

ANNEX I

Flexible Barrier Materials for the Encapsulation of Electronics

Fundamentals, Barrier Concepts, Measurements and Recent Results

Fraunhofer Institut for Process
Engineering and Packaging, Freising



Flexible Barrier Materials for Vacuum Insulation Panels and Electronic Applications

Fundamentals, Barrier Concepts, Measurements and Recent Results

Fraunhofer Institut for Process
Engineering and Packaging, Freising



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 **Fraunhofer**
IVV

Joseph von Fraunhofer (1787 – 1826)



© Deutsches Museum

Researcher

- discovery of the “Fraunhofer lines” in the solar spectrum

Inventor

- new methods for processing lenses

Entrepreneur

- director and partner in a glassworks



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2

Fraunhofer-Gesellschaft Worldwide



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FRAUNHOFER INSTITUTE FOR PROCESS ENGINEERING AND PACKAGING IVV

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Fraunhofer IVV – Location Freising – Location Dresden

Institute Head Fraunhofer IVV: Prof. Dr. Andrea Büttner | Prof. Dr.-Ing. Jens-Peter Majschak



Total workforce	269
Scientists and graduates	143
Postgraduate students	36
Total Budget	€ 22,2 million

Total workforce	56
Scientists and graduates	41
Postgraduate students	5
Total Budget	€ 4,7 million

(Status April 2021)

(Stand: 2021)

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Possible Forms of Collaboration

Publicly funded projects

- Contributions of the companies to the project usually in the form of services, rarely money
- Pre-competitive research possible
- Project results publicly accessible

Bilateral projects

- Offer drawn up to meet the precise needs of the commissioning party
- Project starts immediately after the research is commissioned
- Project results exclusively for the commissioning party
- Confidentiality assured

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Our Business Fields and Core Areas of Expertise



Food

High-quality, healthy and convenient foods and ingredients



Packaging

Safe, customer-friendly and recyclable packaging materials



Processing Machinery

Optimized production and cleaning processes and digital solutions for Industry 4.0



Product Performance

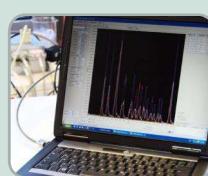
Holistic sensory optimization of raw materials and market-ready products



Recycling and Environment

Innovative recycling technologies, bio-based additives and environmental analysis

Material Development – who we are



Functional Materials

Development of films with barrier properties specially adapted to your requirements

Biobased Materials

Development of bio- and fiber-based materials and packaging

Shelf-Life Modelling

Predicting the shelf life of products in new packaging and optimizing packaging

Packaging pilot plant

Product optimization on a pilot scale with low amount of materials/ test processability of new materials and material combinations

Packaging Lab

Evaluation of mechanical and optical parameters as well as permeability as an elementary component of your material development and quality assurance

Outline

- Barrier requirements of various products
- Gas permeation through polymers
- High barrier films: Production, permeation mechanisms, challenges
- Barrier performance measurements
- Summary and outlook



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9

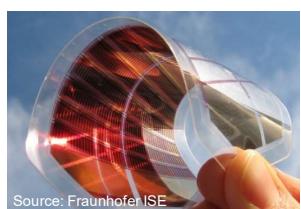
Flexible Electronics



Source: Sony



Source: Fraunhofer IAP



Source: Fraunhofer ISE



Source: Belectric OPV



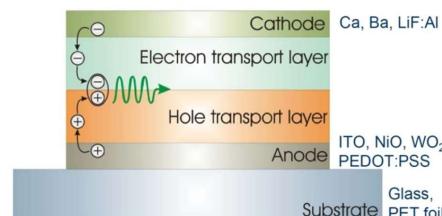
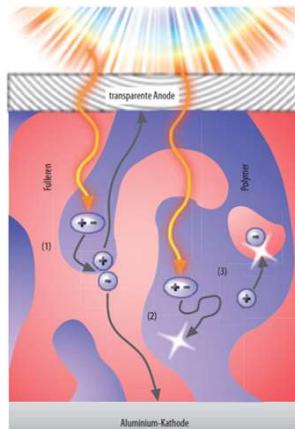
Source: Fraunhofer FEP

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10

Flexible Electronics



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Left: OPVC (Deibel et al., Physik Journal 7 (2008) Nr. 5, 51)
Right: OLED (W. Brüttig, Forum Materialien für die Polymerlektronik, Fürth, 6.11.2008)

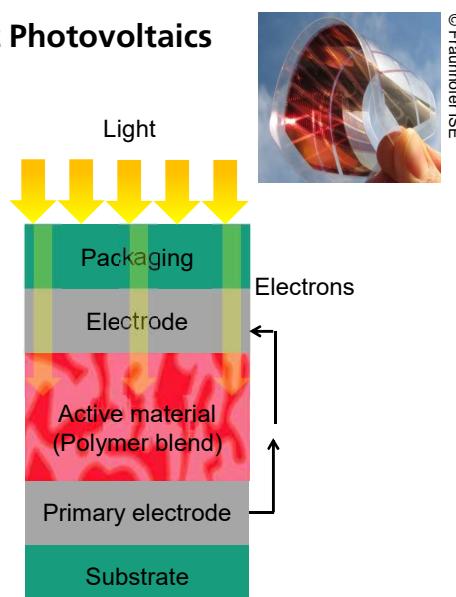


11

Flexible Electronics - Organic Photovoltaics

- Flexible devices are light-weight, unbreakable
- Low-cost roll-to-roll production instead of costly batch processes
- Low consumption of material and energy
- High productivity

Organic active layers are sensitive to oxygen and water vapor!

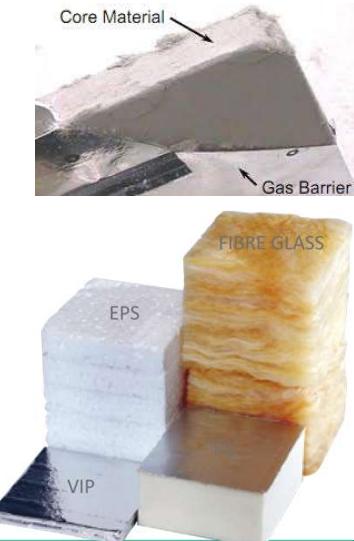


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12

Vacuum Insulation Panels



- Open porous core material
- Evacuation \Rightarrow Low heat conduction



Source: Annex 39

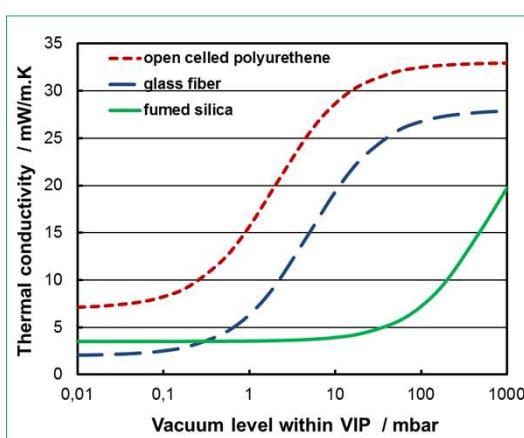
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Upper picture source: Fraunhofer IVV
Bottom picture source: <http://anjestroy.com>



13

Vacuum Insulation Panels



$$\lambda(p) = \lambda_{initial} + \frac{\lambda_{gas}}{\left(1 + \frac{p_{1/2}}{p}\right)}$$



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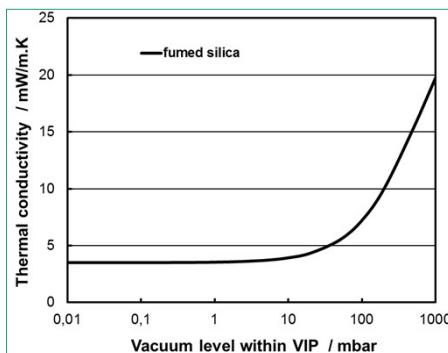
Source picture: <http://www.vacuum-panels.co.uk>



14

Vacuum Insulation Panels

- Evacuation → Low heat conduction
- Air and water vapor permeation → Pressure increase → Loss of thermal insulation

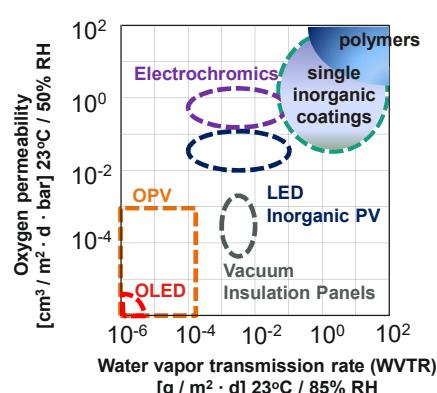
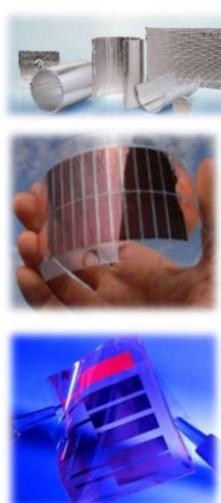


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15

Barrier Requirements of Various Products



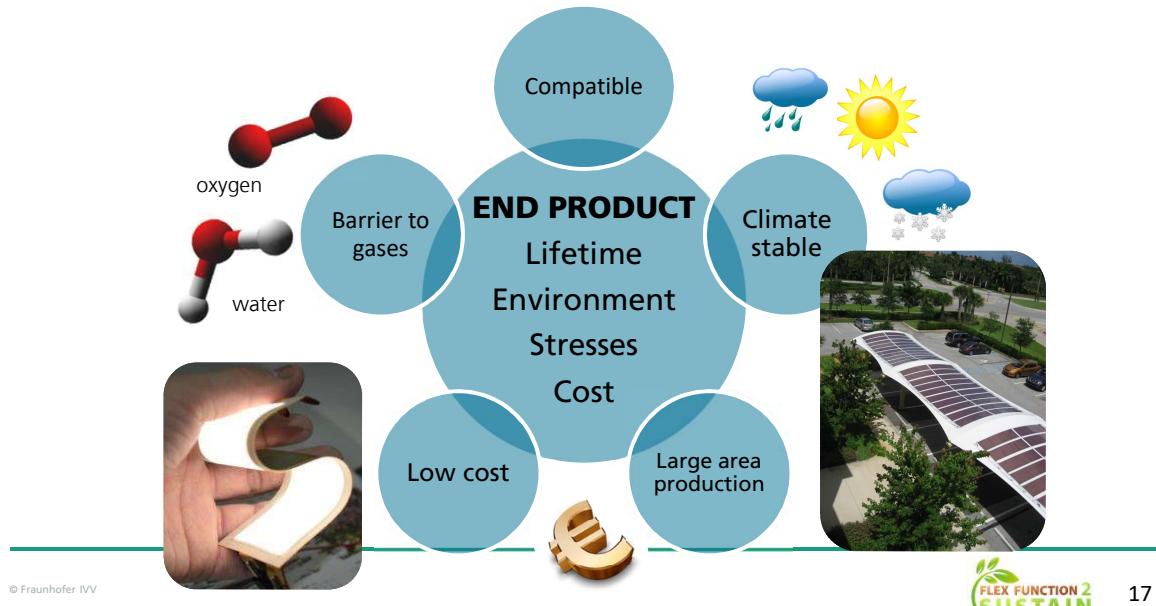
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Left: top, bottom: Fraunhofer ISE, Fraunhofer IAP, Right bottom: Prof. Jan Cremers



16

Requirements for Barrier films Depend on Application



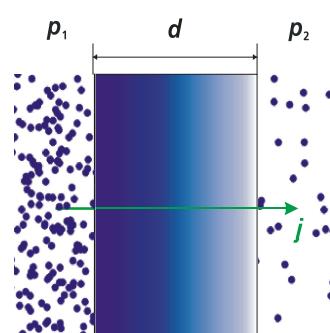
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17

Permeation through Polymers: Solution diffusion model

Steps of permeation process

- Adsorption of permeant's molecules at left polymer surface and dissolution in polymer
- Diffusion through polymer
- Desorption from right polymer surface



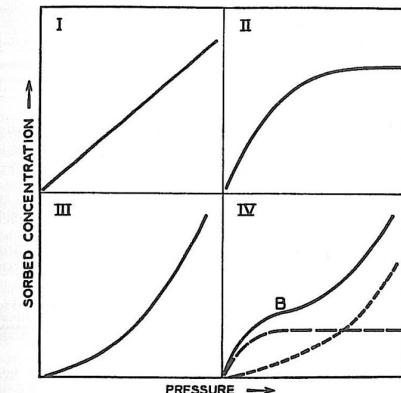
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FLEX FUNCTION 2
SUSTAIN

18

Sorption in polymers

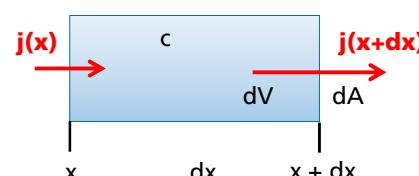
Sorption isotherms (C.E. Rogers, in J. Comyn (Ed.), Polymer Permeability ,1985, 11–73)



Sorption isotherm

- Equilibrium between gas and dissolved phase of permeant
- Simple case: **Henry's law** $c = S p$
 - c : concentration; p : partial pressure; S : solubility
 - Valid for low concentration or for negligible interaction between permeant and polymer

Diffusion in Polymers



Fick's 1st law

- Concentration gradient is driving force of diffusion

$$j = \frac{Q}{At}$$

$$j = -D \operatorname{grad} c = -D \left(\frac{\partial c}{\partial x}, \frac{\partial c}{\partial y}, \frac{\partial c}{\partial z} \right)^T$$

Continuity equation

- Conservation of material amount

⇒ **Fick's 2nd law (Diffusion equation)**

$$\frac{\partial c}{\partial t} + \operatorname{div} j = \frac{\partial c}{\partial t} + \frac{\partial j_x}{\partial x} + \frac{\partial j_y}{\partial y} + \frac{\partial j_z}{\partial z} = 0$$

$$\frac{\partial c}{\partial t} = D \left(\frac{\partial^2 c}{\partial x^2} + \frac{\partial^2 c}{\partial y^2} + \frac{\partial^2 c}{\partial z^2} \right)$$

Permeation through Polymers

Fick's First Law of Diffusion:

$$J = \frac{Q}{At} \quad J = -D \left(\frac{\partial c}{\partial x} \right)$$

Fick's Second Law of Diffusion:

