

# Design of an acoustic sensor for fission gas release characterization devoted to JHR environment measurements

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**Abstract** - Among numerous research projects devoted to the improvement of the nuclear fuel behaviour knowledge, the development of advanced instrumentation for in-pile experiments in Material Testing Reactor is of great interest. In the frame of JHR reactor, new requirements have arisen creating new constraints.

An acoustic method was tested with success during a first experiment called REMORA 3 in 2010 and 2011, and the results were used to differentiate helium and fission gas release kinetics under transient operating conditions. This experiment was leading at OSIRIS reactor (CEA Saclay, France). The maximal temperature during the irradiation test was about 150 °C. [1], [2]. We have developed thick film transducers produced by screen-printing process. They offered a wide range of possible application for the development of acoustic sensors and piezoelectric structure for harsh temperature environment measurements [3]. We proposed a screen-printed modified Bismuth Titanate piezoelectric element on alumina substrate allowing acoustic measurements [4] for JHR environment. In this paper we will focus on the mechanical design of the new sensor. This acoustic sensor is composed of an acoustic element for generation and detection of acoustic waves propagating into a cavity filled with gaz. We will detail the choice of piezoelectric materials, the thickness of the different layers, the cavity shapes, the electrical connections, the means of assembly of the different parts. Theoretical and experimental results will be given. All that point will be discussed in terms of acoustic sensor sensitivity versus dimensional constraints, in the case of a high temperature range working.

**Keywords** — Acoustic sensor, Gaz composition, Screen-printing, JHR

## I. Introduction

The IES laboratory (Montpellier, France), and more precisely the acoustics department, develop acoustic non-destructive testing methods for the nuclear field. And more specifically, a method to control the gas mixture in a fuel rod. Indeed, during a fission reaction, gaseous species such as Krypton (Kr) and Xeon (Xe) and helium (He) can be released and the knowledge of the quantity released can reveal

information on the state of the fuel, but also influence the thermodynamic characteristics of the fuel elements.

In 2010 [2], a device called CACP-1 (in the frame of the Remora 3 project) was tested for the first time in operational condition in the OSIRIS reactor formerly located in Paris Saclay (CEA Saclay, France), at a temperature of 150°C and a thermal neutron flux up to  $3.5 \times 10^{19}$  n.cm<sup>2</sup>. This first prototype used, as piezoelectric elements, a lead zirconate titanate (PZT) bulk piezoceramic commercialised by Meggit® (PZ27 Ferroperm) which had the advantage of having excellent piezoelectric properties ( $d_{33-PZT} = 370-420$  Pc/N [5]) compared to others piezoelectric ceramics. It made it interesting for characterisation in a gaseous medium, but had the disadvantage of having a limited Curie temperature of 350°C. However, this experiment allowed to follow the evolution of the gaseous composition of the fuel element with a precision of about 0.5 % of the molar fraction between helium and the gases coming from fission. The REMORA 3 experiment succeeded in its objective, namely to follow the evolution of the gas mixture in a fuel element from an acoustic measurement. But Improvement and optimization works have been undertaken with the following objectives:

- Increasing the maximum operating temperature of the sensor by using piezoelectric elements with a higher Curie temperature ( $T_{\text{curie-NBT}} = 680$  °C) such as Sodium Bismuth Titanate (NBT) in order to obtain a sensor with an operating temperature around 350°C.
- Direct integration of the piezoelectric elements on a ceramic substrate by screen printing process has been validated. This method avoids the use of metal soldering, which causes many technological issues such as the non-parallelism, the metal migration in the piezoelectric elements as well as the limitation of the maximum operating temperature.

