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BUBBLE FORMATION IN MICROGRAVITY

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INTRODUCTION

A well known problem that has plagued microgravity materials processing and bioprocessing experiments for a very long time is the formation of undesired bubbles during procedures involving liquid transfer and mixing. It is conjectured that often bubble appearance is not due to simple mechanism leakage or lack of proper liquid degassing, but due to a phenomenon associated with microgravity environment. In a number of reported incidents, liquid handling operations performed on flight hardware in 1-g result in no bubble formation, while bubbles were observed when the same operations were performed in microgravity.

As an example, in the recent USML-1 shuttle mission a number of fluid experiments were adversely affected by unexpected and unexplained bubble formation. The Nucleation of Crystals from Solution (NCS) experiment experienced bubble formation in its solution reservoir which greatly interfered with the transfer of solution to the nucleation chamber both by agglomerating in the transfer line and by forming voids in the nucleation chamber. These bubbles appeared to have been generated at the end of the fill line, and sufficient number of bubbles were present in the cell to prevent initiation of nucleation on the first experiment run. An exceptional effort was required by the crew to accomplish a successful second run.

The Onset of Oscillatory Thermocapillary Flow Experiment (OTFE) also had problems with bubbles that were formed in the bottom of the heated liquid chamber. However, in this case the bubbles only interfered with the observations of the flow patterns and did not affect the experiment itself.

In the Protein Crystal Growth Glovebox (PCGG) experiment unexplained bubble formation was observed while extruding solutions from syringes and filling syringes for the experiments. During filling process, these bubbles appeared on the walls of initially dry syringes and were removed by manually centrifuging the syringes. This procedure wasted valuable time. During extrusion of solutions from syringes, bubbles appeared at the exits of the syringes. They were suppressed by greatly slowing the extrusion rate, which again wasted valuable time. This problem was especially serious during the solution mixing process required to initiate crystal nucleation due to the excessive time required to perform the procedure slow enough to avoid bubble formation. In another instance bubbles were observed to form during stirring of PCG solution in a polysulfone container with a needle. When the needle tip accidentally scrapped the surface of the container small bubbles were observed. However, upon repeating the same procedure in terrestrial conditions no bubbles were observed.

In all of the USML-1 examples cited above, bubbles appeared to be associated with the flow of liquids in tubes or through orifices, and occurred in of preflight degassing procedures. The specific examples were discussed in post-flight crew debriefing and subsequent personal communications. It is abundantly clear from these examples that formation of undesired bubbles can arise unexpectedly and with great frequency when transferring liquids in microgravity environment.

PROBLEM DEFINITION

The basic difficulties encountered with bubble formation during the low gravity experiments described above are due to the lack of specific procedures for dealing with bubbles. For example, any bubble generated during filling of an empty container with a liquid solution in terrestrial environment will eventually migrate to the top of the liquid where it will finally break the liquid/gas surface. In this instance buoyancy force works to ensure that the liquid bulk remains bubble free. Thus no special considerations need be adopted in this case to inhibit bubble generation during the filling procedure. In low gravity environment on the other hand, any bubble formed during filling will remain stationary within the liquid host and thus negatively impact the experimental procedure. Such a response of the bubbles is typically due to the absence or diminution of gravity induced buoyancy force. It is clear that the liquid transfer procedures involved in the experiments described above will have to be modified when performed in low gravity environment in order to ensure that bubbles are not formed.

Once bubbles are generated within a liquid host their subsequent development and motion in low gravity environment cannot be easily predicted. This difficulty is essentially due to the absence of the buoyancy force in such an environment. Furthermore, thermocapillary effects cannot be exploited for effecting liquid/gas separation in most of protein crystal growth processes due to the absence of the necessary temperature gradients in the liquid solutions. Thus the most desirable option for dealing with bubbles in this case is to prevent their formation in the first place. In order to ensure that bubbles are not generated requires developing special liquid handling procedures. Such procedures will involve both new fluid transfer protocols as well as novel container designs.

Gas bubbles are formed within liquid bulks in either one of two ways. Either by gas entrainment into the liquid through the gas/liquid free surface or by nucleation and diffusion of the dissolved gases within the liquid bulk. Bubbles may be formed by gas whenever a free gas/liquid interface is broken such as might happen when developed waves on the interface are amplified and subsequently broken. This may occur during filling of an empty container with liquid from a supply tank. Bubbles can also develop by nucleation from a vapor pocket seed through either homogenous or heterogeneous nucleation. In the homogeneous nucleation case the vapor seed is formed due to random molecular action within the liquid under the proper thermodynamic conditions. Homogeneous nucleation may occur whenever the pressure within the liquid falls below the vapor pressure or the temperature rises above the saturation temperature. Heterogeneous nucleation is usually initiated by trapped gas pockets in crevices at solid walls which are in contact with the bulk liquid.

When transferring liquid to an empty container under adiabatic conditions and without large variations in pressure, bubbles are most likely formed by entraining gas through the liquid/gas interface. It is necessary in this case to keep the free surface stable and free from waves during the filling process. These requirements may be accomplished by optimizing the liquid injection rate, as well as by the proper design of the inlet nozzle and the geometry of the receiving container.

When using a syringe to transfer liquids, either through injecting liquid into a host bulk or by withdrawing liquid from a bulk, bubbles may be formed through homogeneous or heterogeneous nucleation processes. In the injection case a cavitation bubble may form in the vicinity of the needle tip where the pressure conditions are extreme and crevices may be present in the external or internal walls of the needle. In the liquid suction case the adiabatic expansion within the syringe piston could reduce the pressure well below the vapor pressure and thus allow bubbles to form. In addition since the volume of some of the liquid bulks is small, the possibility could exist for the needle to break the liquid bulk surface allowing gas to be entrained into the syringe. This is possible in low gravity environment since the liquid bulk inside the vessel may not always be in contact with the vessel wall.

