28th Annual Meeting of the
American Society for Space and Gravitational Research
Nov. 28th – Dec. 2nd, 2012

The DECLIC Research Facility

A Fertile Platform for NASA/CNES Scientific Collaboration

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[4] NASA - Marshall Space Flight Center (NASA - MSFC)
[5] Centre National d'Etudes Spatiales (CNES),

Presentation Outline

- DECLIC Facility - Hardware / Programmatic Overview
- DECLIC Inserts and their Investigations
  - ALice-like Insert
    … ALI, ALI-R, ALI-R2
  - High Temperature Insert (HTI)
    … HTI Experiment
    … Supercritical Water Mixture Experiment (SCWM) in the HTI-Reflight (HTI-R)
  - Directional Solidification Insert (DSI)
    … DSI, DSI-R
- DECLIC - Future plans

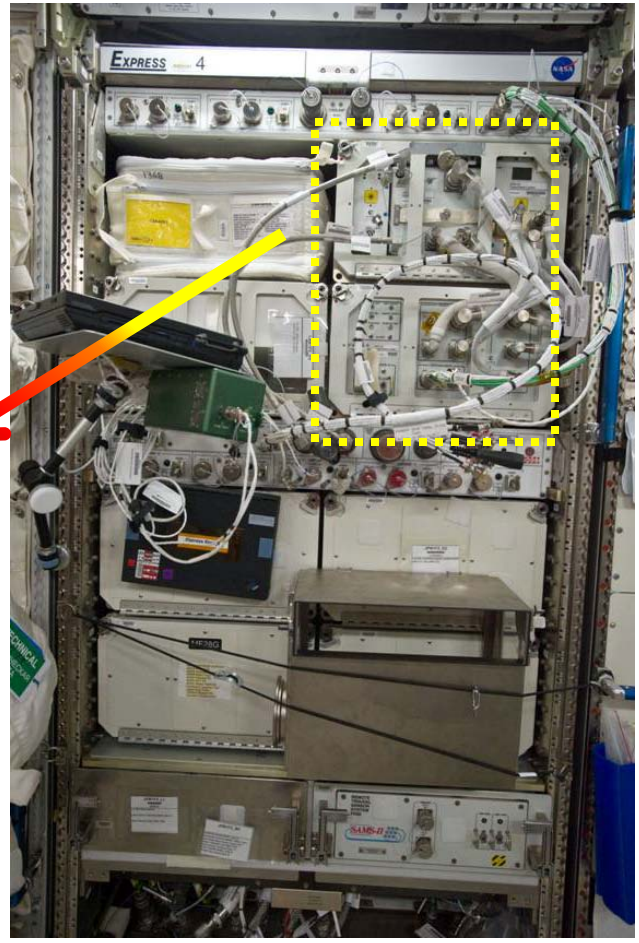
DECLIC Facility Overview

DECLIC Facility

DEvice for the study of Critical Liquids and Crystallization **(DECLIC)**

- Joint CNES/NASA research program
- Launched with STS-128 (August 2009)
- First operations : October 2009

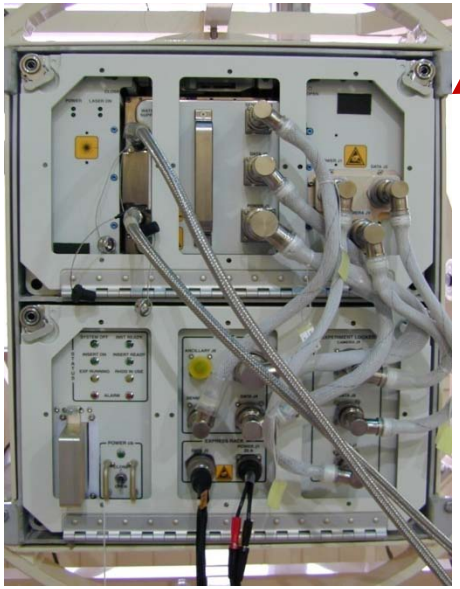
Flight Model onboard the ISS
Japanese Module (JEM) - Express Rack



Experiment locker (EXL), housing the optical bench, light sources and sensors (3 cameras, 2 photodiodes, 3 accelerometers)

Experiment Insert containing the sample cell and the dedicated conditioning and stimulus devices
Size : 200*200*450 mm3

Electronic Locker (ELL) including the power and data handling, precision thermal regulation.



DECLIC Diagnostics (cont)

Longitudinal & transverse observations of sample cell

Direct observation: field of view = ø 12 mm w/ a resolution 10 μm.

Light transmission measurement and grid shadow for turbidity and index gradient

Light Scattering: small angle or 90° for turbidity measurements

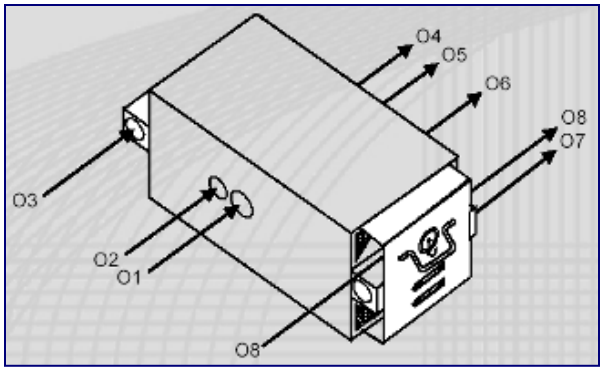
Small field of view (microscopy) 1 mm w/ a resolution of 5 μm

Interferometry: longitudinal or transverse to the fluid cell; resolution 16 μm; on-orbit adjustment w/ piezo-actuator

Cameras: 2 High resolution (HR) and 1 high speed (HS) cameras

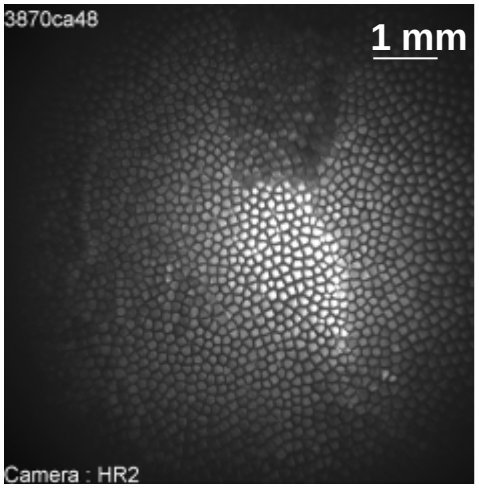
Light Sources: 2 mW He-Ne 633 nm laser with various attenuation filters; several 670 nm LED's

Optical Axis	ALI	HTI	DSI
O1	Interferometry	WF and SF imagery, Grid, transmission, Low Angle Scattering	
O2	WF and SF imagery, Grid, transmission, LAS		Transversal imagery
O3			Interferometry
O4	WF and SF imagery, Grid, transmission, Low Angle Scattering		Transversal imagery
O5		WF and SF imagery, Grid, transmission, Low Angle Scattering	
O6	Interferometry		
O7			WF and SF imagery (HR) Interferometry
O8			Interferometry (reference beam)

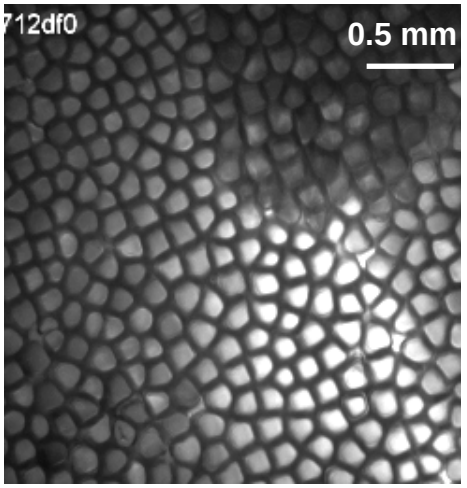


DECLIC Diagnostics (cont)

