4-30-2020

Independent Study Report: Design of Metallic Droplet Generator and Data Analysis for the Project on Wettability of Graphene

Shuhan Zhang
Washington University in St. Louis

Patricia Weisensee
Washington University in St. Louis

Independent Study Final Report

Shuhan Zhang

Washington University in St. Louis

Instructor: Patricia Weisensee

Experiment Date: 01/14/2020 – 04/27/2020

Submission Date: 04/29/2020

Abstract
The outcome of this independent study is different from what I have planned at the beginning of the semester due to the COVID-19. Since the lab has been closed after the spring break, I was not able to continue the project on the fabrication of a metallic droplet generator, which is to be used to conduct research on to analyze the cooling rates and impact dynamics of molten metal droplets. After the spring break, I helped analyze the data for the research on the wettability of graphene, which I have been working on since last summer. Therefore, this report summarized my wiring design of RTDs in chamber, thermocouples in crucible and PID controller for the droplet generator project. It also included my analysis on surface tension of diiodomethane and ethylene glycol from 25-85℃ and the contact angle of graphene in these two liquid for the graphene project. The only step left for the design of droplet generator will be re-drilling the holes on Teflon block. For the graphene project, our next steps will be finishing the data analysis part for all five liquids: water, glycerol, hexadecane, diiodomethane, and ethylene glycol; then a surface energy equation for graphene will be derived.

Introduction
For my Spring independent study, I continued my research under Dr. Patricia Weisensee. My research has been separated into two parts. Before the spring break, I mainly focused on the design and fabricate a metallic droplet generator; after the spring break, I worked on the data analysis on wettability of graphene.

Medal Droplet Generator

The purpose of this project is to use optical and IR high speed camera to analyze the impact dynamic and cooling rate of melted tin droplet. The goal is to find the relation between the droplet impact process and the microstructural development of the deposited material [1]. Figure 1 below shows a rough schematic of the proposed experimental setup.

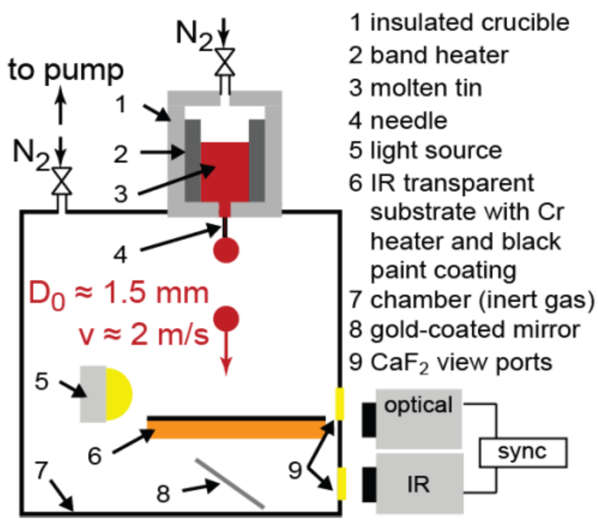

Figure 1. Proposed Experimental Setup for Medal Droplet Generator

In this setup, the tin will be melted in the crucible and pushed down through a tube into a vacuum chamber. The chamber is also designed with viewports to allow IR camera and high speed camera to observe and record the impact of droplets.

For this project, I continued the design and fabrication of the droplet generator apparatus, which was partially finished by the graduate student Devin Williamson. Specifically, I will describe my wirings of RTDs in chamber, thermocouples in crucible and PID controller in following sections.

Wettability of Graphene

The purpose of this project is to characterize the wettability of graphene with the Wilhelmy plate method. Because of graphene's good thermal conductivity and small thickness, researchers were interested in it as an application for enhancing heat transfer. Since all previous research were done in ambient conditions and in short period, our research filled the gaps by analyzing how the contact angle of graphene varies with respect to temperature and environment over long term. My independent study mainly focused on how the contact angle of graphene vary with respect to temperature, by applying Wilhelmy plate analysis.