Between 2011 and 2018 research has been conducted to produce NBT elements by screen printing on alumina substrates [4], [5]. Indeed, the NBT marketed by the company Meggitt® (trade name PZ46) is no longer available, it was necessary to

formulate our own NBT. The NBT used until now is manufactured using the hydrothermal method followed by a crystallisation process. The manufacturing process is carried out by the company *é-Ma*. After various tests, the NBT formulated and screen printed in our laboratory has reached performances close to PZ46 of Meggitt® ( $d_{33-NBT-ies} = 14.1$  Pc/N VS  $d_{33-PZ46 Bulk} = 20$  Pc/N [5]). Piezoelectrical elements has been screen-printed elements with a thickness of 40 micrometers of NBT ceramics. These devices have shown encouraging results with the generation of acoustic wave in a gas mixture of helium at 48 to 61 Bar [6], which can eventually be exploited for the estimation of the gas composition. However, this work has only focused on the design of the "active" part of the device, more precisely the piezoelectric element.

The work that I will present in this conference publication will serve to present the technological choices that have been retained in order to realise the acoustic device as a whole. Namely, the realisation of the piezoelectric element on a ceramic substrate, but also the realisation of the acoustic cavity. In a first long part named materials and methods, I will briefly present the principle of working of the device, and the choice of the materials composing the device by detailing its manufacturing process. Then I will present the results of measurements of the impedance on the piezoelectric elements and the tests in a liquid of the device.

## II. Material and method

In this part, material and method will be presented in a first time the techniques of manufacture employed for the realization of the piezoelectric elements. In a second and third part, I will detail the design of the sensor as well as the choices made for the materials which constitute the sensor as a whole. Finally, a instrumentation part will describe the material used for the measurements and tests.

### A. Fabrication of piezoelectric elements by screen printing

The fabrication of piezoelectric element is done by screen printing deposition, of a piezoelectric ink. The first step is to formulate this ink that will be deposited by screen printing. This mixture is composed of crystallized NBT powder, mixed with a binder agent in order to obtain a liquid composition. The mixture is passed through a roll mill machine to obtain on the first hand a best mix as possible and on the second-hand particles size reduction of the crystallized NBT.

Then a succession of steps of deposition by screen printing allows to deposit successively a layer of electrodes then the multiple layers of NBT ink on an alumina substrate. Between each deposition step, a drying step is carried out in an oven in order to eliminate a part of the organic binder contained in the ink. On Figure 1.1 is represented a picture of a screen-printed

element before the baking step and below a picture allowing to better realize the composition of an electrode-NBT-electrode stack (Figure 1.3). These stacks are realized in a grouped way on 2 inches square substrates allowing to realize up to 8 elements simultaneously.

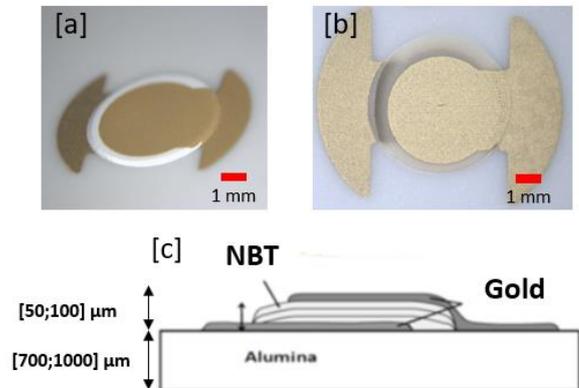


Figure 1 : (a) Piezoelectric element before sintering (b) Piezoelectric element after sintering (c) Cut view of the sensor showing the stacking of gold-NBT-gold

Once the different layers are stacked, an isostatic pressing step in water is performed. The samples are hermetically sealed in a plastic package under vacuum, then pressed at pressures higher than 1500 Bars. This step densifies the material by reducing the porosity of the NBT, which allows to improve the values of  $d_{33}$  and dielectric permittivity compared to the same element not pressed [7]. After pressing the stack, it is fired in a natural convection oven at 850°C, in order to sinter the powder and give the final microstructure. This sintering step can be preceded by a long firing step at a lower temperature (500°C) in order to eliminate the traces of organic solvents present in the binder. On the -Figure 1.2, we have the final form of the piezoceramic element directly printed on the alumina.

