PROJECT FINAL REPORT

Appendix 1 - Figures

Grant Agreement number: 310397

Project acronym: NATURAL

Project title: Standardised metrology of Nano-sTrUctuRed CoAtings with Low surface energy

Funding Scheme: FP7-SME-2013 Research for the benefit of specific groups

Period covered: from 01/03/2013 to 29/04/2016

Name of the scientific representative of the project's co-ordinator[1], Title and Organisation: Amit Rana

Tel: +441642 216346

E-mail: Amit.rana@twi.co.uk

Project website address: http://www.natural-project.eu/

List of Figures

List of Tables

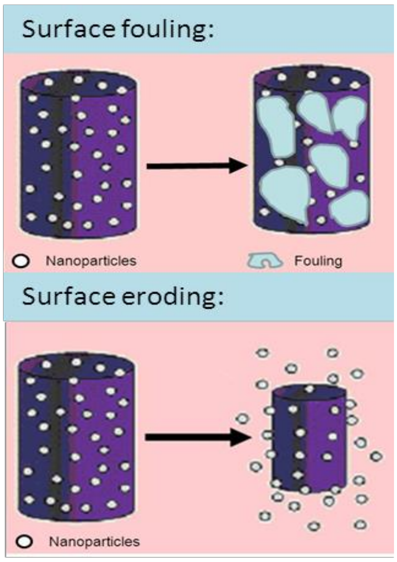


Figure 1 Differences between surface fouling and surface erosion

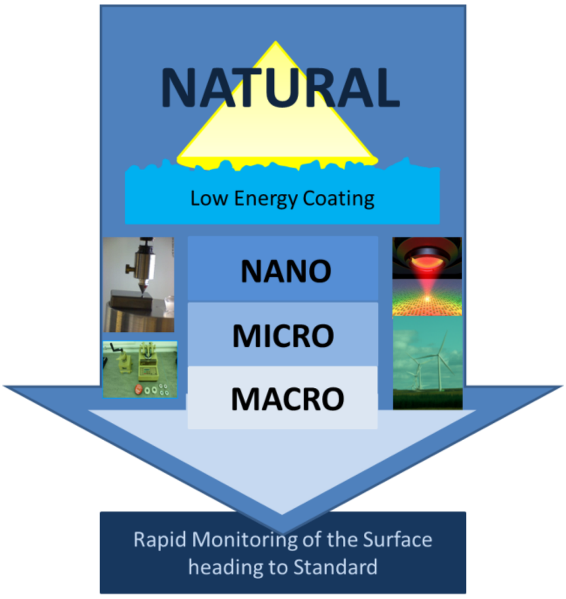


Figure 2 Overview of the NATURAL concept

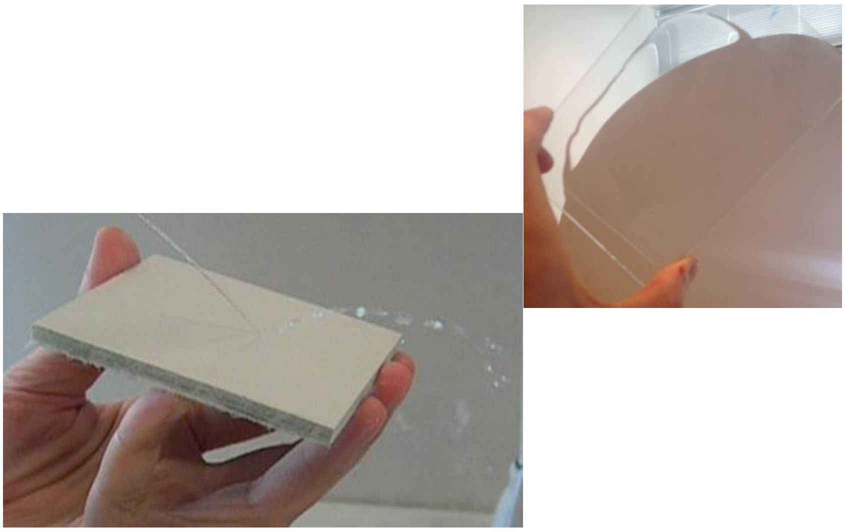


Figure 3 Examples of hydrophobicity and transparency of nanostructured coatings

Figure 4 Optimised spray process for flat lab test substrates (Top) and wind turbine blade sections (Bottom)

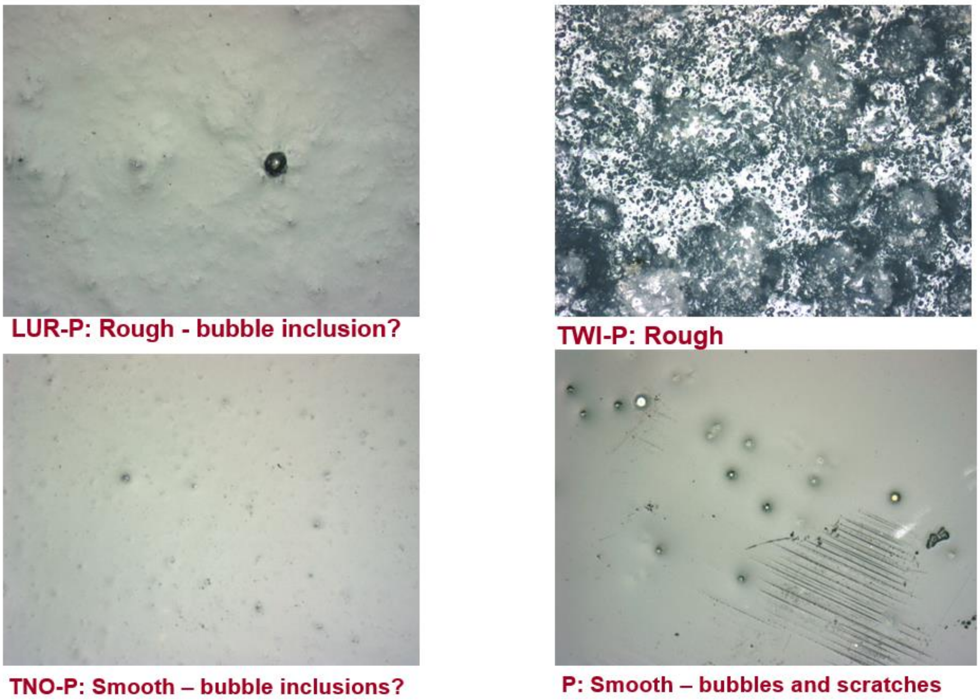


Figure 5 Surface appearance of the pristine samples LPU2 obtained using 3D optical techniques

