Evaluation of VIPs after mild artificial aging during 10 years: focus on the core behavior

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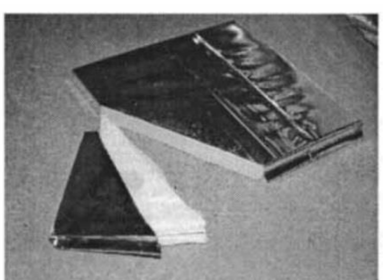
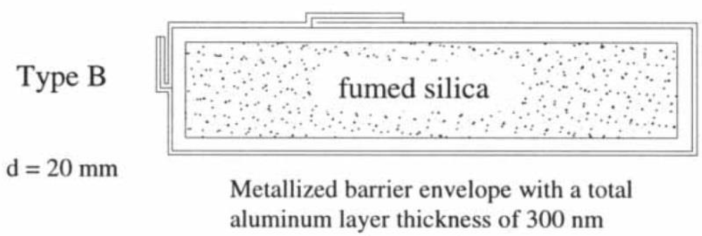




Metallized barrier envelope with a total aluminum layer thickness of 300 nm

This sample type was in Annex 39 as MF4 and as MF2 in Simmler and Brunner 2005
Source of Photo: Ghazi Wakili et al. 2004
The prediction in the Annex 39 were done with the motivation to be near the truth, but above, “the safe side”

Results on weight, pressure and conductivity increase over 10 years

The evolutions are relevant for the aging prediction and the related Service Life Prediction discussion.

Last 3 columns shown already IVIS2015 presentation of **R. Caps** and on Sept 2014 Annex 65 Kick-off in Grenoble

Table 1: weight gain, pressure increase in the VIP of 50 x 50 x 2 cm and thermal conductivity change on during aging (for each condition: average over 2 VIPs)

Sample #	Aging	Envelope area (m^2)	Δm (%)	Δp_{int} (mbar)	$\Delta\lambda_{cop}$ ($mW.m^{-1}.K^{-1}$)
12/13	3948 days at (23 °C, **33% RH**)	0.535	0.57	4.9	0.6
6/7 And samples # 3/4	3881 days at (23 °C, **80% RH**)	0.532	3.13	17.6	2.2

Can differences been "seen" already in the core structure? Hypothesis in 2013 **Hints…**
in Brunner and Ghazi Wakili, Vacuum, 100, 4–6, 2014

About the samples/cooperation

What can we determine about these cores?
Are differences detectable already? (or are 10 years too early?)

Message from conclusion of EDF : **Core has not been fully aged**

This I like to show you now with data revised regarding data in the paper.

6 samples given in April 2014 to EDF.
Additionally sample # 3/4 to start and improve the methods, before cutting # 6/7 and 11/12

-> ***Drying tests***
-> ***Water vapor sorption***
-> ***Nitrogen sorption (BET specific area)***

-> ***Evaluation of the laminate***

Discussion
-> ***Evaluation of the surface hydrophilicity***
-> ***Prediction of the conductivity evolution***

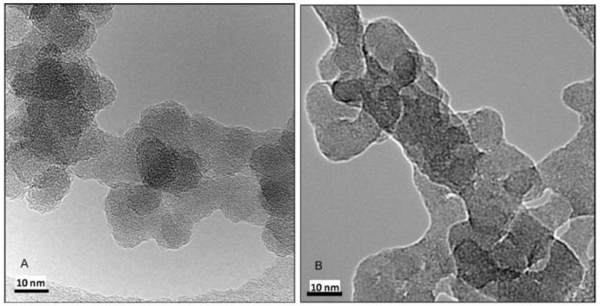


Fig. 9. HDK T30 microstructure observed with TEM before (A), and after 205 days ageing at 60 °C-80%*RH* (B).

As in Yrieix et al.. Energy and Buildings 85 617 -630, 2014 and Brunner and Ghazi Wakili, Vacuum, 100, 4–6, 2014

By the way, at 2003-2005 already the moisture dependence was not understood well enough. For this reason, as well as for the reason of the length of the paper, the 23 ℃ 80%rh data had been not part of the paper Simmler and Brunner 2005 the 80% data. Only in the other cited source.

λ_{cop} measured at EDF after some months of storage at 23 °C - 50 %RH

here are absolute values - table 1 are the deltas

Sample #	Dimensions (mm^2)	Ageing			λ_{cop} initial EMPA ($mW.m^{-1}.K^{-1}$)	λ_{cop} aged EMPA ($mW.m^{-1}.K^{-1}$)	λ_{cop} aged EDF ($mW.m^{-1}.K^{-1}$)
		Conditions	Date start	Date end			
12	500*500	23 °C, 33 %RH	19/05/2003	31/03/2014	3.9	4.5 (24/02/2014)	**4.8** (16/10/2014)
13	500*500	23 °C, 33 %RH	19/05/2003	31/03/2014			**4.8** (16/10/2014)
3	250*500	23 °C, 80 %RH	25/07/2003	31/03/2014	–	–	**7.0** (04/11/2014)
4	250*500	23 °C, 80 %RH	28/07/2003	31/03/2014	–	–	**7.0** (04/11/2014)
6	500*500	23 °C, 80 %RH	25/07/2003	31/03/2014	3.9	6.1 (21/02/2014)	**6.6** (17/10/2014)
7	500*500	23 °C, 80 %RH	25/07/2003	31/03/2014			**6.6** (28/10/2014)

and the pairs
at same conditions
are the same

Differences resulting from difference in instruments and to a small amount form time of measurement.

Drying tests on core

The weight loss **of the core dried under vacuum** 2 h at 140 ℃ and 200 ℃ (% of the wet initial mass).

