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# Motion of Viscous Liquids and Air Bubbles in Narrow Gaps under High Gravity 

by<br>\section*{Saugata Dutt}<br>A thesis submitted to the<br>Graduate School-New Brunswick<br>Rutgers, The State University of New Jersey<br>In partial fulfillment of the requirements<br>For the degree of Master of Science Graduate Program in<br>Mechanical and Aerospace Engineering.<br>Written under the direction of<br>Professor Aaron D. Mazzeo<br>And approved by

New Brunswick, New Jersey.
January 2016.

## Abstract of the Thesis

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## Thesis Director:

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Removal of entrapped air bubbles is generally the rate-limiting step in the conventional bonding of laminates. This thesis describes a new technique to bond laminates together by using centrifugal acceleration to drive a liquid adhesive between laminates and remove initially entrapped air bubbles. The authors studied the effect of "high gravity" ( $\sim 50 \mathrm{~g}$ 's generated using a centrifuge) on air bubbles sandwiched in a 100-micron gap. Some of the bubble diameters were larger than the gap between the laminates (factors ranging from 5 to 80 ), which meant there was significant variation of air bubble shapes due to the walls of the gap. The drag on the bubbles came from both the walls and the surrounding liquid, and these sandwiched bubbles traveled more slowly than theoretical spherical equivalents in an unconstrained medium. Comparisons between experimental drag on the flattened bubbles and theoretical drag on spherical equivalents show that the bubbles studied had significant increases in coefficients of drag - factors ranging from nearly 2 to 280. In addition to studying the motion of air bubbles in a narrow gap, this work provides the experimental protocol to laminate thin transparent sheets together with liquid adhesive. Using this protocol with modest centrifugal acceleration, it was possible to remove bubbles from the gaps with 5 minutes of spinning. This process has the potential
to become an effective manufacturing method for laminating various types of component to each other, such as sheets of polyethylene terephthalate (PET), polymethylmethacrylate (PMMA), or ultra-thin glass used in touchscreens or stacked laminates in transparent, impact-resistant windows.

## Acknowledgement

I would like to thank my thesis committee namely Dr. Jerry Shan and Dr. Hao Lin for taking the time to review my work, along with my research advisor Dr. Aaron D. Mazzeo for guiding me throughout the duration of this research. I would like to thank Rutgers students Khalil Meqdad, Stephanie Bajorek and Brandon Linde who contributed to the development of this work. In addition, Sandesh Gopinath and Jingjin Xie were very helpful in setting up the high speed video setup which aided in further understanding the physics behind bubble removal. I would also like to thank the MAE Senior project administrator John Petrowski for helping with the machining related work. Finally, I would like to thank my parents and family who provided strong educational foundation and supported me in all my academic pursuits.

## Table of Contents

1. Abstract ..... ii
2. Acknowledgment ..... iv
3. List of Figures ..... vi
4. Chapter 1: Introduction ..... 1
1.1 Background ..... 1
1.2 Bubble removal under high gravity ..... 4
5. Chapter 2: Experimental design- ..... 7
2.1 Enclosure design ..... 7
2.2 Imaging setup ..... 9
2.3 Image processing ..... 11
6. Chapter 3: Experimental data ..... 13
3.1 Air bubble velocity ..... 13
3.2 Coefficient of drag ..... 19
3.3 Degassing time ..... 21
7. Chapter 4: Air bubble dynamics in narrow confined channels ..... 25
4.1 Dimensional analysis ..... 25
4.2 Coefficient of drag and the confinement effect ..... 28
4.3 Confined air bubbles smaller than the gap ..... 29
4.4 Occluding air bubbles with diameters larger than the gap (Hele-Shaw cell) ..... 31
8. Chapter 5: Conclusion ..... 35
9. References ..... 37

## List of Figures

$\qquad$
Figure 1
(a) Schematic of the experimental setup installed on the centrifuge rotor and the general direction along which the centrifuge rotates. (b) Top-section view of the inside of the assembly. The liquid is shown rushing into the enclosure towards the direction of centrifugal acceleration (denoted by black arrows). The bubbles shown move radially towards the center of the rotor. (c) Cropped-out, section-view of one of the air bubbles denoted in 1B with an encircled ' C '. The bubble is shown to be elongated and can be seen as being cylindrical in shape.

(a) Exploded view of the experimental enclosure. There is an identical enclosure attached on the opposite side of the rotor in order to balance the mass on the rotor. This prevents vibration due to any mass imbalance when the centrifuge is running. (b) A close-up side view of the placement of the syringe tip in the compression assembly (between the PET sheets).

Figure 3-------------------------------------------------------------------------------------------10
(a) The imaging setup with the light source placed below the centrifuge rotor, along with a high speed camera placed directly above the experimental setup. The direction of the light emitted by the light source is also shown, denoted by a yellow arrow. (b) A side-byside comparison of the images taken by the camera and their processed counterparts. The images follow the progression of one particular air bubble in a set of cropped images. This image showcases how large bubbles tended to wobble.

Figure 4-------------------------------------------------------------------------------------------14
Images showing injection of the fluid into the narrow gap setup and the removal of air bubble from it. The first image is taken 8 seconds into the experimental procedure.

Figure 5----------------------------------------------------------------------------------------------16
A set of five bubbles of different sizes that are tracked for their velocity values. The bubble sizes are arranged in the order of their volumes:

Bubble 1: $19 \mathrm{~mm}^{3}$
Bubble 2: $4 \mathrm{~mm}^{3}$
Bubble 3: $1.7 \mathrm{~mm}^{3}$
Bubble 4: $0.5 \mathrm{~mm}^{3}$
Bubble 5: $0.08 \mathrm{~mm}^{3}$
Figure 6------------------------------------------------------------------------------------------------------------------17
Log-log plot of bubble velocities as a function of volume. The velocities included are measured velocities of flattened cylindrical bubbles, while the velocities of the spherical equivalents are predictions based on the volume and calculated gravity. The $x$-axis shows the $\log$ values of bubble volumes and the $y$-axis shows the $\log$ of velocities. The error bars are $\pm 1$ standard deviation.

