

# Catalogue of physicochemical and functional characterization services



This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement n°862156

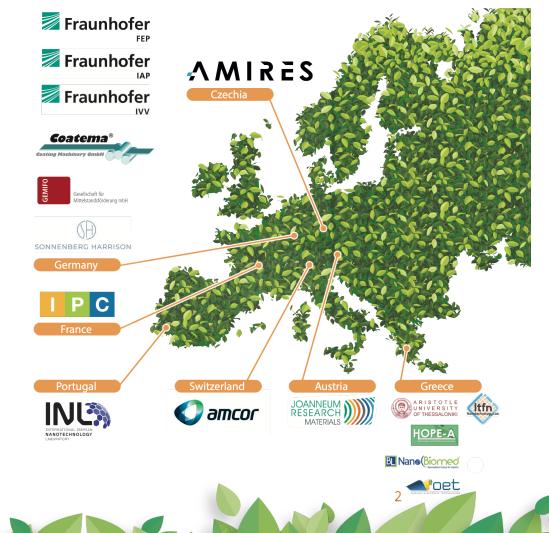


### The catalogue at a glance

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

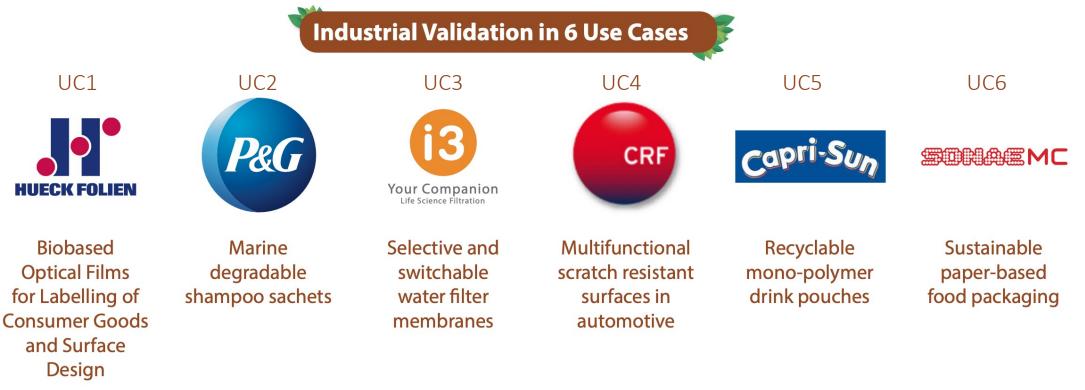




3

### 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#)

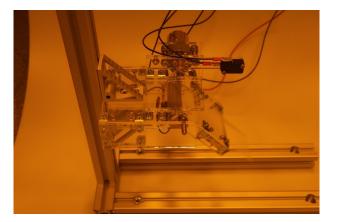


A full description of the six Use Cases can be found in the FlexFunction2Sustain Project Handbook, available at https://flexfunction2sustain.eu

## 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<sup>-6</sup> g/m<sup>2</sup>/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 <sup>-6</sup> 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 mm<sup>2</sup>



#### Surface and microstructure analysis

Functionalities	Equipment	Technical specifications	Application examples
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)



#### 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 <sup>-6</sup> g/m <sup>2</sup> /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 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 x 10 <sup>-3</sup> cm <sup>3</sup> /m <sup>2</sup> /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, mono- material 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 kΩ 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 kΩ/square	Applications in use cases	

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



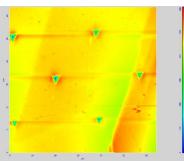
Mechanical properties, integrity, and durability				
Functionalities	Equipment	Technical specifications	Application examples	
Abrasion resistance	CrockMaster Custom-made devices	Possible to test according ASTM D3363, DIN ISO 3537:2018-02, ISO 9352-2018, and on-demand protocols Testing up to 100 mm $\times$ 100 mm; spot for sand trickling: diameter of $\leq$ 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)	

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



F	unctionalities	Equipment	Technical specifications	Application examples
E	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
fa fl	Aulti-axial mechanical ailure testing (shear, lexion, traction, Charpy, zod, 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
Γ	Vanoindentation	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.



