LOTUS

LOW-COST INNOVATIVE TECHNOLOGY FOR WATER QUALITY MONITORING AND WATER RESOURCES MANAGEMENT FOR URBAN AND RURAL WATER SYSTEMS IN INDIA

Deliverable D2.2

First version LOTUS prototype

Lead: EGM

Date: 30/09/2020

- Public -

LOTUS is co-funded by the European Commission under the Horizon 2020 research and innovation programme under Grant Agreement N° 820881 and by the Indian Government, Ministry of Science and Technology.

Project Deliverable

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D2.2 First version LOTUS prototype – public summary

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Abstract
This report describes the chemical, electronical, mechanical and software design of the different elements constituting the LOTUS sensor node.

Keywords
LOTUS node design

Disclaimer

The LOTUS Project

LOTUS is a project funded by DG Environment under the European Union Horizon 2020 Research and Innovation Programme and by the Indian Government. It brings together EU and Indian prominent organisations with the aim to co-create, co-design and co-develop innovative robust affordable low-cost sensing solutions for enhancing India's water and sanitation challenges in both rural and urban area.

The LOTUS solution is based on an innovative sensor and includes tailor-made decision support to exploit the capabilities of the sensor as well as a specific approach to co-creation. LOTUS aims to be co-designed and co-produced in India, and have a wide, diverse and lasting impact for the water sector in India due to intense collaborations with commercial and academic partners in India.

Based on the low-cost sensor platform, solutions for the early detection of water quality problems, decision support for countermeasures and optimal management of drinking and irrigation water systems, tailored on the functionalities of the new sensor, will be developed and integrated with the existing monitoring and control systems.

This sensor will be deployed in five different use cases: in a water-network, on ground-water, in irrigation, in an algae-based waste water treatment plant and in water tankers. The packaging of the sensor, as well as the online and offline software tools will be tailored for each of the use cases. These last will enable to test the sensors and improve them iteratively.

The project is based on co-creation, co-design and co-production between the different partners. Therefore, an important stakeholder engagement process will be implemented during the project lifetime and involve relevant stakeholders, including local authorities, water users and social communities, and will consider possible gender differences in the use and need of water. Broad outreach activities will take place both in India and in Europe, therefore contributing to LOTUS impact maximisation.

The further development and exploitation (beyond the project) of the novel sensor platform will be done in cooperation with the Indian partners. This will create a level playing field for European and Indian industries and SMEs working in the water quality area.

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1 Executive Summary

The LOTUS node comprises the sensing elements, together with the electronic and embedded software allowing proper data acquisition, processing and transmission to a computer for local analysis (required during development phase) and the network so to be consumed by external applications such as SCADA, cloud applications or mobile applications. These components are packaged into a mechanical housing aimed at withstanding use case requirements.

The high-level requirements are firstly been extracted from the LOTUS D6.1 “detailed specification of use cases, sensor requirements, and success criteria” then the LOTUS node is breakdown into its individual components. The design of these different parts is then detailed.

Firstly, the main principles of the Carbon nanotube based water sensor are explained and their deposit and functionalisation by specific polymers using ink-jet process is described.

Then two options to integrate the sensor chip using a flex-pcb within a PVC mechanical body are provided with the mechanical details of the housing as well as glueing procedures.

Finally, the focus is made on the electronic part covering first the analog front-end (transforming analog signal from the sensor chip into pre-processed digital signals) and the LOTUS box interfacing this AFE to other applications of the project.

2 System requirements and overview

2.1Synthesis of use case requirements

The deliverable D6.1 provides an analysis of project use cases, identifying their high-level requirements. From the report, requirements relevant to the WP2 work (thus not considering cloud and application aspects) have been extracted in summarised in Table 1.

Table 1: Synthesis of use case requirements

	UC1 (Guwahati distribution network)	UC2 (tanker-based distribution)	UC3 (Irrigation water distribution network)	UC4 (Groundwater and river water monitoring)	UC5 (Wastewater treatment)
Energy					
Power availability	Electric posts or transformers (except 1 site)	24V on board power supply	Available on spot		Local Computer
Power harvesting	Inline hydrogenerator suggested	Additional battery + PV possible			
Autonomy requirements	Battery recommended			8hrs autonomy battery powered	
Mechanic					
Mounting	Tapping sleeve for 100-700mm pipe			Sensor attached for fish weight at bottom Portable box	
Filter/membrane	Required (dust & biofouling removal)				

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	UC1 (Guwahati distribution network)	UC2 (tanker-based distribution)	UC3 (Irrigation water distribution network)	UC4 (Groundwater and river water monitoring)	UC5 (Wastewater treatment)
Pressure	25 bars max	1 bar max	0.5 to 10 bars	1.96 bar (river water pressure)	
Flow	3500 m^3/h max		5 to 200 m^3/h.	8 m/s (Varanasi city)	
Vibration		Continuous during delivery			
Lotus Sensor IP rating	IP68 (possible flooding)		Exposed to weather. Agriculture conditions		
Lotus box IP rating	??	Outside. 15-35°C. Average humidity	Exposed to weather. Agriculture conditions		N/A
Other			Theft risk		
Connectivity					
Cloud	Wired to RTU (Modbus)	Sim card 3G/4G			
Local		Modbus to PLC (Chlorinator)			Connected to local PC
Functioning					
Reading minimum rate	1 every 5 min	1 every 2 min		Response time < 2 min	Response time < 15 min
Transmission minimum rate	1 per hour			Manual (daily / weekly)	
Data logging	Local storage in lotus box			Minimum storing of 1000 reading (water quality parameter + GPS)	
Other			Need to connect other sensors locally	GPS position	

	UC1 (Guwahati distribution network)	UC2 (tanker-based distribution)	UC3 (Irrigation water distribution network)	UC4 (Groundwater and river water monitoring)	UC5 (Wastewater treatment)
Stability and maintenance					
LOTUS sensor lifetime	10 years		> 5 years	> 3 years	>1 year
sensor maintenance and calibration	Every 6 months (incl. membrane replacement)		6 months		3 months

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2.2 System overview

The lotus node is the edge equipment of LOTUS containing water quality sensor with transmission capabilities to external systems such as SCADA, cloud systems or mobile applications. The simplified view of the data flow is provided in Figure 1. The sensing head is powered by an analog front end which transforms analog signals from the sensing head into digital signals consumed by the lotus box which provides an adaptive connection to external systems. It also ensures energy powering, local data processing and storage. This assembly is packaged into a mechanical housing satisfying mechanical and environmental constraints of the use cases.

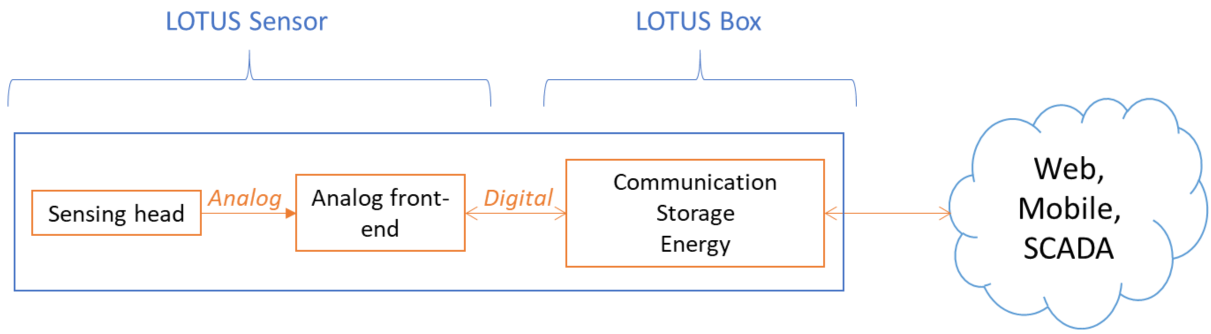

Figure 1: system data flow

2.3System hierarchy

The overall system (named LOTUS NODE) detailed breakdown is provided in Figure 2. The 2 main components are the LOTUS sensor and the LOTUS Box as described earlier.

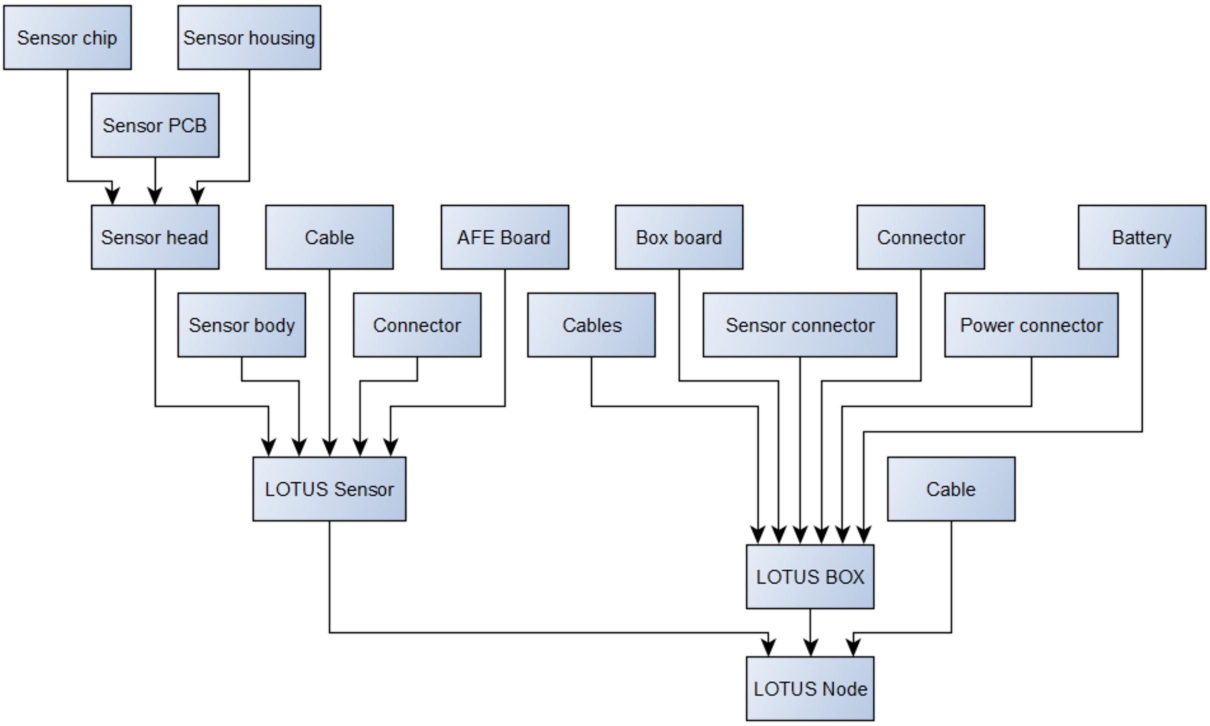

Figure 2: LOTUS Node system breakdown.

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3 System design, fabrication and assembly

3.1 LOTUS Sensor

3.1.1 Principles

This section reports on the different building blocks composing the LOTUS sensor, as shown in the images below (Figure 3 and Figure 4):

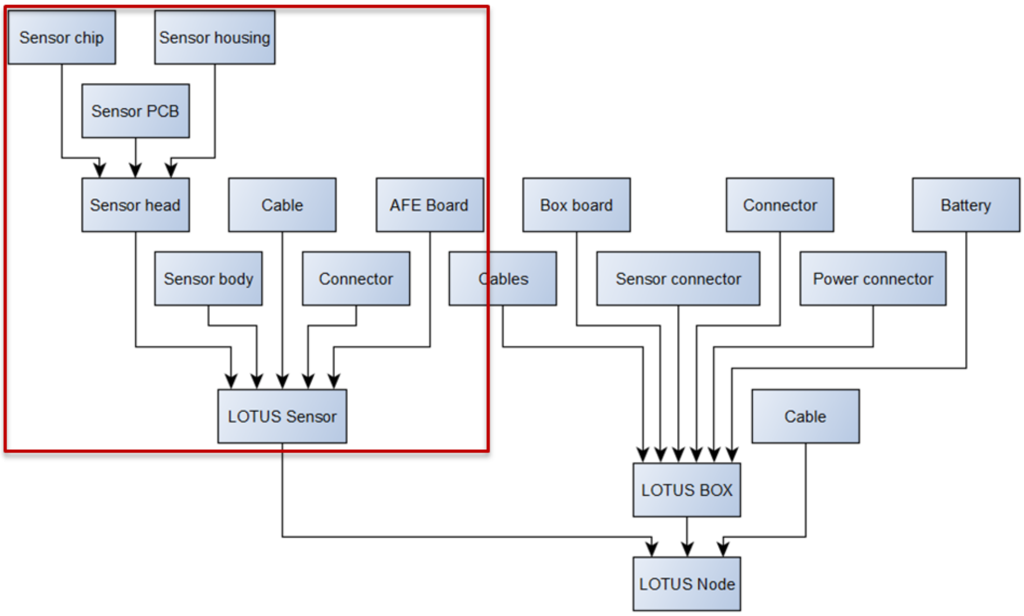

Figure 3: Positioning of the building blocks of the LOTUS sensor with respect to the complete LOTUS node

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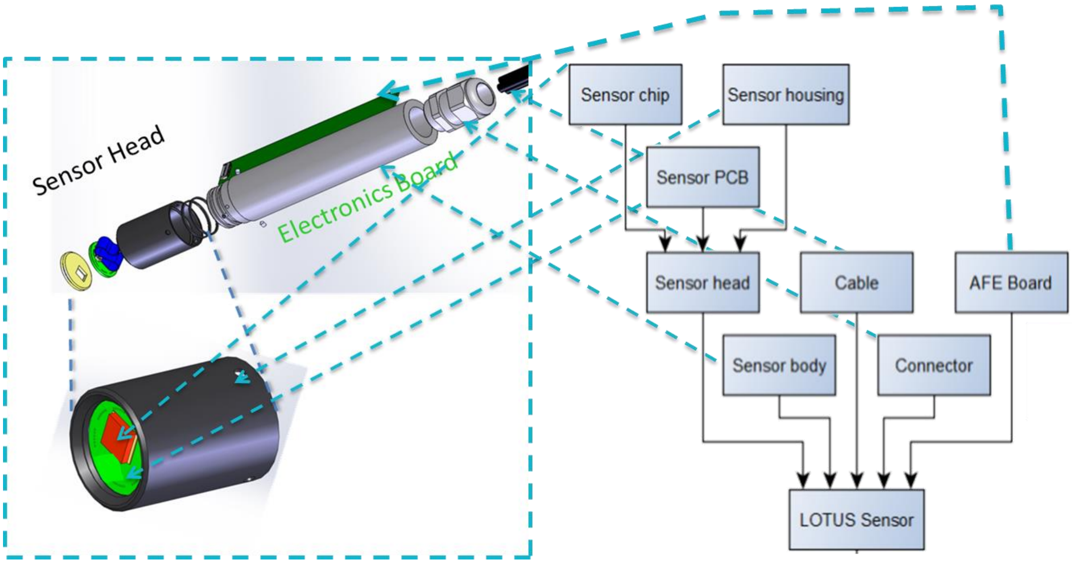

Figure 4: Illustration of the building blocks of the LOTUS sensor

3.1.2 LOTUS Sensor Chip

3.1.2.1 Principles

The parameters targeted by the probes reported in this deliverable are the following:

- Temperature
- Conductivity
- pH
- chlorine

Other parameters will be proposed in later versions, to be reported in deliverables D2.5 and following, such as arsenic and fluoride. The work on those has already started, but completion is only expected in the second period.

3.1.2.2 Sensor chip design

The sensor chip is a 1.44cm² silicon chip. It is fabricated in 4" silicon wafer, 45 chips per wafer

The vertical structure of the chips is shown in Figure 5 :

- Base material: 4" Silicon wafer with Si resistivity between 1 and 10 Ohms.cm; 525µm thickness
- Insulating layer across wafer: Thermal SiO2 layer with 450nm thickness
- Main metal layer (see metal layer description below): 200nm Platinum on Titanium anchoring layer, achieved via cathodic sputtering followed by Lift-off.
- Second metal layer for wirebonding pads only: 500nm Gold layer achieved by cathodic sputtering then Lift-off

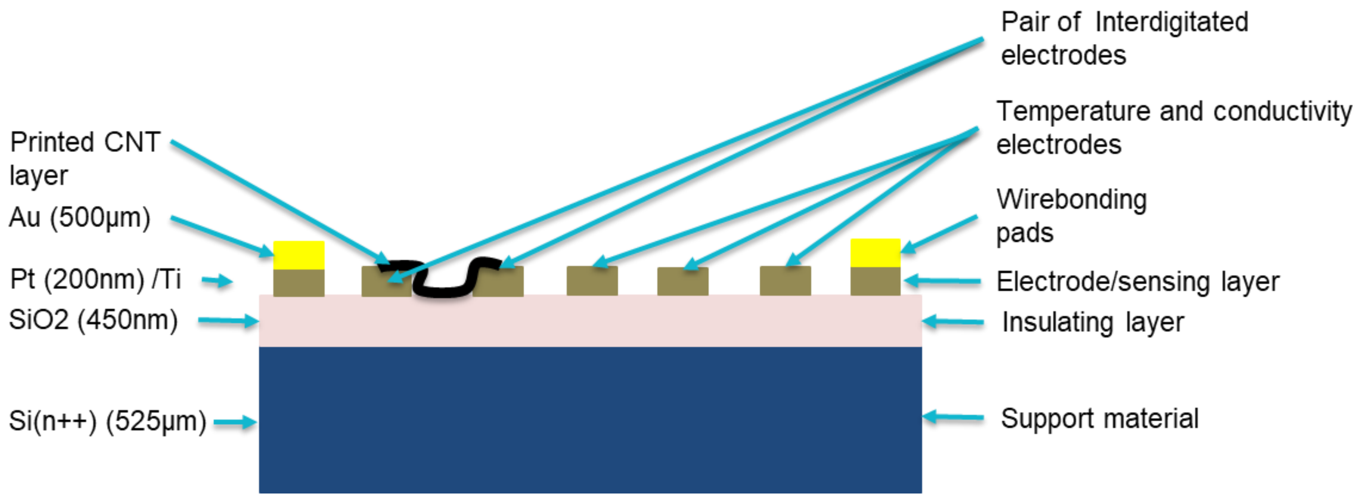

Figure 5: Vertical structure of the chips.