Plot showing the comparison of the drag coefficient for a set of five air bubbles observed and tracked during the experimental run using a narrow gap setup with calculated drag coefficient of a set of five spherical air bubbles. The cylindrical bubbles are referred to as "Flat" and the spherical ones as "Spherical". The dotted line denotes Stoke's Model.

## Figure 8

(a) A set of five images showcasing how the spin time (in minutes) affects the number of bubble left in the setup after curing. (b) A plot quantifying the mean number of bubbles remaining in the cured setup over the duration of spin time in minutes (the red bars show the standard deviation for each spin time for which four experiments conducted).

Figure 9
(a) Plot of Capillary number (Ca) versus effective bubble diameter (mm). (b) Plot of the $\log _{10}$ (Weber Number) versus effective bubble diameter. (c) Plot of $\log _{10}$ (Bond Number) versus the effective bubble diameter.

## Chapter1: Introduction

### 1.1 Background:

In any industrial manufacturing process involving the lamination of components, removal of air bubbles is one of the most time-consuming and critical steps. Removal of air bubbles is especially important if transparency is a requirement for the products being created, as is the case in the manufacturing of touchscreens and impact-resistant windows. Lamination during the production of touch screens is often carried out manually, and the lack of an automated process can lead to inconsistent results. The assembly and lamination are also carried out over several steps, which can increase the probability of occurring defects [1]. The conventional method for removing bubbles involves degassing using a vacuum chamber or autoclave [1], [2]. This step is distinct from the other processes, such as the application of adhesive, curing or assembly.

Loctite 3193 is a high-strength, UV-curable silicone adhesive produced by Henkel which is good for bonding polymethylmethacralite (PMMA)-based cover lenses to ultrathin glass coated with indium tin oxide (ITO). It is commonly used for lamination of touch panels and displays, and vacuum bonding is the bubble removal technique suggested by Henkel for performing degassing of the adhesive [3]. This technique can be costly, as it requires capital (vacuum bonding machine) and time. The adhesive is also applied manually which further prolongs the entire assembly process and requires intricate care.

The new process of centrifugal lamination developed in this thesis is a step toward performing the degassing and introduction of polymer/adhesive without requiring any human intervention. When using the centrifuge for lamination, introduction of the
adhesive, removal of bubbles, and pre-curing occur in a single step. Thus, this new process will remove some of the error associated with manual processing.

With respect to removing bubbles or degassing volumes for complete forming of 3D parts (e.g., components that have gaps and thicknesses larger than those associated with the gaps between laminates), vacuum-based removal of bubbles is common. A study done by Martinez, et al. [4], suggests that it takes up to 30 minutes using a vacuum pump at 36 torr for polydimethylsiloxane (PDMS) to lose entrapped air bubbles. This problem is exacerbated when removing bubbles from a narrow gap due to the additional drag force on the air bubbles as a result of the narrow channel in which they travel.

Centrifugation is widely used to remove air bubbles from liquids in syringes. A patent by Henkel Technologies, Korea [5] expands on the removal of micro bubbles from adhesives used in syringes for semiconductor-based processing. In a study done by Mazzeo, et al. [6] on centrifugal casting to remove air bubbles during the forming of silicone-based components, they predicted the amount of spin time required to produce bubble-free parts. According to Mazzeo, et al., this technique relies on dissolution and buoyancy to remove air bubbles. They further go on to show that an increased spin speed reduces the bubble removal time.

In the research conducted by Chhabra, et al. [7], they suggest that there is a certain "wall effect" that slows down air bubbles and fluids in narrow gaps, which are still larger than the bubble diameter. They introduce a "wall factor" (f) that signifies the ratio of the velocity of a particle in a narrow wall setup to that of a particle in a channel of a much larger diameter. This comparison helps to develop an idea of the additional drag
experienced by particles in a confined medium over the drag experienced by the same particle in an infinitely wide channel under identical conditions. This wall effect also generally describes effects on spherical particles in a Newtonian media. Considering Chhabra's work in describing wall factors, it can be concluded that the drag experienced by air bubbles increases with confinement.

In a study conducted by Figueroa-Espinoza, et al., they looked at confined gas bubbles between two parallel walls for the inertia-dominated regime which was characterized by high Reynolds and low Weber numbers [11]. They studied the movement of air bubbles under the effect of buoyancy in confined channels, extensively, by conducting numerical simulations and getting experimental confirmation. They went on to show that air bubbles experienced increased drag due to the narrow confining channels. In a separate study conducted by Bairstow, et al. [12], they looked at drag on solid cylindrical shapes in a confined channel and they found the confinement to cause an increase in drag by approximately $60 \%$ compared to the unconfined case. In yet another study by Faxen [13], spherical air bubbles moving in a confined channel were shown to experience an increase in drag force, linearly, with an increase in the confinement factor, which is the ratio of air bubble radius to gap size.

The air bubbles analyzed by Figueroa-Espinoza, et al. were all smaller than the narrow gap that they inhabited. For air bubbles larger than the gap, Hele-Shaw cells are a better approximation of the physical conditions. We found several papers on gravity driven Hele-Shaw cells that talk about the presence of thin liquid films around the air bubbles,
which contribute to the overall drag force. This scenario will be discussed in detail in section 4.4.

### 1.2 Bubble removal under high gravity:

The process of bubble removal occurs after our laminating fixture is placed inside a centrifuge at a fixed distance from the center of the rotor. We used a centrifuge with a fixed speed that spins up to $\sim 750$ RPM, which equates to roughly $83.8 \mathrm{rad} / \mathrm{s}$ of angular velocity. The gravitational acceleration acting on our setup ranges from 44 to 55 times the acceleration due to gravity ( g 's) in the region between the closest and farthest points from the axis of rotation.

The buoyant force that would normally act on an air bubble under natural gravity is enhanced under the "high gravity" generated in the spinning centrifuge. This buoyant force is countered by a drag force due to the flow surrounding the bubble and confinement due to the walls above and below the bubble. This additional drag leads to a lowered bubble velocity when compared to a spherically equivalent bubble (unconfined bubble) where the "narrow gap" constraint does not exist. We also consider the buoyant force to be completely balanced by the drag force with a negligible inertial contribution (Mazzeo, et al. [6]), as we model the speed of a moving bubble in a high-gravity environment.


