

# DEVELOPMENT OF STRUCTURAL AND THERMAL ELEMENTS FOR CRITICAL DYNAMICS IN MICROGRAVITY EXPERIMENT

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## Abstract

We report on the design, fabrication and test of cryogenic sample cells and structures for the Critical Dynamics in Microgravity Experiment (DYNAMX), an experiment scheduled to fly on the space shuttle as part of the Microgravity Science Payload (MSP) in February 2001. The design requirements for these components, and the resultant design and implementation experiences will be presented. In particular, experiences with the use of stainless steel, Vespel, Kevlar and gamma alumina composite as materials for use in sample cells and structures with experimental results of the actual thermal characteristics of fabricated prototype structures will be presented.

## Introduction

In the fundamental physics community there is clearly the desire for apparatus to support high precision and high accuracy cryogenic experiments in a microgravity environment. Nearly 100 national and international investigators (U. S., Japan, Germany, the U.K. and Canada) have defined fundamental physics objectives in critical phenomena, relativity, and low temperature physics that are only possible in a microgravity environment. In keeping with NASA's desire to simplify and reduce the cost of flight development, the Jet Propulsion Laboratory (JPL) has undertaken the task of supporting the development of the science instruments for the Principal Investigators of high resolution, low temperature fundamental physics experiments. In doing so, the last of the Space Transportation System ("Space Shuttle") based low temperature microgravity experiments will pave the way for development in the space station era on board the LTMPF<sup>1</sup>.

DYNAMX is a Principal Investigator (PI) experiment competitively peer reviewed and selected for flight definition from the 1991 Fundamental Physics NASA Research

Announcement (NRA). DYNAMX will examine the critical behavior of a second order phase transition driven far from equilibrium by measuring the thermal conductivity of liquid helium, as a function of applied heat flux, at temperatures around the normal/superfluid transition temperature. To achieve the scientific objectives of the experiment requires the apparatus to have sub-nanoKelvin temperature control and resolution, sub-nanoWatt heat control and resolution, and control and readout resolution of the helium superfluid/normal fluid interface within the cell at the micrometer level. In addition to the science requirements, the apparatus must also meet environmental requirements, (e.g., shuttle launch loads), as well as all safety verification requirements that accompany a shuttle-borne experiment. These levels of requirements, however, have not previously been simultaneously met by any cryogenic apparatus, and the current challenge is to meet all requirements within the inherited experiment configuration to the degree possible.

DYNAMX is the fourth in a series of experiments conducted in the cryogenic environment provided by the Jet Propulsion Laboratory (JPL) Low Temperature Platform (LTP) Facility in the Space Transportation System (STS). These previous flights provide a rich legacy of inherited hardware, software, ground support equipment, procedures and experienced personnel on which the implementation of DYNAMX is based,

All experiments that have been, or are planned to be, conducted in the JPL LTP are measurements of the thermal response of liquid helium to known heat inputs. The DYNAMX Critical Thermal Path (CTP) consists of the DYNAMX measurement-specific sample cell, the instrument components directly thermally linked to the cell, and the structure elements joining the cell, instrumentation and cryoprobe. A cryoprobe is a thermal control and structural support platform within a cryostat or dewar.



Figure 1: The JPL Low Temperature Platform

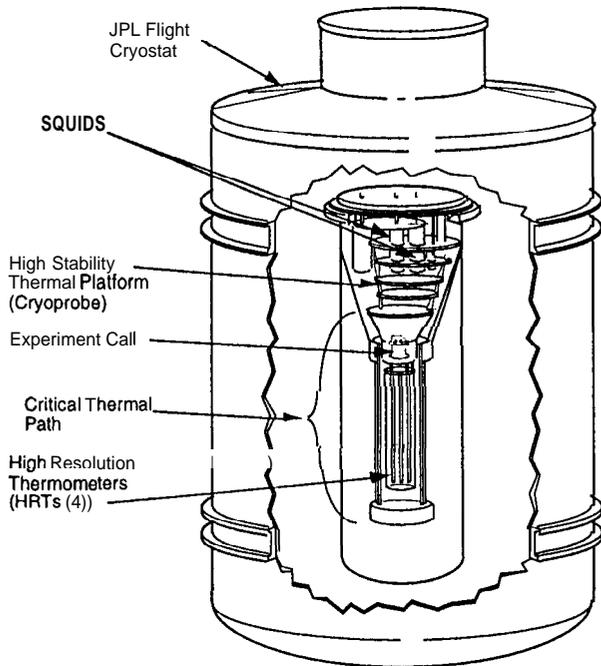


Figure 2: Cryoprobe and CTP within the flight cryostat

In the DYNAMX experiment, heat is applied to one end of a right cylindrical sample cell of helium and is extracted from the other end. Over the range of interest, normal and superfluid phases of helium can simultaneously exist in the sample cell, separated by an interface region. By varying the heat flow in and out of the cell, the interface can be quasi-statically moved back and forth between the hot and cold ends of the cell, allowing the temperature with respect to interface position to be measured by thermal probes embedded in the walls of the sample cell.