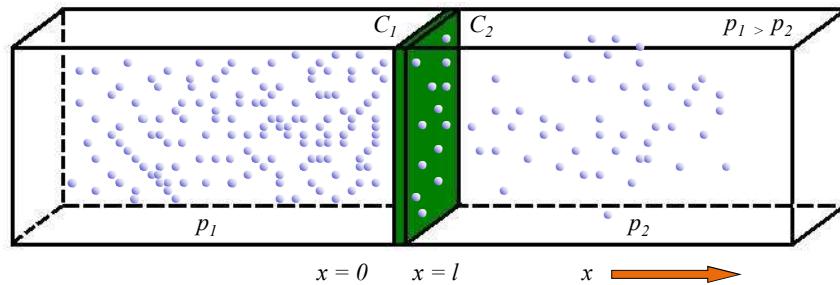
$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2}$$

$$J = -D \frac{dc}{dx} = \frac{D(C_1 - C_2)}{l}$$

$$c = Sp$$

$$J = DS(p_1 - p_2)/l$$

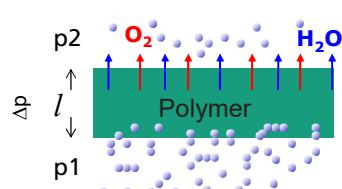
$$P = D \times S$$



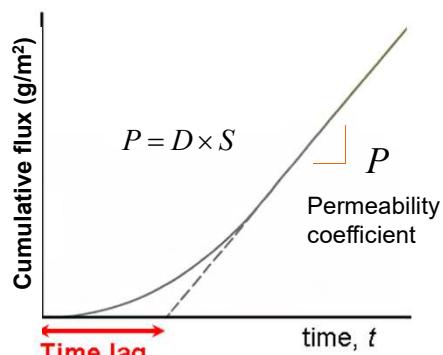
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Basic Principles of Permeation

Solution-diffusion model



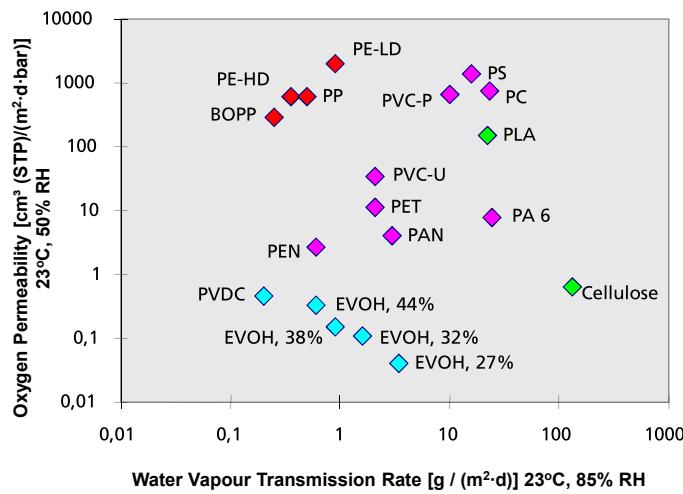
$$\text{Henry's Law: } c = Sp$$



$$\text{Time lag: } l^2 / 6D$$

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Barrier Properties of Polymer Films



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Outline

- Barrier requirements of various products
- Gas permeation through polymers
- High barrier films: Production, permeation mechanisms, challenges
- Barrier performance measurements
- Summary and outlook



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Single Inorganic Barrier Layers

Barrier materials

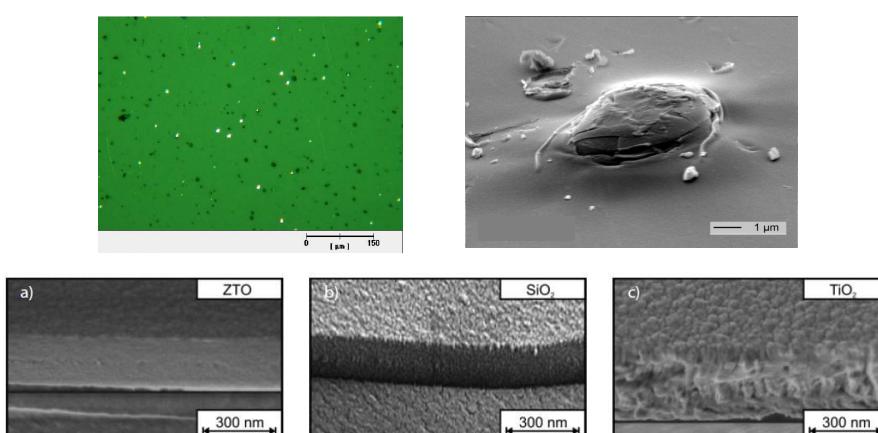
- Metals, e.g. Aluminium
- Transparent oxides and nitrides
 - SiO_2 , Al_2O_3 , Zn_2SnO_4 , Si_3N_4 , ...
- Graphene, ...

Deposition methods

- Physical vapor deposition (PVD)
 - Thermal or electron beam evaporation
 - Sputtering
- Plasma enhanced chemical vapor deposition (PECVD)
- Atomic layer deposition (ALD)

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Permeation Through Inorganic Barrier Layers



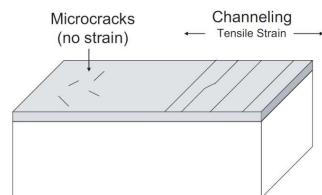
Top: Defects in Al and SiO_x (Fraunhofer IVV)
Bottom: Sputtered layer morphologies (Fahlteich et al., Vakuum in Forschung und Praxis 23 (2011) 4, 29–37)

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Permeation Through Inorganic Barrier Layers

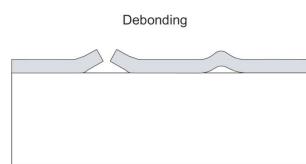
Mechanical stress in inorganic layers

- Intrinsic stress arising during film growth
- Subsequent roll-to-roll processes
- Application in flexible electronic devices



Consequences of mechanical stress

- Formation of cracks within inorganic layers
- Delamination of layers from substrate
- ⇒ Loss of barrier performance



Primary failure modes for brittle films on polymer substrates, Materials Today 9 (2006) 4, 38-45

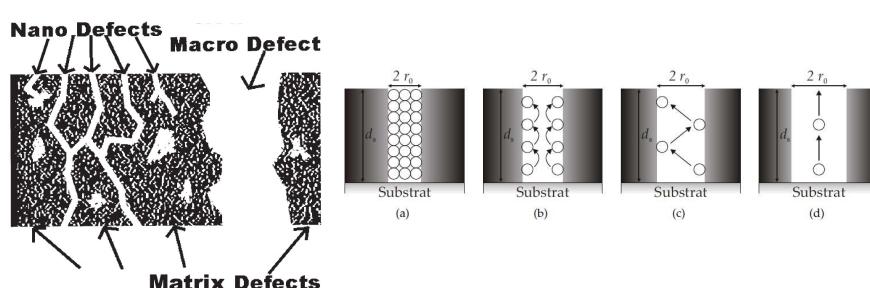
Permeation Through Inorganic Barrier Layers

Macrodefects

- Size: Nanometer up to micrometer; Localized positions
- Unhindered transport: Free diffusion (d), Knudsen diffusion, molecular flow (c)

Microdefects (Nanodefects)

- Size: Sub-nanometer up to nanometer; Quasi-homogeneously distributed
- Hindered transport: Surface diffusion (b), capillary condensation (a)



Left: Affinito, Hilliard, A New Class of Ultra-Barrier Materials, 47th Annual Technical Conference Proceedings SVC, 2004, 563-593
Right: J. Fahleit et al., Transparente Hochbarriereschichten auf flexiblen Substraten, Dissertation, Chemnitz, 2010

Modelling of Permeation Through Barrier Films

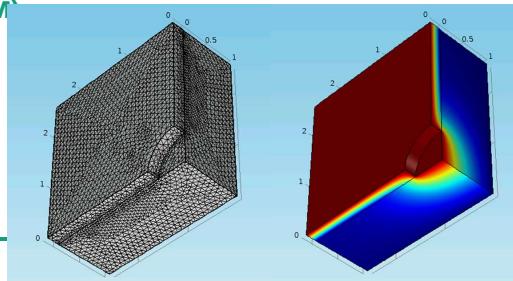
Polymeric materials

- Diffusion equation

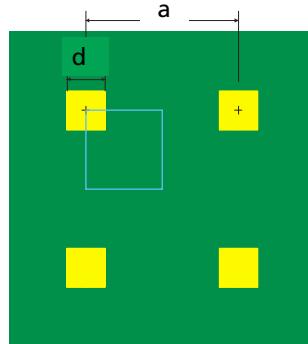
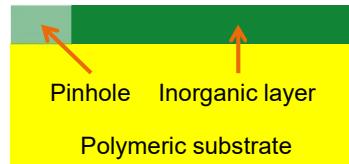
Inorganic layer

- Permeation of oxygen only through pinholes \Rightarrow boundary condition
- Permeation of water vapor also through inorganic matrix (i.e. microdefects)

\Rightarrow Numerical solution of diffusion equation by finite element method (FEM)



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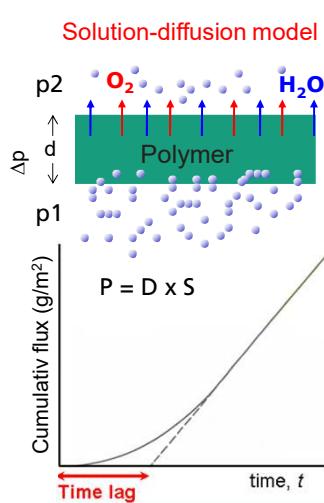


Source:
Oliver Miesbauer

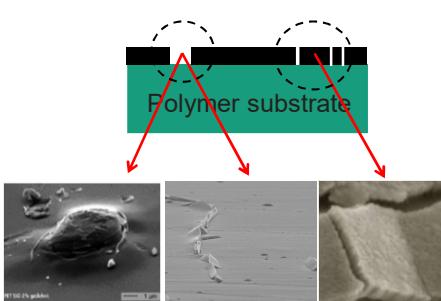


29

Permeation through Single Inorganic Barrier Layers



„Defect-dominated“ permeation mechanisms



SEM picture:
Defects due to
anti-blocking
particles

SEM picture:
Defects resulting
due to thermal
stress

SEM picture:
Porosity and
surface
roughness

Inorganic layer coatings: Al, AlO_x, SiO_x

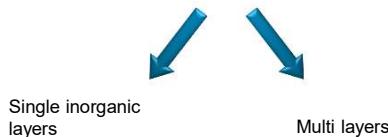
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30

High Barrier Films

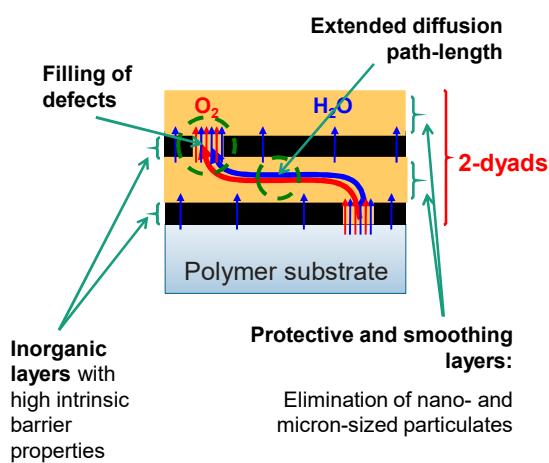
- Permeation through thin films:
Permeation is caused by defects.
- Defects caused by: particles, scratches, non perfect film formation, stress in grown films....
- Approach for the reduction of number and size of defects leading to decreasing permeation



- Large influence of substrate
- Layers are sensitive to mechanical stress
- Defect free deposition needed - ALD
- Combination of barrier layers and smoothening layers, e.g. thin polymer films
- Wet chemical processes for the deposition of the interlayers

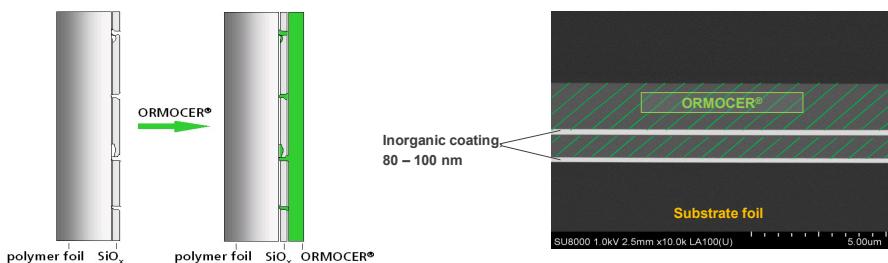
Multilayer High Barrier Films

Ultrabarrier Stack: Alternating inorganic/organic layers



High Barrier Films for Device Encapsulation

Barrier Lacquer ORMOCE®



Fraunhofer
ISC

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<http://www.polo.fraunhofer.de/>
Fraunhofer Polymer Surfaces Alliance POLO®

Fraunhofer
POLO

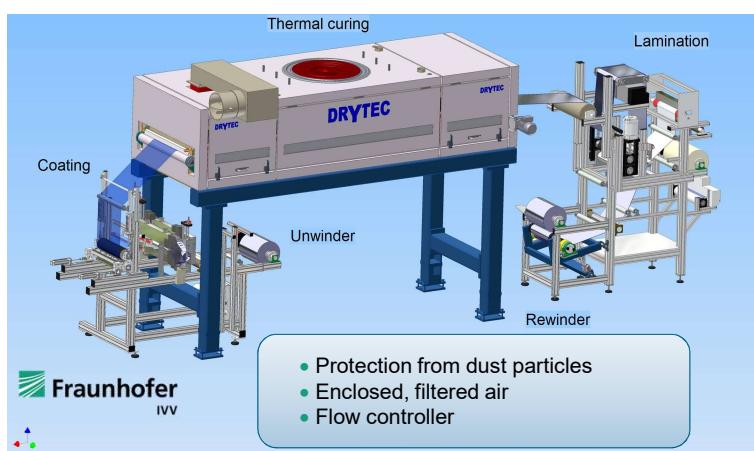


33

Multilayer High Barrier Film Production

Coating Technologies: Roll-to-Roll Processes

Lamination / Lacquering under Clean-Room Environment



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34

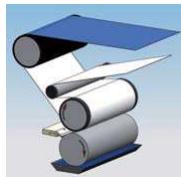
Multilayer High Barrier Film Production

Coating Technologies: Roll-to-Roll Processes

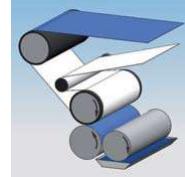
Lamination / Lacquering under Clean-Room Environment

Wet chemical coating process

- Choice of coating and curing process and parameters depends on:
 - Substrate, e.g. surface morphology, surface tension, dimensional stability
 - Properties of the coating material, e.g. rheology, viscosity



Coating with gravure rollers



Coating with smooth rollers

Source: A. Glawe, KROENERT

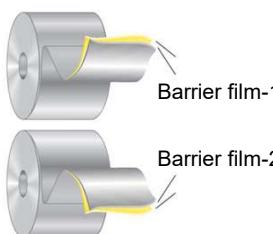


35

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Multilayer High Barrier Film Production: Low-cost

