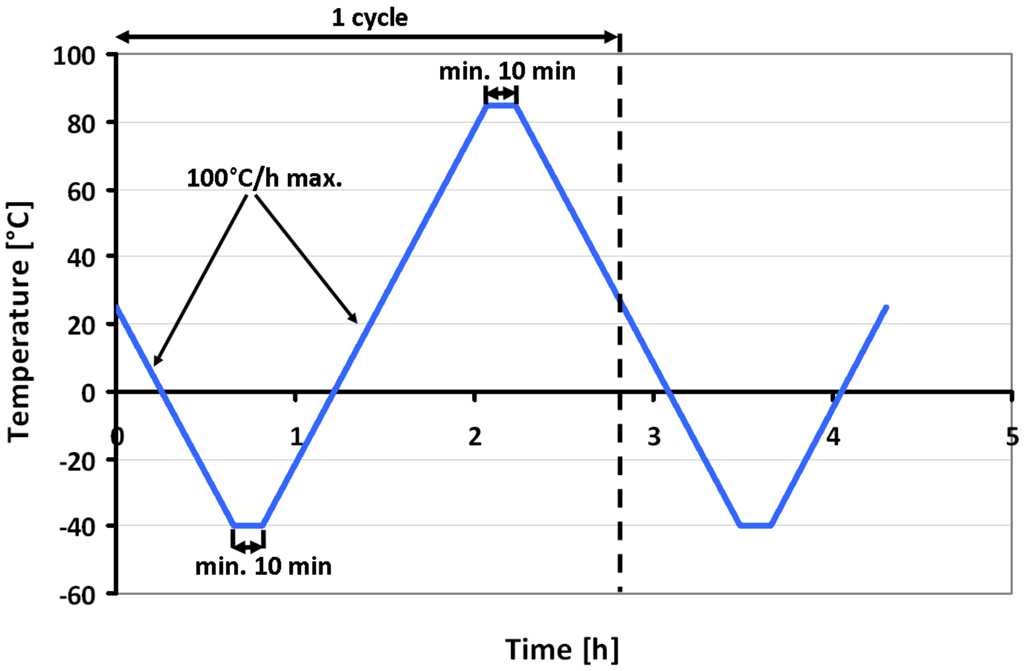
Chamber temperature: 50 ± 2 °C
Humidity: constant 100% relative humidity
Sprayed solution: demineralized water + 50 g/l NaCl + 0.26 g/l $CuCl_2$ (pH 3.1 – 3.3)
Condensation rate: 1.5 ± 0.5 ml/h on a surface of 80 cm²
Sample position: 20 ± 5° respect to vertical
Testing time: 120 – 480 hours

$CuCl_2$

Accelerated aging – laboratory testing

ISO 61215: Thermal Cycling

Chamber temperature: -40°C to +85°C
Humidity: dry
Cycle duration: min. 2h 50min, max. 6h
Recommended cycle number: >100

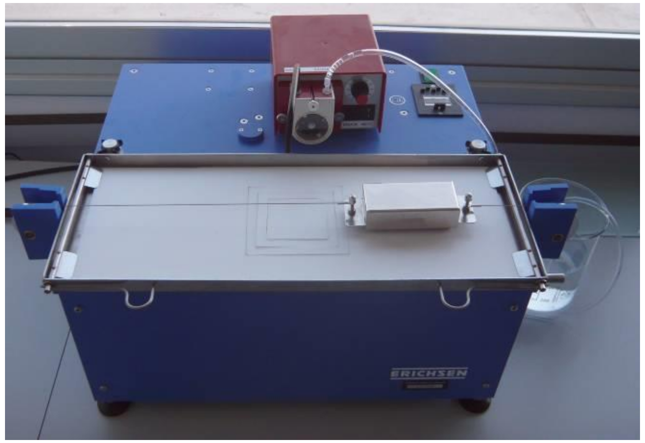


Accelerated aging – laboratory testing

DLR

Abrasion testing

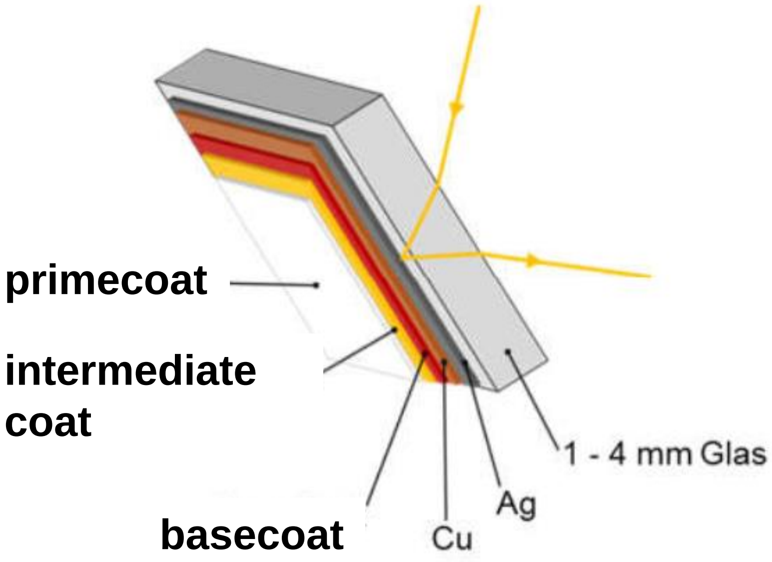
Available standards: ISO 11998, DIN ISO 9211-4