The key element of the chip with respect to sensing is the metal electrode layer. Figure 6 shows the design of the metal layer across a full 4” (10cm diameter) wafer. Each “square” pattern in the image represents a chip of surface area $1.44 cm^2$ (1.2cm x 1.2cm). There are 45 chips per wafer. Those chips are of 4 types, 12 are for research purposes (for testing fabrication process for CNT sensors), 33 are for actual production (for use in actual sensor nodes). All chips have 44 contact pads, 22 on either side.

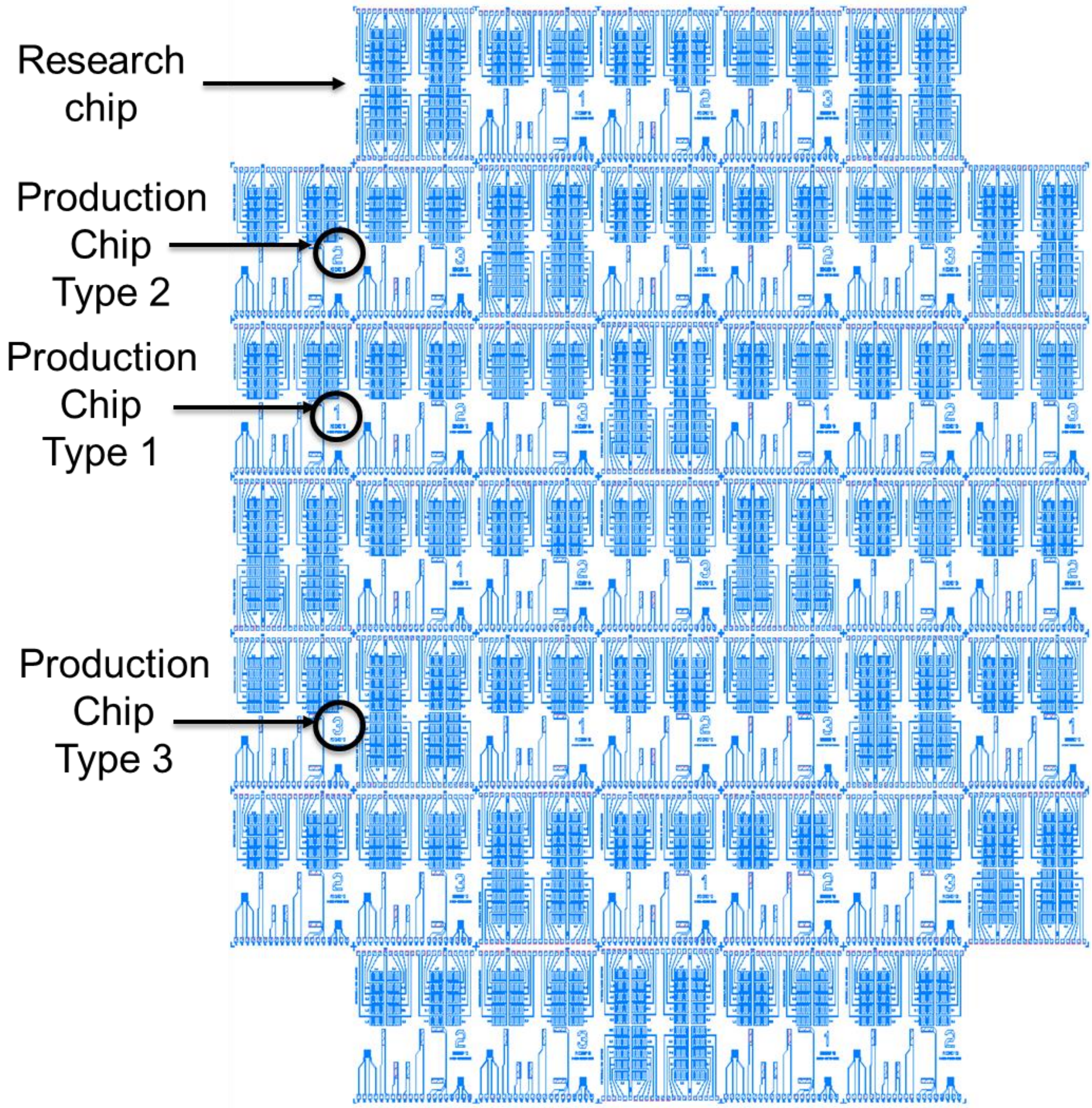

Figure 6: Wafer-scale design of the electrode layer.

The research chips (Figure 7) contain 40 interdigitated electrode (IDE) based devices, with two different geometries of electrodes available on the chip. These IDE electrodes are designed to deposit CNT materials between the electrodes as shown in Figure 5. The production chips contain 20 pairs of

IDE electrodes for CNT sensors, 3 conductivity sensors and 2 temperature sensors as shown in Figure 8.

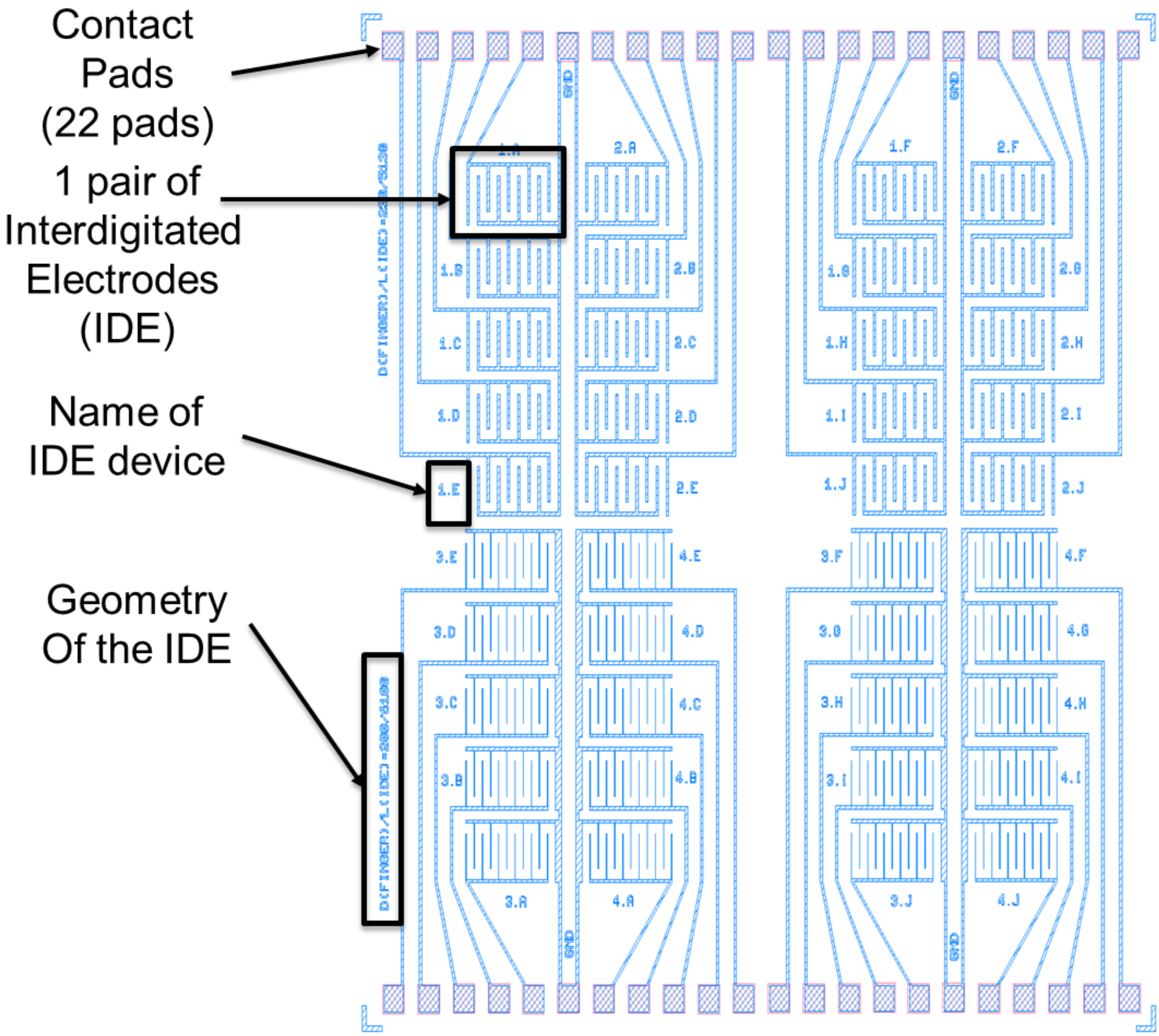

Figure 7: Research chip design with electrodes for 40 CNT sensors

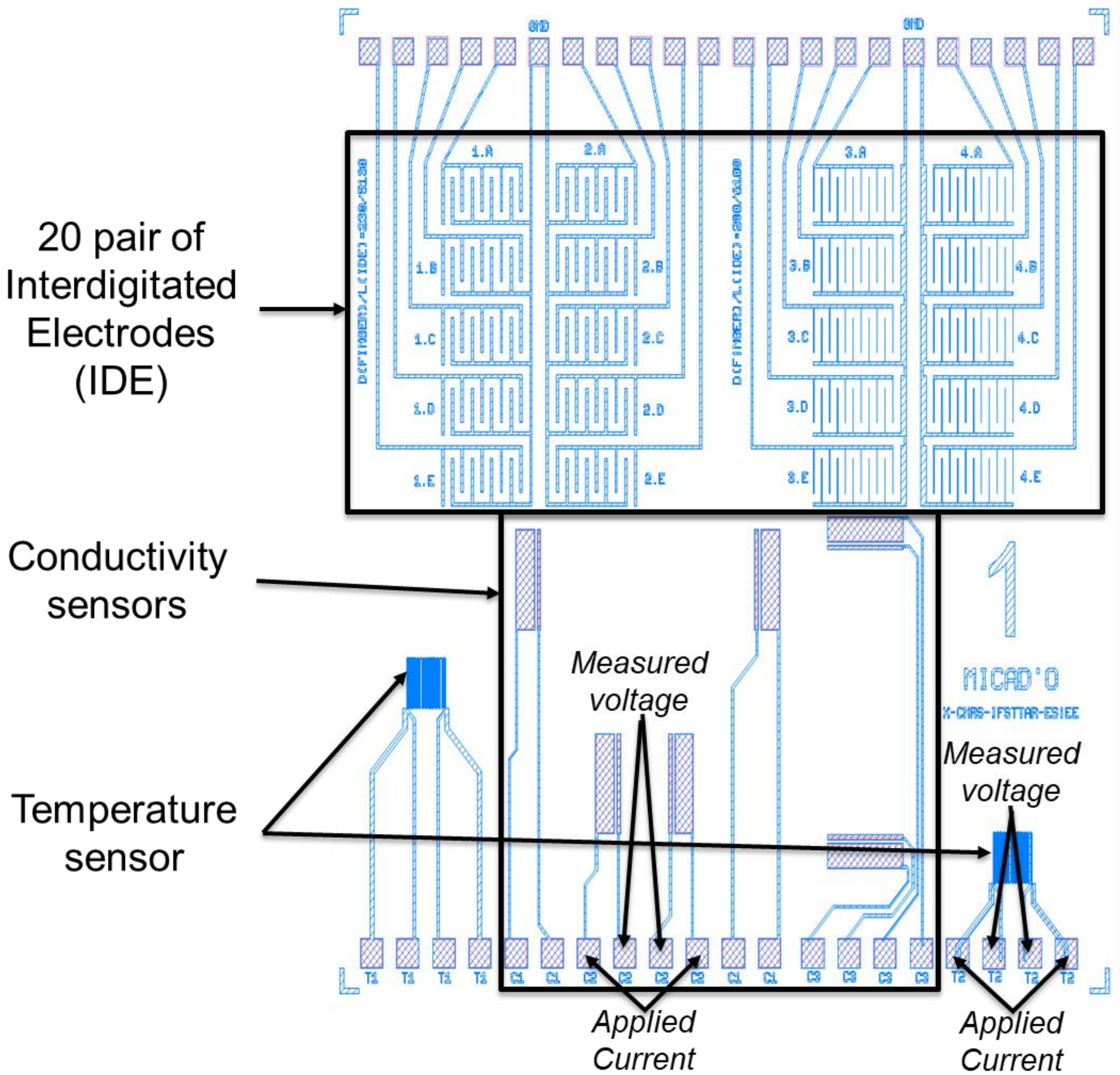

Figure 8: Design of production chip. Example of subversion 1.

The principle of operation of the 3 types of sensors are as follow:

- Temperature sensors: this is an archetypal RTD temperature sensor design. In other words, the platinum layer is coiled between two electrodes. The resistance of the platinum coil is proportional to the temperature. The resistance is measured by 4 wires, two by electrode. Following the classical four wire measurement method, a current is applied between two of the wires, one on each electrode, and the resulting voltage is measured across the other pair of wires.

- Conductivity sensors: the conductivity of the water is measured across two pairs of two electrodes, again in a four-wire measurement approach. The current is applied through the larger electrodes while the voltage drop is measured across the two thinner electrodes. Two difference spacings between pairs of electrodes are proposed (C1 and C3: large spacing; C2: small spacing) as well as two orientations across the chips(C1 and C3) (to confirm the absence of effect of the orientation with respect to water flow).
- Chemical sensors (pH and free chlorine):
  - A thin carbon nanotube layer is deposited between pairs of interdigitated electrodes as in Figure 5. The resistance of the layer is measured in a two wire configuration. Its variation depends on the presence of target chemical in the water.
  - To target N chemicals, N different formulations of CNT ink are needed. To achieve this, carbon nanotubes are functionalized with dedicated functional probes synthesized at Ecole Polytechnique (Patent FR1753131). To provide redundancy, several devices are fabricated with the same functionalization (see Figure 9 and Figure 10-left).
  - For production chips for the first version of LOTUS Nodes, 2 chemicals are target. Thus, two inks are used, one ink with non-functionalized CNT ("pristine CNT" later) (devices 1.X and 2.X on Figure 8) and one ink with CNT functionalized by the FF-UR polymer (see Figure 10-right)(devices 3.X and 4.X on Figure 8).

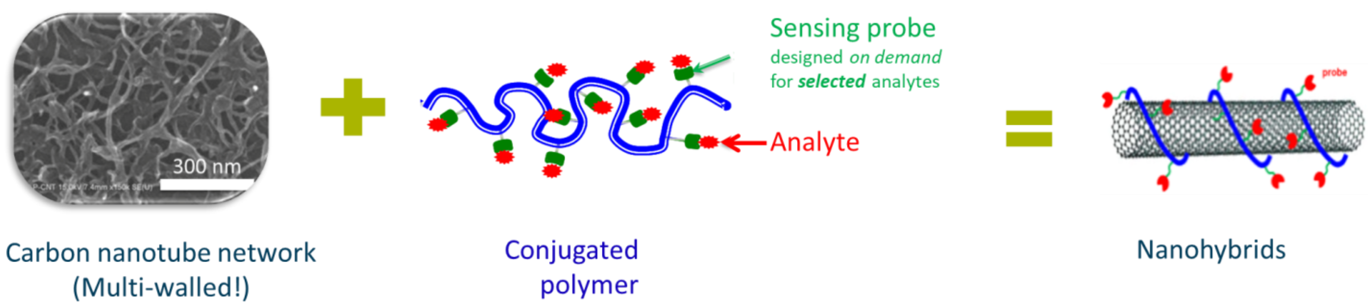

Figure 9: Functionalization of CNT with functional probes based on conjugated polymer

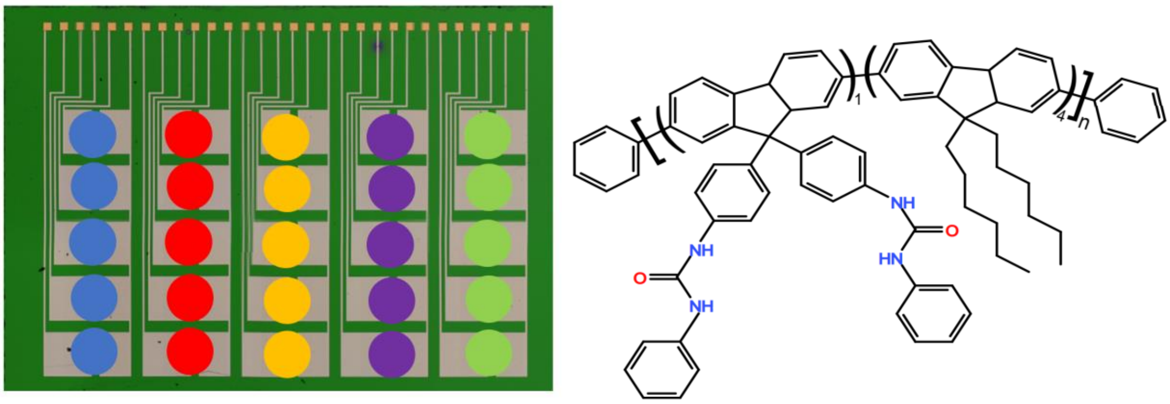

Figure 10: Left - sensor array principle: each colour corresponds to a different ink formulation; there are 5 devices per type of ink (in the schematics, 5 types of ink, 5 devices per type of inks). Right – formula of the FF-UR-14 polymer used in LOTUS sensor chip version 1.0

3.1.2.3 Chip sensor fabrication

The fabrication of CNT sensors atop of the chips described in the paragraph above relies on the following steps:

- Synthesis of FF-UR polymer: process validated in the past (Patent FR1753131) during Proteus project. As no optimization was carried out during LOTUS, the process is reproduced only in the Appendix (Synthesis of FF-UR-14 polymer) for information purpose.
- Preparation of CNT ink and CNT/FF-UR ink: the fabrication process for CNT ink was reused from Proteus, while the process for fabrication of CNT/FF-UR ink was optimized. Details are provided below.
- Printing the two inks on batches of several chips. Optimization was carried out for enhanced reproducibility of the fabrication.