Wilhelmy plate method is used for "measuring the surface tension of a liquid, the interfacial tension (IFT) between two liquids and the contact angle between a liquid and a solid." [2] As shown in Fig. 1, when a plate is inserted into the liquid, contact angle formed between the liquid and the plate.

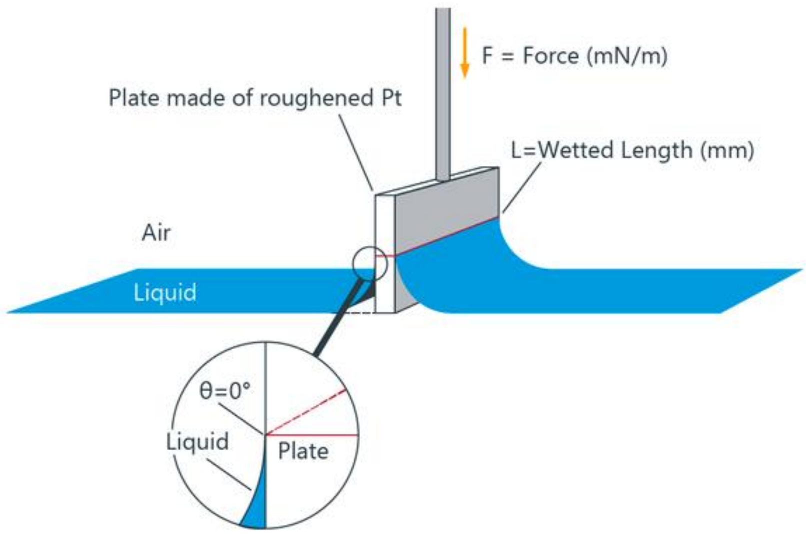

Figure 2. Schematic diagram of the Wilhelmy plate method

The force F acted on the plate correlates with the surface tension or interfacial tension σ and contact angle θ [2]:

$$\sigma = \frac{F}{L \cdot \cos\theta} \tag{1}$$

,where the wetted length L is the perimeter of the plate.

As a material property, the interfacial tension of a liquid is a constant. Thus, we should calculate the interfacial tension first with the plate of a known contact angle such as 0°, and then back calculate the contact angle of graphene using the same method.

Apparatus Design for Droplet Generator

RTD Wirings in Chamber

The RTD sensors, shown in Figure 3, are used to measure the temperature inside the chamber and on the surface of the sample. The large cylindrical sensor is hang approximately in the center of the chamber, but not influence the view of the droplet. The small flat sensor will be attached on to the sample. Both RTDs are connected to USE-TEMP, which will transfer temperature data to computer. The software **InstaCal** is used to warm up the system and recalibrate the unit. The software **tracerDAQ** is used to display the temperature data.