Table 2 rev.: weight loss of the core by drying (assessment of the physisorbed water)

Sample #	Aging	Weight loss by drying (%)	
		at 140 ℃	at 200 ℃
12	10 years at (23 ℃, 33% RH)	0.97	1.16
3	10 years at (23 ℃, 80% RH)	2.30	**2.47**

More than weight gain

Compare with Table 1: weight gain

Sample #	Δm (%)
12/13	0.57
6/7	**3.13**

weight gain of whole VIP

But at such envelope without additional gluing tapes , no relevant effect seen yet.

sample 3 has width 25 cm,

while 6 and 7 have 50cm

I add 3%-w ice for

B = $\lambda_{Xw} = d\lambda / dXw$

since 2009
-> BEST3 or
-> Empa test reports

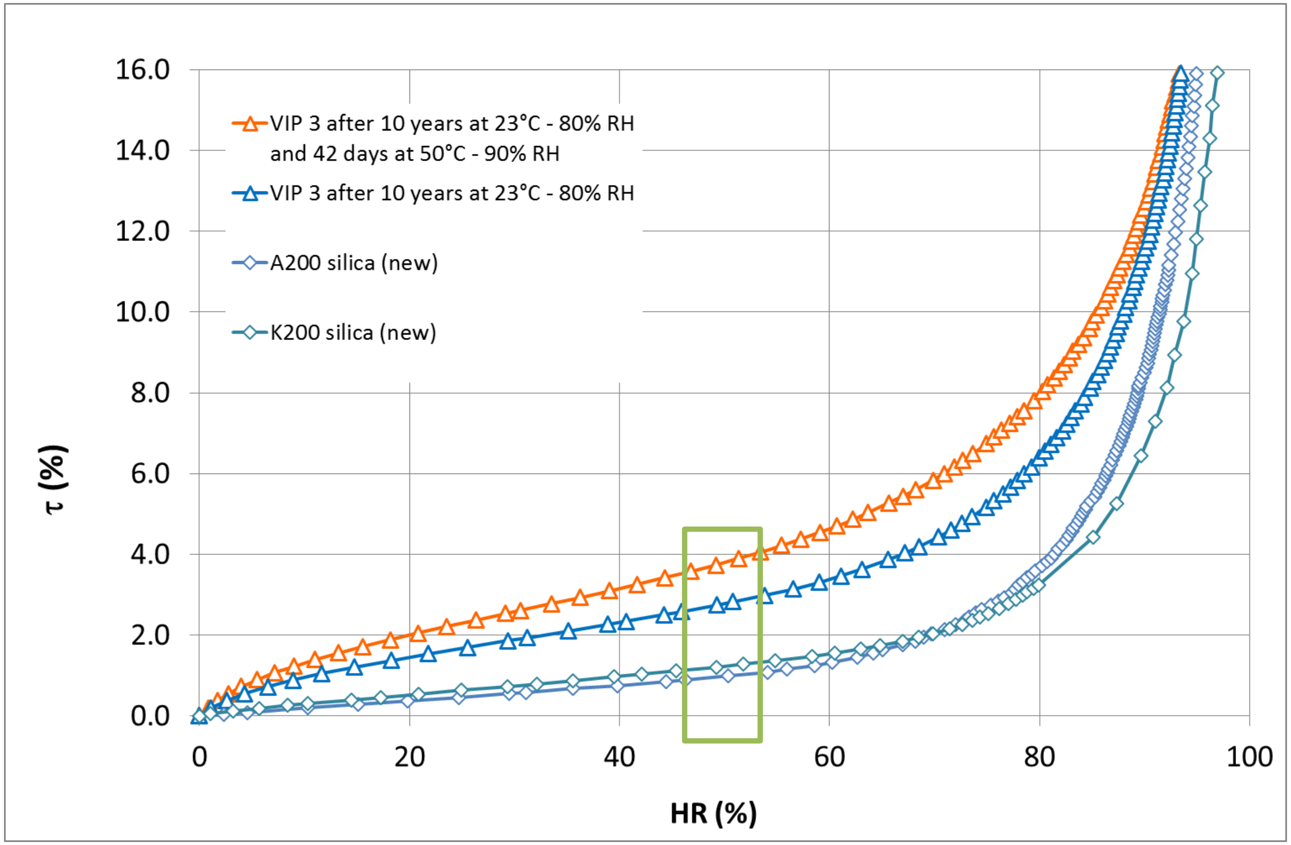
Less than expected

at slightly smaller sample commonly about the same moisture content or more

Water vapor isotherms at 25°C

Adsorption isotherms on formulated core

Preparation of the samples : vacuum drying at 140°C during 2 hours



Fast to values at 50%rh to be in 17 min

The initial isotherm is estimated based on new equivalent products

Results:
Water vapor sorption

Water vapor sorption is a method to detect changes of aging of a core material.

Table 3 rev: water content deducted from the adsorption isotherm

Sample #	Aging	$\tau_{ads@50\%\ RH}$ (%)
3	10 years at (23 ℃, 80% RH)	2.8
3	10 years at (23 ℃, 80% RH) + 1 month core at (50 ℃, 90% RH)	3.9

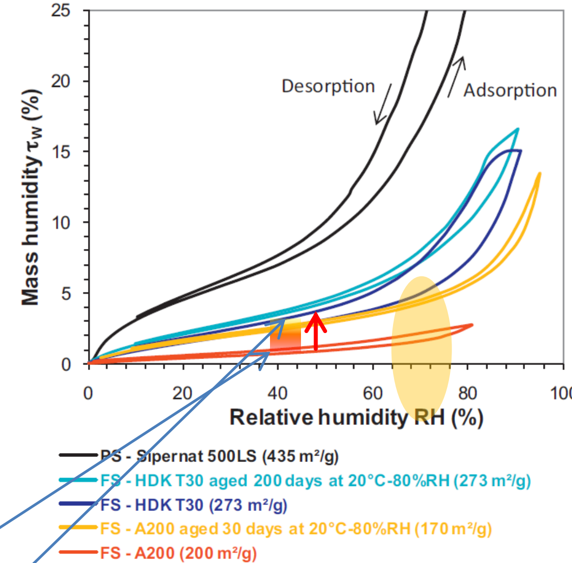


Fig. 4. Typical water sorption isotherms (recorded at 25 °C).

compare with
[Yrieix et al. 2014](#)
a similar fumed silica
in an

VIP core is able to age further age further, despite the VIP was 10 years 23 ℃ 80%

Check again with Bernard

Results: Nitrogen sorption: BET specific area evolution

Table 4: BET specific area (nitrogen measurements) of the core / estimation for the silica

