#### 3D PRINTED PIPES INCLUDING SENSORS AND HEATERS FOR THERMAL MANAGEMENT SYSTEMS IN SPACE AND ON EARTH

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#### 1. INTRODUCTION

The motivation for the development of 3D printed pipes including sensors and heaters for thermal management systems comes from the limitations and difficulties in directly monitoring and acting on fluid properties with existing methods. Sensing parameters such as pressure, temperature and flow rates of refrigerant are of utmost importance for the operation of thermal management systems, on earth and in space. Ideally, these measurements shall be performed directly in the fluid flow with sensors directly in contact with the fluid. In practice, this approach cannot always be applied due to hardto-reach measurement areas, lack of space and limitations on the total mass of the system. Current systems use sensors either located on the outer surface of pipes or inside the pipe as additional elements, introducing additional volume, mass and cable management constraints.

This paper presents the development of pipe segments including temperature sensors, heaters and energy harvesters directly integrated into the pipe thanks to a patented [1] design and manufacturing concept relying on advanced processes such as metal Laser Powder Bed Fusion (LPBF), Aerosol Jet Printing (AJP) of electrically conducting ink patterns as well as thin insulation layers.

#### 2. SMART WALL PIPES AND DUCTS - SWAP

The SWaP project was created within the collaboration between CSEM and CERN, in the frame of ATTRACT Phase 1 [2]. The inspiration of SWaP derived from the increased demands and requirements on thermal management systems and the limitations of direct sensing and monitoring fluid properties of such systems. Taking advantage of the Additive Manufacturing (AM) technology, a pipe with fluidic and electrical interfaces and embedded sensing capabilities was developed for fluid temperature monitoring. The 3D printed segments of pipe were equipped with standard hydraulic fittings and an Aerosol Jet Printed Resistance Temperature Detector (RTD) in the inner wall of the

pipe (Figure 1), providing direct contact fluid's measurements.

The pipe has been tested and integrated in refrigeration test plants at CERN, providing leakfree (leak rate: 10<sup>-10</sup> mbar·l/s Gaseous He) connections with standard commercial fittings. The AJP temperature sensor was tested and compared with commercial sensors (Pt100), mounted on the outer surface of the pipe, and immersed in the fluid's stream (Figure 2). The AJP sensor presented a linear resistance-temperature relationship and a low level of the common effect of hysteresis (Figure 3). The comparison of the AJP sensor with commercial RTDs shows that the AJP sensor response is very similar in terms of linearity and hysteresis, but it is nosier (Figure 4), with one order lower precision (Figure 5, Table 1) than commercial RTDs. Both could be improved by optimized AJP deposition parameters and sintering conditions.

The project addressed the feasibility of combining two AM methods, to produce one single device with fluidic and electrical features, as well as integrated sensing capabilities. The SWaP prototype is a device, fully produced by AM that:

- a. Can be integrated in any hydraulic circuit.
- b. Provides leak tightness even at high pressure ranges (up to 70 bar).
- c. Provides reliable fluid temperature measurements, comparable with immersed commercial Pt100 RTDs.
- d. Does not induce disturbance to the flow (noinvasive measurement)



Figure 1: Smart pipe element developed in SWaP (ATTRACT Phase I)



Figure 2: 3D printed temperature sensing segment of pipe with an embedded AJP sensor compared to two custom-made immersed probes ("Probe 1" and "Probe 2") and two surface sensors ("TS1" and "TS2") sensing the fluid temperature in a circuit.



Figure 3: AJP sensor linear temperatureresistance behavior for the temperature range of – 20°C to +20 °C, with low hysteresis during cooling and heating.



Figure 4: Recording at a constant temperature with a sampling rate of 1Hz to compare the noise among the sensors.



Figure 5: Mean value of temperature of immersed (o), surface ( $\cdot$ ) and AJP sensor ( $\Delta$ ) for seven temperature groups within a cooling – heating cycle, indicating reading variations for a constant temperature.

Table 1: Estimated precision (temperature mean
value plus standard deviation) of the five sensors
for the seven temperature groups.

