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**BUBBLE FORMATION IN MICROGRAVITY**

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## 1. Introduction

Almost all of the experiments dealing with protein crystal growth and with growth of crystals from solution require complicated fluid handling procedures such as filling of containers with liquids, mixing of solutions and stirring of liquids. Such procedures can be accomplished in a straight forward manner when performed under terrestrial conditions in the laboratory. However, in the low gravity environment in space such as on board the Space Shuttle or Earth orbiting space station, these procedures are no longer straight forward. Under terrestrial conditions, liquids are normally positioned below the gas due to the buoyancy effects of Earth's gravity. Consequently any gas bubbles that are entrained into the liquid during a fluid handling procedure will eventually migrate towards the top of the vessel where they can be removed. In a low gravity environment any folded gas bubble tend to remain within the liquid bulk due to the absence of the buoyancy force resulting from the diminished gravity.

An extensive experimental program was initiated for the purpose of understanding the mechanisms leading to bubble generation during fluid handling procedures in a microgravity environment. Several key fluid handling procedures typical for PCG experiments were identified for analysis in that program. Experiments were designed to specifically understand how such procedures can lead to bubble formation. The experiments were then conducted aboard the NASA KC-135 aircraft which is capable of simulating a low gravity environment by executing a parabolic flight attitude. However, such a flight attitude can only provide a low gravity environment of approximately  $10^{-2}g_0$  for a maximum period of 30 seconds. Thus all of the tests conducted for these experiments were designed to last no longer than 20 seconds.

Several experiments were designed to simulate some of the more relevant fluid handling procedures during protein crystal growth experiments. These include submerged liquid jet cavitation, filling of a cubical vessel, submerged surface scratch, attached drop growth, liquid jet impingement, and geysering experiments. To date, four separate KC-135 flight campaigns were undertaken specifically for performing these experiments. However, different experiments were performed on different flights.

Some of these experiments have been thoroughly analyzed elsewhere including the the vessel filling experiment, the surface scratch experiment and the submerged liquid jet cavitation experiment. The results from these tests are found in Antar and Kornfeld (1995). Below we describe some of the other experiments including the jet impingement, the attached drop growth, and the geysering experiments.

## 2. Jet Impingement Experiment

This experiment was designed to investigate the spread mechanism of a liquid jet over a solid surface upon impacting the surface. Jet impingement is a fluid handling procedure that may develop during filling of a vessel when the inflow rate is very high.

It is obvious that the liquid spread over the solid surface resulting from a jet impact in low gravity environment is entirely different from a similar incident under terrestrial conditions. In a terrestrial laboratory the spread mechanism depends very strongly on the orientation of both the jet and the solid surface. Such dependence on geometry is absent in low gravity environment. Another objective of this experiment was to delineate the conditions under which some of the impacted liquid rebounds from the solid surface in the form of droplets that subsequently fold gas bubbles into the liquid bulk.

The test cell for this experiment comprised of a plexiglas cubical box with the dimensions of 2.5 cm on each side. A hole was drilled at the center of one of the cube faces, through which a hypodermic needle was inserted. The needle end was positioned at a set predetermined distance from the opposite wall. The liquid jet in each test of this experiment was established by injecting liquid through the hypodermic needle. The impact of the jet on the opposite face of the cube and the subsequent accumulation and spread of the liquid on the wall was visually recorded using high speed motion picture camera. The liquid flow rate was achieved using a syringe pump with a preset flow rate setting.

The objective of this experiment was to understand the mechanism of jet impact on a solid wall and the subsequent form of liquid spreading for different jet exit velocities, different liquids and different liquid jet lengths. Two hypodermic needle sizes were used in the tests with 0.16 cm and 0.12 cm ID. Three test liquid types were used for this experiment; distilled water, and a mixture of water with 10% PEG, 20% PEG, and 30 % PEG, respectively. Four different jet lengths were tested 0.5 cm, 1.0 cm, 1.5 cm and 2.0 cm. Flow rates of 20, 30, 40 and 50 ml/min were tested corresponding to jet exit velocities of 25, 30, 40, 45, 60, and 74 cm/sec.