Laminated Multilayered Structures



- ✓ Higher mechanical stability
- ✗ Thicker structures

Challenge: Proper adhesive selection
Thermal stability problem: 85°C/85%RH



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36

High Barrier Films for Device Encapsulation

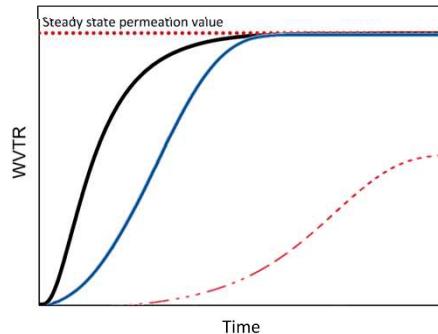
Ultrabarrier Stack: Alternating inorganic/organic layers

Alternating inorganic barrier and organic intermediate layers

- Reduction in WVTR
- Elongation of time-lag

Parameters effecting barrier performance

- Diffusion, Solubility, Permeability Coefficient of each single layer
- Layer sequence and thickness



Experimental and Theoretical Studies on the Time-Dependent Permeation Through Multilayered Encapsulation Films for Flexible Organic Electronics, Oliver Miesbauer, June 5 (15:00), Web Coating and Handling

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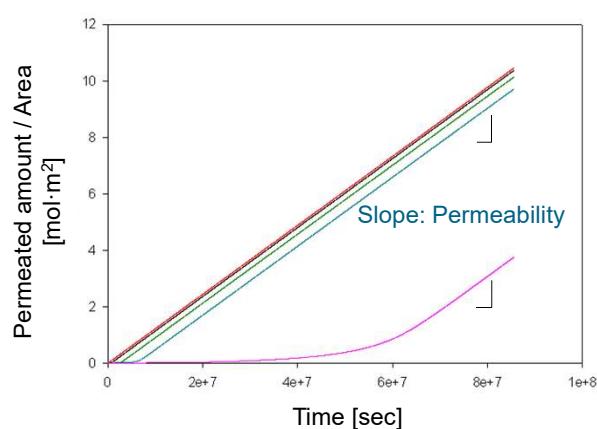


High Barrier Films for Device Encapsulation

Ultrabarrier Stack: Alternating inorganic/organic layers

Optimisation of multilayer films for low permeability and long time lag

- Increase of number of dyads
- Integration of active absorbers into multilayers



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38

High Barrier Film Production

Biggest Challenges

▪ Substrate quality:

- Cleanliness, surface roughness

▪ Thermal and mechanical stability:

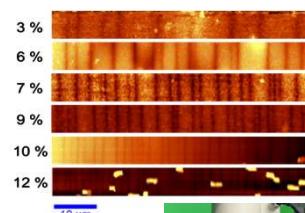
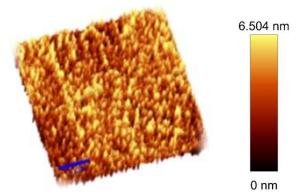
- Substrate and barrier layers

▪ Process conditions:

- Temperature, Pressure (lamination)
- Web-speed, web tension
- Dust-free coating, no contact on coated side

▪ Quality Control:

- Inline (layer thickness, crosslinking degree)
- Off-line (permeation, adhesion, bending stability)



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39



Any Questions?

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40

Outline

Barrier requirements of various products
Gas permeation through polymers
High barrier films: Production, permeation
mechanisms, challenges
Barrier performance measurements
Summary and outlook



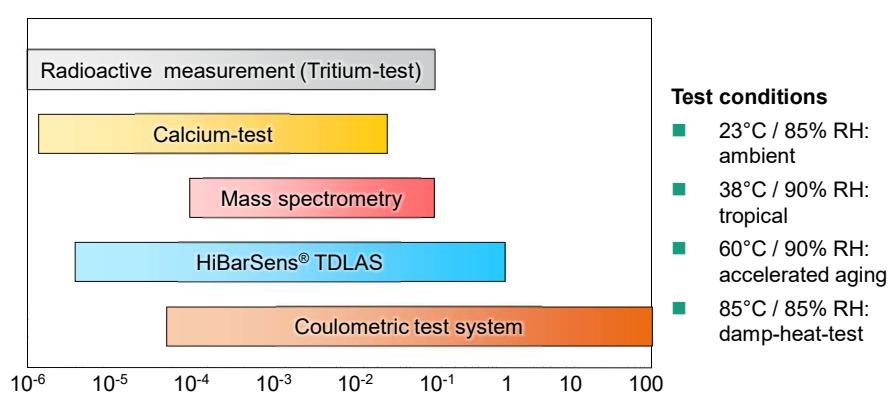
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41

High Barrier Performance Measurements for Electronics

Water Vapor Transmission Rate (WVTR) Measurement Tools



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Graph modified from C. Boeffel et al. "Ultra-high barrier substrates and ALD layers for the encapsulation of organic electronic devices" ISFOE14



42

High Barrier Performance Measurements for Electronics

Water Vapor Transmission Rate (WVTR): Coulometric Test



Mocon® Aquatran®

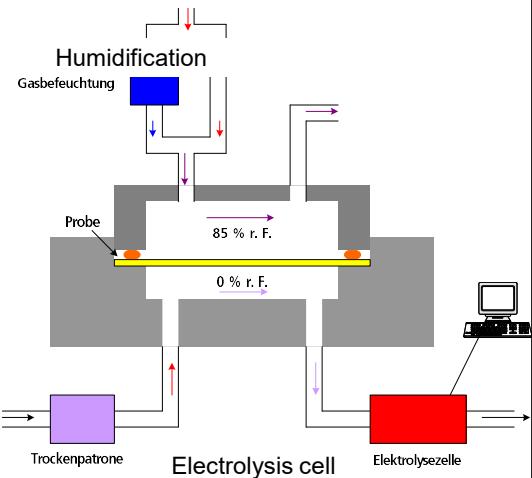
Model 2

Detection limit:

$5 \times 10^{-5} \text{ g}/(\text{m}^2 \cdot \text{d})$

Carrier
gas

Sample



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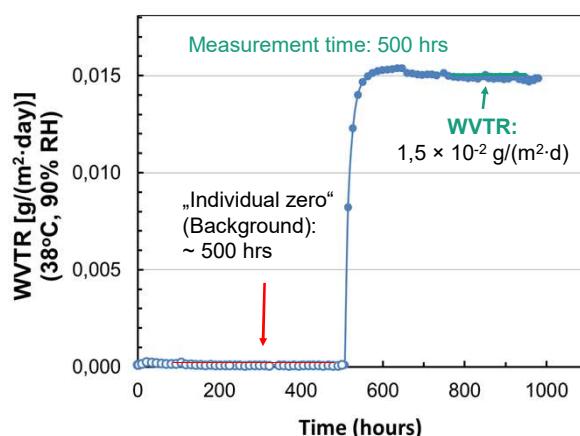


43

High Barrier Performance Measurements for Electronics

Important Factors

- High WVTRs with short time lags → not critical!



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Mocon® AQUATRAN® Model 2

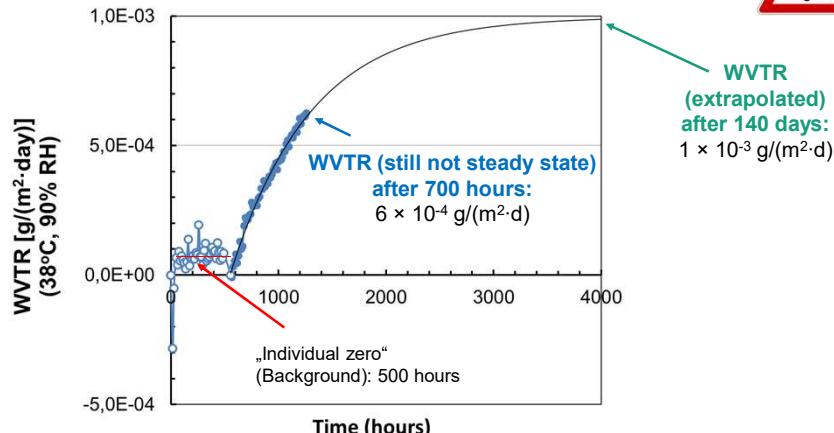


44

High Barrier Performance Measurements for Electronics

Important Factors

- Low WVTRs ($< 5 \times 10^{-3} \text{ g}/(\text{m}^2 \cdot \text{d})$) with long time lags → Pay attention



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Mocon® AQUATRAN® Model 2



45

High Barrier Performance Measurements for Electronics

Important Factors

- Evaluation of measurement curves can be difficult due to the complicated water vapor permeation mechanisms in multilayered structures
- Keep in mind:
 - Pre-conditioning of samples before testing
 - Sufficient measurement duration to ensure the steady state

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46

High Barrier Performance Measurements for VIPs

Important Factors and Limitations



- Barrier performance of VIP envelope before and after VIP production, not comparable
- Values not comparable unless all measurement conditions are the same (Temperature, humidity, measurement duration, ...)
- Measurement limits of the commercially available devices are not sufficient to report the exact values
- Limitation of measurement capacity, when using commercially available devices for permeation → Long measurement times

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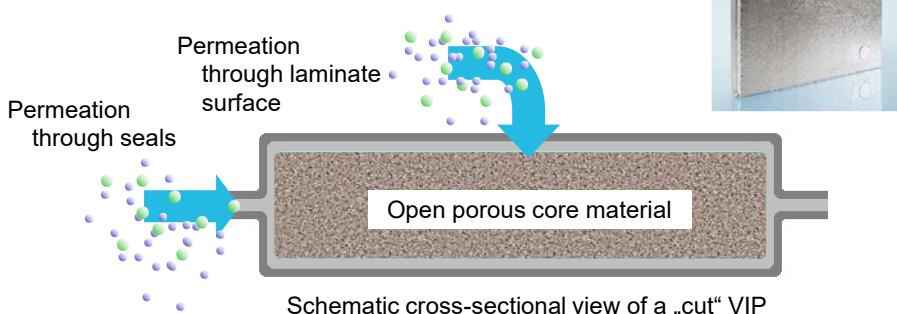
47

High Barrier Performance Measurements for VIPs

Gas and Water Vapor Barrier Performance

Permeation measurements through

- 1) Flat films: VIP laminates before VIP production
- 2) VIP envelopes: VIP laminates after VIP production



Schematic cross-sectional view of a „cut“ VIP

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48

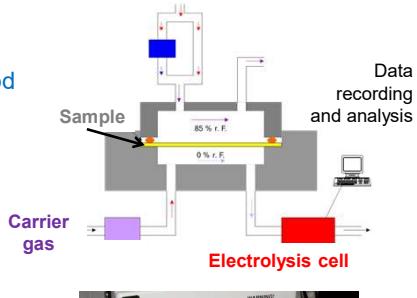
Permeation Measurement Techniques and Conditions

Flat Film Measurements by Coulometric Method

Water Vapor Transmission Rate (WVTR)

Mocon® Aquatran™ Model 2

- DIN EN ISO 15106-3
- Measurement limit: 5×10^{-5} g / (m²·day)
- Sample area: 50 cm²
- Temperature: 40°C, RH: 90%
- Pre-conditioning: 2 days, 60°C, 0.5 mbar



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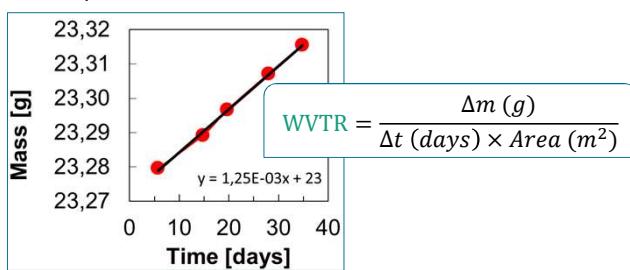
49

Permeation Measurement Techniques and Conditions

VIP Envelopes

WVTR by Water Intake (Gravimetric)

- TC 88 WG 11 "VIP for buildings"
- Measurement limit: 2×10^{-3} g / (m²·day)
- Temperature: 40 °C, RH: 90%



Micro-balance

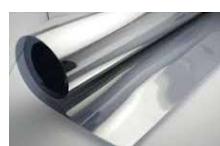
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50

Permeation Measurement Techniques and Conditions

WVTR Measurement Conditions - Summary



Laboratory	Flat Films	Measurement conditions
Lab-1	MOCON® Aquatran™ Model 2 Meas. Limit: 5×10^{-5} g/(m ² ·d)	40 °C / 90 % RH



Laboratory	VIP Envelopes	Measurement conditions
Lab-2	Water Intake (Gravimetric) Desiccant (~ 150 mm x 120 mm x 5 mm)	40 °C / 90 % RH

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51

Water Vapor Transmission Rate (WVTR)

Measurement Results - Summary



Sample Type	Flat films	VIP Envelopes
	Lab-1	Lab-2
@ 38°C / 90% RH g / (m ² ·day)		
Bi-laminate	1.5×10^{-2}	3.0×10^{-2}
Tri-Laminate	2.1×10^{-2}	1.7×10^{-2}

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52

Permeation Measurement Techniques and Conditions

Flat Film Measurements by Coulometric Method

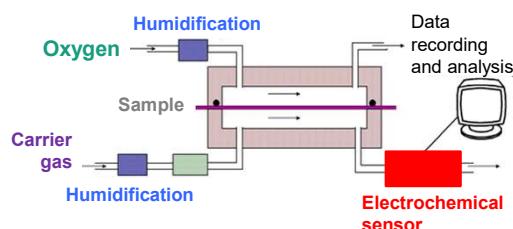
O₂ Permeability

Mocon® Oxtran® Model 2/21

- DIN 53 380, T3
- Measurement limit: 5×10^{-3} cc(STP) / (m²·day·bar)
- Sample area: ~ 50 cm²
- Temperature: 23 °C, RH: 50%



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53

Conclusions and Outlook

- **No significant influence** of VIP production on **water vapor barrier performance** of laminates; similar results for flat films and VIP envelopes
- Comparison of O₂ permeability of the **flat films** to the air permeability values through **VIP envelopes into the VIPs** only by **rough estimation**
- **Air permeability** measurements using **VIP thermal conductivity change** as a function of time proves to be a very promising method
- **European Committee for Standardisation (CEN)** is currently working on the adoption of some of the techniques used within this exercise
- **Further work** still necessary to relate the **real lifetime of VIPs** to **gas/water vapor barrier** properties!

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54

Outline

- Barrier requirements of various products
- Gas permeation through polymers
- High barrier films: Production, permeation mechanisms, challenges
- Barrier performance measurements
- Results from collaborative projects
- Summary and outlook



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55

Summary and Outlook

- **High barrier requirements** of products can be fulfilled by structures of alternating inorganic and polymeric layers
- **Permeation through inorganic layers** limited to defects (pinholes, cracks).
- Barrier performance of multilayer films due to **synergistic effect, tortuous path effect and extended lag time**
- Further barrier improvement possible by **lamination**, and / or integration of **water vapor absorbers**
- **Modelling and numerical simulation** help to optimize barrier film structures
- **Characterisation: Permeability measurements**
New high sensitive WVTR measurement techniques

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56

Summary and Outlook

Requirements for barrier films depend on final application...