	TS1	TS2	Probe1	Probe2	AJP
1	5.5	5.2	5.0	5.2	5.1
	±0.005	±0.005	±0.005	±0.005	±0.014
2	-1.9	-2.4	-2.6	-2.5	-2.5
	±0.006	±0.006	±0.006	±0.006	±0.014
3	-10.4	-11.0	-11.3	-11.1	-11.2
	±0.009	±0.011	±0.013	±0.011	±0.017
4	-18.8	-19.5	-20.0	-19.7	-19.8
	±0.005	±0.005	±0.007	±0.007	±0.015
5	-10.6	-11.3	-11.7	-11.4	-11.5
	±0.007	±0.007	±0.010	±0.009	±0.016
6	-2.1	-2.6	-2.9	-2.7	-2.8
	±0.005	±0.006	±0.006	±0.006	±0.015
7	6.2	5.8	5.7	5.8	5.7
	±0.006	±0.005	±0.006	± 0.005	±0.013

## 3. ADVANCED HEAT EXCHANGE DEVICES – AHEAD

The main goal of AHEAD is to start from the technology bricks developed in SWaP and to develop a TRL7 [3] product, bringing the possibility of sensing fluid parameters with a simple pipe segment to an industrial pre-production level, compatible with natural refrigerants: Carbon Dioxide (for detector applications) and Ammonia (for space applications). To reach this goal, two use-cases are developed: the "MPL" use-case (Mechanically Pumped Loop) targeting thermal management applications for satellite platforms, and the so-called "Refrigeration" use-case targeting earth applications.

AHEAD is organized in two phases. First, five Key Enabling Technologies (KET) bricks will be developed and tested to de-risk the next phase. The preliminary design of the use-case will progress in parallel. In the second phase, the KET bricks will be combined to meet the two use cases from which the project requirements are derived. Validation of the use-cases at system level and in the representative environments will finally be performed.

#### 3.1. Use-case description and key spec

#### 3.1.1.Mechanically pumped loop

The thermal control of a telecommunication satellite with a two phases Mechanically Pumped Loop (MPL) instead of a heat pipes network is a new technical solution that satisfies the need of low mass and compactness while allowing even higher heat transportation and spreading. Some MPL components are now considered to be manufactured by AM, which enables significant reduction of mass (~30%) and material waste during the manufacturing process. This hermetic loop MPL uses a heat transfer fluid which circulates between evaporators (where the thermal energy of the units to control is collected/removed) and condensers (where the thermal energy is evacuated toward space). A two-phase MPL is a complex system that must be monitored in real time since the payload dissipation varies continuously. The MPL control laws relies Negative Temperature Coefficient (NTC) thermistors that are placed on different components of the loop.



Figure 6: Current MPL Evaporator (floor) circulation tubes principle

As short-term application for MPL stainless steel solution, some portions of tubes with embedded NTCs and heaters are the use-case selected for the AHEAD project. At longer term, fully integrated instrumented evaporators embedded into a structural panel aluminum are also foreseen. Once developed, in-situ monitoring of fluid flows could also apply to propulsion applications in spacecraft and local heating to tanks, batteries or every curved surface equipment. In-situ monitoring could also be extended to strain gauges or accelerometers without any large gap in the technology. Another field of application are Single Phase Loop (SPL) systems which are used in some ISS modules and will also be installed on the future lunar space station.

#### 3.1.2.General refrigeration

In the pursuit of its scientific mission, CERN employs innumerable large and complex silicon detectors that require constant precise thermal management for optimal operation. Such a large network of heat-generating units requires an even larger and more complex bespoke system capable of extracting several hundreds of kilowatts from a vast closed volume, while maintaining a controlled temperature over hundreds of square meters of silicon surface.

CERN's thermal management systems rely on a transcritical CO<sub>2</sub> refrigeration cycle in cascade with a CO<sub>2</sub> mechanical pumped loop, and it involves a multi-branch system with complex distribution. Therefore, distributed sensing of local flow parameters is essential for optimizing heat exchange across the thermal circuit. However, the environments where silicon detectors are present are also characterized by strict mass and volume limitations. The use of AM-produced elements within these hydraulic systems provides the necessary freedom of design to answer these constraints, while the inclusion of embedded sensing capabilities allows for the precise monitoring of vital parameters throughout the thermal management system.

#### 3.1.3.AHEAD Use case main requirements

The analysis of the two use cases leads to the Table 2 which presents the key requirements of the project. For each requirement and where possible, a common value has been specified, especially for design driving requirements such as pipe material, pipe dimensions and definitions of mechanical and electrical interfaces.

