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DECLIC, FIRST RESULTS ON ORBIT

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DECLIC is a multi-user facility to investigate critical fluids behaviour and directional solidification of transparent alloys, developed in the frame of a joint NASA/CNES research program. The instrument is a miniaturized thermo optical laboratory in which one can plug inserts containing the materials to be studied.

Three inserts have been built so far.

- The ALI (Alice Like Insert) is dedicated to the study of SF6 as a near-ambient temperature critical fluid.
- The HTI (High Temperature Insert) is dedicated to the study of pure water as a critical fluid.
- The DSI (Directional Solidification Insert) is dedicated to the study of the solidification of transparent alloys.

The DECLIC facility was launched, with the HTI and the DSI inserts, on the 17-A Shuttle flight (August 2009) and is being operated in an EXPRESS RACK onboard the ISS. The third insert (ALI) was launched on the 19-A Shuttle flight (April 2010).

The instrument monitoring is performed from the CADMOS control centre of CNES (France). Received real time data is forwarded to a web-server so that the scientists can look after their experiments from their own laboratory. In order to cope with the communications limitations onboard the ISS, the whole data is transferred to removable hard-disks to be returned to the ground.

During the first HTI sequence, an unexpected radial thermal gradient over the cell was identified. Its origin was a faulty thermo electrical (Peltier) element. A workaround that enabled HTI to recover a good thermal configuration had then to be implemented. After 5 experimental sequences with the HTI insert, a preliminary analysis of the experimental data already indicates outstanding results, as the determination of the critical temperature of water within a precision of 1 mK, to be confirmed by post-flight ground tests.

To this day, two DSI sequences have also been performed, for which the results are very encouraging, with outstanding optical quality of the taken images.

Experimental sequences with the DSI insert are in progress while the ALI commissioning, followed by experimental sequences, should take place in early 2011. The HTI and DSI have to be returned to the ground (respectively with the Shuttle flights ULF-5 and ULF-6) and refurbished to be launched again as soon as possible. The objectives will be to study precipitation phenomena in salted water for the HTI and to change the sample concentration for the DSI.

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I. INTRODUCTION

As the vapour-liquid critical point is approached in pure fluids, the thermal diffusivity tends to zero and the isothermal compressibility tends to infinity. Fluids near their critical point at low temperature (as helium) or high temperature (as water), cannot be studied properly under terrestrial conditions because they are compressed under their own weight. Similarly, the process of solidification from the melt shows strong density differences in the mother phase. Microgravity gives access to a convection-free observation of the interfaces. This is presented in the first part.

Then the second part of the paper is devoted to a presentation of the facility dedicated to the above science objectives. Developed in the frame of a joint NASA/CNES research program, the DECLIC facility is dedicated to multi users and generally to transparent fluid science domains. It has been designed as a service module that can receive experiment-dedicated inserts, which can be either thermostats, fluid cells or furnaces. Its architecture and operations have been designed to comply with an easy access to micro-gravity conditions on board the International Space Station.

Operations are performed in real time from the CADMOS (Toulouse, France) and the telemetry is also forwarded to the scientists' laboratories (User Home Base). This aspect is presented in the third part.

The first results obtained on orbit are presented in the fourth part. Until now, 5 HTI and 2 DSI sequences have been run. We will emphasize a HTI issue (thermal gradient linked to a faulty Peltier element) for which a workaround has been successfully implemented. This is an example of how open and versatile the DECLIC system is.

The last part of the paper is devoted to the remaining program, using already onboard inserts, refurbished inserts, or new inserts.

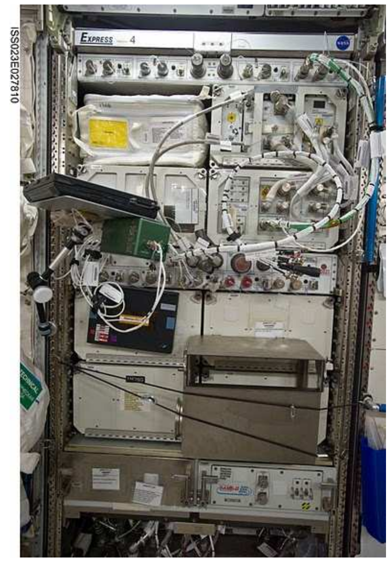


Fig. I: The DECLIC payload fitted in the EXPRESS RACK 4 of the ISS. The two DECLIC lockers are located in the top right quarter of the rack (photo NASA).