Multi-disciplinary approach for successful development

Close cooperation with end-product producers!

- **Low cost deposition techniques, roll-to-roll and large area production:**

High rate evaporation processes combined with lamination

- **Climate stability (weatherability) for outdoor applications:**

UV- stabilised and hydrolysis resistant substrates

- **Compatibility to subsequent processes and added functionality:**

Ready-to-use encapsulation films with adhesives

- **Mechanical stability, adhesion between layers crucial**

Adhesion promoting tie-layers

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57

Thank you for your interest!



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ANNEX II

Barrier Materials in Flexible Packaging for Food Applications

Fraunhofer Institut for Process
Engineering and Packaging, Freising



BARRIER MATERIALS IN FLEXIBLE PACKAGING FOR FOOD APPLICATIONS

Fraunhofer Institut for Process Engineering and Packaging (IVV) - Freising



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IVV

Joseph von Fraunhofer (1787 – 1826)



© Deutsches Museum

■ Researcher

- discovery of the "Fraunhofer lines" in the solar spectrum

■ Inventor

- new methods for processing lenses

■ Entrepreneur

- director and partner in a glassworks

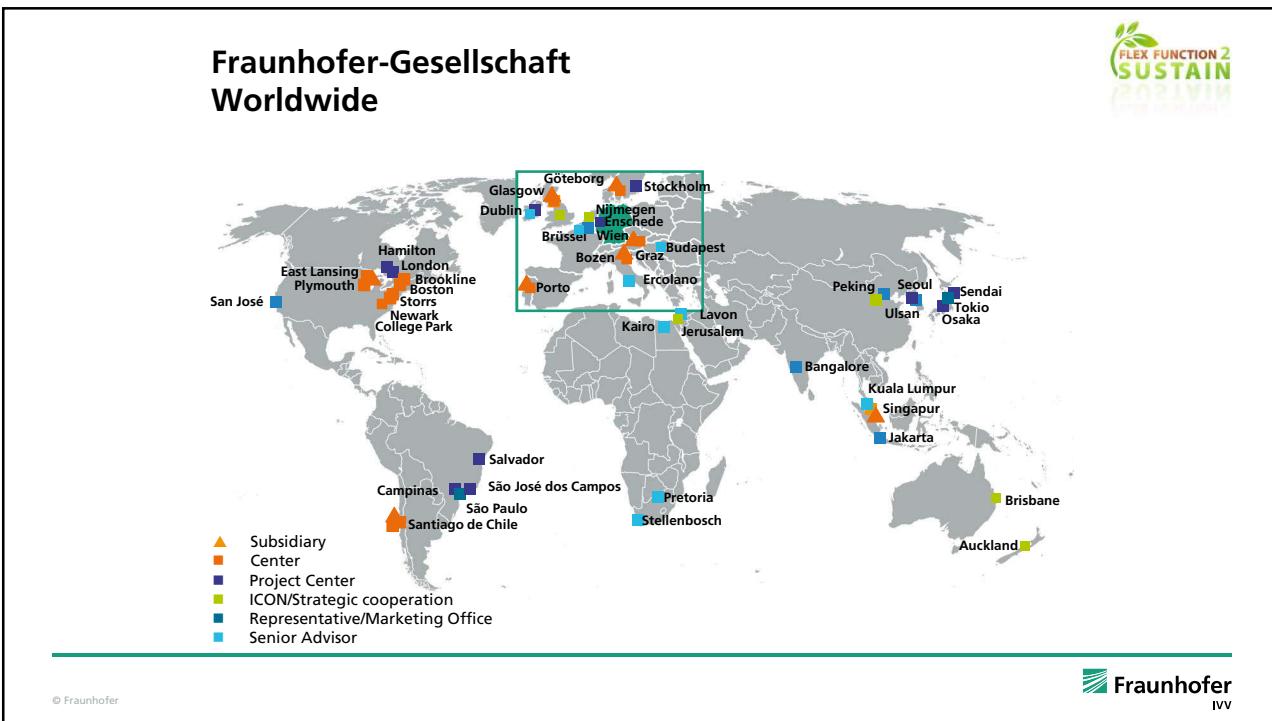
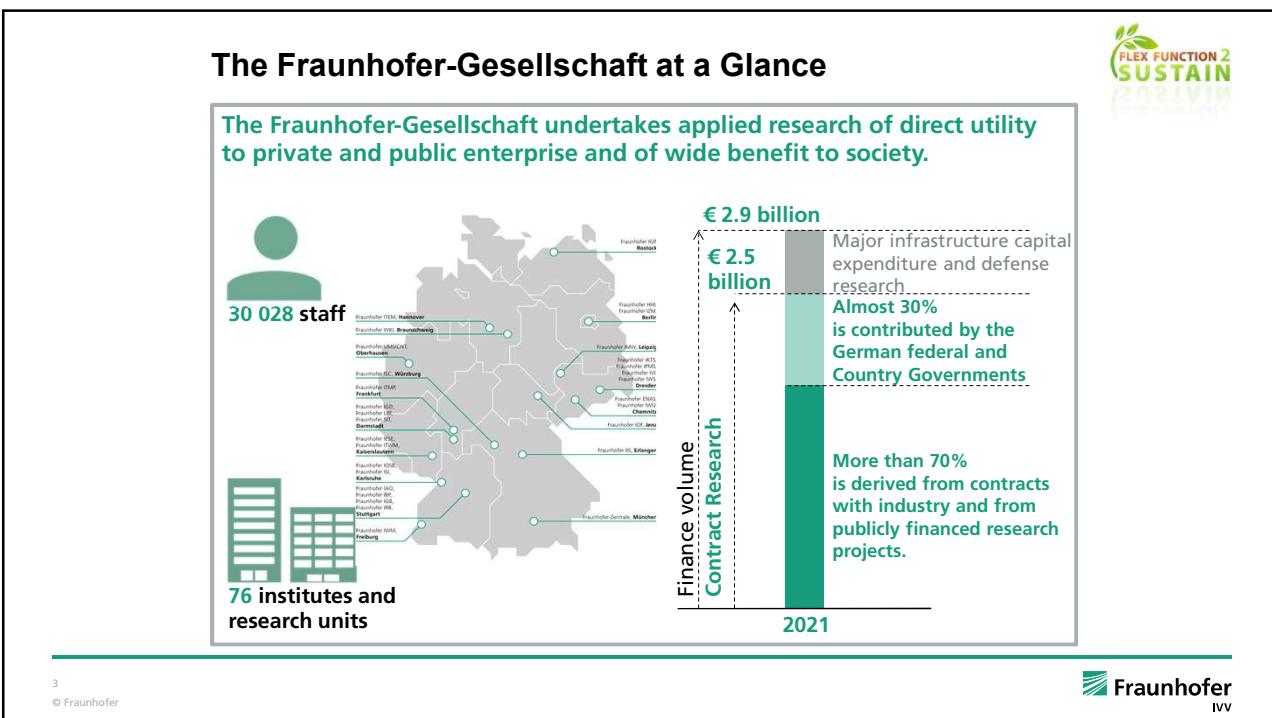


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2
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FRAUNHOFER INSTITUTE FOR PROCESS ENGINEERING AND PACKAGING IVV

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Fraunhofer IVV – Location Freising – Location Dresden

Institute Head Fraunhofer IVV: Prof. Dr. Andrea Büttner | Prof. Dr.-Ing. Jens-Peter Majschak



■ Total workforce	269
■ Scientists and graduates	143
■ Postgraduate students	36
Total Budget	€ 22,2 million

Total workforce	56
Scientists and graduates	41
Postgraduate students	5
Total Budget	€ 4,7 million

(Status April 2021)

(Stand: 2021)

6
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Possible Forms of Collaboration



Publicly funded projects	Bilateral projects
<ul style="list-style-type: none">■ Contributions of the companies to the project usually in the form of services, rarely money■ Pre-competitive research possible■ Project results publicly accessible	<ul style="list-style-type: none">■ Offer drawn up to meet the precise needs of the commissioning party■ Project starts immediately after the research is commissioned■ Project results exclusively for the commissioning party■ Confidentiality assured

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Our Business Fields and Core Areas of Expertise

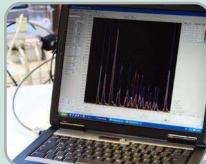


-  **Food**
High-quality, healthy and convenient foods and ingredients
-  **Packaging**
Safe, customer-friendly and recyclable packaging materials
-  **Processing Machinery**
Optimized production and cleaning processes and digital solutions for Industry 4.0
-  **Product Performance**
Holistic sensory optimization of raw materials and market-ready products
-  **Recycling and Environment**
Innovative recycling technologies, bio-based additives and environmental analysis

8
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Material Development – who we are



Functional Materials

Development of films with barrier properties specially adapted to your requirements

Biobased Materials

Development of bio- and fiber-based materials and packaging

Shelf-Life Modelling

Predicting the shelf life of products in new packaging and optimizing packaging

Packaging pilot plant

Product optimization on a pilot scale with low amount of materials/ test processability of new materials and material combinations

Packaging Lab

Evaluation of mechanical and optical parameters as well as permeability as an elementary component of your material development and quality assurance

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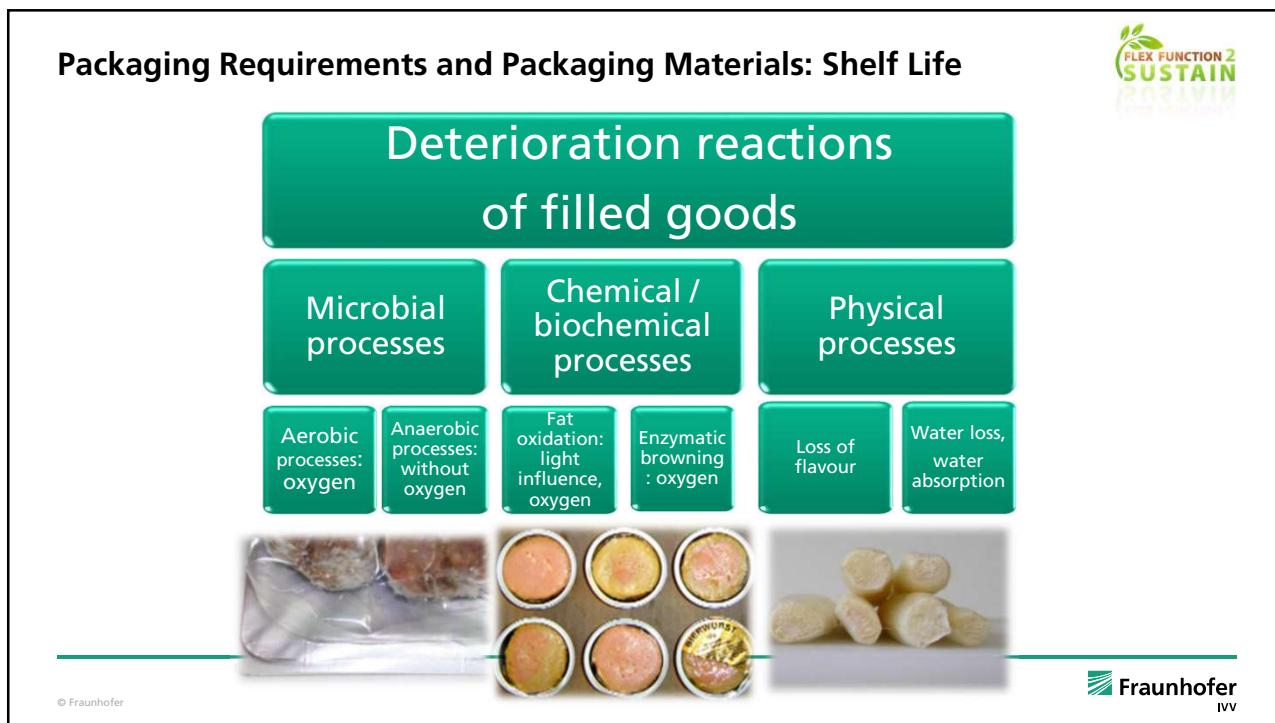
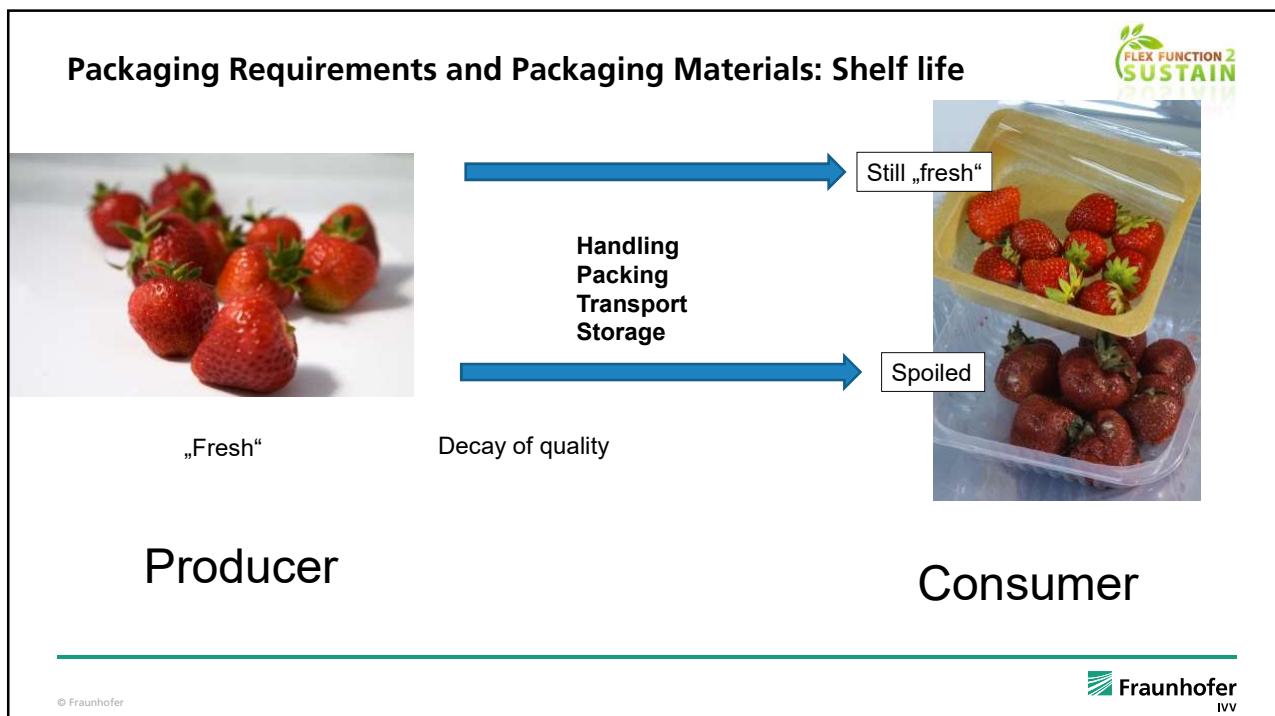
AGENDA