Ink fabrication

Figure 11 summarizes the fabrication process for CNT ink (1), for FF-UR solution (1) and for CNT/FF-UR ink (2) through mixture of CNT ink and FF-UR solution. Figure 12 shows the outcome of the process: images of non-functionalized and functionalized multiwalled carbon nanotubes taken with TEM (transmission electron microscope). Figure 13 shows AFM (Atomic Force Microscope) images of (left) non-functionalized and (right) FF-UR functionalized MWCNT. Analysis of AFM data shows that FF-UR/MWCNTs are wider in diameter than non-functionalized MWCNT by 5.3 nm, which suggests that the FF-UR layer on the MWCNT sidewalls is 2.6nm thick. The process takes 1 full day, plus 1h for finalization 48h afterwards.

<u>Solvent:</u> 1,2-dichlorobenzene (DCB) is used as a solvent to disperse CNT and to solubilize COP.

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Carbon nanotubes: MWCNT from the NC3100™ series purchased from Nanocyl.

CNT ink fabrication process: The process of fabrication of the CNT is adapted from the one reported by the team in Michelis et al, Carbon 95, 1020-1026, 2015.

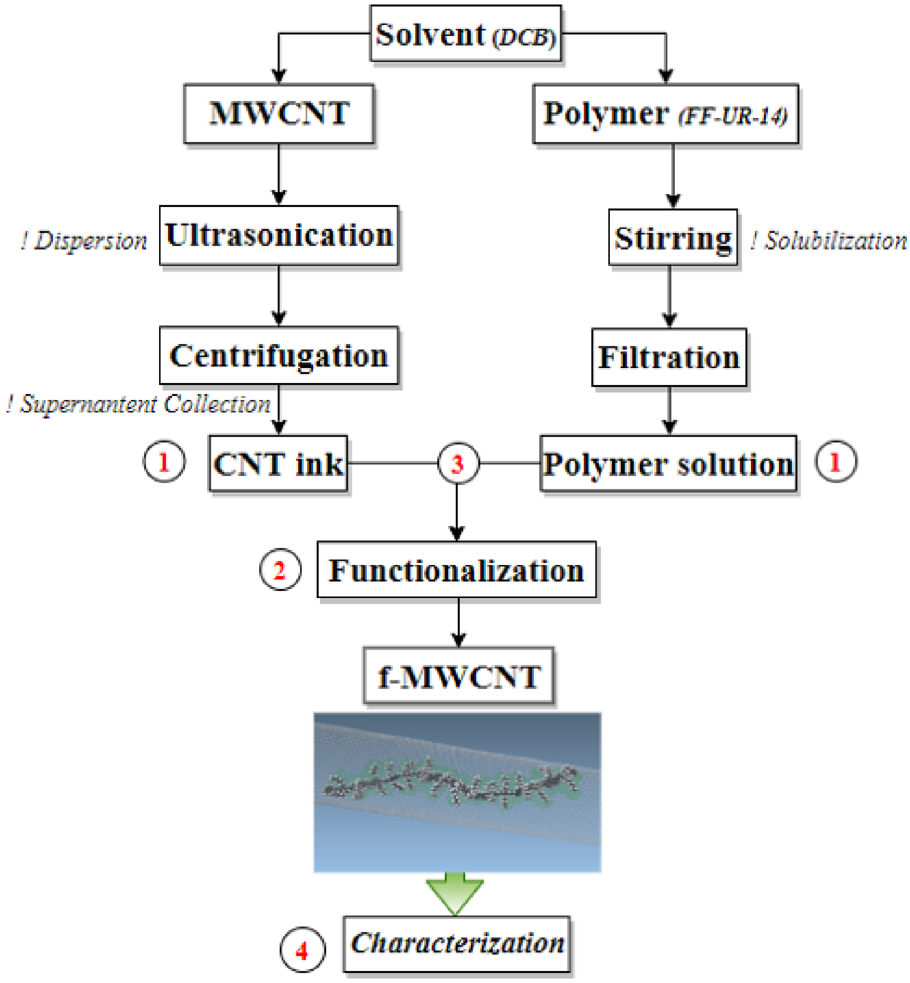

Figure 11: Ink formulation process

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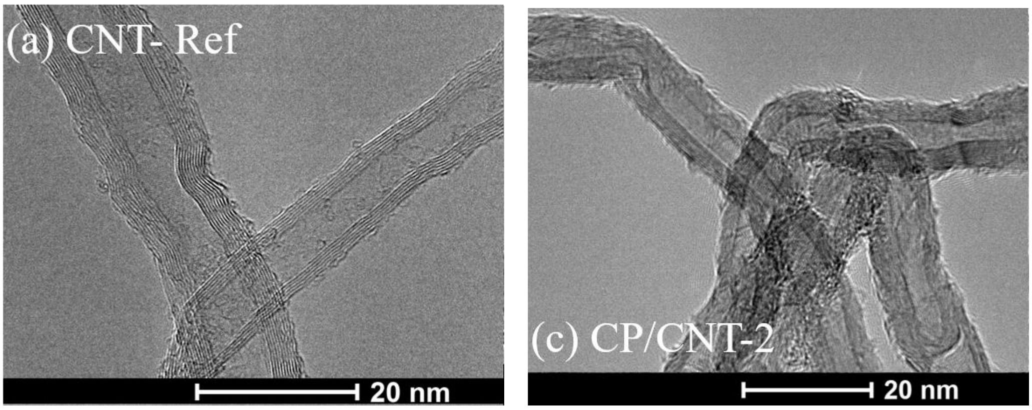

Figure 12: TEM (Transmission Electron Microscope) images of (left) non functionalized MWCNT and (right) FF-UR-functionalized MWCNT (2:1 FF-UR:CNT ratio)

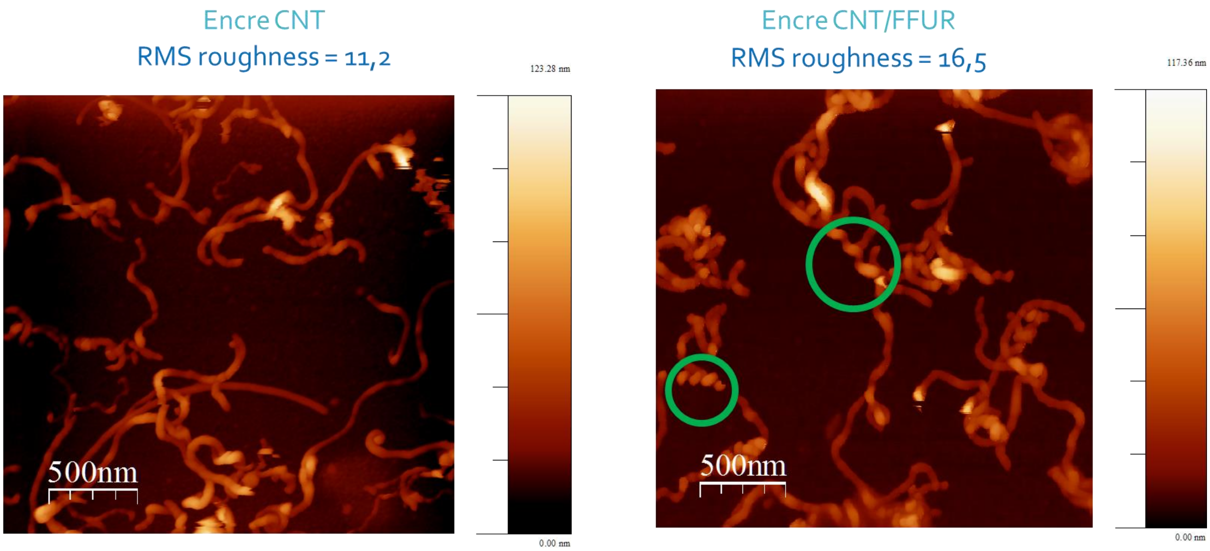

Figure 13: AFM (Atomic Force Microscope) image of (left) non-functionalized and (right) FF-UR functionalized MWCNT. Analysis of AFM data shows that FF-UR/MWCNT are wider in diameter than non-functionalized MWCNT by 5.3 nm, which suggests that the FF-UR layer on the MWCNT sidewalls is 2.6nm thick. The green circle points out obvious coils along some of FF-UR/MWCNT, which are not observed for pristine MWCNT and might be caused by the presence of the FF-UR polymer.

Printing

The printing of the two inks is carried out with the Dimatix DMP 2850 inkjet printer. The equipment is shown in Figure 14.

Four tasks were carried out:

- Optimization of printing parameters separately for each ink
- Development of a process to print both inks on the same chip. The outcome is shown in Figure 15

- Development of a process to print one ink on a batch of 9 or 10 chips. The chips are all annealed together after printing.
- Development of a process to print both inks on a batch of 9 or 10 chips: each ink is printed on the whole batch of chips before moving to the next ink. For two inks, ten chips, the process takes about 12h plus annealing overnight, so can be done over (preferred) 1 long working day or two shorter days.

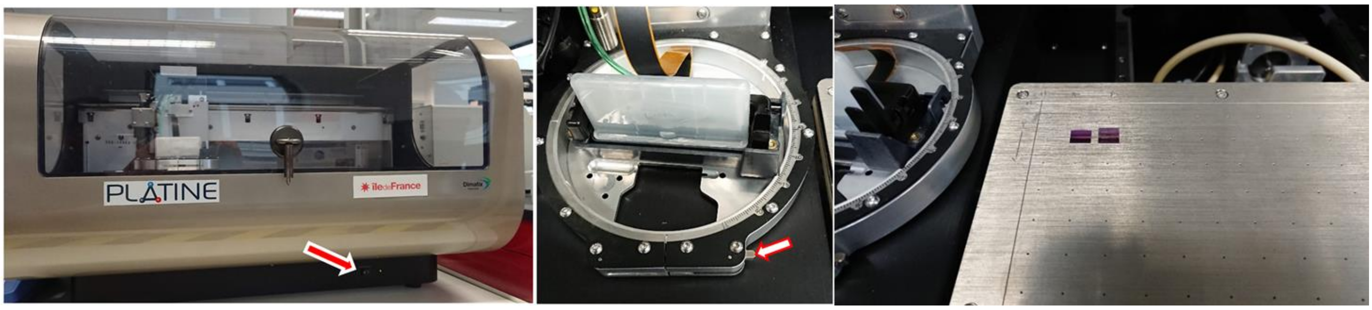

Figure 14: Dimatix DMP 2850 inkjet printer available in Platine facility at Ecole Polytechnique. Left: front view of the printer. Middle: printer head with mounted cartridge. RIght: printing plate with 2 chips

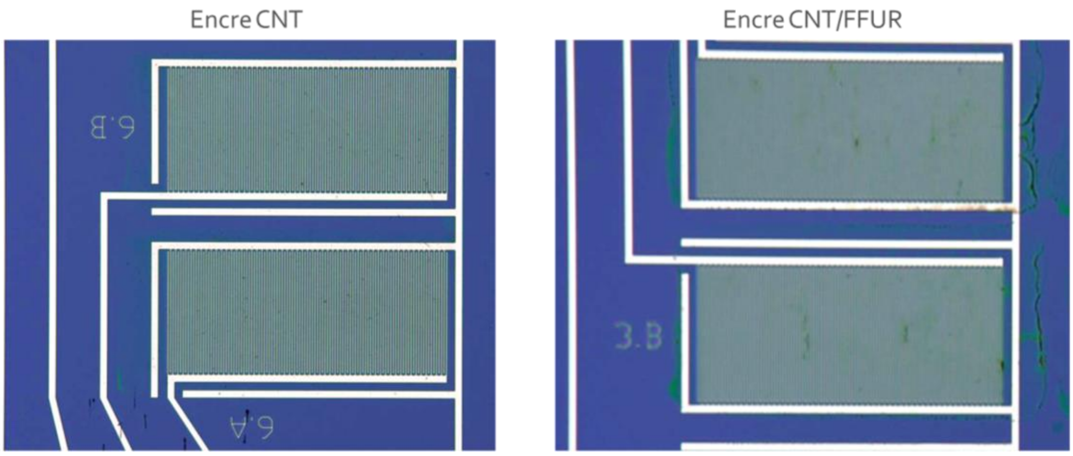

Figure 15: Optical images of a chip with two inks printed on IDE electrodes. Left: CNT ink. Rigt: CNT-FF-UR ink.

Porous membrane deposition

The first characterizations in water of the CNT sensors were very unstable electrically (continuous, rapid increase of resistance in water with time) (Figure 16-left). Comparison of characterization data (Raman spectroscopy) before and after immersion in water showed obvious loss of CNTs in water during trials. (Figure 16-right).

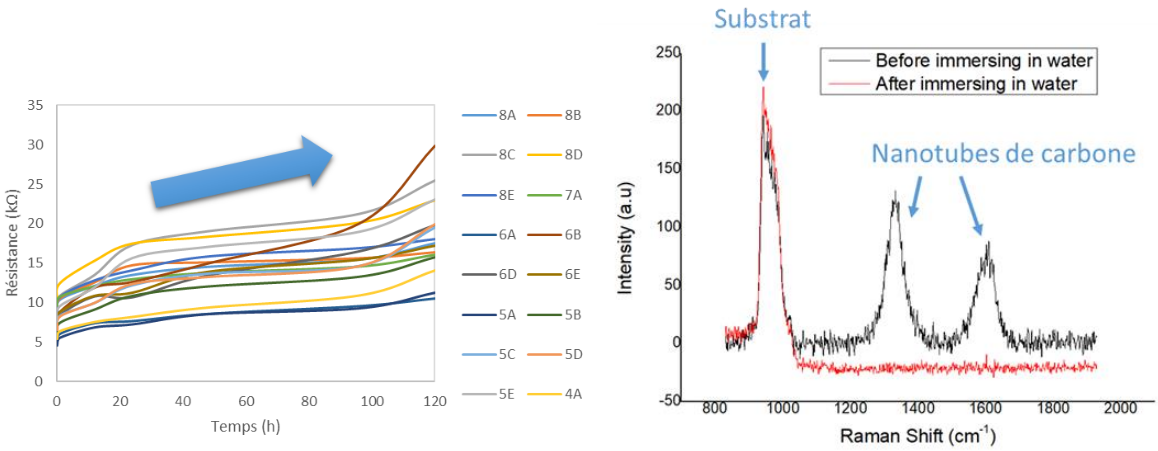

Figure 16: Left-increase of resistances of 16 devices on the same chip during immersion in deionized water. Right – Raman spectroscopy of a chip before and after immersion in water for 5 days. The Raman shift curve before immersion in water shows clear sign of the presence of CNTs (two peaks plus substrate peak – black line) while the measurement taken after immersion in water at the same location does not show any of the CNT-specific features (no peak except substrate peak - red line).

To solve this issue, two additional process steps were carried out:

- Before printing, enhanced cleaning of the chips.
- After printing, fabrication of a porous PMMA layer directly on top of the CNT to anchor them in place.

Figure 17 shows the outcomes of the porous PMMA layer fabrication process through AFM images of CNT layer before (left) and after (middle) porous PMMA layer deposition. The yellow rectangles single out two obvious pores in the layer. Note that there may be smaller pores in the layer, though difficult to observe by this method.

Figure 18 shows that the presence of the PMMA layer successfully stabilizes the device resistance in water. Though a drift in resistance may still be observed over a time scale of 1 month, such a regular and limited drift (around 30% increase over three months in average) is easily manageable through software means.

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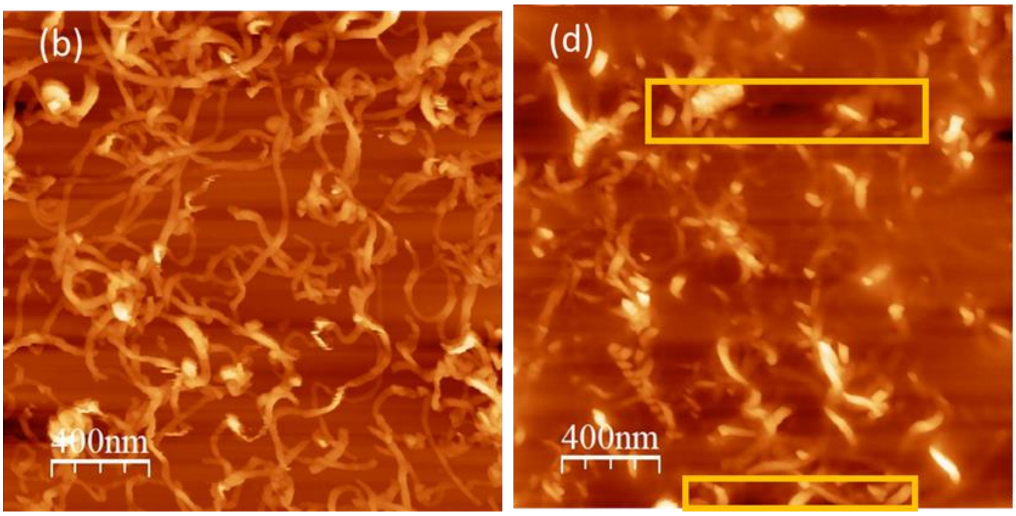

Figure 17: Outcomes of the porous PMMA layer deposition process. AFM images of CNT layer before (left) and after (middle) porous PMMA layer deposition. The yellow rectangles single out two obvious pores in the layer. Right: stable electrical response of the sensors with PMMA covering. Note that immersion for 20 hours is needed before the resistance reaches stability.