The thickness of the piezoelectric element and the substrate has an influence on the resonance frequency of the piezoelectric element which emits the acoustic wave, a mathematical model called KLM [2] makes it possible to estimate this value according to the thicknesses and the acoustic impedances of the substrate's materials and the piezoelectric ceramics. The thickness of the NBT layers produced by screen printing after firing varies from 50 to 100 micrometers and the thickness of the substrate varies between 0.7 and 1 mm. The choice of the thickness depends partly on the desired resonance frequency for the device, which should be between 3 and 7 MHz for acoustic characterization in a gaseous medium [1]. This aspect will be developed in the part III.A of the paper.

### B. Acoustic measurements and design of the device

Before addressing the design and manufacture of the sensor itself, it is necessary to explain in detail the operation of the device. We can distinguish two parts to this device, the first is its active part, namely the piezoelectric element screen printed on alumina and the second is the acoustic cavity which is a cylindrical volume filled with gaz. The acoustic waves

propagate in this cavity and realize multiple reflections on the reflector. This phenomenon is illustrated on Figure 2 by the blue arrows on the two different cavity designs.

By knowing the length of the cavity ( $e$ ), and the time of flight of the acoustic wave ( $\Delta t$ ), it is possible to determine the celerity of the acoustic wave ( $c$ ) by the following equation:

$$c = \frac{2e}{\Delta t} \quad (1)$$

The celerity of an acoustic wave in a gas is related to the thermodynamic parameters of this one, namely its pressure and its temperature, but also to its molar mass. Thus, previous work [2] allowed to link by an analytical model the velocity of the acoustic wave to the ratio of quantity between the gas produced during the fission reaction (Xeon and Krypton) and the helium initially present in the instrumented fuel rod. The devices will be integrated in an instrumented fuel rod, more specifically in a hermetic cavity directly connected to the stack of fuel pellets. The devices have to fit in a volume equivalent to cylinder of 15 mm of diameter and 15 mm of high.

On Figure 2, are represented in a cross-sectional view of the two designs of sensor references. They are both cylindrical in shape with a diameter not exceeding 15 mm and 15 mm in height.

What distinguish the two designs are the fixing methods between the sensor body, the reflector and the alumina pellet on which the piezoelectric element is screen printed.

The first design consists of a hollowed-out cylinder on which the reflector and the disk and the piezoelectric element are glued with alumina cement (not showed on the figure). The second design uses a screw and nut system between which the reflector and the piezoelectric element are tightened.

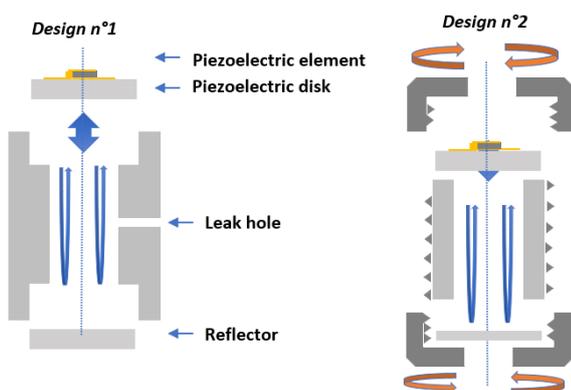


Figure 2 : (left) first design of the acoustic device (right) second design of the acoustic device

In the following part, I will detail the environmental conditions of the device and then the materials that have been selected for the manufacturing of the device.

### C. Technological and material choices for the sensors

In this part I will detail the different materials and shaping techniques used for the realisation of the device, justifying the

technological choices in relation to the technical constraints which are:

- The device has to resist to temperatures of at least 350°C [5]
- The elements must resist to radiative neutrons fluxes, the values of neutron and gamma fluxes are not well defined. But in 2019, NBT screen printed on alumina have been tested with a total dose of  $1.75 \times 10^{17}$  n/cm<sup>2</sup> (sum of thermal, epithermal and fast) with no apparent damage and critical evolution of its properties [8].