Each test was initiated after the low gravity portion of the aircraft flight parabola was attained by activating the pump and the camera. Most of the tests had a liquid injection period no longer than 5 seconds. Each test was completed during the low gravity period of the parabola. Normally one test was conducted during a single parabola. Also every test was performed at least twice, once with a dry wall initial conditions and another with a wet wall initial conditions. For each test one of the four variables, namely, the flow rate, the needle diameter, the test liquid type, or the jet length was varied while the rest of the parameters were kept the same. This experiment was performed in the second and third KC-135 flights campaigns which took place during June 20 - 24, 1994 and January 30 - February 3, 1995.

The results of the tests for this experiment show that the liquid will always accumulate at the jet impact point with the solid wall. The accumulating liquid took the form of a hemispherical dome with its cap at the jet end and the flat surface against the solid wall. As further liquid is injected, the dome expanded radially increasing both its base and its tip. This form of liquid accumulation was consistent regardless of the jet

exit velocity, the jet diameter, or the type of test liquid used. The liquid spread over the solid surface was manifested, in this case, through an expansion of the base of the liquid dome in the radial direction. This was the accumulation configuration observed whenever the wall was dry.

When the liquid jet impacted a wet wall the liquid spreading configuration varied from test to test depending on the shape of the original liquid film on the solid wall prior to the jet impact. In this case the liquid meandered along the wet region of the wall after some liquid mass accumulated at the stagnation region. However, the liquid meandered only after it acquired some mass and when gravity perturbations were present. In other words the accumulating liquid mass was very unstable to small disturbances when the wall was wet. The meandering liquid mass took very interesting forms and shapes which are not the subject of this experiment. No splattering of liquid was ever observed for all jet exit velocities tested in this experiment.

The flow of the liquid over the solid surface was extremely interesting forming unusual patterns that can only be observed under low gravity conditions. One of the most common patterns observed was the motion of the hemispherical dome along the wet path of the wall as also the merging of accumulating liquid volume with existing volume shapes in the container. The resulting liquid configuration took different shapes depending on the material it merged with. In certain instances the hemispherical drop merged with a horizontally flat gas/liquid interface. This pattern evolved as the liquid accumulated from the jet impact on the wall spread and collided with another liquid in the container was partially filled with liquid. In other instances when the test chamber was empty the hemispherical drop evolved into a quarter spherical at the intersection of two walls of the test chamber.

### 3. Attached Drop Growth Experiment

The purpose of this experiment is to investigate the growth process of a drop attached to a solid surface as liquid is continuously added to the drop. The growth process of the attached drop includes in this case the development of the drop geometry with increasing liquid mass as well as the variation of the liquid/solid contact angle as the drop spreads along the solid surface.

All of the tests for this experiment were conducted by injecting liquid through a hole drilled into a solid surface. The liquid drop was formed by injecting liquid through the hole. The drop subsequently was allowed to remain attached to the surface. The test cell for this experiment comprised of a plexiglas cube of 2.5 cm per side. The hole was drilled at the center of one of the cube faces. The liquid was supplied to the drop using a hypodermic needle attached from the other side of the wall. Four different needle sizes were used for injecting the liquid; 0.16, 0.12, 0.084, and 0.058 cm ID, respectively. Five liquid injection flow rates were used resulting in liquid exit velocities ranging from 1.66 cm/sec to 92.4 cm/sec, depending on the needle diameter. Also, four different

liquid types were used in these tests including distilled water, distilled water solution with 10% PEG, 20% PEG, and 30% PEG, respectively. The growth of the drop was recorded visually using high speed movie camera and film.

Each test in this experiment was initiated by injecting liquid through the orifice in the solid surface at a constant flow rate. The variation of the shape of the drop was recorded visually as additional liquid is injected. Each test was conducted during the low gravity portion of the flight parabola. The liquid flow rates, the test liquid and the pore size were varied for each individual test.