1. Packaging Requirements and Packaging Materials
2. Design for Recycling
3. Barrier concepts: „Mono-material“ Multi-layer
 - 3.1. Thin Inorganic Layers on Polyolefin Substrates with Primer Coating
 - 3.2. Nano-composite Coatings
4. Summary

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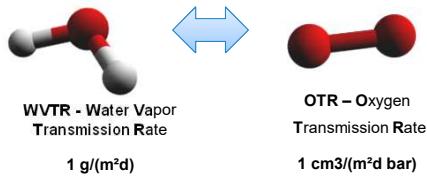




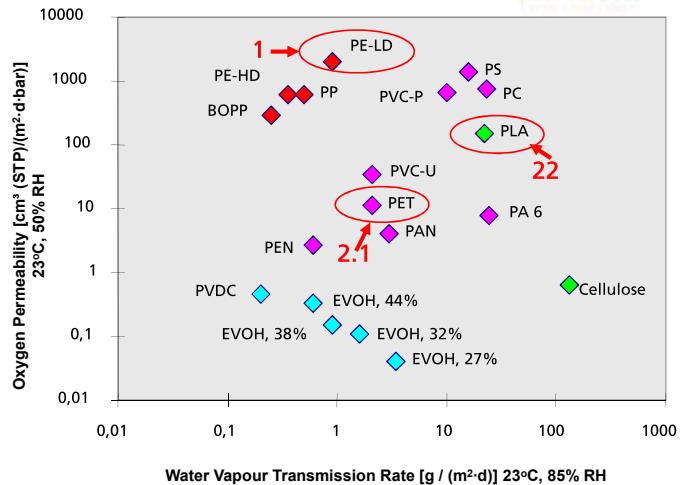
Packaging Requirements and Packaging Materials: WVTR and OTR



At 23°C and 50% RH conditions



- The shelf life of water in a **PET** bottle would be about **10 times longer** than in a **PLA** bottle
- The shelf life of a dry product would be **22 times shorter** in **PLA** than in **PE-LD** of the same thickness



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Design for Recycling



Redesign multi-material flexible packaging to mono-materials, with existing recycling streams

Film conversion process development

Polyolefin based flexible packaging for recyclability

Sorting, recycling, re-use

Packaging requirements – Shelf life modelling

Mono-material design for recyclability

$\text{SiO}_x, \text{AlO}_x$ on polyolefin substrates

Barrier lacquers, adhesives

Film orientation technologies, novel coating processes

Nano-functionalized barrier coatings

Barrier Concepts

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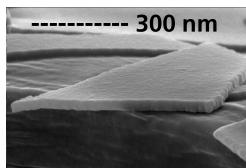
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Barrier Concepts: „Mono-material“ Multi-layer

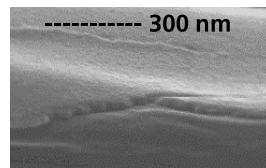
■ Thin Inorganic Layers on Polyolefin Substrates with Primer Coating

- Physical Vapor Deposition (PVD)
 - Thermal or electron beam evaporation
- Inorganic barrier materials
 - Transparent oxides (e.g. SiO_x , AlO_x , Al, ...)

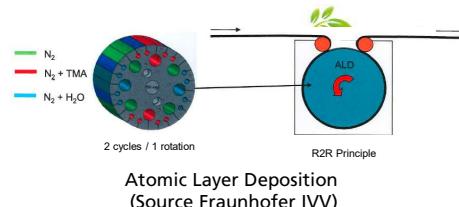
■ Substrates: PET, BOPP, ...



SiO_x (~ 60 nm)



Al metallisation (~ 50 nm)



Upgrade in 2023
FlexFunction2Sustain
Grant Agreement ID: 862156



Electron beam evaporation
(Source: Amcor Flexibles)

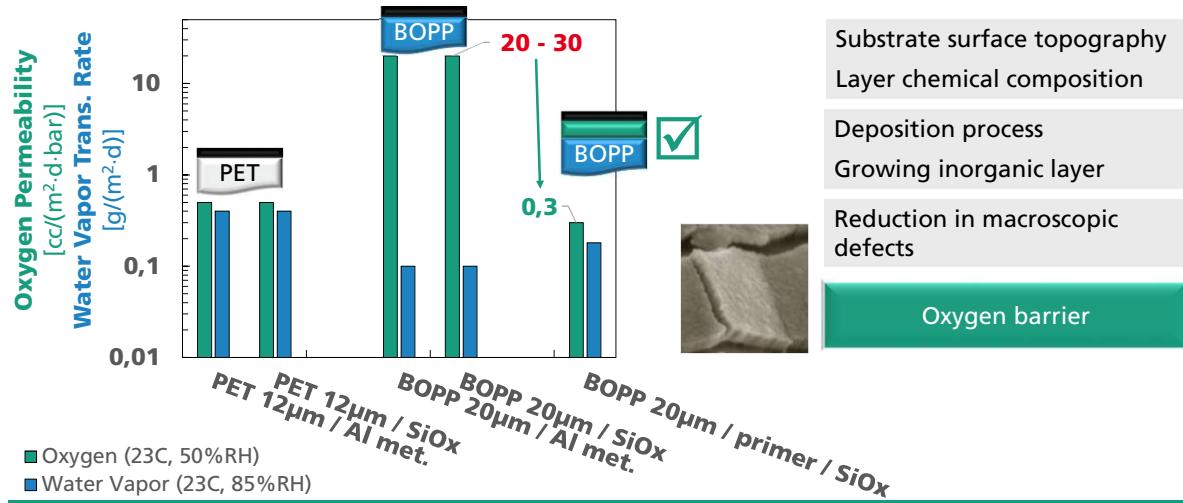
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Barrier Concepts: „Mono-material“ Multi-layer



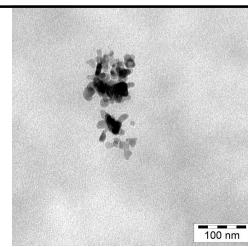
■ Thin Inorganic Layers on Polyolefin Substrates with Primer Coating: Results



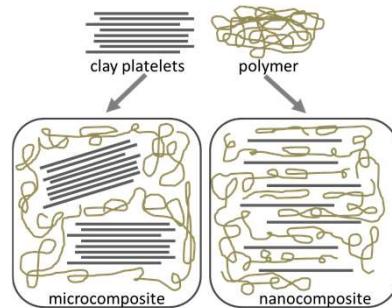
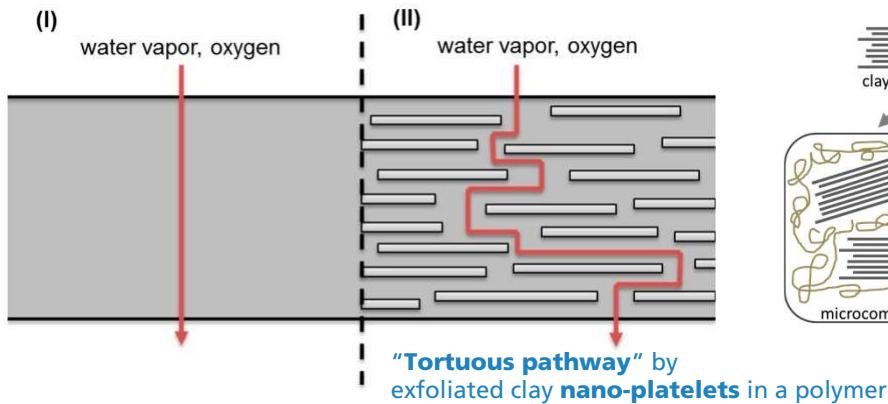
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Barrier Concepts: „Mono-material“ Multi-layer



■ Nano-composite Coatings



K. Müller et al., Nanomaterials (Basel). 2017 Apr; 7(4): 74

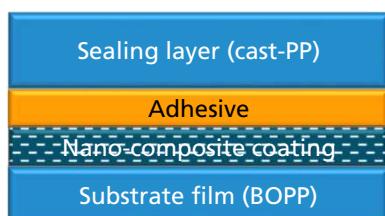


Barrier Concepts: „Mono-material“ Multi-layer



■ Nano-composite Coatings: Biggest Challenges

- Homogeneous dispersion for good surface properties and transparency
- Exfoliation of nanoparticles for nano-metric thickness of platelets and maximum possible surface
- Orientation of platelets for gas barrier → Alignment by shearing during coating



Designed for Recycling: > 90% Polyolefin
„Mono-material“ Multi-layer



© Fraunhofer



Barriflex - Enhanced Performance of Flexible Plastic Materials by Innovative Nanotechnologies for Food Packaging and Technical Applications (2019-2020)



Barrier Concepts: „Mono-material“ Multi-layer



■ Nano-composite Coatings: Biggest Challenges



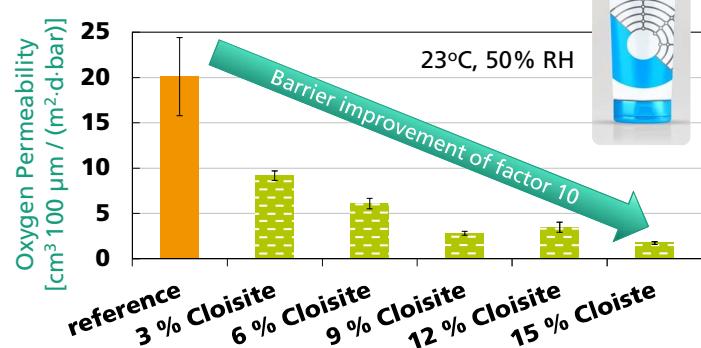
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Barrier Concepts: „Mono-material“ Multi-layer



■ Nano-composite Coatings: Results



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N. Brzosoka, E. Kucukpinar et al., Polymers 2019, 11, 1410

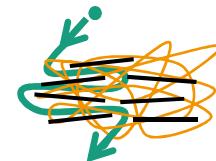
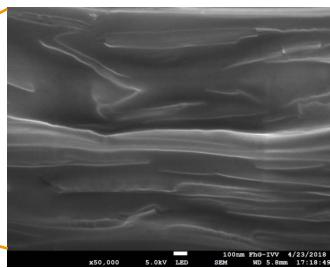
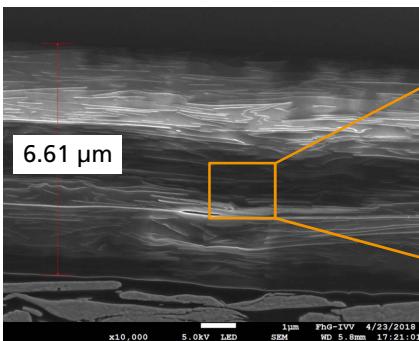
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Barrier Concepts: „Mono-material“ Multi-layer



■ Nano-composite Coatings: Results

- Exfoliation of nanoplatelets in Wheylayer → Orientation during coating process
- Roll-to-roll **reverse gravure** pilot coatings and process optimisation



Tortuosity effect
maximised by optimal
dispersion and
orientation

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K. Müller, M. Jesdziszki, M. Schmid, Hindawi Journal of Nanomaterials Volume 2017



Summary and Outlook



- Requirement: Designing products and packaging for recyclability
- Challenges and Solutions:
 - Mono-material based multi-layers: Polyolefin (BOPE, BOPP) based packaging systems are more feasible due to their proven sorting and recycling technologies at industrial scale
 - Low oxygen barrier performance: Nano-functionalisation enhances the barrier!
 - Nanocomposite coatings increase the oxygen barrier performance and make it possible to use the polyolefin based „Mono-material“ Multi-layers for flexible packaging
 - Fraunhofer IVV has the required coating processes and analytics for the application of functional barrier coatings, including exfoliation and orientation of the nano-particles
- Next Steps: Further production of novel mono-material multi-layers with other types of substrates, and shelf-life tests for sensitive food products

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„Thank you“ for making plastics sustainable!



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ANNEX III



Food Compliance in the EU

INL - International Iberian Nanotechnology Laboratory,
Braga



Food Contact Compliance in the EU

- The Food Contact Material's Regulatory landscape in the EU
- Framework Regulation (EC) No 1935/2004 on Food Contact Materials
- Regulation (EC) No 2023/2006 on Good Manufacturing Practice (GMP)
- Regulation (EU) No 10/2011 on Plastic Food Contact Materials
 - Scope
 - Union List of authorised substances for FCMs
 - Testing for Compliance
- Preparation of a Declaration of Compliance (DoC)
- Non-Harmonized Legislation
- Steps to Achieving Compliance in the EU



ANNEX IV



JOA's R2R Nano-patterning Processes and Offered expertise

JOANNEUM RESEARCH, Graz

JOA's R2R nano-patterning processes and offered expertise

<p>JOANNEUM RESEARCH is a non-profit research institute headquartered in Graz (Austria). At the institute "Materials", located in Weiz, cutting edge technologies and methods that are based on miniaturization, integration and materials optimization are used.</p>
<p>In the photolithography process, light is used to transfer a geometric pattern through a mask to a photosensitive resist on a substrate. The main limitation of this method is the wavelength of the used light, which imposes the geometrical size of fabricated structures - hence only structures down to 1µm are possible. A higher resolution is gained by means of nanoimprint lithography. Here, a geometric pattern is created by the mechanical deformation of the imprint resist. That means, a patterned stamp is pressed into the liquid resin and the resin is then hardened either by UV light or heat. Hence, the nanoimprint lithography is an advanced method for creating patterns down to the nanometer range at a low cost.</p>
<p>At Joanneum Research Materials in Weiz, the nanoimprint lithography process is up-scaled in a R2R-nanoimprint-machine. With this machine it is possible, to continuously produce large-area flexible, patterned substrates at low cost. In this video, the continuous R2R process is shown:</p>
<p>First, a flexible plastic film substrate is coated with a UV curable resin. Then, the coated substrate is guided to the imprinting station. Here a stamp (also called shim) with the desired surface pattern is pressed into the liquid resin. The shim is usually a nickel or polymer sheet with excellent anti-sticking properties which is wrapped around the imprinting roller. During the whole contact time between the stamp and the resin, the resin hardens by UV light. Finally, the substrate with the imprint is demolded from the stamp and wound up.</p>
<p>One key prerequisite is the adjustability of the imprint resin towards the targeted application scenario. At Joanneum Research Materials diverse UV-curable resins that are mainly based on polyurethane acrylates are developed. This NILcure® resin portfolio offers a wide range of possibilities, and the resin can be tailored in terms of mechanical, chemical and optical properties.</p>
<p>These chemically adjusted structured foils achieve for instance drag reducing surfaces, antireflecting or hydrophobic surfaces. Or the foils can just be decorative or used in the lighting sector when they are structured with micro-optics.</p>
<p>The expertise offered by Joanneum Research Materials, as member of the ESNA association, includes:</p> <ul style="list-style-type: none"> • UV- imprint resin (NILcure®) formulation and testing. In particular, formulation and batch/R2R testing of tailored imprint and UV resins. Tailoring of mechanical (e.g. elasticity), optical (e.g. refractive index), surface chemical (e.g. hydrophilicity/hydrophobicity) and reliability (e.g. weathering resistance) properties. • R2R-micro/nanoimprinting. In particular, nano- and micro (optical) structuring of PET, recycled PET, and cellulose based surfaces by the use of R2R UV-NIL machines with inline quality and process control. Applications include Optical films, Security features, Freeform micro-optics, Lighting, Displays & Photovoltaics, 3D printing, Microfluidics, Point-of-Care diagnostics, Lab-on-Foil, Biomimetic/bionic structures. • Micro/Nano characterization. In particular, characterization of materials and structures on the micro/nanoscale (surface, volume-cross sections): Keyence 3D laser scanning microscopy, AFM, SEM, XPS, etc.