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



Mechanical properties, integrity, and durability				
Functionalities	Equipment	Technical specifications	Application examples	
Tensile Properties	Instron 3342 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 HEIIOSa Scan Intelliscan	<ul> <li>Samples Sizes: up to A4 size</li> <li>Possible to test according to DIN 10 050 Part 9 and on-demand conditions</li> <li>Optical characterisation within the UV-VIS-NIR range (220 nm - 2500 nm; 175 nm - 3300 nm in transmission mode)</li> <li>Measurements in transmission, reflection, and diffusion mode.</li> <li>Angle-dependent measurements inside integrated sphere. VN-accessory for measurement of absolute reflectance</li> <li>Optical density of band-stop filters to the limit of OD6</li> <li>Emission and excitation spectra of materials within a spectral range of 200 to 850 nm</li> </ul>	Determination of UV-VIS barrier for light-sensitive food products (UC5, UC6) Optical characterisation and quality control in optical-grade films and coatings (UC1, UC4)		



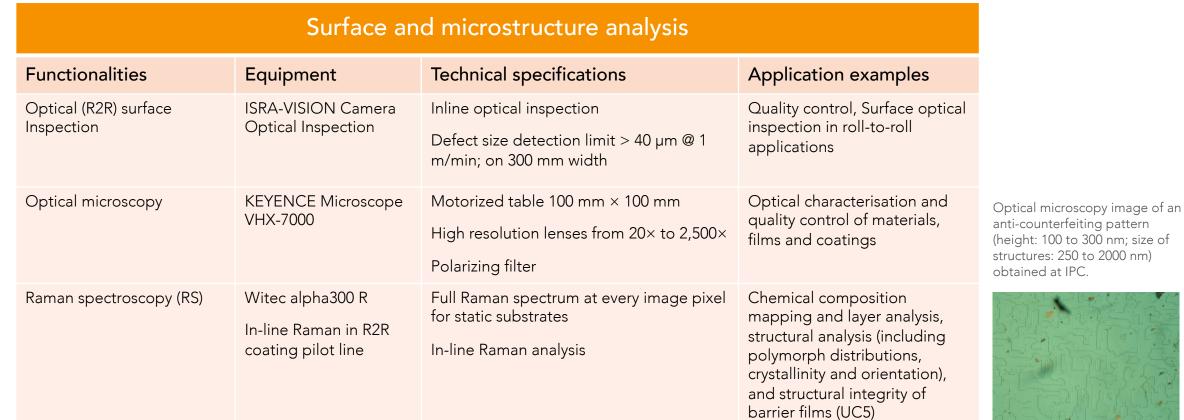
Functionalities	Equipment	Technical specifications	Application examples	
Atomic Force Microscopy (AFM)	Bruker Dimension 3000 Park Systems NX20 Nanosurf Easy Scan 2 NT-MDT NTEGRA SPM Platform	Measurement modes: non-contact mode (topography), Conductive AFM, Piezo Force Microscopy (PFM), and Nanoindentation up to 100 µm × 100 µm scan area Scan height up to 200 nm	Surface topology characterisation, and surface physical properties at the nano-range	Contact angle measurements for surface wettability characterization. Acquired on a Kruss DSA 100E at INL $\theta = 104 \text{ deg}$
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 Bashforth-Adams)	Surface energy determination and hydrophilicity/hydrophobicity characterization of packaging materials Effectiveness of corona /plasma treatment (ability of surfaces to hold a coating)	θ = 78 deg



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	3D mapping of the microstructure of 8 µm filt with confocal microscopy
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	



Surface and microstructure analysis			
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 <sup>-1</sup> Spectral Range: 7900 to 370 cm <sup>-1</sup> 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)



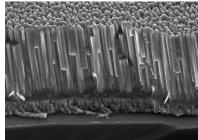


anti-counterfeiting pattern (height: 100 to 300 nm; size of structures: 250 to 2000 nm)



Surface and microstructure analysis				
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 non- coated 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	

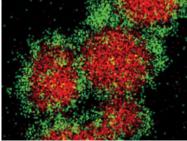
SEM image of a magnetron sputtered TiAlSiN thin film courtesy of S. Calderón). Acquired on FEI Quanta 650 FEG at INL





Surface 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 JEOL JEM 2100 80-200 kV	<ul> <li>TEM point resolution ≤ 0.24 nm at 200 kV</li> <li>AC-STEM information limit ≤ 80 pm (200 kV)</li> <li>Super X energy resolution ≤ 136 eV – 140 eV</li> <li>Alpha tilt: ±70° (Tomography holder)</li> <li>Corrected TEM and STEM Imaging (Resolution 63 pm)</li> <li>Diffraction (Crystallographic analysis)</li> <li>EDX – Super X (Chemical analysis)</li> <li>Electron Holography</li> <li>Differential Phase contrast (DPC) imaging</li> </ul>	Internal structure of thin films including chemical and crystallographic information Size, morphology, and distribution of nanomaterials within films and resin coatings Determination of metallised layer thickness in coated films	

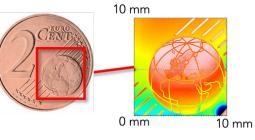
STEM-EDX of Fe3O4@Au nanoparticles (courtesy of E. Carbo-Argibay). Acquired on a Double-Corrected FEI Titan G3 Cubed Themis at INL





