An Investigation of Crystallization in Microgravity

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The Crystallization in Microgravity Experiment payload (CRYME) was built to investigate the validity of microgravity crystalline experiments on sounding rockets. A supersaturated solution of Sodium Acetate Trihydrate (SAT) was used to analyze differences in reaction speed and uniformity between results obtained under Earth and microgravity conditions. To conduct this experiment four wells were constructed out of aluminum. Two 42 mL wells housed the supersaturated solutions, and a seed crystal was introduced via linear actuator to initiate a crystallization reaction. In addition, two 2 mL wells were also constructed to serve as control trials for the experiment. Results of this experiment were recorded on board with a high definition camera and telemetry data was downlinked to a ground station by means of a low resolution camera. The results of this investigation contribute to a wide range of fields, specifically pharmaceutical, semiconductor, and crystallography research.

I. Introduction

The RocketSat-IX team from the Colorado Space Grant Consortium built the Crystallization in Microgravity Experiment (CRYME) payload to investigate the validity of microgravity crystalline experiments aboard sounding rockets through the RockSat-X program. RockSat-X is a national program in partnership with NASA that gives universities around the country the opportunity to launch an experimental payload on a sounding rocket. NASA provides an Improved Terrier-Malemute sounding rocket and launches the rocket from the Wallops Flight Facility (WFF) in Virginia. The Air Force Research Laboratory (AFRL) sponsored the launch this year for the CU team to conduct an experiment involving crystallization in microgravity. BioServe also mentored the team and provided facilities for chemical testing.

Crystal growth is a growing enterprise in the medical and technology industries. Previous experiments completed on the International Space Station have proven that crystals are able to grow larger and more pure when grown in a microgravity environment. This is particularly useful in the production of semiconductors and medicine, as they can be formed into a more potent and pure form. Currently crystal growth research is conducted on either the Space Station or on the Vomit Comet. However, testing on the International Space Station is difficult and expensive and the Vomit Comet only provides a small window of time in which to conduct experimentation. For this reason, sounding rockets could potentially provide a reasonable balance between time, availability, expense, and difficulty of the environment for companies interested in experimenting with crystallization production in space.

CRYME investigated a supersaturated solution of sodium acetate trihydrate (SAT). SAT was used to analyze the differences in reaction speed and uniformity between results that were obtained under Earth and microgravity conditions. Four wells were constructed to house the SAT solutions. Two of these wells were equipped with a linear actuator system to initiate the reaction and two wells served as control trials. The results were captured by both a high definition and low resolution camera. High definition video was stored onboard while the low resolution camera sent still images to the ground station via telemetry.

The requirements placed on the payload from WFF mainly concerned the size and weight of the payload. The requirements were a 32-pound weight limit contained within an 11-inch diameter by 11-inch height volume. Due to the nature of the experiment, an additional requirement was requested to provide an extra seal around the wells holding the chemicals for safety concerns. The rocket produced by Wallops also provided telemetry and power lines to each payload, and specific requirements had to be met for each line that was used.

II. Payload Design

To meet the requirements listed above and complete the mission, the payload was designed with two shells to seal all hardware. The first shell housed two large chemical wells, two small chemical wells, two cameras, and the
triggering mechanisms. Electrical hardware and internal batteries were housed inside the second shell. The reaction in the large wells was initialized using seed crystals, which were introduced into the solution by a linear actuator and the smaller wells were used as control experiments to determine if the rocket vibrations could potentially trigger the reaction without the presence of seed crystals. A combination of external rocket power and internal batteries was used to power the payload.

A. Chemistry

Initially three chemicals were considered for their crystallization properties: sodium acetate trihydrate (SAT), succinonitrile (SNC), and elemental bismuth. During testing of the chemicals, the melting temperature of bismuth was found to be too high to be considered feasible for the project. Although SNC was compatible with the design requirements, research into the microstructure of the hardened compound was determined to be amorphous. Without a clear crystalline structure, SNC was eliminated as a possibility for achieving the mission objective. SAT was thereby selected and analyzed for flight.

SAT is a common chemical that is generally used in potato chip flavoring, but also has the useful property of being able to make a supersaturated solution. A supersaturated solution is an unstable mixture in which too much solute is dissolved in a solvent by heating a solvent to dissolve as much solute as possible before cooling back to room temperature. When a seed crystal is introduced, the solute falls out of the solution and rapidly crystallized leaving no dissolved solute. The rapid solidification was ideal for a sounding rocket flight due to the minimal amount of time in microgravity that was available.