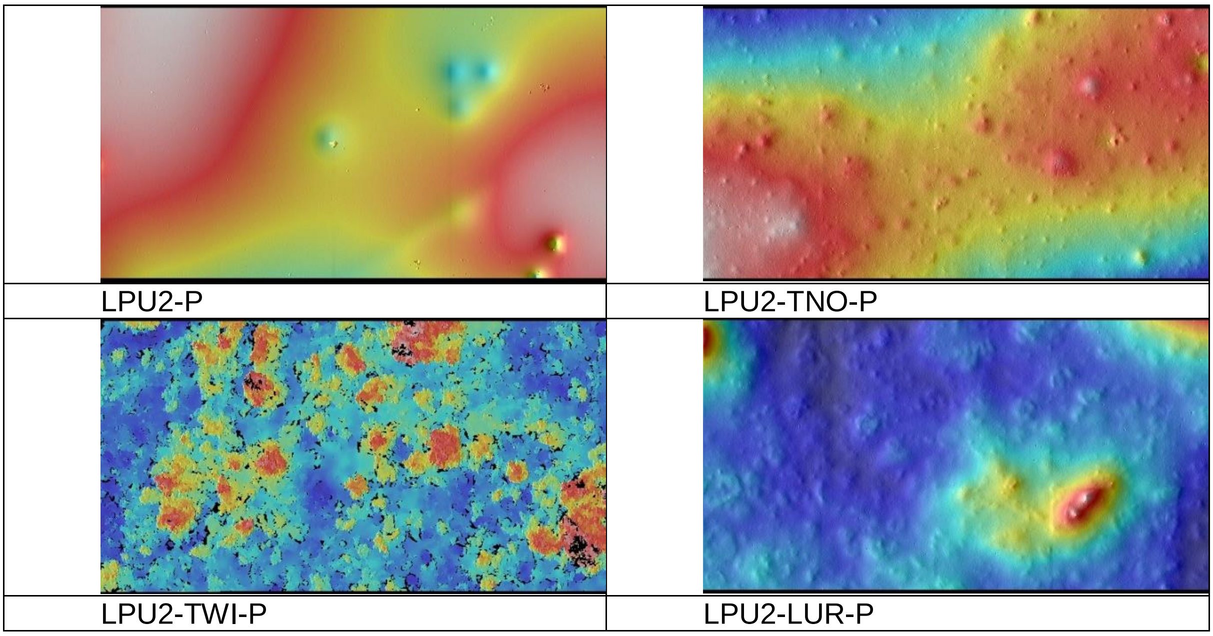


Figure 6 Typical images from different samples (red= high, blue = low) (area: 1.07 x 0.48 mm²)

