DECLIC, SOON TWO YEARS OF SUCCESSFUL OPERATIONS

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DECLIC is a multi-user facility to investigate critical fluids behaviour and directional solidification of transparent alloys.
As part of a joint NASA/CNES research program, the facility was launched with 17-A Shuttle flight (August 2009) and has been operated onboard the ISS since October 2009 : when the IAC 2011 takes place, the instrument will have performed two years of operations, monitored from the CADMOS (CNES, France).

The material samples to be studied are accommodated in dedicated inserts which can be sequencially inserted into the instrument. The three developed inserts have been tested so far.

The DSI (Directional Solidification Insert) is dedicated to the study of the solidification of succinonitrile based alloys, analogue of metallic ones.
The flight sequences are over and have shown unprecedented and unique 3D observations. The results will also be used as benchmark data concerning the solidification interface pattern characteristics.
This insert was brought back with the ULF-6 Shuttle flight (May 2011) for a post-flight sequence and refurbishment.

The HTI (High Temperature Insert) is dedicated to the study of pure water as a critical fluid.
It has produced fundamental results after having faced a perturbating thermal gradient that was finally solved through software modifications [1]. A first water critical temperature value has been approached for the first time in microgravity and interesting turbidity measurements have been performed.
The insert was flown back with the ULF-5 Shuttle flight (March 2011) and is being refurbished.

The ALI insert is dedicated to the study of SF6 (sulphur hexafluoride) as a near-ambient temperature critical fluid.
Its commissioning was performed in March 2011 and three experimental sequences have already been performed. The program covers a whole characterization of SF6, ranging from thermodynamic quantities measurements (thermal diffusivity, heat capacity and turbidity near the critical point...) to boiling effects studies thanks to a cell fitted with a heating window.

The mid-term program is made of existing insert utilization (ALI) and inserts refurbishments and re-utilization (HTI and DSI), while the long-term program plans to develop new inserts.

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I. INTRODUCTION

As DECLIC is close to end its second year of operation on orbit, the three developed inserts have already given interesting results, showing the performances of the payload and increasing the interest of the community for a follow-on of the program.

The aim of this paper mainly is to give a status of the operations and to look at what is expected next.

The first part of the paper gives an overview on the science program, the payload, the inserts, and the ground segment.

Until now, both the HTI and DSI sequences are over, the two inserts have been flown back to the ground, and two ALI operational sequences have been run. The synthesis of the operations is presented in the second part of the paper, as well as some preliminary results for the three inserts.

The mid-term and long-term programs, made of use of existing inserts, inserts refurbishments, and new inserts development, are detailed in the last parts of the paper.

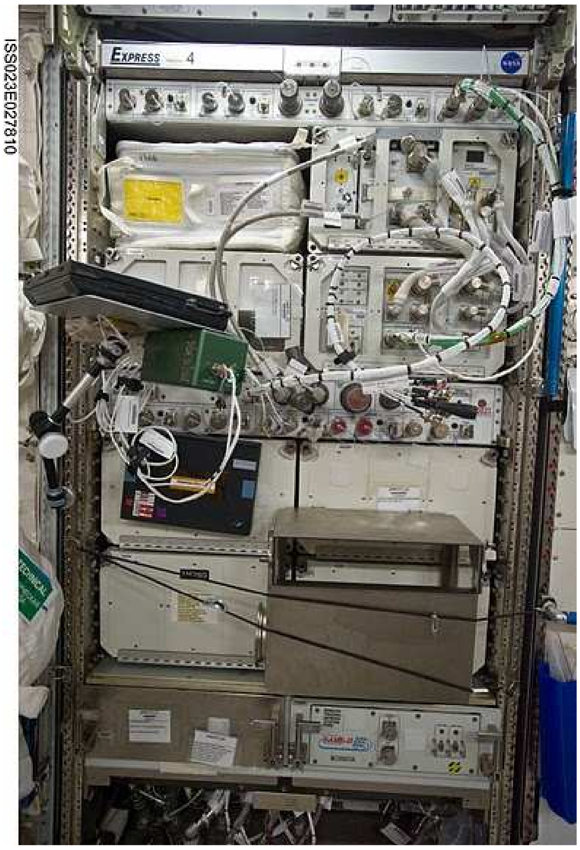


Fig. I: The DECLIC payload fitted in the EXPRESS RACK 4 of the ISS. The two DECLIC lockers are located in the top right quarter of the rack (photo NASA).

II. PAYLOAD, INSERTS AND GROUND SEGMENT

This is a brief overview as those items have already been presented in details during past IACs [1], [2].

The DECLIC instrument is accommodated in two Single Stowage Lockers (SSL), called the EXperiment Locker (EXL) and the ELectronic Locker (ELL).

The general architecture is given in Fig. II.

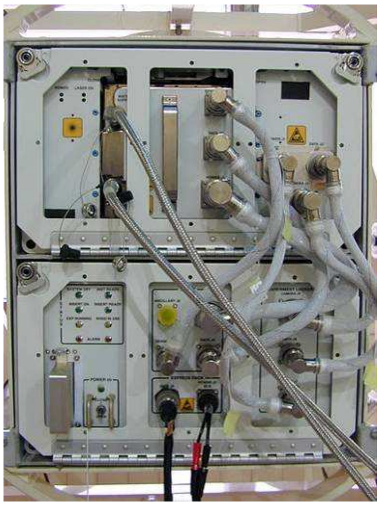


Fig. II: The DECLIC lockers. The ELL is at the bottom while the EXL is at the top and houses the insert. The electrical connections between both component and the water cooling connections (grey “pipes”) to the EXPRESS RACK are also visible. (Photo CNES).