There were 20 different completed successful tests for this experiment over the second and third KC-135 flight campaigns. In all of the tests for which the liquid exit velocity was greater than 40 cm/sec, the liquid issued from the orifice in the form of a jet and consequently a liquid drop did not form at the orifice. For all of the tests in which the liquid exit velocity was less than 40cm/sec a drop formed and developed in almost the same manner for all of the tests except in three cases. In the majority of the tests the liquid drop took an almost perfect hemispherical domed shape immediately upon exiting the orifice with a flat surface against the solid wall . The drop was attached to the orifice in all of these cases and was positioned centrally at that location. As further liquid is added to the drop, the drop expanded radially retaining its hemispherical shape. However, in one test for which the conditions were: 0.16 cm orifice diameter, 4.98 cm/sec for the exit velocity, and 30% PEG test liquid, the hemispherical dome flattened out to a disc covering the orifice as the liquid mass increased beyond a specific value.

In the remaining three cases, for which the orifice diameter and exit velocity were: 0.16cm and 2.49cm/sec; 0.12cm and 2.95 cm/sec; and 0.12cm and 4.3cm/sec, respectively, the liquid took an almost perfect spherical shape upon exiting the orifice with the drop being attached to the orifice. The spherical drop expanded radially with increased injection of the test liquid which was distilled water in all of the three cases. However, the spherical shape became unstable as further liquid was added and subsequently the drop shape changed to a hemispherical dome. The cause of this shape instability could either be due to the effects of the residual gravity or to the increased mass of the drop. The specific cause of this instability could not be identified since that test run was conducted without the benefit of the accelerometer readings being visible simultaneously with the photograph.

No attempt has yet been made at measuring the variation of the solid/liquid contact angle as a function of the advancing liquid front during the growth of the drop. The following general conclusion can be drawn from this experiment. At very low exit velocities, i.e. less than 5 cm/sec the attached liquid drop took a spherical shape. For higher exit velocities a hemispherical domed shape drop was consistently observed.

#### 4. Geysering Experiment

The purpose of the series of tests for this experiment was to validate the critical Weber number criteria at which the free surface is broken by the geysering phenomenon. Previous low gravity experiments have suggested a critical Weber number value of 1.5. The value for the critical Weber number for the geysering experiment were explored by varying the values of some of the parameters that enter into the definition of the Weber number, namely

$$We = \frac{\rho U^2 d}{\sigma}$$

For a specific liquid composition in which  $\sigma$  and  $\rho$  have fixed values, only the liquid expulsion velocity  $U$  and the exit nozzle diameter can be varied in order to cover the desired range of values for the Weber number. The liquid injection velocity  $U$  was varied by changing the fill flow rate through the pump speed. The inlet nozzle diameter  $d$  was varied by using different diameter injection needles. A test matrix was constructed for performing a number of tests in this experiment comprising of four liquid injection rates and two injection needle diameters. The values for these parameters were chosen such that they enveloped the empirically determined critical Weber number value of  $We = 1.5$ . Also since surface tension plays a major role in all free surface phenomena in low gravity environment, three different liquid mixtures were used. The test liquid used in this case was distilled water and water with 10%, 20%, and 30% PEG. This last test item was intended to support the PCG experiments in which water base is used with added surfactants.

The test cell used for the geysering experiment was the same as the one used for the attached drop growth experiment described in section 3. In fact all of the tests for the geysering experiment were performed immediately after completing the drop growth experiment tests by adding more test liquid into the test cell at the end of that experiment. The geysering experiment tests were performed by injecting liquid through the nozzle at a predetermined constant flow rate. The nozzle in all of the tests was covered with a liquid layer of a predetermined depth. The free surface of the liquid layer was observed to determine whether or not, as well as the extent, in which the liquid jet was able to break the free surface. the observations were recorded visually using high speed camera and film.

## 5. References

Antar, B. N. and M. Kawaji (1995) Bubble Generation During Low Gravity Fluid Handling Procedures. AIAA Paper No. AIAA-95-0898.