ANNEX V



Flexible Organic and Printed Electronics from Lab to Fab

Aristotle University of Thessaloniki

Flexible Organic & Printed Electronics from Lab to Fab

**S. LOGOTHEΤΙΔΗΣ, S. KASSAVETIS, A. LASKARAKIS, Ch.
KAPNOPOULOS, Ch. GRAVALIDIS**

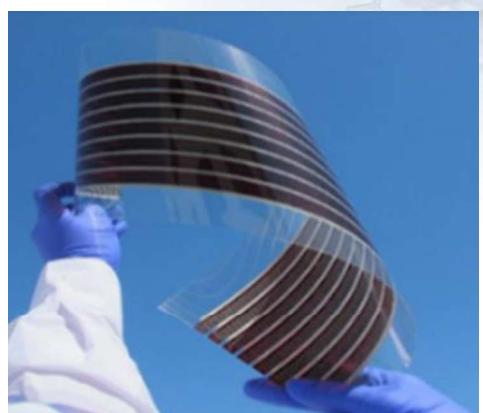
Nanotechnology Lab LTFN
Aristotle University of Thessaloniki,
Thessaloniki, K. Makedonia, GR-54124, Greece



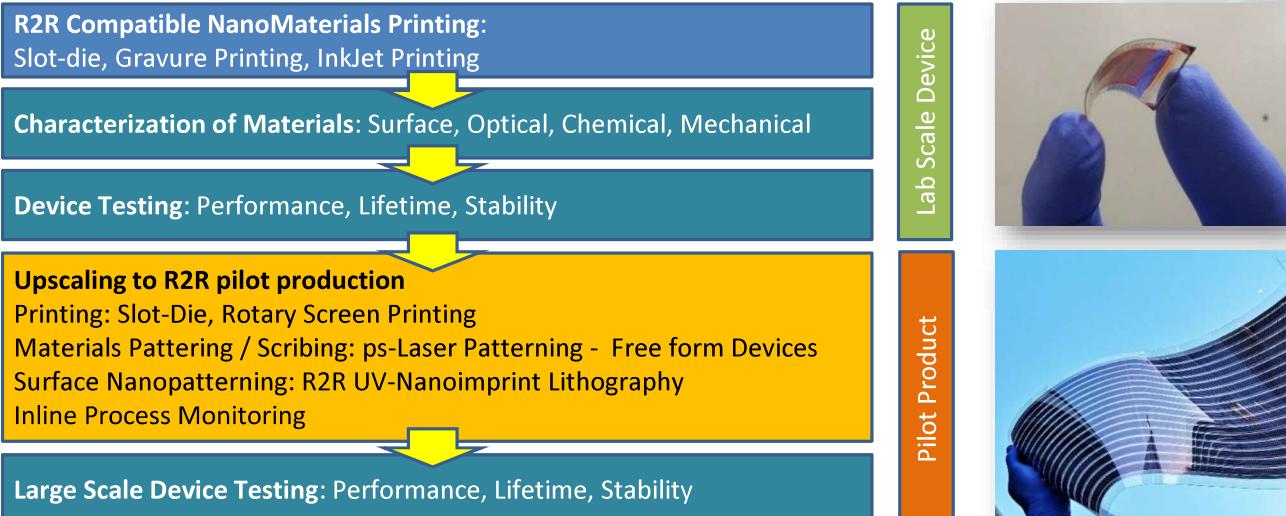
Content

Content

- Printing Techniques for Lab-Scale Organic Photovoltaics
- Upscaling in Sheet-to-Sheet (S2S) pilot line
- Upscaling in Roll-to-Roll (R2R) pilot line
- Summary



Flexible Printed Electronics Devices From Lab to Fab



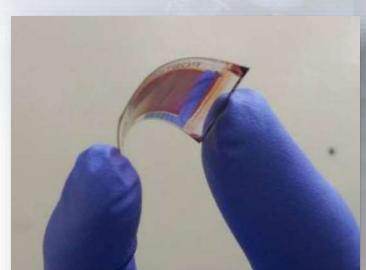
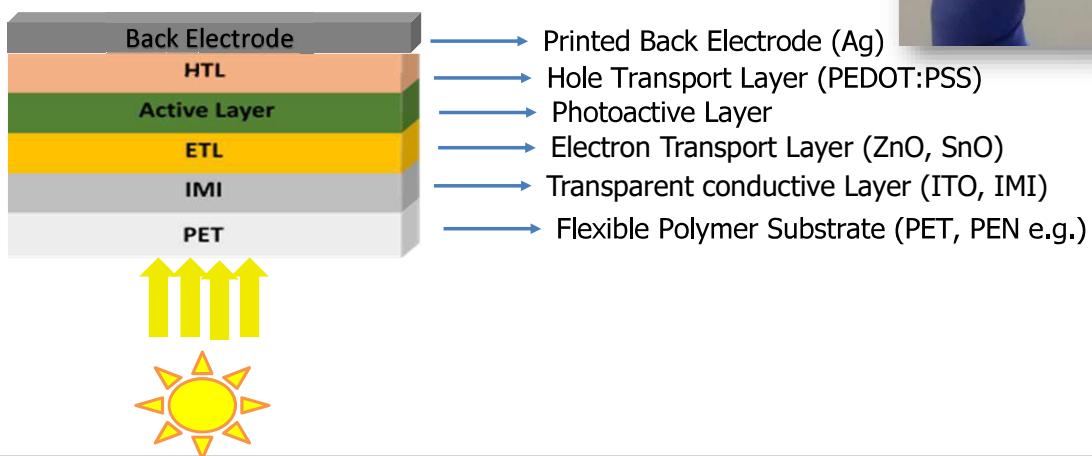
Organic & Printed Electronics from Lab to Fab



Digital fabrication of OEs & Bioelectronics nanomaterials, devices and systems

Flexible Printed Organic Photovoltaics

- Inverted Structure





Digital fabrication of OEs & Bioelectronics nanomaterials, devices and systems

Printing techniques

Screen Printing : The pattern is printed by filling the screen with an emulsion that is impervious to the coating solution in the areas where no print should appear.

The area of the printed pattern is kept open (without emulsion). The screen is then filled with coating solution and brought into proximity of the substrate.

The thickness d of the printed film is given by:

$$d = V_{\text{screen}} k_p c/p$$

where **V_{screen}** is the paste volume of the screen,
k_p is the pick-out ratio,
c is the material concentration in the ink (gr/cm³) and
p is the dry-film density (gr/cm³)



Digital fabrication of OEs & Bioelectronics nanomaterials, devices and systems

Printing techniques

Ink-Jet Printing : Mechanical compression of the ink through a nozzle (piezoelectric) or by heating the ink (and thus creating a pressure increase).

Droplets are electrostatically charged and accelerated towards the substrate by an electric field.

The thickness d of the printed film is given by: $d = N_d V_d c/p$



where **N_d: No of droplets per are (cm⁻²)**

V_d Volume of droplets,
k_p is the pick-out ratio,
c is the material concentration in the ink (gr/cm³) and
p is the dry-film density (gr/cm³)



Digital fabrication of OEs & Bioelectronics nanomaterials, devices and systems

Printing techniques

Slot Die Coating: Printing of stripes of material. Suitable for multilayer devices such as solar cell with overlapping stripes of different materials. The inks are feed to the Slot die head with a pump or a pressure system.

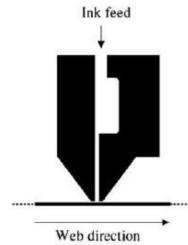
The thickness d of the printed film is given by: $d = \frac{f}{S_w} \frac{c}{p}$

where **f**: Flow rate ($\text{cm}^3 \text{ min}^{-1}$)

S_w Coated width (cm),

c is the material concentration in the ink (gr/cm^3) and

p is the dry-film density (gr/cm^3)



Upscaling on Sheet2Sheet Pilot line:

Hybrid Printing and Vacuum technologies for OE devices with Encapsulation technologies and

Solar Simulator for measuring the efficiency of Solar Cells and Organic Photovoltaics Modules





Upscaling to the manufacturing of pilot products

R2R Pilot-to-Production Line: Large area R2R manufacturing of Organic Electronic devices

Printing technologies: Slot-Die, Inkjet, Screen printing

In-line NanoPattening/Scribing Techniques: Ultra-fast Picosecond Laser Patterning, UV-NanoImprint Lithography

In-line metrology: Vis-UV Spectroscopic Ellipsometry, Raman Spectroscopy

Encapsulation of Devices



Inline nIR-Vis-UV Spectroscopic Ellipsometer

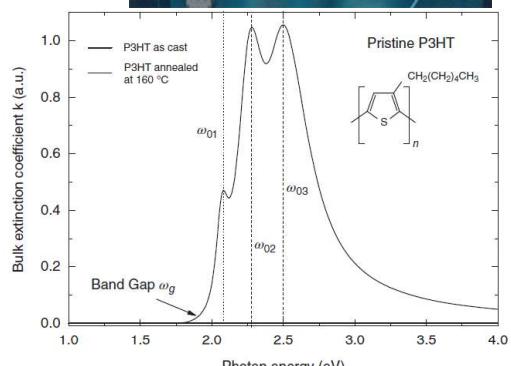
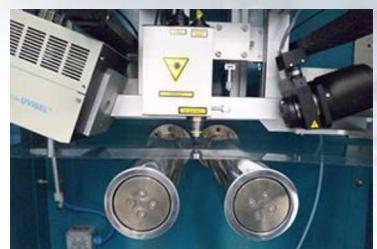
Inline Phase Modulated Spectroscopic Ellipsometer (0.6 – 6.5 eV)

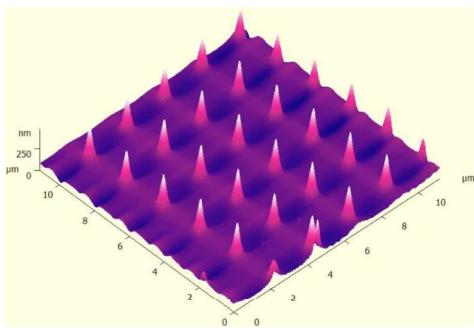
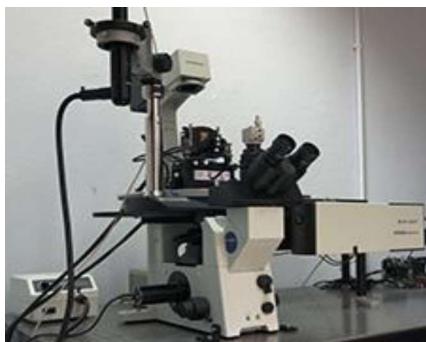
Thickness measurement (subnanometer resolution), optical properties (n , k), optical band gap, interface and roughness thickness, film composition and uniformity in depth and area.

- **Description / specification table :**

- Substrates: Glass, Polymer, Paper
- Sample Processing Range: up to 30 x 30 cm
- Measuring Range / Conditions: 0.6-6.5 eV / RT,

• **Use / Application examples :** Surface and thin film optical characterization, Printed Electronics, Semiconductors, dielectrics, polymers, organics, and metals





Surface Characterisation by Scanning Probe Microscopy

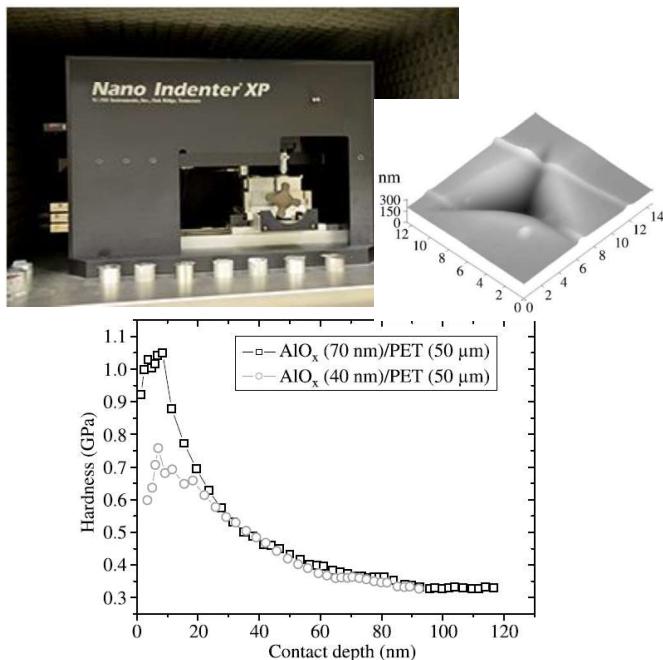
Scanning Probe Microscope (SPM) platforms for surface, Raman and Nano-mechanical characterization of nanomaterials (thin films, polymers, nanoparticles etc.) and Nano systems. The SPM platform supports:

- Atomic Force Microscope scanning in Contact, Tapping and non-Contact modes
- Scanning Tunnelling Microscopy
- Scanning Near-Field Optical Microscopy (532 nm Laser source)
- Atomic Force Acoustic Microscopy (Surface mechanical properties)
- Scanning in Liquid head and nitrogen environment
- Heating Stage (up to 300 °C)

Description / specification table :

- Substrates: Glass, Polymer, Paper
- Sample Processing Range: up to 50 mm x 50 mm
- Measuring Range / Conditions: 100 x 100 µm Scanning Surface Area

Use / Application examples : Surface Characterisation, Nanoparticles characterization, Printed electronics, Organic Photovoltaics (OPVs)



Nanomechanical Characterisation Nano Indenter XP / Scratch Tester

Nano-indentation and Scratch Testing apparatus for quasi-static and dynamic nano-mechanical characterisation

(Hardness, Elastic Modulus, Adhesion, Friction) of surfaces, soft (polymers) and hard thin films and bulk materials.