For the transducer, several piezoelectric materials have been investigated among the ceramics of Aurivillius structure group [5]. These ceramics belong to the family of "hard ceramics", with an average curie temperature above 600°C. Thus, two types of ceramics were tested, namely Bismuth titanate (BIT) Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> and sodium Bismuth titanate (NBT) Na<sub>0.5</sub>Bi<sub>4.5</sub>Ti<sub>4</sub>O<sub>15</sub>. Moreover, it is important to specify that the performances of these piezoelectric materials depend largely on the choice of doping elements. And the method of shaping the piezoelectric elements which is for us screen printing on alumina substrate. The choice of NBT was motivated by the fact that it was the best compromise between a Curie temperature higher than 350°C and a weak influence to neutron fluxes (Thermal and fast). The d<sub>33</sub> performances sufficient for our applications (d<sub>33-BIT-ies</sub> = 4 Pc/N VS d<sub>33-NBT-ies</sub> = 14.1 Pc/N) for ceramics obtained by screen printing [4].

Regarding the electrodes, the choice was made for materials such as gold or platinum. Indeed, the use of silver as an electrode can create problems of electro migration. The passage of metallic silver in the dielectric layer could lead to breakdown phenomena [9]. In order to limit not only this problem and to resist to the different environmental conditions the choice was made to use gold electrodes deposited by screen printing. Screen printing allows to realize layers of 10 micrometers thickness more easily than sputtering method for instance. The reference used comes from the Ferro corporation (Reference: 8844-GI), and has a firing temperature of 850°C and a maximum operating temperature of 600°C.

For the realization of the cavity of cylindrical form of the two designs visible on the Figure 2, the choice fell on alumina and glass-ceramic. The glass-ceramic used is marketed by Corning inc ®, it has as main interest a maximum operating temperature of 1000°C, with no mechanical loading and the possibility of being machined with conventional means. Concerning the substrates of deposits, alumina or MACOR® can be retained, indeed they have both temperatures of use well higher than 350°C [10]. The cutting of the transducers in the form of pellets being ensured by a UV laser (Figure 3), it is completely possible to use the two types of materials. Moreover, the literature studies [10], have not indicated major problems as long as the use of MACOR® as a substrate of deposit.

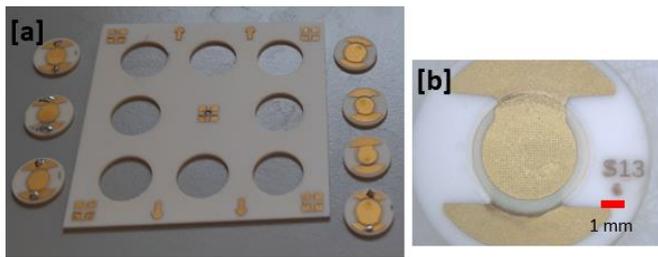


Figure 3 :[a] Alumina substrate with 8 elements cutting by UV LASER [B] Individual piezoelectric element ready to be integrated on a cavity

For the bonding of the elements, the choice was made to use alumina cements marketed by the brand Final material (reference: Final 940HT). This cement can be used at temperatures of more than 1500°C. A summary of the respective properties of alumina MACOR® and alumina cement are presented in Table 1. An important parameter to take into account is the difference of thermal coefficient expansion (TCE) between the different materials that form the sensor, the MACOR®, the alumina, the alumina cement and the NBT. In order to prevent the generation of crack and delamination between NBT ceramic and alumina, the mismatch of TCE between all elements have to be as low as possible.

Table 1 : TCE and maximum operating temperature of the different sensor elements

	Alumina (96%)	Alumina cement	MACOR®	NBT screen printed
TCE (K <sup>-1</sup> )	8.6E-6	7.2E-6	9E-6	7E-6
T <sub>max</sub> (K)	1700	1540	1000	600

T<sub>max</sub> indicates the maximum operating temperature of the elements. The TCE of elements is taken for a range of temperature between [25;300] °C. Data coming from their data sheet

As we can see on Table 1, the materials were chosen in order to limit these differences in TCE between the different parts of the sensor. On Figure 4, a photo represents respectively the design n°1 and n°2 assembled and ready to be tested.