Figure 1: (a) Schematic of the experimental setup installed on the centrifuge rotor and the general direction along which the centrifuge rotates. (b) Top-section view of the inside of the assembly. The liquid is shown rushing into the enclosure towards the direction of centrifugal acceleration (denoted by black arrows). The bubbles shown move radially towards the center of the rotor. (c) Cropped-out, section-view of one of the air bubbles denoted in 1B with an encircled ' $C$ '. The bubble is shown to be elongated and can be seen as being cylindrical in shape.

Figure 1 shows a schematic illustrating the experimental setup. Figure 1A showcases the general layout of the experimental setup inside the centrifuge. Figure 1B further shows a top-section view of the inner chamber of the experimental enclosure when the centrifuge is spinning. Whereas, Figure 1C is a zoomed-in, side-section view of one of the bubbles in the narrow gap inside the enclosure. This depiction illustrates the fact that some of the bubbles observed in our experimental setup are flattened and roughly cylindrical in shape as they are sandwiched in the narrow gap.

It must be noted that there is a thin film of liquid between the flattened air bubble and the parallel sheets of PET seen in the image in Figure 1C. This fact is brought to light on exploring the preexisting studies on gravity driven Hele-Shaw cells. This is discussed further in Section 4.4.

The bubbles observed in the experimental setup move radially towards the axis of rotation denoted, as the centrifuge spins with an angular velocity $\omega$. The distance between the axis of rotation and the liquid-air interface is 70 mm , as noted in 1 B . This liquid-air interface is approximately where the compressed region (i.e., the narrow gap) begins and we track the velocity, diameter, etc. for each bubble up to this point before it leaves the compressed region.

## Chapter 2: Experimental design

### 2.1 Enclosure design:

The goal of the experimental setup was to bond thin sheets of material to each other, remove air bubbles, and prevent any leakage of adhesive while spinning. The experimental setup used two acrylic plates ( $6.35-\mathrm{mm}$ thick), which we laser-cut to fit within our custom fixture. We also laser-cut two PET sheets ( $62.5 \mathrm{~mm} \times 68 \mathrm{~mm} \times 0.1 \mathrm{~mm}$ ) to go between the acrylic plates. The plates clamped and constrained the PET sheets (shown in Figure 2). We screwed the top and bottom acrylic pieces together with seven pairs of screws, nuts, and washers. We cut out a square section from the front edge of each acrylic sheet, which left the PET sheets uncompressed in the small front section near the syringe. This relief allowed the region between the acrylic sheets in the front to catch the excess fluid coming out of the syringe and temporarily store it. Figure 2A shows this feature.

We attached a syringe to the front of the assembly in order to inject any liquid into the narrow gap (100 microns) between the PET sheets. We also attached a tapered syringe tip to the syringe to dispense any viscous liquids. We used a thin latex gasket ( $0.2-\mathrm{mm}$ thick) to prevent any leakage of the liquid from the rear end of the assembly. A sheet of latex was laser-cut into the desired U-shape for the gasket. The gasket used was thicker than the gap $(0.1 \mathrm{~mm})$ so that we could compress it and seal the rear end and sides of the gap properly. This latex gasket helped contain the silicone oil (which was the viscous liquid we used for some of our analysis) within the narrow gap, preventing it from shooting out from the enclosure.

A


B


Figure 2: (a) Exploded view of the experimental enclosure. There is an identical enclosure attached on the opposite side of the rotor in order to balance the mass on the rotor. This prevents vibration due to any mass imbalance when the centrifuge is running. (b) $A$ closeup side view of the placement of the syringe tip in the compression assembly (between the PET sheets).

We also used stainless steel shims to create the desired separation between the PET sheets. These act as a "hard stop" for the acrylic sheets and dictate the thickness of the gap. We placed the shims along the edge of the setup between the PET sheets so that the gasket isolated them from the liquid entering the narrow gap. A 3D printed fixture was used to hold the syringe in place. Figure 2A shows how the holder clamps on the front of the assembly. We mounted the entire assembly directly on the rotor of the centrifuge using a set of screws attached on the rotor that slip into a set of slotted holes on the acrylic sheet.

### 2.2 Imaging setup:

We utilized high-speed imaging to look at the experimental setup during the centrifugation process to analyze the activity occurring inside it. Figure 3 A shows a model of the imaging setup. The enclosure has an open viewing window. This window allowed light to pass through the contents of the enclosure and over to the high-speed camera placed above it. The contents of the enclosure were also transparent, including the liquid being used, hence we could clearly show the air bubbles moving in the polymer.

Imaging the spinning enclosure effectively, required synchronization of a highspeed camera with the spinning centrifuge. In order to take the images at the desired rate, we triggered the camera using a laser tachometer in order to take one image per revolution at the same location of the spinning disc. The laser bounced off a reflective tape on the spinning rotor, and every time it did so, it sent a triggering TTL signal to the camera's frame grabber. This arrangement made the camera capture a single frame every time it received a signal from the tachometer (once per revolution of the centrifuge).

A


B


Figure 3 (a) The imaging setup with the light source placed below the centrifuge rotor, along with a high speed camera placed directly above the experimental setup. The direction of the light emitted by the light source is also shown, denoted by a yellow arrow. (b) A side-by-side comparison of the images taken by the camera and their processed counterparts. The images follow the progression of one particular air bubble in a set of cropped images. This image showcases how large bubbles tended to wobble.

The objects being imaged were spinning at a high speed, so we kept the exposure time of the camera at a low value ( 24 microseconds) to prevent blurring in the images captured. This short exposure meant that the captured images were very dim and required extra lighting. In order to provide enough light we placed a LED grid under the rotor of the centrifuge and cut two circular holes into the rotor using a CNC machine to allow light to traverse through the rotor.

We additionally used a standard power supply to run the grid of LEDs continuously without intermittent flashing. We selected the power supply based on the power rating and voltage requirements of the LED grid. By rigging the battery compartment, we were able to attach the power supply to the LEDs.