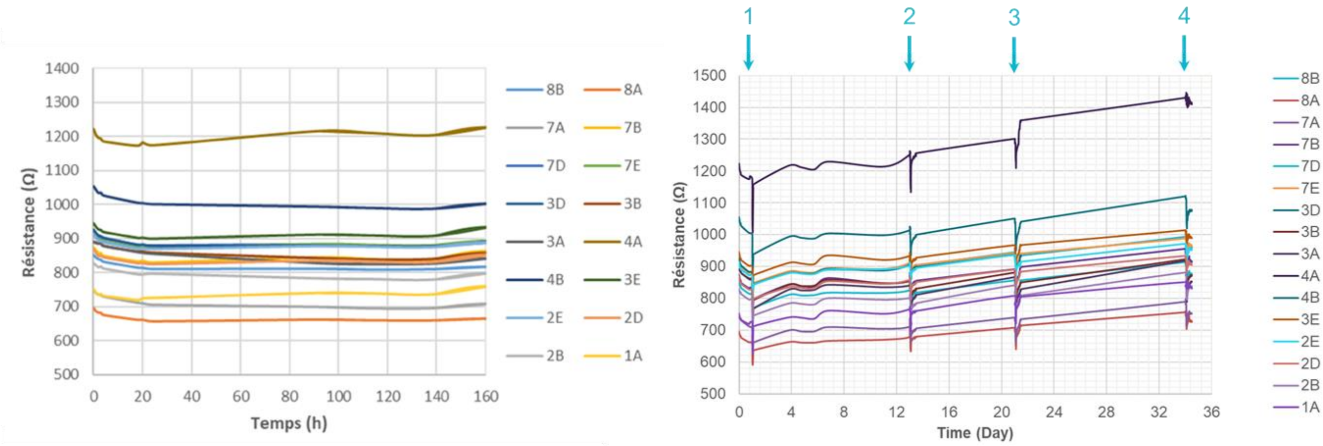

Figure 18: Ageing of devices following the deposition of the PMMA. Left – Ageing over the first 160h of exposure to distilled water. Note that immersion for 20 hours is needed before the resistance reaches stability. Right: drift over 1 month in distilled water. The arrow shows instant of addition of chlorine. The downward peaks indicate the variation of the resistance, even after 1 month, to the addition of chlorine.

Outcomes of the process

The criteria of success for the fabrication process are as follows:

- Devices are said to be operational if they have resistances between 0.5kΩ and 100kΩ.
- The yield per chip is ratio between the number of operational and fabricated devices per chip. The highest yield per chip is desired. The required yield depends on the use of the chip.
    - For development (D) activities (e.g. final validation and calibration, deployment in use case, commercial use), the target yield is set to 80% for now (no more than 2 defective devices per ink)
    - For research (R) activities (e.g. understanding sensitivity and ageing features), the target yield is 50% for at least one of the inks.
    - For process and integration (I) activities (e.g. optimizing fabrication and assembly process, integration, validation of the IT chain), the target yield is 20% for at least one of the inks.
- The yield per batch is the ratio of chips suitable for further use to the total number of chips within the batch. On can differentiate the global yield and the yield with respect to activities of type D, R and I.
- The coefficient of variation CV (%) is the ratio between the standard deviation and the average resistance over devices on the same chip with the same ink and the same geometry. The required variability depends on the use of the chips:
    - Development activities: the lowest possible CV is needed. The CV is preferably below 20%, though higher CV are acceptable as long as the electronics and software are compatible with it.
    - Research activities: high CV are acceptable as they allow to understand the relationship between baseline resistance and sensitivity and ageing features.
    - Process and integration activities: no constraint.

The statistics below are calculated over the four batches of chips with two inks fabricated between February and June. All batches were fabricated with freshly made inks (less than 2 weeks), except the 2nd batch with about 1 month old inks.

1. Early February: 9 chips with 2 inks
2. Early February: 10 chips with 2 aged inks (only 5 out 10 chips with PMMA cover)
3. Mid May: 10 chips with 2 inks
4. Early June: 9 chips with 2 inks

Table 2 summarizes the performances of those batches. **From the strict point of view of yield, all batches have 100% success: all chips are suitable for integration activities or research activities. If one however considers the yield for development activities, 2 batches out of 4 have perfect score, 1 has 78% yield (2 unsuitable chips out of 9), 1 as 10% yield only (1 suitable chip).** Let us note that the unsuccessful batch was fabricated right after the end of 2months closure of the lab due to COVID. Equipment instability right after restart may explain the poor performance of batch 3. **From the point**

of view of variability, the second batch had remarkable variability satisfying the 20% target across the board. For the other batches, the variability lies around 30% to 40% in average. This is an optimization goal for future work, to be reported on in deliverables D2.5.

Table 2: Batch performances. All unit are in % except for the two last lines (Resistances in kΩ).CV: coefficient of variation in %. Yield D/R/I: % of operational devices suitable for activities of type D, R or I. D: development activities. R: research activities. I: Integration activities. Avg: average. Std dev: standard deviation. The coefficient of variation CV (%) is the ratio between the standard deviation and the average resistance over devices on the same chip with the same ink and the same geometry.

Index of batch	1	2	3	4
Yield D	100	100	10	78
Yield D - CV<40%	67	100	0	44
Yield D - CV<20%	11	100	0	11
Yield R 1ink	100	100	100	100
Yield R 2inks	100	100	10	100
Yield I	100	100	100	100
CV CNT avg	39	8	97	41
CV CNT std dev	23	2	0	20
CV CNT min	16	6	97	19
CV CNT max	81	12	97	79
CV CNT/FF-UR avg	32	10	30	37
CV CNT/FF-UR std dev	20	5	16	22
CV CNT/FF-UR min	8	5	5	17
CV CNT/FF-UR max	67	18	55	83
Resistance CNT avg (kΩ)	8,3	1,5	3,3	6,2
Resistance CNT/FF-UR avg (kΩ)	4,5	1,8	2,6	26,0

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3.1.3 LOTUS Sensor head

3.1.3.1 Principles

Two subversions of sensor head are reported here:

- for LNODE V1.0: T-shaped sensor head, expected to be suitable for low flow use cases (groundwater, tanker, waste water use cases).
- for LNODE V1.1: Cylinder-shape sensor head, expected to have more flexible usages (higher flow rate), but at higher unit cost and longer lead time than v1.0

The sensor head is composed of two parts, the sensor head housing and the sensor head PCB. The support PCB is designed so that 1) the chip can be wirebonded on it; 2) it can be mounted onto the sensor head housing and 3) it can be connected to an electronic system able to read the sensor chip (see section 0)

In later versions, to be reported in D2.5 and following, the sensor head will be topped with a membrane filtration system to protect the sensor chip from dust impacts. Preliminary designs are available but require extensive experimental testing.

Likewise, in later versions, the geometry of the sensor head will be adapted to better comply with LOTUS use cases. Designs are available but still need adaptation and testing before release.

3.1.3.2 Chip to sensor head PCB assembly

Sensor head PCB assembly process includes glueing and wirebonding of sensor chip to support PCB, then protection and waterproofing of the wirebondings via globtop. These are standard steps in the microelectronic industry. Hence, while the process can be done in the lab, it has been carried out by service providers for most of the sensor head PCBs assembled so far. Figure 19 shows an example of resulting assembly.

The service providers simply need the following information to proceed:

- Position and orientation of the chip on the PCB
- Correspondence matrix between contact pads on the chip and on the PCB
- Chip-to-PCB Glue
- Wirebonding material and wirebonding method
- Globtop material

New contractors need to adjust their usual process on a case by case basis. In that case, they are provided with test chip and PCB.

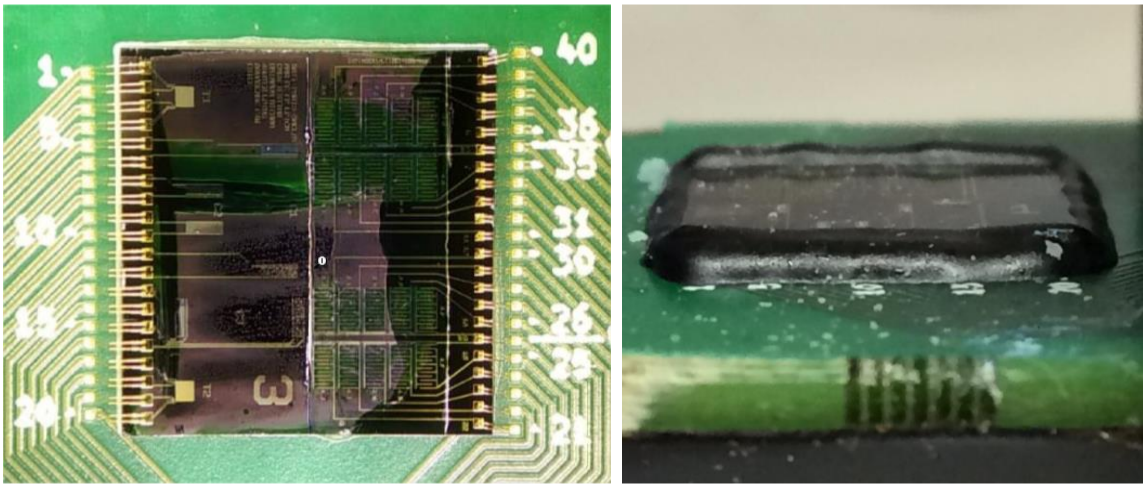

Figure 19:Left - wirebonded chip before globtop. Right: sideview of the globtop layer.

3.1.3.3 Sensor head V1.0 design and assembly

Sensor head V1.0 and Head PCB V1.0 are depicted in Figure 20 while Figure 21 shows how they fit together. The sensor head is manufactured in grey PVC. Figure 22 shows that design details of the sensor head. The geometry of the sensor head PCB is shown in Figure 23 (right). The PCB is manufactured in FR4 with conductive copper lines (Figure 23 left). The sensor head PCB is mounted with a FPC connector which enables to plug and play the flat cable used for connection to the main electronic board (Figure 24). The sensor PCB is glued to the sensor head, then resist material is put in to waterproof the insertion slot.

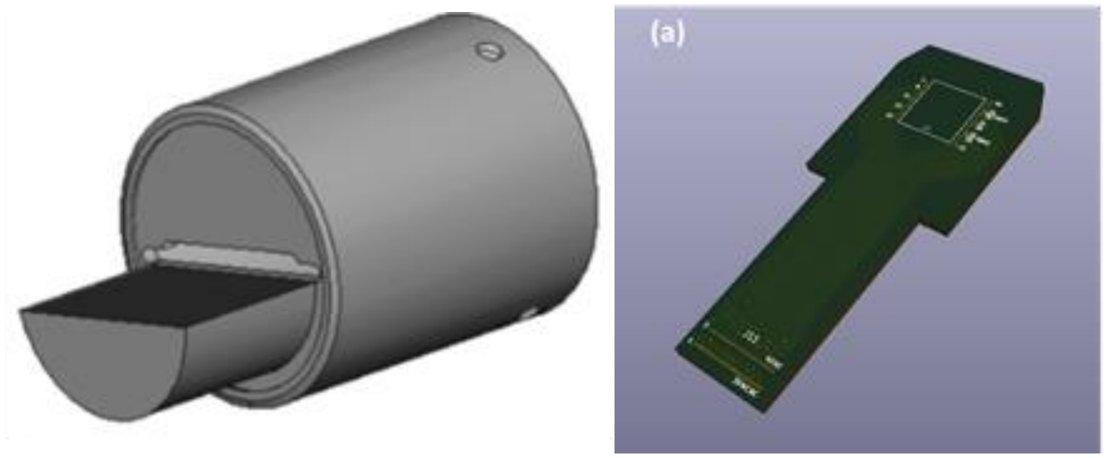

Figure 20: Sensor head V1.0: left - Sensor head housing. Right - Head PCB

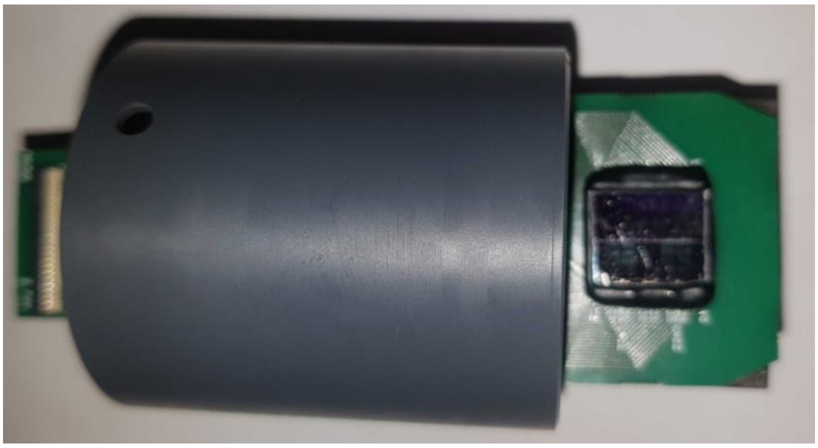

Figure 21: Assembled sensor head (with chip wirebonded on PCB and PCB glued to sensor head)

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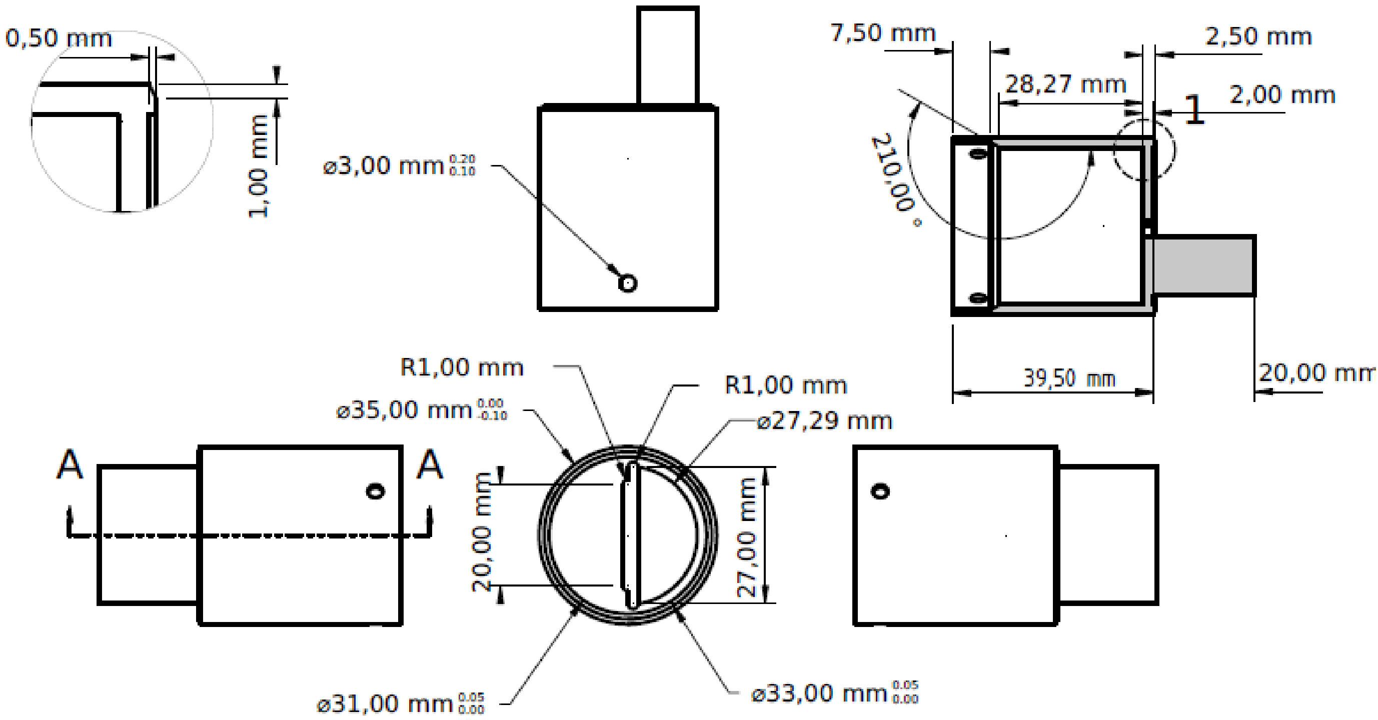

Figure 22: Manufacturing plans for sensor head housing V1.0.

