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FlexFunction2Sustain

Open Innovation Ecosystem for Sustainable Nano-functionalized Flexible Plastic and Paper Surfaces and Membranes

Starting date of the project: 01/04/2020 Duration: 48 months

= Deliverable D4.1 =

Catalogue of physicochemical and functional characterization services

Dissemination level		
PU	Public	X
PP	Restricted to other programme participants (including the	
PP	Commission Services)	
RE	Restricted to a group specified by the consortium (including the	
KE	Commission Services)	
СО	Confidential, only for members of the consortium (including the	
	Commission Services)	



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Executive Summary

Deliverable D4.1 presents the Catalogue of Physicochemical and Functional Characterisation Services. This catalogue describes technological support services provided by the FlexFunction2Sustain project partners for the development of nano-functionalised plastic and paper-based flexible materials and membranes. The catalogue is based on the data of the methodologies provided to meet the characterisation needs of the different Case Studies of the project, as described on the FlexFunction2Sustain website (https://flexfunction2sustain.eu/use-cases) and in the FlexFunction2Sustain handbook (Deliverable D8.10).

The methodology for the selection of services is based on the analysis of market needs, the available technical offer and the experience gained from the analysis and implementation of the Use Cases. The characterisation services described here will be finally integrated in the FlexFunction2Sustain Service Catalogue (D6.4), in which different technical services will be combined to address complex industrial challenges related to nano-functionalised plastic and paper-based flexible materials and membranes.

The catalogue firstly presents the services resulting from improved FlexFunction2Sustain physicochemical and functional characterisation facilities with the ambition to address the specific needs of the development of new sustainable nano-functionalised materials. The stability of these materials is generally reduced compared to current commercial solutions and require special attention in the quality control of the functional properties exhibited by the surfaces based on them.

Finally, all services provided are described in detail and classified into seven categories:

- Gas Permeation Barrier testing
- Electrochemical properties
- Mechanical properties, integrity, and durability tests
- Optical properties
- Surface and microstructure analysis
- Thermal and rheological properties
- Test Specimen Preparation

The catalogue of characterisation services is subject to continuous updating to integrate new methodologies when they become available. This document represents that state of 30th June 2021.

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1. Introduction

D4.1 presents a living digital deliverable, the Catalogue of Physicochemical and Functional Characterisation Services detailing the technical capabilities of the FlexFunction2Sustain Facility Cluster 7 (Physicochemical and Surface Characterisation and Functional Performance Verification Facilities), which will be hosted on the FlexFunction2Sustain Project website (https://flexfunction2sustain.eu) and will be continuously updated as new services become available.

This section presents the context of the Catalogue of Physicochemical and Functional Characterisation Services in the FlexFunction2Sustain project, the objectives sought with the release of the catalogue and, finally, the methodology used to prepare it.

1.1. Catalogue of Physicochemical and Functional Characterisation Services in the context of FlexFunction2Sustain project

D4.1 "Catalogue of Physicochemical and Functional Characterisation Services" is a public document of the FlexFunction2Sustain project, delivered in the context of WP4 "Facilities for Application Verification, Testing, Safety Assessment and Pre-Certification", Task 4.1 "Characterisation and Quality Verification procedures for functional properties of nano-functionalised surfaces".

This catalogue is part of a series of three service catalogues to be created in FlexFunction2Sustain WP4 which, in addition to D4.1, includes D4.2 "Catalogue of services for performance verification in application" and D4.4 "Catalogue of services for safety and regulatory compliance testing". All three together represent the technological offer of the Facilities for Characterisation and Quality Control in FlexFunction2Sustain. D.4.1 is closely linked to two other deliverables, the FlexFunction2Sustain Project Handbook (Deliverable D8.10) and the FlexFunction2Sustain Service Catalogue (D6.4), whose implementation at the end of the project (M42) will benefit from the service catalogues developed in WP4 and from the lessons learned from the interaction between the Facilities for Characterisation and Quality Control and the development of the Use Cases in WP5.

1.2. Objectives of the deliverable

This service catalogue aims to provide a clear and comprehensive overview of the Physicochemical and Functional Characterisation services available within the FlexFunction2Sustain Open Innovation Test Bed (OITB). Herein described characterisation methods and services are evaluated and aligned with customer needs through the industrial Use Cases of the FlexFunction2Sustain project. Furthermore, Pilot Case customers may receive discounted access after successfully applying to the open calls for Pilot Case projects (https://flexfunction2sustain.eu/open-calls).

The Single Entry Point (SEP) company will actively promote and sell the characterisation services described herein as integral part of innovation projects that will be conducted by the FlexFunction2Sustain OITB.

1.3. Methodology for producing the catalogue

The methodology for the selection of services is based on the analysis of market needs, the available technical offer and the experience gained from the analysis and implementation of the Use Cases. In brief, the Use Cases and Letters of Interest received during the application phase were analysed and

the information regarding the characterisation tests required to implement them was compiled in a document based on the different methodologies available at the FlexFunction2Sustain partners' facilities (Facility Cluster 7 Service Portfolio). The specifications of each of the potential services were then summarised to allow a quick interpretation of the technological capabilities available and to make the catalogue more attractive to potential users. For each of the services the following elements were elaborated:

- Service category
- Name of the service
- Functionalities provided
- Technical specifications
- Examples of application in the Use Cases

After this first stage, the FlexFunction2Sustain partners providing Physicochemical and Functional Characterisation services were asked to check the accuracy of the compiled information. Finally, all this information was uploaded to the FlexFunction2Sustain website. Figure 1 summarises the three stages followed to compile the catalogue.