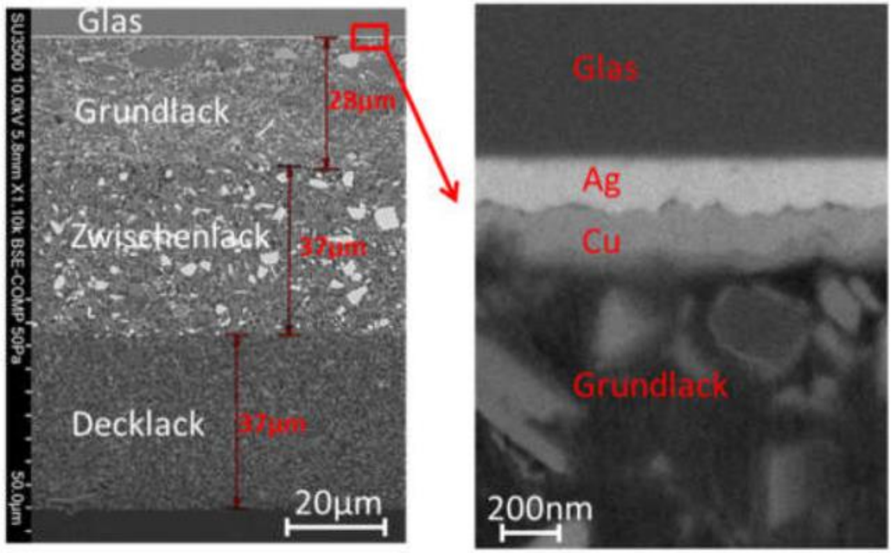


Simulation of cleaning cycles

Scratching of coatings with controlled normal force

Accelerated aging – Corrosion detection - motivation



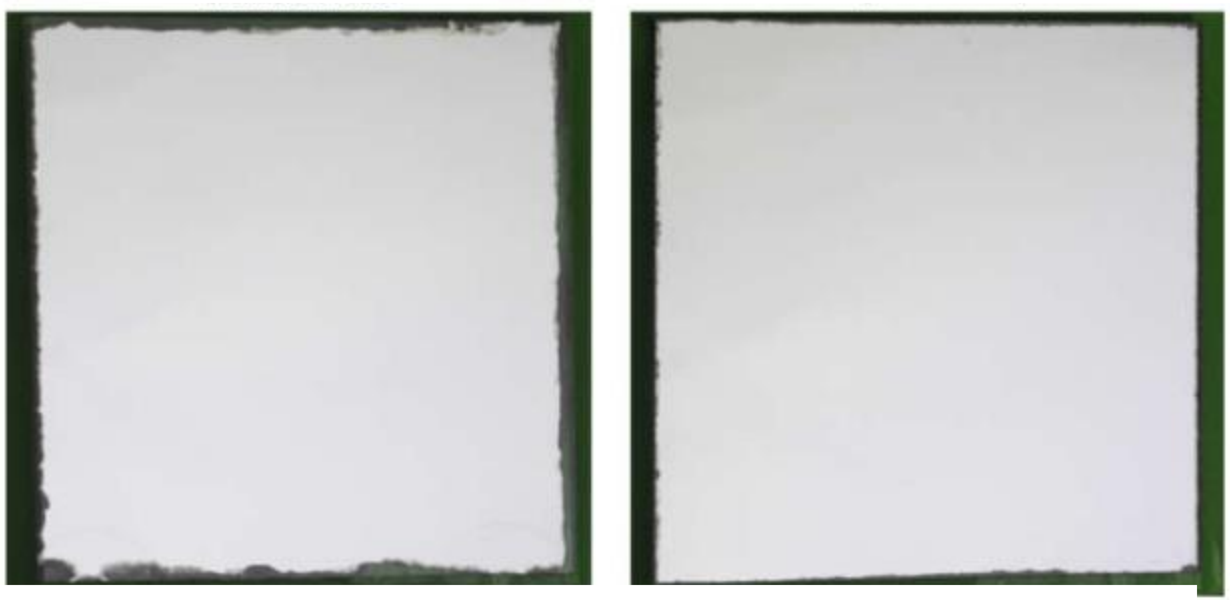


- Glas as corrosion protection
- Ag as reflecting layer and Cu as UV-protection and substrate for basecoat.
- Basecoat as humidity barrier (up to 10% Pb).
- Primecoat (TiO_2) abrasion and humidity protection

Accelerated aging – Corrosion detection - motivation

Traditional sample containing lead

Non lead sample



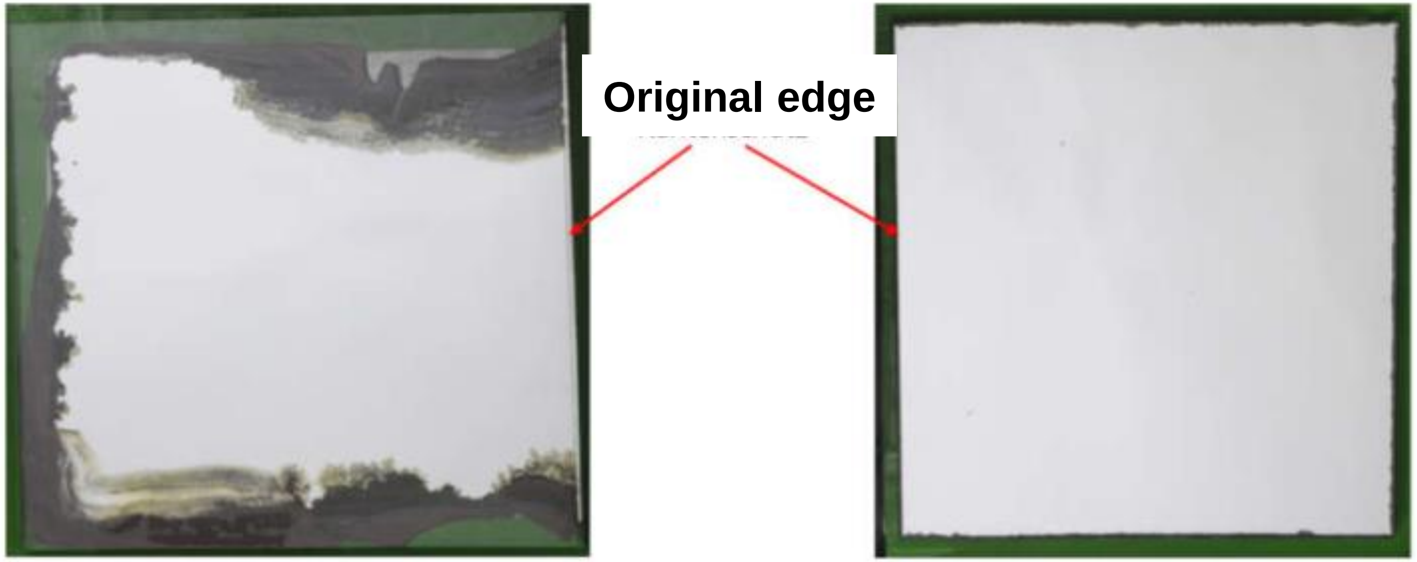
Sample after 2 years exposure in Almeria: coastal site, corrosion class C5 after ISO 9223

Higher degradtion rate for novel non-lead reflectors, especially on edges.

Accelerated aging – Corrosion detection - motivation

Traditional sample containing lead

Non lead sample



Sample after 480 hours of CASS test

Higher degradtion rate for novel non-lead reflectors, especially on edges.

Accelerated aging – Corrosion detection - motivation

<u>State of the art:</u>

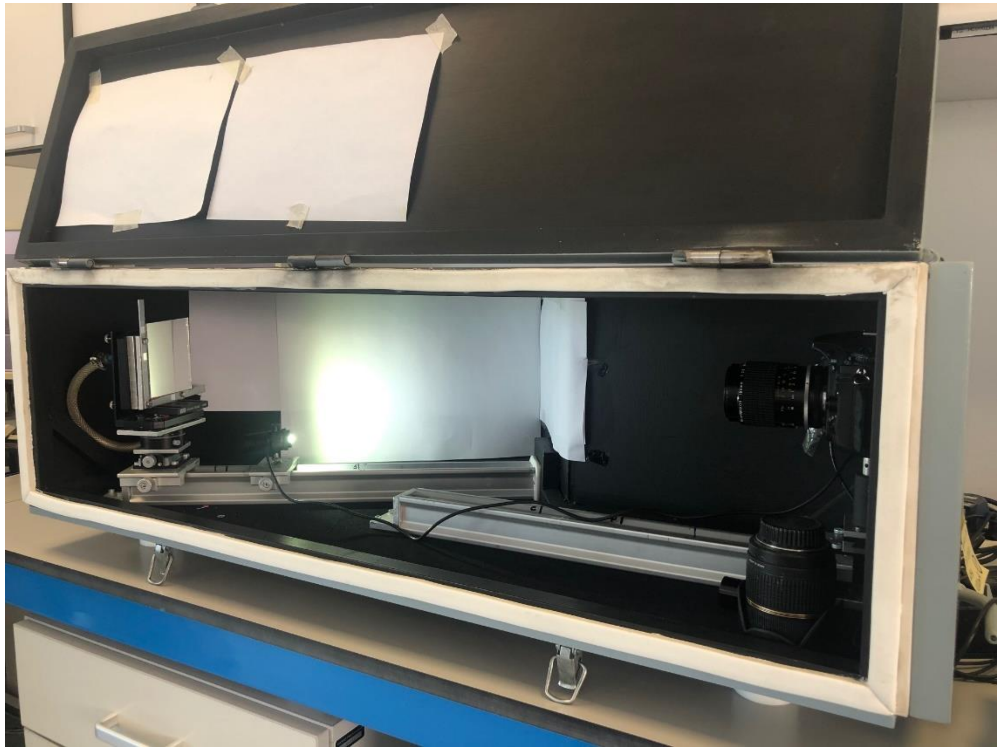
- Count corrosion spots per eye
- Mesure edge penetration with scale