The major difference between the two use cases is the refrigerant considered, anhydrous ammonia  $(NH_3)$  for the space application with MPL and Carbon dioxide (CO<sub>2</sub>) for general refrigeration.

Requirements	MPL	General refrigeration		
Pipe material	LPBF Stainless Steel 316L			
Refrigerant	Anhydrous ammonia, in liquid phase	Carbon dioxide R744		
Flow rate (ml/s) / refrigerant speed (m/s)	Flow rate of 33 to 66	Speed of 0.5 to 10		
Pressure range (bar)	48 at 85 °C	5 to 130		
Temperature range (°C)	-65 to 85	-53 to 90		
Max. pipe length (mm)	1	100		
Pipe external diameter (mm)	about 10	4 to 12		
Max pipe mass (g)	250			
Pipe wall thickness (mm)	About 1			
Heater power (W)	63	N. A.		
Mechanical Connector type	Swagelok (de TIG Welding	(dev. phase) and ng (final product)		
Electrical connection outside of the pipe	Sub-D o	connector		
Temp. sensor accuracy (°C)	±0.5			
Lifetime (year)	15			

Table 2: Summary of AHEAD use-cases main requirements

#### 3.2. Key Enabling Technology (KET) bricks

The first phase of the project consists in designing, manufacturing and testing the KET bricks that are presented hereafter and, illustrated in Figure 7. Those are:

- 1. Pipe segment and mechanical interfaces
- 2. Electrical feedthrough and connector
- 3. AJP temperature sensor and heater
- 4. Commercial of the shelf (COTS) temperature sensor and heater integration
- 5. Energy harvester





Figure 7: Architecture of the two use-cases in terms of Key Enabling Technology Bricks, AJP version (top), COTS version (bottom)

Note that the energy harvester element is not represented in Figure 7 because its integration within the pipe is still in progress.

The following sub-chapters describe the KET bricks design and manufacturing.

#### 3.2.1.Manufacturing workflow

The overall workflow for the manufacture of an instrumented pipe is shown in Figure 8. In order to incorporate the various components into the pipe, the LPBF process will be stopped, in particular to give access to the connectors and the location where the components will be integrated. Then the part will be cleaned and inspected. The entire build plate (BP) will then be shipped to CSEM for the casting of the feedthrough and the integration of the elements. The build plate will then be sent back to LISI to resume the LPBF process and machine the mechanical interface. In between all these steps, proper cleaning and inspection will be performed.



Figure 8: Simplified manufacturing workflow

Note that for the Energy Harvester KET brick, its integration will be performed at the same stage than for a COTS element, as described in 3.2.6.

### 3.2.2. KET Brick 1 – Pipe segment and mechanical interfaces

This KET brick consists of a metal pipe segment with its mechanical interfaces. The main objective of the development of this KET brick is the evaluation of the manufacture of an additive pipe in 316L with its mechanical interfaces. With freeform design in additive manufacturing, it is possible to produce optimized pipes, but the longer the pipe and the smaller the cross-section, the more difficult it will be to remove the powder. The objective of this KET brick is also to validate the quality of the pipes whose printing was interrupted for the integration of AJP/COTS elements and then resumed.

The outer and inner volume envelopes are defined in Table 2. In addition, the pipe geometry shall be optimized specifical keeping the AM LPBF process in mind. Notably:

- The overhang angle shall ideally not be smaller than 45°, and definitely not smaller than 40°.
- The maximal diameter of a horizontal cylinder shall not exceed 8 mm and the minimal diameter shall not be smaller than 2 mm to guaranty that is not clogged.
- The minimal wall thickness shall not be inferior to 0.1 mm and ideally be greater than 1 mm.
- The minimal diameter of a pin shall be greater than 0.2 mm.

The pipe KET brick as represented in Figure 9 has been designed.



Figure 9: 3D image of the pipe after AM print and support removal (left); after interface machining (right)

The internal shape of the pipe is described in Figure 10. The ogive shape allows for printing without addition of support structure. The flat inner bottom is adapted for the integration of the AJP/COTS elements. The bottom outer part of the pipe is designed to ease the integration of structure supporting the pipe during printing. The wall thickness of the tube is about 1 mm to guarantee the pressure resistance.



Figure 10: Pipe cross-section

An opening is added at each end to allow the removal of powder trapped inside the pipe as shown in Figure 11.