Figure 1. Steps for the development of the FlexFunction2Sustain Physicochemical and Functional Characterisation Service catalogue

1.3.1. Example of the process for a single service

In the Facility Cluster 7 Portfolio of Services, the methodologies available for the determination of barrier properties of flexible substrates were described in the following tables:

Table 1. Methodology 1

Test Category	Gas permeation barrier properties
Test	Water Vapor Transmission Rate, WVTR
Application in Use Cases	Assessment of the water vapor transmission rate of flexible barrier materials used in electronic displays (UC4)
Partner (Service Provider)	FHG-IAP

Available equipment	Optical Calcium test
Relevant standards	
Sample dimensions (range)	50 mm × 50 mm
	WVTR 10 ⁻⁶ – 10 ⁻³ g/m ² /d
Measuring range / conditions	Test conditions: 23 °C / 50 % RH; 38 °C / 90 % RH, 60 °C / 90 % RH, 85 °C / 85 % RH
	Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	1 week to 3 months depending on barrier quality
Processing capacity	5 samples per test, parallel testing possible
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4–7
Invoicing model (€/sample or €/h)	€/sample

Table 2. Methodology 2

Test Category	Gas permeation barrier properties
Test	Water Vapor Transmission Rate, WVTR
Application in Use Cases	Assessment of the water vapor transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)
Partner (Service Provider)	FHG-IVV
Available equipment	MOCON Aquatran™
Relevant standards	DIN EN ISO 15106-3
Sample dimensions (range)	2 A4 sheets
Measuring range / conditions	$5 \times 10^{-5} - 1 \times 10^{-2} \text{ g/m}^2/\text{d}$ Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	1 week to over 3 months for ultra-high barrier materials
Processing capacity	2 samples at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 3. Methodology 3

Test Category	Gas permeation barrier properties
Test	Water Vapor Transmission Rate, WVTR
Application in Use Cases	Assessment of the water vapor transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2)
Partner (Service Provider)	FHG-IVV
Available equipment	Gravimetric measurement
Relevant standards	DIN 53 122-1
Sample dimensions (range)	2 A4 sheets
Measuring range / conditions	0.5 – 250 g/m²/d Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	2 – 12 weeks
Processing capacity	4 samples at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 4. Methodology 4

Test Category	Gas permeation barrier properties
Test	Water Vapor Transmission Rate, WVTR
Application in Use Cases	Assessment of the water vapor transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)
Partner (Service Provider)	FHG-FEP
Available equipment	Sempa HiBarSens 2.0 HT
Relevant standards	
Sample dimensions (range)	1 A4 sheet; 1 sample 200 mm × 200 mm
Measuring range / conditions	$1\times 10^{-5}-1\times 10^{-2}~g/m^2/d$ Temperature: 10 – 85 °C Relative humidity: 60 – 95 % Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	Between 1 to 10 days
HR profile - Technician - Engineer / graduate	Technician

- Scientist	
TRL	4-7
Invoicing model	€/sample

Table 5. Methodology 5

Test Category	Gas permeation barrier properties
Test	Water Vapor Transmission Rate, WVTR
Application in Use Cases	Assessment of the water vapor transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)
Partner (Service Provider)	FHG-FEP
Available equipment	Brugger WDDG
Relevant standards	ISO 15106-03
Sample dimensions (range)	1 A4 sheet; 2 circular samples with a diameter of 112 mm
Measuring range / conditions	1 × 10 ⁻³ to 10 g/m ² /d Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	1 day
Processing capacity	2 samples at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 6. Methodology 6

Test Category	Gas permeation barrier properties
Test	Water Vapor Transmission Rate, WVTR
Application in Use Cases	Assessment of the water vapor transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)
Partner (Service Provider)	AUTH
Available equipment	MOCON AQUATRAN MODEL1
Relevant standards	ISO 15106-3
Sample dimensions (range)	Test Sample Sizes: Films - 50 cm²; Packages - up to 3 litres per package
Measuring range / conditions	WVTR Range:

	5 ×10 ⁻⁴ – 5 g/m ² /d
	Sensor: AQUATRACE™
	Temperature Range: 5 - 50 ° C
	RH: films 100 %, RH Packages 100 % RH
	Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	1-3 weeks depending on the properties
Processing capacity	2 samples at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 7. Methodology 7

Test Category	Gas permeation barrier properties
Test	Water Vapor Transmission Rate, WVTR
Application in Use Cases	Assessment of the water vapor transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)
Partner (Service Provider)	INL
Available equipment	Elcometer 5100 Payne Permeability Cups
Relevant standards	ASTM E96/E96M - 10
Sample dimensions (range)	Maximum 30 cm ²
Measuring range / conditions	Up to 600 g/m²/d Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	1 day – 4 weeks
Processing capacity	6 sample at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 8. Methodology 8

Test Category	Gas permeation barrier properties	
Test	OxygenTransmission Rate, OTR	
Application in Use Cases Application in Use Cases Assessment of the oxygen transmis flexible barrier materials used in for packaging (UC5-6), shampoo sache electronic displays (UC4)		
Partner (Service Provider)	AUTH	
Available equipment	MOCON OX-TRAN 2/21	
	ASTM D-3985 Films	
	ASTM F-1927 Films	
Relevant standards	DIN 53380 Films	
Relevant Standards	JIS K-7126 Films	
	ASTM F-1307 packages	
	ISO CD 15105-2	
Sample dimensions (range)	Test Samples Size: Films - 5 in. × 5 in. (12.7 cm × 12.7 cm)	
	O2TR Test Range:	
	Unmasked	
	0.005 to 200 cm ³ /m ² /d	
	0.0003 to 13 cm ³ /100 in ² /d	
	0.000025 to 1.0 cm ³ /pkg./d	
	Masked	
	0.05 to 2,000 cm ³ /m ² /d	
	0.003 to 130 cm ³ /100 in ² /d	
	0.00025 to 1.0 cm ³ /pkg./d	
Measuring range / conditions		
Measuring range / conditions	Sensor: Blue COULOX Sensor	
	Temperature Range: 10 °C to 40 °C	
	Standard Testing:	
	Films - Dry or Ambient	
	Packages - Dry or Ambient	
	Films or Packages at Unknown Wet RH	
	Controlled RH Testing:	
	001111 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	

	Packages - 0%, and 35% to 90% RH ±3%
	Packages - Up to 3 litres per package Controlled RH - Up to 2 litres per package
	Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	1-3 weeks depending on the properties
Processing capacity	2 samples at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 9. Methodology 9