Figure 4 : [a] Design n°1 before and after the sealing step with alumina cement [b] Design n°2 of the screwed version before and after its assembly

#### D. Instrumental bench

Before assembling the piezoelectric elements in the cavity, the devices are polarized in order to give the NBT piezoceramic their ferroelectric properties. The elements are polarized at electric fields up to 150 kV/cm in a silicone oil bath. Once the devices are polarized, an impedance measurement as a function of frequency is performed on each device, this measurement is performed on a Keysight E4990 impedance analyzer. Figure 5 shows the impedance analyzed with the sample holder under electrical contact tips to perform the measurements.

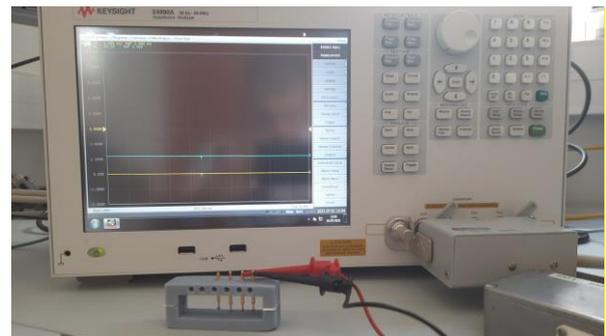


Figure 5 : Instrumentation of impedance meter Keysight E4990

Then tests are carried out in order to check the presence of reflected echoes in the devices n°1 and n°2 by filling them with ethanol. The objective is to ensure the proper functioning of the piezoelectric elements and the good parallelism of the cavities. For this, the transducers will be powered with an Olympus pulser/receiver model 5072R coupled to an oscilloscope allowing to realize an acquisition.

### III. Model and Results

#### A. Estimation of the optimal dimension by KLM modelling

As previously stated, the KLM model allows to calculate an equivalent electrical impedance from the piezoelectric parameters of the materials (the mechanical quality factor, the coupling factor, the dielectric permittivity of the material and the dielectric losses) and from the properties of the layers (mainly the thickness, the density and the acoustic velocity). In the framework of our research, this model allows us to estimate the resonant frequencies of screen-printed piezoelectric transducer on alumina as a function of the thickness of the screen printed NBT and the thickness of the alumina substrate.

The screen-printing manufacturing process limits the thickness of the elements to 100 microns of post-fired ceramic. Thus, the KLM model, allowed to test the following conditions, namely three different thicknesses of alumina substrates (508 µm and 762 µm and 1000 µm) and two different thicknesses of NBT ceramics (50 µm and 100 µm). The objective is to understand the influence of these two parameters on the resonance frequency of the screen-printed element.

For this purpose, 4 combinations of substrates thickness have been tested, namely:

- Alumina: 508  $\mu\text{m}$  and NBT: 50  $\mu\text{m}$
- Alumina: 762  $\mu\text{m}$  and NBT: 50  $\mu\text{m}$
- Alumina: 762  $\mu\text{m}$  and NBT: 100  $\mu\text{m}$
- Alumina: 1000  $\mu\text{m}$  and NBT: 100  $\mu\text{m}$

The results are presented just below (Figure 6).

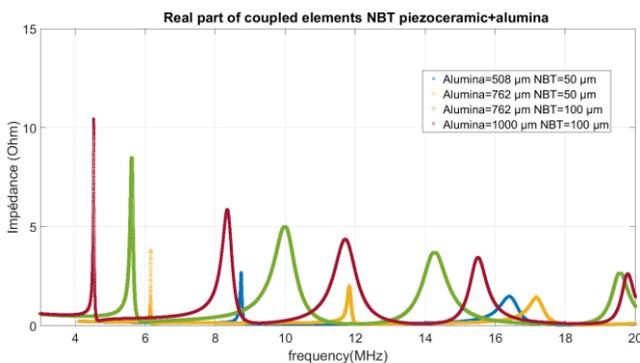


Figure 6 : Estimated real part of electrical impedance Vs frequency for various Alumina-NBT thickness

Two trends emerge from these simulations, the first is that for the same frequency thickness of NBT the more the thickness of the alumina substrate is important, the more the fundamental shifts towards low frequencies. As an example, for a thickness of 1000  $\mu\text{m}$  of alumina and 100  $\mu\text{m}$  of NBT the fundamental is 4.5 MHz. Similarly, the greater the thickness of NBT, the more the fundamental frequency shifts to the low frequency.