Figure 11: Inner shape after AM print (left); Inner shape after machining (right)

Two types of mechanical interfaces are being considered and implemented, Swagelok connectors which allow rapid testing of the prototype during the development phases, and TIG welded interfaces which will be used for the final product. Machining operations are performed on the pipe detached from the build plate to achieve the mechanical interfaces.

To integrate the temperature sensors, heaters and energy harvester devices into the pipes, the LPBF process must be stopped, and the build plate disassembled and removed from the AM machine. The integration of the components will then be completed, and the build plate will be reintegrated into the AM machine for the LPBF process to resume. There are several main challenges that need to be overcome when stopping and restarting a batch, as they can impact the quality and consistency of the final product. Some of these difficulties include:

- 1. Residual Heat: The laser beam melting process generates a high amount of heat, which can make it challenging to stop the process and restart it later without affecting the quality of the product. Residual heat can cause warping or cracking, and the process parameters must be carefully managed to prevent these issues.
- Material Degradation: LPBF process typically uses metallic powders as feedstock, which can degrade over time if exposed to air, moisture, or other environmental factors. This can impact the properties of the final product and affect the consistency of the process, making it challenging to restart a batch.
- 3. Part Distortion: The heat generated during the LPBF process can cause the part to expand and contract, which can result in distortion. Stopping and restarting the process can make this problem even more pronounced, requiring careful process management to ensure consistent results.
- 4. Calibration: The laser beam melting process is highly dependent on accurate laser calibration, which can be affected by stopping and restarting the process. Any deviation from the ideal laser position or power can result in inconsistencies in the final product, making it challenging to restart a batch.

In conclusion, restarting a batch in laser beam melting process is challenging due to residual heat, material degradation, part distortion, and calibration issues. Careful management of the process parameters and ensuring that the materials and equipment are in optimal condition will minimize these difficulties and ensure consistent, high-quality results. A large panel of tests will be put in place to monitor the quality of the pipes produced. Pipes whose printing has been interrupted and resumed will be compared with pipes printed at one time.

#### 3.2.3.KET Brick 2 – Electrical feedthrough and connector

The electrical feedthrough and connector KET brick shall provide electrical connection between the interior and the exterior of the pipe while ensuring the tightness of the pipe. The geometry of the external connector is chosen as a standard 9-pins D-sub male connector so that it can be universally used across a large variety of applications.



Figure 12: 3D image of the electrical feedthrough KET brick (left); electrical connection within the pipe with insulating resin in blue (right)

The concept of built-in wires relies on CSEM's patent [1].

To provide a compact design, to minimize the needs for support structures during LPBF and to ease the resin casting process, the feedthrough is U-shaped. On the D-Sub side, the wires are connected to the structure by the "bottom of the U". To minimize the length of the wire while maintaining an adequate surface area along its path, the shape is designed to be oblong.

Two versions of the feedthrough are designed on the inner side of the pipe, one to connect AJP elements, and the other to connect COTS elements. The AJP elements are connected by direct printing of their conductive silver ink wires onto the top surface of the feedthrough wire, while for the COTS elements, female connectors crimped onto the element wires are pressed onto the feedthrough pins.



#### *Figure 13: AJP (top left) and COTS (top right) electrical wire designs* The selection of an appropriate insulation resin is a

key element of the development of this KET brick. The resin must provide good electrical insulation and withstand the mechanical load induced by the pressure of the fluid inside the pipe. In addition, it must be leak-proof and be compatible with the type of refrigerant used. Its mechanical properties should not vary too much in the temperature range considered. Finally, the coefficient of thermal expansion (CTE) and elastic modulus of the material shall be as close as possible of their 316L SS counterpart to avoid high stress levels during thermal cycling.

Taking all these aspects into account and looking for materials readily available on the market, two families of resin materials are considered, epoxies and polyimides. UV-curing epoxies were excluded as optical access cannot be guaranteed. In addition, the curing temperature of AJP elements is approximately 200°C, so the casting resin must withstand this temperature without damage. For the casting process, the ideal resin viscosity should be in the range of 100 to 5000 mPa·s.

The feedthrough manufacturing process is divided into three main phases:

- 1. LPBF printing of the metallic parts including support structure to hold the wires in place.
- 2. Casting of a resin that provides electrical insulation and support for the electrical wires.
- 3. Removal of the wire metallic support structure.