Test Category	Gas permeation barrier properties
Test	Oxygen Transmission Rate, OVTR
Application in Use Cases	Assessment of the oxygen transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)
Partner (Service Provider)	FHG-FEP
Available equipment	MOCON OX-TRAN 2/20 OTR
Relevant standards	DIN 53380-3 and ASTM D 3985
Sample dimensions (range)	1 A4 sheet 2 samples with a diameter of 92 mm
Measuring range / conditions	0.1 - 200 cm ³ /m ² /d/bar with pure oxygen; 0.5 - 1000 cm ³ /m ² /d/bar with compressed air Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	1 day
Processing capacity	2 samples at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 10. Methodology 10

Test Category	Gas permeation barrier properties
Test	Oxygen Transmission Rate, OTR
Application in Use Cases	Assessment of the oxygen transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)
Partner (Service Provider)	FHG-IVV
Available equipment	MOCON OX-TRAN
Relevant standards	
Sample dimensions (range)	2 A4 sheets
Measuring range / conditions	$5 \times 10^{-3} \text{cm}^3/\text{m}^2/\text{d/bar}$ up to 40 °C / 90 % RH Customer specific measurement conditions or testing protocols available upon request
Processing time per sample	Depending on the barrier performance
Processing capacity	Two samples at a time
HR profile - Technician - Engineer / graduate - Scientist	Technician
TRL	4-7
Invoicing model (€/sample or €/h)	€/sample

Table 11. Methodology 11

Test Category	Gas permeation barrier properties	
Test	Oxygen Transmission Rate, OTR	
Application in Use Cases	Assessment of the oxygen transmission rate of flexible barrier materials used in food packaging (UC5-6), shampoo sachets (UC2), or electronic displays (UC4)	
Partner (Service Provider)	JOA	
Available equipment	Own construction	
Relevant standards		
Sample dimensions (range)	Circle with 45 mm diameter, thickness up to 1 mm	
Measuring range / conditions	Down to $1 \times 10^{-4} \text{cm}^3/\text{m}^2/\text{d/bar}$ Customer specific measurement conditions or testing protocols available upon request	
Processing time per sample	Up to two weeks (depends on sample and layer sequence)	
Processing capacity	1 sample at a time	
HR profile	Technician	

- Technician - Engineer / graduate - Scientist	
TRL	7
Invoicing model (€/sample or €/h)	€/sample

The information in the above tables was then compiled and integrated into the public Catalogue of Physicochemical and Functional Characterisation Services and uploaded to the FlexFunction2Sustain website (Table 12).

Table 12. Description of Gas Barrier Properties characterisation in FlexFunction2Sustain's Catalogue of Physicochemical and Functional Characterisation Services

Determination of Barrier Properties flexible materials			
Functionalities	Equipment	Technical specifications	Application examples
Water Vapor Transmission Rate, WVTR	Optical Calcium testing Elcometer 5100 Payne Permeability Cups Sempa HiBarSens 2.0 HAT Brugger WDDG MOCON Aquatran™	Samples Sizes: from 3 × 3 cm to 20 × 20 cm Possible to test according to ASTM E96/E96M - 10, ISO 15106-3, DIN 53 122-1, NIST standards, and ondemand protocols upon request Sensitivity down to 10-6 g/m²/d Possible to test under controlled RH and temperature: 23 °C / 50 % RH, 38 °C / 90 % RH, 60 °C / 90 % RH, 85 °C / 85 % RH, and on-demand conditions upon request	Determine the water vapour transmission rate of flexible, fully recyclable, monomaterial laminates for drink pouches (UC5). Determine the water vapour transmission rate of paper-based packaging for moisture-sensitive food products (UC6) and shampoo sachets (UC2). Determine the water vapour transmission rates of barrier films for flexible electronics applications (UC4)
Oxygen Transmission Rate, OTR	MOCON OX-TRAN MOCON OX-TRAN 2/20 MOCON OX-TRAN 2/21 Own-developed equipment	Samples Sizes: from 3×3 cm to 20×20 cm Possible to test according to ASTM D-3985, ASTM F-1927, DIN 53380-3, JIS K-7126, ISO CD 15105-2, and ondemand conditions Sensitivity down to 5×10^{-3} cm ³ /m ² /d/bar Possible to test under controlled RH and temperature: $23 ^{\circ}\text{C} / 50 ^{\circ}\text{K}$ RH, $38 ^{\circ}\text{C} / 90 ^{\circ}\text{K}$ RH, $60 ^{\circ}\text{C} / 90 ^{\circ}\text{K}$ RH, $85 ^{\circ}\text{C} / 85 ^{\circ}\text{K}$ RH, and on-demand conditions upon request	Determine the oxygen transmission rate of flexible, fully recyclable, monomaterial laminates for drink pouches (UC5). Determine the oxygen transmission rate of paper-based packaging for oxygen-sensitive food products (UC6).

The comparability of the test conditions at the different partner sites is regularly proven by interlaboratory round robin tests on representative materials for those characterization techniques available at different partner sites. This guarantees the same quality of measurements in the different labs.

2. Catalogue of Physicochemical and Functional Characterisation Services (public document online)

The public version of the Catalogue of Physicochemical and Functional Characterisation Services is available on the FlexFunction2Sustain website in form of a PDF (Portable Document Format) file. The following sections are included in the online catalogue:

- Use Cases of the FlexFunction2Sustain project
- Services from Upgraded Facilities: methodologies beyond the state of the art
- Full catalogue of physicochemical and functional characterisation services

2.1. Contents of the Catalogue

The structure of the catalogue is briefly described in this section of the document to provide readers with a quick overview of the content (page 2 of the Annex).

2.2. Use Cases in FlexFunction2Sustain project

For users' reference, the six Use Cases of the project are presented as they were described in the FlexFunction2Sustain Handbook, with a short description and identification of the industrial partner. Each Use Case has been labelled with a numerical code (UC1 to UC6), which will be used to identify them in the application examples for the services described in the catalogue (page 3 of the Annex).