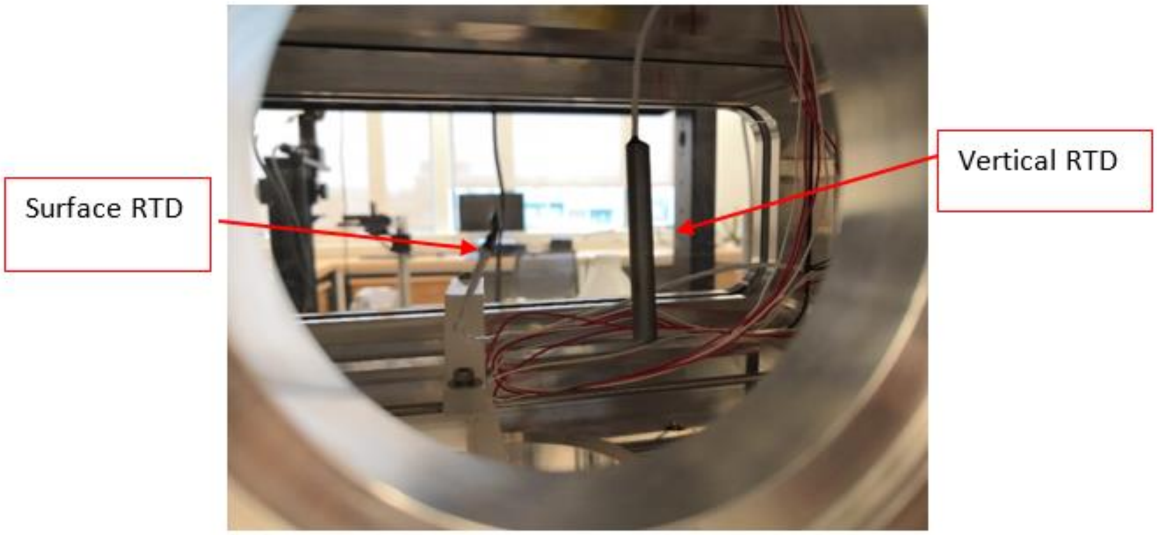

Figure 3. RTDs inside the chamber

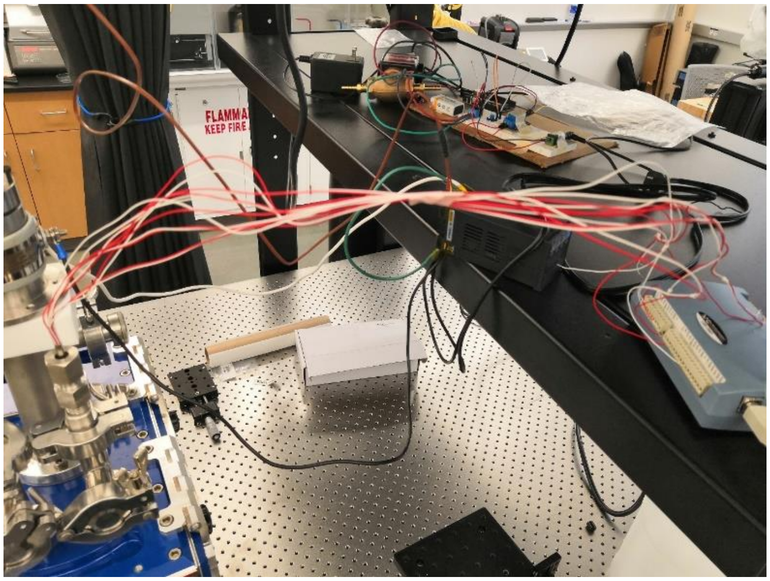

Figure 4. Wirings of RTDs and USB-TEMP

Figure 5 shows a sample plot for the measurement of temperature using the two RTDs. The upper lane is for the cylindrical RTD, and the lower lane is for the surface RTD.

Figure 5. Sample Temperature Plot for RTDs

Crucible Setup and PID Controller Wirings

The crucible, shown in Figure 6, is used to melt tin. PID controller will control the input voltage to heat the band heater to around 240℃. The thermocouple inserted through the crucible cap is used to measure the temperature inside. Nitrogen is pumped into the crucible through the nozzle is used to push the molten droplets out through the bottom tubing.