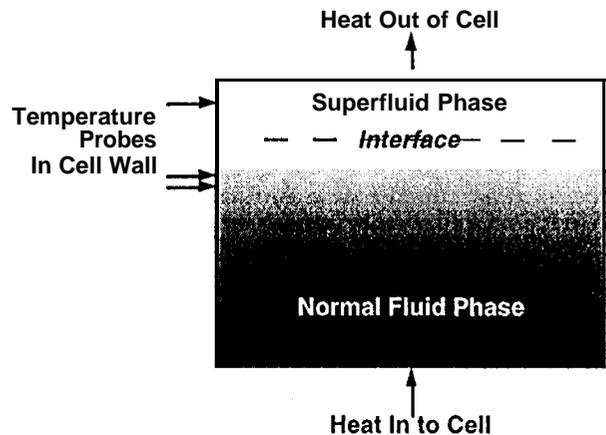


Figure 3: DYNAMX measurement method

The cell contains three sidewall probes, which are made from rings of thin high thermal conductivity material hermetically sandwiched between cell wall segments of low thermal conductivity material. The endcaps of the sample cell needs to be made from material that has high thermal conductivity to provide uniform heat flow across the endcap, and that has low mass to minimize the stray heating that comes from the cosmic ray environment in earth orbit.

Temperature readout is obtained with High Resolution Thermometers (HRTs) of design similar to those used in the Lambda Point Experiment (LPE) and the Confined Helium Experiment (CHeX)<sup>2</sup>. Unlike LPE and CHeX, for DYNAMX each of the four HRTs supported by the LTP is an independent data channel (LPE and CHeX used the HRTs for two data channels with redundancy on each channel). To control thermal crosstalk between thermometers (each reading a different temperature), as well as to control crosstalk between the thermometers and different locations on the cell, a new supporting structure has been developed.

### Development Requirements

The functional requirements of the laboratory prototype development are:

- The prototype shall interface with the PI's existing cryoprobe.
- The prototype shall be modular and permit access to allow the hardware to be iterated as the performance requirements are better

understood through a combination of testing and analysis

- . The prototype shall include:
  - . A sample cell with three sidewall temperature probes
  - . A nano Kelvin stable thermal reservoir (as a heat sink for the cell)
  - . A superfluid leak tight cryogenic valve
  - A pressure stabilization device
  - . Germanium resistance thermometry
  - . Cell and thermal reservoir heaters
  - Mounting structure for the cell, reservoir and all instrumentation.
- Fast (< 6 month) design and implementation to maximize PI test time.
- The prototype shall be adaptable to a design that will fit within the existing JPL LTP.

The performance requirements of the laboratory prototype are:

- The prototype shall apply heat current densities of  $5 < Q < 70 \text{ nW/cm}^2$  through the cell with better than 1 % accuracy and **precision**.
- The prototype shall read the temperature of helium at the level of each sidewall probe over the range of measurement ( $2.2\text{K} \pm 10\mu\text{K}$ ) with better than 0.2 nK accuracy and precision
- The prototype shall read the temperature of the helium at the level of each sidewall probe with better than 12  $\mu\text{m}$  accuracy and precision.
- The prototype shall stabilize the Pressure within the cell such that the (pressure dependant) superfluid/normal fluid transition temperature is accurate and precise to better than 0.2 nK.

The flight environmental requirements on the laboratory prototype development are:

- The prototype cell shall be superfluid leak tight after launch load shake,
- The prototype materials shall be chosen to reduce the effects of on orbit heating due to cosmic rays to within the performance

requirement levels (to be confirmed by analysis),

### Design

The principal challenges in developing the sample cell were:

- Identifying cell wall materials and dimensions, and implementations that yielded the required thermal and spatial accuracy and precision.
- Developing an implementation that was superfluid tight and rugged enough to withstand launch load vibration and repeated thermal cycling.
- Accomplishing the development within the schedule.