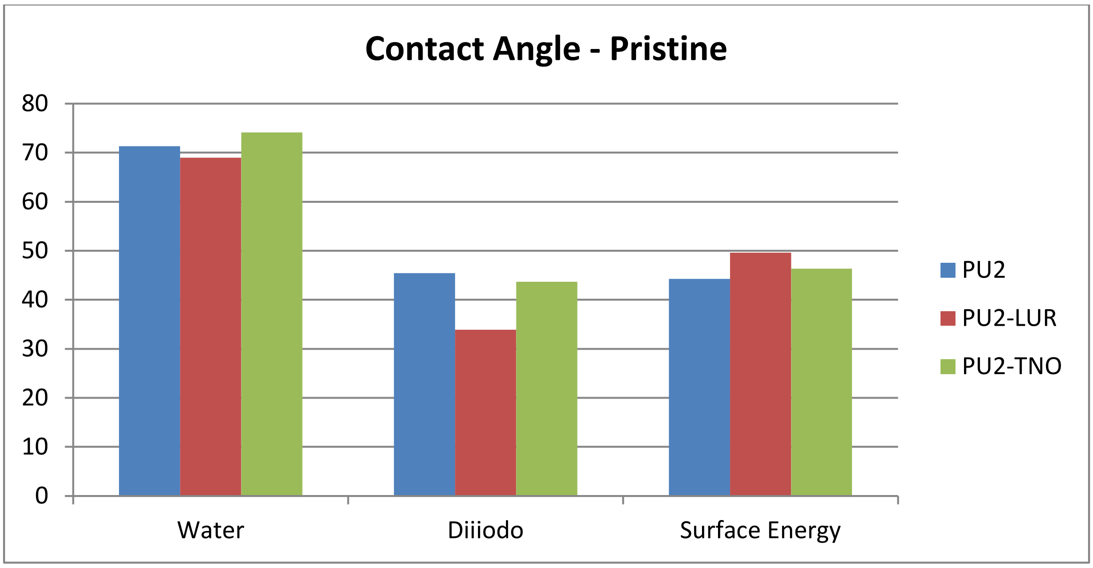


Figure 7 Contact angles and surface energy of pristine surfaces

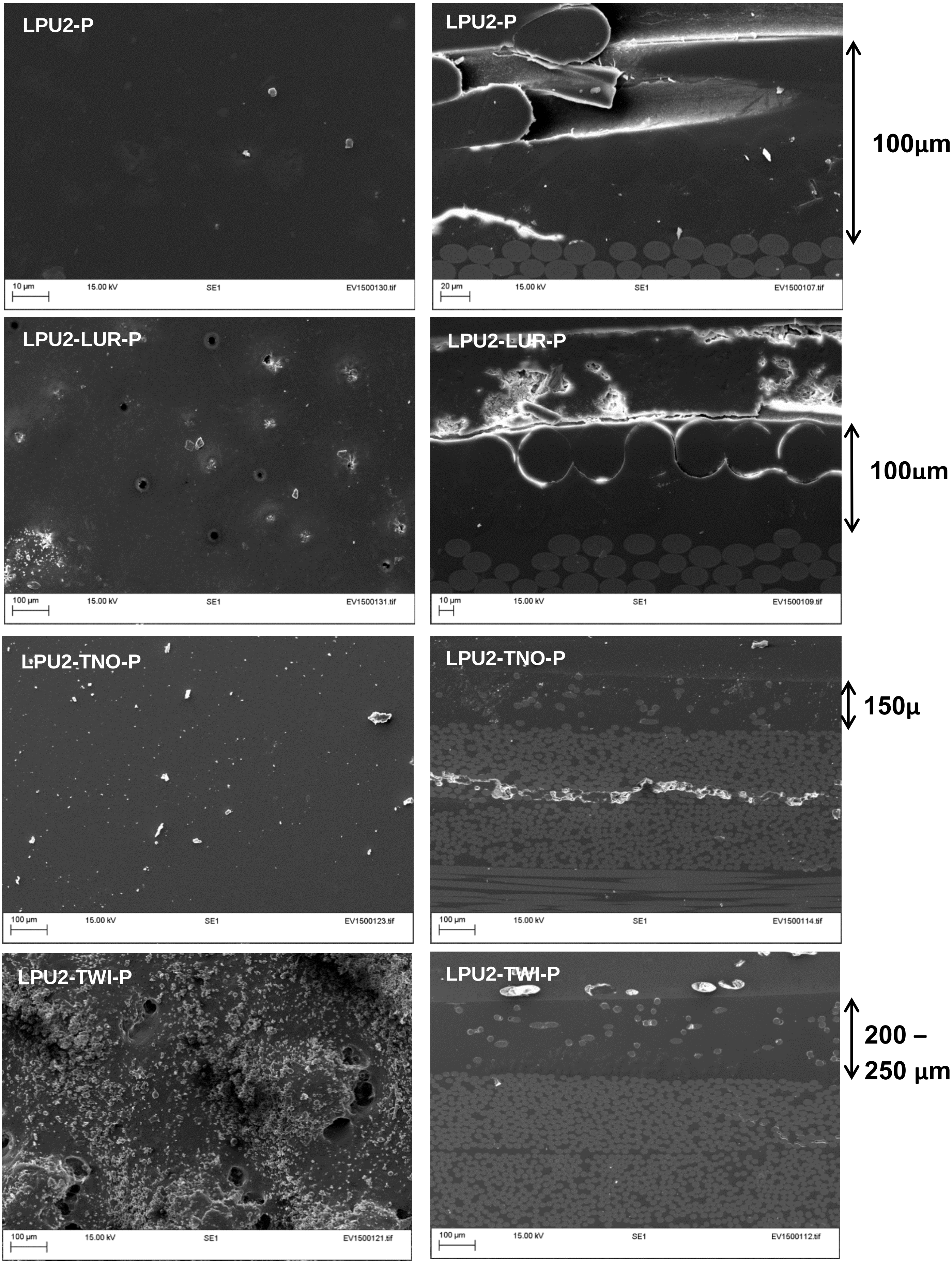


Figure 8 SEM micrographs of pristine coatings showing topography and cross-sectional views

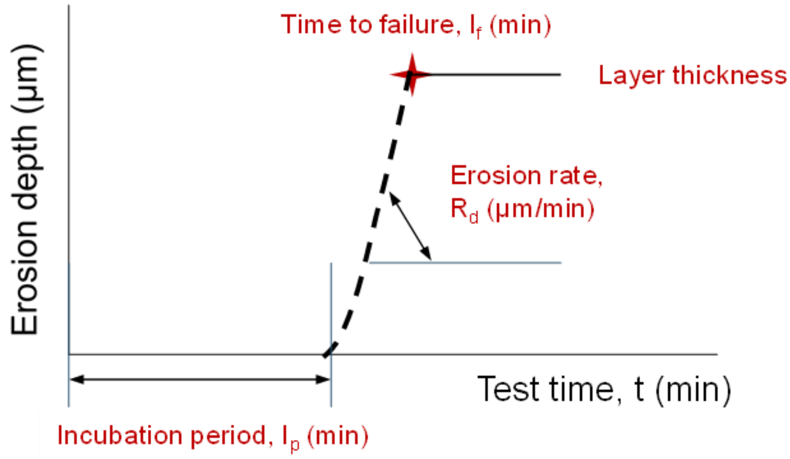
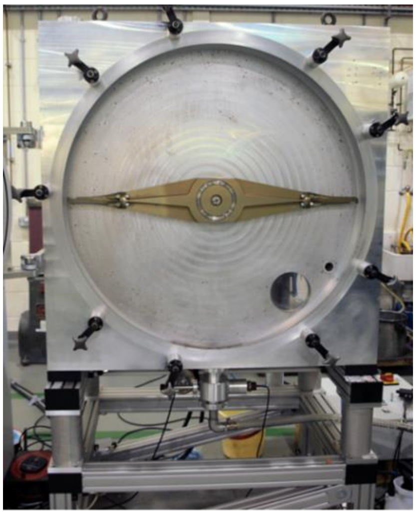


Figure 9a Key test criteria for a coating material determined with droplet erosion tests



Arm installed in chamber

Figure 9b 2Water droplet erosion equipment at NPL

Figure 9c 1 Natural seawater mist cabinet for biofouling test at ENDURES B.V. in Den Helder