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

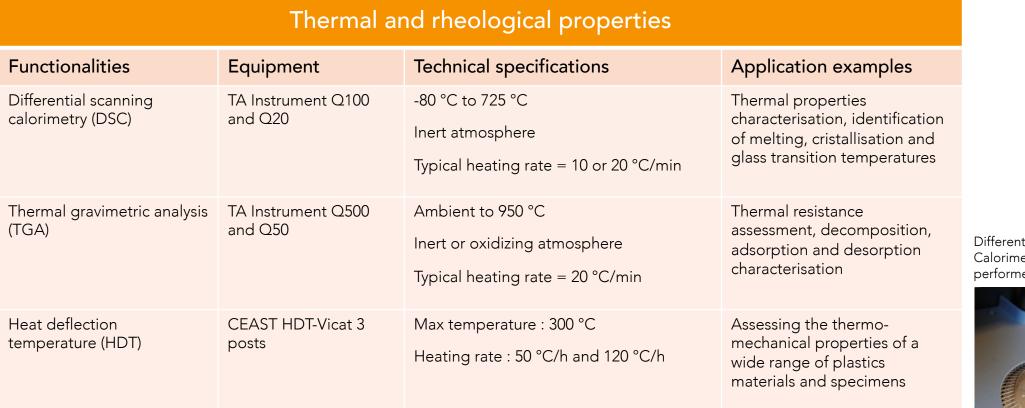




Surface and microstructure analysis			
Functionalities	Equipment	Technical specifications	Application examples
X-Ray Photoelectron Spectroscopy (XPS)	Thermo Scientific Escalab 250 Xi Omicron + own construction (DAR 400 X-ray source, XM 500 quartz crystal monochromator) Axis 165 - Auger (SAM/AES) (Kratos)	Measurements in samples up to $3 \times 3$ cm, Depth resolution up to 1-10 nm via depth profiling, and lateral resolution down to ~1 µm Electron Analyser ( $0 - \pm 5000 \text{ eV}$ ) X-ray Sources (Monochromatic Al K $\alpha$ and twin anode Mg K $\alpha$ / Al K $\alpha$ ) Heating and Cooling of Specimen UV Source Flood Sources (Charge compensation and REELS) Monoatomic and Gas Cluster Ion Source for depth profiling "soft" (cluster mode) and solid (monoatomic mode) materials	Bonding state analysis and quantitative elemental composition of materials Composition uniformity in thin films Analysis of residues in surfaces and coatings, surface contamination penetration



# Catalogue of physicochemical and functional characterisation services Thermal and rheological properties





Differential Scanning Calorimetry (DSC) measurement performed at IPC



# Catalogue of physicochemical and functional characterisation services Thermal and rheological properties

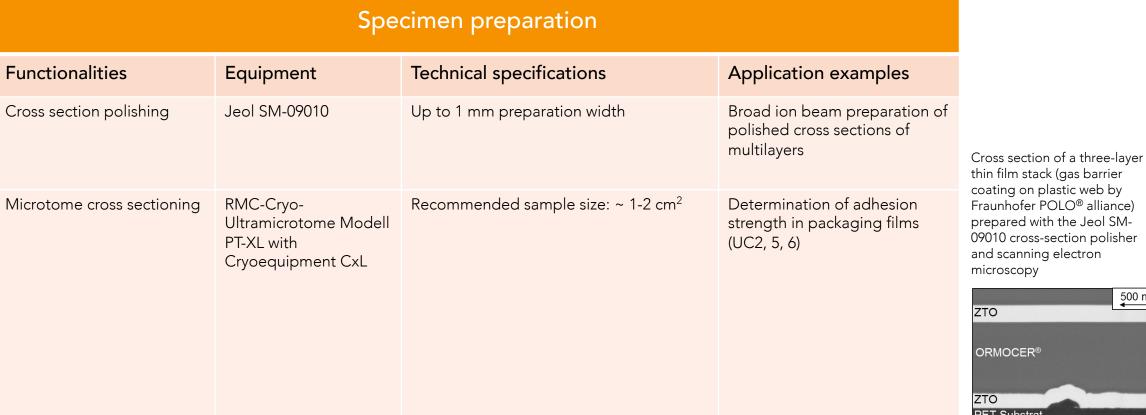


FLEX FUNCTION 2 SUSTAIN

Parallel-plate rotational rheometry measurement performed at IPC



#### **Catalogue of physicochemical and functional** characterisation services **Specimen preparation**





thin film stack (gas barrier coating on plastic web by Fraunhofer POLO<sup>®</sup> alliance) prepared with the Jeol SM-09010 cross-section polisher and scanning electron microscopy