It is then possible to estimate a couple of thickness of alumina substrate and NBT "optimal dimension" for our applications in order to obtain a fundamental frequency between 4 and 7 MHz. So substrates of thickness 762  $\mu\text{m}$  or 1000  $\mu\text{m}$  seem quite indicated, with NBT elements whose thickness will be between 50  $\mu\text{m}$  and 100  $\mu\text{m}$ .

### B. Impedance of the NBT Layers

As previously stated, the transducers must resonate at a frequency between 4 and 7 MHz. Experimentally, several samples have been manufactured, their characteristics are given in the Table 2.

Table 2 : Characteristics of various sample tested

ID	Thickness alumina ( $\mu\text{m}$ )	Average Thickness of NBT ( $\mu\text{m}$ )
S4	508	50
S12	762	100

The objective of these measurements is to validate, the trends of the model. More specifically, the increase and decrease of the frequencies of the fundamental by changing the thickness of the substrate and NBT. The impedance spectra between 1 MHz and 20 MHz of two piezoelectric elements, namely S4 and S12,

are shown in Figure 7.

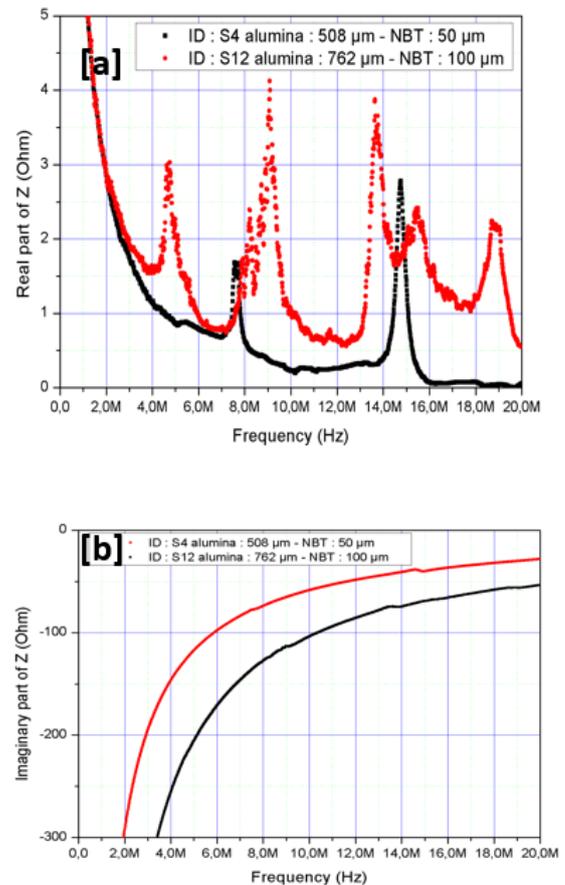


Figure 7 : Electrical impedance real part [a] and imaginary part [b] of S4 and S12 sample

The impedances confirm the trend of an increase in the resonant frequency of the fundamental by jointly increasing the thickness of the alumina substrate and the NBT ceramic. Thus, the couple 762 micrometers of alumina and 100 micrometers of NBT is validated for the fabrication of devices because its fundamental is about 4 MHz. However, it remains differences in amplitudes and quality factors concerning the resonances. This can be due to several parameters, namely:

- The piezoelectric parameters of the NBT used for the simulation do not exactly match the NBT used for the S4 and S12. Indeed, parameters are taken from the publication of O.Gatsa and other [4]. The reasons of differences is the addition of chemical elements in our NBT slightly different than the NBT tested by O.Gatsa and other.
- Moreover, the values of the impedances depend on other parameters which linked on the process of manufacture of the elements. For instance, the time and the duration of the sintering which will define the state of the ceramic microstructure. But also, the presence or not of a densification step during the manufacturing process.