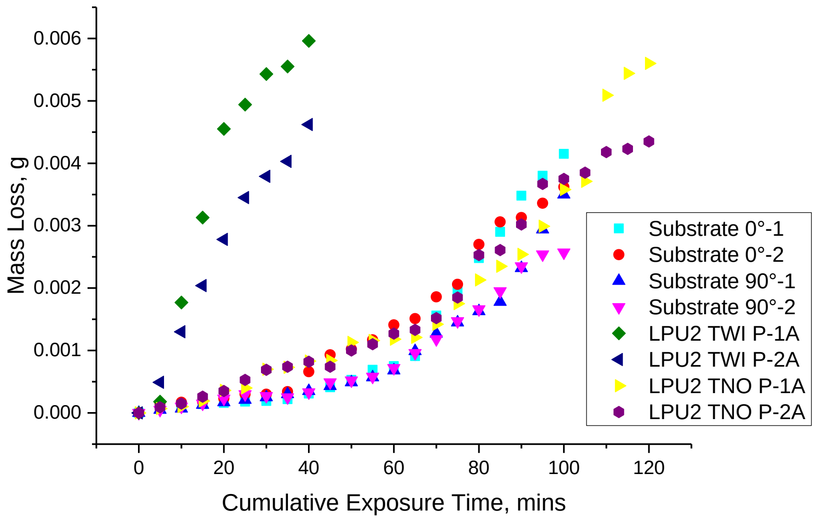


Figure 10 Droplet erosion tests showing time to failure

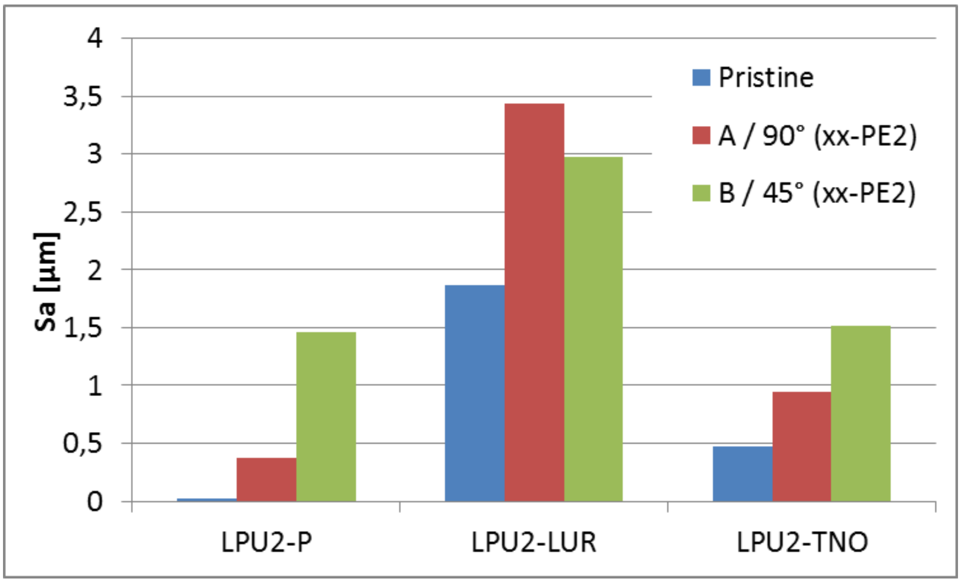
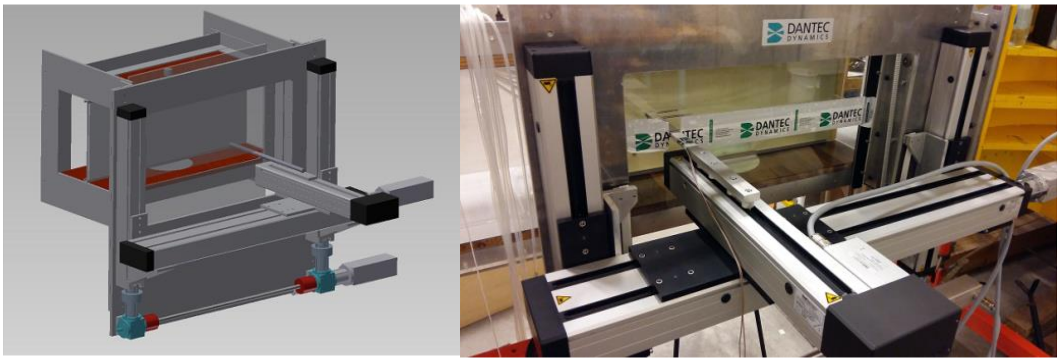


Figure 11 Surface roughness after ageing of the PU2 surface by particle erosion



(a) (b)

Figure 12 Computer-controlled three-axis traversing system (a) the design schematic (b) the actual unit.

(a) (b)

Figure 13 Test section view as seen by the flow (a); angle of attack adjustment for the plate carousel and the angle indicator (b).

Figure 14 Boundary layer and freestream hotwire probes (a) during insertion into probe support outside the test section, (b) during measurements in the boundary layer

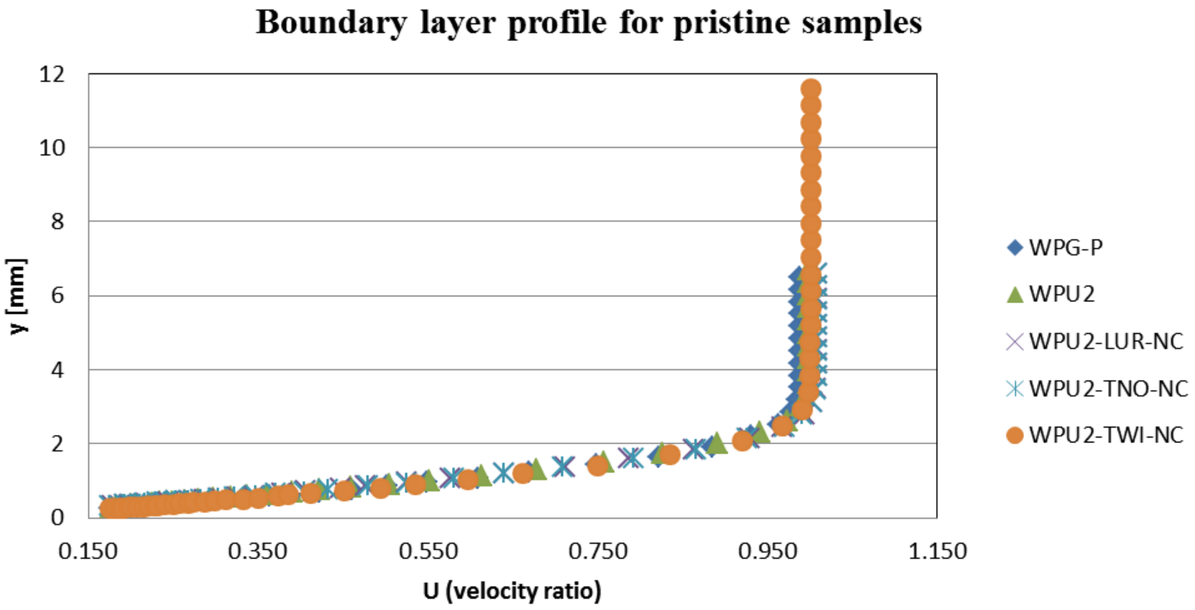


Figure 15 Boundary layer average profile at mid-span at the middle length of the sample plates

