

# Microscopic Investigation of Laminates for barriers of **V**acuum **I**nsulation **P**anels

presented by

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# Structure of the two investigated laminates for the barrier envelopes of VIP's

## Laminate L1 92 $\mu\text{m}$

<b>J</b>	PET	Polyethylene terephthalate	12 $\mu\text{m}$
<b>I</b>	AL	Aluminium	100 nm = 0.1 $\mu\text{m}$
<b>H</b>	PU	Polyurethane	2 $\mu\text{m}$
<b>G</b>	PET	Polyethylene terephthalate	12 $\mu\text{m}$
<b>F</b>	AL	Aluminium	100 nm = 0.1 $\mu\text{m}$
<b>E</b>	PU	Polyurethane	2 $\mu\text{m}$
<b>D</b>	PET	Polyethylene terephthalate	12 $\mu\text{m}$
<b>C</b>	AL	Aluminium	100 nm = 0.1 $\mu\text{m}$
<b>B</b>	PU	Polyurethane	2 $\mu\text{m}$
<b>A</b>	LDPE	Low Density Polyethylene	50 $\mu\text{m}$

Vacuum side

## Laminate L2 108 $\mu\text{m}$

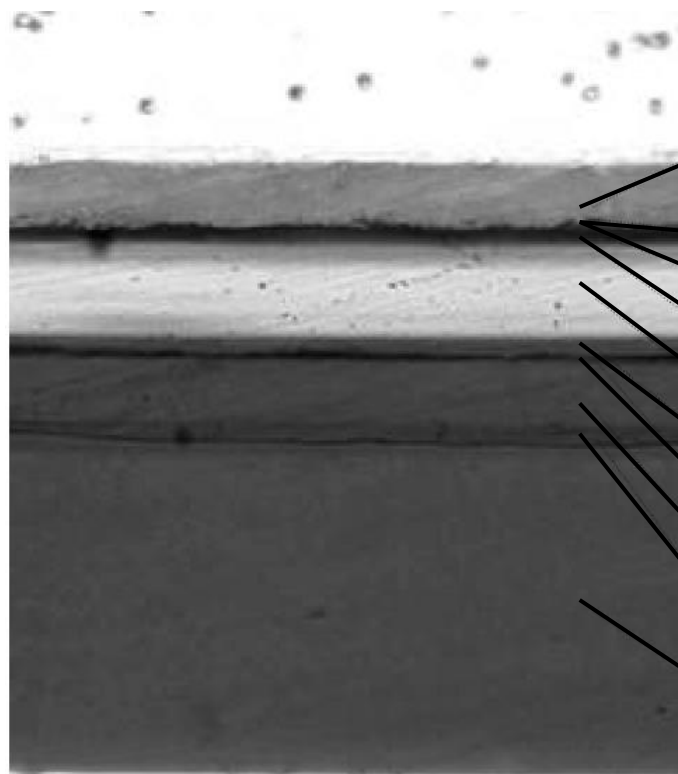
<b>J</b>	PET	Polyethylene terephthalate	12 $\mu\text{m}$
<b>I</b>	AL	Aluminium	60 nm = 0.06 $\mu\text{m}$
<b>H</b>	PU	Polyurethane	2 $\mu\text{m}$
<b>G</b>	AL	Aluminium	60 nm = 0.06 $\mu\text{m}$
<b>F</b>	PP	Polypropylene	18 $\mu\text{m}$
<b>E</b>	PU	Polyurethane	2 $\mu\text{m}$
<b>D</b>	AL	Aluminium	60 nm = 0.06 $\mu\text{m}$
<b>C</b>	PET	Polyethylene terephthalate	12 $\mu\text{m}$
<b>B</b>	PU	Polyurethane	2 $\mu\text{m}$
<b>A</b>	LDPE	Low Density Polyethylene	60 $\mu\text{m}$

Vacuum side

(Not scaled)

