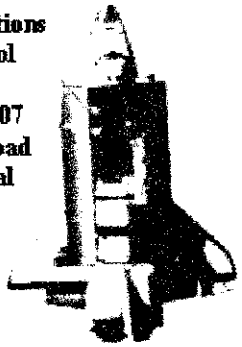
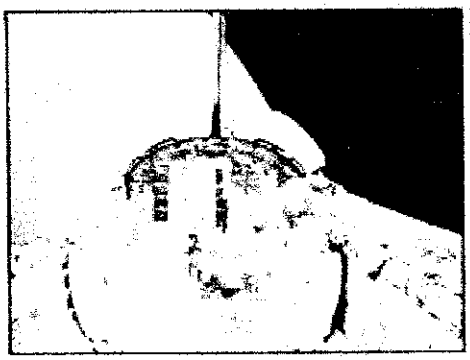
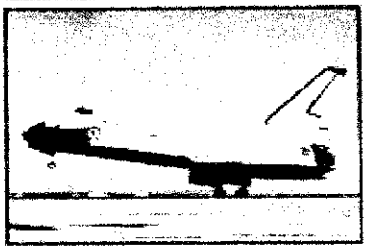
Lockheed Martin Space Operations Sponsored Elementary School Inorganic Crystal Growth Experiments Onboard STS-107 Columbia in the CIBX-2 Payload (Commercial ITA Biomedical Experiments)



Final Report

March 7, 2003





Prepared by:
Valerie A. Cassanto
Student Outreach, ITA, Inc.

Ulises R. Alvarado
Consultant, ITA, Inc.

INSTRUMENTATION ITA TECHNOLOGY ASSOCIATES
Box 671, Exton, PA 19341
(215) 524-1987

This report is dedicated to the brave crew of Columbia, in particular, Mission Specialist Kalpana Chawla, Ph.D. and Mission Specialist Laurel Blair Salton Clark, M.D., who operated the CIBX-2 payload containing Lockheed Martin - sponsored student crystal growth experiments in Space.

Lockheed Martin Space Operations Sponsored Elementary Schools Inorganic Crystal Growth Experiments Onboard STS-107 Columbia in the CIBX-2 Payload (Commercial ITA Biomedical Experiments)

Final Report

March 7, 2003

Presented to:
Robert R. J. Mohler, Ph. D.
Lockheed Martin Space Operations

Prepared by:
Valerie A. Cassanto
Student Outreach, ITA, Inc.

Ulises R. Alvarado
Consultant, ITA, Inc.

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I. Introduction

The Lockheed Martin Space Operations Elementary student crystal growth experiments were designed to show the benefits of the microgravity environment and space research on inorganic crystal growth for students. These experiments, led by Dr. Robert Mohler, Lockheed Martin, represent the work of 420 children, mostly fifth graders, from four different schools:

- ❖ LaVance Stewart Elementary (Kemah TX):
- ❖ Sam Houston Elementary (Bryan TX):
- ❖ Eagle Nest School (Eagle Nest, NM):
- ❖ Helen M. Knight Intermediate School (Moab, UT).

Due to the Space Shuttle Columbia Tragedy, as of this date no data from the space samples were recovered. This will be further discussed in section V (Results).

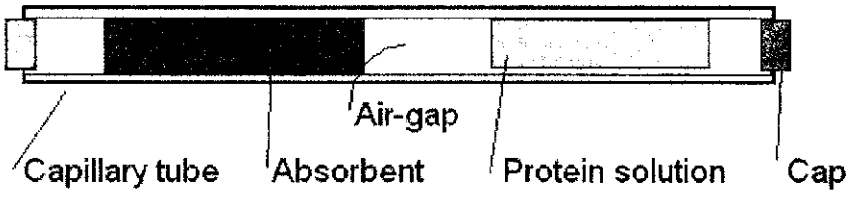
ITA performed the following tasks relative to these Lockheed Martin sponsored student experiments:

- Chose experiment parameters,
- Pre-flight controls of chemicals to fine-tune the final concentrations,
- Performed precision loading of liquids into capillaries and DMDA's for flight,
- Performed precision loading of liquids into capillaries for ground controls,
- Loaded LMA's and DMDA's containing student space experiments into complete payload and performed all necessary engineering to have it flown on Space Shuttle Columbia,
- Performed microscope analysis of ground control crystals and prepared final report.