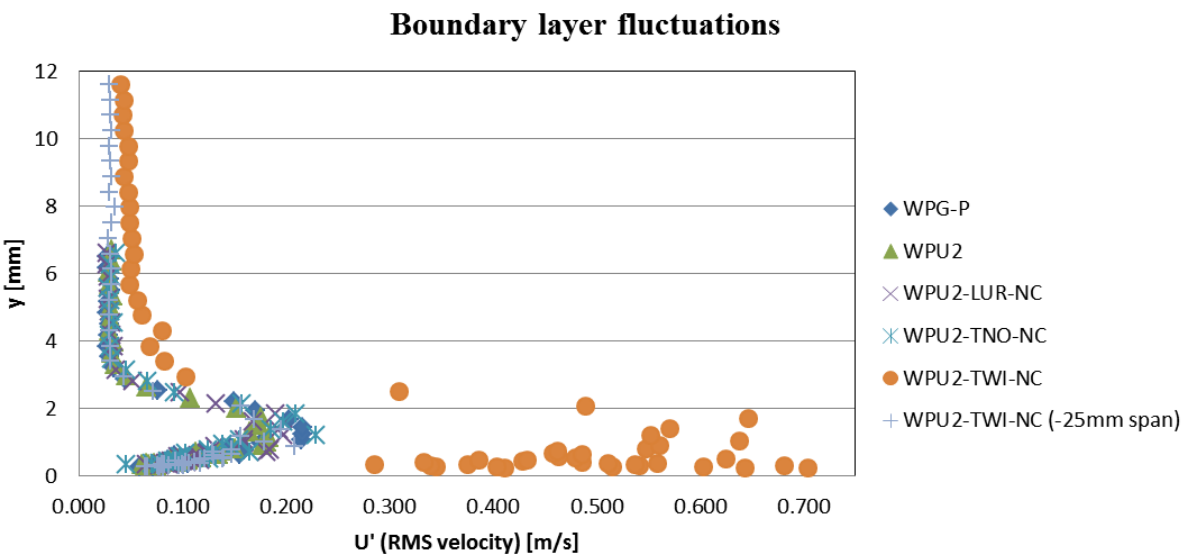


Figure 16 Boundary layer fluctuations at mid-span at the middle length of the sample plates

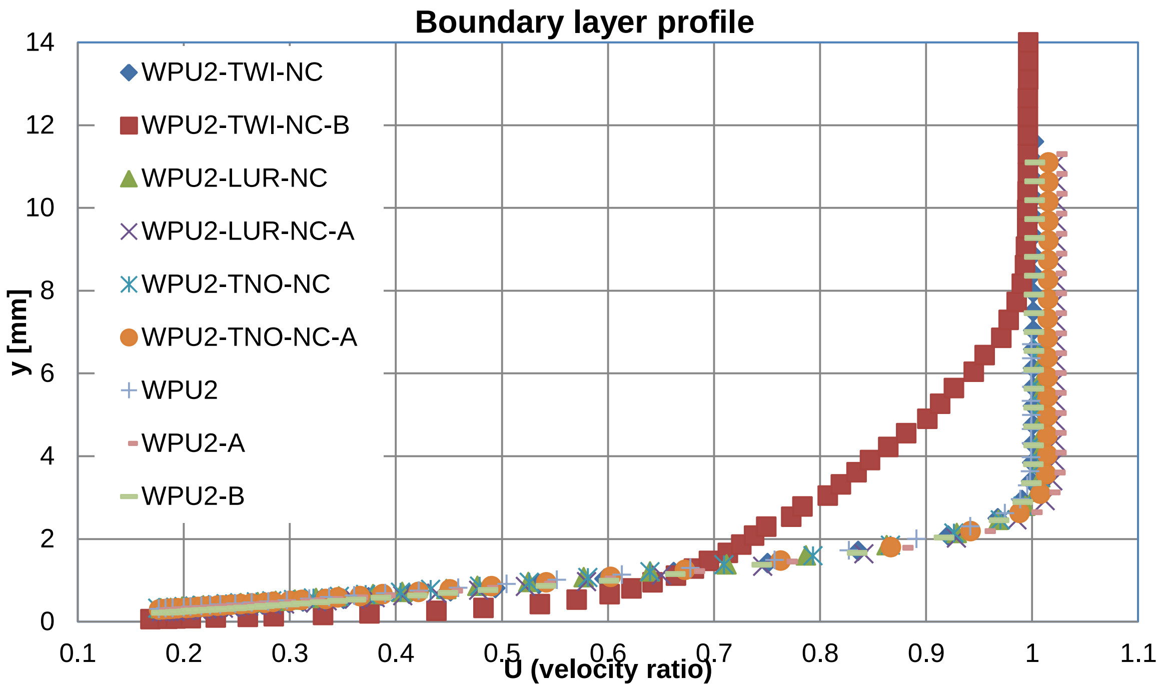


Figure 17 Boundary layer average profile at mid-span at the middle length of the sample plates