### 2.3 Image processing:

We used MATLAB to process all the images. The image processing code used an algorithm to detect the edges, fill in the completely enclosed geometries with white colored pixels, and further filter out the background to show the air bubbles against a black background. This aided in determining the area and coordinates of the centroid of the air bubbles in the image frame. We used the tip of the syringe (at a predetermined position with respect to the rotor) as a reference point to indirectly calculate the distance of the centroid of bubble from the center of the rotor. We used this estimation since the center of the rotor was not visible in any of the images.

Each image also came with a time stamp, which along with the centroid data, helped us to find the velocity of the bubble. In order to calculate the values for gravity acting at
the bubble's location (which cannot be measured directly) we used the time stamps on the images to calculate the angular velocity of the centrifuge rotor. As mentioned previously, the tachometer sent a triggering signal to the camera once per revolution of the rotor, therefore the differences in the time stamps on the images gave us the time interval between each image acquisition. Thus, the calculated angular velocity, along with the distance of the bubble from the center of the rotor, allowed us to calculate the gravity at the location of the bubble.

Figure 3B shows a bubble moving in the narrow gap over four successive image frames. Each image is displayed next to a processed version of itself in order to show how the data related to area and centroid were collected.

## Chapter 3: Experimental data

### 3.1 Air bubble velocity:

Silicone oil (viscosity of 1000 cSt ) was the liquid used in our experiments for studying the effect of high gravity on the dynamics of air bubbles generated in a narrow gap. Figure 4 shows how the liquid began entering the gap ( $\mathrm{t}=0 \mathrm{~s}$ ). We used a syringe to inject the liquid into the gap, and the acceleration acted on the inertia of the fluid to drive it forward. The syringe had a tapered tip which facilitated injection of the viscous liquid with minimal spilling. As the liquid filled the narrow gap, various pockets of air formed $(t=8 \mathrm{~s}$ and $\mathrm{t}=12 \mathrm{~s})$, individual bubbles formed $(\mathrm{t}=16 \mathrm{~s})$, and the bubbles migrated toward the center/axis of rotation of the spinning centrifuge. Once the bubbles left the compressed region (marked by the nearly horizontal line below the liquid-air interface just below the syringe), the air bubbles moved faster as the gap widened.

The bubble sizes observed (i.e., effective diameter of the cross-section of cylindrical bubbles) ranged from 8 mm to as small as 0.5 mm in diameter. It must be noted that the silicone oil was not agitated before the start of the experiment hence the air bubbles seen are just broken-up pockets of air present within the setup. In the experiments conducted with Sylgrad-184 (discussed in Section 3.3) agitation of the liquid introduces many tiny bubbles into it. These micro bubbles are much smaller in size (some even smaller than the gap size of 0.1 mm ). The micro bubbles were not tracked because the camera used was not able to resolve air bubbles that small. However we have discussed this scenario in detail while citing several studies conducted on air bubbles in a similarly confined space
(this is discussed further in Section 4.3) while including experimental and numerical evidence.


Figure 4: Images showing injection of the fluid into the narrow gap setup and the removal of air bubble from it. The first image is taken 8 seconds into the experimental procedure.

During our experiments we tracked a set of five air bubbles shown in Figure 5 from the point where they appeared in the experimental setup to the point where they left the enclosure at the liquid-air interface. In this way, we were able to measure the velocity of different sized occluding air bubbles seen in this gap. These air bubbles were also analyzed for their drag coefficients. There was an increased amount of drag force acting on the air bubbles seen here due to the confinement induced by the sheets of PET enclosing the liquid. We illustrate the difference in the effect of drag between the flattened air bubbles in a narrow gap setup and spherical air bubbles of the same volume under similar conditions in a wider channel (unconfined setup) in a set of plots (Figure 6) showing bubble velocity. This comparison helps us understand the extent to which an occluding bubble is slowed down and the excess drag that acts on the bubble due to the confinement.

We used a method similar to the one in the study done by Mazzeo, et al. [6], to determine the theoretical velocities of spherical air bubbles of the same volume in a similar, but unconstrained experimental setup. In a paper by Eck and Siekmann [8] they used a similar method of calculating the velocity of gravity driven air bubbles in a confined Hele Shaw cell by balancing the drag and buoyant forces. The velocities calculated are for the spherical bubbles (as opposed to the cylindrical ones observed in our image) in a liquid of the same viscosity and density. Mazzeo, et al. used the expressions for velocity for spherical air bubbles under conditions similar to ours by considering that the drag force and buoyant force on an air bubble balanced each other while neglecting any inertial contribution. They use the Hadamard-Rybczynski relationship for the drag force expression [14]-[16] . We used a similar method to determine the equation for
the velocity of the spherical air bubbles but use Stokes Law (in order to take a conservative approach) instead of the Hadamard-Rybczynski relationship.


Figure 5: A set of five bubbles of different sizes that are tracked for their velocity values. The bubble sizes are arranged in the order of their volumes:
Bubble 1: $19 \mathrm{~mm}^{3}$
Bubble 2: $4 \mathrm{~mm}^{3}$
Bubble 3: $1.7 \mathrm{~mm}^{3}$
Bubble 4: $0.5 \mathrm{~mm}^{3}$
Bubble 5: $0.08 \mathrm{~mm}^{3}$


Figure 6: Log-log plot of bubble velocities as a function of volume. The velocities included are measured velocities of flattened cylindrical bubbles, while the velocities of the spherical equivalents are predictions based on the volume and calculated gravity. The x-axis shows the log values of bubble volumes and the $y$-axis shows the log of velocities. The error bars are $\pm 1$ standard deviation.