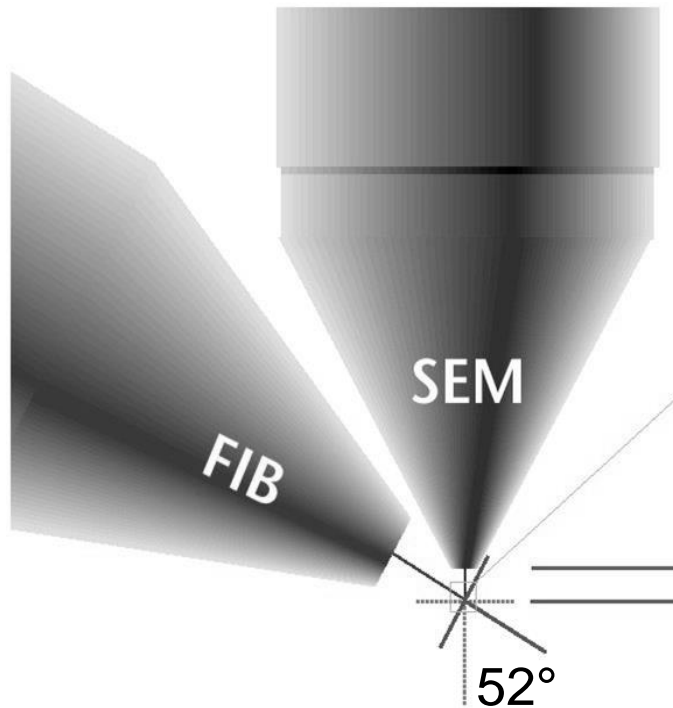
# Laminate seen by optical microscopy (scaled)

Laminate L2 - low visibility of aluminium layers

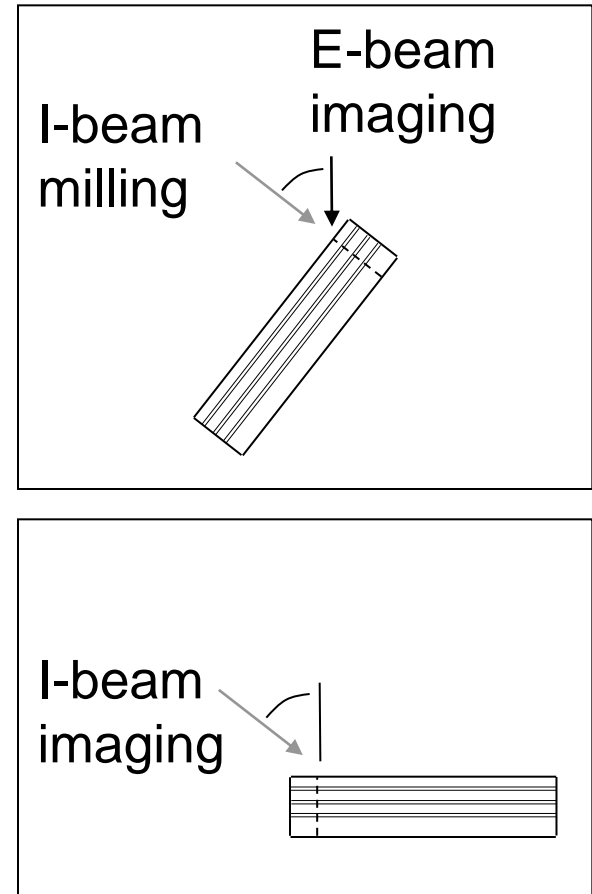


Layer #	Material	Function	Thickness
1	PET	Protecting layer (substrate for 2)	12 $\mu\text{m}$
2	ALU	Barrier layer	30 nm
3	PUR	Glue	2 $\mu\text{m}$
4	ALU	Barrier layer	30 nm
5	PP	Substrate for 4	18 $\mu\text{m}$
6	PUR	Glue	2 $\mu\text{m}$
7	ALU	Barrier layer	30 nm
8	PET	Substrate for 7	12 $\mu\text{m}$
9	PUR	Glue	2 $\mu\text{m}$
10	PE-LD	Sealing layer	60 $\mu\text{m}$