II. SCIENCE PROGRAM

The science program has been given in details in [1].

Two main fields are addressed in the initial science program: phenomena near the critical point and the directional solidification of transparent model materials. Three inserts (see §III) 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, properties of high temperature supercritical fluid like water and microstructures dynamics during the solidification of model materials.

II.I. Critical and supercritical fluids.

The hydrostatic pressure induces density gradient that adversely affects ground based observation of near critical fluids. It is the main reason why it is mandatory to perform microgravity experiments.

The initial program for this domain was built on 2 inserts, ALI and HTI, respectively dedicated to room temperature and high temperature critical fluids.

II.I.I. Room temperature supercritical fluids

ALI (Alice Like Insert) provides a relative temperature regulation of a mK. It aims to follow the program conducted onboard the MIR station between 1992 and 2000 with the ALICE apparatus [2].

The scientific objectives are directed to the coupling phenomena involving hydrodynamics in highly compressible supercritical fluids and forced piston effect. After the series of successful experiments on board the MIR station in the ALICE facility, it was decided to pursue the research program on the ISS. The main points to be addressed by this insert are :

- Visualisation of the boiling crisis. To address this point, a specific planar heater was specifically developed.

- Phase separation at very low volume fraction

- Critical density fluctuations in the direct space.

To reach those objectives, the ALI insert contains two cells containing SF6, whose critical temperature is close to room temperature (318.68 K).

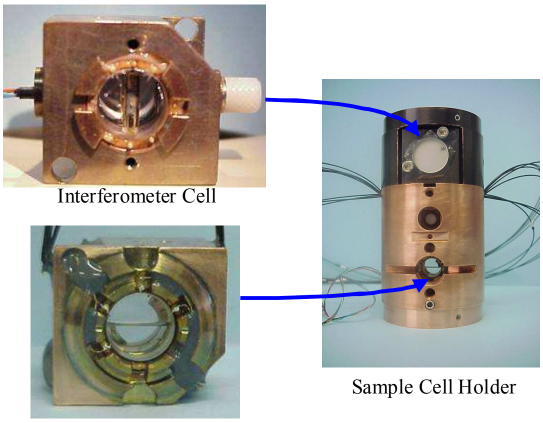


Fig. II : The ALI cells (Interferometer and Direct Observation) and their integration in the sample cell holder [3].

II.I.II. High temperature supercritical fluids

The HTI (High Temperature Insert) is dedicated to the study of supercritical water (critical temperature 647 K, critical pressure 22 kPa).

The main objective, for this insert, is to check if the basic hydrodynamic behavior of water, including the thermo mechanical couplings and the transport processes, does not differ drastically from those occurring in supercritical fluids at lower temperatures. This is called the universality of critical phenomena. More generally speaking, all the water properties close to the critical temperature will be checked for the first time in microgravity conditions.

To reach those objectives, the HTI insert contains one cell filled with pure water, as close as possible to the critical density.

The development of this cell, in order to withstand high pressures and temperatures and, at the same time, meeting the ISS safety requirements, has been a real technical challenge.

Fig. III: The HTI cell assembly. The cell itself is in the middle while the large screws are devoted to withstand high pressures (up to 500 bars) at high temperature (up to 600°C) [3].

II.II. The solidification of transparent model materials

The DSI (Directional Solidification Insert) contains a directional solidification furnace that gives very accurate temperature gradient inside a sample of succinonitrile- based model alloy.

The program addresses both fundamental physics and applied metallurgical questions of interest for ground based processing. In the field of the self organization of matter, several topics are to be explored:

- the growth and Patterns in Non-linear Physics
- the dynamics of extended 3-D systems
- the birth of Morphological Instabilities
- the dynamics of formation and selection of cellular and dendritic a arrays
- the recoil of the solidification front
- the coupling between solidification and convection.

In material processing DSI gives convection free observations of microstructures engineering and well defined model experiments on technical alloys for the ground based process benchmarks.