The equation for the velocity of a spherical air bubble based on Stokes Law is given by Equation 1 below:

$$
\begin{align*}
& \mathrm{v}_{\mathrm{r}}=\frac{\left(\rho_{\mathrm{a}}-\rho_{\mathrm{f}}\right)}{18 \mu_{\mathrm{f}}} \mathrm{r} \omega^{2} \mathrm{~d}^{2}  \tag{1}\\
& \mathrm{~F}_{\mathrm{D}}=6 \pi \mu_{\mathrm{f}}\left(\frac{\mathrm{~d}}{2}\right) \mathrm{v}_{\mathrm{r}}  \tag{2}\\
& \mathrm{~F}_{\mathrm{Br}}=\left(\rho_{\mathrm{f}}-\rho_{\mathrm{a}}\right) \mathrm{V}_{\mathrm{r}} \mathrm{~g}_{\mathrm{r}} \tag{3}
\end{align*}
$$

The $\mathrm{v}_{\mathrm{r}}$ seen here is the velocity of air bubble derived by balancing the equation for buoyant force and drag force experienced by each air bubble and further solving the equation for
the velocity. The drag force $\left(\mathrm{F}_{\mathrm{D}}\right)$ based on Stokes Law is given by Equation 2 above and the buoyant force $\left(\mathrm{F}_{\mathrm{Br}}\right)$ under high gravity is given by Equation 3. The variable $\mu_{\mathrm{f}}$ is the dynamic viscosity of liquid (which is $\sim 1 \mathrm{~Pa}$-s in our case). The $\rho_{\mathrm{a}}$ and $\rho_{\mathrm{f}}$ are the densities of air and silicone oil, respectively (they come out to $1.27 \mathrm{~kg} / \mathrm{m}^{3}$ and $\sim 970 \mathrm{~kg} / \mathrm{m}^{3}$ ). The d is the diameter of a given air bubble and $\mathrm{V}_{\mathrm{r}}$ is its volume. The position of the air bubble is signified by $r$ with respect to the axis of rotation which spins at an angular velocity of $\omega$. The "high gravity" acting at the location of the air bubble for a given angular velocity is given by $\mathrm{g}_{\mathrm{r}}$, or $\omega^{2} \mathrm{r}$.

The velocities recorded during our experiments were for roughly cylindrical bubble shapes because of the sandwiching effect that the sheets of PET had on them. Therefore, there is a large drag force exerted by the surface of the PET on the bubbles along with the drag due to the surrounding liquid (discussed further in Section 4.4). The comparison of bubble velocities of the five air bubbles observed in the experiment (shown in Figure 5) are compared with those of spherical bubbles of the same volume (calculated theoretically using Equation 1) moving in an unconstrained medium.

We plotted the velocities for the air bubble pairs (shown in Figure 6) in an aggregate bar plot. We numbered the bubbles 1 through 5 in Figure 5 in a descending order based on their volumes. Figure 6 shows a log-log plot of the bubble velocities along with the standard deviation shown as error bars. As discussed earlier the bubble velocity drops off considerably for larger bubbles showing an increased contribution from the constraining walls.

### 3.2 Coefficient of drag:

We used the expression for the coefficient of drag with Stokes' Law as a metric to compare the values of drag coefficient for spherical air bubbles:

$$
\begin{gather*}
C_{D}=\frac{24}{R e}  \tag{4}\\
F_{D}=\frac{1}{2} C_{D} \rho v^{2} A \tag{5}
\end{gather*}
$$

Here $C_{D}$ is the drag coefficient and the Re is the Reynold's number. $v$ is the velocity of the air bubble in question and A is the area of the front face air bubble in contact with the viscous liquid (projected area of both flattened and spherical air bubbles). $\rho$ is the density of the surrounding fluid.

We calculated the drag force using the buoyant force values from Equation 3 (Section 3.1). We simply considered the buoyant force equal to the drag force by considering a negligible inertial contribution, similar to Mazzeo, et al. [6]. We further used the Equation 5 with the drag force to work out the drag coefficient. We used this technique to calculate the drag coefficient for our flattened cylindrical air bubbles and for spherical air bubbles of equivalent volume moving in an unconstrained medium.

The drag coefficients of the flattened air bubbles are compared with spherical equivalents in a plot against their Reynold's numbers in Figure 7. In order to confirm the results for the drag coefficient of the spherical air bubbles, we used Equation 4 and plotted it along the other two data sets in Figure 7.

In order to calculate the Reynold's numbers we used the velocity for the five air bubbles observed in our experiment (seen in Figure 5), as well as, those of the spherical air bubbles calculated using Equation 1 (Section 3.1). The effective diameter of the crosssection of the flattened cylindrical air bubbles were used as the bubble diameter. The Reynold's number for a spherical air bubble was different from the cylindrical ones observed in our experiment, for the same volume. This is due to the discrepancy in their velocities and diameters.


Figure 7: Plot showing the comparison of the drag coefficient for a set of five air bubbles observed and tracked during the experimental run using a narrow gap setup with calculated drag coefficient of a set of five spherical air bubbles. The cylindrical bubbles are referred to as "Flat" and the spherical ones as "Spherical". The dotted line denotes Stoke's Model.

The values for the coefficients of drag for the spherical bubbles calculated using Equation 3 follow the Stoke's model, as expected. However, the plots for the cylindrical air bubbles seem a bit shifted with an increased discrepancy with increasing bubble size. The huge difference in the drag coefficient is clearly due to the confinement of the air
bubbles. The reasons behind the confinement and resulting change in the air bubble dynamics are related to bubble behavior in Hele-Shaw cells. The air bubble dynamics for similarly confined bubbles are discussed further in chapter 4.4.

### 3.3 Degassing time:

A critical factor for a successful and efficient degassing procedure is the total degassing time. In the paper by Mazzeo, et al. [6] increased gravity has been shown to reduce the degassing time by using progressively higher spin speeds. In their study they conduct several experiments and numerical simulations showcasing how the increased spin speed can aid in reducing the degassing time for air bubbles in their setup.

We tried to determine the correlation between the spin time of the experimental setup in the centrifuge and the number of bubbles that remained in the liquid within the enclosure. This relation allowed us to predict the amount of time required for degassing in a narrow gap setup similar to ours.

For our experiments we used Sylgrad-184 as a curable liquid to conduct our degassing experiments. We prepared the sample by taking ten parts of the polymer to one part of the curing agent, by weight, in a container and mixing them together vigorously. We needed the two components to react sufficiently enough for the entire sample to cure in due time (roughly 6 hours). When the two components were stirred together, however, we introduced many tiny micro bubbles in the liquid. We experimented with spinning our enclosure with the Sylgrad-184 mixture loaded into its syringe over several different durations of time to establish the total degassing time.