• Description / specification table :

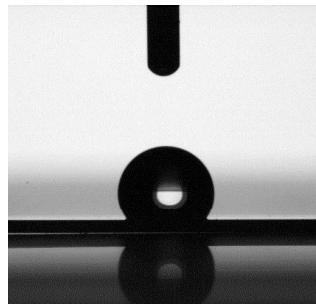
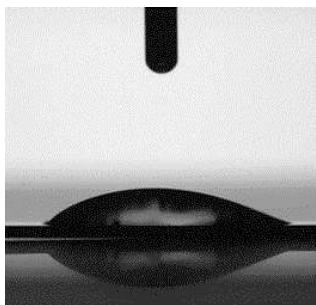
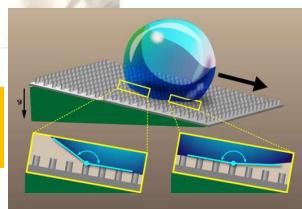
- Substrates: Various (Glass, Polymer, Paper)
- Sample Processing Range: up to 50 mm x 50 mm
- Measuring Range / Conditions: 100 mN maximum applied load, 20 µN lower normal applied load

• Use / Application examples : Nano indentation and Scratch Tests of bulk materials, nanocomposite materials, hard protective coatings and thin films, barrier coating for flexible electronics packaging, soft materials (polymers and organic layers) fibrous scaffolds, nanocomposite concrete.

Hardness of a AlO protective layer on top on PET substrate



Superhydrophilic &
Superhydrophobic Surfaces



Contact Angle System

for measuring contact angle and free surface energy. A CCD firewire camera (512x480) with telecentric zoom optics combined with LED based background lighting allows capturing images (image area 5.7 x 5.4 mm²) at frame intervals from 10 ms to 1000s.

Description / specification table :

- Sample Processing Range: 150x50 mm
- Measuring Range / Conditions:
 - 0 - 180° degrees
 - Frame interval: 40ms - 1000s
 - Inaccuracy: +/- 5 degrees
 - Curve fitting to Young Laplace equation, cycle, polynomial and Bashforth/Adams

Use / Application examples : Determination of surface or interfacial tension, contact angles, absorption or surface free energy of :

- Surfactants & Detergents, Emulsions
- Polymers, Papers, Films & Inks, Substrates
- Sprays, Paints & Coatings



Nanotechnology Lab LTFN (www.ltfn.gr)



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TESTBEDS

Nanotechnology Lab LTFN is equipped with 10 Pilot to Production Lines and several Testbed facilities in Organic & Printed Electronics and Photonics, Thin Film Technologies, Nanoengineering, Nanomedicine & 3D Bioprinting, Nanometrology & Optical Technology, Computational & Modelling at Nano- to Micro-Scale.

Organic & Printed Electronics

- **R2R Pilot & Production line:** Large area R2R manufacturing of Organic Electronic devices, equipped with Ultra-fast Laser scribing and in-line metrology. Main technologies used: Printing (Slot-Die, Inkjet, Screen printing), Ultra-fast Laser Patterning, Encapsulation module, Raman Spectroscopy & In-line Spectro-Ellipsometry
- **Sheet2Sheet Pilot line:** Hybrid Printing and Vacuum technologies for OE devices with Encapsulation technologies and Solar Simulator system
- **OVPD Cluster - Gas Transport Pilot line:** Scalable OVPD Pilot Line equipped with in-situ optical metrology systems (Raman Spectroscopy, Spectro-Ellipsometry) for high precision fabrication of OE devices
- **Lab Scale Printing:** Printing techniques (S2S Gravure, Slot-Die, Inkjet) for Digital fabrication of OEs & Bioelectronics nanomaterials, devices and systems
- **Ex-situ Laser System:** High energy laser systems for ultra fast processes (laser ablation, laser annealing, patterning) for fabrication and functionalization of novel nanomaterials and nanoparticles

Thin Films, Nanomaterials & Nanoengineering

- **CVD Pilot line:** Thermal and Plasma CVD Pilot line for Graphene and 2D nanomaterials growth in 6" wafers. The system is equipped with real-time optical monitoring techniques (Vis-UV SE and Raman) for in-situ characterization and process optimization
- **Pilot lines:** equipped with state-of-the-art PVD techniques (Magnetron Sputtering, HIPIMS, Thermal, Electron-gun Evaporation) for

ABOUT DIGITAL INNOVATION HUB
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DIH MENU

TESTBEDS EXPERTISE

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COLLABORATIVE RESEARCH

EDUCATION & SKILLS DEVELOPMENT

ACCESS TO FUNDING

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DIGITAL INNOVATION HUB

ACTIVE PROJECTS



Flexible Organic & Printed Electronics from Lab to Fab

**S. LOGOTHEΤΙΔΙΣ, S. KASSAVETIS, A. LASKARAKIS, Ch.
KAPNOPOULOS, Ch. GRAVALIDIS**

Nanotechnology Lab LTFN
Aristotle University of Thessaloniki,
Thessaloniki, K. Makedonia, GR-54124, Greece



ANNEX VI



Mechanical Recycling for Flexible Plastics

Centre Technique Industriel
de la Plasturgie et des Composites, Bellignat



Centre Technique Industriel
de la Plasturgie et des Composites

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Plastiques et Composites.**

Technical teaching course

Mechanical recycling for flexible plastics

ct-ipc.com



Recycling, what is it ?

Definition

'Recycling' means any recovery operation by which waste materials are reprocessed into products, materials or substances whether for the original or other purposes. It includes the reprocessing of organic material but does not include energy recovery and the reprocessing into materials that are to be used as fuels or for backfilling operations.

Article 3(17), Waste Framework Directive

Packaging recyclability, what is it ?

Definition

A global definition of “recyclability” was developed by [The Association of Plastics Recyclers](#) (APR) and [Plastics Recyclers Europe](#) (PRE) in 2018.

4 conditions are required for a packaging to be considered as “recyclable” :



The product must be made of **plastic that is collected** for recycling, has market value, and/or is supported by a legislatively mandated program.



The product must be **sorted and aggregated into defined streams** for recycling processes.



The product can be **processed and reclaimed/recycled** with commercial recycling processes.



The recycled plastic becomes a raw material that is **used in the production of new products**.

Source: RecycClass website, <https://recyclass.eu/recyclability/definition/>

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Recycling technologies

Recycling technologies can be classified under two categories:

Mechanical recycling

Selective dissolution



Physical Recycling

Technologies and processes whereby plastic waste materials are recycled back into plastics without altering the chemical structure of the materials.



Chemical Recycling

Technologies and processes whereby clean plastic streams undergo a chemical process which includes a purification step, to potentially obtain virgin-like polymers to be used in new plastic articles.

Pyrolysis

Gasification

Hydro-cracking

Depolymerisation

Selective dissolution

Source: PRE website, <https://www.plasticsrecyclers.eu/plastic-recycling/how/>

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A complete service offer at IPC



MATERIAL SELECTION

- Materials substitution or research
- Materials comparison

FORMULATION AND COMPOUNDING

- Specific formulations development
- Integration of additives, reinforcements...
- Sampling from some kilograms to tons
- Post-consuming or post-production regeneration
- Standardized samples, injection, lab testing and analysis

RECYCLABILITY EVALUATION

- Rigid and films// Commodities (PE, PP) or technical (PA, PPS, PEEK...)
- Material preparation (sorting, grinding), washing, densification, granulation, filtration, post-transformation, characterization
- Recyclability evaluation based on protocols (RecyClass, COTREP...)

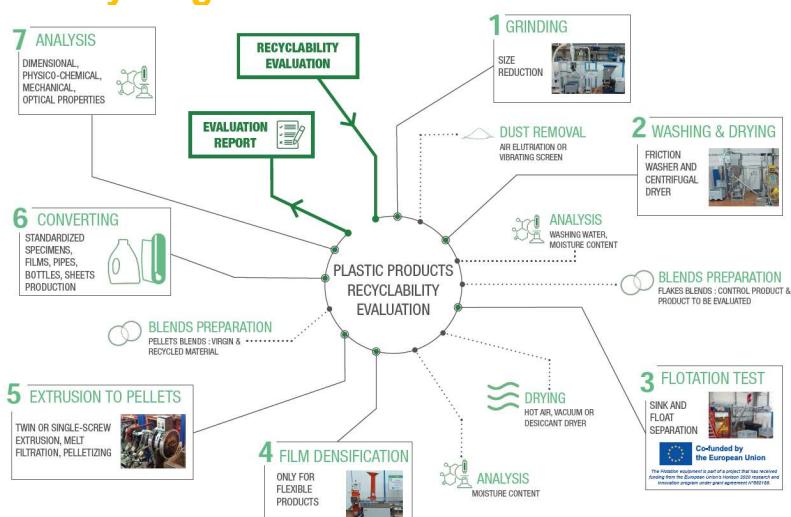
EXTRUSION PROCESS

- Profiles, films, sheets, plates, containers...

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Mechanical recycling at IPC



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A unique recycling line in France : REMIX pilot line

Objectives:

- Give access to a technological platform that is representative of industrial means.
- Evaluate recyclability for both flexible and rigid plastic products
- A modular and upgradable pilot line to address different kind of materials

Evaluation process:

- Reproduction on a pilot scale of sorting and treatment of plastic waste (grinding, washing, sifting, metallic parts removal, drying, sorting)
- Regeneration and formulation (compounding, compatibilization, reactive extrusion / Custom formulation to meet specifications)
- Injection and extrusion process (profiles, plates, films, containers,..)

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REMIX : Size reduction, sorting and washing unit



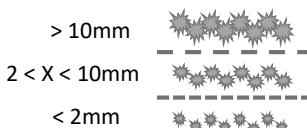
**Knives grinding unit
(Rigid Materials)
5 mm Grid**



2 Grids vibrating sieve



**Shredder
(Flexible materials)
Grilles 10, 20, 30 mm**



**SOREMA Washing /
drying unit**

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REMIX: Material separation - sorting

Density based separation



Flotation bath
(Static)



Flotation bath
(Dynamic with rollers)

Electrostatic separation



HAMOS KWS
*(Ferrous / non ferrous materials
separation)*

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REMIX: Homogenization and densification



Vertical or rotational
mixer



WANNER densifier and granulator
(for flexible materials)

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REMIX: Granulation / filtration

- Material homogenization using lamination and filtration (filter size 500µm, 300µm, 150µm, 125µm...)



bi-vis extruder



Filtration unit

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REMIX: Quality control (granulates / parts / samples)

➤ Parts Production / test samples

- Injection of standardized test samples
- Extrusion-inflation and films extrusion
- Extrusion of profiles (tubes,..)
- Extrusion-blow molding of containers



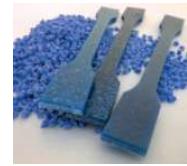
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REMIX: Laboratory evaluation on granulates, test samples

➤ Physico-chemical characterization

- Heavy metals detection
- Ash content
- Humidity rate
- Infrared analysis
- Rheological properties (MFI, viscosity...)
- Thermal properties (DSC, ATG,...)
- Optical properties



➤ Mechanical characterization

- Mechanical properties (tensile, bending, impact, tearing, perforation...)



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Recyclability certification

RecyClass

➤ IPC as Recognized Certification Body for RecyClass to deliver certificates based on an audit of products



➤ IPC as Recognized Laboratory for Recyclability evaluation based on RecyClass protocols on the following streams



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14

Recycled content certification

LABORATOIRE
NATIONAL
DE MÉTROLOGIE
ET D'ESSAIS



- IPC and LNE "French National Testing and Metrology Laboratory" developed in a joint effort a certification scheme that assess the quantity of Recycled content incorporated in products.
- This certification scheme has recently been approved by PolyCert Europe, giving French industry access to a certification scheme, compliant and harmonized with other European existing schemes
- This certification scheme consist of a two part audit:
 - Part 1: Global RPM (Recycled Plastic Materials) quantity used within a production site
 - Part 2: Specific quantities of RPM contained in a product / range of products



Company XXX has used 6750 tons of RPM in 2021



Contains 55 % of RPM

ANNEX VII



Life Cycle Assessment

Centre Technique Industriel
de la Plasturgie et des Composites, Bellignat



Centre Technique Industriel
de la Plasturgie et des Composites

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Life Cycle Assessment (LCA)

ct-ipc.com



Life Cycle assessment, what is it ?

Definition

ECO DESIGN

**Life Cycle
Assessment**



Eco Design: « taking the environment into account during the design or improvement phase of a product (good or service), by considering its entire life cycle »

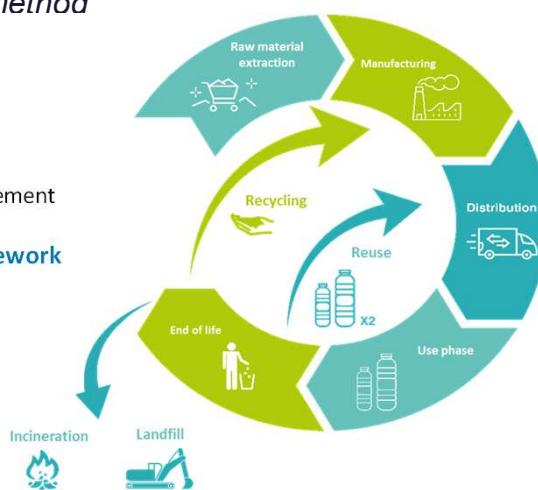
LCA : « compilation and evaluation of the inputs, outputs and potential environmental impacts of a product system over its life cycle »

Life Cycle assessment, what is it ?

A *normalized* method

ISO 14040 : 2006

Environmental management
life cycle assessment
principles and framework



ISO 14044 : 2006

Environmental management
life cycle assessment
requirements and guidelines

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3

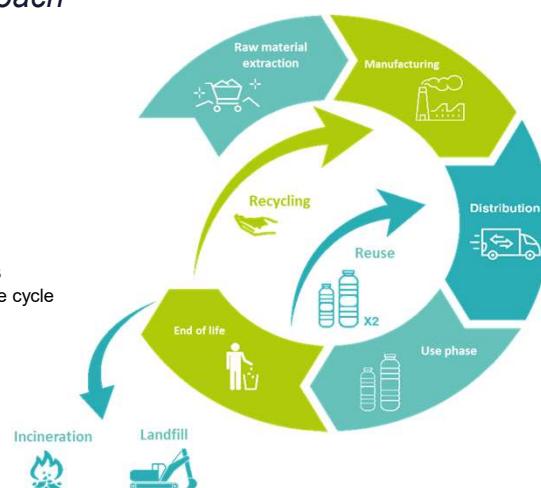
Life Cycle assessment, what is it ?