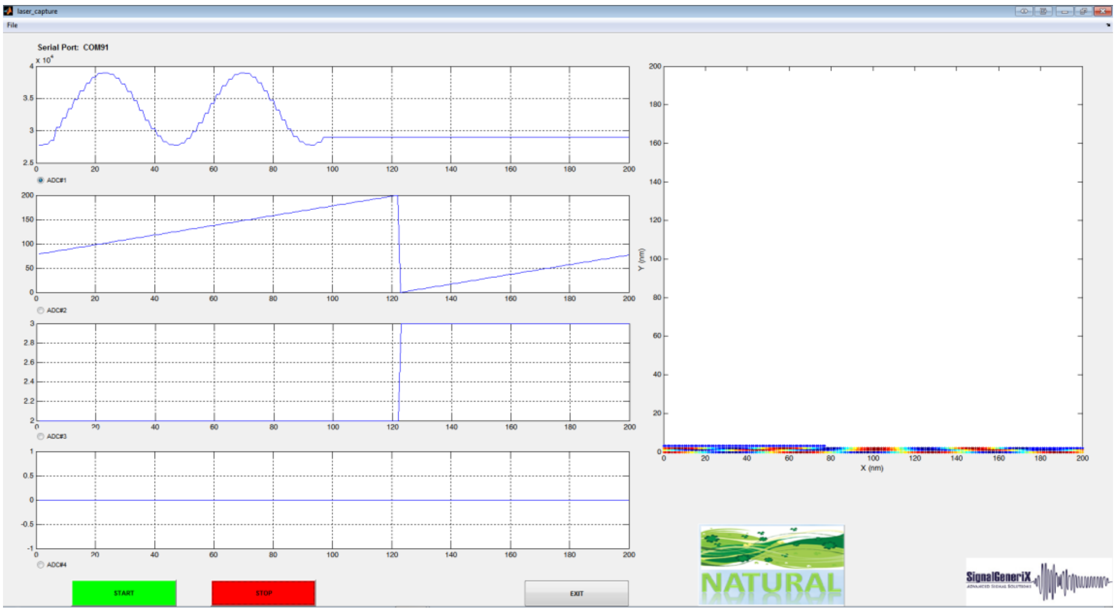


Figure 18 Live signal from the profiler during operation

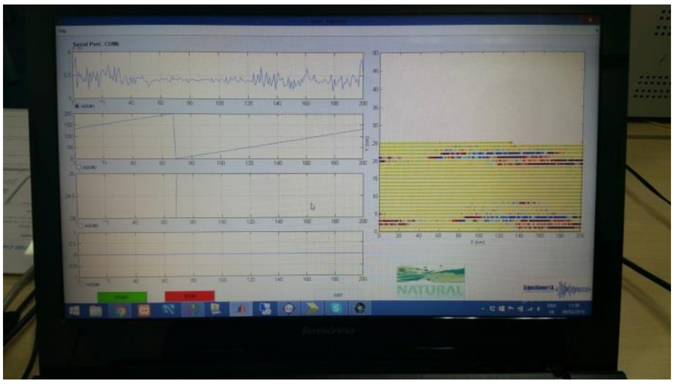


Figure 19 Real-time data during a live scan of a wind turbine blade

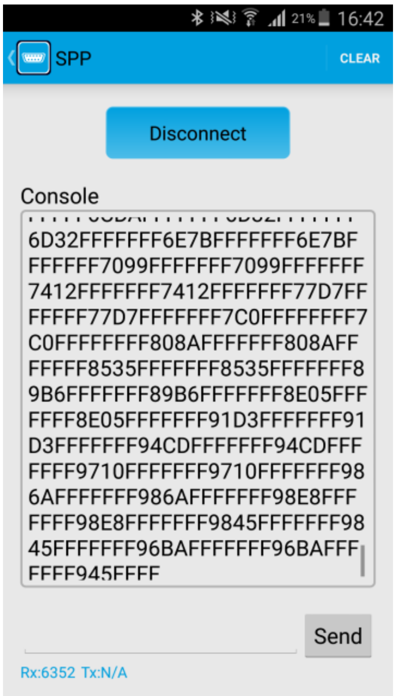


Figure 20 Android data streaming to mobile phone

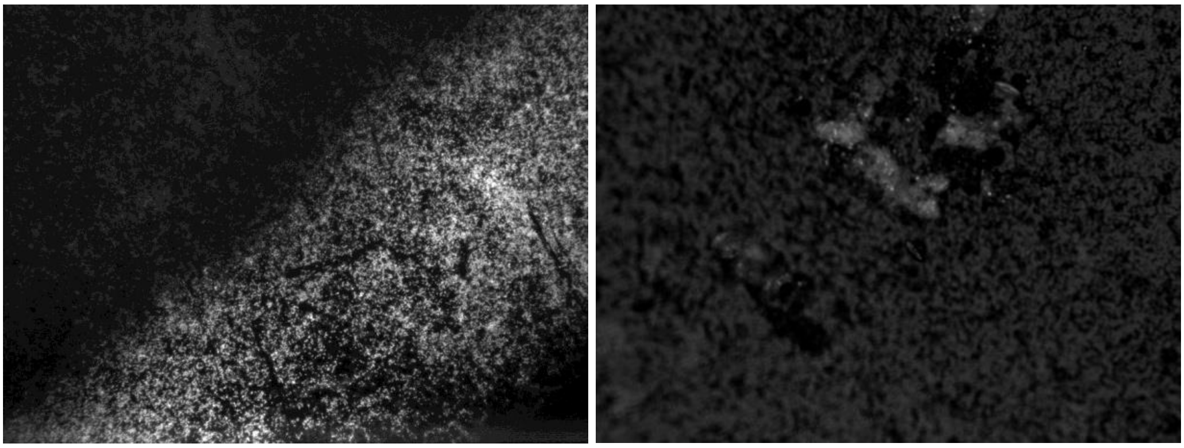
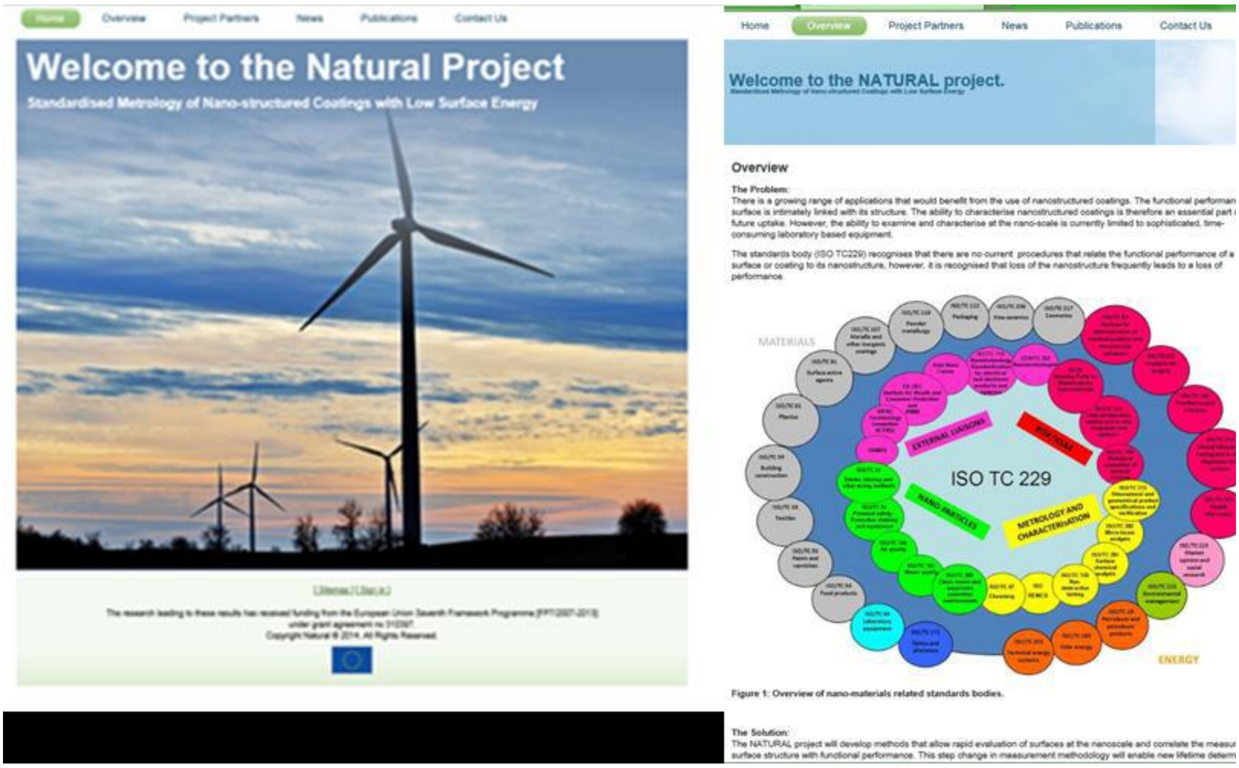