2.3. Services from Upgraded Facilities: methodologies beyond the state of the art

This section (pages 4-5 of the Annex) presents the services resulting from upgrading the FlexFunction2Sustain Physicochemical and Functional Characterisation Facilities. These new services arise from the need to address the specific needs for the development of new sustainable nano-functionalised materials. These special characteristics demand increased attention in the assessment of failure in the functional properties exhibited by the surfaces based on them. Consequently, services such as in-situ assessment of gas barrier properties under strain load or increased temperature are highly demanded in the industry but currently require the combination of several services, with extended execution times. Thus, these new set of upgraded services for providing characterisations beyond the state of the art have the ambition to accelerate services such as the verification of thermo-mechanical failures in packaging films by up to 75 % and, overall, increase confidence in test results and speed up the material verification process.

An example of an upgraded service tool is the combination of a bending test with an optical calcium test implemented at FhG-IAP. The test setup is sketched in Figure 2. The bending diameter currently available is either 1 cm or 1.8 cm and can be adjusted for different sizes upon request. The current device setup consists of a calcium layer between gas barrier films of a size of 18 mm x 26 mm, which is located in the centre of a substrate with sizes of 150 mm x 50 mm (Figure 3). Defect formation upon bending is visualized via a camera located on top of the bending roll. From the defect analysis the WVTR can be conducted. This construction also allows the investigation of active devices such as OLEDs or in conductive films. The functionality of the OLED and possible defect formation can be visualized with the camera as well. In the case of conducting layers conductivity and resistivity is accessible by a four-point measurement. Thus, this upgrade allows application-related lifetime testing of functional substrates as well as devices.

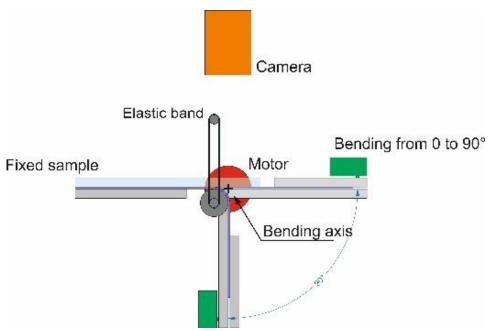


Figure 2. Sketch of the combined bending and Ca-test constructed at FhG-IAP

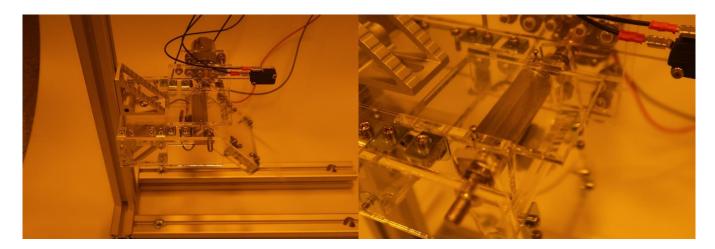


Figure 3. Images of the construction of the combined instrument with the view on the bending roll and the substrate holder at ~45° angle (left) and 90° angle (right). The total length of the substrate is 15 cm, bending rolls of 1cm and 1.8 cm in diameter are implemented, and the investigated device size has a length of 26 mm and is centred on the roll.

2.4. Full catalogue of physicochemical and functional characterisation services

This section describes the full range of characterisation solutions available in FlexFunction2Sustain to assess the structure, surface properties, chemical composition and functional performance of nano-functionalised flexible surfaces and membranes. All the main methodologies relevant to this market segment are made available to industry through the FlexFunction2Sustain Open Innovation Test Bed (pages 6 to 25 of the Annex), and interlaboratory tests implemented to validate methodology among partners when required.

The individual characterisation services available through this catalogue will be integrated into complete workflows, tailor-made for specific materials and products (initially for the Use Cases and pre-commercial Pilot Cases and later for the services commissioned by the SEP customers). These workflows will combine physicochemical and functional characterisation tests at the material level (available through FlexFunction2Sustain's Facility Cluster 7 and included in the catalogue published with D4.1), test for performance verification in application at component and prototype level (available through FlexFunction2Sustain Facility Cluster 8 and listed in the catalogue to be published with D4.2) and safety and regulatory compliance tests (available through FlexFunction2Sustain Facility Cluster 9 and listed in the catalogue to be published with D4.4). Examples of such workflows will be presented in detail in the next deliverables D4.2 and D4.4 (M24).

3. Conclusions

Deliverable D4.1 presents the set of physicochemical and functional characterisation techniques available in the Facility Cluster 7 of the Open Innovation Test Bed project FlexFunction2Sustain. As a part of D4.1, a public catalogue of physicochemical and functional characterisation services for flexible materials has been produced and made available on the FlexFunction2Sustain website (https://flexfunction2sustain.eu). The methodology for setting up the catalogue is described for the example of the gas barrier characterization techniques available in the consortium. The comparability of available testing methods across several partners is proven in interlaboratory round robin tests. Testing protocols for different customer requirements are defined and agreed on in the consortium. The catalogue of characterisation services is subject to continuous updating to integrate new methodologies when they become available. This document represents that state of 30th June 2021.

4. Degree of progress

Deliverable 4.1 is fulfilled by 100 %. A public catalogue of physicochemical and functional characterisation services for flexible materials has been delivered and will be integrated into the Service Catalogue to be offered by the FlexFunction2Sustain OITB through the Single-Entry Point (SEP). This service catalogue will be a living document, which will evolve together with the technological capabilities of FlexFunction2Sustain's partners and the needs of the project and future customers.

5. Dissemination level

Deliverable D4.1 "FlexFunction2Sustain Catalogue Physicochemical and Functional Characterisation Services" is a public document, and version 1.0 is available for download.

6. Appendix

The online Catalogue Physicochemical and Functional Characterisation Services is attached in this section.