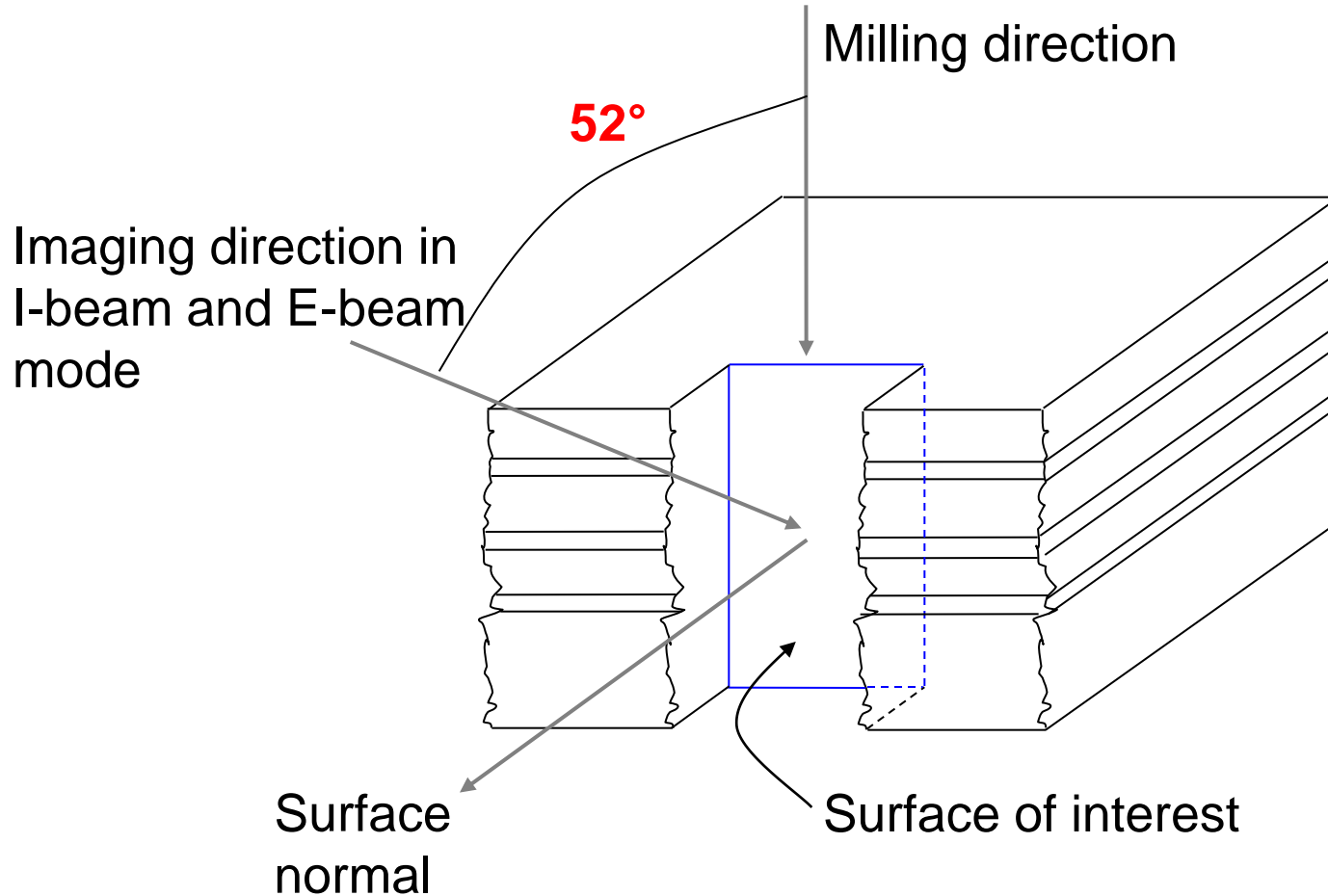
# Schematic representation of the Focused Ion Beam method (FIB)