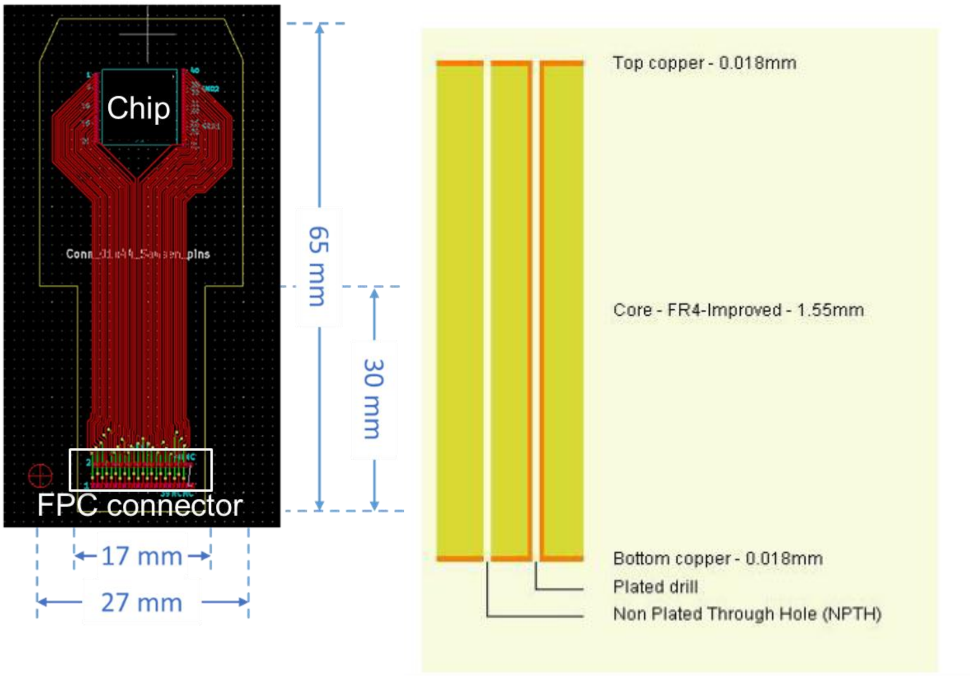

Figure 23: Geometry (left) and buildup of the sensor head PCB version 1.0

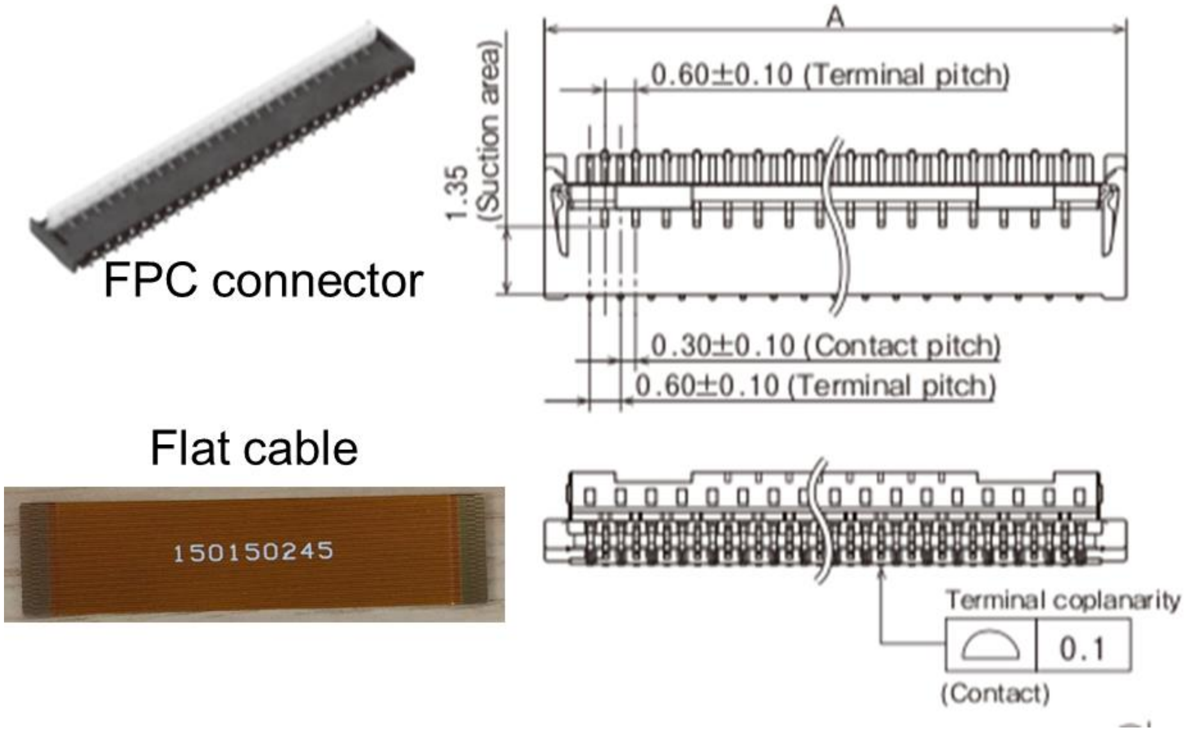

Figure 24: FPC connector for the sensor head PCB version 1.0 and associated flat cable

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3.1.3.4 Sensor head V1.1 design and assembly

Sensor head V1.1 and Head PCB V1.1 are depicted in Figure 25 while Figure 26 shows how they fit together. The sensor head v1.1 is manufactured in grey PVC as well. Figure 27 shows its design details. By design, the head PCB v1.1 can be connected directly to an electronic board carrying a FPC connector through its flexible part.

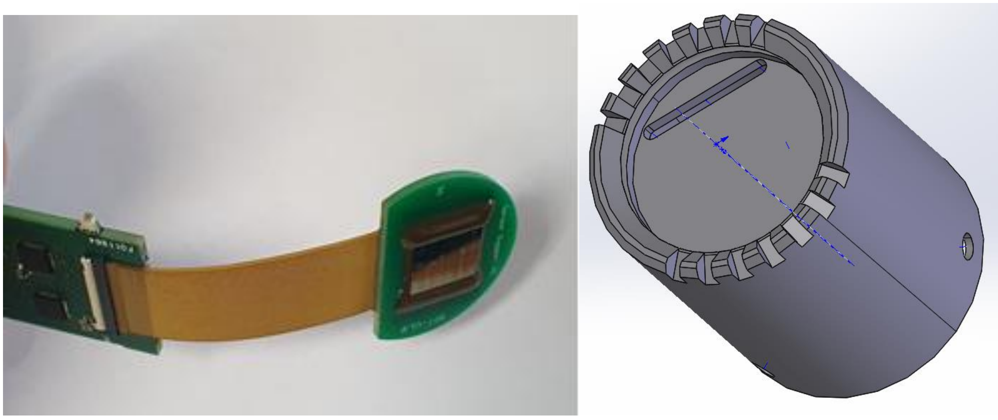


Figure 25: Left - semi-flex head PCB v1.1. Right - sensor head version 1.1

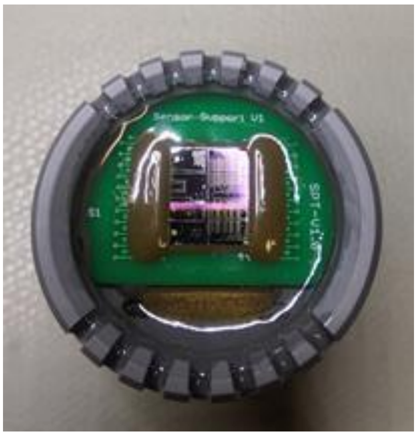


Figure 26: Assembled sensor head version 1.1

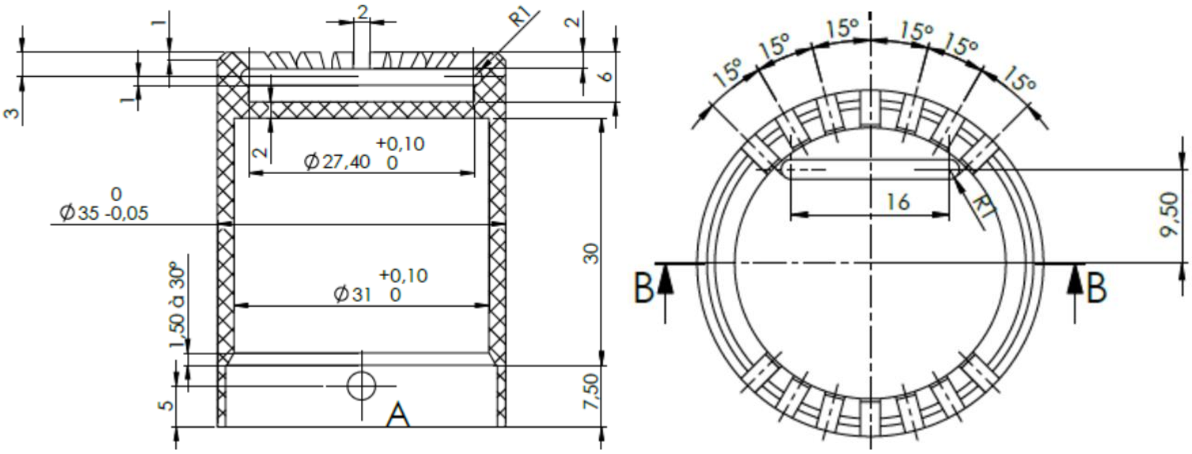
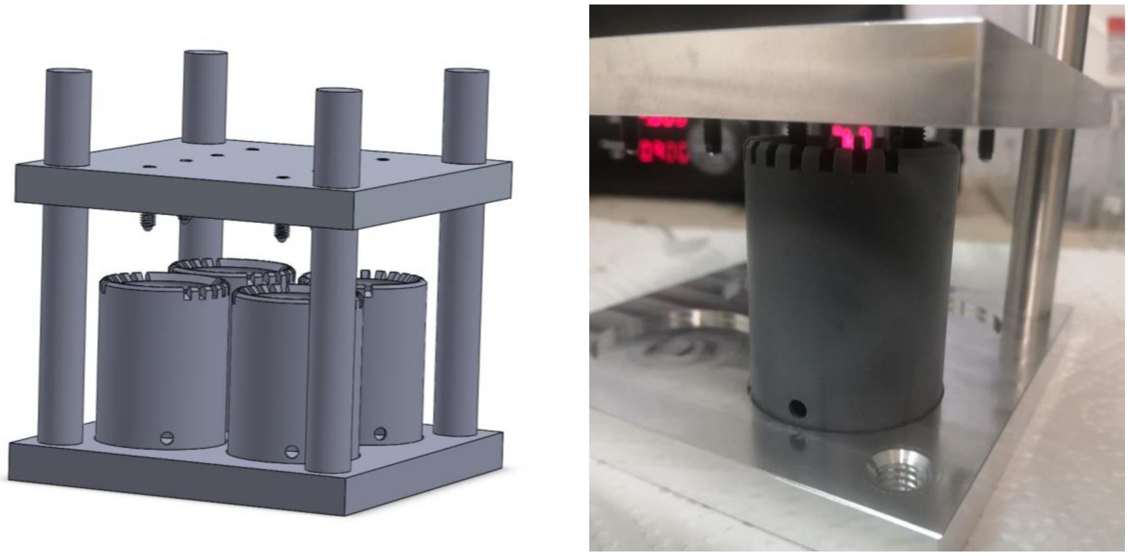


Figure 27: Sensor head design v1.1. Units are mm.

For the mounting, a special holder has been designed (Figure 28) to maintain up to 4 assemblies during the gluing process. Small springs are used to apply controlled pressure over the head PCB during the process described in this section.



(a) (b)

Figure 28: LOTUS head holder on gluing bench (a) CAD view; (b) holder equipped with one LOTUS head

Coating strategy

In order to glue the sensor PCB in the sensor housing, a bi-component epoxy adhesive was selected. To provide electrical insulation while at the same time maintaining sensor exposure to the water flow,

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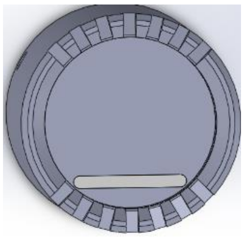
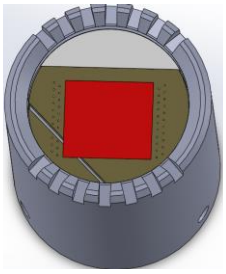
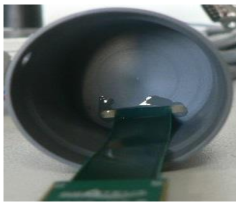
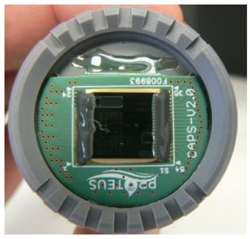
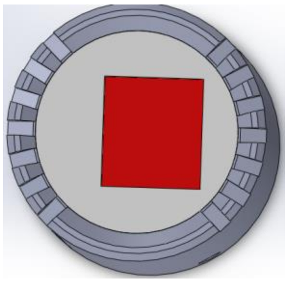
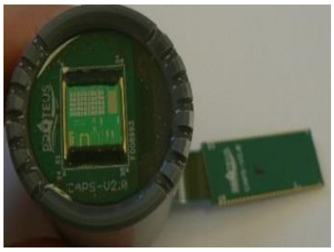
a dam and fill process is used. For this process, another bi-component epoxy was selected based on electrical, chemical, physical properties.

Table 3: Epoxy resin quantity needs

Step	Description	Design	Result
1-GLUING of semi-circular PCB in the housing	Bi-component epoxy resin deposit on the PVC part		
2- Deposit of a bead of glue all around the slot	Bi-component epoxy resin (thixotropic) deposit in the slot -------------- deposit in the empty part around the slot	 ----------------------- 	 ------------------------ 
3- Covering the sensor chip	Bi-component epoxy resin (low viscosity) Drop deposit to cover around the sensor chip		

Targeted process

The assembly process of the sensor cap is as follows:

1/ Cleaning of sensor housing:

Dip sensor housing inside a beaker of IPA and put the beaker inside an ultrasonic cleaner; all the mechanical parts have been cleaned and dried in oven.

Figure 29: cleaned sensor housing

2/ Gluing of the sensor PCB into the sensor housing:

The sensor PCB is glued into the sensor housing with a bead of a thixotropic bi-component epoxy resin on the inner edge of the part and on the middle in form of two vertical axis. After thermal curing in oven, the assembly is ready for the coating steps.

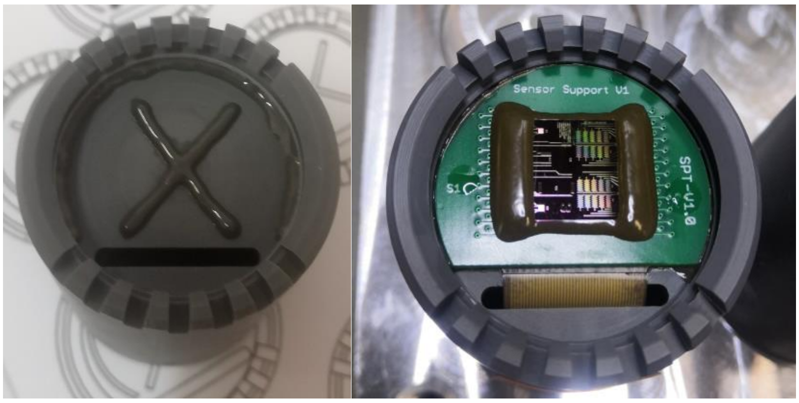


Figure 30: Glueing and damp process

3/ Coating around the slot, the flex link and the sensor chip

This step is to close the remaining free space around the flex link with epoxy.

A low viscosity bi-component epoxy resin is used to cover the PCB around the sensor chip.

After thermal curing in oven we can move to the last step.

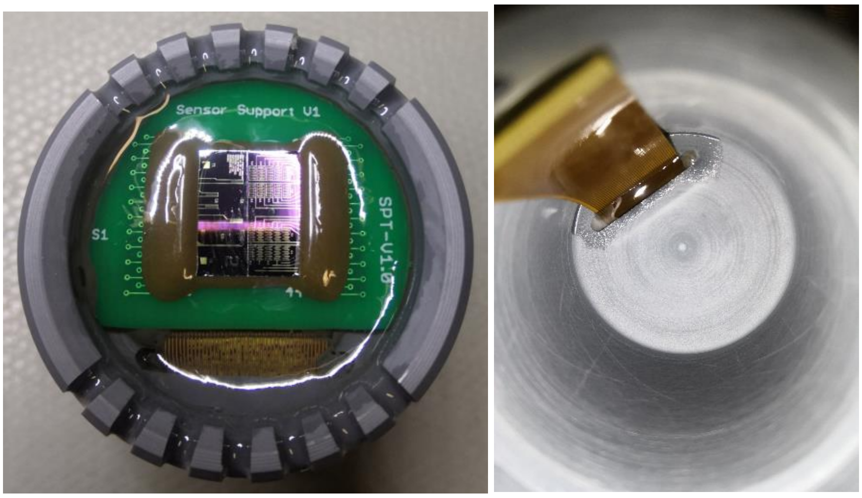


Figure 31: filling process

4/ Coating the inside face of the sensor cap

Also, a low viscosity bi-component epoxy resin is used to fill the inside of the sensor cap.

In this case, a thermal curing is also realized in oven.

Figure 32: additional internal waterproofing protection.

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3.1.4 LOTUS Analogic Front End

The Analogic Front End is the electronic board which interfaces the sensor chip to any end-user system (LOTUS box, PC…). It main function is to connect all the sensors in the chip (Analog Multiplexer – Mux), bias each different sensor in the appropriate manner, then detect and digitize its analogic output (Mixed-mode Processor; e.g. analog & digital), finally communicate it to an end-user system following the appropriate protocol and connector (here USB or UART). This is summarized in Figure 33. Figure 34 shows the AFE front and back sides (size 9.1x2.2cm) with the positioning of its component on the board.

Importantly, most of the processing is carried out within a programmable system on chip (PSOC) instead of the more traditional approach to use discrete components. Besides yielding a drastically more compact system, it also allows to modify by software the data acquisition parameters (such as gain, amplification, biasing…) which significantly reduces the time needed optimization of signal processing parameters.