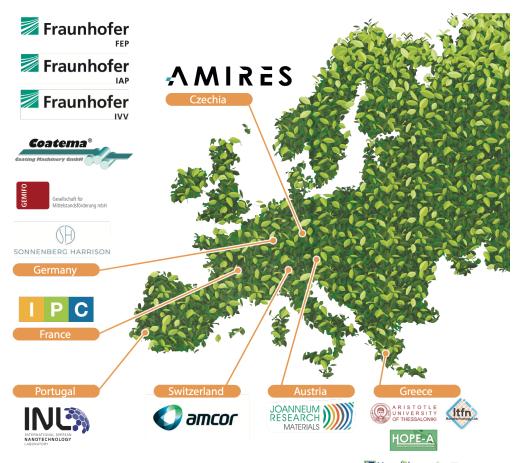
Catalogue of physicochemical and functional characterization services



The catalogue at a glance

- Use Cases of the FlexFunction2Sustain project (p. 3)
- Catalogue of Services (p. 4)
 - Services from Upgraded Facilities: methodologies beyond the state of the art (pp. 4-5)
 - Full catalogue of physicochemical and functional characterisation services (pp. 6-25)
 - Gas barrier testing (pp. 6-7)
 - Testing of electrochemical properties (pp. 8-9)
 - Assessment of mechanical properties, integrity, and durability (pp. 10-12)
 - Optical properties characterisation (pp. 13-14)
 - Surface and microstructure analysis (pp. 15-22)
 - Thermal and rheological properties (pp. 23-24)
 - Test specimen preparation (p. 25)

Service Providers in FlexFunction2Sustain











Use Cases of the FlexFunction2Sustain project

FlexFunction2Sustain's technical facilities and the performances of novel nano-functionalised surfaces will be demonstrated and validated by Europe's leading companies within six industrial application scenarios. Application examples for the services related to the Use Cases are labelled with a code specific for each one (UC#)

Industrial Validation in 6 Use Cases

UC1



Biobased

Optical Films

for Labelling of

Consumer Goods

and Surface

UC2



Marine degradable shampoo sachets UC3



Selective and switchable water filter membranes

UC4



Multifunctional scratch resistant surfaces in automotive

UC5



Recyclable mono-polymer drink pouches

UC6



Sustainable paper-based food packaging

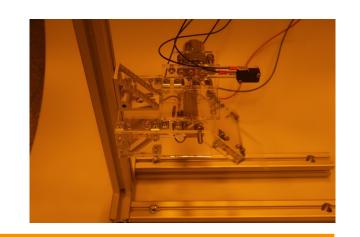
Design A full description of the six Use Cases can be found in the FlexFunction2Sustain Project Handbook, available at https://flexfunction2sustain.eu



Services from Upgraded Facilities: methodologies beyond the state of the art

Mechanical testing with inline WVTR characterization

- Coupling of optical calcium test for WVTR with mechanical bending test in device geometry
- Sensitivity down to 10⁻⁶ g/m²/d



Combined gas barrier and mechanical testing

Functionalities	Equipment	Technical specifications	Application examples
Flexural Testing with inline Water Vapor Transmission Rate via Optical Calcium test for ultra high barrier flexible materials	Own-developed	Sample sizes: 50 mm × 150 mm Sensitivity down to 10 ⁻⁶ g/m²/d Testing according to on-demand protocols upon request	Mechanical testing with inline water vapour permeation measurement of flexible displays (UC4) Mechanical testing of active OLED devices (UC4)

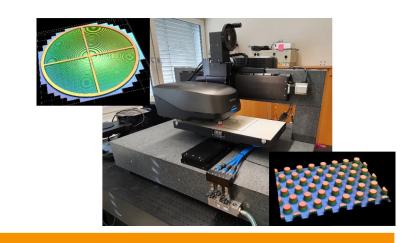




Services from Upgraded Facilities: methodologies beyond the state of the art

Large area 3D Confocal Laser **Scanning Microscopy**

- Surface profiling, defect density characterisation
- Sample processing up to 300 x 600 mm²



	Surtace ar	nd microstructure analysis
unctionalities	Equipment	Technical specifications

Large-Area Laser Scanning 3D Confocal Microscopy

Keyence VK-X1050 + Custom-made platform for large area measurements

Up to 300×600 mm scanning area

X,Y-resolution: 200 nm

7-resolution: 10 nm

Application examples

Depth profiling and defect density characterisation in masters for nano/micro patterning and quality control of nano-imprinted films (UC1)





Catalogue of physicochemical and functional characterisation services Gas barrier testing



Gas barrier testing

Functionalities	Equipment	Technical specifications	Application examples
Water Vapor Transmission Rate, WVTR	Optical Calcium testing Elcometer 5100 Payne Permeability Cups Sempa HiBarSens 2.0 HAT Brugger WDDG MOCON AquatranTM	Samples Sizes: from 3 × 3 cm to 20 × 20 cm Possible to test according to ASTM E96/E96M - 10, ISO 15106-3, DIN 53 122-1, NIST standards, and on-demand protocols upon request Sensitivity down to 10 ⁻⁶ g/m²/d Possible to test under controlled RH and temperature: 23 °C / 50 % RH, 38 °C / 90 % RH, 60 °C / 90 % RH, 85 °C / 85 % RH, and on-demand conditions upon request	Determine the water vapour transmission rate of flexible, fully recyclable, monomaterial laminates for drink pouches (UC5). Determine the water vapour transmission rate of paper-based packaging for moisture-sensitive food products (UC6) and shampoo sachets (UC2). Determine the water vapour transmission rates of barrier films for flexible electronics applications (UC4)



Catalogue of physicochemical and functional characterisation services Gas barrier testing



Gas barrier testing				
Functionalities	Equipment	Technical specifications	Application examples	
Oxygen Transmission Rate, OTR	MOCON OX-TRAN 2/20 MOCON OX-TRAN 2/21 Own-developed equipment	Samples Sizes: from 3 × 3 cm to 20 × 20 cm Possible to test according to ASTM D-3985, ASTM F-1927, DIN 53380-3, JIS K-7126, ISO CD 15105-2, and on-demand conditions Sensitivity down to 5 × 10 ⁻³ cm ³ /m ² /d/bar Possible to test under controlled RH and temperature: 23 °C / 50 % RH, 38 °C / 90 % RH, 60 °C / 90 % RH, 85 °C / 85 % RH, and on-demand conditions upon request	Determine the oxygen transmission rate of flexible, fully recyclable, monomaterial laminates for drink pouches (UC5). Determine the oxygen transmission rate of paper-based packaging for oxygen-sensitive food products (UC6).	