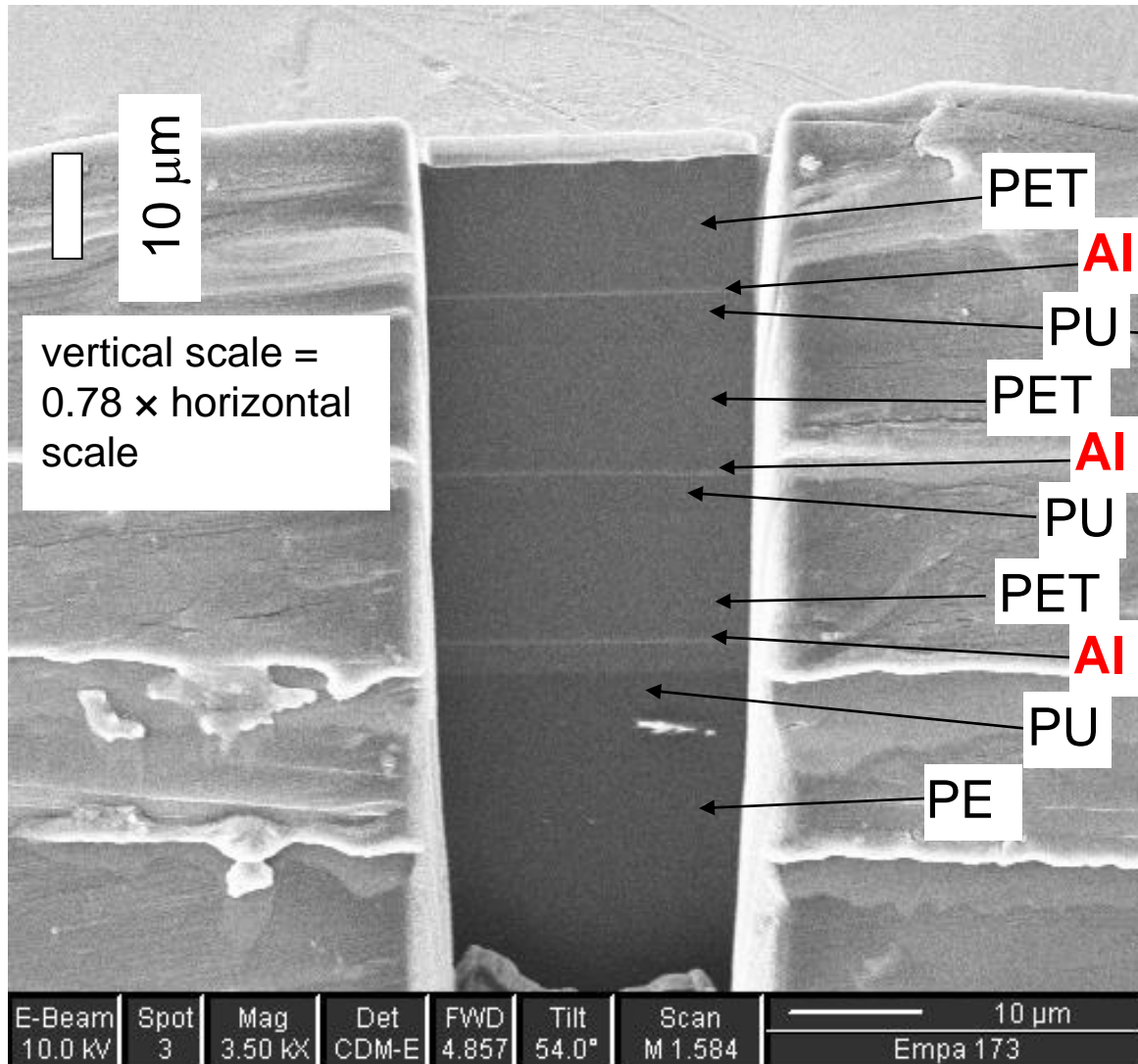
Dual Beam - 2 switchable sample positions



prepared area is seen under an angle of  $52^\circ$

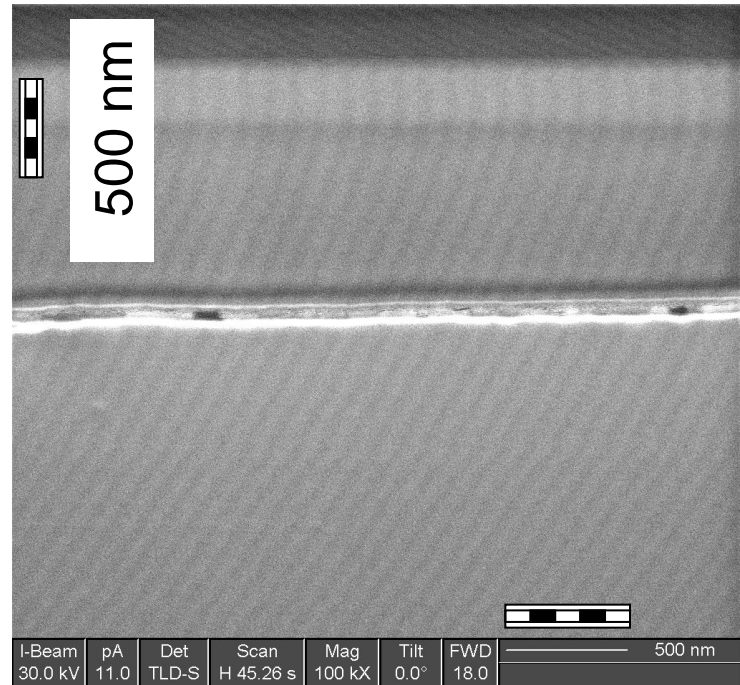
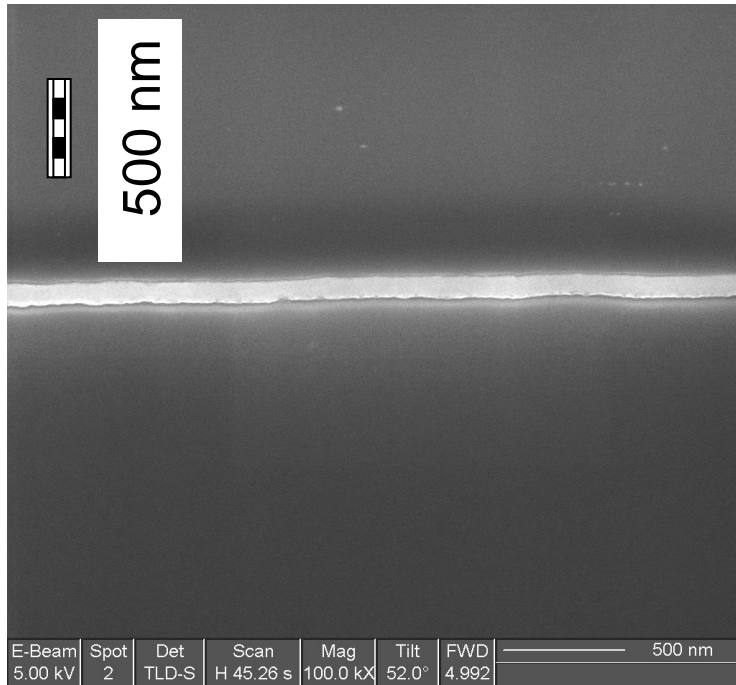