A total of twenty samples were spun for $1,2,3,4$ and 5 minutes of overall spin time. Four samples were spun at each designated amount of spin time for these five durations and further cured in an oven at $50^{\circ} \mathrm{C}$ for about 6 hours.

None of the experimental samples had any observable air bubbles left in the gap when we took them out of the centrifuge in a pre-cured condition, however, they developed air gaps during the curing process. This emergence of visible bubbles alludes to the fact that there are micro/nano bubbles (that cannot be seen by the naked eye) left in the gap much smaller than the occluding bubbles. These take much longer to remove than the larger bubbles seen earlier. The small volume of these bubbles helps generate a very tiny buoyant force despite a large drag force acting on these bubbles from the surrounding liquid and the confining walls (the physics is discussed in detail in Section 4.3).

These micro bubbles seemed to remain in the narrow gap after we removed the setup from the centrifuge. This was confirmed after the curing process as the bubbles expanded and could then be seen in the cured sample. Any trapped air bubble would reduce the effectiveness of the bond created between the laminates and removing them is one of the most important factors when it comes to creating an effective bond. This behavior was also noted by Mazzeo, et al. [6] during the bubble removal process in their setup and was resolvable by spinning for sufficiently long periods of spin times.

We also observed elongated air bubbles along the edge of the gasket as seen in Figure 8A in the experimental setup in all the runs. These bubbles were essentially caught in bubble traps under the gasket as the liquid rushes into the gap near the gasket. The immense amount of drag acting on the air bubbles under the compressed gasket prevented
the air from moving out from under it in the short time the setup is spun in the centrifuge. However, as the setup was put in the oven for the curing procedure the air bubbles expanded due to the heat in the oven and seep out from under the gasket appearing as elongated bubbles along the gasket.

These bubbles are not as big of an issue as the ones that appear away from the gasket because they might be away from our region of interest and can be avoided with proper cutting away of the outer perimeter or by a future innovation of a better clamping mechanism (we were limited by the requirement of having to image our setup).

Using the centrifuge for degassing, however was pretty effective despite any issue regarding the degassing of trapped micro bubbles. This is showcased in Figure 8B where even very modest centrifugal forces resulted in almost complete degassing from our enclosure within 5 minutes of spinning.


Figure 8: (a) A set of five images showcasing how the spin time (in minutes) affects the number of bubble left in the setup after curing. (b) A plot quantifying the mean number of bubbles remaining in the cured setup over the duration of spin time in minutes (the red bars show the standard deviation for each spin time for which four experiments conducted).

## Chapter 4: Air bubble dynamics in narrow confined channels

### 4.1 Dimensional analysis:

In order to understand the dynamics of the air bubbles, we analyzed the physical parameters associated with their behavior. We used Buckingham-Pi theorem to carry out the dimensional analysis to find dimensionless terms that govern the behavior of the air bubbles seen. The selected governing variables were the viscosity of the liquid ( $\mu$ ), the velocity of air bubbles ( v ), the diameter of the air bubbles (d), surface tension at the liquidair interface $(\sigma)$, the size of gap between parallel plates $(\mathrm{t})$ and the high-gravity generated due to the spinning centrifuge $(\mathrm{g})$. The repeating variables were the diameter of air bubble, surface tension and viscosity of the liquid. The dimensional analysis we performed produced the confinement parameter or, the dimensionless ratio of air bubble radius to gap size $(s)$, coefficient of drag $\left(C_{D}\right)$ and the Capillary number $(\mathrm{Ca})$ as the dimensionless parameters. These parameters point at the effect that the different experimental designs have on the outcome of the process such as material properties of liquid used, gap size between the parallel plates, and speed of the spinning centrifuge.

The Capillary number $(\mathrm{Ca}-$ Equation 6$)$ determines if the air bubble behavior is dominated by capillary effects or viscous effects (viscos drag) for a bubble of a certain size (Saylor and Bounds [28]). Figure 9A shows the Capillary number plotted against the five air bubble diameters for the bubbles analyzed in Section 3.1. The effect of capillary number values seen here are discussed in detail later in Section 4.4.


Figure 9 (a) Plot of Capillary number (Ca) versus effective bubble diameter (mm). (b) Plot of the $\log _{10}$ (Weber Number) versus effective bubble diameter. (c) Plot of $\log _{10}$ (Bond Number) versus the effective bubble diameter.

$$
\begin{align*}
& \mathrm{Ca}=\frac{\mu \mathrm{v}}{\sigma}  \tag{6}\\
& \mathrm{We}=\frac{\rho \mathrm{v}^{2} \mathrm{~d}}{\sigma}  \tag{7}\\
& \mathrm{Bo}=\frac{\Delta{\rho g d^{2}}_{\sigma}^{\sigma}}{\mathrm{s}=\frac{\mathrm{d}}{2 \mathrm{t}}} \tag{8}
\end{align*}
$$

The $\Delta \rho$ shown above is the difference in the density of air and the liquid surrounding it. The $\rho$ in Equation 7 is the density of the liquid used. The other physical parameters are same as the ones used during the dimensional analysis.

The relative change in confinement parameter (s - Equation 9) with the change in drag coefficient $\left(\mathrm{C}_{\mathrm{D}}\right)$ helps determine how the confinement affects the dynamics of the air bubbles contained in it. The reason behind the drag experienced by the air bubbles is discussed in the following sections along with the experimental data related to coefficient of drag seen in Section 3.2. We also discuss different confinement scenarios to show how the drag would scale with a change in the confinement.

In order to further look into the bubble dynamics we also used Weber Number (We - Equation 7) and Bond Number (Bo - Equation 8) as a reference. We plotted the log of Weber Number and Bond Number values against the air bubble diameters for the five air bubble sizes analyzed earlier (Figure 9 B and C ) to see how confinement affected the bubble dynamics. The Weber number looks at the relative effect of inertia over surface tension (Saylor and Bounds [28]) whereas the Bond number looks at the effect of body
forces over the surface tension forces (Hager [29]). The effect that Weber number and Bond number had on the air bubbles in our setup are discussed in the following sections.