A combination of low<sup>3</sup> and high<sup>4</sup> resolution thermal modelling successfully identified a small number of candidate materials. “Proof of concept” single probe cells were made using the candidate materials and testing results revealed the most promising approach for a cell meeting all of the development requirements (see discussion in the Fabrication section below).

The final cell design utilizes Vespel as a sidewall material, with 190  $\mu\text{m}$  X 76  $\mu\text{m}$  steps at the joints to the 50  $\mu\text{m}$  copper foil probes. The segments are bonded to the copper foils using Hysol 9330 epoxy in a channel accessible from the exterior of the cell. High purity (> **99.99%**) aluminum has been chosen for the endcap material to meet the cosmic ray impact requirement.

The stringent limits on heat leaks into the CTP led to a design which utilized Vespel for the flexures isolating the cell and thermometers from the cryoprobe. The stringent limits on heat leaks within the CTP led to a design which utilized 127 and 254  $\mu\text{m}$  thick 304 stainless steel shim stock, bent and spot welded into “box” like elements for structural elements supporting the cell and thermometers while maintaining their thermal isolation from each other. The thermal reservoir is filled with Copper Ammonium Bromide (CAB) which has a Curie temperature just below the reservoir operating point, and provides the necessary heat capacity per unit volume for the unit’s use as a stable heat sink.

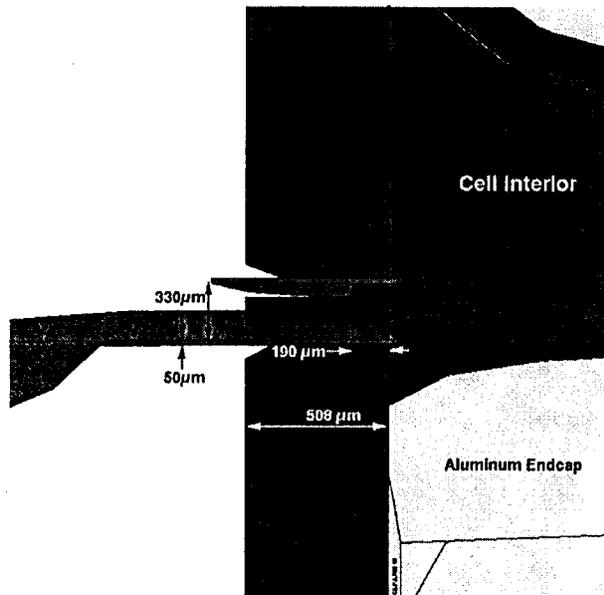


Figure 4: Rendering of cell cross section

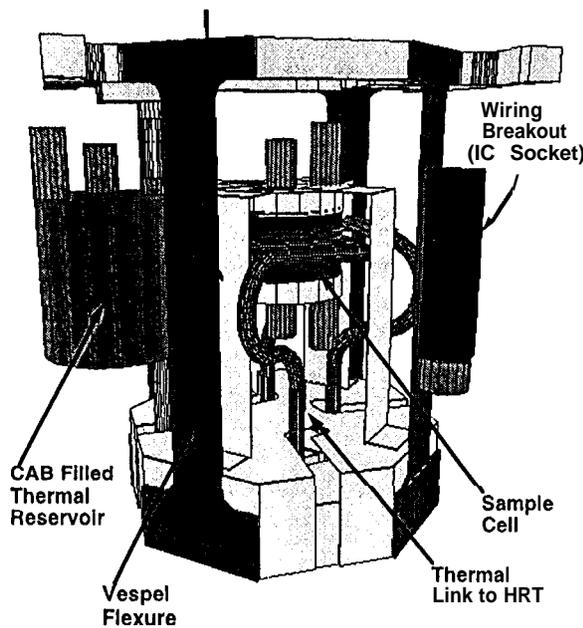


Figure 5: Rendering of prototype CTP

### Fabrication

“Proof of concept” single probe cells were made using the candidate materials identified by thermal modelling. Diffusion welding of cupro-nickel sidewalls to copper probes was abandoned after repeated attempts failed to produce a joint that would remain hermetic over the full range of expected operating pressures within the cell. Although hermetic cells were

produced using gamma alumina composite (tube) sidewalls epoxied to copper probes, the approach was ultimately abandoned after it was decided that producing cells with the necessary probe spacing accuracy and precision (particularly parallelism) would lead to significant schedule risk,

Vespel sidewall cells containing three sidewall probes made from rings of high thermal conductivity copper as thin as 25 μm can now be routinely built and tested. Cells of interior diameter 1,8 cm and wall segments of 508 and 762 μm thickness have been tested. Probe spacings (segment lengths) as small as 330 μm have been achieved in a superfluid leak tight cells.