# Overview of the different layers



- PET a relatively hard plastic with superior flat surface, an excellent substrate for the
- metallic barrier layer (Al).
- polyurethane (PU) is the glue between films
- Polyethylene (PE) is the sealable layer, lowest melting temperature

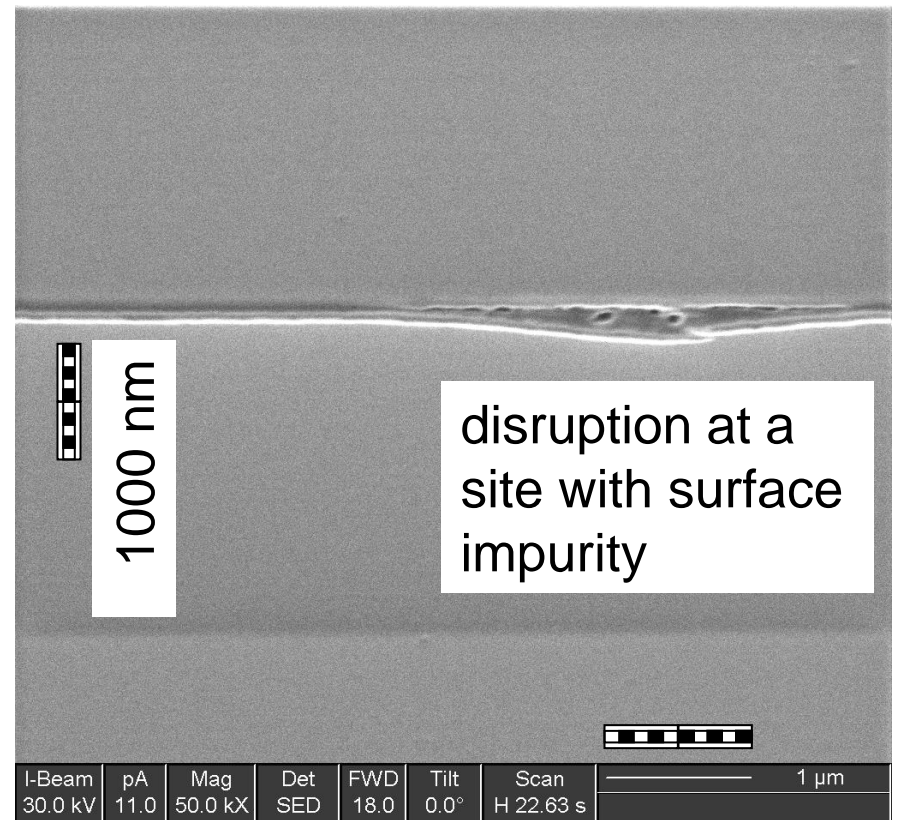
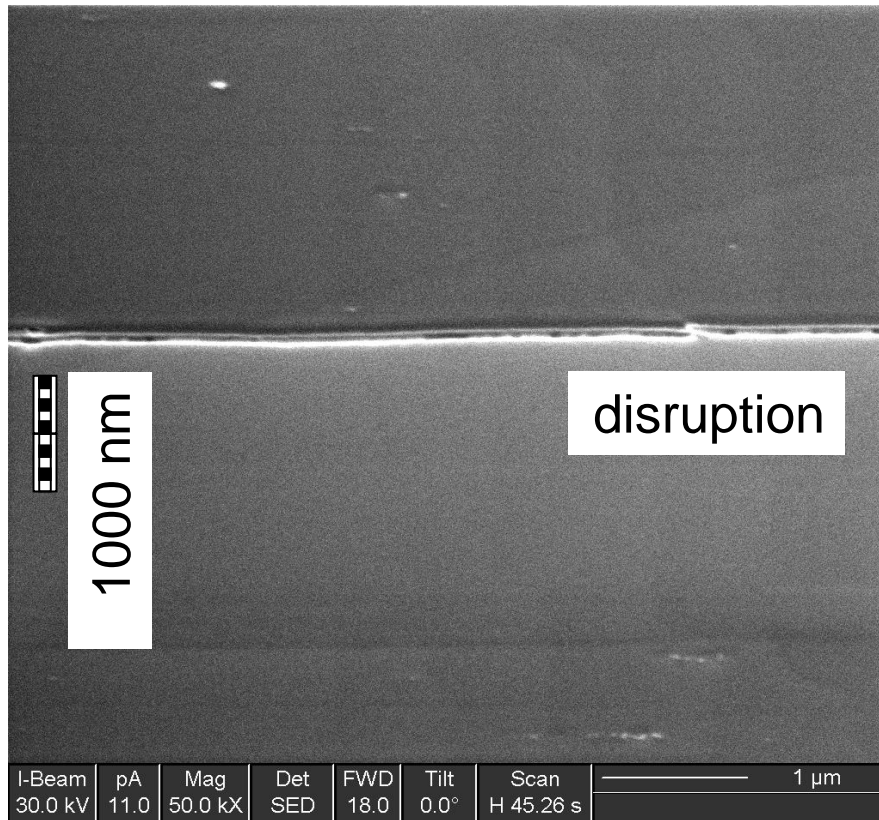
# Barrier layer - unaged