The casting process is done in two steps. The resin is poured through the first pipe (Figure 14 left). After curing, a second layer of resin is poured through the second pipe (Figure 14 right). For the COTS version, the COTS element is connected to the pins before the second casting process (Figure 14 bottom).



Figure 14: illustration of the casting process; AJP version (top), COTS version (bottom)

After the casting process, the part is removed from the AM build plate. On the underside of the part, a milling cut is made to disconnect the wires from the part. The two slots for the D-Sub nuts are also milled. Finally, a final resin casting is performed to isolate the pins.



Figure 15: Bottom view of the feedthrough after machining of the bottom surface and milling of the D-Sub nut slots.

An image of a casted feedthrough connector sample is given in Figure 16.



Figure 16 : Image of the feedthrough KET brick (AJP version) casted with epoxy

### 3.2.4.KET brick 3 – AJP temperature sensor and heater

The objective of this KET Brick is to develop a RTD with similar thermal sensitivity, accuracy, and repeatability to a standard 4-wire PT1000 and to develop a heater using the same technology and material to locally heat the refrigerant. Aerosol jet printing (AJP) is an advanced version of the recently developed technologies for high resolution patterning of functional materials. AJP is distinguished from other printing technologies concerning the high-resolution patterning i.e., down to 10 µm compared to the ≥ 60 µm pattern sizes achieved by other competitive technologies. Prospects of processing a wide range of materials including conductors, semiconductors, insulators, and epoxies make AJP a versatile tool for printed electronics. Colloids as well as chemical resins at different viscosities ranges (from 1-1000 cP) and less process parameters are the attractive and promising traits of this fast-emerging technology. The high resolution of printed patterns especially electrically conductive structures makes AJP ideal for dense integration of electronic components on planar as well as 3D topographies.

The AHEAD AJP elements are made of three different layers:

- Insulation/planarization layer: provide electrical insulation and planarize the surface for support the electrically conductive track deposition.
- Deposition of an electrically conductive silver

ink: the ink is deposited with a particular precise geometry to serve either as an RTD equivalent to a commercial PT1000 temperature sensor or as a heating element.

 Encapsulation layer: to protect and insulate the electrically conductive ink from the environment.

Both insulation and encapsulation layers are made of the same materials, which can be either an UV-curable epoxy or polyimide material. The process parameters are being optimized for both materials and tests performed to check their compatibility with the refrigerants. Thickness of both layer is about 20  $\mu$ m.

The silver ink track is deposited on the insulation layer in a meandering pattern and with a thickness and length tuned to obtain the targeted resistance of 1000  $\Omega$  at 0°C similar to a PT1000.



Figure 17: 3D view of a AJP RTD sensor (left) and image of a RTD sensor and a heater deposited on a 316L substrate (right)

A view of the ink pattern sandwiched between two PI layers printed on an AM representative 316L substrate is given in Figure 18. The ink pattern is composed of 40  $\mu$ m wide lines at 20  $\mu$ m pitch size. RTD covers an area of 12×5 mm<sup>2</sup> with a track length of 500 mm.



Figure 18: AJP RTD silver ink track sandwiched between two PI layers.

Thermal tests have been performed on RTDs deposited on commercial PI foils and encapsulated with a thin layer of PI. The results show good linearity, low hysteresis, and good reproducibility of temperature measurements.



Figure 19: Thermal behavior of 4 RTDs deposited on commercial PI foil and encapsulated with polyimide by AJP.

Thermal testing has also been performed on two samples deposited on a representative AM 316L substrate and gives promising results as shown in Figure 20. One can observe practically no hysteresis on the measured resistance curves and almost identical temperature coefficients for both RTDs in Table 3.



Figure 20: Temperature vs resistance curves for the RTD 1 (top) and RTD2 (bottom) deposited on representative AM substrate by AJP with silver ink in between two thin layers of polyimide.

Table 3: Temperature coefficient of the two RTDs of Figure 20.

RTD sample	Temperature coefficient
AJP-RTD-Ag-1000Ohm-AM-ENC-001	0.002281
AJP-RTD-Ag-10000hm-AM-ENC-002	0.002281

Further tests will be performed on additional PI samples and on samples printed between two layers of epoxy in order to validate the materials, the manufacturing process and the behavior of the sensors.