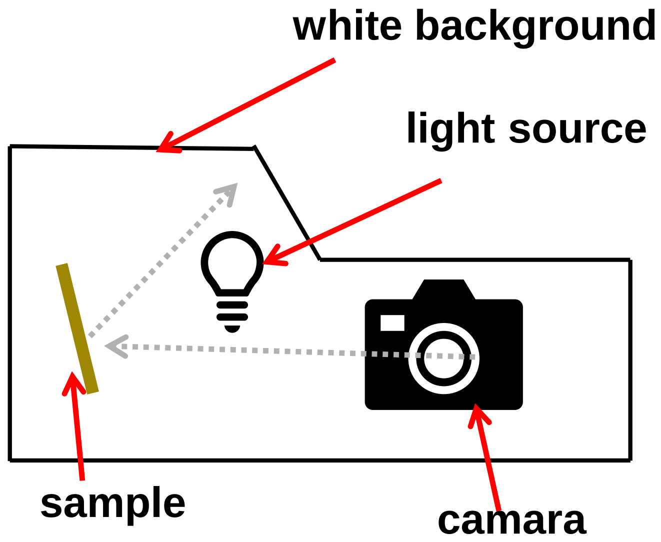
<u>Problems: Result highly depending on operator</u>

- Ilumination changes
- different sample materials
- spot / no spot
- one spot / two spots
- calculation of area imposible

→ Development of automatic image detection algorithm

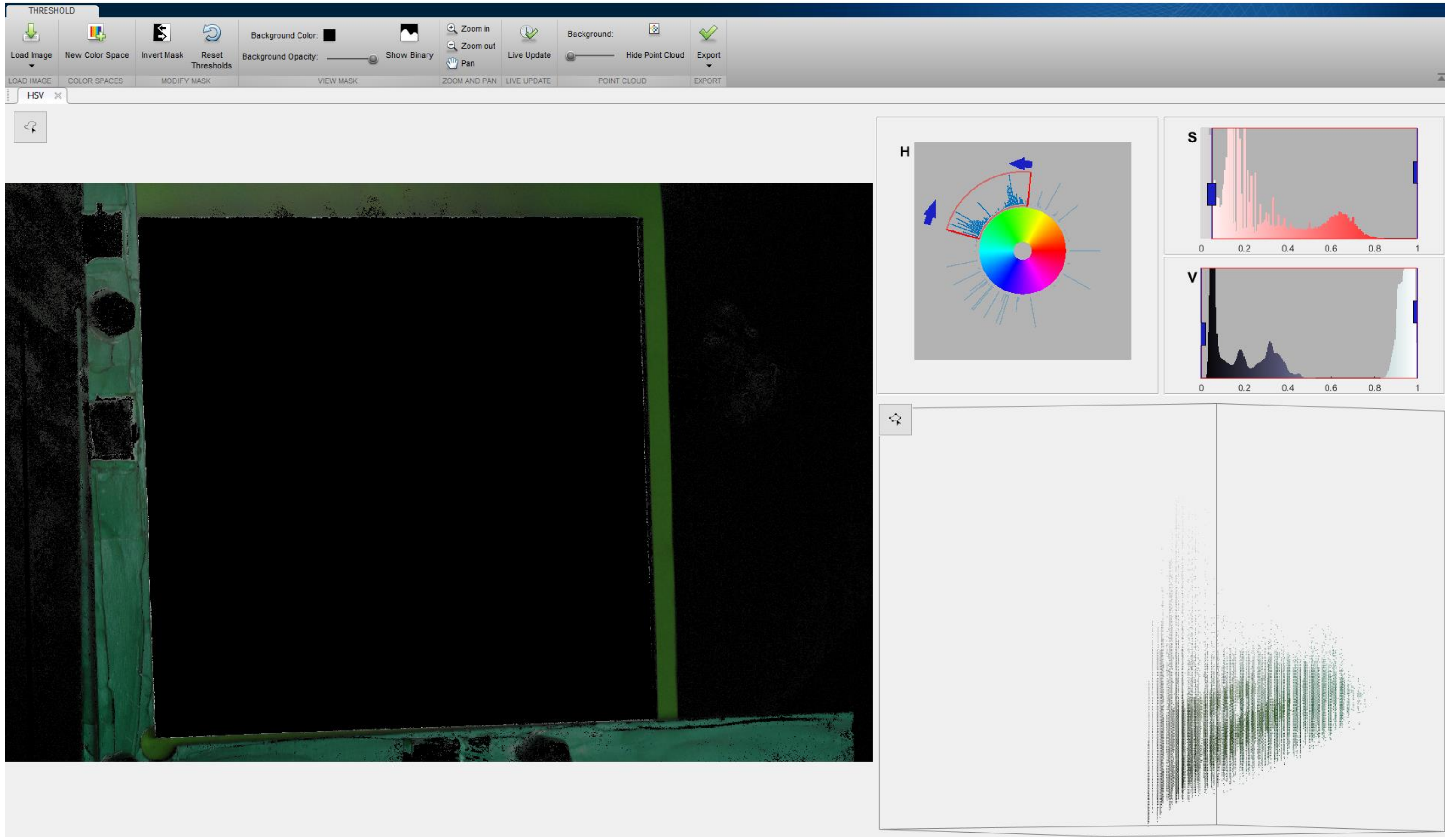
Accelerated aging – Corrosion detection



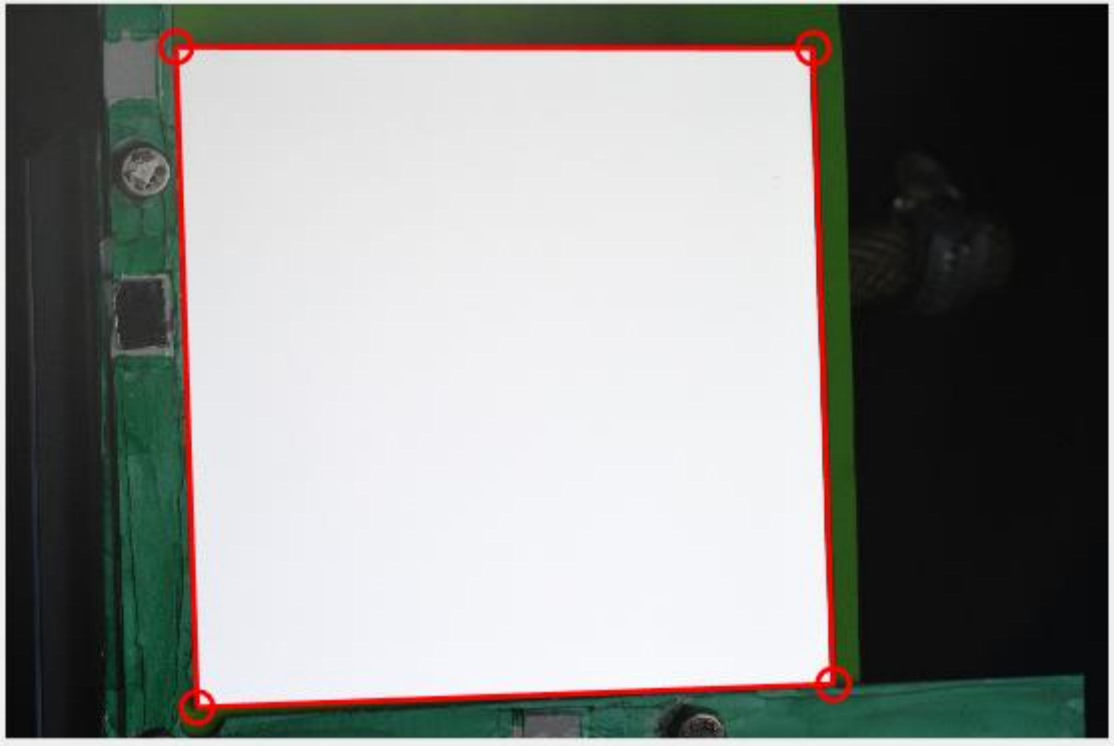


10 x 10 cm² sample results in ca. 5000 x 5000 pixel area.
→ 1 pixel = 20µm

Accelerated aging – Corrosion detection



Accelerated aging – Corrosion detection

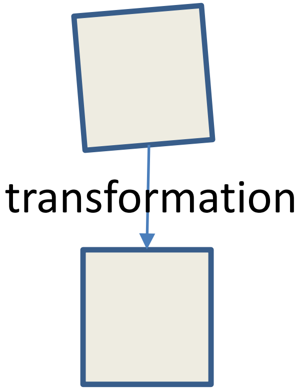


Next step is to detect the corner points of the large inner black area, assuming that it is a tetragon.