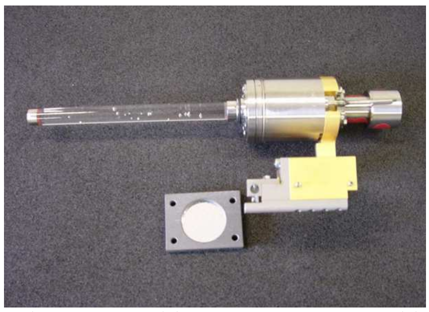


Fig. IV : the DSI cartridge. One can see on this picture the quartz cartridge containing the succinonitrile-camphor alloy (left), the compensator and motorization arm (right) [3].

III. PAYLOAD

The payload has been described in detail in previous papers [1] [3]. Its main functions are to provide services that are needed for experiments dedicated to the study of transparent media. Thermal regulation of the sample, temperature measurements, optical diagnostics, data storage and tele-operation are the main functions.

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

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. Coherent and incoherent light sources and optical sensors are combined in order to provide 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.

An insert accomodates 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 general architecture is given in Fig. V.

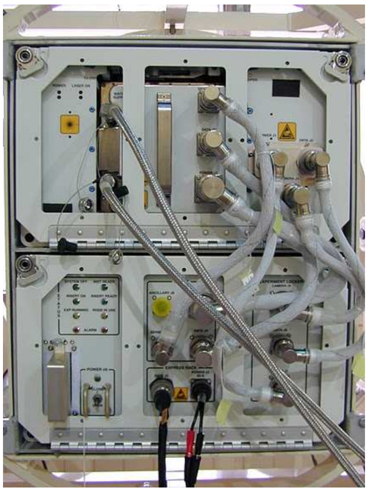


Fig. V: The DECLIC lockers. The ELL is at the bottom while the EXL is at the top and houses the insert. The electrical connections between each component and the water cooling connections (grey "pipes") to the EXPRESS RACK are also visible. (Photo CNES).

IV. GROUND SEGMENT

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).

Fig. VI depicts how the data is transmitted from the payload to the final user (the scientist).

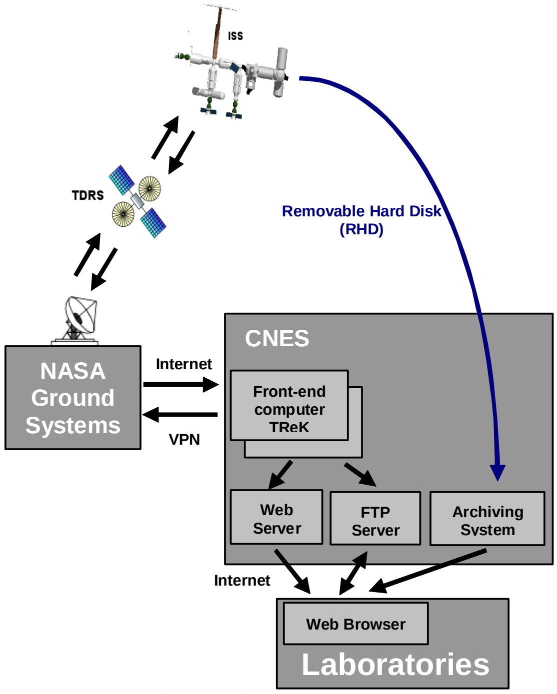


Fig. VI: DECLIC data paths overview. Command and Telemetry follow the black paths while the data stored on RHDs follow the blue path.

Let us consider the Telemetry path as an example. The data is transmitted from the payload to the NASA's ground system through classical ways : TDRS satellites, ground stations and networks.

The Health and Status (H&S) packets are processed at POIC (NASA's Marshall Space Flight Center, Huntsville, USA) level in order to check the payload status.

The other packets are transmitted via UDP to the CADMOS. They are then processed by TReK (Telescience Resource Kit) workstations. The data is then forwarded to a webserver called VisuWeb. VisuWeb allows the scientists to look after their payload in realtime from their laboratory. Fig. VII shows a typical VisuWeb's screen.

Once a day, the whole previous day data is also made available to the scientists on a dedicated FTP server.

As the bandwidth is limited onboard the ISS, and because DECLIC generates numerous data, it is not possible to download all the data through telemetry means. This is the reason why, at the end of each sequences, all the data is recorded on a Removable Hard Disk (RHD) which is sent back to the ground as soon as possible once it is filled. DECLIC was sent with 6 RHDs.

These data are the reference data that will be archived in the CNES' dedicated systems, to which the scientist will be allowed to connect in order to retrieve needed data.