Figure 21 Fluorescence image taken from an undamaged biofouled surface (left) and a damaged biofouled surface (right) of a wind turbine blade



(a) **(b)**

Figure 22 Screenshot of NATURAL's public website

Figure 23 NATURAL logo types. The image on the left is the most commonly used for the project

Table 1 Coating Nomenclature

Coating Name	Nanoparticle supplier
PU2	
PU2-TNO	TNO (Netherlands)
PU2-TWI	TWI (UK)
PU2-LUR	Lurederra (Spain)

Table 2 XPS measured elemental composition summary of PU2 samples

	Atomic %													
Sample	Al	Au	Ba	C	Ca	Ce	F	N	Na	O	Si	Sn	Ti	Cl
LPU2-P	0.0	0.0	0.0	72.4	0.0	0.0	0.0	2.7	0.0	23.5	1.4	0.0	0.0	
LPU2-P	0.0	0.0	0.0	72.1	0.0	0.0	0.0	2.6	0.0	23.7	1.5	0.0	0.0	
LPU2-P	0.0	0.0	0.0	72.4	0.0	0.0	0.0	2.4	0.0	23.7	1.4	0.0	0.0	
LPU2-TNO-P	0.0	0.0	0.0	69.2	0.0	0.0	0.0	3.8	0.0	23.4	3.5	0.0	0.0	
LPU2-TNO-P	0.0	0.0	0.0	69.5	0.0	0.0	0.0	3.6	0.0	23.3	3.6	0.0	0.0	
LPU2-TNO-P	0.0	0.0	0.0	69.6	0.0	0.0	0.1	3.7	0.0	23.0	3.5	0.0	0.0	
LPU2-LUR-P	0.0	0.0	0.0	71.3	0.0	0.0	0.0	2.3	0.0	24.0	2.4	0.0	0.0	
LPU2-LUR-P	0.0	0.0	0.0	70.3	0.0	0.0	0.0	2.0	0.0	24.3	3.4	0.0	0.0	
LPU2-LUR-P	0.0	0.0	0.0	71.3	0.0	0.0	0.0	2.6	0.0	23.9	2.1	0.0	0.0	
LPU2-TWI-P	0.0	0.0	0.0	32.1	0.0	0.0	0.0	1.8	0.0	43.0	23.0	0.0	0.0	
LPU2-TWI-P	0.1	0.0	0.0	32.9	0.0	0.0	0.0	2.1	0.0	42.3	22.6	0.0	0.0	
LPU2-TWI-P	0.1	0.0	0.0	32.8	0.0	0.0	0.0	2.1	0.0	42.6	22.5	0.0	0.0	

Table 3 Nanoindentation results

Variant	Hardness (GPa)
LPU2-P	0.20
LPU2-LUR-P	0.16
LPU2-TNO-P	0.26
LPU2-TWI-P	0.32

Table 4 Results from Tubular impact testing

Sample	mm-kg
LPU2 - P	375
LPU2-TNO-P	250
LPU2-LUR-P	1000
LPU2-TWI-P	600

Table 5 Percentages of covered surface of bio-fouled samples

Sample	Min [%]	Max [%]	Average [%]
LPU2-B (25samples)	3.06	12.71	**6.53**
LPU2xTWI-B-B (21samples)	3.90	18.18	**10.80**
Glass reference (4samples)	1.13	7.90	**5.64**

Table 6

Table 7 XPS measured elemental composition summary of biofouled samples

	Atomic %																	
Sample	Al	Au	Ba	C	Ca	Ce	F	N	Na	O	Si	Sn	Ti	Cl	Mg	S	Sr	Br
LPU2-B	0.0	0.0	0.0	60.8	0.0	0.0	0.0	5.2	1.2	26.6	0.8	0.0	0.0	2.1	1.8	1.4	0.0	0.1
LPU2-B	0.0	0.0	0.0	60.3	0.0	0.0	0.0	5.0	1.3	27.8	0.8	0.0	0.0	2.0	1.4	1.3	0.0	0.1
LPU2-B	0.0	0.0	0.0	61.4	0.0	0.0	0.0	5.2	1.2	26.4	0.8	0.0	0.0	1.8	1.7	1.2	0.0	0.1
LPU2XBB TWI	0.0	0.0	0.0	63.7	0.0	0.0	0.0	3.9	1.4	25.2	1.1	0.0	0.0	1.8	1.2	1.7	0.0	0.0
LPU2XBB TWI	0.0	0.0	0.0	64.6	0.0	0.0	0.0	3.6	1.1	25.3	0.7	0.0	0.0	1.5	1.3	1.8	0.0	0.0
LPU2XBB TWI	0.0	0.0	0.0	63.6	0.0	0.0	0.0	3.2	1.4	25.7	0.9	0.0	0.0	1.5	1.5	2.1	0.0	0.0

Table 8 Water droplet erosion tests before and after ageing

Substrate: Glass fibre reinforced epoxy
Thickness: 5 mm
Coating thickness: ± 100 µm

Sample No.	foil/coating		Coating density (mg/mm^3)	Droplet erosion coating Incubation time, I_p (min)	Erosion rate R_e (µm/min)	Failure time, I_f (min)
1	LPU2-P	Pure	0.95	24	9	32
2	LPU2-LUR	Nano-particles LUR (3%)	1.00	1	100	1
3	LPU2-TWI	Nano-particles TWI (40%)	1.63	7	17	12
4	LPU2-TNO	Nano-particles TNO (1%)	0.97	120	3	139
5	LPU2-P-B	pure	0.95	3	34	5
6	LPU2-TWI-B	Nano-particles TWI (40%)	1.63	5	32	8

Table 9 Hardness values of pristine vs. biofouled coatings

Variant	Hardness (GPa)
LPU2-P	0.20
LPU2-LUR-P	0.16
LPU2-TNO-P	0.26
LPU2-TWI-P	0.32
LPU2B	0.26
LPUxB-B (TWI)	0.24