### Test Results

Cells of both preliminary (gamma aluminum wicks) and present (Vespel SP-1) walls designs have been subjected to series of tests to verify their leak tightness and survivability in operation. Since it is known that the cell will be subjected to several thermal cycles during testing of the apparatus, the cells are subjected to at least 5 thermal cycles between the room temperature and 77K. After thermal cycles the cells are leak tested in the configuration maximally resembling operating environment: each cell is placed in a vacuum can. It is cooled down to 1.5 K and filled with Helium, which condenses into the cell, forming superfluid there. The level of Helium content in the vacuum can is constantly monitored with a mass spectrometer leak detector. Stationary increase of the Helium content indicates failure of the cell. This test is more sensitive than regular leak test with Helium gas. No cell that passed through the preliminary screening failed superfluid leak test. Finally, the pressure of superfluid Helium inside the cell is increased to 0.4 MPa to ensure that the cell will survive internal pressure it will be subjected to during operation and tests (less than 0.1 MPa). Two of the cells have been tested to destruction at 1.5 K by increasing the internal pressure. One of the cells leaked when the internal pressure reached 1.5 MPa. The other cell survived the maximum pressure achievable in this type of tests: 2.4 MPa (Helium solidification pressure).

Several tests have been performed to verify that the implementations are consistent with the design assumptions, In particular, the thermal

conductivity of the materials utilized in the construction of the CTP have been measured and found to be in the range of assumptions used in the thermal modelling.

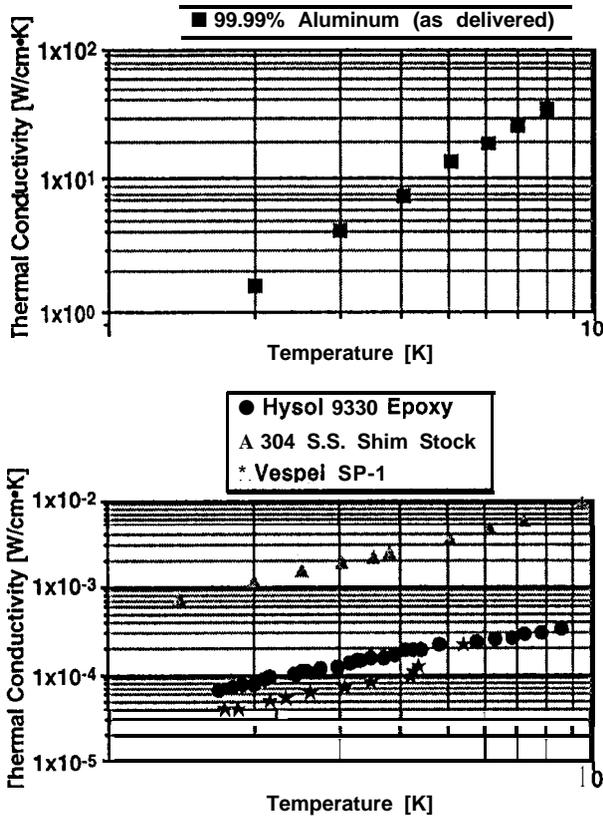


Figure 6: Measured thermal conductivity of materials used in prototype CTP construction

In addition, the interior surface and dimensions of a completed cell have been examined with optical and scanning electron microscopy. The surface roughness and probe spacing accuracy and precision were found to be within the allowed variation.

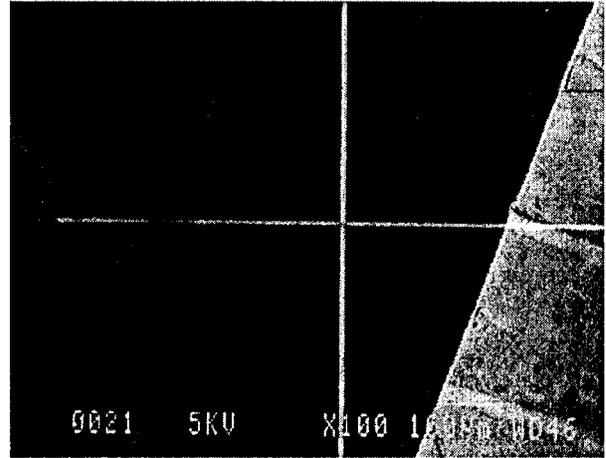


Figure 7: Scanning Electron Micrograph of a sectioned Vespel sidewall cell.