- Depending on the nature of the Beam ( Ion / Electron ) different contrast are produced at the barrier layer (aluminium)
- The different orientation of the metallic crystallites can lead to strong contrast in the Ion-beam image (left)



# Fresh, unaged laminates



- Smallest defects are visible, which are hardly avoidable during industrial production



During a typical service life, might there be an aging factor other than permeation?

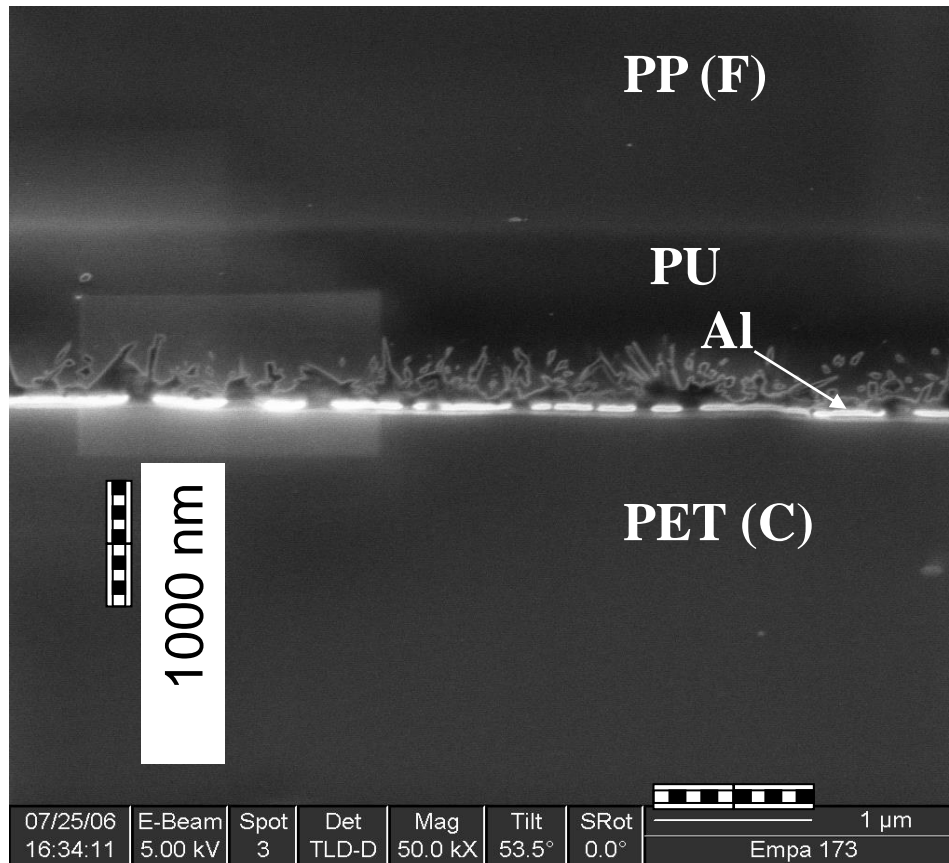
- 1) What kind?
- 2) Does it reach the failure criteria earlier than permeation?