Accelerated aging – Corrosion detection

Transformation from tetragon to square to correct for the distortion and crop.

Aged image (AI), foto of a sample type in ist aged state.

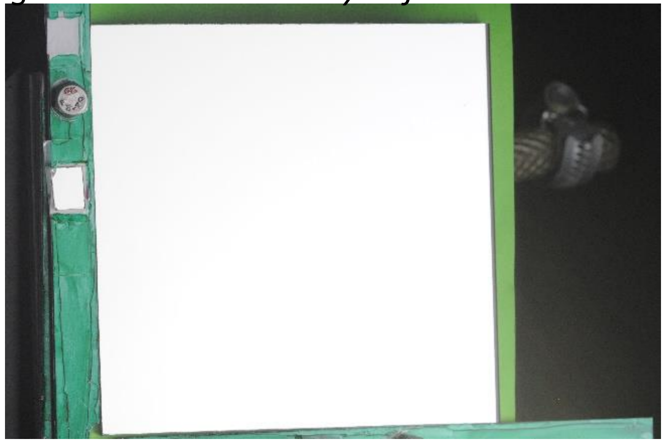


Aged image (AI) cropped and transformed

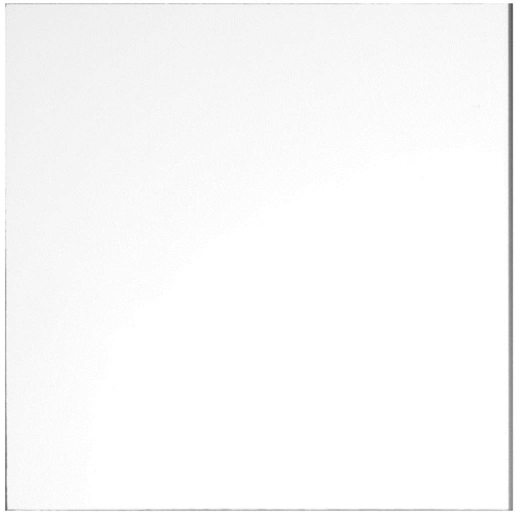
Problem: What is defect, what is shadow or coating or initial defect?

Accelerated aging – Corrosion detection

Reference image (RI), foto of a certain sample type in its original state without any defects or corrosion.



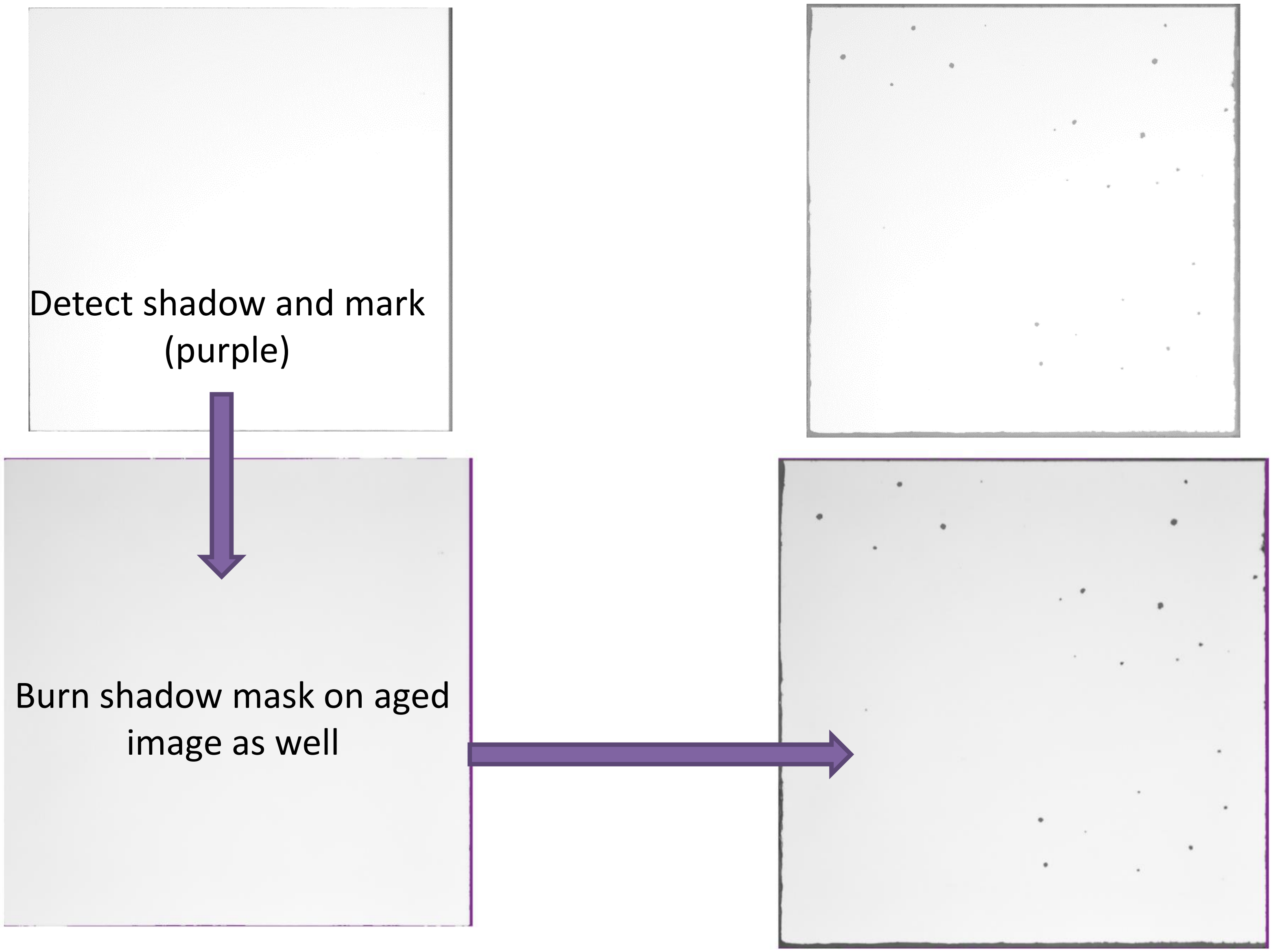
Aged image (AI), foto of a sample type in ist aged state.



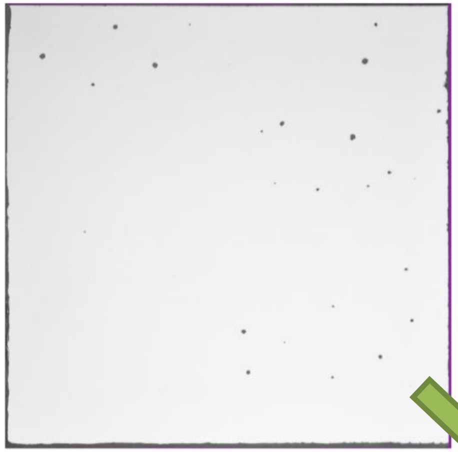
Reference image (RI) cropped and transformed