The ELL houses all the power, data handling and high accuracy thermal regulation electronics, and manages the whole system and the scientific experiment execution. It is the interface, for signal and power, with the EXPRESS RACK.

The EXL hosts the optical bench, providing various diagnostics like wide or narrow field of view, grid shadowing, light scattering, interferometers, light transmission measurement etc...). The EXL also receives the experiment container which is called the insert.

Three inserts have been built so far, each of them being dedicated to a science domain:

- Phase transitions near the critical point at room temperature, critical fluids and boiling crisis are studied thanks to the ALI (Alice Like Insert).
- Properties of high temperature supercritical fluid like water are checked thanks to the HTI (High Temperature Insert).
- Microstructures dynamics during the solidification of model materials are studied thanks to the DSI (Directional Solidification Insert).

An insert accommodates a thermostat (ALI, HTI) or a pulling furnace (DSI), respectively containing a sample cell unit (SCU) or a cartridge with the material to be studied. It also contains most of the electronics associated with user dedicated sensors (temperature, pressure...).

The control centre for DECLIC is the CADMOS. The CADMOS is a User Support Operation Centre (USOC) located at the CNES (Centre National d'Etudes Spatiales) centre of Toulouse (France).

As the team is not 24/7 on console, the payload's Health and Status data is also monitored at the POIC (NASA's Marshall Space Flight Center, Huntsville, USA) where Ground Commands Procedures allow to quickly react to non nominal situations.

The real time data is made available to the scientists via a webserver. In addition, an archiving system (to which the scientists can also connect via a web browser) allows to store the exhaustive reference data, which is retrieved via Removable Hard Disks, returned to the ground when full.

The operations are organized in insert dedicated sequences which typically last 3 weeks and are separated by several weeks (3 at least typically). That kind of arrangement allows the scientists to perform some data processing of a given sequence in order to tune the following sequence program.

V. RESULTS

V.I Operational Synthesis and anomalies

As of July 2011, the instrument has seen 14 flight sequences, leading to the following statistics :

- More than 8000 issued commands
- 5800 operating hours
- 1 TB of generated data

Since the beginning of the operations, DECLIC has experienced several kinds of anomalies. Those having had impact on DECLIC performances or inducing strong operational constraints are listed below.

V.I.I HTI Thermal Gradient

As stated in [1], an unexpected thermal gradient was seen during the first HTI flight sequences. This thermal gradient was suspected to be linked to a non linear phenomenon occurring in one of the two Peltier elements involved as sensors (Seebeck effect) in the regulation loops.

We were able to implement a workaround in order to put the thermal gradient within the cell back at expected values or so ; the faulty Peltier element was replaced, in the regulation loops, by a linear combination of two neighbour sensors.

During the post-flight sequence, the other Peltier element also failed.

As the HTI is being refurbished for the HTI-R program (see § VI.II), the Peltier elements have been retrieved and some cracks were find in the alumina plates that accommodates the thermoelectrical material ($FeSI_2$) legs. That certainly explains the observed phenomena but further investigation is needed to conclude.

V.I.II Reboots and Shutdowns

Like the preceding anomaly, this one was already stated in [1].

Since the beginning of the operations, the payload has experienced 7 events called shutdowns and reboots.

What is called a reboot is when the payload completely reboots by itself, while a shutdown is when the payload is found in a very low power consumption mode and that we cannot interact with it anymore.

Mainly because those kinds of events appeared with two different inserts (DSI and HTI) at very different steps of the sequences (science activities, data recording...), and also because nothing was found in the parameters evolutions, nor in the logs generated by the payload, the troubleshooting led us to propose two explanations : Single Event Processes (SEU, SEL), or micro cuts in the 28V EXPRESS RACK power line.

We have not concluded on the origin of the anomaly yet, but there hasn't be any occurrence since July 2010.

V.II DSI

The main objective of the present project is to identify and understand the physical phenomena which govern the dynamics of the microstructure selection in 3D directional solidification [3]. Acquisition of experimental benchmark data in diffusive mode (ISS experiments) is also necessary to provide the scientific community with a database; this is critical for the validation and development of reliable theoretical and numerical models. This database will be established as a function of the control parameters of the microstructure (solute concentration, thermal gradient, pulling rate) so that it is mandatory that these parameters are unambiguously controlled during one experiment. If we consider a pattern grown on ground (fig. III a), it is characterized by a very clear radial variation of size going from large cells in the centre to small cells and even smooth interface at the crucible border; this variation is due to convection that induces radial gradients in the microstructure control parameters along the interface [4]. In contradistinction with 3D ground patterns, the microgravity ones are very homogeneous as illustrated in fig. III : radial variation of size is no longer noticeable on the pattern grown in microgravity. In this last case, the width of the size histogram is then fully representative of the selection process (fig. III c).