However, depending on the temperature and concentration of the solution, the reaction proceeded slightly differently. As the concentration decreased or the temperature increased, the reaction spread more slowly and less uniformly, creating fingers of crystal that spread across the solution. An example of these “fingers” can be seen below in Figure 1. Based on testing, an ideal concentration of 4:1 SAT to water ratio and a temperature of 37°C was determined to give the best reaction for analysis of the difference between full gravity and microgravity conditions.

![Figure 1. SAT reaction with "fingers"](image)

Many trials were performed to test different methods to trigger the reaction. Initial research lead to the belief that the reaction could be triggered using temperature differentials or shockwaves. After extensive research and through trial and error it was determined that only the introduction of a seed crystal into the solution could initiate the reaction. Articles received from the HotSnapz Reusable Hand Warmer Company, described how seed crystals were used to create reusable hand warmers and supported these realizations. The articles also described how a seed crystal could be stored in a small imperfection in a material and then released if the material was flexed. For example, one design of the wells used a silicon gasket reinforced with a fiberglass layer. The fiberglass layer contained many small cavities that stored seed crystals between tests and caused crystallization to begin prematurely.

B. Structures

The structural design of the RocketSat IX payload was significantly dependent on the chemistry subsystem so the design was constantly adapting to meet the new discoveries. The payload required individual housings for the multiple Sodium Acetate Trihydrate (SAT) solutions, electrical components, low and high definition cameras, and the payload portion above the base plate. It was determined that a water and vacuum seal would be crucial in providing protection
for internal components from reentry conditions, splashdown and launch, as well as protecting the environment from potential leakage of chemicals from the wells.

The rectangular shell was placed on top of the heat sink and maximized the size of the solution containers while accommodating for both Hack HD and LinkSprite focal length, container height, and camera housings. The shell was manufactured out of a solid block of T6-6061 Aluminum which had 1/8” thick walls and included a margin for electrical wiring and other components. The top shell was a total height of 6”, 1/2” of which was used to helicoil the camera housings into an additional section centered above the shell. Due to the rigor that all seals would undergo throughout flight, both shells were sealed using High-Temp Silicone Rubber gaskets which were placed on the outer flanges and compressed by an array of 1/4” diameter, 20 thread/in bolts. The top shell was attached to a heat sink 1.1” in height whose dimensions matched those of the keep out zone. Similarly, the bottom shell was helicoiled into an additional aluminum base plate below the payload 0.5” in thickness that also allowed for batteries, and PCB standoffs. Both shells were then fully integrated into the base plate with 3” through bolts from either side. The fully integrated SolidWorks model can be seen below in Figure 2.

The rectangular containers within the shell were chosen to accommodate the size of the top shell while maximizing the area for crystallization spread. A total of four wells were flown, two of which were 2.5” X 2.75” and .750” in height and contained 2mL of SAT versus 38mL for the remaining two wells, 5.0” X 2.75” and 1.25” in height. All four wells used 1/8” transparent polycarbonate sheets which compressed between two hollow rectangular neoprene rubber gaskets and fully placed within the aluminum wells. These wells can be seen below in Figure 3.

A total of eight Peltier Devices were used as both a heating and cooling mechanism and were placed into grooves machined into the heat sink and placed directly beneath the wells. All four wells were fastened onto the heat sink by
helicoils and individually compressed together in the same manner but with the use of both shorter helicoils and bolts.

![Figure 4. Full integration of heat sink](image)

A custom manufactured plunger sat within a permanently epoxied tube positioned through the polycarbonate sheets of the two largest wells. An actuator motor plunged a rubber stopper a distance of 1cm, thus exposing seed crystals to the supersaturated solution and initializing crystallization. The plungers were constructed from a polyethylene plastic and threaded into a portion of the Firgelli actuators. In order to maintain the actuators stability, two separate housing plates were permanently attached around the components and fastened to the SAT containers by two threaded rods. The final SolidWorks model can be seen below in Figure 5

![Figure 5. Principle views of linear actuator configuration](image)

The entire payloads wiring configuration was compiled by the use of hermetically sealed connectors, one 42 pin connector on the top shell and two on the electrical housing. The electrical housing was fully vacuum sealed after incorporating a bolt and gasket that was compressed by a nut positioned on the outside of the shell. This design was used to cover a redesigned machining plan. The fully assembled payload can be seen below in Figure 6.
C. Electrical

Microcontrollers were used to communicate with each component in our system and to allow for control of the payload via software. The principle components in our system included motor drivers, Peltier devices, linear actuators, a high resolution camera, a low resolution camera, an accelerometer, and several temperature sensors. The motor drivers regulated the polarity of the Peltier devices and linear actuators. The linear actuators introduced seed crystals into the SAT solutions after the rocket achieved apogee and initiated the reactions. The Peltier devices cooled the wells after the SAT reaction to increase the probability of crystal survival through the heat of reentry. Internal batteries supplied power to the Peltier devices, as these devices had a large current draw beyond the capabilities of the power supplied by the rocket. Additionally, the 28V line supplied by the rocket was converted to 5V using a small transformer IC to power the rest of the system. A functional block diagram of the electrical system can be seen below in Figure 7.