Sample #	Aging		$A_{BET\ core}$ *measured* ($m^2.g^{-1}$)	$A_{BET\ silica}$ *calculated* ($m^2.g^{-1}$)
12	10 years at (23 °C, 33% RH)	difference to humid storage	219 - 220	235
3	10 years at (23 °C, 80% RH)		175 - 205	203
3	10 years at (23 °C, 80% RH) + 1 month core at (23 °C, 90% RH)		168	180

+7% of the averaged of measured to consider weight of fibres and opacified

Clear change with + 1month without envelope

Calculation of the surface hydrophilicity

$$\Psi_{ads} = \frac{\tau_{ads@50\%HR}}{A_{BET}}$$

water content at equilibrium at 50% RH (%) divided by the specific area (m2/g)

Table 3, 2nd line
Table4

Table 6 rev.: assessment of the surface hydrophilicity of the VIP core

Sample #	Aging	Ψ_{ads} ($\mu g.m^{-2}$)
3	VIP 10 years at (23 ℃, 80% RH)	147
Reference fumed silica	silica not aged	47
	silica, 30 days at (23 ℃, 80% RH)	170

VIP core seem not been fully aged, despite the VIP was 10 years 23 ℃ 80%

Calculations for the laminate

with results from table 1:

Is L1 in Brunner2006, Brunner2008
MF4 in Annex 39 and
MF2 in Simmler and Brunner 2005
Type B in Ghazi Wakili et al. 2004

Table 5 rev: Permeance of water vapor assessed from weight and
permeance air/gas from pressure increases over 10 years
(mean values over 2 VIPs)

To be compared with Annex 39 Subtask A (Table 28 on page 76)

Sample #	Aging	Π_{wv} (kg.m^{-2}.s^{-1}.Pa^{-1})	Π_a (kg.m^{-2}.s^{-1}.Pa^{-1})	Π_a (kg.m^{-2}.s^{-1}.Pa^{-1})
12/13	10 years at (23 ℃, 33% RH)	3.2E-14	1.5E-18	-
3/4 6/7	10 years at (23 ℃, 80% RH)	7.5E-14 * 7.4E-14	_ 5.2E-18	 1.2E-18
		Base on Table 1 weight gain data of whole VIP * width 25cm	Base on Table 1 **pressure increase data** (of whole VIP)	Base on estimation of the DRY air pressure inside the VIP $p_g = p_{dry\ air} = p_{int} - p_{wv}$

To compare e.g. with
Pons et al 2014, Energy and Buildings 85 (2014) 604–616

See big table 8 in the proceedings too with 8, resp. 7 different conditions

Calculation on the laminate and core

Simplified version $p_g = p_{int}$ in the simplified version, pressure inside the VIP)
A robust calculation and a prediction near the "safe side"

Table 7: calculated values by the model Eq. 4
and measured values of the thermal conductivity increase

$$\Delta\lambda = B \times \tau_W + G \times p_g$$

Sample	Aging	Δm (%)	Δp_{int} (mbar)	$\Delta\lambda_{calc}$ (mW.m^{-1}.K^{-1})	$\Delta\lambda_{meas}$
12	3948 days (23 ℃, 33% RH)	0.57	5.1	0.5	0.6
13		0.57	4.8	0.5	
6	3881 days (23 ℃, 80% RH)	3.16	17.8	2.2	2.2
7		3.11	17.5	2.2	

$$\Delta\lambda_{(t)} = B \times \tau_{W\infty} \times \left(1 - \exp\left(\frac{-t \times \Delta m_{(t)}}{\tau_{W\infty}}\right)\right) + G \times \Delta p_{g\,(t)} \times t \qquad \text{(Eq. 5)}$$

See big table 8 too.

Conclusions on core

The evaluation of VIPs after mild artificial aging during 10 years revealed that the silica **core has not been fully aged**, even at high relative humidity (80% RH).
highlighted by:

i) the **moisture content at equilibrium**, which is not so high as the moisture content that could be reached by short-term additional aging at higher humidity levels,

ii) the evolution of the **specific area**,

The water sorption isotherm indicates that the moisture content inside the VIPs, deducted from the weight increase, corresponds to a humidity level of 43 % and so a water vapor partial pressure of 13.6 mbar (at 23 °C). It corresponds to a decrease of the WVTR by a factor 2.

The parameters *B* and *G* used for the simplified model, previously determined with short term tests, appear valid for 10 years aging.

To go further, similar investigations of VIPs from real applications with changing boundary conditions should give clearer answers.

And the VIPs can even be dried, if the environmental conditions change, as it was confirmed by the weight loss of the VIPs over the last year, where they were placed at (23°C, 50% RH).

Calculation on the laminate and core

More complex, detailed model

$p_g = \ldots$ ($p_g = p_{int}$ in the simplified version, internal pressure inside the VIP)

$p_g = p_{dry\ air} = p_{int} - p_{wv}$ in the more detailed version

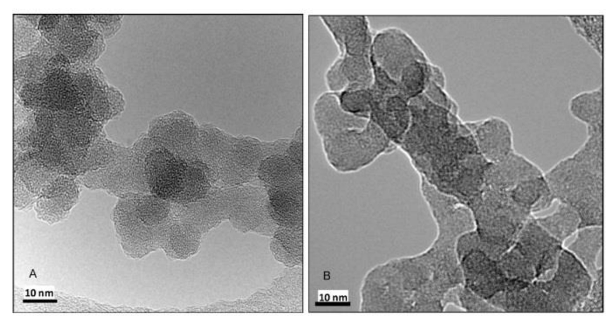
as used in the papers Simmler and Brunner 2005 and Brunner and Simmler 2008

and also $p_{dry\ air}$ in Schwab et al. 2005 and based on this Wegger et al 2011, resp. P. Johansson et al. 2014

Table 8:

Sample #	Aging	Size (cm³)	$\Delta m/\Delta t$ (%.y⁻¹)	$\Delta p_{int}/\Delta t$ (mbar.y⁻¹)	Π_{WV} (kg.m⁻².s⁻¹.Pa⁻¹)	Π_g (kg.m⁻².s⁻¹.Pa⁻¹)	$\Delta\lambda_{cop}$ forecasted Eq.4 (mW.m⁻¹.K⁻¹)	$\Delta\lambda_{cop}$ forecasted Eq.5 (mW.m⁻¹.K⁻¹)	$\Delta\lambda_{cop}$ measured (mW.m⁻¹.K⁻¹)
12/13	3948 days at (23 ℃, 33% RH)	50x50x2	0.05	0.46	3.2E-14	1.5E-18	-	-	0.60
[3], [9]	180 days at (23 ℃, 50% RH)	25x25x2	0.16	1.4	6.0E-14	4.0E-18	-	-	-
		25x50x2	0.13	1.3	5.1E-14	3.8E-18			
		50x50x2	0.12	1.0	4.9E-14	3.0E-18	-	-	-
3/4	222 days at (23 ℃, 80% RH) [9]	25x50x2	0.22	1.1	5.6E-14	3.4E-18	-	-	-
	4115 days at (23 ℃, 80% RH)		0.30	-	7.5E-14	-	-	-	-
6/7	236 days at (23 ℃, 80% RH) [9]	50x50 x2	0.21	1.3	5.3E-14	5.1E-18			
	3881 days at (23 ℃, 80% RH)		0.30		7.4E-14	5.2E-18	1.59	1.45	2.20

further work needed
to compare with
Yrieix et al. ENB, 2014
left pristine



End

And my general message

"Lambda measured" is not the lambda of "a VIP".
It is only the Lambda – center of panel value
There is all times
thermal bridging effects at the edges of
vacuum-insulation-panels (VIP)

Answer 1
to Question on corrosion on Präsentation Jelle.

FIB prepared SEM- Images of the laminate of this IVIS2015 presentatition are L1 Type in
Brunner et al 2008, Surface & Coatings Technology 202 (2008) 6054–6063
(see also with Brunneret al 2006 Surface & Coatings Technology 200 (2006) 5908–5914,)

And Brussel 2012 (?)
Nominally the same laminate also in Brunner and Simmler 2008
Brunner2014 (got published shortly before last IVIS (you might remember)
Newer, improved laminate generation nowadays, like visible in the Jelle2015IVIS related full paper version.

And my message

"Lambda measured" is not the lambda of "a VIP",
It is only the Lambda – center of panel value
There is all times
thermal bridging effects at the edges of
vacuum-insulation-panels (VIP)

Answer 1
to Question on corrosion on Präsentation Jelle.

FIB prepared SEM- Images

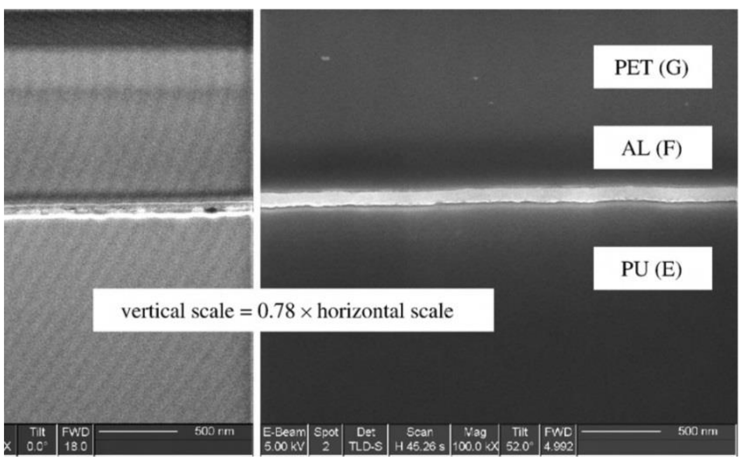
of another laminate L2 Type in at 65°C 75%rh 1.5 year

[Brunner et al 2008, Surface & Coatings Technology 202 (2008) 6054–6063]

And Brussel 2012 in t[he booklet] related to the International Symposium Superinsulating materials Brussels, Belgium, 26 April 2012

see also [Brunneret al 2006 Surface & Coatings Technology 200 (2006) 5908–5914],

S. Brunner et al. / Surface & Coatings Technology 202 (2008) 6054–6063



ayers (100 nm) in I-beam imaging (left) and in E-beam imaging (right). The latter is identical to the common

L2 Type with 60 nm alu.
(resp. 30nm)