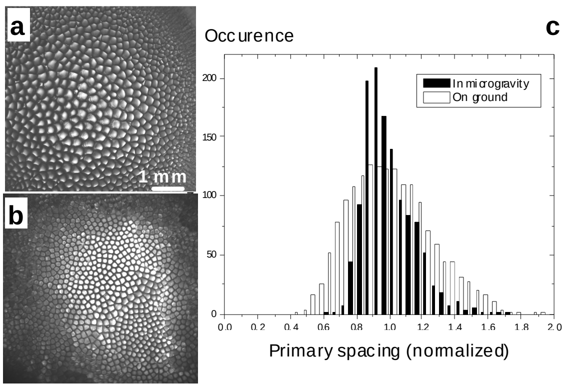


Figure III: Comparison between patterns grown
- a) on ground
(SCN - 0.1 wt%, VP = 10 µm/s, G = 17 K/cm)
- b) in microgravity
(SCN - 0.24 wt% Camphor, VP = 4 µm/s,

The solute concentration of the sample is adjusted on ground so that it can not be changed. Two sets of thermal conditions, associated to two values of thermal gradients, have been used. Long solidifications of the whole cartridge at a given pulling rate, as well as experiments with a velocity jump, have been performed. Long solidifications are dedicated to the exploration and characterization of the microstructure map as a function of growth parameters. Experiments with a velocity jump are of prime importance in order to understand how a quasi-stationary pattern responds to a variation of parameter, with which dynamics and by which mechanisms.

Quantitative characterization of the patterns obtained is still in progress but let us illustrate our work with the analysis of a long solidification at a constant pulling rate. The starting interface corresponds to $V_P = 0$ (no microstructure). After triggering of the pulling, morphological instability initiates by forming linear ridges along sub-boundaries that finally underline a rather complex array (fig. IV a). Between these defects, in interface areas that are still smooth, some poxes may be present: they can be described as circular ondulations of the interface. A quite uniform corrugation that corresponds to the initial visible wavelength of morphological instability then invades the interface (fig. IV b). The amplitude of all these interface modulations starts to increase, channels are forming but it is still difficult to identify cells. At this stage, the interface dynamics is extremely fast and pattern disorder high. There is then a progressive decrease of disorder and a clear pattern of cells is eventually reached (fig. IV c). The dynamics then clearly slows down and is limited to progressive size adjustment and array ordering (fig. IV d). All this process is quantitatively characterized, for example in term of primary spacing evolution, and the mechanisms of spacing adjustment are investigated.

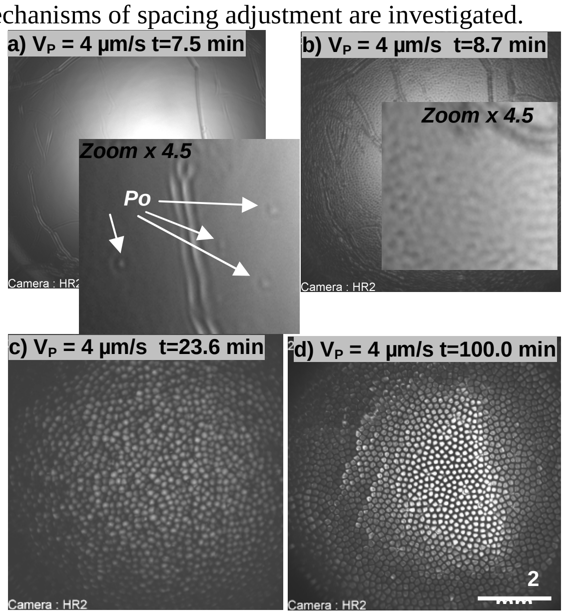


Figure IV: Patterns evolution as a function of time

Thanks to the combination of in situ observation and extended homogeneous patterns, it was also possible to obtain very unique and of major interest observations of some secondary instabilities such as multiplets or oscillating patterns. Analyses of these phenomena are also in progress.

V.III HTI

As the HTI was the first insert to be tested onboard DECLIC, few preliminary results have already been presented at IAC 2010 [1] and more detailed results will be presented during IAC 2011 in [5].

V.IV ALI

V.IV.I Hardware

The microgravity experimental program to investigate the boiling crisis uses the ALI insert. For that purpose, two optical pressurized cells integrate respective in-situ heater devices, i) as a form of massive flux meter appropriate for interferometry imaging of the bubble shape deformation during heating, and ii) as a form of a transparent resistive layer (~ 50 mm^2 area), appropriate for light transmission observation of the liquid film boiling.

Each cell is filled by SF_6 and the critical coordinates (temperature, pressure, density) of the gas-liquid critical point of SF_6 are T_c=318.737 K, (45.587 °C), p_c=3.73 MPa, and ρ_c=742.6 kg m^{-3}.

As the results presented in this paper are focused on the Direct Observation Cell (DOC), the design of this cell is reminded below. For more about the cells design, see [6].