Finally, with respect to the printed heater, tests have been performed to demonstrate that a large surface area can be covered by AJP using silver ink and PI insulation and encapsulation layers. It is expected that the entire area of the tube dedicated to the heater can be used, approximately 40 x 8 mm<sup>2</sup>.



Figure 21: Silver ink-based heater deposited between two PI layers on an AM representative 316L substrate.

Preliminary tests were performed on a sample heater printed on PI foil with a measured resistance of approximately 1.3 k $\Omega$ . The heater was placed in a vacuum chamber and a potential difference of 30 V was applied across its terminals.



Figure 22: AJP heater long-term temperature measurement, in vacuum, with 30 V input

Figure 22 shows the long-term stability of the printed heating element. The heating power of the heating element is low, about 0.8 W, and further testing will be done with larger devices with higher electrical resistance and higher input voltage.

## 3.2.5. KET Brick 4 – COTS temperature sensor and heater integration

The objective of this KET brick is to demonstrate that COTS components such as temperature sensors and heaters, can be integrated in the pipe segment of KET brick 1. This will open the door to the integration of other elements such as pressure sensors and accelerometer for instance and expend the field of application beyond limitations of AJP.

The integration of COTS temperature sensor and heater in the pipe segment is illustrated in Figure 7 (bottom).

For the MPL use case, a heater cartridge is used in conjunction with a Negative Temperature Coefficient (NTC) temperature sensor. For the GR use case, only a RTD sensor is used.

The COTS elements are connected to the electrical feedthrough using commercially available sockets. The elements are fixed on the bottom of the pipe and covered with protective resin. This is the same resin used for the insulation of the electrical feedthrough connector. The fixation is ensured by flexible clamps printed on the pipe flat inner bottom. The geometry of these clamps has been optimized to ensure a good fit of the element to integrate.

#### 3.2.6. KET Brick 5 - Energy Harvester

The energy-harvesting KET brick shall provide electrical energy to the RTD sensor of CERN's refrigeration use case. Nevertheless, the development and integration of energy harvesting systems will enable standalone sensing and wireless data transmission for a wide range of industrial and scientific IoT applications in the near future. Based on the results obtained in the "Energy4Oil" project from ATTRACT Phase I [4], the chosen solution relied on the tailoring of triboelectric nano-generator (TENG) membranes and their hybridization with commercially available piezoelectric (PZE) membranes able to transduce the mechanical motion of the refrigerant into electrical energy (Figure 3-11).



Figure 3-11: Design for the triboelectric energy harvesting system inside a straight tube.

By using numerical simulations, a study to optimize different tube structures and configurations to enhance the turbulence and pressure drop and thus increase the amplitude and frequency of the oscillation of the membranes was conducted. These results determined that a Venturi tube is the best approach for additive manufacturing. Therefore, the following study focused on optimizing the corresponding internal slopes, diameters, and the throat length relative to the inlet (Figure 3-12).



numerical simulations.

The configurations and materials of the triboelectric membrane were then studied experimentally. Therefore, after the optimization of the electrode preparation method (Figure 3-13), the chosen assembly is constituted by silver ink deposited on chemically-activated Kapton, coupled with a Polytetrafluoroethylene (PTFE) membrane. It is also important to note that a single electrode configuration is being used, where the silver ink works both as a triboelectric material and electrode for the external electrical connection.



Figure 3-13: Output performance of different triboelectric devices assemblies.

In the hybrid device, the developed triboelectric membrane is connected in parallel with a commercial piezoelectric membrane using a female FFC connector, which is then fixed in grooves inside the tube.

Furthermore, the energy harvester performance in both straight and Venturi tubes under an airflow of 40 L/min, a pressure of 2 bar, and using a full-wave rectification circuit to enable energy storage were studied. The experimental results shown in Figure 3-15(A) demonstrate that there is a clear advantage in using the Venturi tube over a straight one, with an increase in the maximum voltage reached in a 1  $\mu$ F capacitor (from 3.8 V to 13.6 V) when using the TENG alone. Finally, employing the optimized Venturi tube and a gas flow of 25 L/min at 2 bar, the hybridized device shows a significant performance

improvement, reaching 53.9 V in 8 minutes (Figure 3-15B).



Figure 3-15: (A) Capacitor charge resorting to a straight and Venturi tube using the TENG energy harvester. (B) Capacitor charge resorting to a Venturi tube, and the hybrid energy harvester.