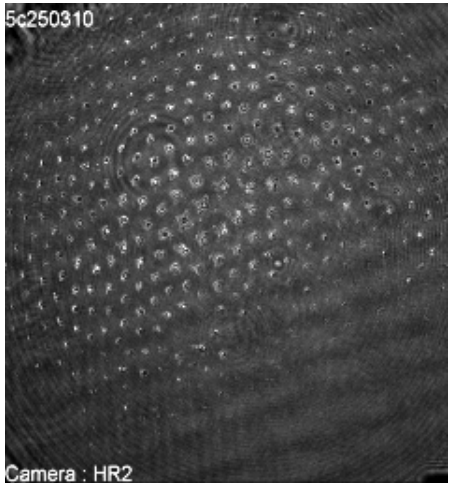
DSI Images



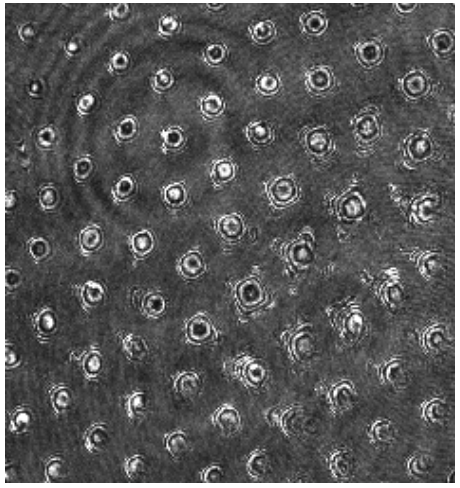
Axial
Wide Field of View



Axial
Small Field of View

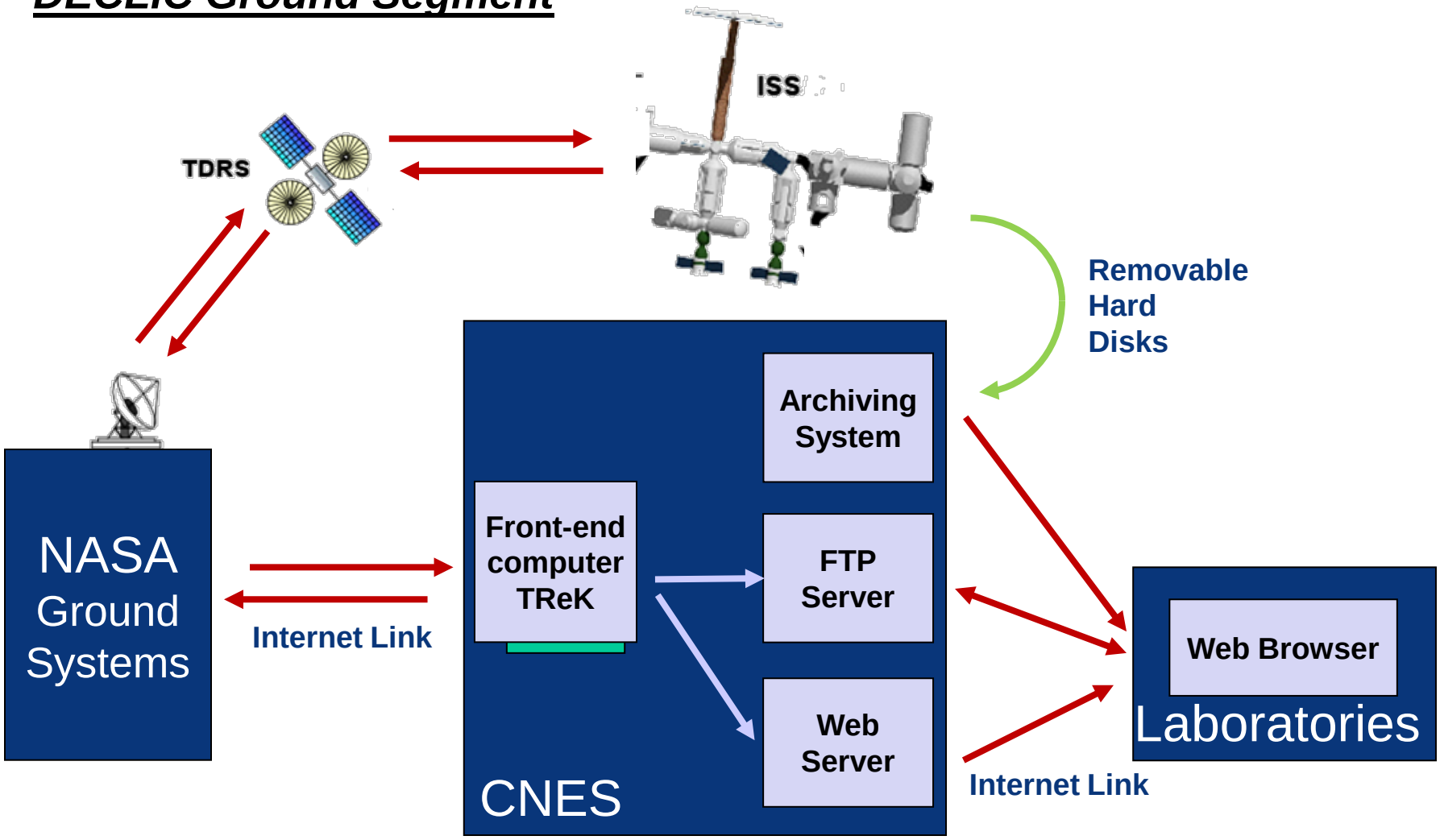


Interferometry
Wide Field of View



Interferometry
Small Field of View

DECLIC Ground Segment



DECLIC Experiments (current)

- **Fluids** (SF6) close to their near ambient critical point in a dedicated insert **(ALI)**

  PI's Lab : ICMCB

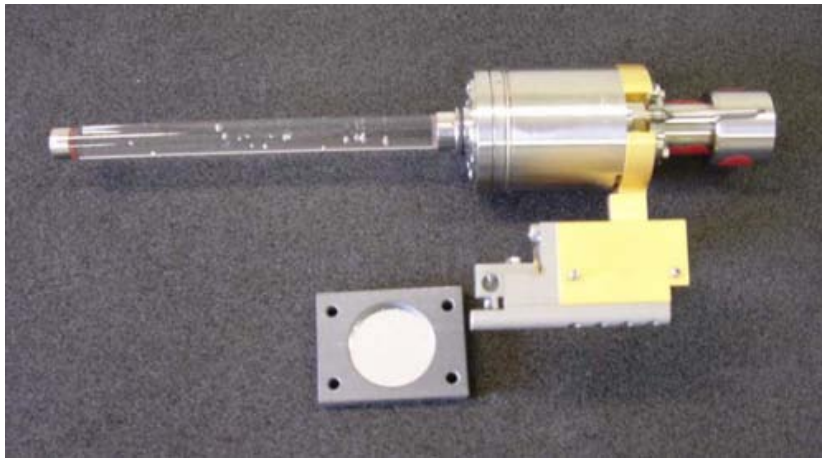
- **High temperature, and high pressure Critical Fluids** (H_2O) in a dedicated insert **(HTI)**