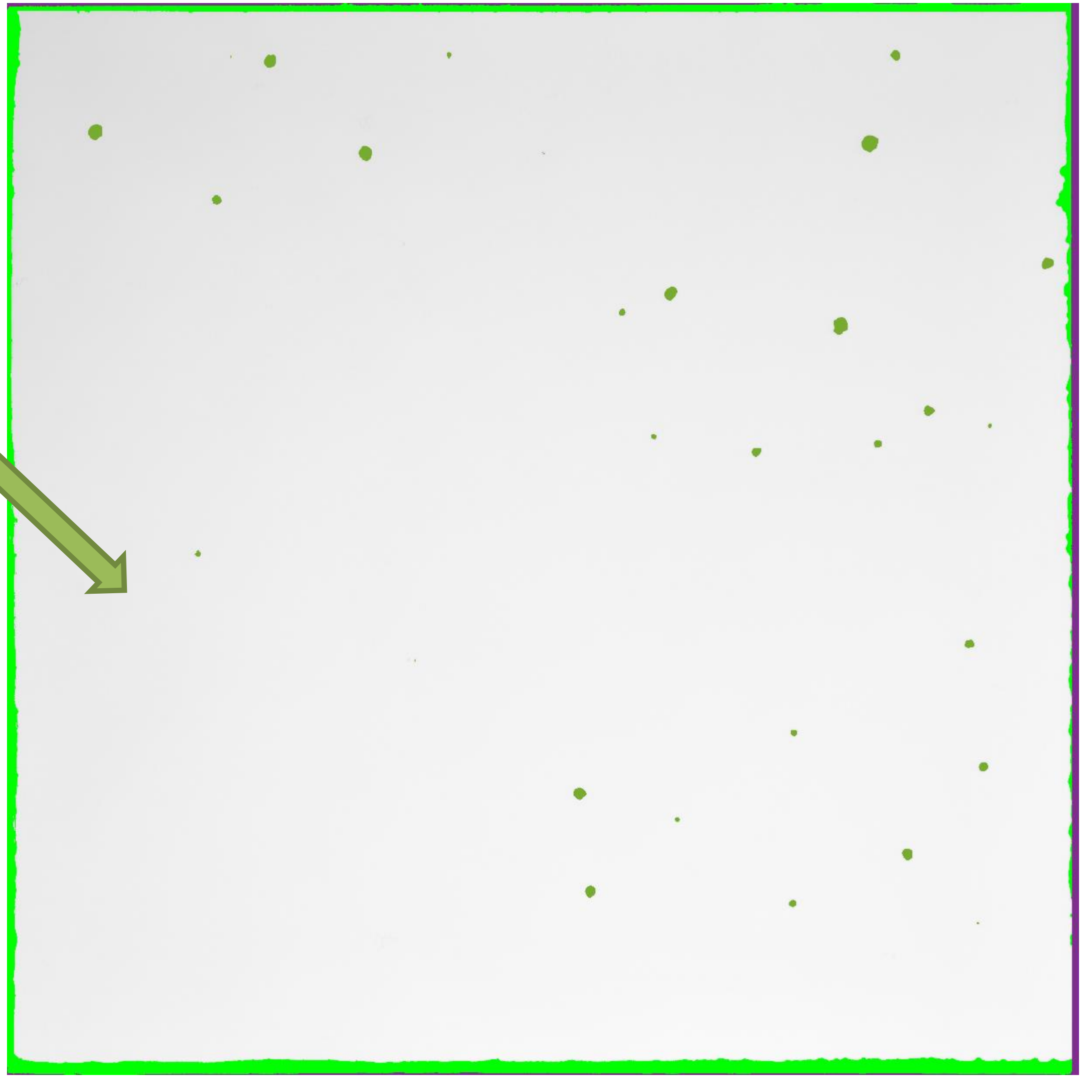
Accelerated aging – Corrosion detection

Aged image (AI) cropped and transformed



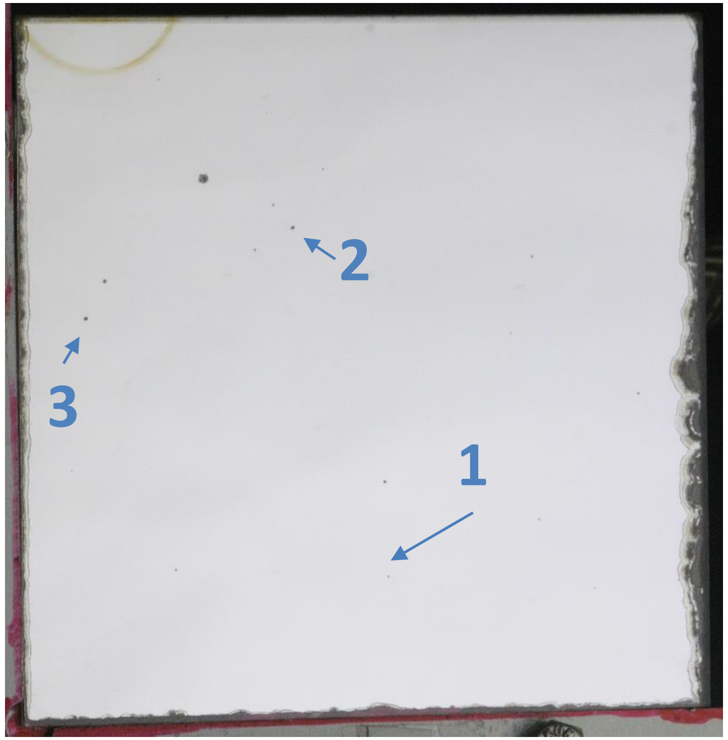
Accelerated aging – Corrosion detection

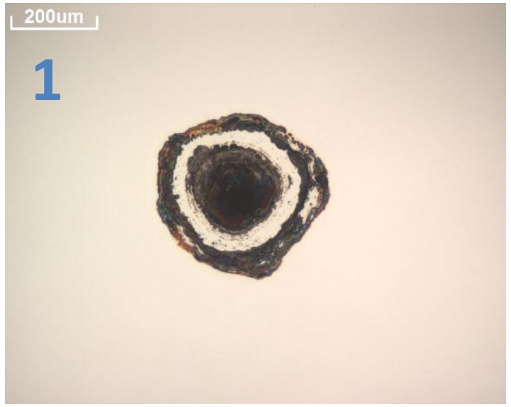


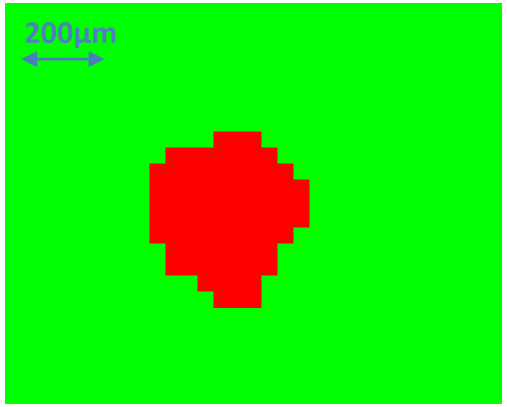


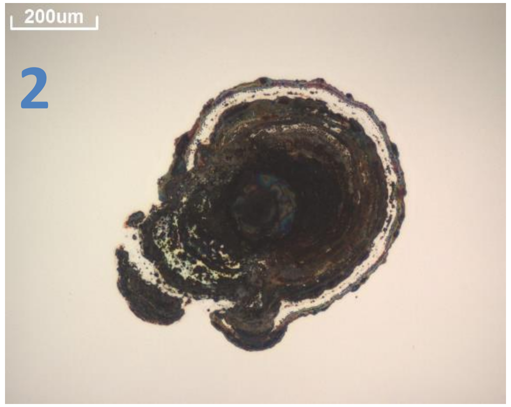
Divide Aged image (AI) by Reference Image (RI) and detect every pixel with less than certain brightness ratio of initial brightness as corrosion.