Catalogue of physicochemical and functional characterisation services Electrochemical properties



Electrochemical properties				
Functionalities	Equipment	Technical specifications	Application examples	
Cyclic Voltammetry	Autolab PGSTAT302N	Samples from 5 to 15 µL of active ink or 1 × 2 cm to 1.5 × 3 cm of polymer substrate with deposited active material Redox potential onsets (ranges: -2,5 to 0,5 V) Possibility to test according to EN61326-1 (1997) + A1 (1998) + A2 (2001) + A3 (2003), EN61000-3-2 (2006), EN61000-3-3 (1995) + A1 (2001) + A3 (2003)	HOMO-LUMO determination Biosensor's quality testing	
Squarewave Voltammetry	Autolab PGSTAT302N	Δ-current from forward and backward pulse (Range 1 nA – 1 mA) Measurements up to 10 mA Possibility to test according to EN61326-1 (1997) + A1 (1998) + A2 (2001) + A3 (2003), EN61000-3-2 (2006), EN61000-3-3 (1995) + A1 (2001) + A3 (2003)	HOMO-LUMO determination Biosensor's quality testing	

Catalogue of physicochemical and functional characterisation services Electrochemical properties



Electrochemical properties

Functionalities	Equipment	Technical specifications	Application examples
Electrochemical Impedance Spectroscopy	Autolab PGSTAT302N	Working electrode diameter 4 mm -10 mm Resistance measurement 0.1 - 10 k Ω in PBS solution Possibility to test according to EN61326-1 (1997) + A1 (1998) + A2 (2001) + A3 (2003), EN61000-3-2 (2006), EN61000-3-3 (1995) + A1 (2001) + A3 (2003)	HOMO-LUMO determination Biosensor's quality testing
Sheet resistance	Custom-developed equipment Jandel 4-point probe stand	Adaptable sample sizes and testing protocol Measurement range 0.1 Ω /square - 100 k Ω /square	Applications in use cases

Catalogue of physicochemical and functional characterisation services Mechanical properties, integrity, and durability



Mechanical properties, integrity, and durability

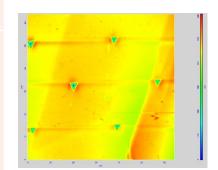
Functionalities	Equipment	Technical specifications	Application examples	
Abrasion resistance	CrockMaster Custom-made devices	Possible to test according ASTM D3363, DIN ISO 3537:2018-02, ISO 9352-2018, and on-demand protocols Testing up to 100 mm × 100 mm; spot for sand trickling: diameter of ≤ 10 mm Pencil hardness test: 6B to 6H; Abrading wheels CS10F; 500 gf; 1000 revolutions; measurement of haze	Coating resistance to mechanical damage by rubbing, scraping, etc (UC4)	
Adhesion strength	Peel tester Sebastian V Universal tensile testing machine with video recording (Zwick Z005)	Sample size up to 1 A4 sheet Testing according IPC-TM-650, DIN 55543-5, and the standard EAA peel test of the Fraunhofer IVV/EMA (European Metallizers Association) Max force 3.5 N/15 mm	Determination of adhesion strength in packaging films (UC2, 5, 6)	





Mechanical properties, integrity, and durability				
Functionalities	Equipment	Technical specifications	Application examples	
Bending / rolling testing	Self-made equipment	Testing surface up to 400 mm × 200 mm On-demand protocols Bending and rolling testing with inline electrical measurements	Bending Strain and Bending Fatigue of flexible films incorporating metal electrodes	
Multi-axial mechanical failure testing (shear, flexion, traction, Charpy, Izod, dynamic)	4 Dynamometers (Zwick and Instron)	Load cells ranging from 200N up to 250kN, Climatic chambers (-70 °C up to 250 °C), and extensometers. Multiple test fixtures for shear, bending, tensile, drop, compression	Determination of elastic modulus, breaking force, elongation at break of plastic samples	
Nanoindentation	Nano Indenter XP	Testing up to up to 50 mm × 50 mm 100 mN maximum applied load, 20 µN lower normal applied load Testing according DIN EN ISO 14577 and on-demand protocols	Testing of thin films (metallic and polymeric) and protective (barrier) coatings. Calculation of hardness and Young's modulus. Scratch test: coatings cohesion adhesion to substrate, friction coefficient measurement	

Atomic Force Microscopy image of nanoindentation imprints (triangles in the image) acquired at AUTH with a Berkovich-type diamond indenter on top of an aluminum coated membrane. The maximum penetration depth of the indenter was 500 nm.







Mechanical properties, integrity, and durability

Functionalities	Equipment	Technical specifications	Application examples
Tensile Properties	Universal tensile testing machine with video recording (Zwick Z005) Self-made equipment with Zeiss microscope and AXIO-Vision camera set up and heating module	Testing according DIN EN ISO 1924, DIN EN ISO 527, and on-demand protocols Typical sample sizes: 10 mm × 60 mm / 150 mm × 40 mm <5 kN load capacity (0.5 % load accuracy); Determination of elastic modulus up to 150 °C	Determination of elastic modulus, breaking force, elongation at break of films and paper (UC1, 2, 5, 6)

Catalogue of physicochemical and functional characterisation services Optical properties



Optical properties			
Functionalities	Equipment	Technical specifications	Application examples
Spectroscopic ellipsometry (thin film thickness; refractive index, dispersion)	HORIBA: UVISEL-VASE	Samples Sizes up to 10 × 10 cm Spectral Range: 250 -1100 nm Possibility of in-line measurements in R2R pilot line Variable angle, 0.6-6.5 eV, real-time measurements	Determination of thin film thickness and optical constants for optical-grade functional coatings (UC4)
Gloss, colorimetry, and Haze characterisation	Erichsen Picogloss master 500 BYK Haze-gard plus Spectro-guide sphere gloss BYK Gardner	Gloss range 0 to 199.9 GU, 60° Haze range from 0.1 to 30 % Testing according to DIN EN ISO 2813, ASTM D1003, and on-demand protocols Colorimetry: CIELab and CIELCH; Spectro colorimetry with spectral range: 400 - 700	Quality control in surface treated optical-grade display covers (UC4) Plastic films and samples quality control (UC3), surface aesthetics characterisation

In-line optical metrology tools (UV-Vis Spectroscopic Ellipsometer, Raman Spectrometer) on the AUTH R2R pilot line for real-time optical characterisation of nanomaterials printed on flexible polymeric membranes for printed electronics applications.