  PI's Lab : ICMCB

- **Directional Solidification of transparent materials** (succinonitrile alloy) in a dedicated insert **(DSI**)

  PI's Lab : IM2NP

HTI Sample Cell - 500 bar and 600°C



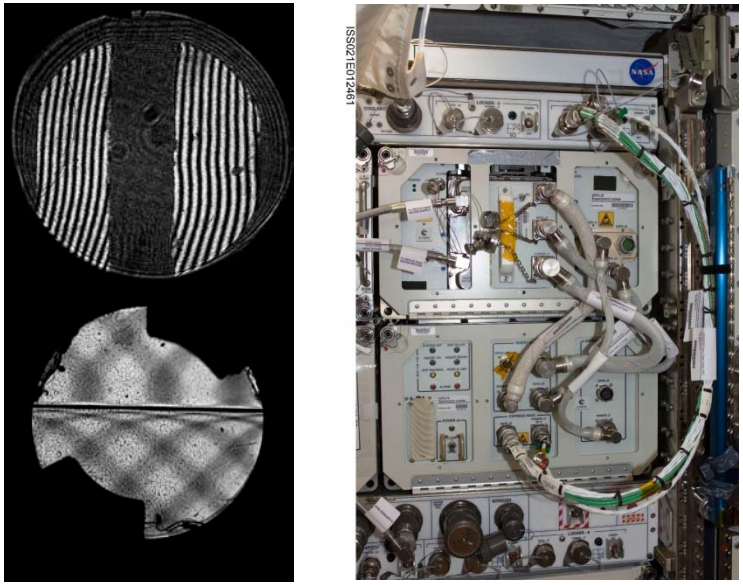
DSI Cartridge

Alice-like Insert (ALI)

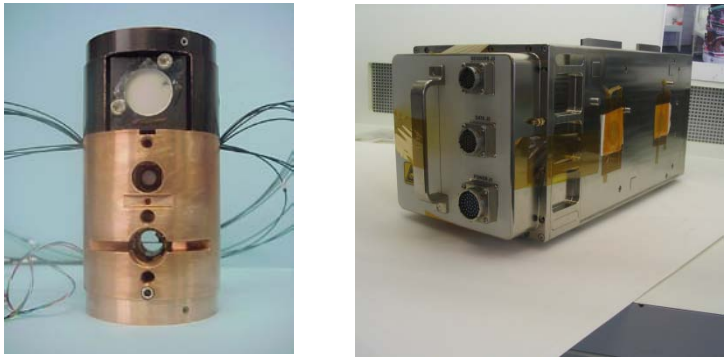
ALI , ALI-R and ALI-R2 Investigations

ALI Experiment - Background

- The ALI ("ALice-like" Insert) is built to study the boiling crisis utilizing properties of fluids near the critical point
  - high stable thermostat
  - two sample cells with SF6 near the critical density
- Interferogram, shadowgraph, heaters, thermometers
- NASA/CNES joint investigation additionally measure
  - thermal diffusivity, heat capacity
- Turbidity (correlation length, compressibility)
- Density in two-phase (coexistence curve)
- **Investigation Team:**
  - I.Hahn, Y. Garrabos, C. Lecoutre, D. Beysens.

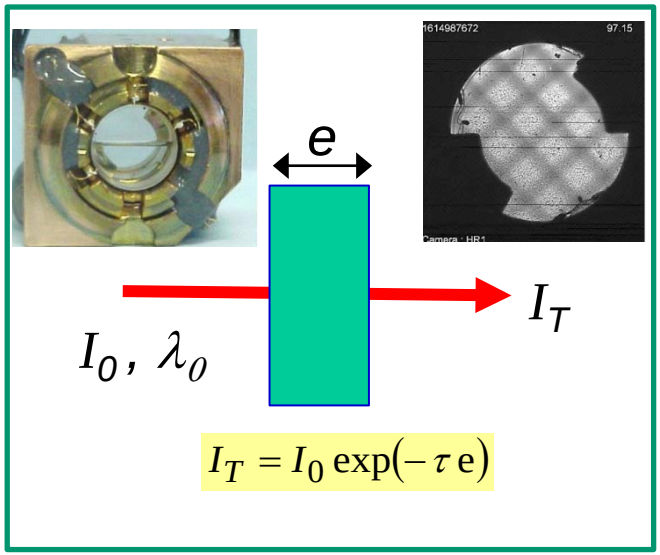


Shadowgraph and interferogram (left) DECLIC on ISS Express Rack (right)

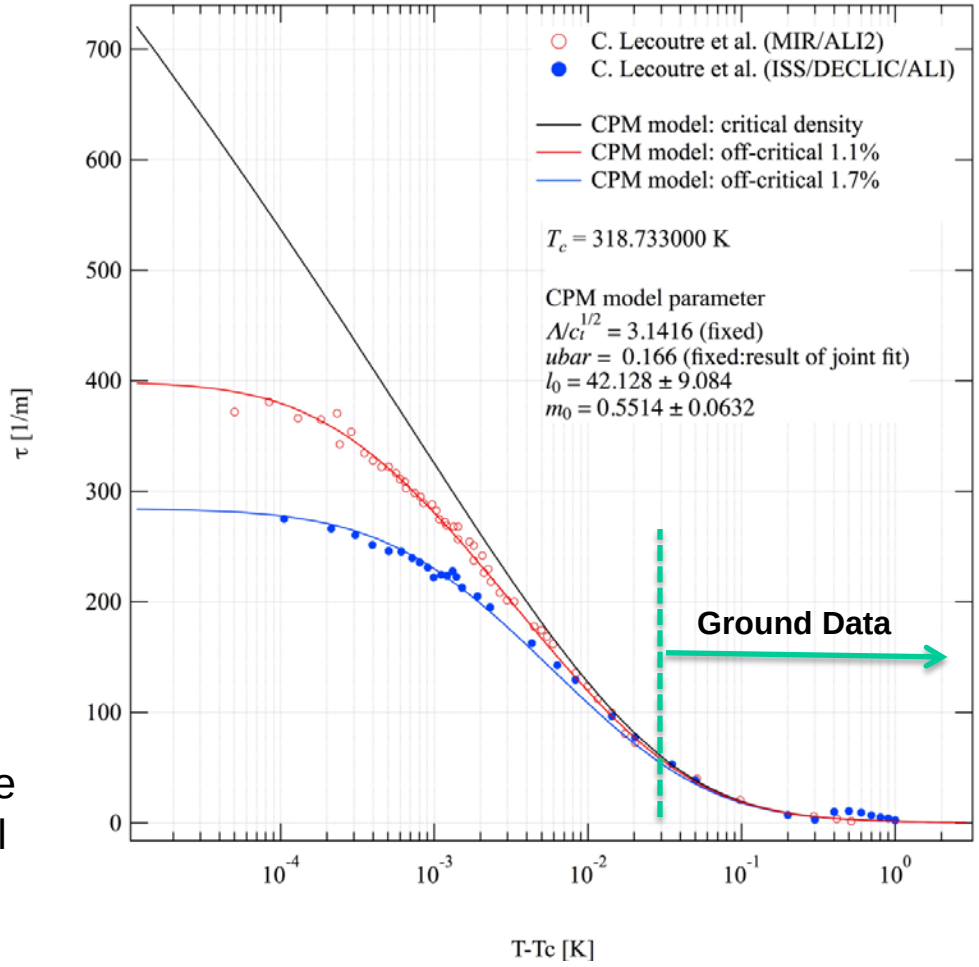


ALI sample cell (left) ALI insert (right)