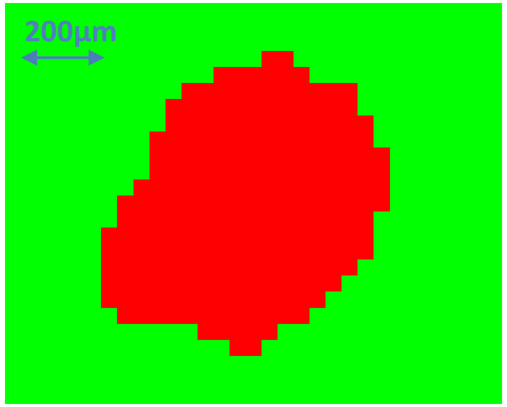
Inner corrosion (dark green) and edge corrosion (light green)

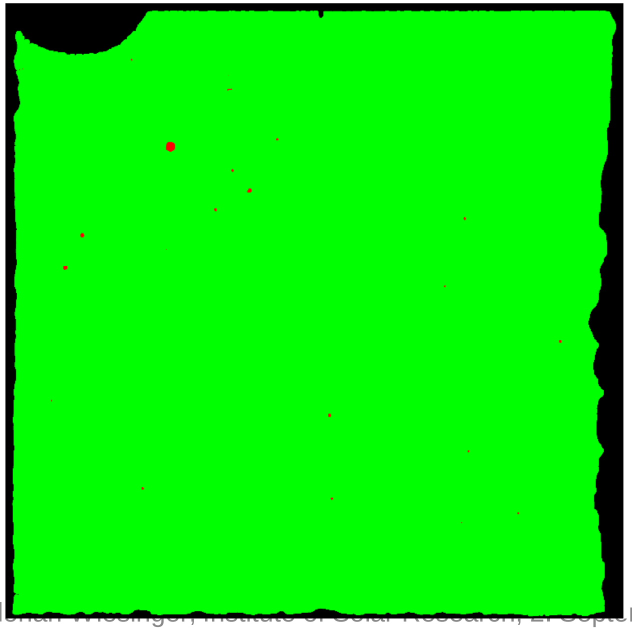

2
3
1

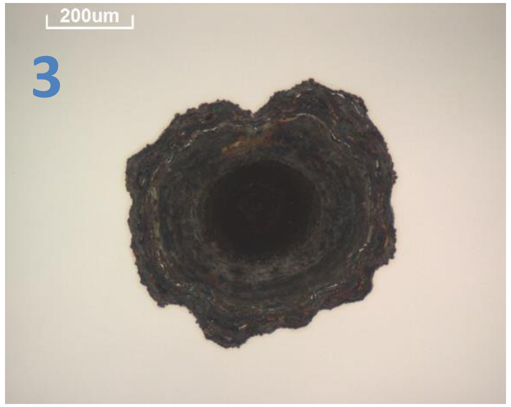

200um
1

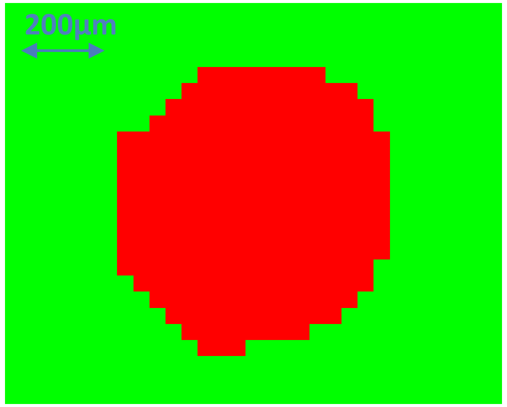

200µm


200um
2


200µm



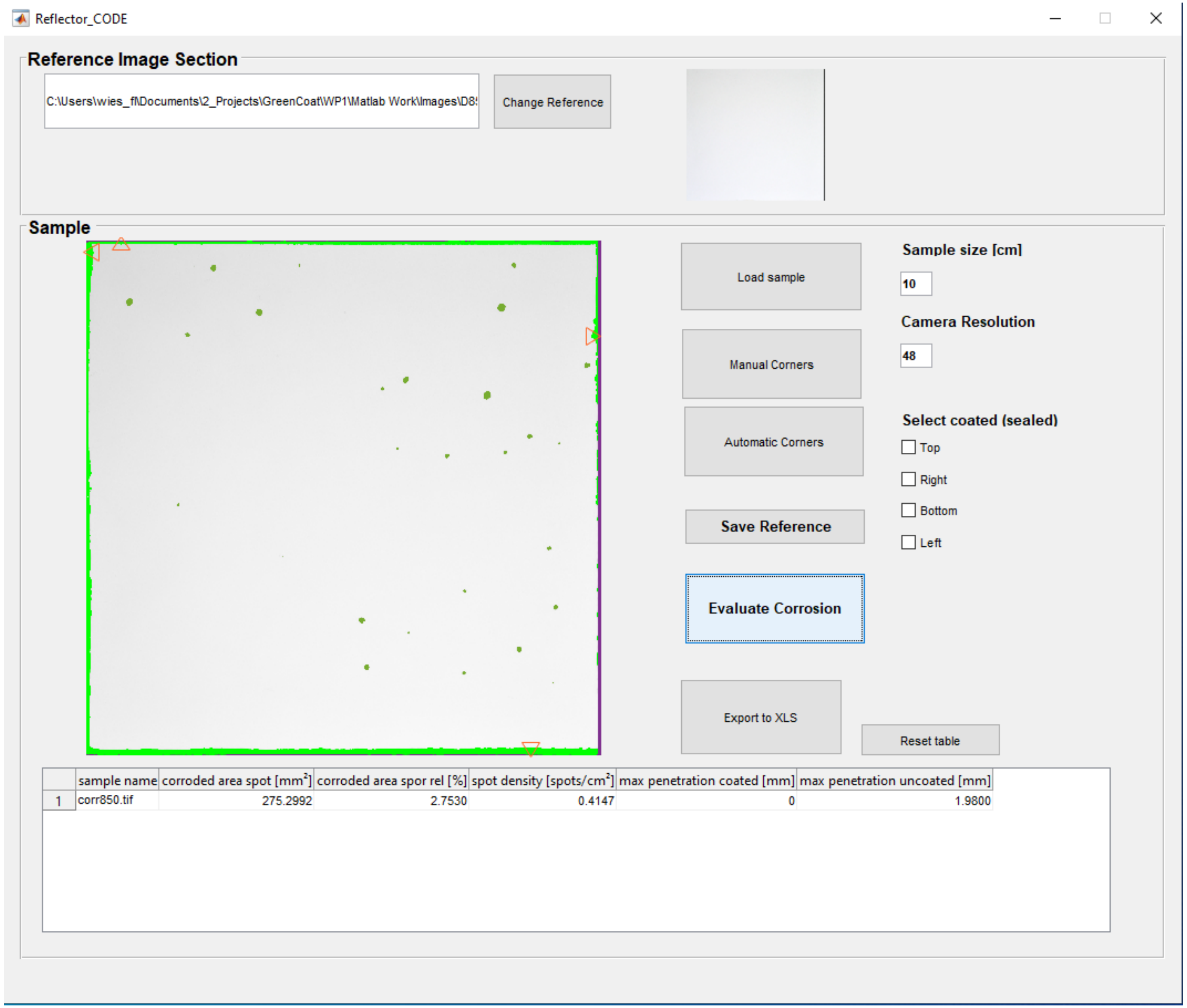

200um
3


200µm

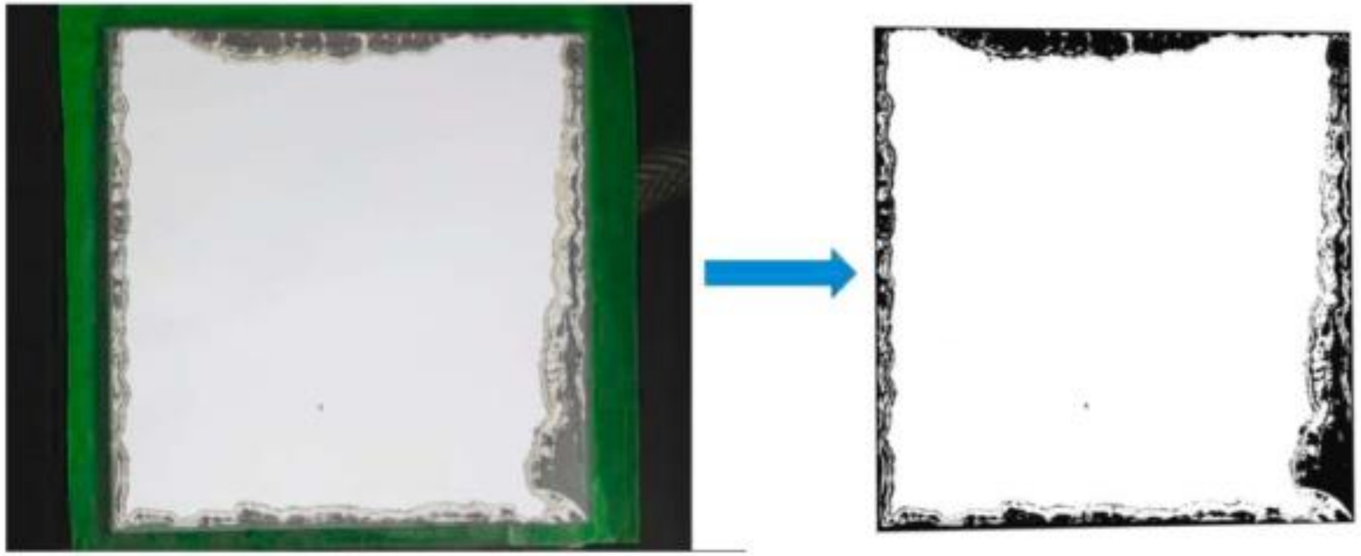
Accelerated aging – Corrosion detection

GUI:

- Simple selection of RE and corresponding AI
- Adapt basic parameters like sample size and cam res →should be automated as well.
- Selection of the coated edges.
- Marking of maximum edge penetration.
- Export important parameters in a table.



Corrosion Testing - modeling



Analysis via total reflectance: $$\rho = \rho_{NC} \cdot \frac{A_{NC}}{A_{Total}} + \rho_C \cdot \frac{A_C}{A_{Total}}$$

When ρ_C is considered to be negligible → $$\rho = \rho_{NC} \cdot \left(1 - \frac{A_C}{A_{Total}}\right)$$

[6]

Corrosion Testing - modeling