### C. Acoustic test with ethanol

Impedance testing showed that a thickness of 762  $\mu\text{m}$  alumina and 100  $\mu\text{m}$  NBT is a potentially satisfactory combination for gas composition study. The devices will be tested in a non-conductive liquid such as ethanol with two cavity designs, the cavity sealed by alumina cement bounding (design n°1) and the screwed cavity (design n°2). The objectives of these measurements are the following:

- To verify the proper functioning of the device by observing the emission of echoes. Indeed, if in a liquid medium the device does not manage to emit echoes, then it will surely not be able to emit and measure any echoes in a gas. Because gases are less dense than liquids, the acoustic waves for frequencies between 1 and 20 MHz are more attenuated than liquid middle.
- To validate the good functioning of the acoustic cavities. More specifically, the flatness between the piezoelectric element and the reflector must be the best as possible. It is possible to observe these problems on an echogram, with in particular the appearance of non-periodic echoes and a sudden variation of reflected echoes.

In Figure 8 below, the devices are mounted with a functional piezoelectric element. For the first design the reflector at the bottom of the device is glued to the bottom with alumina cement (Figure 4 [a]) and the active element is only maintained against the cavity. The absence of glue allows to test several elements per cavity, but also simplifies the filling of the cavity with ethanol. The second device is screwed on. The devices are electrically connected using single stranded wires connected with Sn/Pb solder. Nevertheless, connection method is not representative of the final device.

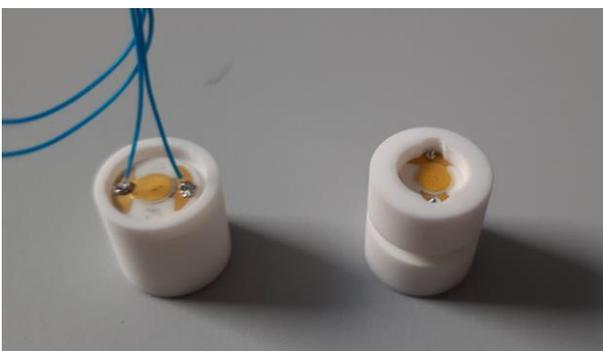


Figure 8 : Acoustics elements ready to be tested

In Figure 9, are presented the results of several measurements made successively with the design n°1 and n°2 using the same piezoelectric transducer. 7 measurements were made, 4 on design n°1 and 3 on design n°2. Between each measurement the device is dismantled and purged of ethanol and then fill a new

time in order to validate the reproducibility of the measurements.

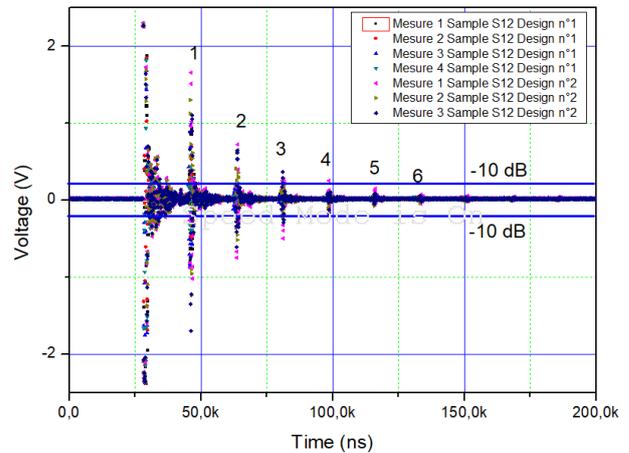


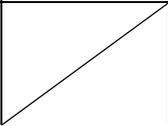
Figure 9 : Acoustics echoes measuring in Pulse/echo mode for designs n°1 and n°2

The measurements show several things: the first thing is a series of several reflected echoes, whose amplitude decreases exponentially in agreement with the acoustic models. The attenuation mechanism is linked to the phenomena of propagation and reflection. When the acoustic wave propagates in a medium, its energy decreases because of the transmission of acoustic energy in the material medium to set in motion the particles constituting this medium.