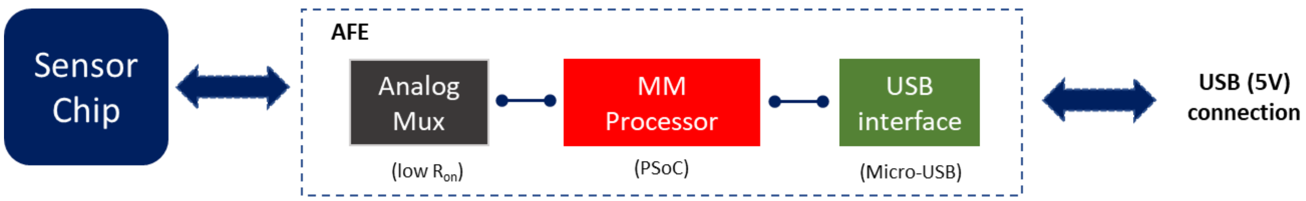

Figure 33: Main building blocks of the AFE

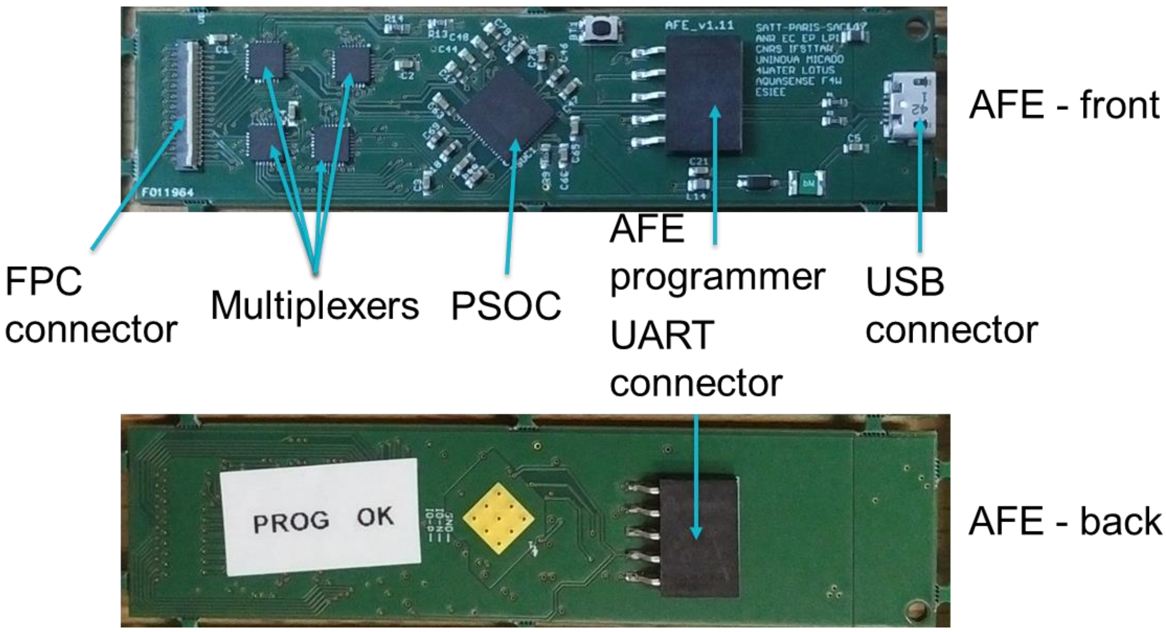

Figure 34: Front and backside of the AFE with position of the components. The length is 91.3mm and the width is 22.1mm.

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3.1.5 LOTUS Sensor body

The Sensor body is designed to encapsulate in a waterproof manner the AFE and to allow its connexion to the sensor head via flat cable on one side and to the desired end-user system by USB or UART cable. Figure 35 depicts the sensor body while Figure 36 details its size: 18cm long cylinder of diameter 3.5cm. With respect to Figure 35 orientation, the sensor head is screwed on the left side of the sensor body, while the AFE lies within. The USB cable goes out from the AFE through a cable gland mounted on the right side of the sensor body. Both versions of the sensor heads are compatible with this design.

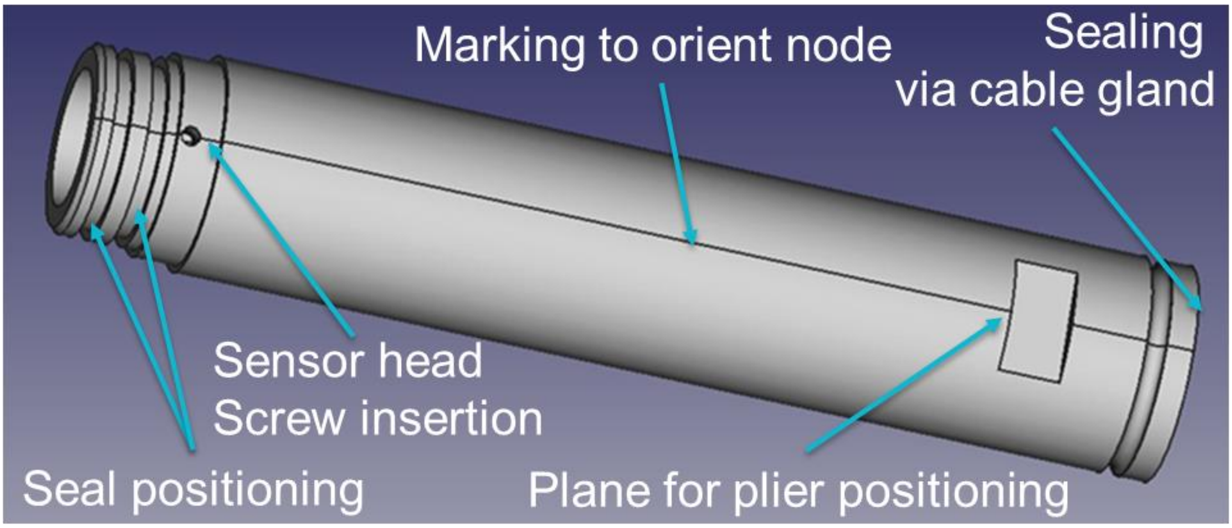

Figure 35: Sensor body

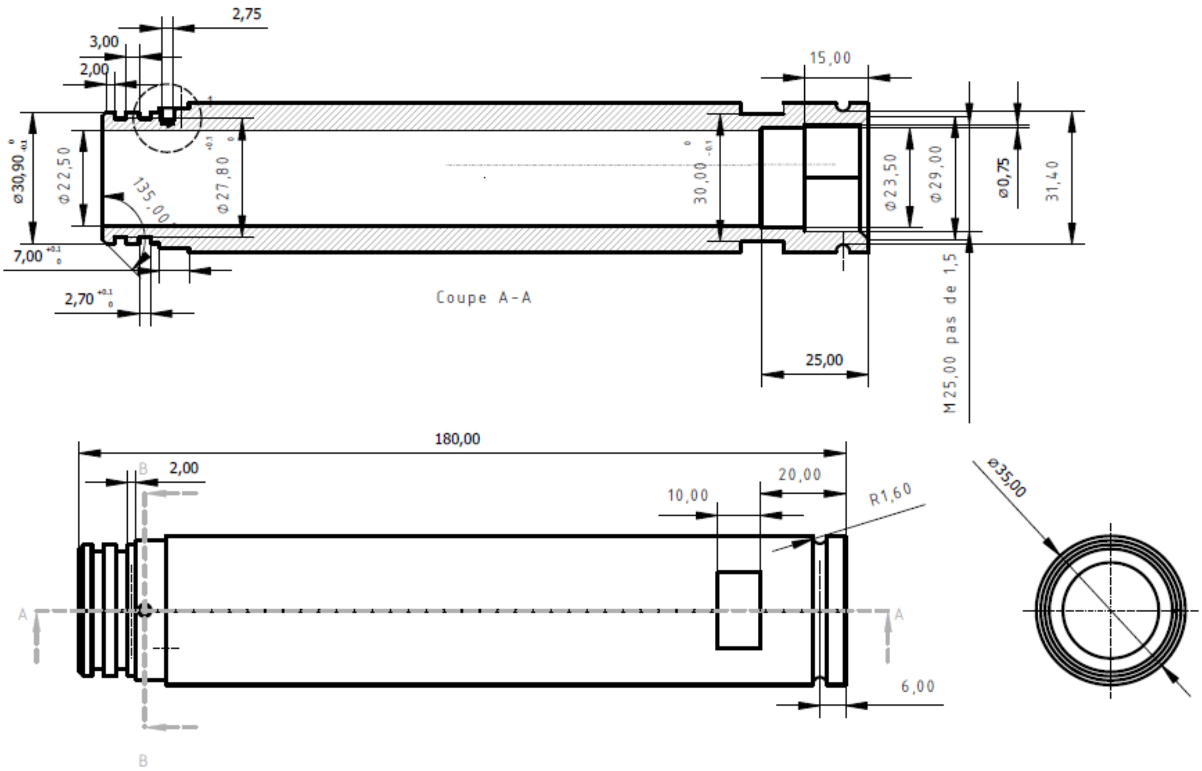

Figure 36: Design of the sensor body

3.1.6 LOTUS Node assembly

The spare parts needed to assemble a sensor node are as follows:

1. Sensor body
2. Assembled sensor head
3. AFE
4. Flat cable
5. USB cable
6. Cable gland
7. Screws
8. O-rings
9. Thin wire

Figure 37 shows a decomposed view of the end-to-end system as well as an assembled view. To manage this assembly, the following steps are carried out:

- Prepare AFE: mount USB cable to AFE, pass USB cable through cable gland, anchor the thin wire to the hole in the AFE
- Prepare the sensor body: mount 2 O-rings, insert AFE (with USB cable) within sensor body, connect flat cable to AFE
- Mount the sensor head: plug the other side of the flat cable to the sensor head PCB, insert sensor head and screw it in.
- Close the cable gland and use the thin wire to keep the AFE in place inside the sensor body.

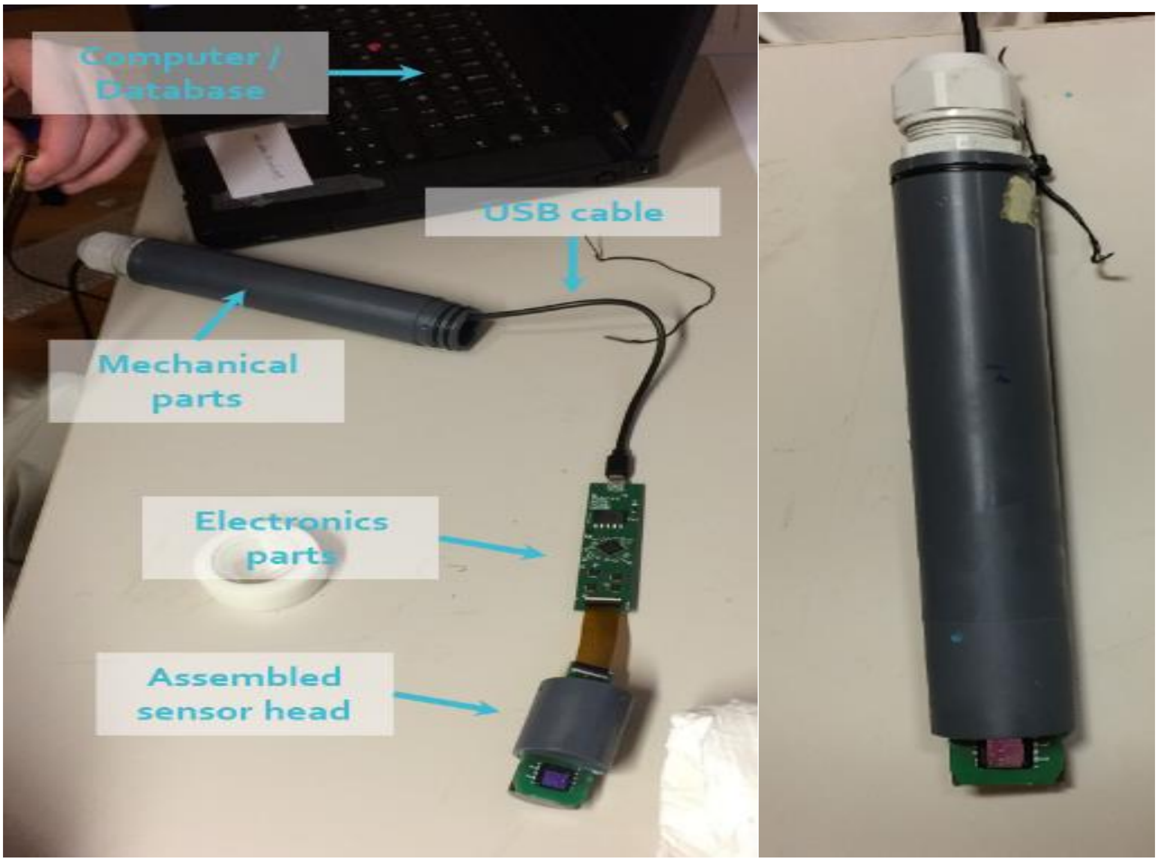

Figure 37: Assembled sensor node v1.0. Left - split view. Right - assembled view

3.2LOTUS Box

3.2.1 Functional requirements

The functional requirements for the LOTUS Box have been extracted from D6.1 and summarised in the next table

Function	Expression	Criterion	Possible tech.	Flexibility level
FP0	Computation	Must have an internal computation	Microcontroller	Mandatory
FP1	Modularity	The overall device functionalities must be changed or extended according to customer need	Pluggable internal module	Mandatory
FP2	Sensor input	One or more must be attached to the device. Could be done by FP1	Eg. UART, RS232, RS485, 4-20mA, BLE5, SPI, I2C…	Mandatory
FP3	Actuator output	Must operate a device like a valve	Relay, silicon switch…	Mandatory
FP4	Communication output	Device must transmit data from the sensors to the cloud. Could be done by FP1	Wireless (eg. Lora, LTE-M, BLE5, Iridium, NB-IoT, LTE-M, 3/4G…) or wire (modbus slave, USB, RS232…)	Mandatory
FP5	Data logging	Must record all datas from connected sensors	On-board memory, Removable memory	Mandatory
FP6	Real time clock	Must add a timestamp to recorded data		Mandatory
FP7	Internal t°/hum	Must provide internal health status for internal diagnostic	On board sensor	Mandatory
FP8	Autonomy	May have several hours/days of autonomy	Battery. Can be external ?	Optionnal. Mandatory in case of for portable version and also because of low power reliability in India

Function	Expression	Criterion	Possible tech.	Flexibility level
FP9	Internal charging unit	May charge an internal battery, in conjunction with FP8	LiFePo4 Lion…	Optionnal
FP10	External power	Must be externally powered	USB powered (5V) Car battery (12V), Industrial power supply (24V) Or any other DC power adapter	Mandatory
FP11	Energy harvesting	May source energy from environment	Solar panel	Optional
FP12	Core power	Must provide power supply to all internal component		Mandatory
FP13	Power supply output	Must supply power to the sensor (see FP2)	5V 12V 24V (optional)	Mandatory
FP14	Power control	Must control each module (see FP1) power supply to shut them down	Switch or transistor	Mandatory
FP15	Security	Must encrypt data or similar	AES encryption	Mandatory
FP16	Updatable	Must be externaly updatable (OTA updates)	Wire or wireless. Eg. USB port, Wifi, BT	Mandatory
FP17	HMI / configuration	Human machine interface. Must be externally or internally configurable	Eg. On board screen and button, on board touch screen or wire/wireless external interface	Mandatory

These functions are represented in the functional diagram (Figure 38) in which communication interfaces are proposed as pluggable modules to allow addressing the diverse needs of the use cases and their possible evolutions. Network access technologies are indeed unclear, and situation may vary depending on the user location.

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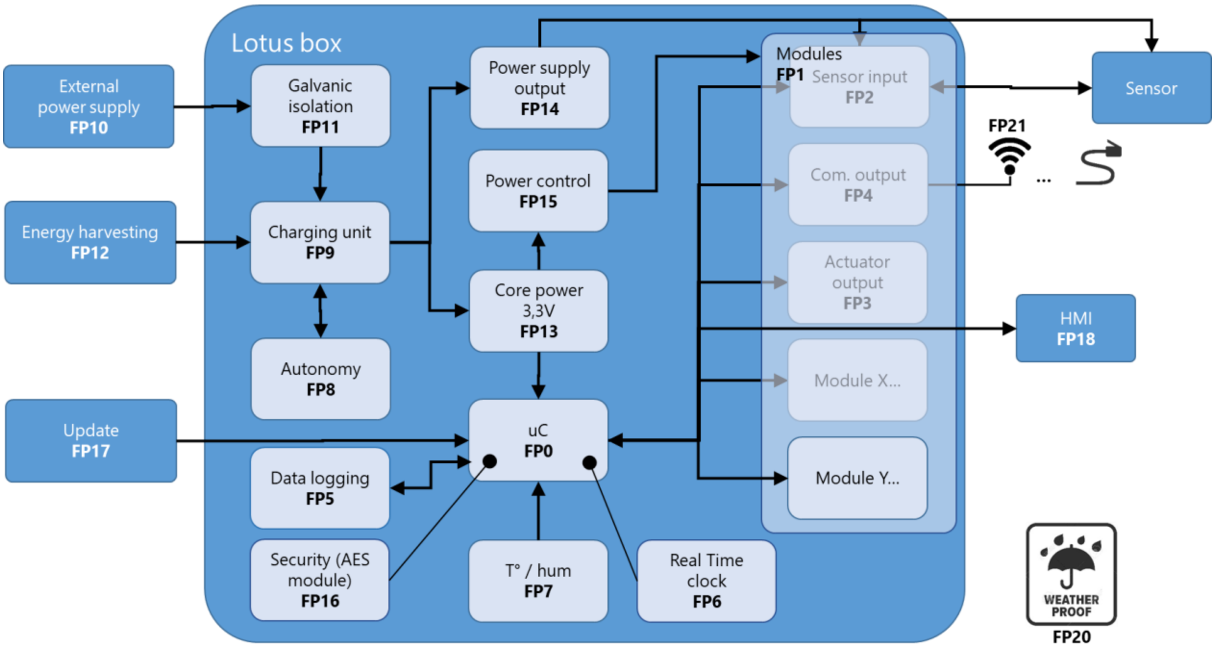

Figure 38: LOTUS Box functional diagram

3.2.2 Main design elements

3.2.2.1 µController / FP0

Microcontroller offer is wide either by their architectures (8, 16, 32 bits), their number of pins, their size, their advertised consumption, the available development tools, the development community, etc.