ALI Experiment - Results



- Current ALI cell is at off-criticality
- Crossover Parametric equation-of-state Model (CPM)* fits data reasonably well with off-critical density (1.7%)
- The ALI flight experiments are still in progress onboard ISS (until Apr-2013)



Validation of CPM for slightly "off-critical" fluid cells *

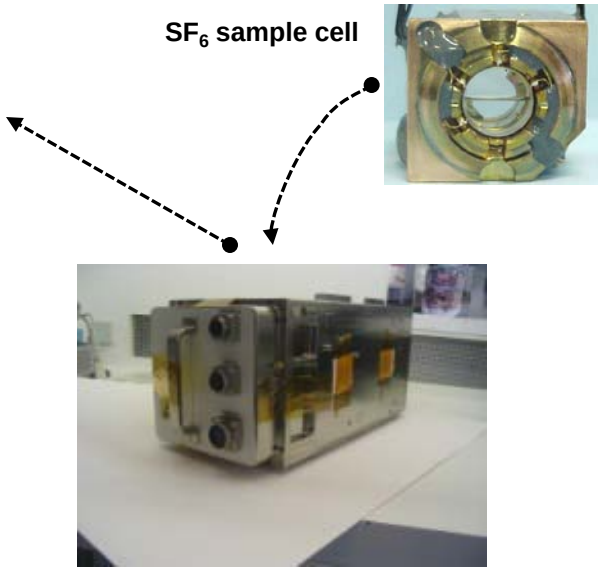
* **V. Agayan** (Ph.D. thesis, Univ. Maryland 2000)

*** C. Lecoutre-Chabot, Y. Garrabos, D. Beysens, I. Hahn** (Boulder, 2012)

ALI-R Experiment – Objectives

- Current ALI cell is at off-critical density.
- Baseline Approach … **no new insert development**
  - bring ALI insert to ground
  - re-fill the current flight cell closer to critical density and re-flight
  - operation 7/2014 - 9/2015
  - no engineering change of DECLIC facility

DECLIC on board ISS



ALI Insert

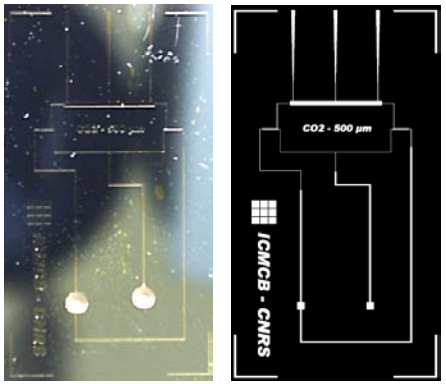
- Turbidity will be measured more precisely at the critical point.
- Effect of Green-Fisher exponent near singularity will be tested.
- Crossover behavior of the SF_6 fluid near the critical point will be studied.
- On-going ground-based R&D
  - development of variable density cell using affordable micro-fluidic techniques.
  - variable density cell may be used for ALI-R

ALI-2 Experiment - Objectives

Plan: 3 years development, launch 2016
PIs: I. Hahn (USA) & Y. Garrabos
Team: F. Zhong, S. Marre, D. Beysens, C. Lecoutre

- Entails development of new hardware (variable density cell w/ new insert) to study critical phenomena utilizing the DECLIC facility
- Precision turbidity measurement
  - Multiple densities + critical density (0.01%)
  - Will provide data 2 orders of magnitude closer to critical point than previous ground experiments
- Critical isotherm measurement & isothermal Compressibility measurement
  - Multiple isotherm measurements using electrostriction technique
  - Determination of singular behavior (exponent δ, γ)
- Critical isotherm & isothermal compressibility measurements
  - Multiple isotherm measurements using electrostriction technique
  - Determination of singular behavior (exponents δ, γ)
- Dielectric constant anomaly measurement
  - 2 orders of magnitude closer to the critical point than previous ground based experiments
  - Anomaly is related to the exponent α.

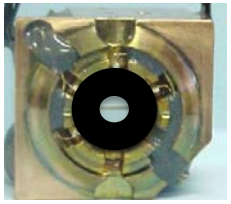
Micro-fluidic Technique



silicon-Pyrex prototype *
micro-fabricated cell

Electrostriction Technique

Capacitor plates



Electrostriction Cell **

* **S. Marre** (DECLIC Workshop, 2012)

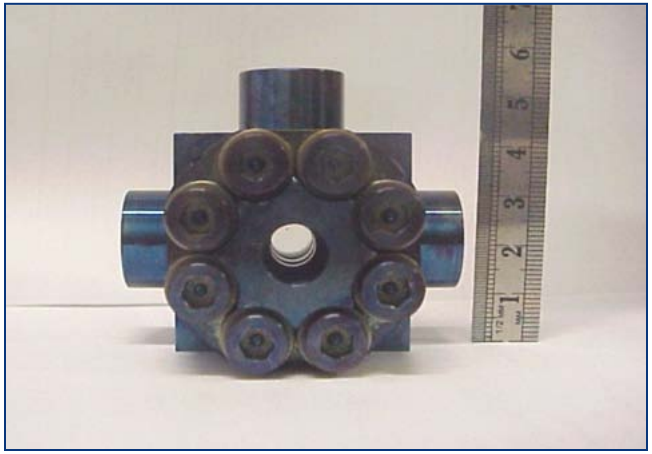
** **I. Hahn** (DECLIC workshop, 2012)

High Temperature Insert (HTI)

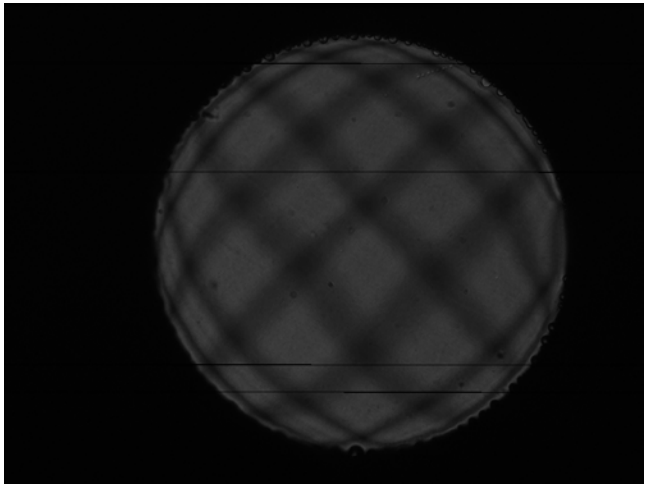
HTI and HTI-R (SCWM) Investigations

HTI Experiment – Background

- The HTI (High Temperature Insert) is built to study near-critical phenomena of pure water
  - pure water critical point Tc ~647 K; Pc ~218 atm
  - represents the high end of fluids in the *3D Ising-like universality class*
- Diagnostics same as that used with ALI … interferometry, shadowgraph, turbidity measurement
- Five test sequences from Sep '09 to Jul '10
  - relative value of Tc measured within 1 mK; absolute value of Tc within 50 mK
  - turbidity measurements made in non-homogenous temperature field make it difficult to correlate w/ density … final analysis awaits development of theoretical tools from ALI experiment
  - further observations … phase separation processes and unexpected transport of localized vapor bubbles due to temperature gradients
- **Investigation Team:**
  - Y. Garrabos, C. Lecoutre, D. Beysens, B. Zappoli