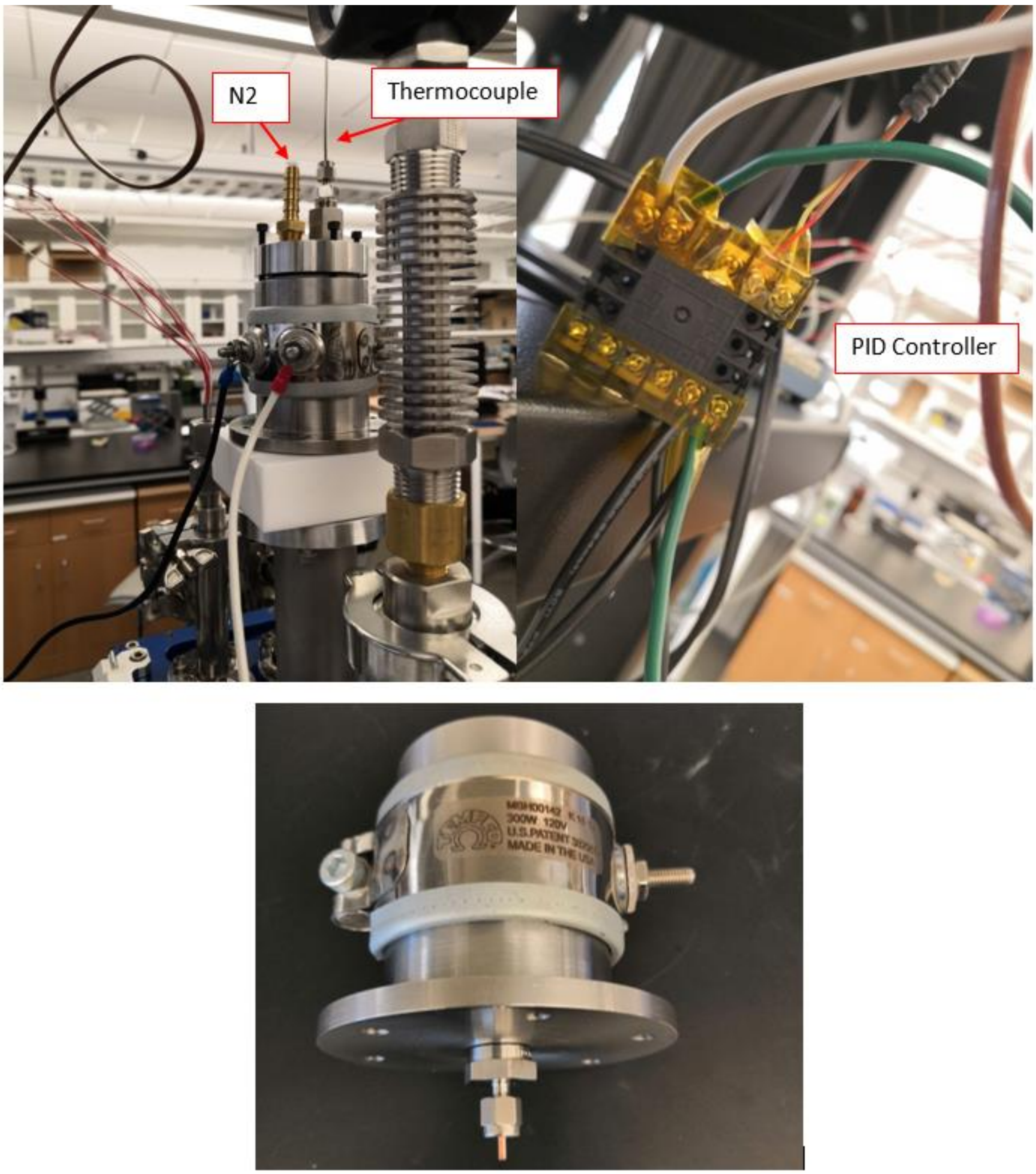

Figure 6. Components of Crucible and PID Controller

The wiring diagram for the 48 VFL PID Controller is shown below. I adjusted this PID controller to appropriate unit, temperature range and settling time. The following researcher can simply adjust the desired temperature to melt the medal.