Catalogue of physicochemical and functional characterisation services Optical properties



Optical properties			
Functionalities	Equipment	Technical specifications	Application examples
UV VIS Spectroscopy	PerkinElmer Lambda900 PerkinElmer LS50B HEllOSa Scan Intelliscan	Samples Sizes: up to A4 size Possible to test according to DIN 10 050 Part 9 and on-demand conditions Optical characterisation within the UV-VIS-NIR range (220 nm - 2500 nm; 175 nm - 3300 nm in transmission mode)	Determination of UV-VIS barrier for light-sensitive food products (UC5, UC6)
	TTE II G G G G G T II I C III G G G G G T II I C II G G G G G T II I C II G G G G G T II I C II G G G G G G G G G G G G G G G	Measurements in transmission, reflection, and diffusion mode. Angle-dependent measurements inside integrated sphere. VN-accessory for measurement of absolute reflectance	Optical characterisation and quality control in optical-grade films and coatings (UC1, UC4)
		Optical density of band-stop filters to the limit of OD6 Emission and excitation spectra of materials within a spectral range of 200 to 850 nm	

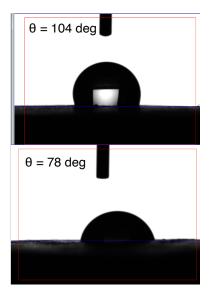




Surface and microstructure analysis

Surface and interestructure analysis			
Functionalities	Equipment	Technical specifications	Application examples
Atomic Force Microscopy (AFM)	Bruker Dimension 3000 Park Systems NX20 Nanosurf Easy Scan 2 NT-MDT NTEGRA SPM Platform	Measurement modes: non-contact mode (topography), Conductive AFM, Piezo Force Microscopy (PFM), and Nanoindentation up to 100 μm × 100 μm scan area	Surface topology characterisation, and surface physical properties at the nano-range
Contact Angle Measurement (CA)	Krüss DSA100 Kruss DSA 100E KVS Cam 200 GBX photo goniometer	Up to A4 sheet size Range: 0-180°, resolution: up to 0.1°, accuracy: 1° Advancing, receding, static, dynamic, tilting modes Curve fitting to multiple models (Young Laplace equation, cycle, polynomial and	Surface energy determination and hydrophilicity/hydrophobicity characterization of packaging materials Effectiveness of corona /plasma treatment (ability of
		Bashforth-Adams)	surfaces to hold a coating)

Contact angle measurements for surface wettability characterization. Acquired on a Kruss DSA 100E at INL







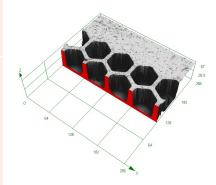




Surface and microstructure analysis

Functionalities	Equipment	Technical specifications	Application examples
Confocal laser microscopy	OLYMPUS LEXT 3D Measuring laser microscope OLS4000	Motorized table 100 mm × 100 mm Magnification ranges from 108× - 17,280× 3D measurement - Mapping - Non- contact roughness	Plastic films surface characterisation, quality control
Contact roughness	MAHR PERTHEN Perthometer S4P	Probing length = 17.5 mm max Ra - Rz - Rmax - R3z - Pt - Wt and more	Plastic films surface characterisation, quality control

3D mapping of the microstructure of 8 µm filters with confocal microscopy at IPC



Catalogue of physicochemical and functional characterisation services Surface and microstructure analysis



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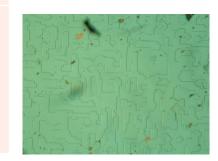
Functionalities	Equipment	Technical specifications	Application examples
Fourier Transform Infrared Spectroscopy (FTIR)	Bruker VERTEX 80v vacuum FTIR spectrometer Tensor 27 ATR-FTIR spectrometer from Bruker Optics PerkinElmer Spectrum 2000 Nicolet iS10 FTIR Nicolet iN10 µFTIR	PEAK resolution up to 0.06 cm ⁻¹ Spectral Range: 7900 to 370 cm ⁻¹ Modes: transmittance, reflectance, ATR (attenuated total reflection)	Polymer fingerprinting and quality control of plastic and paper films (UC1, 2, 5, 6)
Large-Area Laser Scanning 3D Confocal Microscopy	Keyence VK-X1050 + Custom-made platform for large area measurements	Up to 300 × 600 mm scanning area X, Y-resolution: 200 nm Z-resolution: 10 nm	Depth profiling and defect density characterisation in masters for nano/micro patterning and quality control of nano-imprinted films (UC1)





Surface and microstructure analysis **Functionalities** Equipment Technical specifications Application examples Optical (R2R) surface ISRA-VISION Camera Inline optical inspection Quality control, Surface optical Optical Inspection inspection in roll-to-roll Inspection Defect size detection limit > 40 µm @ 1 applications m/min; on 300 mm width Motorized table 100 mm × 100 mm Optical microscopy **KEYENCE** Microscope Optical characterisation and VHX-7000 quality control of materials, High resolution lenses from $20 \times$ to $2,500 \times$ films and coatings Polarizing filter Raman spectroscopy (RS) Witec alpha300 R Full Raman spectrum at every image pixel Chemical composition mapping and layer analysis, for static substrates In-line Raman in R2R structural analysis (including coating pilot line In-line Raman analysis polymorph distributions, crystallinity and orientation), and structural integrity of barrier films (UC5)

Optical microscopy image of an anti-counterfeiting pattern (height: 100 to 300 nm; size of structures: 250 to 2000 nm) obtained at IPC.