The electrical system was comprised of two printed circuit boards (PCB). One board contained all voltage regula-
tion and motor drivers. The other board controlled the communications with the rocket, the operation of the cameras, and stored all data taken by the system. Video from the high resolution camera was stored on an external SD memory card housed in the camera itself. The pictures from the low resolution camera were sent to the second PCB, where the data was stored on a microSD card and was also sent to the WFF via telemetry. An accelerometer on the second PCB took g-loading measurements which were also stored on the internal memory of the system. Finally, temperature sensors were placed next to the chemistry wells and recorded their thermal signature and was also stored in the internal memory system.

III. Testing

A. Chemistry

Chemical testing began early on in the semester, as the results drove the design of the entire payload. Bismuth was tested on a heating pad to determine the approximate temperature the metal would require to melt and solidify as desired. It was determined that bismuth required a far too much heat and did not cool slowly or uniformly enough during the tests to be a possible flight chemical.

SCN was a simple chemical to test, as it only required about 60°C of heat to melt and solidified quickly when cooled below this temperature. Tests were first performed on a heating pad set at a specific temperature and was timed to determine heating and cooling times. After the Peltier devices were obtained by the electrical team, SCN was melted and cooled in a prototype well via the Peltier devices. The heating and cooling times were determined to be reasonable but the difficulty of SCN was determining the crystalline structure. A simple optical microscope did not provide enough information, and the team could not interpret the results well due to inexperience. Oscar Resto generously offered to use his Scanning Electron Microscope (SEM) to obtain pictures of the microstructure. This proved that SCN solidified in an amorphous manner, making it impossible to use to achieve the mission objective.

SAT testing began first with simple solutions in beakers, heating the water first on a heating pad and then cooling the supersaturated solution in a refrigerator. A seed crystal was then introduced to the solution to view the speed and uniformity of the reaction and to determine the ideal conditions for the rocket flight. The reaction had to proceed slow enough that analysis of the uniformity and speed of the reaction could be easily seen. The presence of the fingers was desirable because of the hypothesis that they would decrease in size and amount during microgravity conditions. After multiple tests, the team discovered that the solutions needed to be covered from the atmosphere because even small dust particles acted as seed crystal substitutes that caused premature reactions to trigger.

After testing the solution in the beakers, it was tested in a prototyped aluminum chemical well. The well was required to be very smooth and extremely clean to prevent the solution from premature crystallization. The many different triggering mechanisms were tested both in beakers and in the well. The SAT testing also proved to be difficult due to the pressure created in the well when the solution was heated. Because there was very little air inside the container, the liquid expanded when heated causing immense pressure on the gasket seals and the well. The liquid broke the seals due to this pressure, requiring a new process for filling and cooling the wells.

Premature crystallization of the chemicals was a constant problem. Two wells were procured for launch that remained in their liquid form for launch. However, a clear procedure was never found to recreate these results and to prevent the solution from crystallizing. More testing would be necessary to pin-point the process that created these successes, but the time line of the project prevented this analysis.

B. Structures

In order to test the structural integrity of the payload, simulation loading of both the external shell and electrical housing was conducted using SolidWorks models, which subjected the shells to both vertical and compressive forces. The shells withstood 25 gs to forces acting perpendicular to the walls, simulating suppressed g-loading. They withstood 50 gs in the z-direction which simulated instantaneous g-loading. In both cases an FOS of 1.5 was used and the structure successfully exhibited minimal or insignificant deformation when experiencing touchdown forces and potential collisions prior to, during, or after launch. The results of these simulations can be seen below in Figure 8. A fully integrated payload was also placed on a simulation rocket at Wallops Flight Facility and underwent vibration testing. All structural components and subsystem hardware remained secure and was thus demonstrated to be fully functional.
Pressure testing was also conducted using a Bell Jar within the Colorado Space Grant facility in which the fully integrated payload was situated and exposed to space-like vacuum conditions for two hours and was successfully able to remain within 10 percent of the initial pressure of one atmosphere.

The other structural component which was rigorously tested was the linear actuators used to plunge and expose seed crystals to the saturated solutions of the wells. The actuator braces were adjusted and ran through a full simulation of crystallization in order to determine the best height off the wells to perform the actuation.