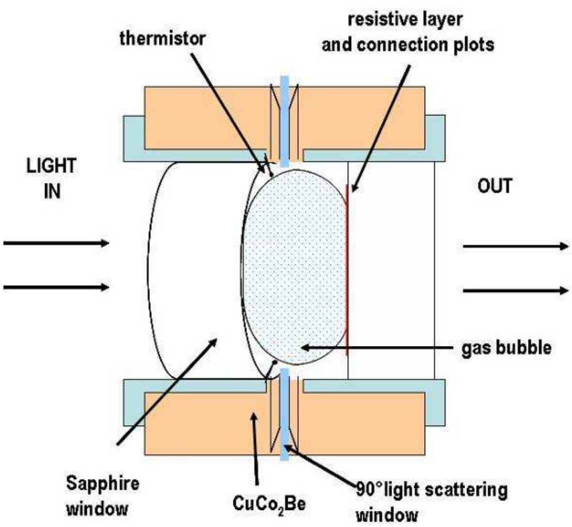


Fig. V Schematic cross section (not to scale) of the expected microgravity gas-liquid distribution in the Direct Observation Cell (DOC) dedicated to study the liquid film drying due to boiling phenomena, using a transparent resistive layer as a flat local heating source.

The fluid sample volume observed by light transmission corresponds to a cylindrical volume of D=10.6 mm inner diameter and inner thickness e=4.115 mm. Moreover, the direct observation cell also allows collecting the light scattered at small angles and at 90°. The total fluid volume of the cell is 0.463 cm^3 (including a dead volume mainly due to filling holes), corresponding to a total SF_6 mass of 0.353 g, leading to the filling mean density $\rho = \rho_c$+2% (to be refined).

Three small (250 µm bead diameter) thermistors (THERMOMETRICS B10, 10 kΩ resistance at 25 °C}) are located inside the fluid volume, so that three local temperatures are measured close to the gas-liquid interface in microgravity environment, and labeled R5, R6 and R7, respectively. The thermistors are visible on the picture of Fig. VI.

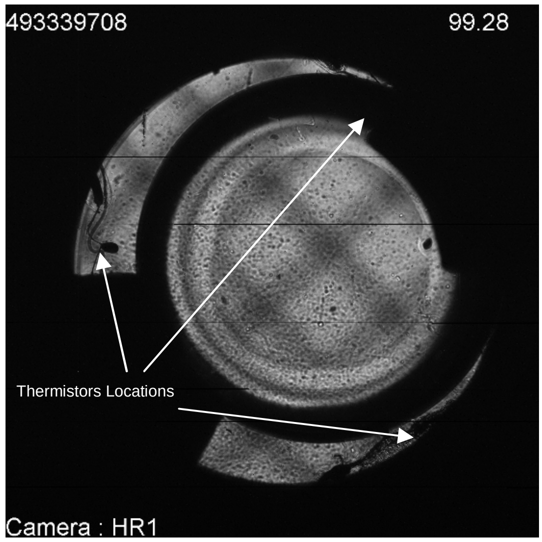


Fig. VI. Wide field and grid shadowscopy image of Direct Observation Cell (DOC) at equilibrium in the two-phase range (T~Tc-300mK)

V.IV.II Preliminary Results

Only the preliminary results obtained from the direct observation cell are presented in the following. These results are focused to i) the T_c-determination and phase separation process after the T_c-crossing down from a temperature quench of -100 µK depth, and ii) the relaxation of the boiling phenomena after heat pulses delivered by the transparent resistive heater.

Turbidity measurements and phase separation process are used to determine the DOC critical temperature with a resolution better than 50 µK. The Tc-crossing down is illustrated by the series of images given in Fig. VII which show the density changes from the initial (homogeneous) one-phase domain above T_c (a) to the (growing) two-phase domains when the temperature goes to below T_c due to a temperature quench of -100 µK depth. We note that the two-phase evolution is here observed over a period of 34 hours following the temperature quench, thanks to the high temperature stability (±15 µK) of the regulation system. We also note that the final equilibrium state where a single vapor bubble is surrounded by the liquid is not still reached.