#### 3.2.7.KET bricks validation strategy

Based on a risk analysis, the critical points to be addressed for each KET brick as de-risking before printing the final prototypes have been identified. Early actions have been performed in the first step of the project such as co-engineering between the partners for the design of the pipe segment, tradeoff of technical solutions and early testing.

The aim of dividing the prototype into KET bricks is that working smaller by on parts/processes/systems, a concrete analysis can be obtained for each sub-system individually. Consideration and collection of parameters and information for each distinct system increases the likelihood of producing a failure-free final prototype. It is essential to recognize the initial and highest development risks, their causes, circumstances and consequences. Taking into account the severity and probability of such incidents, the following major risks have been identified:

• **Resin chemical compatibility** with the refrigerant is one of the first and main risks to consider. An initial study based on the literature, the technical specifications, and the supply availability on the market of resin products was a mandatory step to find out proper candidates of resins. De-risking further the possibility of bad compatibility, resin samples will be exposed to the refrigerants and be compared with non-

exposed samples.

- Electrical feedthrough by itself and with *casted resin* is a critical aspect, firstly because of the surface finishing of AM pins which is very different than commercially available ones and secondly due to the large volume of casted resin, which will be subjected to different thermal processes and temperatures (curing, AJP sensor curing, resume of the AM process). Testing the quality of electrical contact and the robustness feedthrough of the toward connection and disconnection is essential. Process parameters might affect the structure or the surface of the casted resin and lead to unsealed segments which will cause leakages to the refrigerant's flow. However, if these defects are minimal, they might not be easily detected under ambient conditions (temperature and pressure). It is essential that the feedthrough is exposed to the fluid flow and tested for the operational temperature and pressure range of the refrigerant.
- Stop and resume AM process can mainly affect the AJP sensor and its substrate of resin during the resume process. High temperature is induced due to the laser power, and therefore the temperature in the AJP sensor region is also increased. The associated risk is that the resin degrades, and the electrical properties of the sensors are altered.
- Thermal and mechanical Loading can affect lifetime of the product. Several thermal cycling tests are necessary to evaluate thermal fatigue impact especially on the encapsulation resin in addition with vibrations tests to address launch fatigue for MPL case. The first batch of test on KET Bricks will allow to make necessary adjustment on the prototype design to achieve targeted performances.
- Energy Harvester (EH) functionality with the refrigerant should be validated at a preliminary level, to verify the EH durability for temperature and pressure ranges of the final application, as well as sufficient electrical power generation. Thus, the design, the geometry, and the material of the EH device could be modified or optimized to ensure adequate level of functionality before the final protype is being produced.

To address all the identified risk at KET brick level, a test plan has been edited with the contribution of each partner.

As an example, the compatibility with ammonia of the resins to be used for the casting of the electrical feedthrough will be evaluated in 4 progressive steps:

- 1. Perform a trade-off and select a few realistic candidates according to literature and compatible with a supply within the project. Epoxies and Polyimide have been found as credible candidates and procured.
- 2. Perform simple optical inspections and weight of the eligible resins after a life duration

representative immersion into ammonia at the maximum operating temperature. This test will allow to detect potential damage to the resins to make a preselection of a few candidates with high ammonia resistance capability.

- 3. Assess all the risks at elementary level, testing the resins all together (for feedthrough and AJP) in a printed SS container to be representative of future use conditions with potential crossing interactions: The gas plug measurements are in addition to optical and weight inspection to detect potential physical damage to the resins and chemical reaction with the pipe segment and ammonia. In parallel, the fatigue behavior of the casting resin will be evaluated.
- 4. Investigate the thermo-elastic behavior by performing thermal cycling of the equipped pipe segment and examine the potential consequences of resin fragmentation on the particle cleanliness requirement, which is crucial MPL systems.