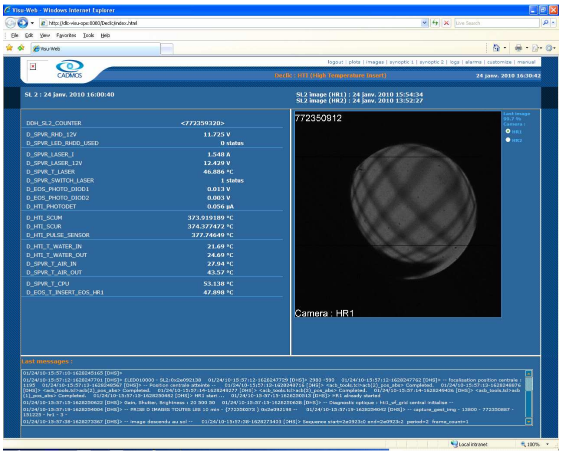


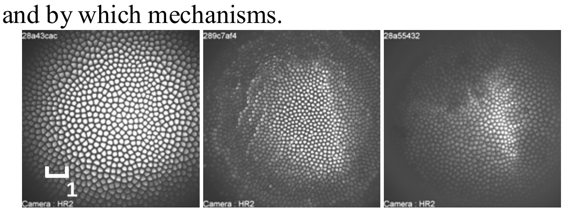
Fig. VII: VisuWeb server's main page, showing real time values of parameters, last taken image and text logs coming from DECLIC

V. FIRST RESULTS

V.I 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 [5]. Acquisition of experimental benchmark data in diffusive mode (ISS experiments) is necessary to provide the scientific community with a database; this is critical for the validation and development of reliable theoretical and numerical models. In parallel, a ground experimental plan similar to the flight one is conducted at the IM2NP laboratory for µg-1g comparative study.

Acquisition of benchmark data on the formation and characteristics of the microstructure in diffusive condition has been initiated. Two types of experiments have been conducted accordingly: long solidifications of the whole cartridge at a given pulling rate V, as well as experiments with a velocity jump from V_1 to V_2 ($V_1 \neq V_2$) occurring in the middle of solidification. The pulling rates range from 0.5 to 16 µm/s. Long solidifications are dedicated to the exploration and characterization of the microstructure map as a function of growth parameters; an illustration of typical microstructures obtained for different pulling rates (solute concentration and thermal gradient fixed) is depicted in Fig VIII. Experiments with a velocity jump are of prime importance in order to understand how a quasi-stationary pattern responds to a variation of parameter (i.e. the pulling rate), with which dynamics and by which mechanisms.

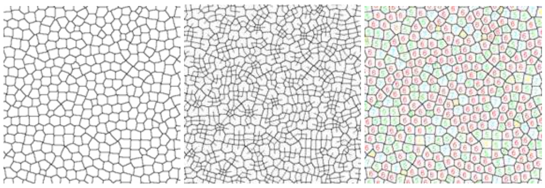


V = 1 µm/s	**V = 4 µm/s**	**V = 8 µm/s**
Shallow cells	Deep cells	Dendrites

Fig VIII: Microstructure type versus pulling rate (Succinonitrile – 0.24 wt% camphor ; G = 20 K/cm)

Quantitative characterizations of the patterns obtained during the first flight experiments are in progress. The study consists in investigating the evolution, as a function of time and growth parameters, of the parameters which characterize the structures morphology, such as the primary spacing, tip radius, order/disorder of pattern, shape of structures.

Image treatments and analyses procedures have been developed on both analySIS and Visilog softwares to facilitate the exploitation of results. In order to illustrate this point, let us consider in Fig VIII the raw image obtained at 4 µm/s (quasi-stationary state). The first step is to obtain a binary image (Fig IXa) that is then used as an input for, on one hand, the cell detection to get the primary spacing distribution and, on the other hand, the use of specific procedures for characterizing pattern order/disorder and type: Minimal Spanning Tree (MST) (Fig IXb) [6], number of first neighbors (Fig IXc), etc.



(a) (b) (c)

Fig IX : Images treatment and analysis procedures: a) Binarisation, b) Minimal Spanning Tree and c) Number of first neighbours superposed to the image (yellow: 4 neighbours ; green: 5; red: 6; blue: 7)

For example, some results obtained during the commissioning of the DSI are presented on Fig X.