Further correlation approaches

$$\rho = \rho_{NC} \cdot \left(1 - \frac{A_C}{A_{Total}}\right)$$

$$\rho_{NC} = \rho_0 - a \cdot t_{outdoor}$$

Non corroded area shows linear degradation of reflectance, due to erosion, UV-degradation or soiling deposition

$$\frac{A_C}{A_{Total}} = 1 - e^{-b\left(\frac{t_{outdoor}}{a_f}\right)^c}$$

Correlation derived from CASS test. Taking into account the acceleration factor a_f

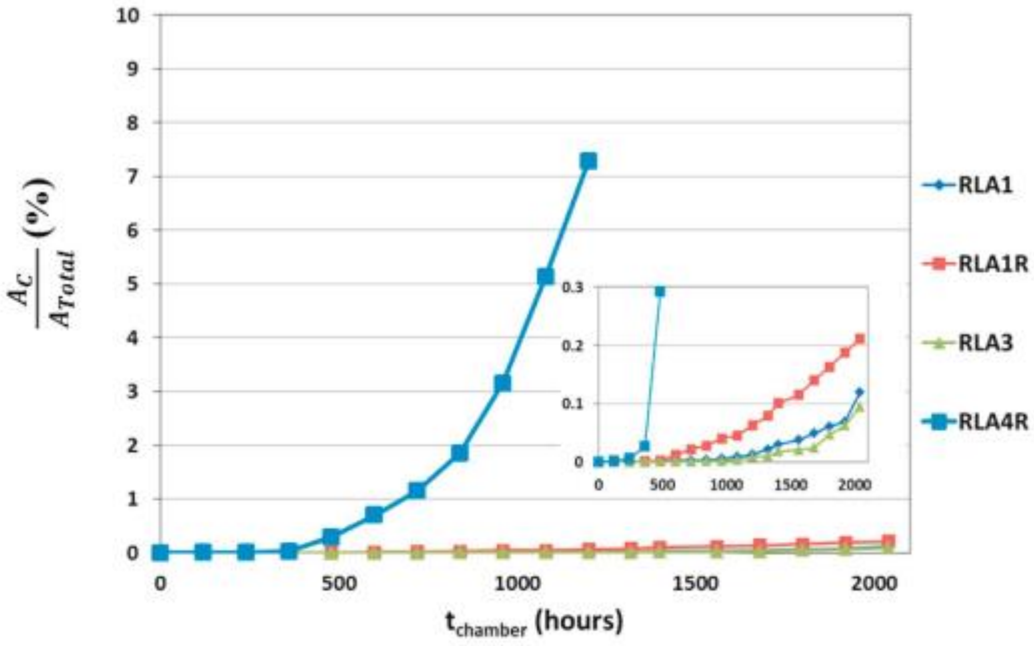
[6]

Corrosion Testing - modeling

Further correlation approaches

$$\rho = \rho_{NC} \cdot \left(1 - \frac{A_C}{A_{Total}}\right)$$

To find reasonable model: CASS test

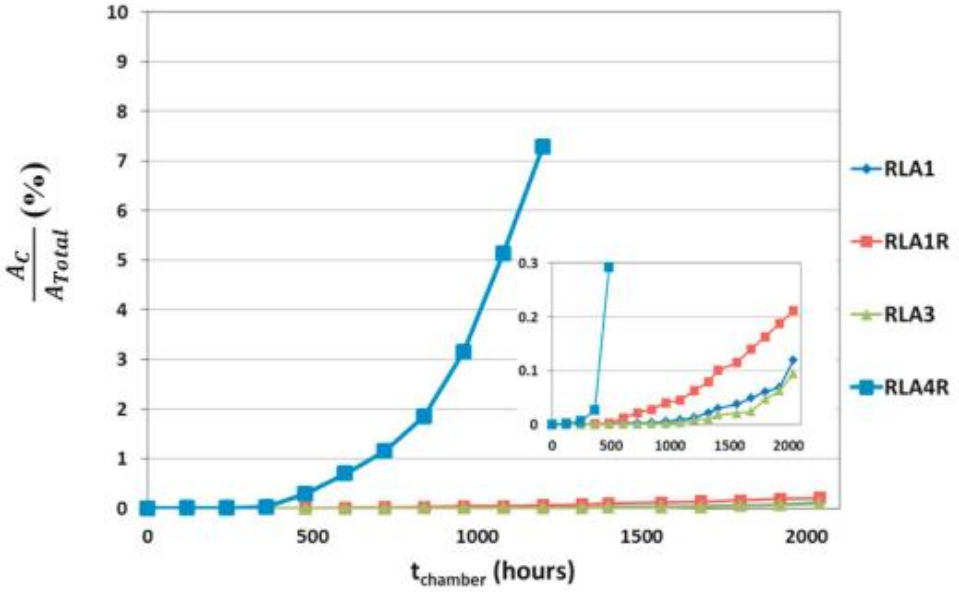


$$\frac{A_C}{A_{Total}} = 1 - e^{-b\left(\frac{t_{outdoor}}{a_f}\right)^c}$$

Acceleration factor a_f derived from comparison of CASS test with outdoor results.

[6]

Corrosion Testing - modeling



Find testing time in CASS to reproduce same corrosion area as after 3 years outdoor.