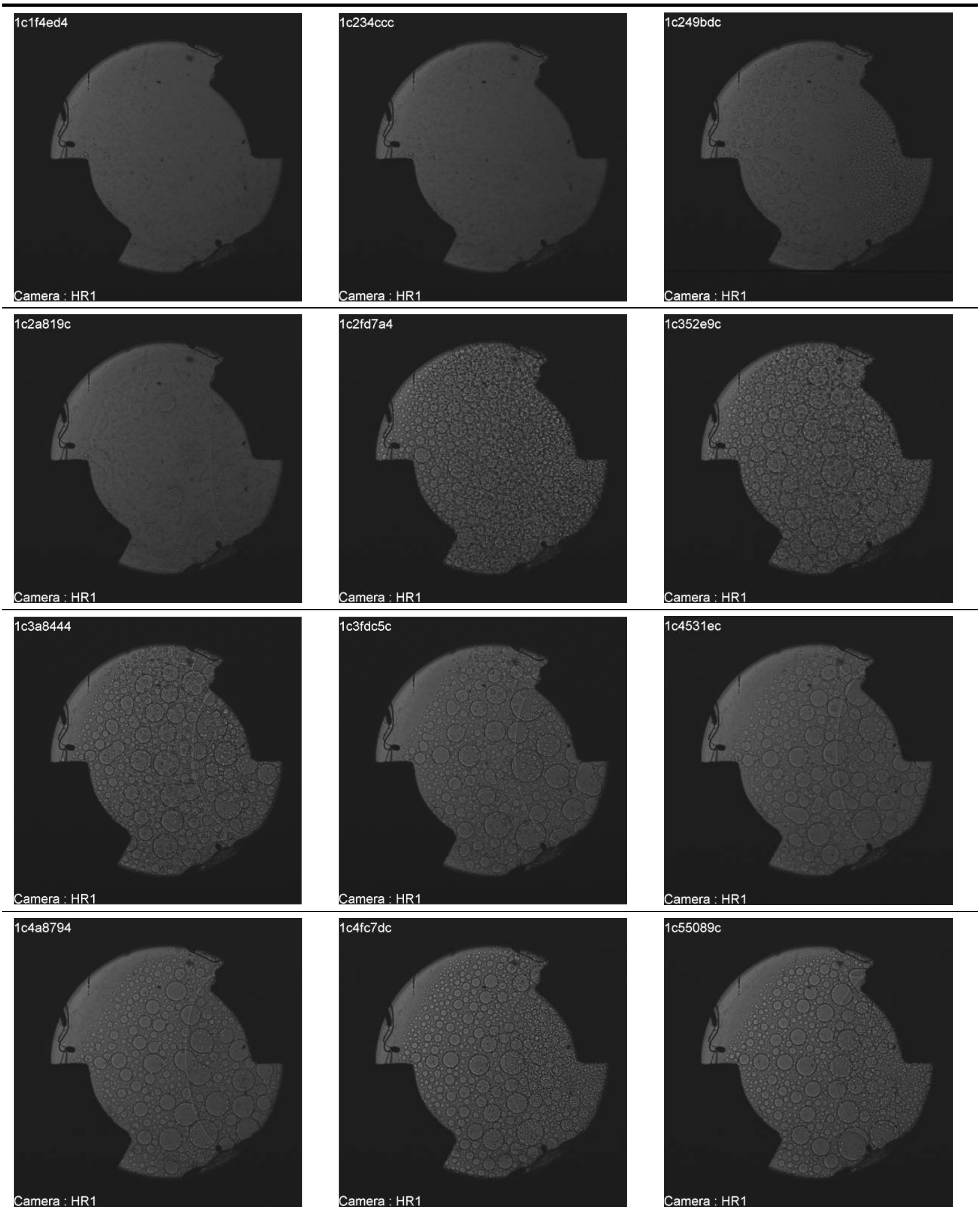


Fig VII. Wide field images of Direct Observation Cell (DOC) for a Tc-crossing down when the temperature change from above T_c- to below T_c is due to a temperature quench of -100 µK depth. Image (top-left) corresponds to the initial (homogeneous) one-phase domain above T_c. The following images (from right to left and top to bottom) illustrate the density changes due to the evolution of the (growing) two-phase domains. About 34 h total time between (top-left) and (right-bottom) images.

As shown in Fig. [V], the cell is fitted with a transparent resistive layer.
By using this layer, heating of the two-phase domain, boiling phenomena and drying of this liquid film can be analyzed, through in situ measurements of the temperature changes (see below Fig. VIII) and direct observations of the boiling and drying processes during heating pulses and their related relaxation after heating stop (see below Fig. IX).

We recall that the resistive layer is made from a Sn alloy oxide [7], 8 mm diameter and 200 nm thickness. The value of its resistance (106 Ω) is temperature independent ($dR/dT \leq 7$ m Ω K^{-1}) in the temperature range of present interest. The heater resistance, covering a typical area of 50 mm^2, is supplied at constant adjustable voltage using two gold plate layers which induce two non-transparent regions in the fluid volume (see the corresponding black areas in Fig. VI). The Declic instrument allows injecting an electrical power covering the typical range 0-3.4 mW, of 12-bits resolution. The heat power per unit area which is dissipated by Joule effect can then be controlled in the range 17 mW m^{-2} to 68 W m^{-2}, i.e., a range presently not available in (generally non-transparent) technical devices of same area (and significant larger thickness). The realistic variation of the heat flux dissipated into the fluid can be estimated as a function of $T-T_c$, thanks to the effective heater design and the precise knowledge of the singular thermal properties of SF_6. At $T=T_c+10$ mK for example, 90% of the electrical power will be dissipated into the fluid. In addition, the heating period (from a minimum time of 43 ms) can be adjusted (from time increments of 43 ms) in a synchronized manner to the fluid temperature measurements and images.

Fig. VIII and IX are the first illustrations of the high-level performances of such heating device used in microgravity conditions.

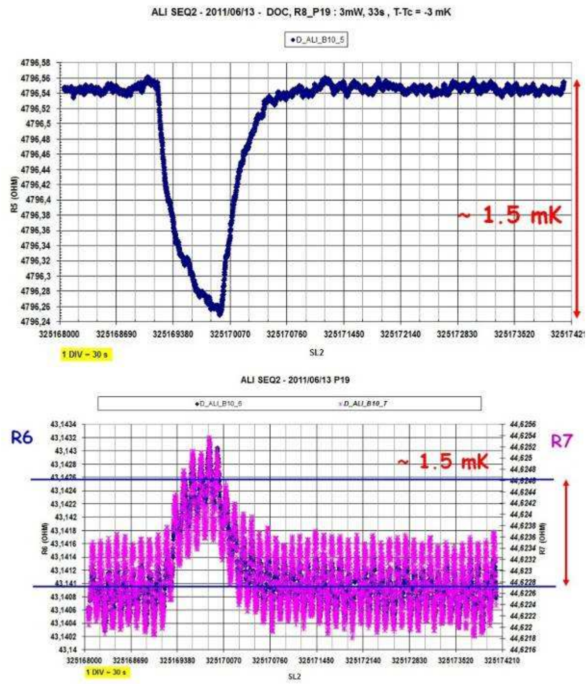


Figure VIII: (a) Measured resistance change for the R5 thermistor during the electrical pulse (3 mW power, 33 s duration) in the two phase domain ($T=T_c$-3 mK); related temperature change directly measured by R6 and R7 thermistors.