literature on permeation in VIP :

- Simmler, H., Brunner, S., Energy and Buildings 37 (2005) 1122
- Brunner, S, Simmler H., Vacuum 82 (2008) 700
- Schwab et al, Journal of Thermal Envelope & Building Science, (2005) 28, 293-374

# After accelerated aging under extreme heat and moisture load

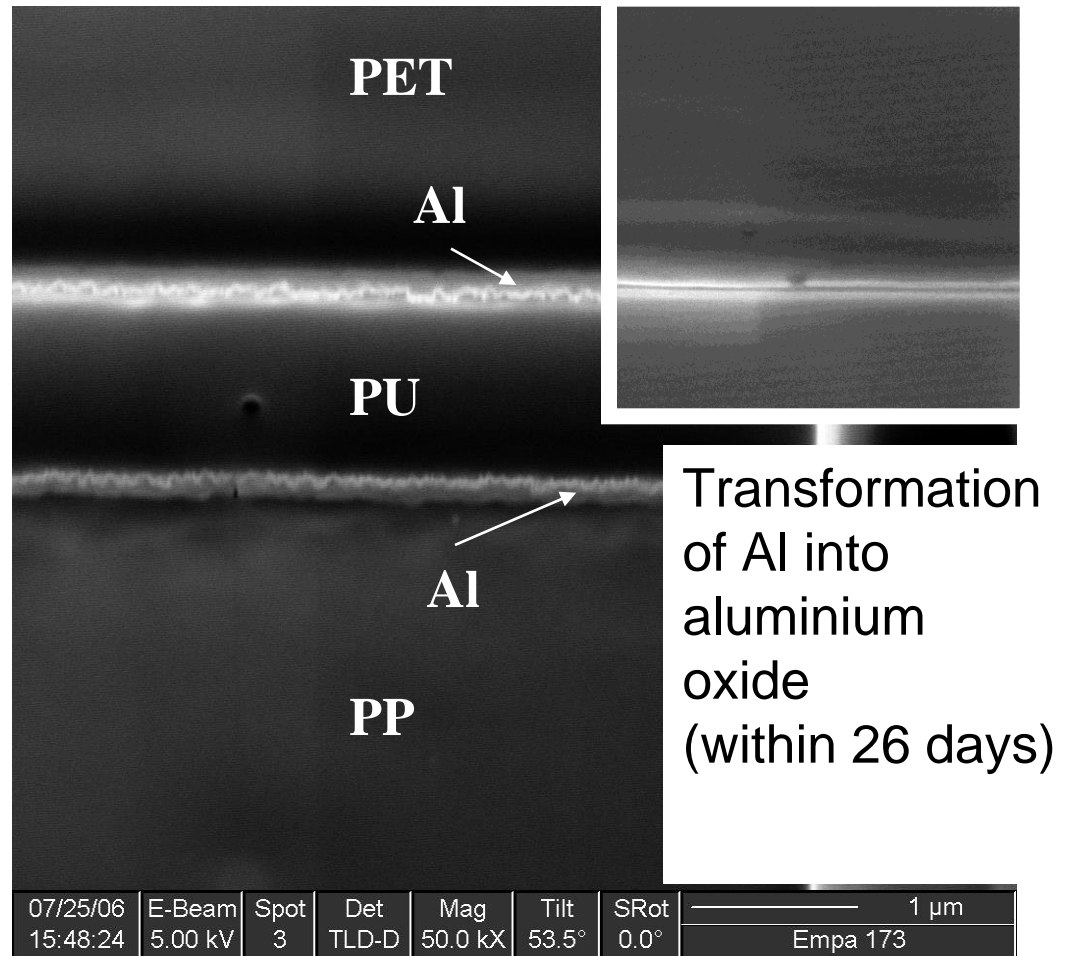
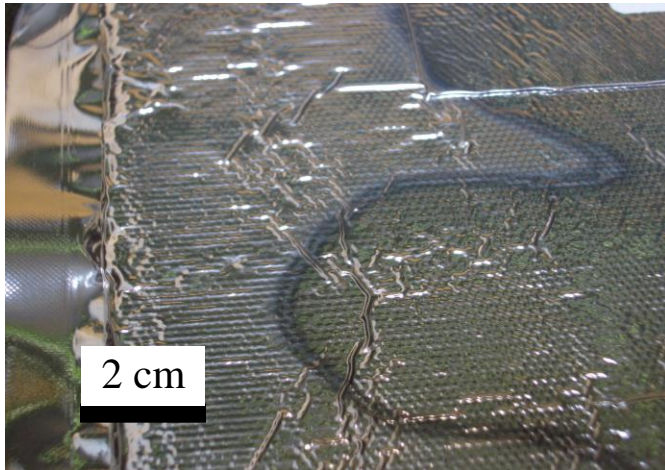


- Barrier layer with defects (corrosion?)