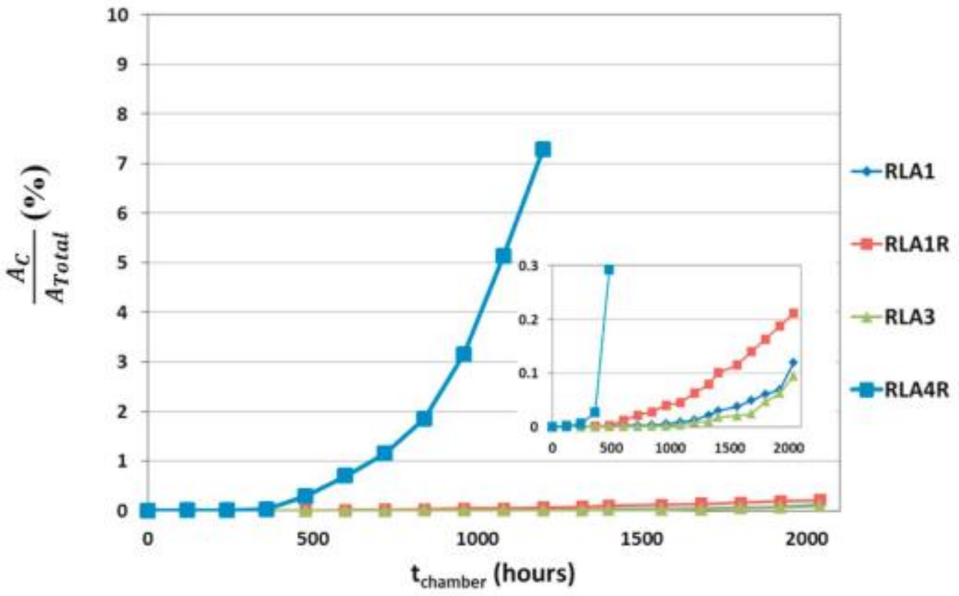
Table 4

Ratio of area corroded by corrosion spots $\left(\frac{A_C}{A_{Total}}\right)$ after approximately 3 years of outdoor exposure, depending on the material and site.

Material	Site (corrosivity class)		
	Almería (C4)	Tabernas (C3)	Atacama Desert (C3)
	$A_C/A_{Total}(-)$	$A_C/A_{Total}(-)$	$A_C/A_{Total}(-)$
RLA1	0.000001	0.000000	0.000000
RLA1R	0.000007	0.000002	0.000002
RLA3	0.000037	0.000004	0.000001
RLA4R	0.000210	0.000021	0.000055

[6]

Corrosion Testing - modeling



Acceleration factor a_f derived from comparison of CASS test with outdoor results.

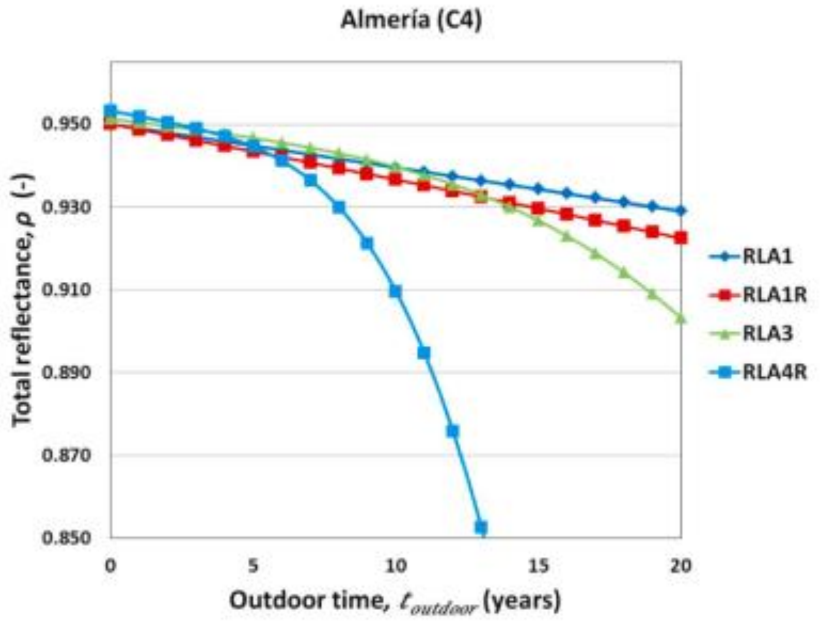
Material	Site (corrosivity class)					
	Almería (C4)		Tabernas (C3)		Atacama Desert (C3)	
	$t_{chamber}$ (h)	a_f (–)	$t_{chamber}$ (h)	a_f (–)	$t_{chamber}$ (h)	a_f (–)
RLA1	234	106	118	204	131	200
RLA1R	230	108	130	185	144	182
RLA3	935	27	493	48	331	79
RLA4R	280	88	157	153	201	131

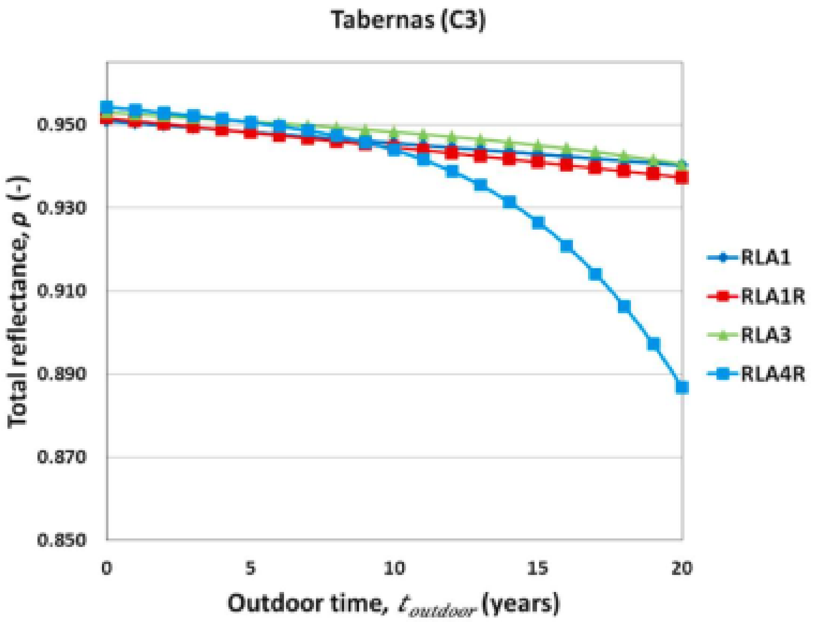
[6]

Corrosion Testing - modeling

Material	Site (corrosivity class)					
	Almería (C4)		Tabernas (C3)		Atacama Desert (C3)	
	$t_{chamber}$ (h)	a_f (−)	$t_{chamber}$ (h)	a_f (−)	$t_{chamber}$ (h)	a_f (−)
RLA1	234	106	118	204	131	200
RLA1R	230	108	130	185	144	182
RLA3	935	27	493	48	331	79
RLA4R	280	88	157	153	201	131

Using the acceleration factor a_f the lifetime of each reflector type and every site can be calculated.





[6]

Thank you for your attention

Contact: Florian.Wiesinger@dlr.de

References

[1] https://arm.ual.es/arm-group/facilities-and-infrastructures/plataforma-solar-de-almeria-2/.

[2] Torresol website/media, Gemasolar

[3] Meyen, Stephanie. "Parameters and method to evaluate the solar reflectance properties of reflector materials for concentrating solar power technology-Official SolarPACES reflectance guideline verstion 2.5." (2013).

[4] Meyen, Stephanie, Florian Sutter, and Peter Heller. "Near specular scatter analysis method with a new goniophotometer." *Reflection, Scattering, and Diffraction from Surfaces IV*. Vol. 9205. SPIE, 2014.

[5] J. Wette, F. Sutter, M. Tu, A. Fernandez-Garcia, F. Buendia, M. Carvalho, T. Diamantino: Advanced cyclic accelerated aging testing of solar reflector materials. AIP Conference Proceedings 2126, 160007 (2019); https://doi.org/10.1063/1.5117670

[6] Buendía-Martínez, Francisco, et al. "Lifetime prediction model of reflector materials for concentrating solar thermal energies in corrosive environments." *Solar Energy Materials and Solar Cells* 224 (2021): 110996.