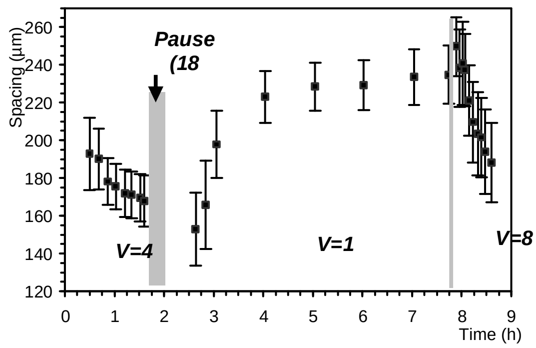


Fig X: Primary spacing = f(t) (Commissioning experiment ; SCN – 0.24wt% ; G = 20 K/cm)

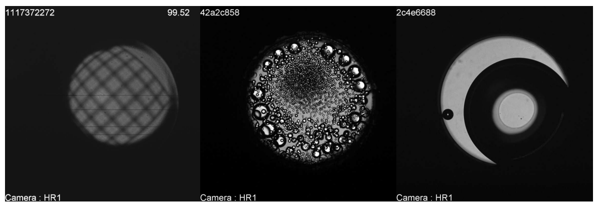
The experiment consisted in a sequence of three consecutive solidifications (about 20 mm each) that started at V = 4 µm/s, followed by 1 µm/s and completed at 8 µm/s. Regarding the primary spacing evolution as a function of time (refer to Fig X), it is evident that the primary spacing depends on the pulling rate, and tends to stabilize at the end of each solidification step. This is more striking for the experiment at V= 1 µm/s, where the time allocated for the solidification enables to observe a quasi-stationary state. Each solidification step is associated with an evolution towards a better ordered pattern, with mainly an hexagonal tiling (cells with 6 neighbors). However, our analyses pointed out a strong dependence on the initial interfacial pattern on the order/disorder evolution: starting from rest or from already existing pattern involves different mechanisms.

V.II HTI

As stated above, the HTI is dedicated to the basic study of pure water in a high pressure and high temperature optical cell.

The main objective is to provide quantitative data concerning the critical properties of water. For this purpose, measurements of turbidity and light scattering intensity are performed together with the thermal dynamics from the piston effect time scale (a few ms) to the diffusion time scale (a few hours). The optical cell is also designed to study the kinetic and morphology of the vapour-liquid phase separation process by means of full-field and microscopy observations. Eventually, the HTI-insert tests thermal stimuli for monitoring the temperature gradients. This latter study aims to prepare future studies in aqueous solutions that have to fit the NASA safety standards concerning high temperature and high pressure. (A more detailed presentation of the optical cell and the HTI-insert can be found in [4].

Illustrations of the local density distribution inside the cell under microgravity (ISS) are provided by the three typical pictures given in Figure XI. In part (a) of this figure, the supercritical one-phase water at Tc+1mK is observed by optical transmission. The fluid picture (grey) shows a circular turbid area (dark grey) corresponding to a radial density gradient close to the external cell body (black part). (The cell is made with inconel alloy). The internal bulk fluid (clear grey) is nearly homogeneous in density, as shown by the very weak deformation of the grid shadowgraphy. The remaining axis-symmetrical density gradients are due to the coupling between the radial piston effect that originates from the thermal exchanges with the thermostat and radiative thermal losses along the optical axis throughout the sapphire windows.



(a) (b) (c)

Figure XI: (a). Supercritical one-phase water at Tc+1mK observed by optical transmission. (b) Two-phase water at Tc-99mK around eight hours after a cooling ramp of -2mk/mn rate, starting from the initial state of the previous picture. (c) Gas-bubble distribution in the two-phase domain at T=351 °C, i.e., ~23°C below the critical temperature of water.

Fig. XI (b) shows a liquid-vapour distribution in the two-phase domain of water at T= Tc-99mK. The cell was cooled from an initial one-phase state as in Fig. X (a) with a cooling ramp of -2mk/mn rate. The above Fig.

XI (b) pattern corresponds to 8 h. equilibration time. The external bubble distribution is initiated by the boiling at the fluid-body cell surface, while the central two-phase distribution is due a nucleation-growth process occurring in the bulk liquid. The long-time stability of the bubble distribution in the cell is presumably correlated with the very low value that the liquid-vapour surface tension exhibits at such small temperature distance from Tc.