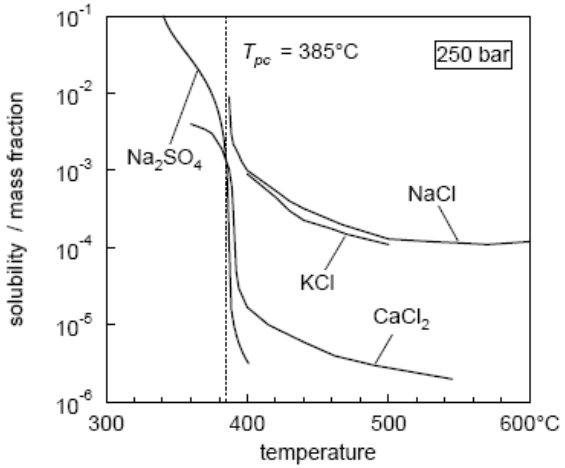
Flight Sample Cell Unit (SCU) to be integrated into the HTI



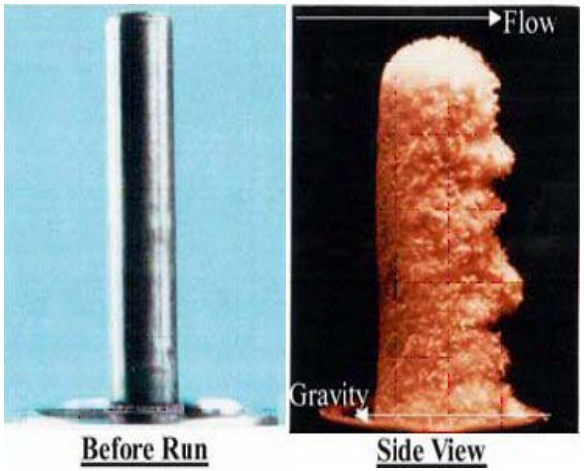
Grid image of the two-phase water cell at 1mK below T_c. Density gradients, in central part of the cell with liquid droplets wetting the internal wall.

HTI–R / SCWM Experiment – Objectives

- The *Supercritical Water Mixture* (SCWM) experiment (2nd Qtr 2013) is a "follow-on" experiment to an earlier ISS experiment (HTI)
  - collaborative effort between NASA / CNES / ICMCB
  - uses a refurbished High Temperature Insert (HTI-R)
  - designed to study precipitation and transport phenomena of a solute near the critical point of the salt/water mixture
- SCWM Motivation :
  - key technological hurdle limiting application of SCWO technology is control of corrosion and fouling caused by deposition of salt precipitates
- Science Objectives:
  - quantify critical point for a specific salt/water mixture (0.5%-w Na_2SO_4)
  - observe/quantify (i) incipient precipitation and solvation at near critical, (ii) observe/quantify transport processes of the precipitate in the presence of thermal/salinity gradients
- **Investigation Team:**
  - M. Hicks, U. Hegde, Y. Garrabos, C. Lecoutre, D. Beysens, B. Zappoli



Solubility profiles of salt in water near Tc



Test in 1-g showing illustrating rapid build-up of salt precipitate; Na_2SO_4 aqueous solution 4%-w at (T_{BF} = 356C, P=250 atm) flowing past unheated rod (left) and heated rod (right) (Hodes, M. '04)

Directional Solidification Insert (DSI)

DSI (MiSOL-3D, DSIP), DSI-R (MiSOL-3D, SPADES) Investigations

DSI Experiments – Background

Microstructures de Solidification 3D (MiSOL-3D)

Investigation Team: B. Billia, N. Bergeon, A. Ramirez, L. Chen

Dynamical Selection of 3-D Interface Patterns (DSIP)

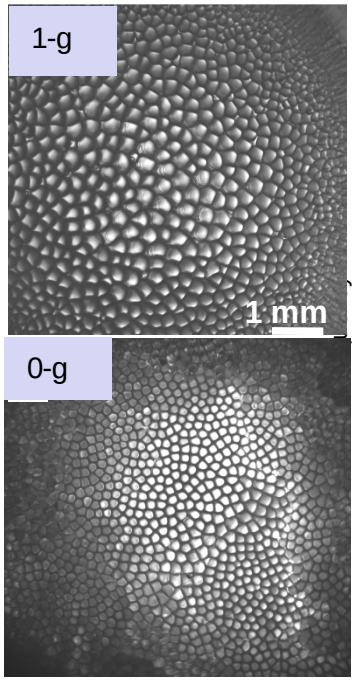
Investigation Team: R. Trivedi, J. Gu, A. Karma, D. Tourret

Relevance/Impact:

- Microstructures created during solidification control material properties
- Applicable to formation of high temperature/strength, complex alloys and molten metal joining (e.g., welding, brazing and soldering)
- Also, micro-gravity effects need to be understood for any potential in-situ metal fabrication/processing

Science Objectives:

- Understand the interface dynamics leading to uniform and reproducible pattern formation in materials, particularly in alloys
- Obtain benchmark data required for establishing the role of interface dynamics on spatial arrangement of three-dimensional interface patterns



Comparison of cell growth patterns at solid-liquid interface in 1-g and 0-g. Radial variation in size, from center outward, due to convectively induced disturbances during cell growth. In 0-g, radial variations in cell structure are absent .

***1-g** mixture = SCN 0.10%-w Camphor w/ VP = 10 µm/s*

***0-g** mixture = SCN 0.24%-w Camphor w/ VP = 4 µm/s*

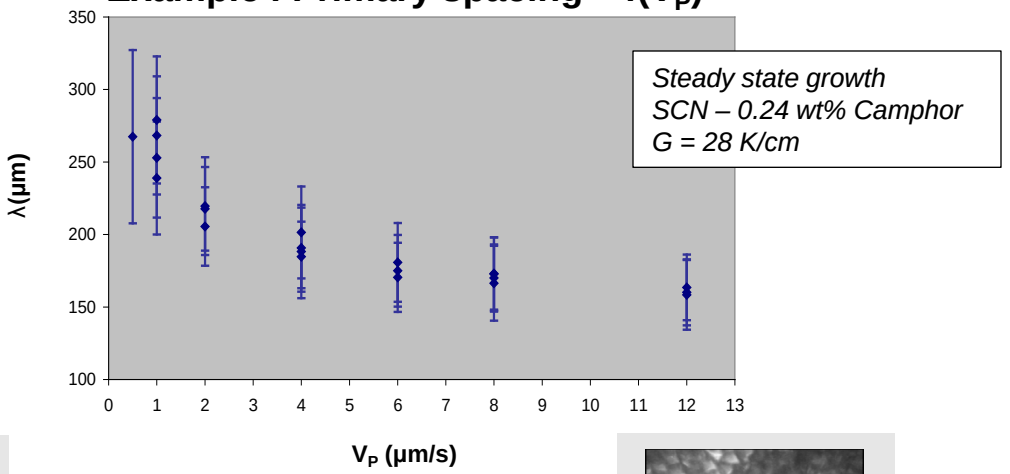
Development Approach:

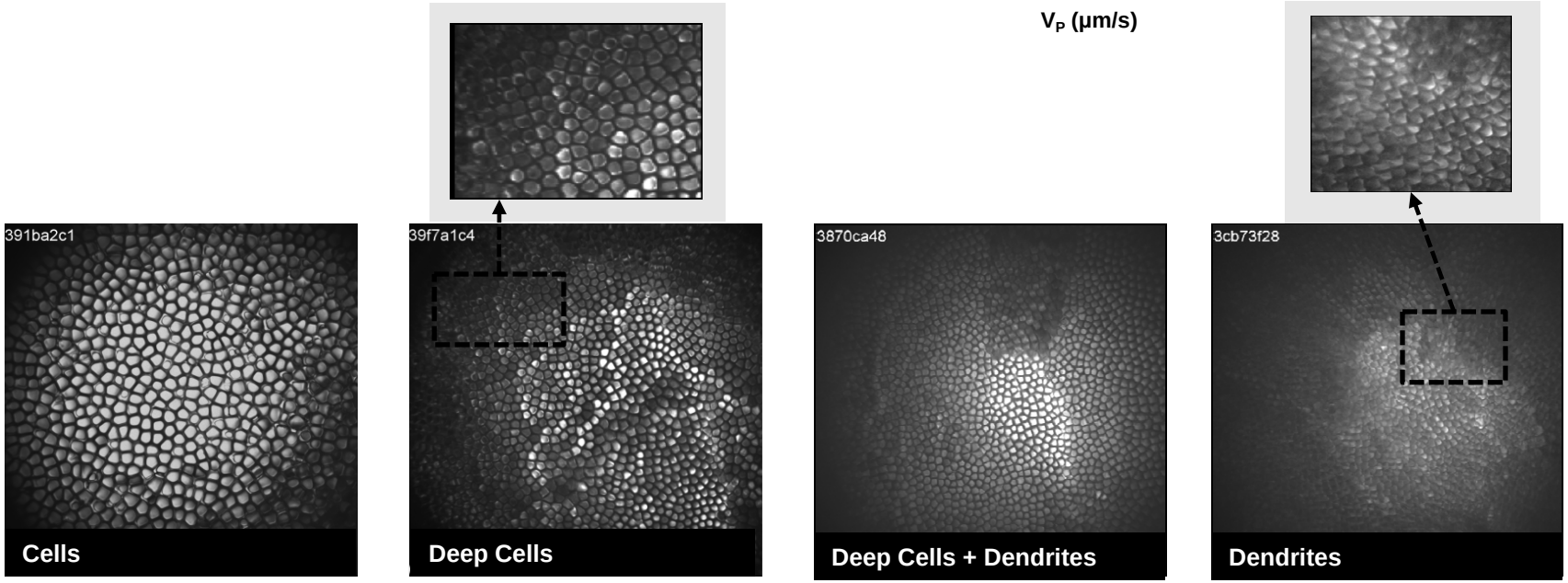
- Transparent alloy of succinonitrile (SCN) - camphor is used as analog of metallic alloy
- Sample included with DSI at launch: (SCN –0.24 wt% Camphor) was re-run with various temperature gradients.
- Step changes in velocity were initiated to examine the role of interface dynamics on the selection of three-dimensional patterns

DSI Experiments – Results

- Data bank being created through systematic analysis of the interface patterns to extract characteristic parameters … **f**(C_0, G, V_p)
- Homogenous patterns observed over a large range of experimental conditions

Example : Primary spacing = f(V_P)



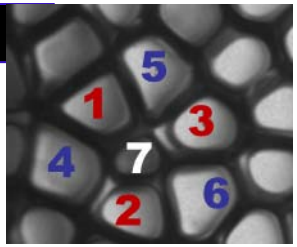


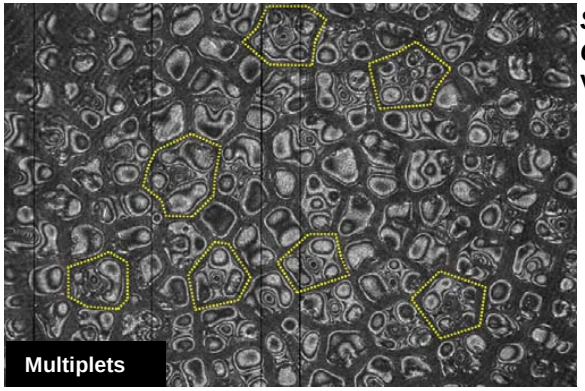
DSI Experiments – Results

Unique observations in extended 3D patterns included:

- Existence of **oscillatory growth of cells** in a diffusive growth regime
- Formation of **multiplets**
- **Coherent side-branching**

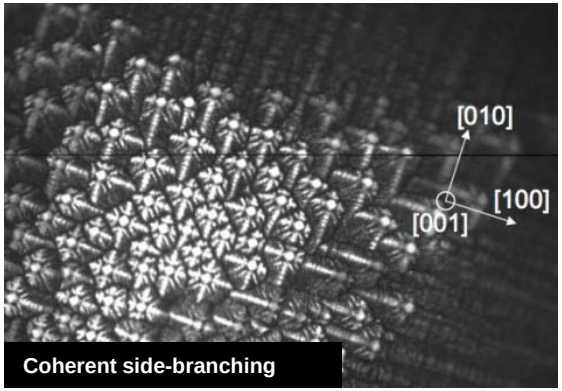
Oscillatory Growth of Cells





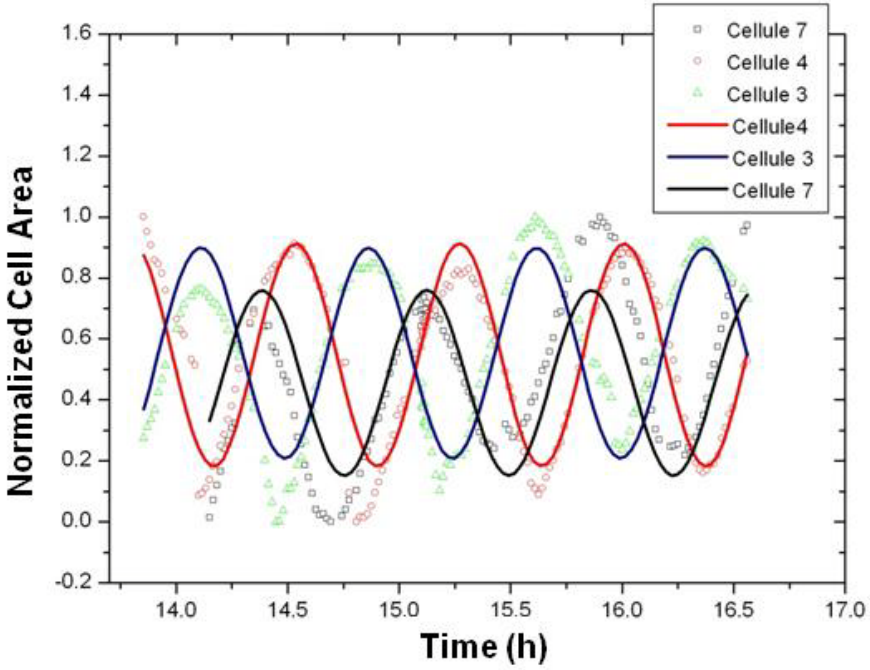
Multiplets

SCN –0.24 wt% Camphor
G = 12 K/cm
V_P= 0.25 µm/s



Coherent side-branching

SCN –0.24 wt% Camphor
G = 12 K/cm
V_P= 30 µm/s



Total of 6 experimental sequences

- 15 –20 days ~ 100 days of experimental runtime performed on-orbit during 2010 and early 2011

DSI-R Experiments – Objectives

Microstructures de Solidification 3D (MiSOL-3D) (continuation)

Investigation Team: B. Billia, N. Bergeon, A. Ramirez, L. Chen, J.M. Debierre, R. Guerin

SPAtiotemporal evolution of 3D DEndritic array Structures (SPADES)

