The DECLIC Research Facility
A Fertile Platform for NASA/CNES Scientific Collaboration

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2 National Center for Space Exploration Research (NCSER)
3 Jet Propulsion Laboratory (JPL)
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

**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 = \( \varnothing \) 12 mm w/ a resolution 10 \( \mu \text{m} \).

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 \( \mu \text{m} \)

Interferometry: longitudinal or transverse to the fluid cell; resolution 16 \( \mu \text{m} \); 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

<table>
<thead>
<tr>
<th>Optical Axis</th>
<th>ALI</th>
<th>HTI</th>
<th>DSI</th>
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<tbody>
<tr>
<td>O1</td>
<td>Interferometry</td>
<td>WF and SF imagery, Grid, transmission,</td>
<td>Transversal imagery</td>
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<tr>
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<td>Low Angle Scattering</td>
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<tr>
<td>O2</td>
<td>WF and SF imagery, Grid,</td>
<td>Transversal imagery</td>
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<td></td>
<td>transmission, LAS</td>
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<tr>
<td>O3</td>
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<td>Interferometry</td>
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<tr>
<td>O4</td>
<td>WF and SF imagery, Grid,</td>
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<td></td>
<td>transmission, Low Angle Scattering</td>
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<tr>
<td>O5</td>
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<td>WF and SF imagery, Grid, transmission,</td>
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<td>Low Angle Scattering</td>
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<tr>
<td>O6</td>
<td>Interferometry</td>
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<td>O7</td>
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<td>WF and SF imagery (HR)</td>
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<td></td>
<td></td>
<td>Interferometry</td>
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<tr>
<td>O8</td>
<td></td>
<td>Interferometry</td>
<td>(reference beam)</td>
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**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
DELCIC Ground Segment

NASA Ground Systems

TDRS

Internet Link

Front-end computer TReK

Archiving System

FTP Server

Web Server

Removable Hard Disks

Web Browser

Laboratories

Internet Link
**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** (**H₂O**) 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
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:**
**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)

\[ I_T = I_0 \exp(-\tau e) \]


**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
- 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
**ALL-2 Experiment - Objectives**

- 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 $\delta, \gamma$)
- Critical isotherm & isothermal compressibility measurements
  - Multiple isotherm measurements using electrostriction technique
  - Determination of singular behavior (exponents $\delta, \gamma$)
- Dielectric constant anomaly measurement
  - 2 orders of magnitude closer to the critical point than previous ground based experiments
  - Anomaly is related to the exponent $\alpha$.  

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

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**Micro-fluidic Technique**

* S. Marre (DECLIC Workshop, 2012)

**Electrostriction Technique**

** 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 $T_c \approx 647$ K; $P_c \approx 218$ 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 $T_c$ measured within 1 mK; absolute value of $T_c$ 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
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 Na₂SO₄)
  - 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

Test in 1g showing illustrating rapid build-up of salt precipitate; Na₂SO₄ aqueous solution 4%-w at (T_{BF} = 356°C, 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

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

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
**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

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**Example:** Primary spacing = \( f(V_p) \)

Steady state growth
SCN – 0.24 wt% Camphor
\( G = 28 \text{ K/cm} \)

![Graph showing primary spacing as a function of \( V_p \)](image)

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**Images:**
- Cells
- Deep Cells
- Deep Cells + Dendrites
- Dendrites

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**Table:**

<table>
<thead>
<tr>
<th>( V_P ) (( \mu \text{m/s} ))</th>
<th>Primary Spacing (( \lambda ) (( \mu \text{m} )))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 ( \mu \text{m/s} )</td>
<td>( \lambda ) (( \mu \text{m} ))</td>
</tr>
<tr>
<td>4 ( \mu \text{m/s} )</td>
<td>( \lambda ) (( \mu \text{m} ))</td>
</tr>
<tr>
<td>8 ( \mu \text{m/s} )</td>
<td>( \lambda ) (( \mu \text{m} ))</td>
</tr>
<tr>
<td>16 ( \mu \text{m/s} )</td>
<td>( \lambda ) (( \mu \text{m} ))</td>
</tr>
</tbody>
</table>
**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**

![Image of oscillatory growth of cells](image1)

**SCN –0.24 wt% Camphor**
- G = 12 K/cm
- \(V_P = 0.25 \mu m/s\)

![Image of multiplets](image2)

**SCN –0.24 wt% Camphor**
- G = 12 K/cm
- \(V_P = 30 \mu m/s\)

**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)**

**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

ALI  HTI-R  DSI-R  ALI-R  DSI-R2  HTI-R2  ALI2

ALI-R (Hyp: 1 year dev)

DSI-R2 (Hyp: 1 year dev)

HTI-R2 (Hyp: 1 year dev)

ALI2: Explore NASA sponsorship, DECLIC hardware
(Hyp: no technology gap, starts beg 2013, 3 years development)

SCWO: Explore NASA sponsorship, non DECLIC hardware
(Hyp: technology gaps, starts 2013, 5 years development)

DECLIC Operations

Green: approved
Black: planned
Supercritical Water Mixture Experiment (SCWM)
in the High Temperature Insert – Reflight (HTI-R)

BACKUP
**DECLIC Diagnostics**

OEB: Optical Emitter Box

ORB: Optical Receiver Box

EOS: Electronics of Optical Subsystem

GSE: Ground Support Equipment
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:

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

From CDR - 2005

<table>
<thead>
<tr>
<th>PLANNING</th>
<th>INSERT</th>
<th>COMPOSITION</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHORT TERM DURATION (including the 1st increment)</td>
<td>Insert DSI-1</td>
<td>SUCCINONITRILE – 0.25 % CAMPHOR</td>
</tr>
<tr>
<td>MIDDLE AND LONG TERM DURATION</td>
<td>Insert DSI-2</td>
<td>SUCCINONITRILE – 0.50 % CAMPHOR</td>
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<tr>
<td></td>
<td>Insert DSI-3</td>
<td>SUCCINONITRILE – 0.10 % CAMPHOR</td>
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<td></td>
<td>Insert DSI-4</td>
<td>SUCCINONITRILE – 0.90 % CAMPHOR</td>
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<tr>
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<td>Insert DSI-5</td>
<td>SUCCINONITRILE – 10 % CAMPHOR</td>
</tr>
<tr>
<td>(LONG TERM DURATION)</td>
<td>Insert DSI-6</td>
<td>PURE SUCCINONITRILE</td>
</tr>
</tbody>
</table>

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

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

DSI: Large scale side-branch coherence observed

30 µm/s