C. Electrical

In order to test the electrical system, several electrical full mission simulations were conducted in order to verify power needs and functionality. These simulations ran all of the electrical components as they were run during flight. From these tests, the team verified that the motor drivers powered the Peltier devices correctly, that the timing of the linear actuators was correct, and that all data was stored properly. Power measurements were also taken to ensure that the system did not draw more power than was allotted to the payload.

From these tests, it was concluded that all power needs fell below the maximum allotted by the rocket and that the internal batteries supplied sufficient power for the Peltier devices. Additionally, all data was correctly stored and formatted on the microSD cards and were accessed after each test. However, the method of saving on the HackHD high-resolution camera was not verified, which led to a memory problem experienced during launch. Additionally, time restrictions did not allow for the payload to be assembled and isolated for any long period of time. Such a test would have lead to the discovery of a faulty component by measuring the voltage drop across all batteries.

IV. Results

Before launch, Wallops regulation called for two sequence tests to be run on the rocket for integration and then the payloads were to remain unpowered until launch occurred. However upon arrival for integration, it was discovered that this was not the case. The payloads were actually powered a total of 16 times during the integration process at Wallops. The team had not prepared nor tested the payload for this sequence of testing but it was determined that this would not adversely affect the payload as long as it was not powered for more than ten minutes.

The rocket was launched and recovered successfully out of WFF on August 13, 2013. Post-launch investigation of the payload showed that both housings held a dry seal and all hardware housed within sustained no damage and all internal electrical connections were intact. However, all four wells remained un-crystallized, contrary to expectations.

To prove if the seed crystals had been loaded properly, the linear actuators were connected to a 5V power supply and the reactions in both wells were initiated. This verified that the seed crystals had been loaded into the plunger correctly and therefore the actuators did not run during flight. To verify this conclusion, video from the HackHD was analyzed to determine the events that occurred during flight. However, due to the testing procedures conducted during integration, the external memory in the HackHD was filled to capacity and thus no video was recorded during flight.

Without the high-resolution video to reference whether the wells crystallized during flight, the team turned to the telemetry pictures taken by the low-resolution camera. The pictures verified that the wells remained un-initialized throughout the entire flight and the triggering mechanisms never fired. It was later determined that the motor driver that controlled the linear actuators had a small short. Over the duration of pre-launch testing, the battery connected
to this motor driver to initiate the chemical reaction was drained to 2.14V. This voltage was too low to run the linear actuators, thus no crystallization occurred during flight.

Data analysis was also conducted on the payload using a MatLab script developed to analyze the logger files recorded from the flight. It was quickly discovered that the accelerometer had not been calibrated properly prior to launch. Thus the raw voltages obtained through the telemetry lines were required in order to re-calibrate the accelerometer data. After this re-calibration, Figure 9 was obtained by plotting the data. As seen in the figure, the average reading on the accelerometer prior to launch was .985 gs as expected. At apogee the average reading was only .302 gs, thus implying that had the experiments been initiated they would have been conducted at roughly a third of the gravity as those conducted on the ground. The max G loading calculation however was limited by the constraints of the accelerometer. The accelerometer chosen for the mission was only capable of reading a maximum of 6 gs in any of the x, y, or z directions. After analysis it was determined that the z direction maxed out, failing to read as high of a max G loading as expected.

![Figure 9. G Loading throughout flight](image)

V. Conclusions

The objective of this experiment was to compare crystallization results obtained in Earth gravity and microgravity. However, the failure to initialize the SAT inhibited the realization of this objective. Several conclusions can still be drawn from this experiment. The hypothesis before flight was that the vibrations of launch would not crystallize a solution of SAT and only a seed crystal could trigger the reaction. This hypothesis was validated by post-launch investigations. Visual inspection and low-resolution images proved that the SAT solutions remained un-crystallized throughout flight. Activating the mechanical triggers after launch further verified that a seed crystal is the only known way to initiate the reaction.

The failures experienced were recreated to find the source of each issue. The faulty motor driver was proven to cause the critical failure of the mission. The HackHD external memory filled to capacity after being powered repeatedly during pre-launch testing, but this issue could have been prevented in the software. These issues were addressed and verified as corrected using full mission simulations post-launch.

The payload held structural integrity throughout flight and none of the hardware sustained damage. Both external shells held dry seals and all electrical connections remained intact. Post-flight investigation showed that without the faulty motor driver and unexpected pre-launch testing procedure, the payload would have fulfilled all expectations. By replacing this failed component and restoring the payload, it is ready for another launch opportunity. To verify the plausibility that a sounding rocket payload can be used to investigate crystallization, this payload can be flown again.
Appendix: A
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Bibliography

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