Fig. VIII(a) gives the R5 thermistor response from a heat pulse of 3 mW, 33 s duration, performed 3 mK below the critical temperature, while Fig. VIII(b) gives the related temperature changes measured by the two other in situ thermistors R6 and R7. These results illustrate the resolution of the temperature measurements from the B10 sensors, which is of the order of 100 µK, typically. We note that the temperature increase of order of 1.5 mK remains lower than the temperature distance to T_c, probing that the thermal energy supplied to the fluid by the heat pulse does not change the two-phase nature of the cell. Such a resolution in temperature control and measurements is essential working at a few mK from T_c.

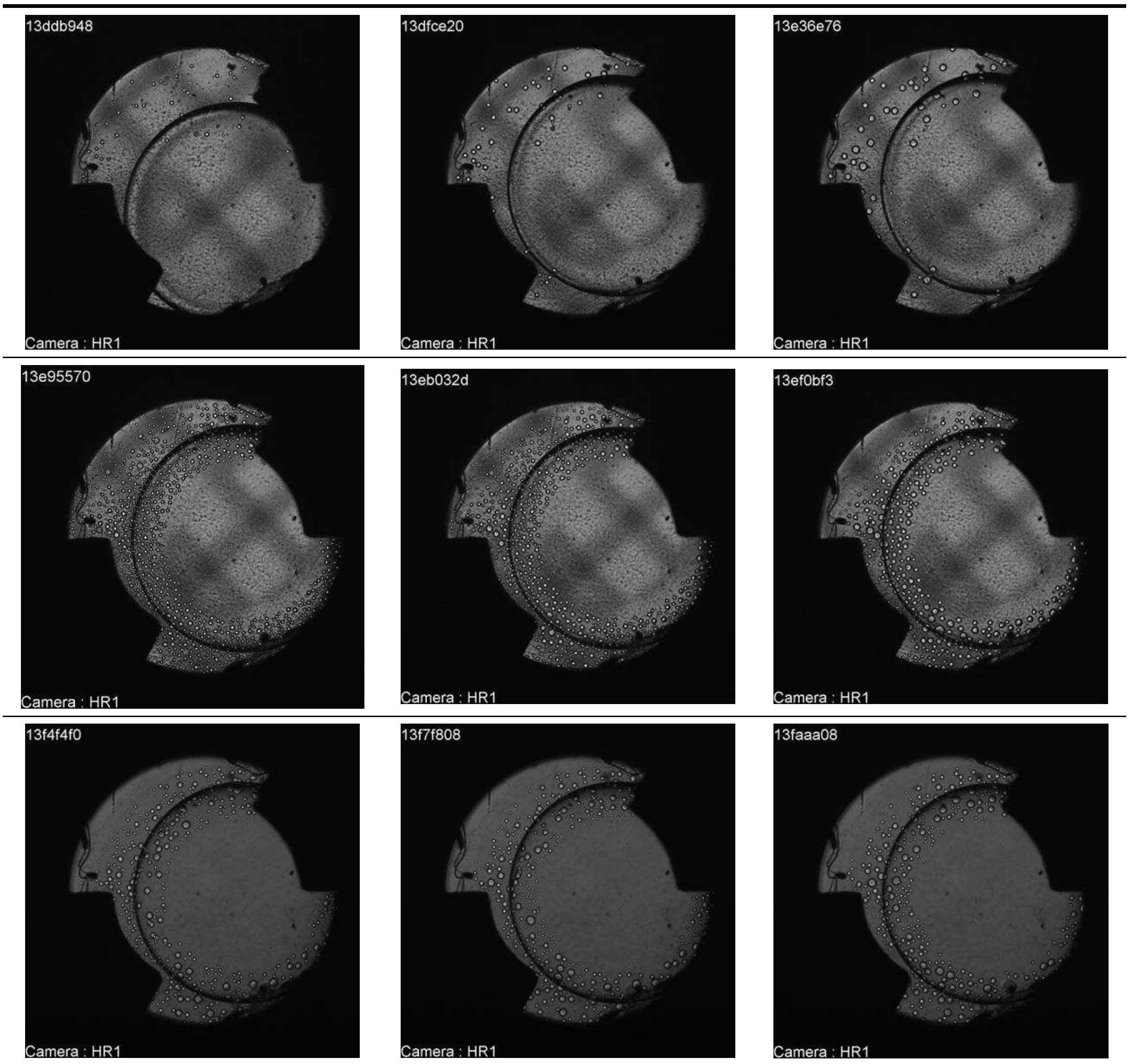


Fig. IX : Wide field images of SF_6 DOC at T_c-T=10 mK, 20 mn (left column), 2 hours (mid column) and 5 hours (right column) after three heating pulses of 0.02 mW / 33 s (upper line), 0.1 mW / 22 s (mid line) and 1 mW / 3 s (lower line). The relaxation of the nucleated small vapor bubbles due to the boiling phenomena close to the triple contact line liquid-gas-transparent heater is clearly observed.