Presented in this report will be details on the experiment preparation, the capillary crystallization technique, and the results from ground control experiments.

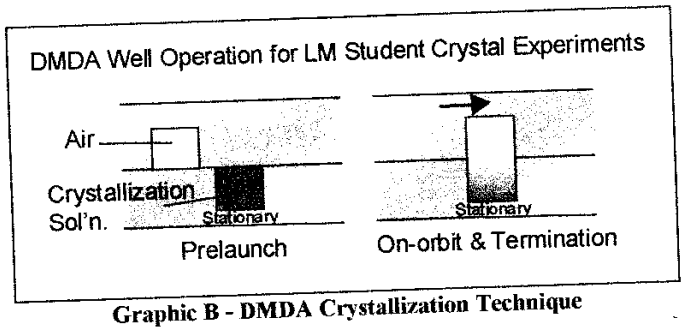
II. Experiment Protocol

Two techniques were utilized for crystal growth: 1) Capillary Crystallization technique in the LMA (Liquids Mixing Apparatus) (see Graphic A), and 2) Fluid evaporation in the DMDA (Dual Materials Dispersion Apparatus), (See Graphic B).



Graphic A - Capillary Crystallization Technique

Two sets of samples (space and ground) were loaded into five (5) LMA capillaries for a 16-day space mission. The goal was to obtain a high quality space crystal and compare this with ground controls.



Graphic B - DMDA Crystallization Technique

One set of space samples were loaded into seven (7) DMDA wells for the 16-day space mission. Once in orbit, the astronaut activated the DMDA blocks to move the Lockheed Martin Student Crystal growth solution into contact with a chamber containing air to start the evaporation process to activate crystal growth.

III. Capillary Crystallization Technique

A vapor diffusion technique for growing crystals of inorganic materials and proteins has proven to be effective in laboratory and space experiments. It uses self-contained capillary tubes where water evaporated from the crystallization solution is absorbed by one or two suitable absorbents. The time-rate of evaporation is programmed and controlled by setting the appropriate volumes and concentrations of the crystallization solution and absorbent, and exposing or interrupting the evaporation process.

In addition to its scientific advantages in crystallography this method is particularly suitable for hands-on educational experiments both in space and the laboratory. The question invariably arises as to why some crystal growth experiments are performed in space. Briefly, the answer is that the process of crystal nucleation and growth is affected by gravitation-induced convection. In some cases the crystals grow faster and more perfect in the absence of gravity.

Description of the Method

A sealed glass capillary tube less than 10 centimeters long, two millimeters external diameter, and 1.6 millimeters internal diameter serves as a compact, self-contained evaporation reactor for each experiment sample. In the basic configuration shown in Graphic A, a quantity of the crystallization solution (protein) is contained in a liquid column, and a volume of air or other suitable gas separates this protein solution from a column containing a liquid absorbent such as sodium chloride. Evaporated medium from the protein solution (usually water) transfers to the air column (or air-gap)

and is absorbed by the absorbent in the opposite column, leaving the air-gap length constant. The evaporation and absorption process increases the concentration of the protein solution and a corresponding dilution of the absorbent. During the evaporation process the air column can be observed to remain constant in length and migrate in a direction from the absorbent column to that of the protein solution. The diameter of the capillary is selected to provide sufficient surface tension to prevent liquid-to-liquid contact between the protein column and the absorbent column.

This method offers the experimenter flexibility in developing the desired rate of evaporation and a time-dependent evaporation profile.