### 4.2 Coefficient of drag and the confinement effect:

The drag experienced by air bubbles in a narrow gap is an interesting concept to explore, especially when it comes to flattened occluding air bubbles in a high gravity setting. The air bubble motion between closely spaced flat plates is affected by several factors: gravity, viscous forces, inertia and surface tension. The parameters mentioned earlier (Weber Number, Capillary Number, Reynolds Number etc.) help determine which phenomenon contributes to the behavior of air bubbles the most (inertia, gravity, surface tension etc.).

It must be noted that the air bubbles analyzed during our experiments were all larger than the gap by at least a factor of five and they were removed fairly quickly during the degassing procedure. The micro bubbles formed in our setup that were the same size as the gap or smaller could not be detected using the high speed imaging and image processing code due to the inability of the imaging system to resolve bubbles of that size. In order to successfully carry out degassing in a reasonably short amount of time, the micro bubbles are of the biggest concern to us. Any air bubble which is approximately the same size as the gap or smaller is observed to require a much larger amount of time to leave the compressed region compared to any of the larger air bubbles studied. This is because the bubble dynamics differ depending on whether they are smaller or larger than the gap size. The different bubbles here could be put into two different categories depending on their
diameter: air bubbles with a diameter smaller or equal to the gap size and occluding air bubbles with a diameter larger than the gap size.

### 4.3 Confined air bubbles smaller than the gap:

The air bubbles smaller than the gap size in our setup had a very small volume and therefore naturally did not have a high buoyant force. There was, however, a large drag force acting on these small confined bubbles that slowed them down considerably. The work done by Figueroa-Espinoza, et al. [11], looks at air bubbles in a confined setup with similar values for confinement parameter (s) as the smaller bubbles seen in our experimental setup. Their setup involved the use of silicone oil between two parallel plates with air bubbles rising in the oil under natural gravity due to buoyancy. They were mainly concerned with air bubbles with a confinement of $s=0$ (unconfined) all the way to $s=1 / 2$ (bubbles as large as the gap).

Figueroa-Espinoza, et al. normalized their drag coefficient values with the drag coefficient obtained from the study done by Moore [20] (Moore looked at the drag coefficient of freely rising unconfined ellipsoidal air bubbles in a quiescent liquid). They further plot this normalized drag coefficient against their confinement parameters. The drag coefficients for the air bubbles are seen to increase monotonically with the increasing confinement parameter (increasing ' $s$ ' means decreasing gap size). In their study for the most confined case (air bubbles with a diameter equal to the gap size) the increase in the coefficient of drag for the air bubbles came out to be greater by just under $\mathbf{2}$ in comparison to an unconfined equivalent [11].

They mention that the drag coefficients for these air bubbles, based on their research, is dependent on two factors: the Reynolds number and the confinement effect. The confinement effect is a result of the resistance experienced by the air bubbles due to the walls of the container. The Reynolds number component essentially referred to viscous drag due to the liquid around the air bubble which would presumably depend on the surface tension at the liquid-air interface. They mention that their study focused on cases where inertial effects were bigger than viscous ones ( $\mathrm{Re}>1$ ) and surface tension was dominant over inertia $(\mathrm{We}<1)[11]$. When surface tension forces dominate the inertial effects (usually $\mathrm{We}<1$ ) for an air bubble, it has the tendency to remain intact and not break up (Evans, et al. [27]). The effect of surface tension dominance also manifests itself by minimizing the surface area of the bubbles thus making the bubbles spherical or oblate which lowers the amount of drag the bubble would experience due to the liquid moving around the bubble.

They further cite the work done by Legendre [21] regarding the argument that drag forces for Stokes Flow could be represented as a function of maximum vorticity. They go on to use Legendre's method of calculating the drag coefficient and plot its normalized values (normalized using coefficient of drag from Moore's paper [20]) versus the confinement parameter thus showing that the drag increases with the confinement similar to their experimental results. The study conducted by Legendre essentially splits the drag force into two subcomponents: the drag on the air bubble in an unconfined case and the additional drag on the bubble due to the vorticity generated due to the confining walls. Figueroa-Espinoza, et al. further go on to show that the maximum wall vorticity increases with an increase in the confinement parameter (increases with $\mathrm{s}^{3}$ ) and contributes to an increase in the drag coefficient.

### 4.4 Occluding air bubbles with diameters larger than the gap (Hele-Shaw Cell):

The air bubbles analyzed in our study here belong to the "occluding bubble" case. These bubbles had a bubble diameter much larger than the gap size and are similar to air bubbles seen in a Hele-Shaw cell. We have a somewhat limited understanding of the dynamics of occluding air bubbles in these narrow gaps, however, we made an attempt to string together available literature that explains the behavior of air bubbles in a Hele-Shaw cell to help us understand the air bubble dynamics.

In a study conducted by Maxworthy [22], he used flat plates placed at an angle to construct a gravity driven Hele-Shaw assembly. In a manner very similar to our setup he used Lucite sheets with viscous silicone oil to create the Hele-Shaw cell. For his analysis of individual bubbles moving in the setup Maxworthy mentions that there is a flow of liquid around the air bubbles and that the bubbles do not completely fill the gap. The presence of thin films around a bubble in a Hele-Shaw cell is confirmed by a theory by Park and Homsy [24] as well, where they look at two-phase displacement in Hele-Shaw cells.

In a study conducted by Kopf-Sill and Homsy [26], they look into the shape of immiscible bubbles in a moving fluid along with the discrepancy of the bubble velocity compared to preexisting simplified theories on bubble velocity in Hele-Shaw cells. In the experiments by Kopf-Sill and Homsy, a Hele Shaw cell was placed at an angle and used to generate some buoyant force to move the air bubbles in their setup. They used a glycerinewater mixture between glass plates for their analysis. They also pumped the liquid through their apparatus to generate a liquid velocity in the direction of the air bubble. They state that the discrepancy in their measured bubble velocity values and existing theoretical
calculations of velocity could be explained by the stresses introduced by thin films around the air bubbles that retarded the flow of the bubble. A similar stress could contribute to the drag force acting on the air bubbles seen in our setup.