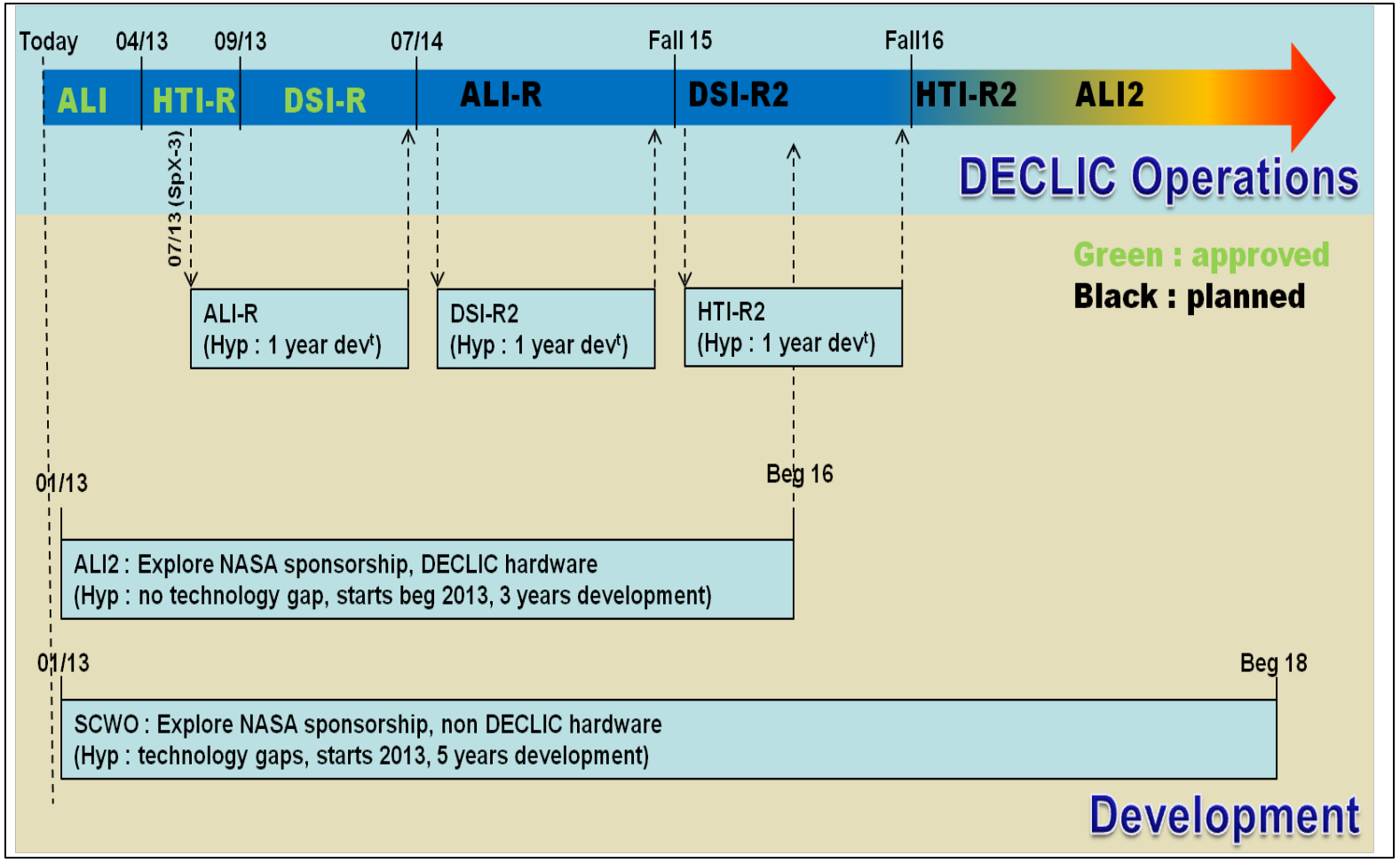
Investigation Team: R. Trivedi, A. Karma, D. Tourret, D. Young

Outstanding Issues Still to be Addressed:

- Effect of composition on dynamics of pattern evolution
- Characterization of variation in cell and dendrite tip radius with velocity (prefer lower solute concentration)
- Effect of cell-dendrite transition on primary spacing
  - Experiments needed: near cell-dendrite transition point w/ variations in V_p to determine effect of transition on cell spacing
- Further experiments are needed to understand the physics of coherent branch formation
  - Experiments performed at the low G, high V_p regime in DSIP revealed dendrites formed with *coherent branches*
- Identification of regime in which the multiplet branch is stable