Thus, a medium such as a gas even at pressures of several hundred bars will have a density less important than a liquid at an equal pressure and temperature. And thus, the liquid will be far less attenuating because requiring less energy for its setting in movement. Furthermore, gas and liquid being dispersive media, the losses also depend on the frequency of the acoustic signal. Various model existing so as to estimate the acoustic attenuation in a liquid or gas mixture, but not presented in this paper.

The second type of notable losses are those by reflection, the acoustic energy is lost during each interaction with a solid medium. They can be modelled as a function of the acoustic impedances (Table 3 (2)) specific to the two media, with the formula Table 3 (3) representing the share of energy reflected during each reflection. For example, for the acoustic impedance of alumina ( $Z_{\text{Al}_2\text{O}_3} = 3.89 \times 10^7 \text{ Pa.m. s}^{-1}$ ) and that of ethanol ( $Z_{\text{C}_2\text{H}_5\text{OH}} = 9.16 \times 10^5 \text{ Pa.m. s}^{-1}$ ) the reflected acoustic energy corresponds to nearly 95% of the initial energy. While for a gas such as helium at 25 ° C and 200 Bar, the acoustic impedance is much lower than alumina ( $Z_{\text{He}} = 600 \text{ Pa.m. s}^{-1}$ ), so that the reflected energy is reflected to more than 99.99%.

Table 3 : Definition of acoustic impedance and Rate of reflected energy

<b>Acoustic impedance</b>	$Z = \rho c$	Pa.m.s <sup>-1</sup>	(2)
<b>Rate of acoustic energy reflected</b>	$R = \frac{Z_2 - Z_1}{Z_2 + Z_1}$		(3)

$\rho$  and  $c$  represent respectively the volumic mass and the celerity of sound of the material, and  $Z_2$  and  $Z_1$  represent respectively the acoustic impedance of alumina and gaz (or liquid)

Thus, in ethanol and for the fourth echo, the acoustic wave has made nearly 7 reflections on the alumina which corresponds to  $0.95^7 = 0.69$  of the initial energy at the end of the fourth echo just by the reflection of the waves. And on the fourth echo almost 70% (-10 dB) of the energy is lost compared to the initial signal.

Thus, it is for the moment impossible to validate at least analytically the functioning of the device in gas medium. Because the types of acoustic losses are different and the acoustic attenuation complex to model in a gas. Nevertheless, this experiment has permitted to validate the emission of acoustic wave in a liquid medium. The first experiments in gaz medium are planned for the third quarter of 2021.

Besides, the time difference between each echo of the measurements on S12 is an average of  $17.47 \mu\text{s} \pm 0.37$ . The speed of acoustic wave estimated for this time of flight is 1144 m/s for a cavity of 10 mm height (equation 1). This is within the measurement interval of  $1142.9 \text{ m/s} \pm 1$  measured by Erich C Meister [11] for pure ethanol at 25°C by another acoustic method. Finally, the echogram does not show any parasite echoes between the different reflections, which indicates a satisfactory parallelism for our applications.

#### IV. Conclusions and perspectives

This work showed the study, the realization and the test of an innovative acoustic sensor usable in an experimental fuel rod of the future JHR. The choices concerning the materials of the sensor are respectively, NBT for the piezoelectric ceramic which is deposited by serigraphy on alumina. Glass-ceramic such as MACOR® is used for the fabrication of the acoustic cavity and the reflector. These choices were motivated by the low thermal expansion of these materials taken individually, their resistance to the radiative fluxes and their maximum operating temperature well higher than 300°C

In parallel to the technological aspects, with the help of an analytical model, the choice of the thickness of the substrate and the piezoelectric element were fixed at respectively a thickness of 762 micrometers of alumina and 100 micrometers of NBT. These choices have been confirmed by the experimental results which showed the presence of an acoustic wave reflected in the cavity filled by ethanol. The shape of the signal and the time between each echo seem coherent in comparison to the state of the art.

Next results will concern, the tests in a gas to validate the sensitivity of the device immersed in a gas mixture of helium. The device must also be tested at a temperature of at least 300°C to verify that its piezoelectric performance remains stable despite the temperature.

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