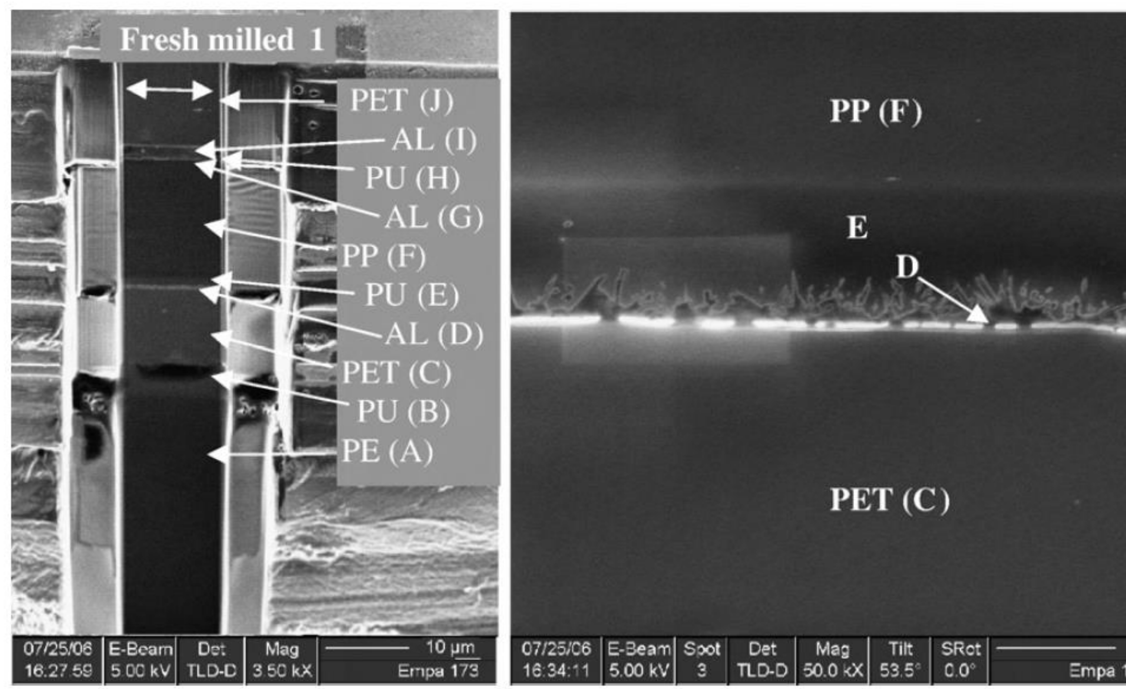


Fig. 13. Moderately aged L2 laminate with a fresh milled part (left). Layers I, H and G are visible again (compared to Fig. 11 right). The alum shows a lot of disruptions (right). The PU layer E shows a coral shaped structure.

Answer2

to Question on corrosion on presentation Jelle.

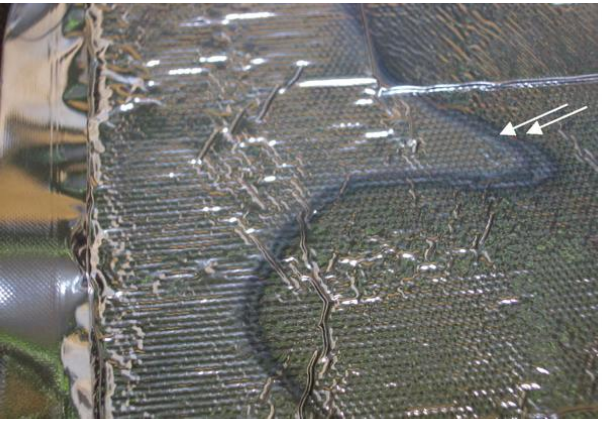
FIB prepared SEM- Images

of another laminate L2 Type in at 80°C 80%rh cyclic

Brunner et al 2008, Surface & Coatings Technology 202 (2008) 6054–6063

And Brussel 2012 in the booklet related to the International Symposium Superinsulating materials Brussels, Belgium, 26 April 2012

see also Brunneret al 2006 Surface & Coatings Technology 200 (2006) 5908–5914,



a rigorous regime of alternating conditions between
θ=80 °C, RH=80%, t=8 h and θ=25 °C, RH=50%, t=4 h was chosen,

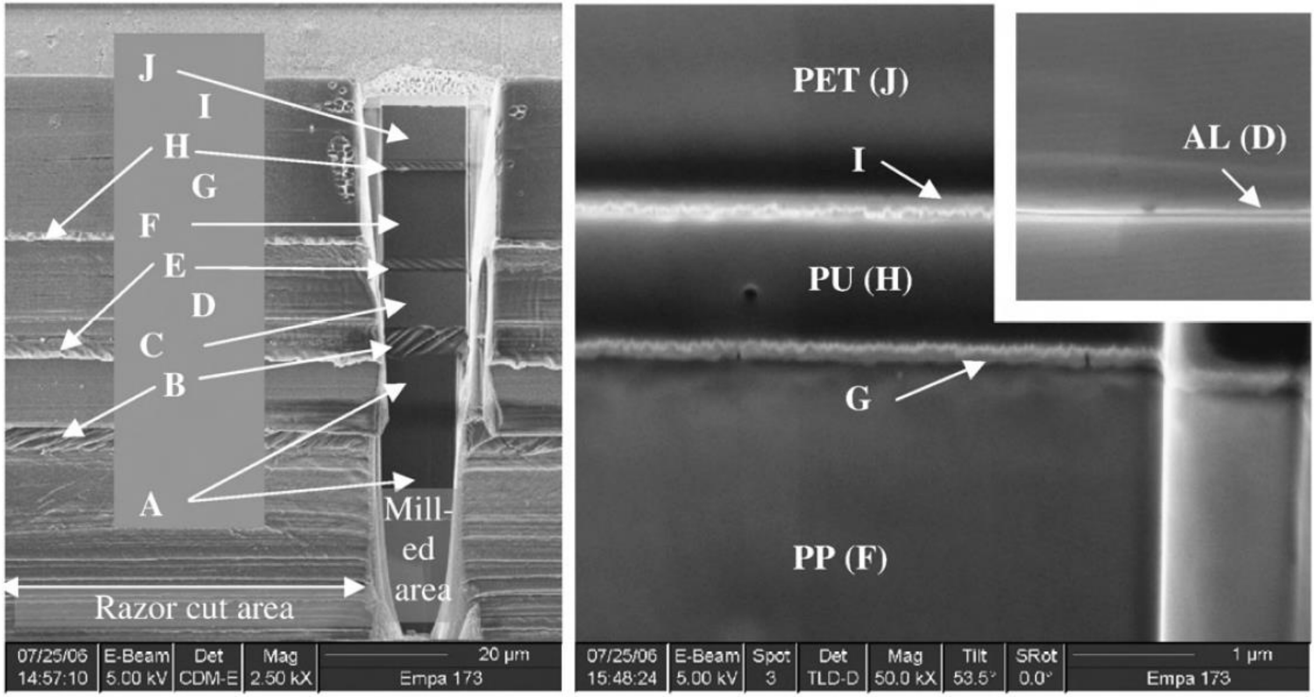


Fig. 18. Laminate L2 after rigorous ageing for 26 days, a condition where aluminium easily oxidizes. Image taken 4 weeks after milling (left). Detail of a with the oxidized layers I and G (right) and the unaffected layer D (inlay right).

Version for discussions –in Annex or small groups

Drying tests on core

The weight loss of the core dried 2 h at 140 and 200 ℃ under vacuum (% of the wet initial mass).