Table 10 Overview of the pristine, abraded and biofouled samples

Sample ID	Description
WFG-P	Non-coated
WPU2	Polyurethane coating
WPU2-TWI-NC	TWI nanoparticles with polyurethane base
WPU2-TNO-NC	TNO nanoparticles with polyurethane base
WPU2-LUR-NC	LUR nanoparticles with polyurethane base

Table 11 Overview of the pristine, abraded and biofouled samples

Sample ID	Description
WFG-P	Fiber Glass – Pristine (ZPG alignment)
WMANK-P	M2 - Pristine
WPU2-P	PU2 - Pristine
WPU2-TWI-P	PU2 – TWI nanoparticles - Pristine
WPU2-TNO-P	PU2 – TNO nanoparticles - Pristine
WPU2-LUR-P	PU2 – LUR nanoparticles - Pristine
WPU2-B	PU2 - Biofouled
WPU2-TWI-B	PU2 – TWI nanoparticles - Biofouled
WPU2-A	PU2 - Abraded
WPU2-TNO-A	PU2 – TNO nanoparticles - Abraded
WPU2-LUR-A	PU2 – LUR nanoparticles - Abraded

Table 12 Physical parameters and morphology

Parameter	Must have (minimum specification)	Comments
Detection requirements		
Depth resolution in nanostructure measurement area (z-direction)	150nm	Miniaturisation challenge
Transverse resolution in nanostructure measurement area (x- and y-directions)	1 µm	Damage over a large area on the blade should be made visible
Transverse resolution to detect large surface defects, microstructure measurement area (x- and y-directions)	50-150 µm	Larger surface defects are to be determined otherwise?
Final output	- Roughness - Fluorescent spectrum - Coating topography - Water droplet shape analysis (contact angle)	Quantified / semi-quantified assessment to assist service operatives

Table 13 Parameters for optical device

Parameter	Must have (minimum specification)	Nice to have (target specification)
Measurement area	5mm x 5mm	500mm x 500mm
Measurement on a curved area	Measure single curved surface	Double curved surface
Weight	15kg	<10kg
Size	30x20x20cm	< shoe box
Other properties	Waterproof, dustproof (e.g. IP65)	Floatable Shock proof
Mounting of device to the blade.	Stable fixation of the device to the blade to gain optimal resolution	Measurement on various spots.
Power supply/charging Voltage	240V	12V USB connectivity (5V)
Power source and Battery life time **- On operational time** **- On stand by**	 8hrs 24hrs	 24hrs 100hrs
Acceptable materials	ROHS compliant materials	

Table 14 Parameters for data processing

Parameter	Must have (minimum specification)	Nice to have (target specification)
Data processing	Off-line (PC)	Real time (on equipment on a 'live' turbine (e.g. up in the air, free hanging))
Communications to PC	USB or Serial Port	Wireless (e.g. Bluetooth, WLAN, GPRS)
Status/warnings	Displayed on equipment	Displayed on equipment and remotely on PC
3D Graphics Plot	Displayed on PC	Displayed on equipment
Reporting options and analysis	Exporting to .xls or .txt format to allow CAD 'cloud'	Allow statistical analysis of the data
Control of Laser	Via equipment (keypad or touchscreen)	Via equipment and remotely from PC

Table 15 A list of dissemination events attended during the duration of the Project

Dates	Type	Type of audience	Countries addressed	Partner responsible /involved
22-23/09/13	International conference on surface coatings and nanostructured materials - Nanosmat 2013	Nano-materials community	Spain	TWI
20-21/05/14	International Conference on Structural Nano Composites Nanostruct 2014	Nano-materials and characterisation community	Spain	TWI
27-29/05/14	E-MRS 2014 Spring meeting – Symposium H: ALTECH 2014 - Analytical techniques for precise characterization of nanomaterials	Nanomaterials and characterisation research community	France	TWI
6-8/05/14	European Symposium on Friction, Wear and Wear Protection 2014	Materials characterisation community	Germany	TWI
5-6/06/14	Congress on Surface Finishing in the Aeronautics and Aerospace Industries - Surfair 2014	Aerospace coating community	France	TWI
3-5/09/14	European Technical Coatings Congress - ETTC2014	European coatings community	Germany	TWI
9 /10/13	Gamesa	Advisory board	Germany	TWI
30/01/14	Mankiewicz	Advisory board	Germany	TWI
23-24/10/13	ISO/TC35/ SC9/WG32	ISO standard for Coating materials for wind-turbine rotor blades and tidal-steam driven rotor blades.	Germany	TWI
16 /10/14	Coatings conference (eg Itapic, or VVVF)	Coatings producers	NL	TNO
28/01/2016	Presented research	Scientists community	Spain	SO

	plan of the doctoral thesis entitled “Device and strategy for surface energy measurement”			
15/07/2015	Poster presented at OPTOEL congress	Specialists in the field of photonics and optoelectronics.	Spain	SO
10/2015	Poster presented at IOM3 UK Tribology Network Launch event	Physicists, chemists, material scientists, biologists, mathematicians and computer scientists involved in tribology and industrialists involved in R&D.	UK	TWI
11/2015	Flyers at RSC Chemistry for Collaboration networking event	Chemical community	UK	TWI
28-30/06/2015	Talk at Microscience Microscopy Congress 2015 (MMC 2015)	Scientists community	UK	NPL
22-26 June 2015	COSI 2015	Academia and industry	Netherlands	TNO
12 November 2015	Day of Surface technology	Academia and industry	Netherlands	TNO

Table 16 List of key exploitable results proposed at the start of the project

No.	Key Exploitable Result (KER)	Abbreviation
1	Prototype for nano-characterisation measurement system capable of rapid non-destructive evaluation in the field.	Prototype
2	Algorithms linking the nanostructure of the surface to the performance. Develop understanding of practical characterisation methods for nano-structured coatings with a focus on low energy coatings for anti-fouling applications and/or for low friction applications such that the results can be interpreted in a meaningful fashion in a real-world application.	Algorithms
3	Lead position via standardisation and know-how in characterisation of nanostructured coatings. Initialisation of standardised method to determine the lifetime of nanostructured coatings.	Standardisation & Know-How

Table 17 List of key exploitable results proposed at the end of the project

No.	Key Exploitable Result (KER)	Key Partner
1	Device for measuring the topography and evaluating the surface energy of a solid surface by interferometric profilometry	SO
2	AMSCAN : Totally innovative – miniature portable	AMT, SG