Another example concerns the Energy Harvester functionality with carbon dioxide refrigerant. Before using the EH with CO<sub>2</sub>, several tests have been performed, to select proper materials that can be electrically charged and the type of configuration, which can generate sufficient electrical power and energy storage can also be provided. The testing sequence consists of the following steps:

- 1. Materials, operational principle, and configuration of the nanogenerators have been selected according to the literature and COMSOL simulations.
- 2. Three types of nanogenerators have been developed (piezoelectric, triboelectric and hybrid) and integrated into two different types of tube (straight and Venturi). Besides the simulations, tests performed for each possible combination of them with different fluids, in order to find out which configuration and under which conditions generates sufficient electrical power.
- 3. The EH devices have been then integrated inside standard stainless-steel tubes, to be easily connected with standard fittings to a refrigeration circuit test setup.
- 4. Further tests performed on the nanogenerators after their integration to the standard tubes, to verify the electrical power generation.
- 5. The EH devices will be then connected to a circuit to be charged with CO<sub>2</sub>, where pressure, temperature and flow will be recorder during the test. Therefore, the functionality of the EH devices with the working refrigerant can be verified.

#### 3.3. Work in progress

KET bricks design and validation activities are planned until 3<sup>rd</sup> quarter of 2023. Then, fully equipped pipe segments dedicated to both use cases will be validated in representative environment with the objective of reaching TRL7. Concerning the pipe segment (KET brick 1), 316L AM material properties are validated, and the mechanical and material properties of pipe segments printed without and with the stop and resume process are being validated. Measurement of the surface temperature when the AM process is resumed have been performed and shows that the region where the AJP elements will be printed does not exceed the limits of the AJP ink and resins. The mechanical interface and the overall geometry are well defined. Since the cleanliness level of the parts is of utmost importance for the MPL system, standard cleaning procedures will be tested and the particular contamination level will be monitored to demonstrate that the applicable cleanliness level can be achieved. Finally, pressure resistance of the pipe segment will be demonstrated.

Regarding the feedthrough, KET brick 2, dedicated test samples for leakage and pressure resistance validation are produced and testing is planned. Thermal cycling of these samples is also planned to reduce the risk induced by thermal fatigue on pressure resistance. The pressure test sample design is shown in Figure 23 (left). Samples with different casting resins will be tested. The various materials in contact with the refrigerant are tested using, among others, resin dogbone samples (Figure 23; right) that will be exposed to the refrigerant, and then tested in fatigue or tensile. Resin material properties will be compared to those from samples not exposed to refrigerants.



## Figure 23: Design of the feedthrough pressure test samples (left); Resin refrigerant dogbone samples (right).

Preliminary functional and performance tests of the AJP KET brick 3 have been successfully completed and the results are promising. The AJP KET brick will now be combined with the feedthrough KET brick 2 to test the electrical connection and sensor/heater functional and performance tests will be performed. Design of the AM test sample is shown in Figure 24.



## Figure 24: Design of the AM test sample for the AJP KET brick combined with the feedthrough KET brick.

Procurement of COTS components, KET brick 4, is ongoing and the design and test of their clamping interface to the pipe segment is done. The next step consists in combining the COTS KET brick with the feedthrough KET brick and test the correct functioning of the system and use the result to benchmark performance test done on AJP KET brick.

Regarding the EH KET brick, robustness and mechanical tests under liquid and gaseous  $CO_2$  flow for the whole operational temperature and pressure ranges will be performed along with electrical power generation and durability test. Based on the obtained results, an EH geometry will be selected and its design will be optimized and integrated to the KET brick 1.

#### 4. CONCLUSION

After nine months project execution dedicated to the specification of the requirements, the definition of the validation philosophy and the development of the KET bricks, the project now enters in the KET bricks validation phase.

This phase will allow defining the functional and performance limitations of the KET bricks. Those experimental results will allow finalizing the design of the use-cases and defining mitigation actions if some of the identified risks would materialize. Since the KET bricks were defined is such a way that they can apply to a variety of other use-cases, the results of the validation phase will be of utmost importance to define their scope of applicability to other applications.

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#### 5. REFERENCES

- Saudan H., Kiener L. Method for manufacturing a 3D electromechanical component having at least one embedded electrical conductor. European patent 3740382 B1, 2022-05-25.
- Chrysoula Manoli, Nicholas R. Hendricks, Jérôme Noël, Paolo Petagna, and Sébastien Lani. (2021). Smart Wall Pipes and ducts (SWaP). Public deliverable for the ATTRACT Final Conference.
- 3. https://attract-eu.com/about-attract-phase-2/
- Correia J. M., Nunes D. R., Esteves R. N., Rodrigues C., Pereira A. M., Ventura J. O. Energy Harvesting Under Harsh Conditions: Towards a Safe Oil & Gas Industry (Energy4Oil). Public deliverable for the ATTRACT Final Conference.