Fig. XI (c) corresponds T=Tc-23 °C. The presence of the large vapour bubble is presumably related to the large value of the water surface tension at such a large distance from Tc.

Concerning the value of the critical temperature of water, a first approach has been made for the first time in microgravity, but the absolute value is still to be confirmed by ground testing when the insert is brought back to the ground (sensors calibration).

V.III Main Anomalies

Since the beginning of the operations, DECLIC has experienced several kinds of anomalies. The two main (i.e. having impact on DECLIC performances or inducing strong operational constraints) anomalies are depicted here below.

V.III.I HTI Thermal Gradient

In order to reach the needed thermal regulation performances at high temperature (the water critical temperature being 647.096 K), a dedicated control system, based on several PIDs, had to be developed and optimized. This control system takes into account data coming from the sensors shown on the figure below.

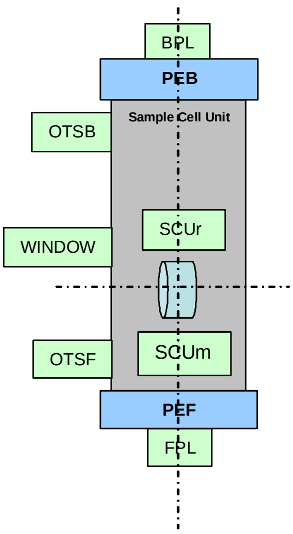


Fig. XII : HTI temperature sensors. The platinum sensors are shown in green while the Peltier elements (PEF and PEB) are shown in blue. The cell (light blue) is located in the Sample Cell Unit.

Most of the sensors are classical platinum sensors, used as absolute or relative sensors. But the Peltier elements (called PEF and PEB on figure XII), which are used in Peltier mode during quenches, are also used in Seebeck mode during fine regulation. In this passive mode, a Peltier element generates signal depending on the thermal difference between its two faces. It is a kind of thermal flux sensor. One of the regulation loops' goal is to minimize the signal given by the Peltier elements (i.e. the flux going through the Peltiers).

During the first HTI sequence, an unexpectedly high thermal gradient (500 mK or so, which is 10 times the expected value), radial with respect to the cell axes, was observed. This was very upsetting for the scientist because, when the fluid critical temperature is neared, the thermal expansion coefficient ($\partial \rho / \rho \ \partial T$) diverges ($\rho$ is density and T is temperature). The situation was paradoxically close to the one experienced on the ground with the effect of gravity!

After some data processing, it was found that one of the Peltier elements (the PEF) signal suddenly stuck to a zero value while it was not the case for the other.

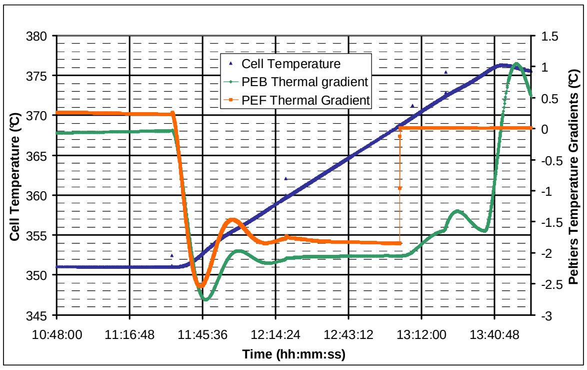


Fig. XIII: HTI thermal behaviour during heating. When the cell approaches 369°C or so, the PEF signal sticks to a zero value.

As the signal remained stuck to zero during all the end of the heating, the thermal control could not correctly regulate the temperature and the thermal gradient value remained very high all sequence long.

A workaround to minimize the gradient had then had to be elaborated.

As the PEF gives the thermal flux going through one side of the Sample Cell Unit (or SCU), a good correlation was found, on the Ground Model, between the PEF value and a linear combination of SCUm and SCUr sensors difference.

$$PEF \approx \alpha(SCUm - SCUr) + \beta \qquad \text{[eq 1]}$$

The PEF being included in one of the PIDs loops, this loop was inhibited in the regulation system and a new loop was implemented, taking into account the calculation of the PEF as shown in [eq 1].

After some successful tests on the Ground Model, the modifications were uploaded on the Flight Model and, after tuning α and β values, the results were even better than expected. The thermal gradient value was back at the expected value (50 mK or so).