Table 2 rev.: weight loss of the core by drying (assessment of the physisorbed water)

Sample #	Aging	Weight loss by drying (%) at 140 ℃	Weight loss by drying (%) at 200 ℃	
12	10 years at (23 ℃, 33% RH)	0.97	1.16	More than weight gain
3	10 years at (23 ℃, 80% RH)	2.30	2.47	Less than expected

sample 3 has width 25 cm,

while 6 and 7 have 50cm

Difference 3.13-2.47 is understand as chemisorped water

at slightly smaller sample commonly about the same moisture content or more

Compare with Table 1: weight gain

Sample #	Δm (%)
12/13	0.57
6/7	3.13

weight gain of whole VIP

But at such envelope without additional gluing tapes , no relevant effect seen yet.

I add 3% ice for

$\lambda_{Xw} = d\lambda / dXw$

since 2009

-> BEST3 or

-> Empa test reports

Results on weight, pressure and conductivity increase over 10 years

The evolutions is relevant for the aging prediction and the related Service Life Prediction discussion.

Last 3 columns shown already IVIS2015 presentation of **R. Caps** and on Sept 2014 Annex 65 Kick-off in Grenoble

Table 1: weight gain, pressure increase in the VIP of 50 x 50 x 2 cm and thermal conductivity change on during aging (for each condition: average over 2 VIPs)

Slide 2 version full paper

Sample nr.	**Aging**	**Envelope area (m^2)**	**Δm (%)**	**Δp_{int} (mbar)**	**$\Delta\lambda$ ($mW.m^{-1}.K^{-1}$)**
12/13	3948 days at (23 ℃, **33% RH**)	0.535	0.566 0.57 0.573	0.94 6.02 4.9 0.99 5.80	0.6
6/7	3881 days at (23 ℃, **80% RH**)	0.532	3.16 3.13 3.11	0.86 18.62 17.6 0.89 18.39	2.2

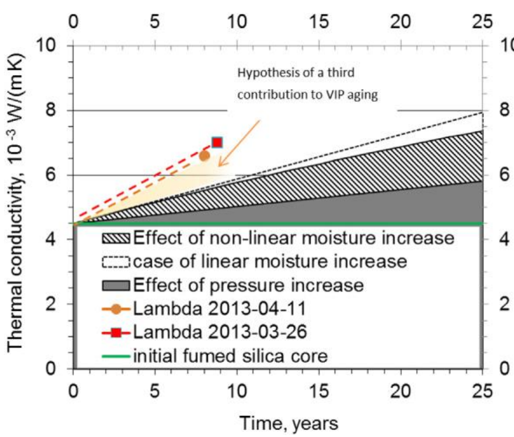
Can differences been ”seen” already in the core structure?
Hypothesis in 2013 Hints… ~0.8%, 18mbar
on VIP with nominal same laminate (slightly different core, different sealing type)

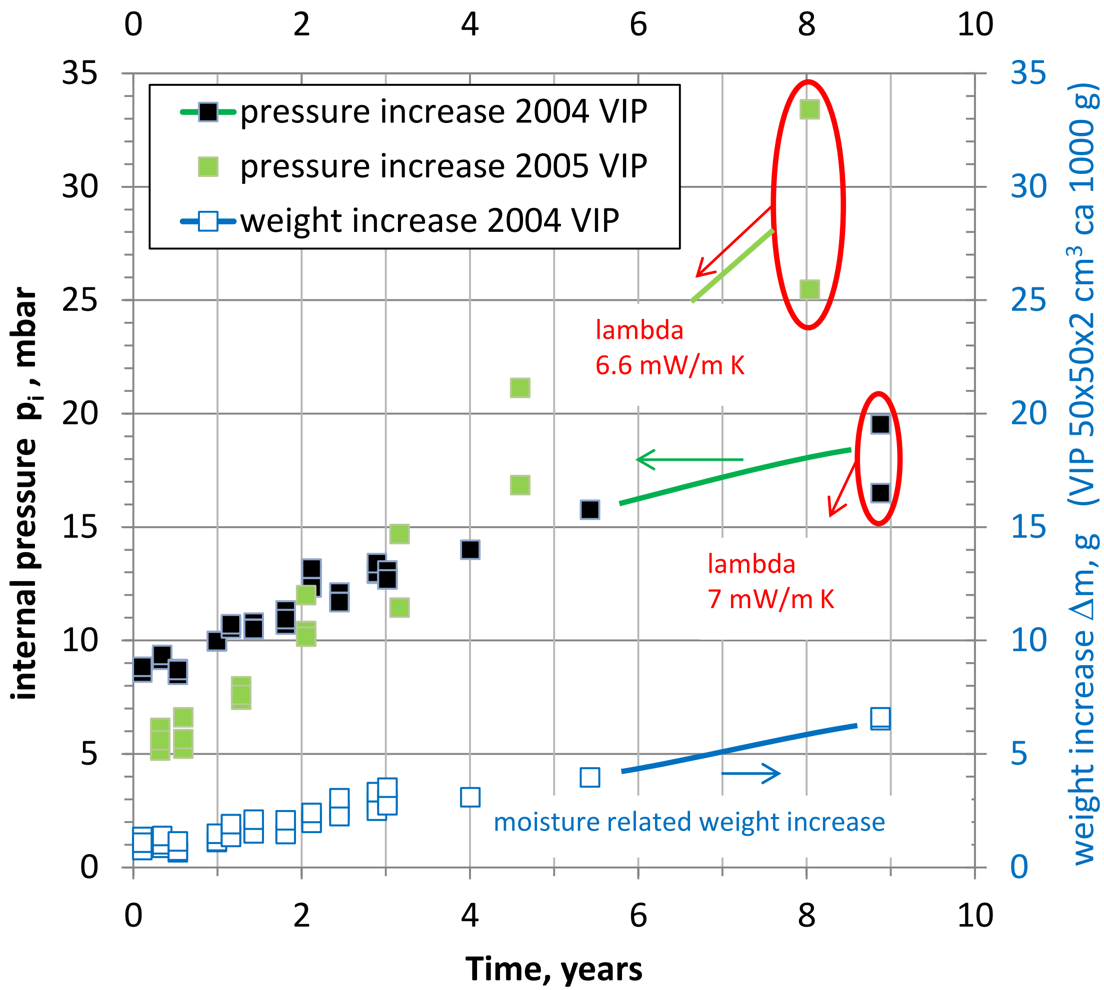
2013 PAPER IN VACUUM

100TH VOLUME

SPECIAL EDITION

2013 roof opening data:





Paper: [Brunner, et al. Vacuum, 2014:100:4–6]

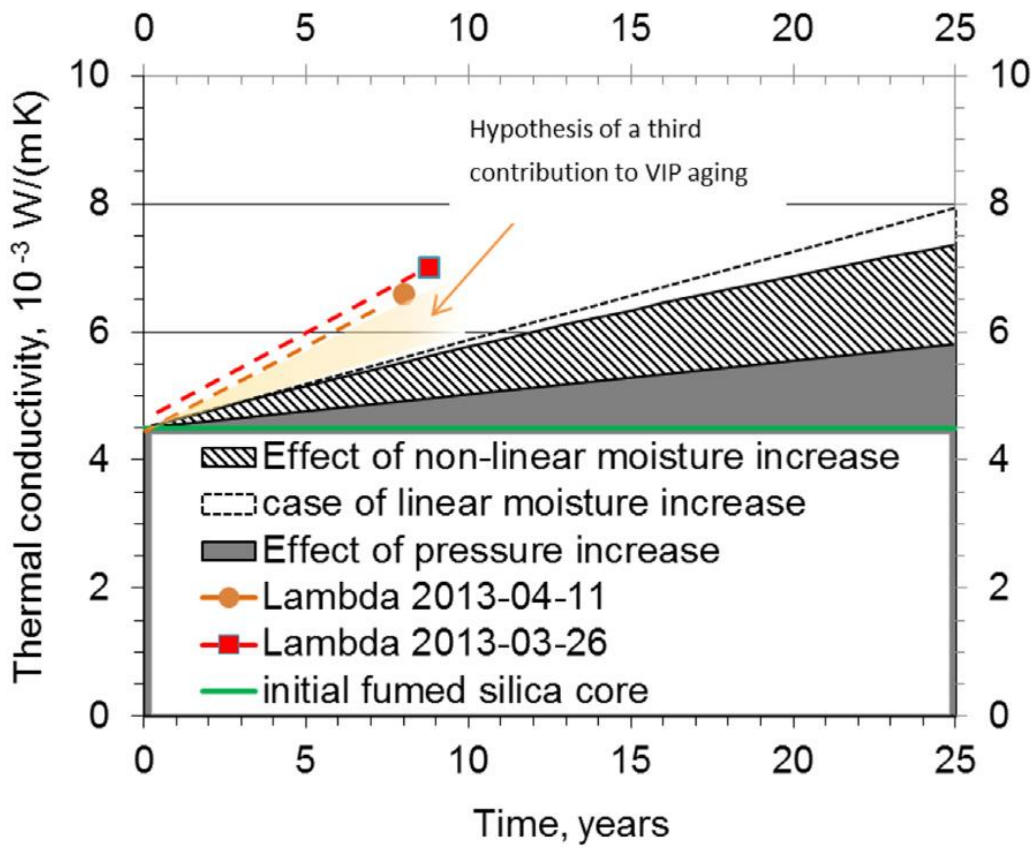
Conclusions- from former IVIS2007

25-year-extrapolation (from 2007):

– Cold and dry environment: $\Delta\lambda < 0.5 \cdot 10^{-3}$ W/(m K)

– Roof (Façade?) applications: $\Delta\lambda \rightarrow 3 \cdot 10^{-3}$ W/(m K)

2013 roof data:



Coloured and B&W part of graph
Paper: [Brunner, et al. Vacuum, 2014:100:4–6]

B&W part of graph from Paper [Brunner and Simmler Vacuum 82 (2008) 700–707] where measured temperatures got used, Simmler and Brunner 2005 was with simulated temperatures. Both with 80%rh pi/a and mass-%/a. In 2005 for MF1, in 2008 for MF2, as this laminate got used by the VIP producer from 2004 on.

Version for discussions –in Annex or small groups

Results: Nitrogen sorption: evolution of the BET specific area

Table 4: BET specific area (nitrogen measurements) of the core / estimation for the silica

Sample	**Aging**		$A_{BET\ core}$ *measured* **(m².g⁻¹)**	$A_{BET\ silica}$ *calculated* **(m².g⁻¹)**
12	10 years at (23 °C, 33% RH)	difference to humid storage	219 - 220	235
3	10 years at (23 °C, 80% RH)		175 - 205	203
3	10 years at (23 °C, 80% RH) + 1 month at (23 °C, 90% RH)		168	180

Clear change with + 1month without envelope

Relative comparison with Table 2: weight loss

Sample	**Aging**	**Weight loss by drying (%)** at 140 °C
12	10 years at (23 °C, 33% RH)	0.85
3	10 years at (23 °C, 80% RH)	2.30

Big difference to humid / dry storage

Calculations for the laminate

with results from table 1:

Is L1 in Brunner2006, Brunner2008
MF4 in Annex 39 and
MF2 in Simmler and Brunner 2005
Type B in Ghazi Wakili et al. 2004

Table 5 rev: Permeance of water vapor assessed from weight and
permeance air/gas from pressure increases over 10 years
(mean values over 2 VIPs)

Sample	Aging	Π_{wv} (kg.m^{-2}.s^{-1}.Pa^{-1}) *((g.m^{-2}.d^{-1}) 50%HR)*	Π_{a} (kg.m^{-2}.s^{-1}.Pa^{-1}) *(cm^3.m^{-2}.d^{-1})*
12/13	10 years at (23 ℃, 33% RH)	**3.2E-14** *0.0039*	**1.5E-18** *0.011*
3/4 6/7	10 years at (23 ℃, 80% RH)	**7.5E-14 *** **7.4E-14** *0.0090*	– **5.2E-18** *0.039*

Base on Table 1 weight gain data of whole VIP

* width 25cm

Base on Table 1 pressure increase data (of whole VIP)

See big table 8 in the proceedings too with 8, resp. 7 different conditions

to be compared with Annex 39 Subtask A (Table 28 on page 76)

Table 28: Perimeter (P) and area (A) related permeation data for barriers MF3 and MF4 at 23°C, 50% RH. The yearly rates for the format 1.0 x 0.6 x 0.02 m³ are calculated values. "for 2 x A" stands for the permeation through the whole panel surface.