The most important thing in these "internet of things" times is to have a low power consumption device. All manufacturers claim to have the "lowest power mcu", so it became a commercial argument. It is therefore very difficult to have a truly reliable comparison on this.

There is decade of microcontroller manufacturer, however we will limit our choice to the two widely recognized in industry: ST and Microchip / ATMEL.

Objectively comparing all the characteristics of microcontrollers from different manufacturers is quite difficult to perform, especially since it is necessary to go into detail on the datasheets and it is quite tedious. One will have such an advantage that the other will not have and so on.

We made a first comparative table in order to "target" the series of two major manufacturers that are ST and ATMEL, because they use the same core architecture.

The list of candidate µcontrollers was then filtered with the following criteria:

- Non-DIE package (die = silicon without case)

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- with D / A Converters (12 bit)
- with Crypto-HASH
- without SMPS (it's a ST specific DC/DC power supply to reduce consumption)

Looking additionally at cost and available librairies, we selected the STM32L462VE.

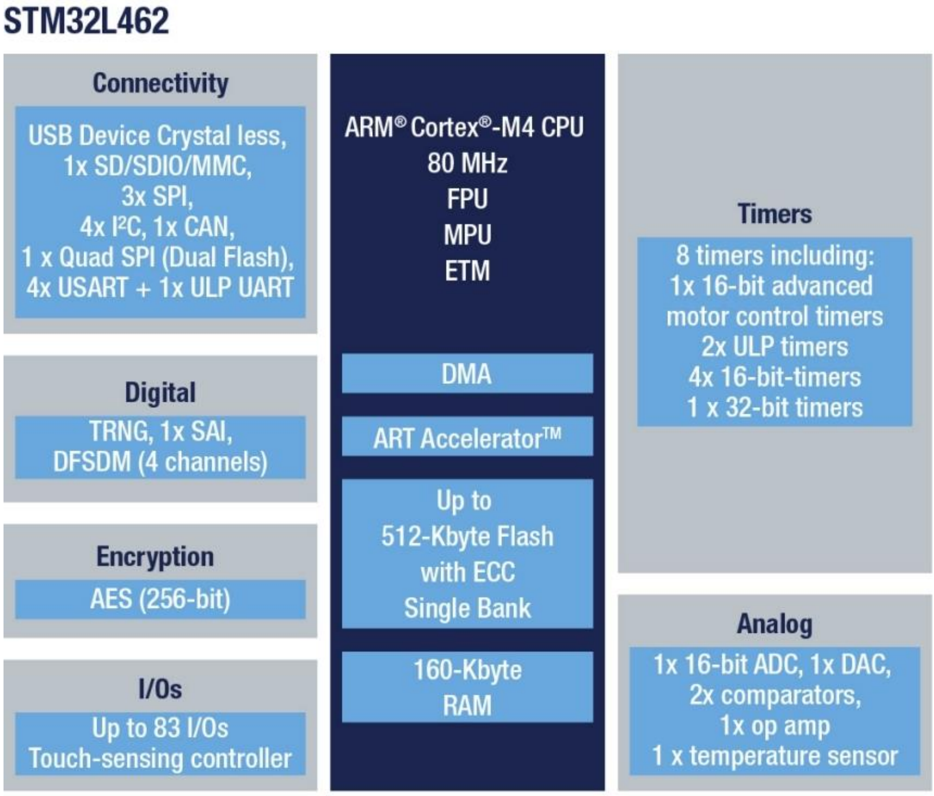

Figure 39: STM32L462 main characteristics

3.2.2.2 Modularity / FP1

One particular format already exists and is object of an open standard: mikroBUS™ from MikroElectronika [1]. It defines mainboard sockets and add-on boards used for interfacing microcontrollers or microprocessors (mainboards) with integrated circuits and modules (add-on boards). The standard specifies the physical layout of the mikroBUS™pinout, the communication and power supply pins used, the size and shape of the add-on boards, the positioning of the mikroBUS™ socket on the mainboard, and finally, the silkscreen marking conventions for both the add-on boards and sockets.

The purpose of mikroBUS™ is to enable easy hardware expandability with a large number of standardized compact add-on boards (more than 700), each one carrying a single sensor, transceiver, display, encoder, motor driver, connection port, or any other electronic module o

[1] https://www.mikroe.com/mikrobus

integrated circuit. More over, mikroBUS™ is an open standard adopted by many uC manufacturer — anyone can implement mikroBUS™ in their hardware design, as long as the requirements set the standard are being met.

This has been retained as a base standard for the LOTUS Box.

3.2.2.3 Sensor input / FP2

Theses can use numerous ways of transmitting data, by wire or wireless, eg. analog, RS232, RS485, SPI, BLE, …

From D6.1:

- “The disinfectant system will be operated based on the results from the LOTUS sensor by a PLC controller placed in the tanker”: §3.5.2 page 24
- “The level of the water available in the tanker will be monitored using a level sensor. The output from the level sensor should be concatenated with LOTUS sensor output.”: §3.6 page 25
- “There should be the possibility to add signals from additional sensors to the LOTUS box for online computations. E.g. a flow sensor could be added if this functionality is not included the LOTUS sensor.”: §4.6 page 30

Thanks to “Modularity / FP1”, this can be changed as needed.

3.2.2.4 Actuator output FP3

From D6.1:

- “automatic valves can be operated from the LOTUS box”: §4.7 page 30

Thanks to “Modularity / FP1”, this can be changed as needed.

3.2.2.5 Communication output FP4

The main goal of such IoT devices is to send data they collect to the cloud to be processed further. It can be done thought the same medium as used in “Sensor input / FP2” (BLE, RS485, RS232, USB…), or thought LPWAN (Low Power and Wide Area Network): LTE-M, NB-IoT, Lora, Sigfox…

It can use wired connexion like ethernet.

Other wireless technologies like 2G/3G/4G can be used too, but they consume more energy.

Iridium is another option, this technology use satellites for communication, but consume a lot of energy.

Furthermore, from the needs expressed in D6.1, these functions are mandatory:

- “The data from LOTUS sensor should able to transfer directly to SCADA using LOTUS box and, in another aspect, multiple LOTUS sensor can be connected to intermittent data transfer unit (similar to RTUs), then data will be transferred to SCADA” : §2.4 page 11
- “should be able to communicate these recorded values every hour to SCADA/cloud” §2.5.4 page 14
- “The data from the LOTUS sensor will be transferred from LOTUS box to cloud and from cloud to SCADA.”: §2.7 page 15
- “Connectivity to cloud or SCADA (at administration office) from LOTUS box”, and “Modification of the currently available SCADA for receiving and storing the data from LOTUS box”: §2.8 page 15
- “In case of a connection failure, data should be extractable using USB from LOTUS box”: §2.8 page 15
- GSM / Wifi /LoRa and Modbus: figure 6 page 21
- “The wireless connection between the LOTUS box, cloud system, and data storage system will be based on the SIM (3G/4G) network.”: §3.5.2 page 24
- “The output from the LOTUS box has to be connected to the cloud with appropriate connecting protocol (e.g., LOTUS application (Figure 11))”: §5.5 page 41

Thanks to “Modularity / FP1”, this can be changed as needed.

3.2.2.6 Data logging / FP5

All collected data must be recorded locally as backing-up purpose, or to exploit data in case of Communication output FP4 isn’t available.

From D6.1:

- “In case of a connection failure, data should be extractable using USB from LOTUS box.”: §2.8 page 15
- “The mobile memory must store a minimum of 1000 water analysis reading”: §5.4.3 page 40
- “Both level and quality will be measured onsite and data will be stored in the LOTUS box”: §5.5 page 41

We can use cheap and widely available removable memories like SD card. Drawback of these cards are their reliability.

An onboard memory is a good alternative, but to extract data, it is mandatory to have a USB connexion (see Communication output FP4). In terms of technology, FRAM is a promising memory technology compared to FLASH, consuming les power.

3.2.2.7 Real time clock / FP6

The chosen µController / FP0 integrate a real-time clock

3.2.2.8 Monitoring Internal temperature & humidity / FP7

There are plenty of temperature / humidity sensor, a majority of them use I2C interface. Each I2C devices has got a specific address, it seems that cheap sensors use the same address.

So, we choose a sensor providing:

- Fully calibrated, linearized and temperature compensated digital output
- Wide supply voltage range, from 2.15V to 5.5V
- I2C Interface with communication speeds up to1 MHz and two user selectable addresses
- Typical accuracy of +/-1.5%RH and +/-0.2°C
- Very fast start-up and measurement time
- Tiny 8-Pin DFN package
- High reliability and long-term stability
- Industry-proven technology with a track record of more than 15 years

3.2.2.9 Power autonomy FP8

In some case, the devices may be self-powered, notably in some country where energy supply isn't reliable or accessible. From D6.1:

- "The battery backup for sensor must be for at least 8 hours and should be easily rechargeable on site": §5.4.2 page 39
- "the required power for each sample testing should not be more than 25mAh out of 5000mAh mobile battery backup": §5.4.3 page 39
- "The energy required for the water quality measurement by sensor and data communication by LOTUS box will be supplied by the battery integrated with the LOTUS box (as shown in figure 11)": §5.5 page 41

Among batteries chemistries, we choose the LiFePO4 technology, because of its advantages:

- Cycle life up to 2000 times or more,
- Wide working temperature (-20°C/~55°C), good performance in high temperature,
- Have no memory effect, the battery can be charged and used no matter what state it is in, and it is not necessary to wait the power runs out and then charge for it.
- Light weight, the volume of the LiFePO4 battery is 2/3 of the lead-acid battery in the same specification capacity, the weight is 1/3 of the lead-acid battery
- Good safety performance, without any heavy metals and rare metals, non-toxic, no pollution, in line with European RoHS regulations, is a green battery.

LiFePO4 batteries are widely used in electric vehicles, power tools, solar & wind power energy storage equipment, UPS and emergency lights, warning lights and miner's lamps, small medical equipment and portable instruments and other fields.

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A LiFePO4 battery cell has nominal voltage of 3.2V and exist in several standard format: eg. 18650 or 26650. Several cells could be assembled with each other to have superior voltage or current/capacity. The voltage needs are mostly driven by the need to power

- The electronic (3.3V & 5V)
- Sensor modules (LOTUS AFE, external sensors), with voltages ranging in standard from 5 to 12V.

In addition, the price of a battery pack is proportional to its number of cells and/or its capacity whereas its long term reliability is inversely proportional to the number of cells. We may think that a single cell would be sufficient, but generating 12V from it is fairly difficult. The Webench Power Design from Texas Instrument may help in the choice of a regulator, and we saw that the minimum acceptable voltage is 2.7V. For these reasons, we select a 2 cells, 6.4V battery pack.

3.2.2.10 Powering / FP9 - FP15

From D6.1:

- “Power supply to the LOTUS box by either the grid/hydrogenator”: §2.8 page 15
- “A battery system is recommended to provide uninterrupted power supply to the LOTUS sensor. An inline hydro-generator has to be installed in the pipes in combination Li-ion/Li-po battery for the location without permanent power source”: §2.5.2 page 13
- “The estimated power demand of about 10 W for LOTUS sensor, level sensors, and disinfectant dosing system will be supplied by the Tankers 24 VDC on board power supply”: §3.5.2 page 24
- “Alternatively, an additional backup battery will be installed which can be charged either by the power supply of the truck or an attached solar PV panel.”: §3.5.2 page 24

Most common and used energy harvesting is photovoltaic panel. The internal charging unit needs to integrate a “Maximum Peak Power Tracking” to maximize power from panel. Its input must be 3.3V greater than the programmed output battery float voltage for reliable start-up. In our case, floating is 7,2V so an optimal solar panel voltage would be 10,5V minimum.

Some of the most used charging circuit are compared in the next table.

Table 4: comparison of solor charging circuits.

	Buck/boost/ buck boost	MPPT	Battery quiescent current (µA)	Prices / 100
Analog Devices LT3652	Buck only	Integrated	50 (0 by using relay)	14
Texas Instruments BQ25703	YES	Partially integrated	22 - 45	12.3
Analog Devices LTC4000-1 + LT3789	YES	Integrated	10 - 20	28

To power the different system part, the differ voltage levels are generated through step up/down voltage controllers.

3.2.3 Design and fabrication

The LOTUS Box has been designed using Altium Designer. A 6 layers PCB has been chosen to allow appropriate placement and routing of all the components. An IP67 casing has been retained and custom 2 LiFePo4 cells batteries ordered. The Figure 40 shows the initial version of the board produced in 5 samples for test and debugging, in it intended casing, with 3 daughters board providing WiFi, LoRaWAN and additional modbus connectivity.

2 connectors will be provided by default: 1 for powering (12VDC or solar) and one for sensors/actuators connectivity. Different casing may be provided to adjust to use case needs.

Figure 40: initial version of LOTUS Box electronic box in its target casing. 3 daughters boards are mounted providing LoRaWAN, WiFi and additional modbus connectivity.

4 Software

4.1 AFE firmware

The principle of the PSOC – the core component of the AFE - is akin that of a FPGA: the PSOC can be reconfigured at the hardware level dynamically to accomplish a series of complex functions – analog front end for multisensor chip – via software commands. More convenient than true FPGA, the PSOC can be programmed in C using a software called PSOC-creator. These software commands make up what we call here the AFE firmware. It is composed of a suite of building blocks relevant for sensor data processing.

To read the sensors, the AFE is controlled by data acquisition software run on external hardware, here the LOTUS box or a regular computer. To enable this, the AFE firmware offers a series of API that are called by the data acquisition software. There are two types of API, the Configuration APIs and the Operations APIs. Available APIs allow notably to:

- CONFIGURATION APIs:
    - Configure type of chips and types of sensors inside each chip
    - Configure Biasing and Readout parameters for each sensor
    - Configure sensor routing through the multiplexers
- OPERATION APIs
    - Return AFE version , sensor chip type.
    - Select sensor to acquire
    - Start and stop measurements
    - Stop AFE

4.2 Python acquisition code

4.2.1 Core acquisition code

The Python acquisition code – in short PAC; is a Python code that runs in the PC and communicates with a User, AFE and a Mongo database. It manages the following operations:

1- Gets AFE configuration from user and save it in the database
2- Gets Experiment configuration from user and save it in database
3- Loops over all or part of the 25 sensors on the chip (each sensor is activated sequentially)
4- Sends binary commands to configure the AFE
5- Starts and stops measurement
6- Reads and parse the received binary data

7- Converts values from binary to decimal (see example of output for 5 sensors on Figure 41)
8- Creates time scale
9- Saves the measured values in a local MongoDB database (see Figure 41)
10- Saves measured data from database to csv if requested
11- Gets calibrations data from user

The current version of the PAC is 1.6. There are different subversions P, S or B depending on context of use, respectively Platine Lab, Sense-City, Business/Partners

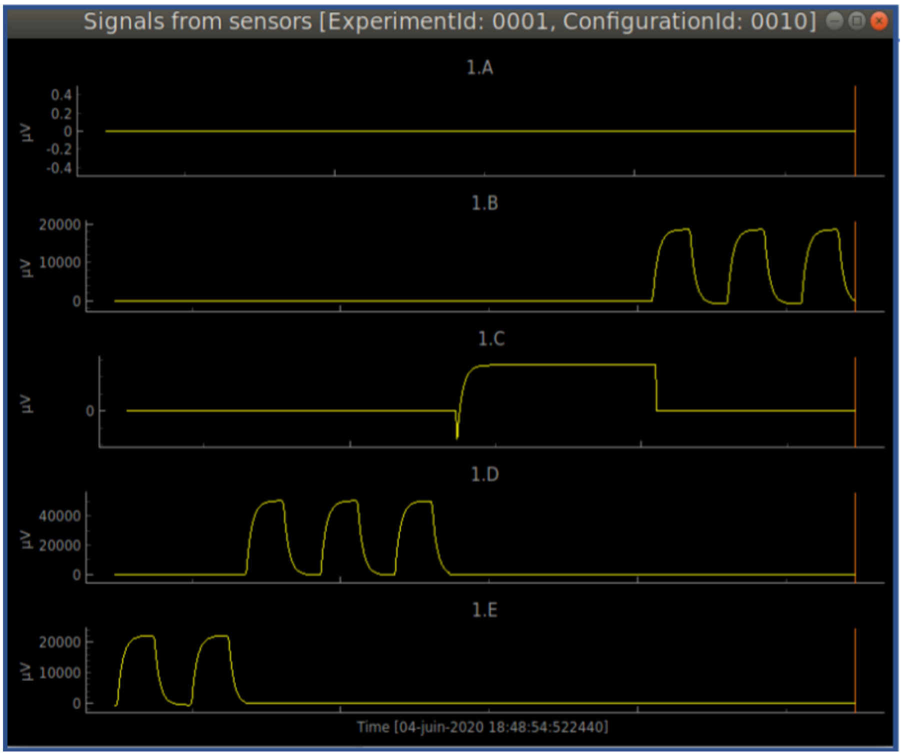

Figure 41: Example of data readout for 5 sensors read sequentially. Sensor 1A is non-functional, the other four are functional. The vertical axis are in µV, the horizontal axis is time (1 peak = 1s)

4.2.2 Graphical User Interface

Figure 42 shows examples of the Graphical User Interface (**GUI**) developed to work with the **PAC** in graphical mode instead of through command lines.