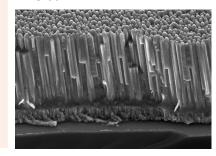




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Functionalities	Equipment	Technical specifications	Application examples
Scanning Electron Microscopy (SEM) and Environmental SEM	FEI Quanta 650 FEG Environmental SEM (including Peltier and Heating Stage) Hitachi SU-8000 JEOL JSM-IT-300LV JEOL Neoscope JCM- 5000	Voltage: 1 – 30 kV SEM Imaging (Resolution 1 nm) Low vacuum and Environmental SEM Imaging for sensitive materials and noncoated samples EDXS (Chemical analysis) Cooling/Heating stage (in-situ): –20 °C – 1500 °C SE/BSE detectors (Topographical/Structural analysis)	Ultra-high resolution surface imaging for morphological/topographical characterization, failure analysis and or contamination detection

SEM image of a magnetron sputtered TiAlSiN thin film (courtesy of S. Calderón). Acquired on FEI Quanta 650 FEG at INL







Surtace and microstructure analysis				
Functionalities	Equipment	Technical specifications	Application examples	
Transmission Electron Microscopy (TEM)	Probe-Corrected FEI Titan G2 80-200 kV ChemiSTEM Double-Corrected FEI Titan G3 Cubed Themis 60-300 kV	TEM point resolution ≤ 0.24 nm at 200 kV AC-STEM information limit ≤ 80 pm (200 kV) Super X energy resolution ≤ 136 eV – 140 eV	Internal structure of thin films including chemical and crystallographic information Size, morphology, and distribution of nanomaterials within films and resin coatings	
	JEOL JEM 2100 80-200 kV	Alpha tilt: ±70° (Tomography holder) Corrected TEM and STEM Imaging (Resolution 63 pm)	Determination of metallised layer thickness in coated films	

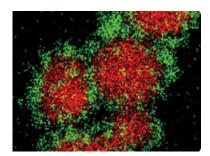
Diffraction (Crystallographic analysis)

Differential Phase contrast (DPC) imaging

EDX – Super X (Chemical analysis)

Electron Holography

STEM-EDX of Fe3O4@Au nanoparticles (courtesy of E. Carbo-Argibay). Acquired on a Double-Corrected FEI Titan G3 Cubed Themis at INL

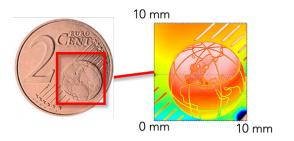


Catalogue of physicochemical and functional characterisation services Surface and microstructure analysis



Surface and microstructure analysis			
Functionalities	Equipment	Technical specifications	Application examples
White Light Interferometry	Smart WLI	Up to 100 mm × 100 mm Sub nanometre height resolution	Surface characterisation, micro/nanostructure characterisation; surface roughness; defect characterisation; layer thickness determination
X-Ray Fluorescence (XRF)	SPECTRO XEPOS III	Element analyses from Na to U For flat and solid samples (powder and liquid samples on-demand)	Determination of and trace elements in materials

False color image of the surface profile of a 2 Euro Cent coin acquired at FEP with the Smart WLI white-light interferometer







Catalogue of physicochemical and functional characterisation services Surface and microstructure analysis



Surface and microstructure analysis			
Functionalities	Equipment	Technical specifications	Application examples
X-Ray Photoelectron Spectroscopy (XPS)	Thermo Scientific Escalab 250 Xi Omicron + own construction (DAR 400 X-ray source, XM 500 quartz crystal monochromator) Axis 165 - Auger (SAM/AES) (Kratos)	Measurements in samples up to 3×3 cm, Depth resolution up to 1-10 nm via depth profiling, and lateral resolution down to ~1 μ m Electron Analyser ($0-\pm5000$ eV) X-ray Sources (Monochromatic Al K α and twin anode Mg K α / Al K α) Heating and Cooling of Specimen UV Source Flood Sources (Charge compensation and REELS)	Bonding state analysis and quantitative elemental composition of materials Composition uniformity in thin films Analysis of residues in surfaces and coatings, surface contamination penetration

Monoatomic and Gas Cluster Ion Source for depth

profiling "soft" (cluster mode) and solid

(monoatomic mode) materials

Surface and microstructure analysis





Thermal and rheological properties				
Functionalities	Equipment	Technical specifications	Application examples	
Differential scanning calorimetry (DSC)	TA Instrument Q100 and Q20	-80 °C to 725 °C Inert atmosphere Typical heating rate = 10 or 20 °C/min	Thermal properties characterisation, identification of melting, cristallisation and glass transition temperatures	
Thermal gravimetric analysis (TGA)	TA Instrument Q500 and Q50	Ambient to 950 °C Inert or oxidizing atmosphere Typical heating rate = 20 °C/min	Thermal resistance assessment, decomposition, adsorption and desorption characterisation	
Heat deflection temperature (HDT)	CEAST HDT-Vicat 3 posts	Max temperature : 300 °C Heating rate : 50 °C/h and 120 °C/h	Assessing the thermo- mechanical properties of a wide range of plastics materials and specimens	

Differential Scanning Calorimetry (DSC) measurement performed at IPC







Thermal and rheological properties

Functionalities	Equipment	Technical specifications	Application examples
Melt Flow Index (MFI) & Melt Volume Index (MVI)	Göttfert MI40	Max temperature = 500 °C 1.2kg - 2.16 kg - 3.8 kg - 5.0 kg - 10.0 kg - 21.6 kg	Thermal properties characterisation, identification of melting, crystallisation and glass transition temperatures
Rheology	TA Instrument AR2000ex	Heating rate up to 20 °C/min Angular Velocity Range CS : 0 to 300 rad/s Normal/Axial Force Range : 0.005 to 50 N Peltier Plate up to 200 °C	Characterisation of low viscosity fluids, polymer melts, solids and reactive materials

Parallel-plate rotational rheometry measurement performed at IPC







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5	pecimen	pre	para	tion

Functionalities	Equipment	Technical specifications	Application examples
Cross section polishing	Jeol SM-09010	Up to 1 mm preparation width	Broad ion beam preparation of polished cross sections of multilayers
Microtome cross sectioning	RMC-Cryo- Ultramicrotome Modell PT-XL with Cryoequipment CxL	Recommended sample size: ~ 1-2 cm ²	Determination of adhesion strength in packaging films (UC2, 5, 6)

Cross section of a three-layer thin film stack (gas barrier coating on plastic web by Fraunhofer POLO® alliance) prepared with the Jeol SM-09010 cross-section polisher and scanning electron microscopy