DECLIC Future Plans

DECLIC Future Plans



BACKUP

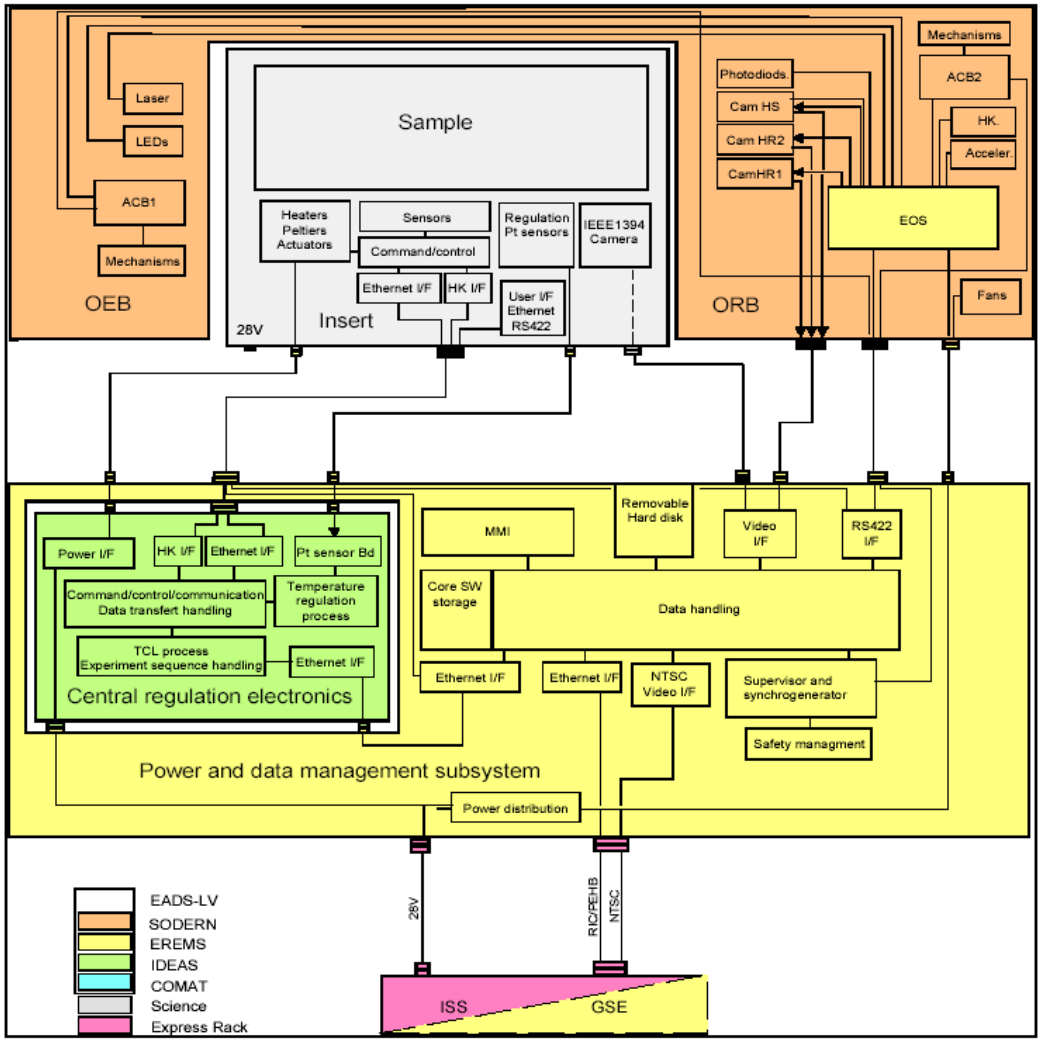
DECLIC Diagnostics

OEB: Optical Emitter Box

ORB: Optical Receiver Box

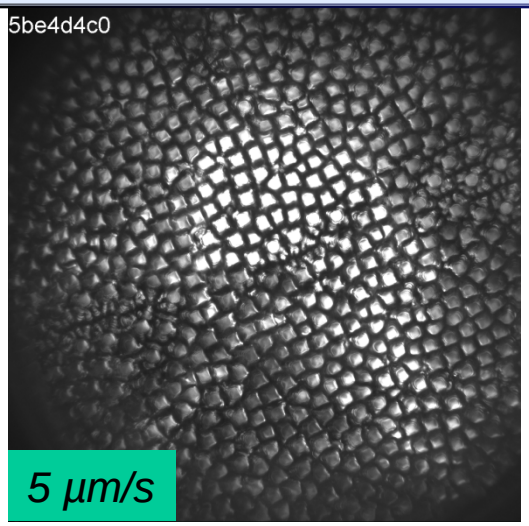
EOS: Electronics of Optical Subsystem

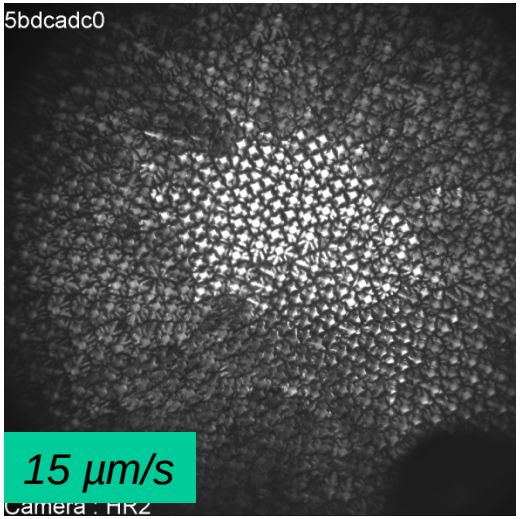
GSE: Ground Support Equipment

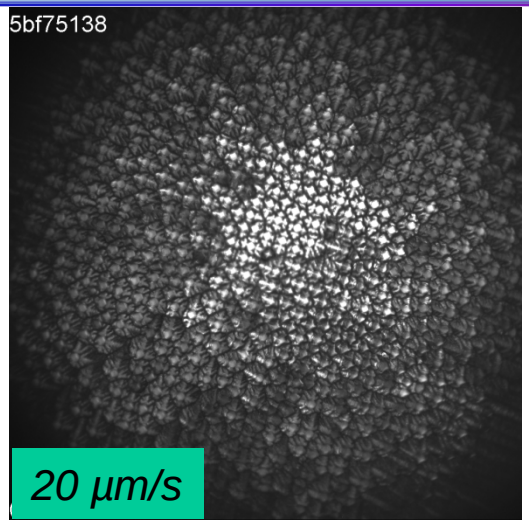


Functional Diagram - Optical System

Supercritical Water Mixture Experiment (SCWM) in the High Temperature Insert – Reflight (HTI-R)







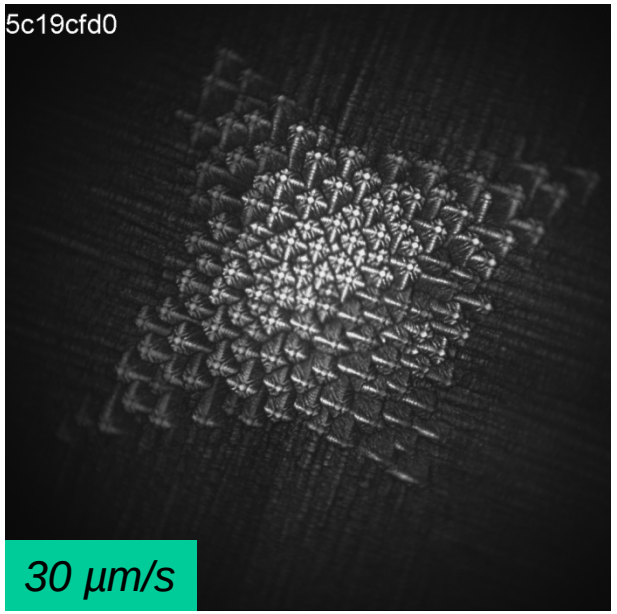
DSI: Large scale side-branch coherence observed

Phase-field modeling will be used to tailor DSI-R/SPADES experiment to investigate dynamical origin of side-branch formation:

noise amplification vs. limit cycle

- *Oscillatory branch of growth solutions bifurcates subcritically and exists over a finite range of large array spacings*
- *Noise-induced side-branching amplitude increases exponentially with spacing up to non-linear saturation due to overlap of diffusion fields from neighboring cells*

Echebarria, Karma, Gurevich
Phys. Rev. E 81, 021608 (2010).



Supercritical Water Mixture Experiment (SCWM) in the High Temperature Insert – Reflight (HTI-R)

From CDR - 2005

PLANNING	INSERT	COMPOSITION
SHORT TERM DURATION (including the 1st increment)	Insert DSI-1	SUCCINONITRILE – 0.25 % CAMPHOR
MIDDLE AND LONG TERM DURATION	Insert DSI-2	SUCCINONITRILE – 0.50 % CAMPHOR
	Insert DSI-3	SUCCINONITRILE – 0.10 % CAMPHOR
	Insert DSI-4	SUCCINONITRILE – 0.90 % CAMPHOR
	Insert DSI-5	SUCCINONITRILE – 10 % CAMPHOR
(LONG TERM DURATION)	Insert DSI-6	PURE SUCCINONITRILE

Outstanding Issues Still to be Addressed:

- ***The effect of composition on the dynamics of pattern evolution.***
- ***Characterization of variation in cell and dendrite tip radius with velocity. This is best achieved with lower solute concentration where cell-to-dendrite transition occurs at higher velocity.***
- ***Effect of cell-dendrite transition on primary spacing. DSIP results indicated continuity in spacing in contrast to prior thin sample ground-based experiments. A set of experiments needs to be carried out close to the cell-dendrite transition through change in velocity to determine precisely the effect of the transition on primary cell spacing. At higher solute concentration the dendrite formation occurs at a lower velocity while the macroscopic interface remains reasonably planar due to limited latent heat release.***
- ***Experiments performed at the low G, high Vp regime in DSIP revealed that dendrites formed with coherent branches. Further experiments are needed to understand the physics of coherent branch formation.***
- ***Identification of regime in which the multiplet branch is stable.***

Phase-field modeling will be used to tailor DSI-R/SPADES experiment to investigate dynamical origin of side-branch formation: *noise amplification vs. limit cycle*

- *Oscillatory branch of growth solutions bifurcates subcritically and exists over a finite range of large array spacings*
- *Noise-induced side-branching amplitude increases exponentially with spacing up to non-linear saturation due to overlap of diffusion fields from neighboring cells*

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DSI: Large scale side-branch coherence observed