A shake test was performed to verify the mechanical integrity of the DYNAMX probe cell. The applied levels shown in the figure are based on typical STS launch levels modified by the transfer function of the cryoprobe which amplifies certain frequencies. The transfer function is based on a NASTRAN (finite element) model of the cryoprobe that was developed for the LPE flight. The different acceleration levels in the two directions are based on the different response of the cryoprobe in the two directions (longitudinal and lateral). The cryoprobe has a lateral bending mode near 55 Hz. Although the levels for these tests are quite severe, the cell survived easily and was found to remain leak-tight to superfluid helium after the shake. Crude estimates predicted factors of safety for the epoxy joints much greater than 5, so there was no expectation of structural failure. However, prior to the test, there was uncertainty about whether or not the joints would remain superfluid leak tight. The shake was performed with the cell surrounded by gaseous nitrogen at temperatures below 100 K. In cooling from room temperature, most of the deformation occurs by this temperature,

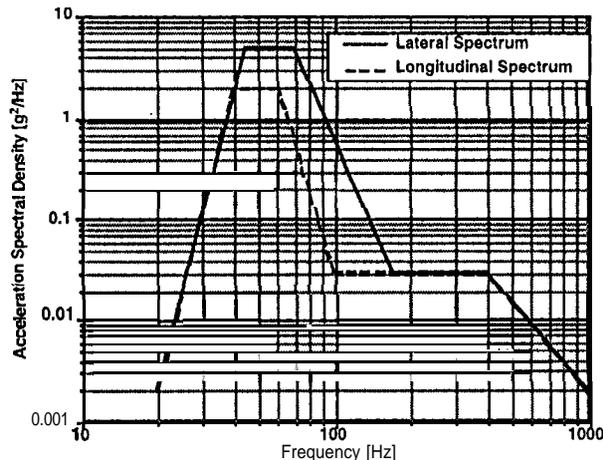


Figure 8: DYNAMX cell qualification shake levels

Performance characterization obtained through data collection with the prototype units has led to modifications of the original design. The probe foil implementation has been changed to meet the temperature precision requirement. Noise in the type of high resolution thermometry used is well modelled by the fluctuation dissipation theorem where the noise in a one Hertz bandwidth is given by

$$\sqrt{\frac{2k_B T^2}{\pi C} \tan^{-1} 2\pi\tau} \approx \sqrt{4Rk_B T^2}$$

Here  $k_B$  is Boltzman's constant,  $T$  is the temperature, and  $\tau = RC$ , where  $R$  is the thermal resistance along the path from the helium to the thermometer and  $C$  is the heat capacity of the thermometer. The probe foils have been increased from 25 to 50  $\mu\text{m}$  in thickness to reduce the thermal resistance between the helium and the probe foil due to Kapitza resistance, and the foils are annealed to further reduce their thermal resistance so that the required precision can be obtained.

It was also discovered during performance characterization obtained through data collection with the prototype units that the reentrant structure for supporting the thermometers contributed to sensitivity to microphonics in the thermometer readout instrumentation. A mechanical support system utilizing Kevlar thread<sup>5</sup> has been designed which should provide thermal isolation of 10%/W between elements about 2 cm apart,

## Summary

We have successfully designed, fabricated, tested and integrated into the PI's cryoprobe prototype Critical Thermal Path units that:

- Interfaces with the Principal Investigators's existing equipment
- Allows for design iteration as requirements are better understood
- Can be adapted to a design that will fit within the existing JPL Low Temperature Platform
- Will perform with the required performance in terms of sub nano Kelvin thermal, micrometer spatial, and sub nanoWatt heat flux accuracy and precision,
- Utilizes a cell implementation that can withstand launch load vibrations.

## Acknowledgment

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## References

- <sup>1</sup> See "Low Temperature Microgravity Physics Space Station Facility", R.M. Ruiz, U. Israelsson, M. Lee, and R. Reinker, 34th Aerospace Sciences Meeting, Reno, 1997.
- <sup>2</sup> T.C.PChui, P. Day, I. Hahn, A.E. Nash, D.R. Swanson, J.A. Nissen, P.R. Williamson, and J.A. Lipa, *Cryogenics*, 34, 417, (1994).
- <sup>3</sup> See for example A.E. Nash, *Advances in Cryogenic Engineering*, 41, 1979, (1996).
- <sup>4</sup> Analyses conducted by D. Hensinger, Sandia Laboratories using Coyote II, a finite non-linear thermal analysis program
- <sup>5</sup> See L. Duband, L. Hui and A. Lange, *Cryogenics*, 33, 643, (1993) for the basis of our design.