A *holistic* approach



1 – Multi-steps

Takes into account all life cycle stages



2 – Multi-indicators

Calculates impacts on several
indicators to avoid impact
shifting



3 – Multi-systems

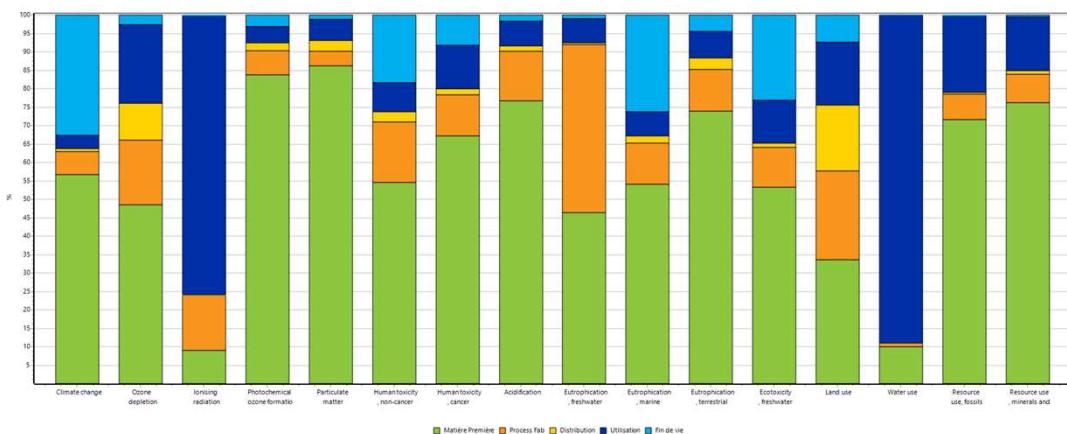
takes into account the
product's environment and its
life cycle

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4

Why making an LCA ?

Identifying

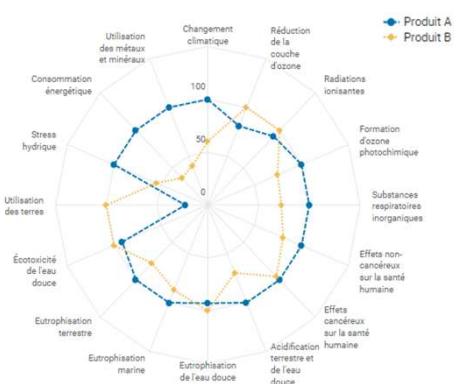
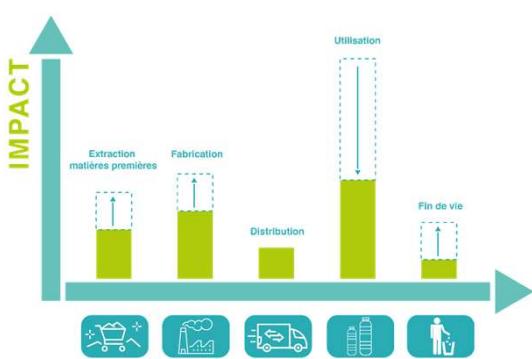


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Why making an LCA ?

Comparing and avoiding impact shifting



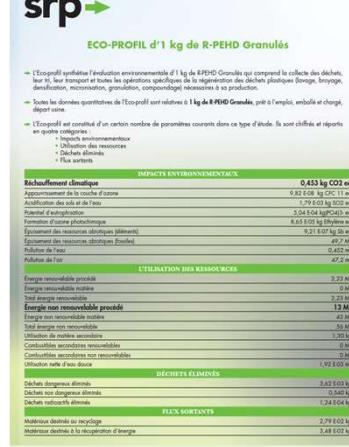
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Why making an LCA ?

Communicating, using an Environmental Product Declaration

CRITICAL REVIEW

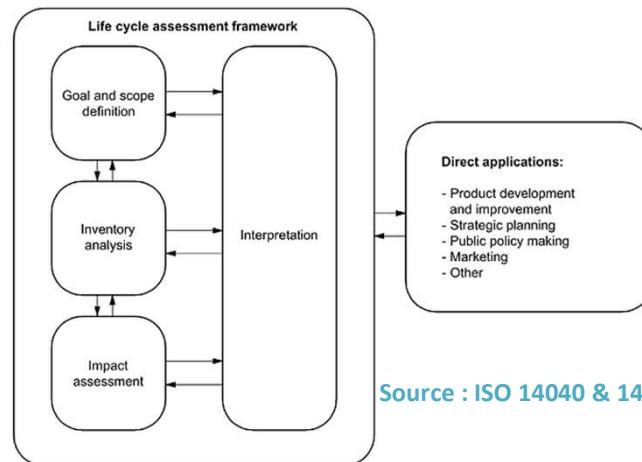


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How to make an LCA ?

LCA methodology



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8

How to make an LCA ?

1. Goal and scope definition

- Defining the objective behind making the LCA
- Defining the **functionnal unit (FU)** and the **reference flow**

The functional unit

« Quantified performance of a product system for use as a reference unit » (ISO 14040 :2006)

Example: Orange Juice Brick

FU = "Contain, protect, store 250 ml of orange juice during its lifetime"

RF = One 250ml capacity brick

How to make an LCA ?

1. Goal and scope definition

- Defining **system boundarie**

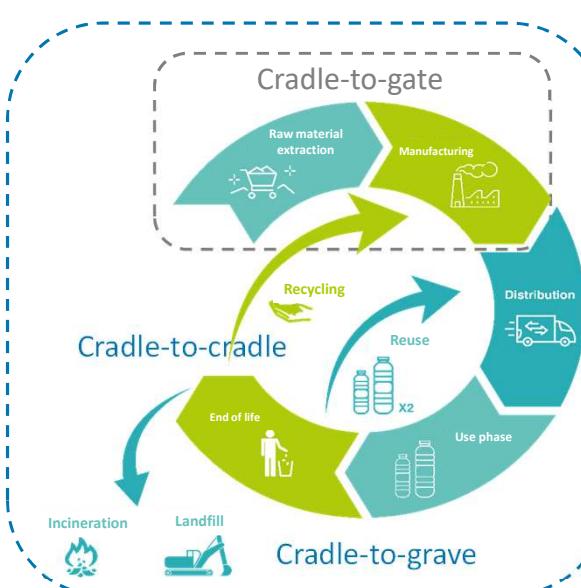
System boundaries

« Set of criteria specifying which unit processes are part of a product system » (ISO 14044 :2006)

Gate-to-gate is a partial LCA looking at only one value-added process in the entire production chain.

Cradle-to-gate is an assessment of a partial product life cycle from resource extraction (cradle) to the factory gate (i.e., before it is transported to the consumer). The use phase of the product is omitted in this case. The disposal phase can be included depending on the objective of the analysis

Cradle-to-cradle is a specific kind of cradle-to-grave assessment (the full Life Cycle Assessment from resource extraction ('cradle') to use phase and disposal phase ('grave')), where the end-of-life disposal step for the product is a recycling process.



 IPC

LCA tools at IPC

 SimaPro

- World leader LCA software
- Proposed for any type of application and for any field of activity
- Suitable for complex and simple LCA models
- Several databases including **Ecoinvent V3.9**, covering over 15,000 data
- Different methods of impact assessment, including the **PEF method (Product Environmental footprint)** developed and recommended by the EU commission

 C3R'
IMPACT
L'ACV PAR IPC

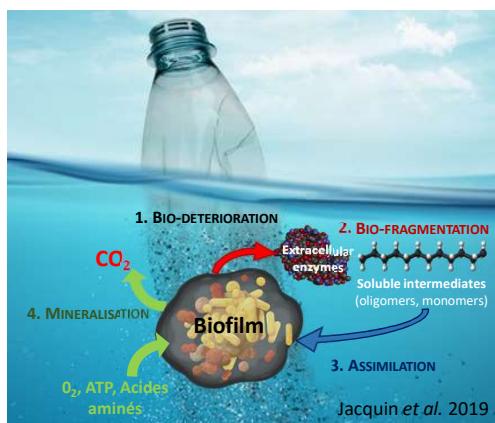
- **Simplified LCA software developed by IPC**
- Intended for **Plastic and Composite industry**
- Uses Simapro as a background for calculation
- Includes data from Ecoinvent 3.9, specific data collected by IPC, and SRP (plastic recyclers union in France)
- Uses **PEF calculation method**

ANNEX VIII



Presentation of the biodegradation experiments carried out within IPC

Centre Technique Industriel
de la Plasturgie et des Composites, Bellignat

*Technical teaching course***Presentation of the biodegradation experiments
carried out within IPC**ct-ipc.com**Biodegradation of a plastic by the plastisphere**

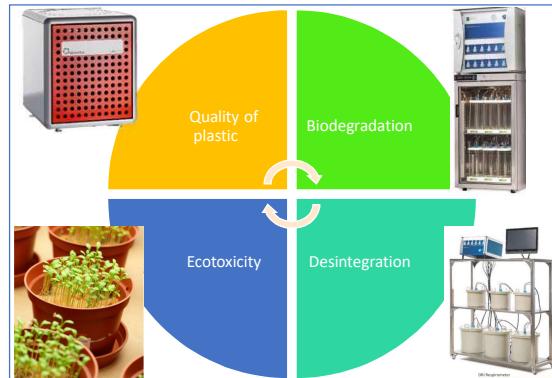
Conversion of **organic carbon** into **biogas** and **biomass** associated with **microbial community activity**

Study of biodegradation in industrial or domestic composting conditions

Standards EN 13 432 and NF-T51800

➤ The standards require a complete study of biodegradation

- Physico-chemical parameters of the material
- Organic carbon mineralization
- Study of polymer fragmentation
- Impact on plant toxicity



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3

Study of the biodegradation of materials in domestic/industrial compost



- Temperature: 58°C
- Biodegradation (6 months max):
 - Reference : 70% in 45 days
 - Test: 90% absolute or relative to cellulose
- Disintegration (3 months max):
 - NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- Ecotoxicity
 - OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control



- Temperature: 25°C
- Biodegradation (12 months max):
 - Reference : 70% in 90 days
 - Test: 90% absolute or relative to cellulose
- Disintegration (6 months max):
 - NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- Ecotoxicity
 - OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control



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4

Study of the biodegradation of materials in domestic/industrial compost



INDUSTRIAL

- **Temperature:** 58°C
- **Biodegradation (6 months max):**
 - Reference : 70% in 45 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration (3 months max):**
 - NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- **Ecotoxicity**
 - OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control



HOME

HOME

- **Temperature:** 25°C
- **Biodegradation (12 months max):**
 - Reference : 70% in 90 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration (6 months max):**
 - NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- **Ecotoxicity**
 - OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control



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Study of the disintegration of materials in domestic/industrial compost



INDUSTRIAL

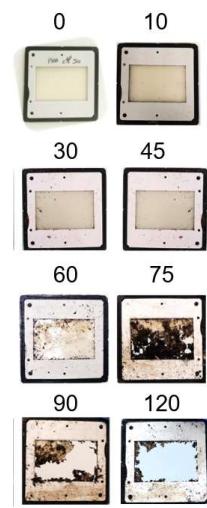
- **Temperature:** 58°C
- **Biodegradation (6 months max):**
 - Reference : 70% in 45 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration (3 months max):**
 - NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- **Ecotoxicity**
 - OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control



HOME

HOME

- **Temperature:** 25°C
- **Biodegradation (12 months max):**
 - Reference : 70% in 90 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration (6 months max):**
 - NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- **Ecotoxicity**
 - OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control



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Study of the biodegradation of materials in domestic/industrial compost

 TÜV AUSTRIA INDUSTRIAL	 TÜV AUSTRIA HOME
<ul style="list-style-type: none">› Study time: < 6 months› Temperature: 58°C› Biodegradation:<ul style="list-style-type: none">▪ Reference : 70% in 45 days▪ Test: 90% absolute or relative to cellulose› Disintegration:<ul style="list-style-type: none">▪ NF ISO 20 200 or ISO 16929▪ <10% of DM after sieving at 2 mm› Ecotoxicity<ul style="list-style-type: none">▪ OECD 208▪ Between 14 and 21 days after germination of 50% of the control seedlings▪ Germination rate > 90% relative to control	<ul style="list-style-type: none">› Study time: < 12 months› Temperature: 25°C› Biodegradation:<ul style="list-style-type: none">▪ Reference : 70% in 90 days▪ Test: 90% absolute or relative to cellulose› Disintegration:<ul style="list-style-type: none">▪ NF ISO 20 200 or ISO 16929▪ <10% of DM after sieving at 2 mm› Ecotoxicity<ul style="list-style-type: none">▪ OECD 208▪ Between 14 and 21 days after germination of 50% of the control seedlings▪ Germination rate > 90% relative to control



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7

Study of the biodegradation of materials in the marine environment

ASTM D 6691

- › The standards require a complete study of biodegradation
 - Physico-chemical parameters of the material
 - Organic carbon mineralization
 - Study of polymer fragmentation
 - Impact on plant toxicity



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8

Study of the biodegradation of materials in the marine environment

ASTM D 6691

- **Temperature: 30°C**
- **Biodegradation:**
 - Reference : 70% in 45 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration:**
 - Incubation according to ASTM D6691
 - Test over 84 days, < 10% of DM after sieving at 2 mm
- **Ecotoxicity**
 - OECD 202
 - Study on the leachate obtained after 6 months of incubation at 30°C
 - No effect on daphnia magna



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9

Study of the biodegradation of materials in the marine environment

- **Temperature: 30°C**

- **Disintegration:**

- Incubation according to ASTM D6691
 - Test over 84 days, < 10% of DM after sieving at 2 mm

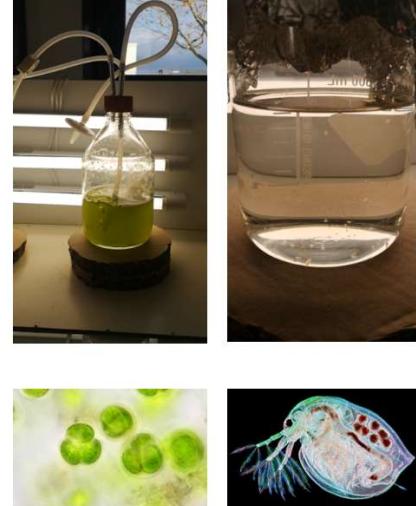


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10

Study of the ecotoxicity of materials in the marine environment

- **Temperature:** 30°C
- **Biodegradation:**
- Reference : 70% in 45 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration:**
- Incubation according to ASTM D6691
 - Test over 84 days, < 10% of DM after sieving at 2 mm
- **Ecotoxicity**
- OECD 202
 - Study on the leachate obtained after 6 months of incubation at 30°C
 - No effect on daphnia magna



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11

Study of biodegradation according to the type of medium

- OK compost
INDUSTRIAL
- **Biodegradation (6 months max):**
- Reference : 70% in 45 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration (3 months max):**
- NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- **Ecotoxicity**
- OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control

- HOME
OK compost
- **Biodegradation (12 months max):**
- Reference : 70% in 90 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration (6 months max):**
- NF ISO 20 200 or ISO 16929
 - <10% of DM after sieving at 2 mm
- **Ecotoxicity**
- OECD 208
 - Between 14 and 21 days after germination of 50% of the control seedlings
 - Germination rate > 90% relative to control 0

- ASTM D 6691
OK bio-degradable
MARINE
- **Temperature: 30°C**
- **Biodegradation:**
- Reference : 70% in 45 days
 - Test: 90% absolute or relative to cellulose
- **Disintegration:**
- Incubation according to ASTM D6691
 - Test over 84 days, < 10% of DM after sieving at 2 mm
- **Ecotoxicity**
- OECD 202
 - Study on the leachate obtained after 6 months of incubation at 30°C
 - No effect on daphnia magna

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12