There are no explanations yet on what happened to the PEF element. A thermo mechanical event that affects the Peltier circuit electrical connections is suspected. As the HTI will be returned to the ground within a few months (see §VI), the faulty Peltier will, of course, be assessed and replaced.

Proving the extraordinary versatility of the DECLIC system, this successful workaround implementation also shows that the cell's radial thermal gradient can be tuned. And this is an excellent result for the HTI-R program that will be detailed later in this article (§VI).

V.III.II Reboots and Shutdowns

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 :

- Influence of sensitive locations (South Atlantis Anomaly, poles). Despite the locations of the events (Single Event Processes (SEU, SEL) leading to reboots and shutdowns) do not all correspond to these sensitive locations, their influence cannot be ruled out.

- Micro cuts of the power line. The 28V EXPRESS RACK power line has experienced micro cuts of various durations. Because DECLIC is a kind of furnace, it needs quite a large amount of power (up to 500 W when heating up the HTI inserts). Thus, it is not fitted with capacitances large enough to withstand long duration power cuts.

Typically, for power cuts of durations shorter than 100µs or so, the DECLIC supervisor (called SPVR) will cut all the other subsystems but itself, leading to a power consumption of the order of magnitude of the one observed after the shutdowns. This could explain the shutdowns.

For power cuts of durations longer than 100 µs or so, the SPVR is also shut down with all others subsystems and DECLIC is thus completely powered down, until the power comes back. This could explain the reboots.

Unfortunately, the EXPRESS RACK has a voltage trip that rises only when the power cuts are of duration longer than 1000 µs, so we cannot formally say that the shutdowns and reboots are linked to power cuts.

In some configurations, a shutdown or a reboot can be dangerous for the sample because it leaves DECLIC without thermal regulation. For the HTI, an unregulated cooling down can lead to a leak of the cell, while, for the DSI, it can lead to a cartridge breakdown, depending on the thermal configuration and on the cartridge's position in the furnace.

As DECLIC team is not on console 24/7, and because DECLIC has to be quickly configured in order to recover a safe thermal configuration, procedures had to be set up in order to face that kind of event.

The Health and Status data packets coming from DECLIC are looked after at POIC. A reboot, a shutdown, or even a loss of thermal regulation are tracked in those packets and the POIC has the ability, in case nobody is on console at CADMOS, to issue DECLIC emergency commands by applying Ground Command Procedures (or GCPs) jointly defined by the CADMOS and the POIC. The goal of the corresponding commands being to recover, as quickly as possible, a safe thermal configuration.

As a result, a shutdown or a reboot is not dangerous for the material sample contained in the insert, mainly because the ground procedures have been adapted to react as quickly as possible. Nevertheless, in some cases, that kind of anomaly can lead to an important loss of time. When it arises with the HTI insert, for example, close to the critical temperature, the amount of lost days can be up to 4, mainly because the scientists sometimes want to get close to the critical temperature as slow as possible, with as less thermal disturbances as possible.

VI. THE MID-TERM PROGRAM

The DSI sequences should last until the end of January 2011 and will be followed by the ALI program (starting with the commissioning sequence).

But the inserts utilizations will not end and the following is planned for those two inserts :

- HTI-R. the HTI insert will be returned to ground with the ULF-5 Shuttle flight (November 2010). The cell, containing pure water, will then be replaced by exactly the same cell containing salted water. The main objective is to study salt precipitation phenomena close to the critical temperature. To do so, and in order to master the precipitation directions, the workaround implemented to solve the thermal gradient issue (see above) will be very useful. The objective is to launch the so-called HTI-R insert with a Progress flight in the early days of 2012.

- DSI-R. the DSI insert will be returned with the ULF-6 Shuttle flight (March 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 mid-2012.

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.

VII. CONCLUSION

Launched in August 2009 with two inserts (DSI and HTI), DECLIC has already shown very good results and the HTI campaign already is over. The DSI tests will be performed all fall 2010 long and the ALI tests should start in the early days of 2011.

Of the two main anomalies encountered, one is still unexplained (unexpected shutdowns and reboots of the payload) while the other (high thermal gradient over the HTI cell) has been gone around by implementing a new control process. This workaround implementation was made possible by DECLIC architecture which is opened and versatile and really allows to perform many tasks from the ground.

So far, despite most of the data has to be processed, the results are of first order.

The HTI and DSI inserts should be 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.

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The PIs' labs 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).

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