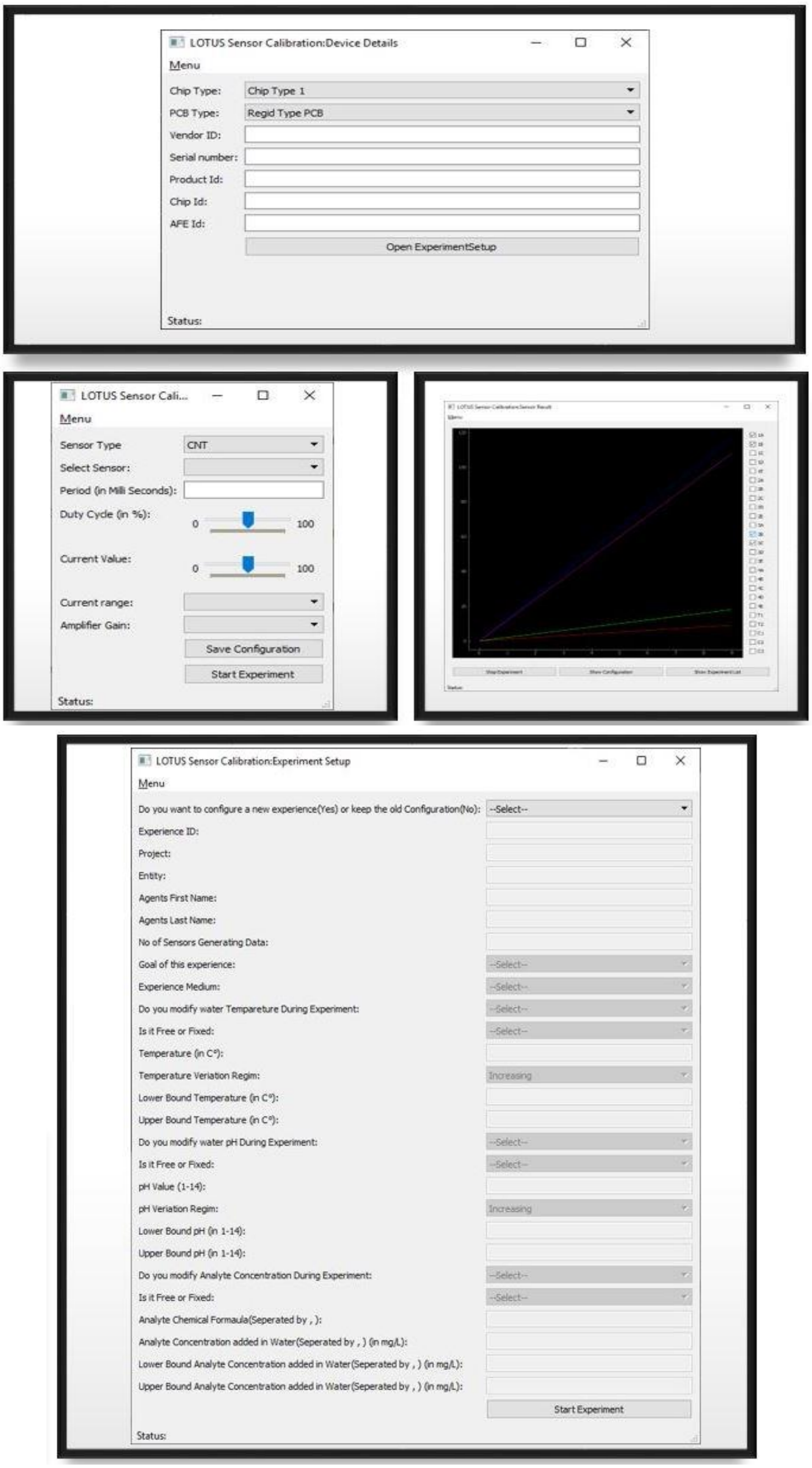

Figure 42: Graphical User Interfaces developed for PAC 1.6: top – sensor calibration data; middle left – biasing and acquisition configuration ; middle right – real time data display; bottom right – experiment configuration

LOTUS is co-funded by the European Commission under the Horizon 2020 research and innovation programme under Grant Agreement N° 820881 and by the Indian Government, Ministry of Science and Technology.

4.2.3 Database management

The PAC is interfaced with MongoDB databases. The PAC is based on predefined data models. It includes the connexion methods to each database and database read&write functionalities. Different databases were created depending on use cases.

Currently, most of the databases are local and hosted on local servers. To make this architecture more convenient to use and more resilient, it was elected to create cloud replica of these databases with daily syncing. After benchmarking currently available commercial options, AWS cloud services were chosen, notably the documentDB database service. Note that the LOTUS box software described below includes real time data upload to cloud, but it is a function not needed at this time for computer-controlled sensor node and tricky to implement.

Figure 43 shows the high-level connexion schematics between client and documentdB cluster. So far, the syncing commands in Python to carry out the syncing with documentDB are available, but the full migration has not been carried out. Briefly, the syncing operates as a DB upload to cloud, namely an insertion of new documents in the database. To manage this upload properly, the PAC records the status of the local database at the time of syncing; it allows to upload only the new or modified documents generated between syncing.

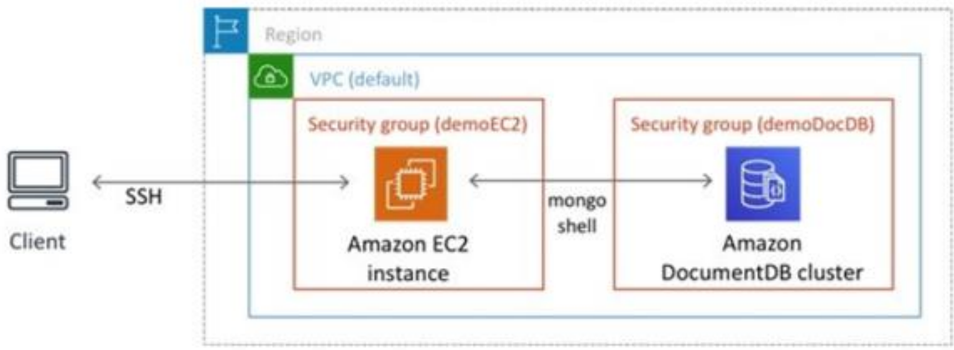

Figure 43: connexion schematics to the cloud

4.2.4 Data postprocessing

As can be seen on Figure 41, the sensor output signals are complex. A zoom on a single peak can be seen on Figure 44. In this version of the software, it was elected not to use the raw data of each peak for data exploitation but only an average of the signal over the stabilized part of each peak.

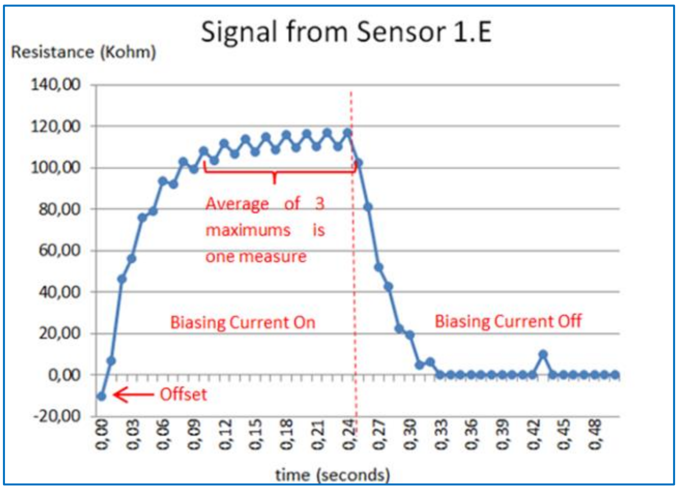

Figure 44: Details of a single peak

In short, the data post-processing steps include the following parts:

- Extract a relevant indicator from the raw signal of each peak, for instance the average of 3 maxima of each peak. The output signal is either a resistance (CNT and temperature sensor) or a voltage (conductivity sensor)
- Filter the resulting signal, notably to remove high frequency noise. The filtering approach used here yields a reduction of noise by a factor 8 to 10.
- Interpolate linearly successive data in time to provide a continuous time response
- Apply the calibration relationship to the sensor data to convert them into the target unit based on calibration coefficients entered by the user (for instance, linear calibration relationship with slope and y-intersect coefficients to enter).
- If necessary, apply data fusion algorithm on the calibrated sensor data to improve their accuracy (for instance averaging of selected sensors).

The performances of this data post-processing method, the derivation of the calibration relationship and the choice of the data fusion algorithm are discussed in the validation deliverables D2.3 and D2.9.

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4.3LOTUS box software

The LOTUX Box software is built using the STM32 development ecosystem. An hardware abstraction layer is built to abstract the sensor and communication hardware interfaces. The software itself is foreseen to run as a state machine which periodically poll the AFE to query the sensor and send the values to the cloud after possible data filtering. A previous phase has been analysed to allow gathering measurements from the AFE and publish it both a Mongo database and a FIWARE cloud platform with the preliminary logic described on Figure 45 where the sensors are sequentially queried, their data flow buffered and processed (average) to get the value measurement.

This model will be adjusted as processes are main on the AFE side as it is expected that most of the signal pre-processing will be done on the AFE side.

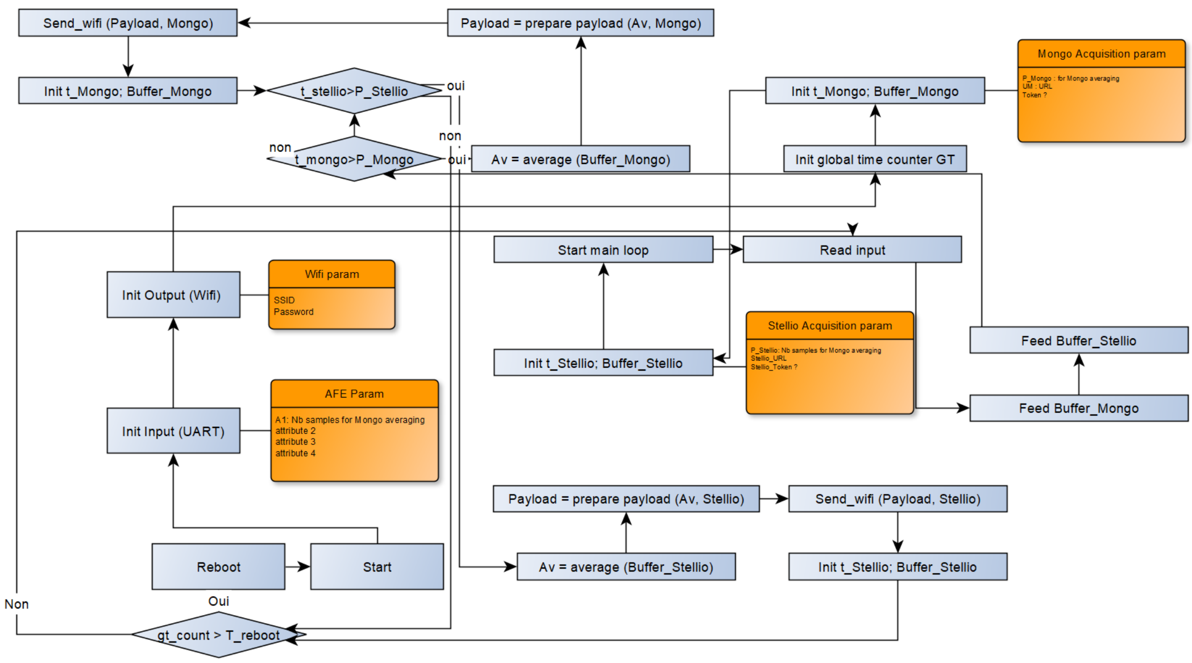

Figure 45: LOTUS Box initial data acquisition flow

5 Conclusion

All elements of a LOTUS node have been specified and designed. First realisation shave been made.

The initial operation tests have been run, focusing on the interoperability between the different components' interfaces. These are documented in LOTUS deliverable D2.9 "Validation of the first version of LOTUS Prototype".

Appendix

1. Synthesis of FF-UR-14 polymer

The section describes the synthetic scheme for FF-UR-14. This section is reproduced from the deliverable D3.1 of Proteus Project (https://cordis.europa.eu/project/id/644852), as this deliverable is not public so cannot be found easily online. The synthetic scheme is displayed in Figure 46.

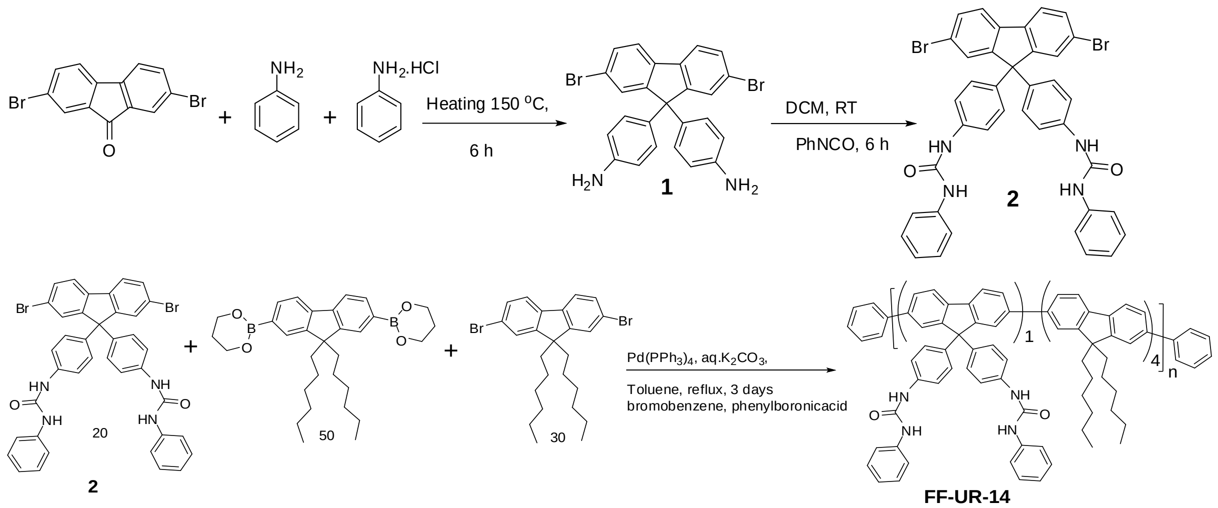

Figure 46: Synthesis of FF-UR-14 polymer.

Compound (1): Synthesis of 4,4'-(2,7-dibromo-9H-fluorene-9,9-diyl)dianiline (1)

To a mixture of 2,7-dibromo-9-fluorenone (0.5 g, 1.5 mmol) and aniline hydrochloride (0.23 g, 1.5 mmol), aniline (5 mL) was added under argon atmosphere (turns to red colour) and heated at 150 °C for 6 h, while heating it turns to dark colour. After completion of the reaction, cool it to room temperature and the reaction mixture slowly poured into water (100 mL) and extracted with ethyl acetate (3 X 50 mL). The combined organic extracts were dried over Na_2SO_4, concentrated and recrystallized from THF-*n*-hexane (1:1) to obtain compound **1** as dark gray solid (0.55 g, 73%).

Compound (2): Synthesis of 1,1'-(4,4'-(2,7-dibromo-9H-fluorene-9,9-diyl)bis(4,1-phenylene))bis (3-phenylurea) (2)

To a solution of 1 (0.4 g, 0.8 mmol) in dichloromethane (10 mL), phenyl isocyanate (0.22 mL, 1.9 mmol) was added dropwise under an argon atmosphere at room temperature and stirred for 4 h. Then it was diluted with diethyl ether (20 mL) filtered and washed with diethyl ether (50 mL) and DCM (20 mL) to obtain 2 as gray solid (0.41 g, 69%).

Synthesis of FF-UR-14: Polymer of 1,1'-(4,4'-(9',9'-dioctyl-9H,9'H-2,2'-bifluorene-9,9-diyl)bis(4,1-phenylene))bis(3-phenylurea)

Compound **2** (0.25 g, 0.34 mmol), 2 7-dibromofluorene (0.25 g, 0.5 mmol), 2,2'-(9,9-dihexyl-9H-fluorene-2,7-diyl)bis(1,3,2-dioxaborinane) (0.5 g, 0.85 mmol) and $Pd(PPh_3)_4$ (50 mg, 0.04 mmol) were dissolved in toluene (30 mL) purged with argon for 30 min. Then, aq. K_2CO_3 (0.59 g, 4.3 mmol) (8 mL) was added slowly to the above mixture and refluxed under an argon atmosphere for 3 days. Bromobenzene (22 L, 0.21 mmol) had added and continued for 6 h, before phenylboronicacid (26 mg, 0.21 mmol) added and continued for another 6 h. Cool it to room temperature and slowly poured into methanol: water (400 mL, 10:1) filtered and repeated twice, and washed with water and methanol and further purified by soxhlet extraction with acetone (2 days). Weight of the sample is 450 mg.

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