In a study conducted by Eck and Siekmann [8] (mentioned previously in section 3.1), they claimed that the air bubbles were considered asymmetrically positioned in the Hele-Shaw cells based on high Bond Number values (0.5 and 0.8). According to them this leads to an uneven thickness of the liquid film between the air bubbles and the flat plates on either sides of the bubble. Considering that our Bond numbers were considerably higher for all the air bubbles we can assume that our air bubbles experience uneven drag from liquid films above and below the air bubbles. This uneven drag force leads to a wobble in the bubble boundary which is observed during our experiments.

According to Maxworthy [22] in a gravity driven flow a smaller film thickness is present around the air bubble when compared to the absence of gravity. For the five air bubbles studied in our experiments in Sections 3.1 and 3.2 the Bond number values were much higher than 1 due to the high gravity generated in our centrifuge (even for the smallest air bubble which has a 0.5 mm diameter seen in Figure 9C). The high Bond numbers allude to the dominance of body forces (or gravity), so we can assume that there is a much thinner film present around an air bubble of this volume for the given gap size in a Hele-Shaw cell. It must also be noted that a thinner liquid film around the bubbles would lead to lower or negligible capillary effects (capillary effects would further slow the bubble down). This is also reflected by the Capillary numbers calculated for the air bubbles, which were quite high (meaning negligible capillary effects) as seen in Figure 9A. Maxworthy also talks in
detail about air bubbles that split up into smaller bubbles as seen during his experiments. In our experiments all the air bubbles observed remained intact and colliding air bubbles combined together to form a larger bubble. This discrepancy can be easily explained by the Weber number values for our air bubbles (Figure 8B). The bubbles of different sizes observed during our experiments all yielded Weber number values much less than 1 indicating a dominance of surface tension over inertial effects (Saylor and Hager [28]). This dominance of surface tension forces the air bubbles to stay together instead of breaking up (Evans, et al. [27]). The dominance of the surface tension is also echoed by the circular (for smaller air bubbles) or oblate shape for the air bubbles (for larger bubbles with a higher Weber number) seen in our setup.

In another study by Maruvada and Park [25], the retarded motion of bubbles in a Hele-Shaw cell were specifically studied. Similar to Maxworthy, they use an inclined HeleShaw cell to introduce a buoyant force into their experimental setup. They also claimed that air bubbles have a thin liquid film around them. During the calculations for bubble velocity they mention that the film thickness is a function of the Capillary Number. They also mentioned that the flow in the liquid film could be represented by a Couette flow if the bubble surface is rigid (this might be relevant for the smaller air bubbles seen in our experiments). It also must be pointed out that the Couette flow has a zero contribution if the surface of the air bubble surface is clean and stress free. They go on to add that the drag experienced by the air bubbles are proportional to the film thickness. It must be noted that the shape of the largest bubble observed during our analysis did not have a well-defined shape and tended to wobble a lot over its travel range. Maruvada and Park state that for a large enough bubble high values of Capillary number would lead to a lot of irregularity of
air bubble shapes. This fact is further bolstered by the knowledge that the Weber number for the largest bubble is the highest thus lowering the contribution of surface tension in retaining the bubble shape.

## Chapter 5: Conclusion

The study conducted here works to explain the principles of air bubble removal from viscous liquids under high gravity in narrow gaps. It introduces a contrasting theoretical situation where air bubbles of a spherical shape travel under high gravity in viscous liquids in an unconstrained medium.

An analysis involving high-speed imaging was conducted to show that bubbles in a narrow gap move much slower than they do in an unconstrained medium due to a comparatively higher drag experienced by them under these conditions. The drag coefficient of the confined bubbles (we looked at flattened bubbles larger than the narrow gap) when compared to unconfined spherical equivalents were calculated to be higher by factors ranging from 2 to 280 . The smallest of the five air bubbles monitored during the high-speed imaging procedure, which had a bubble diameter five times the size of the gap $(0.5 \mathrm{~mm})$, had a coefficient of drag greater than an unconstrained equivalent by a factor of 2.

After gathering all the data, an analysis of the observed phenomenon was conducted by citing several similar studies on air bubbles smaller than a constrained gap between two flat plates, which mimics the behavior of the smaller bubbles seen in this setup. For air bubbles smaller than the gap size, vorticity generated due to the constraining walls in the liquid around the air bubbles was believed to be the reason behind the additional drag experienced by them. The drag coefficient was also shown to increase with increasing confinement of an air bubble in a narrow gap (which could result from decreasing gap size or increasing bubble diameter or both). The air bubbles with a diameter equal to the narrow
gap were shown to have a drag coefficient greater by a factor of just under 2 when compared to an unconstrained case [11]. An air bubble with a $100 \mu \mathrm{~m}$ diameter in our case would have the same confinement parameter and hence would have a drag coefficient differ by the same factor in comparison to an unconfined equivalent.

For occluding air bubbles larger than the gap, experimental observations appeared to agree qualitatively with previous studies on Hele-Shaw cells. Based on the information available the excess drag on these air bubbles was believed to be a result of the stress applied on the bubble due to the presence of thin liquid films around the air bubbles. The overall drag experienced was dependent on the film thickness which in turn depended on the balance between surface tension effects, inertia, body forces and capillary effects.

An experimental design was also developed to accomplish the use of a centrifuge to introduce an adhesive between a pair of laminates in a repeatable manner and successfully carry out degassing from the gap between them. Several experiments were conducted to develop a degassing procedure capable of producing bubble free laminates using a pair of PET sheets with Sylgrad-184 as the curable adhesive. A bubble-free sample measuring $68 \mathrm{~mm} \times 62.5 \mathrm{~mm}$ (with a 0.1 mm gap size) was created with only 5 minutes of centrifugation. It was observed that the air bubbles the same size as the gap or smaller took considerably longer to be removed from the liquid in the narrow gap because of their small terminal velocity as a result of their small volume. The larger occluding bubbles experienced a much higher drag, however, the high buoyant force generated due to the size of these bubbles resulted in a much higher terminal velocity thus lowering the time required to remove them from the narrow gap.

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