Steps in Planning, Preparing and Executing the Experiments

The following tasks were completed by ITA:

1. Determine the type of inorganic crystal and the concentration of the crystallization solution.
2. Select the absorbent experiment parameters such as volumes of the crystal solution and absorbent liquids, for each experiment sample using guidelines and/or computer model provided by ITA, Inc.
3. Calculate capillary length on the basis of (crystallization solution volume) + (10mm nominal air gap length) + (absorbent solution volume) + (20 mm cap length on each side).
4. Mark the tube with the basic four lengths in 3 above.
5. Load crystallization solution first, using a pipette with a resolution of one microliter or better.
6. Establish air gap length established nominally at 10 mm.
7. Load absorbent using the pipette, leaving spaces for sealing the ends.
8. Leaving the crystallization solution side engaged in the pipette, seal the absorbent end of the capillary with silicone grease, using a no. 18 needle on a 10 cc plastic syringe partially loaded with silicone grease.
9. Remove capillary tube from pipette and seal absorbent end without applying any significant compression on the liquids and air within the tube.
10. Mark the capillary tube with an identification that can be correlated with experiment sample name and date.
11. Measure fluid column lengths and record corresponding volumes, loading date and loading time.
12. Store tube at the prescribed temperature preferably secured on a board, appropriately labeled.
13. Repeat procedure steps 1-12 for all the samples to be tested.
14. Inspect the grown crystals using medium magnification optics.
15. Prepare a report on the findings.

Chemicals Used in the Crystal Growth Experiment

Inorganic Crystal Chemicals:

- Potassium aluminum sulfate
- Potassium chromium sulfate

Absorbents:

- Sodium chloride 3.3 molar

Desiccants:

- Glycerol, 25 and 50% concentrations

Procedure for mixing chemicals:

1. Measure 4 cc [proper amount of Crystallization chemicals (Potassium Aluminum Sulfate and] Potassium Chromium Sulfate) [using precision scal
2. Boil 10 cc of Water.
3. [Measure proper amount of boiling water and mix into the measured crystallization chemical using a spoon or stirring device until chemicals dissolve in water.] Add approximately ½ cc of room temperature water (afte water has cooled down to approximately 200 degrees).
4. Let mixture cool to room temperature.

V. Results

STS-107

Results from the spaceflight were dependent on the payload being returned. However, we do know that in the case of the DMDA samples for this experiment, all units were activated at the designated time and that all equipment operated well.

As of writing this report, ITA is in the process of trying to obtain the intact DMDA unit spotted in photos and news reports of the Shuttle debris. (See Graphic C).

Graphic C: DMDA (Dual Materials Dispersion Apparatus) found in Nacagoshes, Texas, February 2, 2003. Photo published in February 2, 2003 New York Times

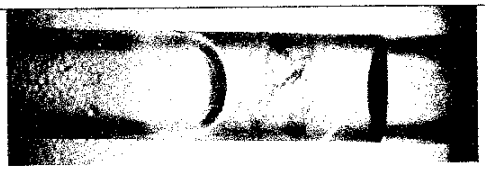
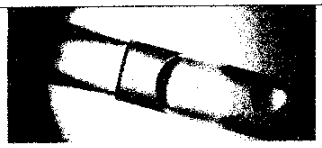
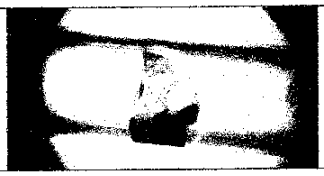
There is a possibility that some of the samples may have survived and that we may be able to gleam some potential results if the samples survived the heat of re-entry.

Ground Controls

Ground controls for the LMA capillary experiments were performed. A total of five (5) capillaries (See Graphic D, below) mimicking the flight capillaries were loaded then observed "post-flight". The detailed results record is shown in Appendix 2 (in the attached Excel Spreadsheet).

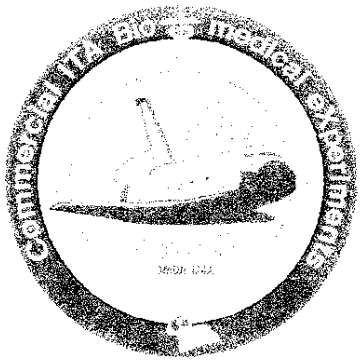
Graphic D – Ground Control Capillaries loaded "pre-flight"

Of the five samples, 3 produced crystals, summarized below:

Capillary Number	Results	Photos
1	Hard Crystallized "plug". No crystals.	
2	1 Large beautiful crystal – pyramid shaped. The best.	
3	2 small crystals growing together.	
4	No crystals.	
5	Small crystal or half a crystal. Completely dewatered.	