		MF3	MF4
WVTR (for 2 x A)	[g/(m² d)]	0.0075	0.0069
$WVTR_A$	[g/(m² d)]	0.0030	0.0048
$WVTR_L$	[g/(m d)]	0.0008	0.0006
ATR (for 2 x A)	[cm³/(m² d)]	0.0660	0.0171
ATR_A	[cm³/(m² d)]	0.0034	0.0087
ATR_L	[cm³/(m d)]	0.0090	0.0018
Yearly rates (1.0 x 0.6 x 0.02 m³)			
$X_{w,A}$	[%-mass/yr]	0.050	0.080
$X_{w,L}$	[%-mass/yr]	0.033	0.027
$X_{w,total}$	[%-mass/yr]	0.083	0.107
dp/dt_A	[mbar/yr]	0.12	0.32
dp/dt_L	[mbar/yr]	0.87	0.17
dp/dt_{total}	[mbar/yr]	1.00	0.49

ATR_A

ATR is for dry air/gas from pressure increases over 6 month and extrapolated to bigger sample size

B. Yrieix et al. / Energy and Buildings 85 (2014) 617–630 625

Table 4
VIP mass variations, specific area A_{BET}, mass humidity τ_W measured in study 2 (measurements on pure silica FS_x are presented for comparison).

Material	Treatment	VIP mass variation (%)	A_{BET} (m^2/g)	τ_W (% at 25 °C 50%*RH*)
Core 1	VIP aged 70 °C 90%*RH*, 540 days	+5.0	134	2.24
Core 2	VIP aged 70 °C 90%*RH*, 400 days	+5.3	155	2.41
Core 3	VIP aged 50 °C 90%*RH*, 540 days	+5.0	142	2.23
FS_x	Initial state	–	200	0.94
FS_x	23 °C-80%*RH*, 30 days	–	170	2.89

Water vapor sorption is a method to detect changes of aging of a core material.

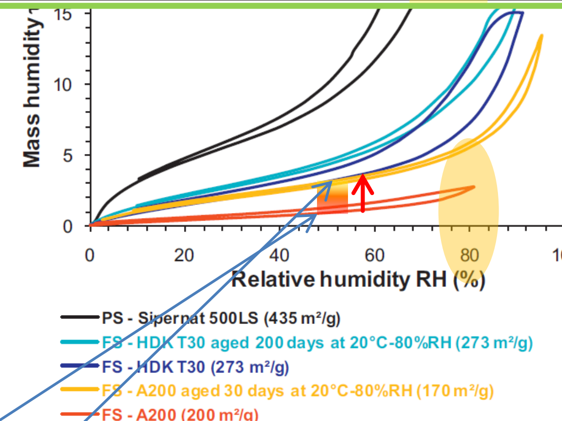


Fig. 4. Typical water sorption isotherms (recorded at 25 °C).

Table 3: water content deducted from the adsorption isotherm

Sample #	Aging	$\tau_{ads@50\%\ RH}$ (%)
3	10 years at (23 ℃, 80% RH)	0.9
3	10 years at (23 ℃, 80% RH) + 42 days core at (23 ℃, 90% RH)	2.3
3	10 years at (23 ℃, 80% RH) + 1 month core at (50 ℃, 90% RH)	3.9

Old, as in paper

compare with
Yrieix et al. 2014
a similar fumed silica
in an

VIP core seem unaged, despite the VIP was 10 years 23 ℃ 80%
(in this testing method)

Old version as in paper

Conclusions on core

The evaluation of VIPs after mild artificial aging during 10 years revealed that the silica **core has not been or slightly aged**, even at high relative humidity (80% RH). highlighted by:

i) the **moisture content at equilibrium**, which is far from the moisture content that could be reached by short-term additional aging at higher humidity levels,

ii) the evolution of the **specific area**, and

iii) the validity of the parameters *B* and *G* for the simplified model.

On the other hand, the water sorption isotherm indicates that the moisture content inside the VIPs, deducted from the weight increase, corresponds to a humidity level over 80%. So the water ingress must have been stopped (driving force for water permeation), if the value is near 80%.
And the VIPs can even be dried, if the environmental conditions change, as it was confirmed by the weight loss of the VIPs over the last year, where they were placed at (23°C, 50% RH).

In parallel, although the stationary conditions have avoided higher relative humidity periods, the high current water content reached inside the VIP (corresponding to 80% RH at 25 °C) can now strongly activate the core aging of the silica.

Old version as in paper

Conclusions on core (part2)

… In parallel, although the stationary conditions have avoided higher relative humidity periods, the high current water content reached inside the VIP (corresponding to 25 °C 80%rh) can now strongly activate the core aging of the silica.

Indeed, it is **now just in the capillary condensation domain** and any temperature decrease will lead to micro condensation and maybe saturation and therefore to a strong aging of the core itself.

As the main lever to age the silica is the humidity, one can deduct that the VIP aging is divided in two steps:
1st : no aging of the silica, because of too dry conditions inside, and
2nd: : where the silica ages because of the high humidity inside.
For the studied samples the results above lead to estimate the **duration of the 1st period at just under ten years.**

To go further, similar investigations of VIPs from real applications with changing boundary conditions should give clearer answers.

compare with
Yrieix et al. ENB, 2014
left (pristine) represent 1st step

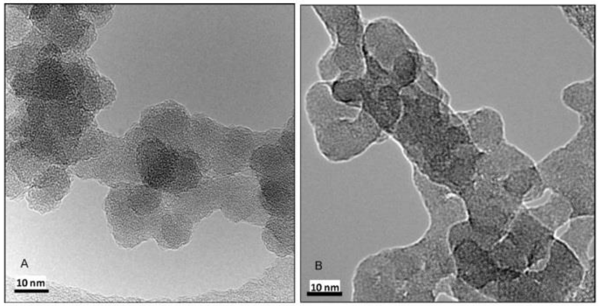


Fig. 9. HDK T30 microstructure observed with TEM before (A), and after 205 days ageing at 60°C-80%RH (B).