*Extremely aged at 65°C,  
75%r.h. for 1.9 years  
aging continued long after  
critical pressure was  
reached in the panel.*

S. Brunner et al., Surface & Coatings  
Technology 202 (2008) 6054

High humidity  $>80\%r.H.$  and temperature  $>80^{\circ}C$  leads to oxidation of aluminium

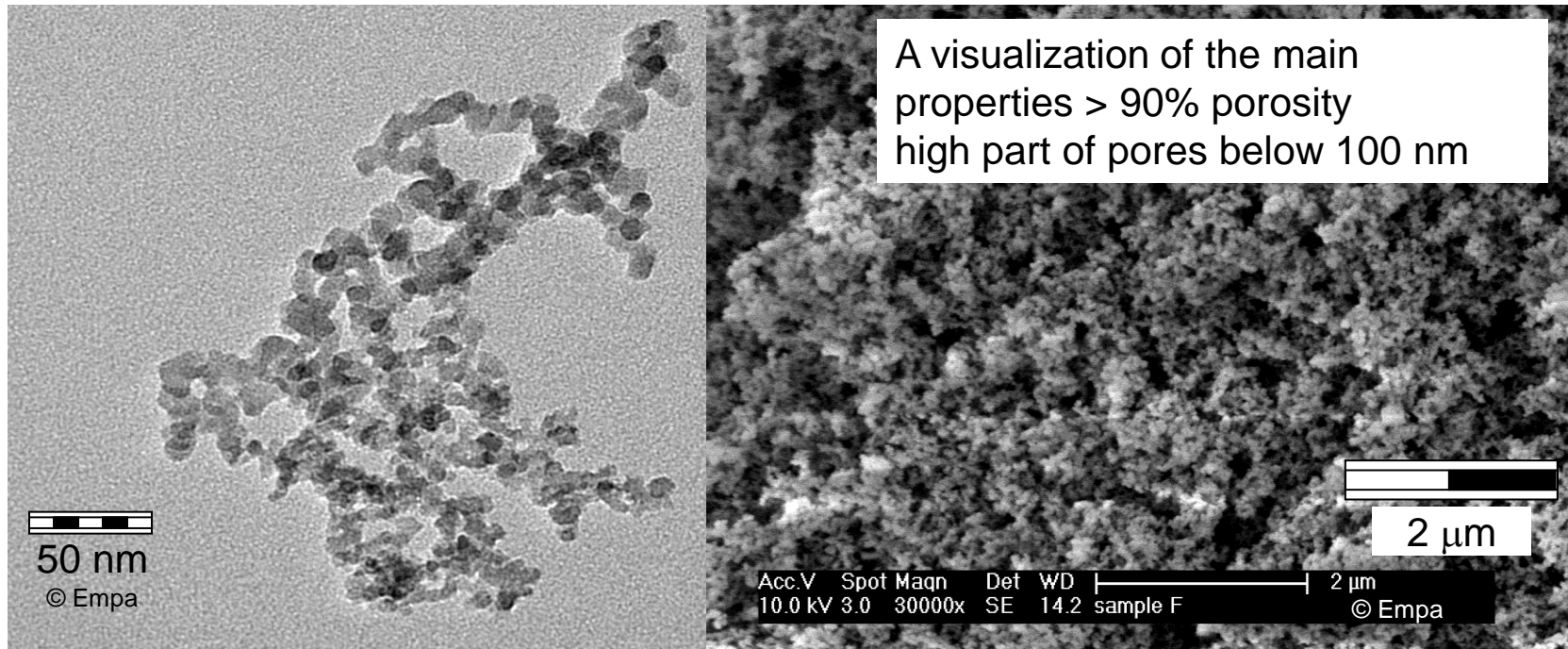


# Summary of Focused Ion Beam results

- Damages induced by high moisture and heat load do not appear in the barrier samples naturally aged during 5 years.
- Service Life Prediction based on the permeation rate through the whole VIP is the most appropriate approach for environmental conditions met in buildings in central Europe.
- Corrosion related failure mechanisms will appear much later than permeation related failure mechanism for the described application (as long as pH-conditions are not to extreme e.g. basic water from concrete).



The mentioned statements apply to VIP's with core made of pyrogenic silica.



TEM (Transmission electron microscopy)

SEM (Scanning electron microscopy)

**Thanks for your attention**