APPROACH

In order to understand the fundamental physics in bubble formation in microgravity a number of experiments were designed to study this phenomenon under controlled conditions. Specifically, a number of tests were designed to be performed onboard the NASA KC-135 airplane for the purpose of understanding the conditions that lead to bubble formation during microgravity liquid transfer. The KC-135 airplane provides approximately 40 parabola each of 30 seconds duration with a gravity level of $10^{-2} g_0$ per flight day. Normally, 4 days are available for testing during a flight campaign.

The first flight in this effort took place on March 14 - 18, 1994. In that flight three types of low gravity experiments were performed. In the first set of experiments water with PEG solution was injected, at various flow rates, through different needle diameters into a group of empty containers configured to duplicate the NCS experiment that was carried onboard USML-1. The purpose of these tests is to investigate bubble formation due to breakdown of the liquid/gas interface as the test cell is being filled with liquid. In the second set water with PEG solution was injected into liquid filled container of unique design to investigate cavitation phenomenon for submerged jets. Different needle sizes were used in these tests as well as different flow rates. In third set a number of "scratch tests" were performed by scrapping a needle with a sharp tip over various plastic surfaces that were submerged in water or PCG type solutions. These tests were intended to duplicate specific incidents reported to occur during the Glovebox PCG experiments.

All of the test chambers were constructed from transparent material such as plexiglass in order to allow for visual recording of the fluid motion within the test cells. High speed motion picture cameras were used to record all of the tests. In addition, both temperature and pressure data were collected for the submerged jet cavitation tests. Two pressure transducers were used in that test, one located at the entrance to the jet inlet line, and another at the inner wall of the chamber to record the background pressure. For each experiment a sequence of individual tests were designed and fitted within a test matrix. All of the items in the test matrices for these experiments were successfully conducted during the four flight of the campaign. All of the movie record-

ings were performed at a speed of 400 frames per second to allow for reasonable resolution of the events. fifteen rolls of films were used during that flight. The analysis of the films was conducted at 30 frames per second which was recorded on video tapes for convenience.

The NCS filling tests yielded the most dramatic images. For the high inflow rates the exiting liquid was seen to spread along two paths. One path is along the floor of the cell in the form of a very thick liquid film with a front advancing radially from the exit corner. The second path of the liquid is along the two side walls adjacent to the exit nozzle (the exit nozzle in all of these tests was located at the bottom corner of the test cell). In this case the liquid took the configuration of growing drops along the cell sidewall. Also the liquid was observed to climb along the inlet tube in the cell corner and spread along the top surface. Eventually, the liquid for these tests took a shape similar to an hourglass form. Needless to say that for such a complex liquid configuration the liquid engulfed a large number of bubbles and finally was full of bubbles. In the low flow rate tests as the liquid exited the nozzle it took a spherical form which expanded outwards in the radial direction. The spherical shape become more symmetrical with decrease in the flow rate. A spherical shape for the liquid/gas interface is the theoretical limit for zero gravity. The number of engulfed bubbles in all of these tests decreased with decrease in the flow rate.

The submerged jet cavitation tests did not show any bubbles forming at the jet exit nozzle. This was expected since the hardware design criteria, i.e. jet diameter and jet exit speed, were well below the critical values for cavitation to take place. These tests were repeated in the second flight campaign in which the jet conditions were brought closer to the critical values.

Careful analysis of the scratch tests have lead to the conclusion that the the gas bubbles observed during the shuttle experiments must have been entrained through the gas/liquid free surface with the assistance of the needle. This conclusion was made after the films were slowed down to 6 frames per second and then down to a single frame observations. In other words, the entrainment mechanism occurred extremely fast. Single frame analysis shows very clearly that a fast oscillation of the needle tend to engulf gas from the downstream side of the needle at the liquid/gas interface and then down into the liquid bulk along the needle axis. The gas is initially entrained in the form of a cloud surrounding the submerged portion of the needle which subsequently breaks up into bubbles. The bubbles will then spread throughout the liquid bulk along the motion streamlines of the liquid. There are two characteristic feature of these tests, one is that there exists a lower limit of the needle oscillation speed below which gas is not entrained. The other feature is the speed with which gas is entrained. the gas cloud appears in single frame advance indicating that it occurs in under 2.5 milliseconds.

These tests are far from being complete and further experiments are planned in order to advance the tests to the point of establishing a final conclusion on bubble formation in microgravity fluid handling processes. Also the results from all the tests will indicate the optimum design criteria for microgravity hardware for liquid handling.

CONCLUSIONS

Two KC-135 flight campaigns have been conducted to date which have specifically dedicated to study bubble formation in microgravity. The first flight was conducted during March 14-18, 1994; and the other during June 20-24, 1994. The results from the June 1994 flight have not been analyzed yet, while the results from the March flight have been partially analyzed. Thus the conclusions detailed in here are based on the results from first flight.

In the first flight three different experiments were performed, one with the specific aim at determining whether or not cavitation can take place during any of the fluid handling procedures adopted in the shuttle bioprocessing experiments. The other experiments were concerned with duplicating some of the procedures that resulted in bubble formation namely the NCS filling procedure and the needle scratch of a solid surface. The results from this set of experiments suggest that cavitation did not take place during any of the fluid handling procedures. The results clearly indicate that almost all the were generated as result of the breakup of the gas/liquid interface. This was convincingly demonstrated in the scratch tests as well as in the liquid fill tests.