Fig. IX shows typical wide field images of the direct observation cell at three times - 20 mn (left column), 2 hours (middle column) and 5 hours (right column) - of the relaxation period of the boiling phenomena after supplying three different pulses - 0.02 mW / 33 s (upper line), 0.1 mW / 22 s (mid line) and 1 mW / 3 s (lower line) - at T_c-T=10 mK. Here we illustrate the use of the grid shadowscopy on the images of the upper lines, which allows visualization of the density differences between gas and liquid to probe the nucleation and coalescence of the small vapor bubbles due to the boiling phenomena close to the triple contact line liquid-gas-transparent heater.

V.IV.III Conclusions on the ALI Results

We have started the microgravity study of the triggering mechanism at the origin of the boiling crisis by direct observation of a gas bubble spread over a transparent heater surface, during the heating of a critical two phase sample cell filled with SF_6. The preliminary investigations show that the high capabilities of this instrument are irreplaceable powerful tools for studying the boiling crisis for a three-dimensional spheroidal shape of the gas bubble, especially by monitoring of low heat fluxes and by fine tuning of the distance to the critical point to control the liquid-vapor properties.

VI. THE MID-TERM PROGRAM

Currently, the ALI is the last insert remaining on board the ISS, and more ALI sequences to study the boiling crisis phenomenon are planned until the beginning of 2012. Further utilizations of the insert to study the critical phenomena have recently been added to the program. The following scientific objectives are defined for the mid-term agenda of the DECLIC activities.

VI.I ALI-2

The objectives of the new proposed NASA science investigation are to measure thermal diffusivity, heat capacity at constant volume, turbidity, and coexistence curve in two-phase domain of a fluid sample near the liquid-gas critical point. This investigation will utilize the existing ALI insert as is. After completion of the original scientific objectives of the ALI insert, additional experimental scripts will be added to the DECLIC activities on board ISS.

To measure the thermal diffusivity and heat capacity, a heat pulse technique will be used using a heater and thermistors in the Interferometric Observation Cell (IOC). By monitoring the sample temperature after the heat pulse, thermal diffusivity and heat capacity at constant volume can be obtained. The thermal diffusivity will be also determined by monitoring the evolution of density profile of the sample after the heat pulse using the interferogram. We will also use the Direct Observation Cell (DOC) to measure the turbidity and coexistence curve of the sample. As the average density of the sample is ≈2±05% higher than the critical density, we will measure the turbidity of the sample at this off-criticality as a function of temperature. The results will be compared with the previous MIR/ALICE 2 results with the sample density at 0.8% off criticality [8]. These turbidity measurements at two different densities in microgravity condition will enlighten the comparison with Ornstein-Zernike theory. Density measurements in the two-phase domain will also be performed by determination of the volume of the gas bubble in the DOC. This is feasible because the bubble is expected in middle of the DOC cell due to its curved window. By measuring the volume of the gas bubble at different reduced temperatures, the coexistence curve of the fluid can be determined. The experimental result will be compared with predictions of the recent crossover equation-of-state theoretical models.

VI.II SCWM/HTI-R

In many supercritical water applications, a solid phase consisting of inorganic salts exists which arises from chemical reactions during oxidation or from impurities in non-reacting systems [9]. The presence of salts can severely limit the lifetime of system components due to their corrosive behavior and tendency to build depositions on critical thermal control surfaces, plug valves, and flow passages, etc. Precipitation occurs in the near-critical and supercritical regimes due to an order of magnitude decrease in the dielectric constant, ε, of water compared to its value under room conditions (i.e., $\varepsilon \cong 80$ at $T = 300K$ compared to $\varepsilon \cong 5.4$ at Tc) [10]. The Debye ionic screening distance is proportional to $\varepsilon^{1/2}$ so that at near-critical conditions the ionic charges are not well shielded. This leads to a recombination and eventual precipitate agglomeration from dissolved salts.

The HTI insert was returned to the ground with the ULF-5 Shuttle flight. The cell, containing pure water, is being replaced by a similar cell containing a salt- water mixture. This reflight cell is referred to as the HTI-Reflight (HTI-R).

The main objective is to study salt precipitation phenomena close to the critical temperature and in the presence of a temperature gradient. To do so, the workaround implemented to solve the thermal gradient issue (see [1]) will be very useful. The objective is to launch the so-called HTI-R insert in 2012.

The Super Critical Water Mixture (SCWM) experiment, using the HTI-R, will investigate the behavior of an aqueous mixture of a Type 2 salt (sodium sulfate) in the vicinity of the water-salt mixture's critical point. Of particular interest is the point at which precipitation occurs, the rate of precipitate agglomeration, the precipitate's spatial distribution, the precipitate's transport mechanism in the presence of salinity and temperature gradients, and the potential accumulation on the walls of the test section.

A test cell, whose dimensions are close to the HTI cell, has been built and tested at Glenn Research Center (GRC) in order to prepare for the SCWM experiment. The testing includes verification of the filling process in order to make sure that the expected salt concentration is reached when the cell is closed.