VI. Concluding Remarks

Despite the great tragedy of the STS-107 mission, much information can be gained from the ground control experiments which produced good results, as well as the experience the students had in preparing the experiments, analyzing pre-flight experiments, and now analyzing the ground control data. It is clear that the LMA capillary technique for crystallization (originally designed for biological protein crystal growth) is an excellent tool for crystallization. It is also a wonderful way for students to learn the scientific method and become motivated to learn math and science.



Left: Kaplana Chawla (K.C.) with the CIBX-2 payload behind her at Day 14 in the mission.
Right: CIBX-2 Mission Sticker

Appendix 1. Loading Record Pre-flight for LMA Capillaries and Corresponding Ground Controls

Appendix 2. Ground Control Capillaries Results

STS-107 Passive Capillaries Loading Record (Princ. Investigators: Dr. Mohler)

LMA TRAY NUMBER	VIAL NO. & TYPE	CAPILLARY NUMBER	PROTEIN I.D. NUMBER	PROTEIN VOLUME	ABSORBENT I.D. NUMBER	ABSORBENT VOLUME	AIRGAP LENGTH	LOADING DATE	LOADING TIME	PERSON'S NAME
11	D (# 925)	6	Inorg.Crystal	20	50% Glycerol	12 ul	8 ul	1/12/03	22:49	URA/MB
	D (# 925)	7	Inorg.Crystal	20	50% Glycerol	12 ul	8 ul	1/12/03		
	D (# 925)	8	Inorg.Crystal	20	25% Glycerol	12 ul	8 ul	1/12/03		
	D (# 925)	9	Inorg.Crystal	20	3.3 M NaCl	12 ul	8 ul	1/12/03		
	D (# 925)	10	Inorg.Crystal	20	3.3 M NaCl	12 ul	8 ul	1/12/03		
Ground Controls										
n/a	*n/a*	1	Inorg.Crystal	20	50% Glycerol	12 ul	8 ul	1/15/03	14:30	URA/MB
n/a	*n/a*	2	Inorg.Crystal	20	50% Glycerol	12 ul	8 ul	1/15/03		
n/a	*n/a*	3	Inorg.Crystal	20	25% Glycerol	12 ul	8 ul	1/15/03		
n/a	*n/a*	4	Inorg.Crystal	20	3.3 M NaCl	12 ul	8 ul	1/15/03		
n/a	*n/a*	5	Inorg.Crystal	20	3.3 M NaCl	12 ul	8 ul	1/15/03		

Appendix 1

STS-107 Ground Control Capillaries Results (Princ. Investigator: Dr. Mohler)

Capillary Number	Crystal Sol'n. I.D.	Crystal Sol'n. Volume	Absorbent I.D.	Absorbent Volume	Airgap Length	Loading Date	Loading Time	"Unloading" Date	"Unloading" Time	Remarks	*Photos Taken 2/25/03* Photo Roll #	Photo Frame #
1	Potassium Al. & Pot. Chromium Sulfate	20	50% Glycerol	12 ul	8 ul	1/15/03	14:30			Hard crystallized "plug". No crystal.	1 2	18-20 14
2	Potassium Al. & Pot. Chromium Sulfate	20	50% Glycerol	12 ul	8 ul	1/15/03				1 Large crystal - pyramid shape	1 2	9-17,22-23 16-23
3	Potassium Al. & Pot. Chromium Sulfate	20	25% Glycerol	12 ul	8 ul	1/15/03				Small crystal (or two crystals growing together?) in fluid	1	24-25 12-13
4	Potassium Al. & Pot. Chromium Sulfate	20	3.3 M NaCl	12 ul	8 ul	1/15/03				No crystals. No de-watering occurred	1 2	21 11
5	Potassium Al. & Pot. Chromium Sulfate	20	3.3 M NaCl	12 ul	8 ul	1/15/03				Crystal-pyramid shape half a crystal?. Completely de-watered.	2	6-8, 15

Appendix 2