Fig. X. Experimental observation cell used in 1-g testing showing holes for four cartridge heaters and one of the two orthogonal optical axes.

Results from experiments conducted suggest that while salt is present in the vapor phase below, but close to, the critical temperature, precipitation occurs mainly in the liquid phase side, nucleating on the surfaces (e.g., windows) in contact with the liquid, and possibly with smaller particulates (less than 15 microns) in the liquid phase. An example of salt deposition patterns on the cell window is shown in Fig. XI.

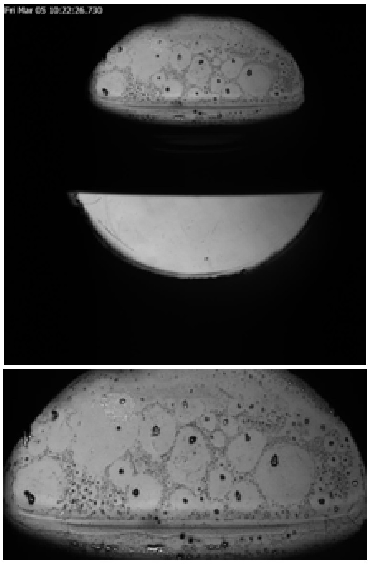
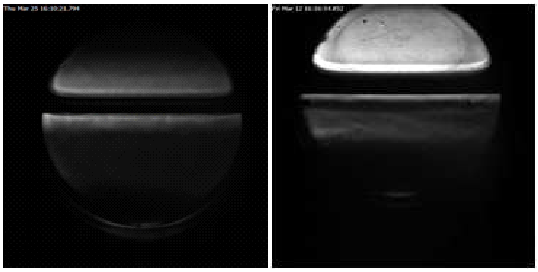


Fig. XI. Salt deposit patterns visible on the cell window (a) in the vapor region after heating to pre-critical temperatures and subsequent cool down and (b) magnified view of salt deposits in the vapor region.

An interesting stratification pattern begins to appear in the liquid during a quench after heating to critical temperature. As the temperature goes below critical, a dark band just below the meniscus appears which sits above a lighter region. This stratification pattern of alternating dark and light regions (Fig. XI) might be explained by the thermal gradient that begins to develop upon cooling. As the test cell cools, a thermal gradient, from high temperature fluid at the meniscus to lower temperature fluid at the base of the cell, begins to develop. The downward drift of the salt-laden fluid (i.e., the darker fluid) is momentarily retarded by the fact that it is at a higher temperature, and consequently less dense, than the cooler, less saline but denser fluid. This creates a salinity inversion that gradually diminishes as salt diffuses out of this dark band.

It was also found that the critical temperature increased slightly with addition of the salt. For example, for a 1.5% weight Na_2SO_4 solution the critical temperature was measured to be 647.8 K compared to 647.1 K for pure water, which makes the SCWM experiments fully compatible with the HTI insert.



(a) (b)

Fig. XII Images of the fluid during cool down. (a) Pure water (b) 10% weight Na_2SO_4 solution. Note the stratification pattern in the salt-water solution.

VI.III DSI-R

The DSI insert has been returned with the ULF-6 Shuttle flight. After a post-flight sequence to take place in October 2011, the cartridge will be replaced by another cartridge containing a different camphor concentration. Consequently, another parameter (camphor concentration) will be added to the parameters available onboard the payload (furnace's temperatures and cartridge's speed). Again, the so-called DSI-R insert will be launched as soon as possible, but likely not before end-2012.

VII. THE LONG-TERM PROGRAM

In a longer term view, and as a logical follow-on of the HTI and HTI-R experiments, a new insert in envisaged. This insert, called SCWO for Super Critical Water Oxidation, will be dedicated to the study of oxidation phenomena in supercritical water. Very preliminary activities have been performed and the development is not formally decided yet.

There is also a strong interest in studying Super Critical fluids as a function of density (i.e. a cell with a tunable volume) and a new DECLIC insert could be a good candidate in doing so.

VII. CONCLUSION

First operated onboard the ISS in October 2009, DECLIC is close to 2 years of successful operations on orbit.

Despite some ALI sequences are still to be performed and most of the data has still to be processed for all the inserts, the results already are of first order.

The HTI and DSI inserts are being refurbished in order to extend the science program by, respectively, replacing pure water by salted water, and changing the camphor concentration, and a new insert, dedicated to the study of oxidation phenomena in supercritical water, is planned.

With that mid-term program well engaged, DECLIC will remain active until 2015 at least.

ACKNOWLEDGMENTS

The authors would like to thank all the companies involved in the development of DECLIC. ASTRIUM-ST (France), which was the prime contractor, but also SODERN (France), University of Amsterdam (The Netherlands), COMAT (France), EREMS (France), the Thermoelectric materials laboratory from Ecole des Mines de Nancy (France)...

The science teams were also involved in the development phases, not only by expressing their needs, but also by developing the cells (ALI and HTI cells were built by ICMCB-CNRS, France), or filling the DSI cartridges (IM2NP-CNRS, France). Thanks to them for their involvement and constant inspiration.

ICMCB team gratefully thank Eric Georgin for his assistance during the cell integration, ARCOFLUID company for it numerical simulation support and Jean Pierre Manaud, Iyad Saadedin, and Guy Campet for development and processing of the thin resistive layer made of Sn alloy oxide.

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