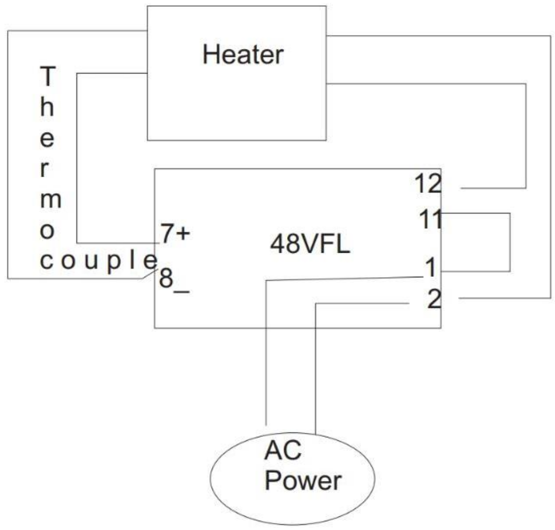

Figure 7. Wiring Diagram for PID Controller

To mount the crucible to the fittings or directly to chamber, a Teflon block should be added in between. The design of the Teflon block was designed by graduate researcher Devin Williamson. Unfortunately, the Teflon block fabricated in the machine shop was not accurate enough to seal the threads. The holes on the Teflon block under the crucible is still not lined up with the holes on the crucible. Although I re-drilled the holes in makerspace with Dr. Okamoto, only two threads can fit into the holes. The rest of three threads are only partially inserted. For future steps, I recommend to drill the hole one size larger, so that the threads can fit in.

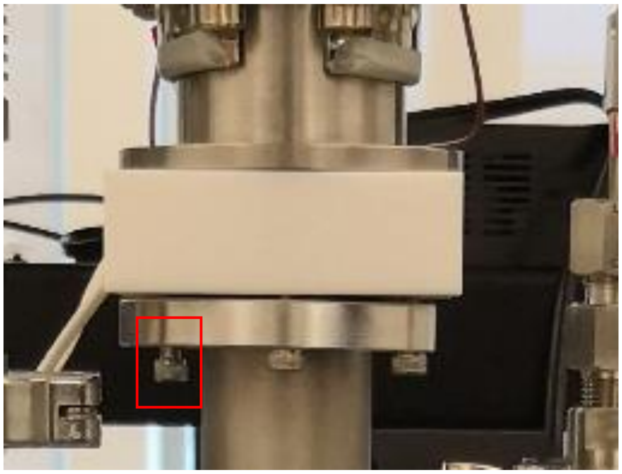

Figure 8. Threads not Perfectly Fit into the Teflon Block

Data Analysis for Surface Tension and Contact Angle of Graphene

Beginning with glass sample inserted in ethylene glycol at 25℃, the mass change was recorded by the microbalance. As the immersion depth increased, the buoyancy force F_b also increased, which can be calculated with the equation [3]:

$$F_b = \rho g V \tag{2}$$

,where ρ is the density of liquid, g is gravity and V is the immersion volume. The total surface tension F, can be calculated by the equation:

$$F = F_m + F_b = mg + \rho g V \tag{3}$$

,where F_m is the force given by the microbalance and m is the mass measured by microbalance.

The wetted length of slide was the diameter of bottom surface:

$$L = 2 \times (0.025 + 0.001) = 0.052m \tag{4}$$

With Eqn. 1, the interfacial tension can be calculated. Matlab scripts are generated to combine the temperature data recorded by Arduino, and the mass data recorded by microbalance. For each measurement, I took the average of temperature and interfacial tension. The relationship of interfacial tension and temperature for water is shown in Fig. 9 below:

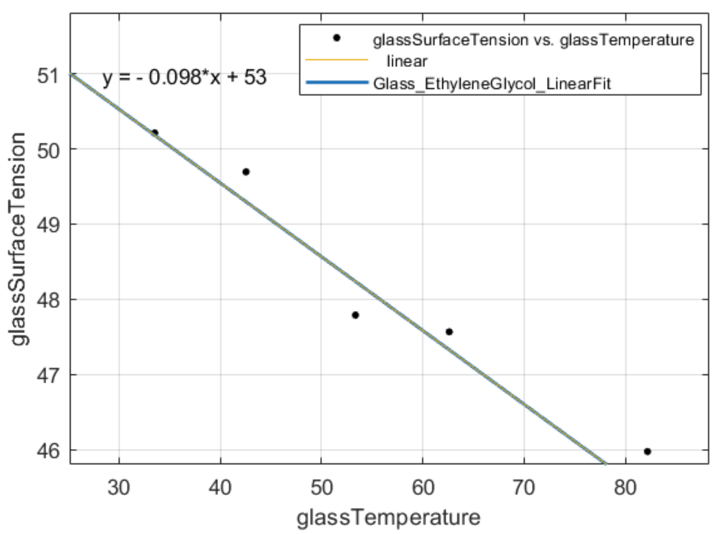

Figure 9. Interfacial Tension for Ethylene Glycol

I did not find a theoretical relation between the surface tension and temperature of ethylene glycol. However, the R squared for the linear fit is large enough to assume the fitting is highly appropriate. Thus, I can use the equation generated in this fit to calculate the surface tension of the liquid at the temperature I measured on graphene samples.

Since graphene slide has a different dimension, the wetted length became:

$$L = 2 \times (0.0255527 + 0.0004064) = 0.0518668m \tag{5}$$

Contact angle can be derived by converting Eqn. 1:

$$\theta = arccos(\frac{F}{L \cdot \sigma}) \tag{6}$$

Figure 10 below shows the contact angle of graphene at different temperatures. The advancing contact angle increased when temperature was larger than 50℃. This situation seemed unreasonable. One reason might be when the liquid was heated, it extracted water faster than before. Thus, there might be some change in the liquid density.

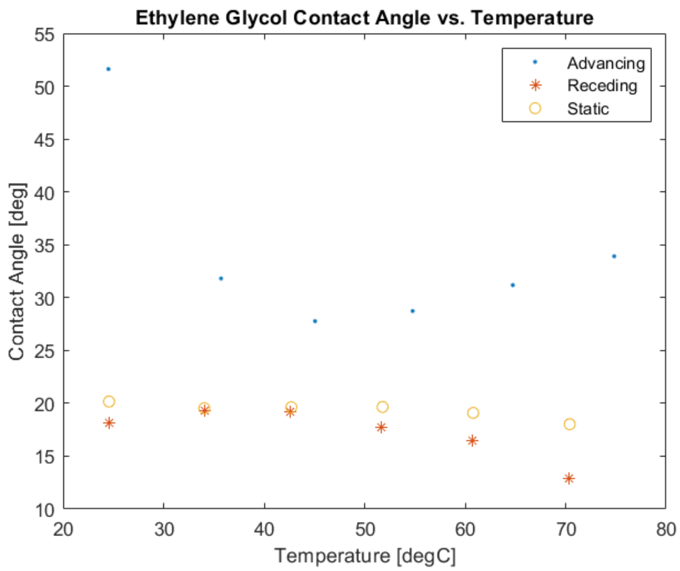

Figure 10. Contact Angle of Graphene in Ethylene Glycol

The surface tension of diiodomethane and the contact angle of graphene in diiodomethane have been calculated using the same method. The plots are shown below.

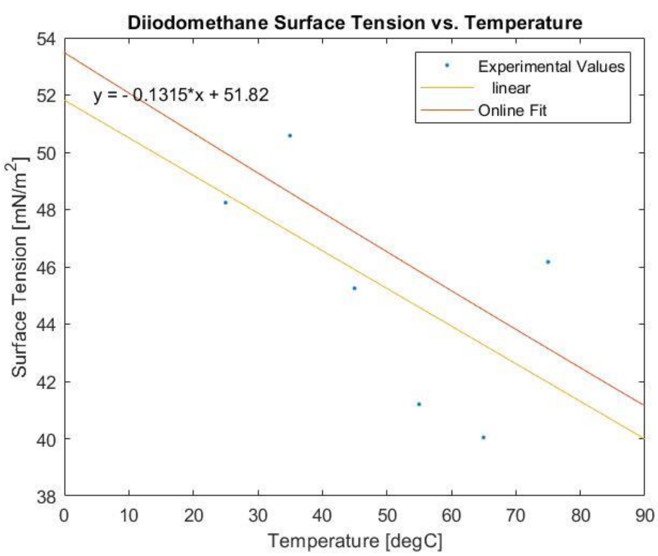

Figure 11. Interfacial Tension for Diiodomethane

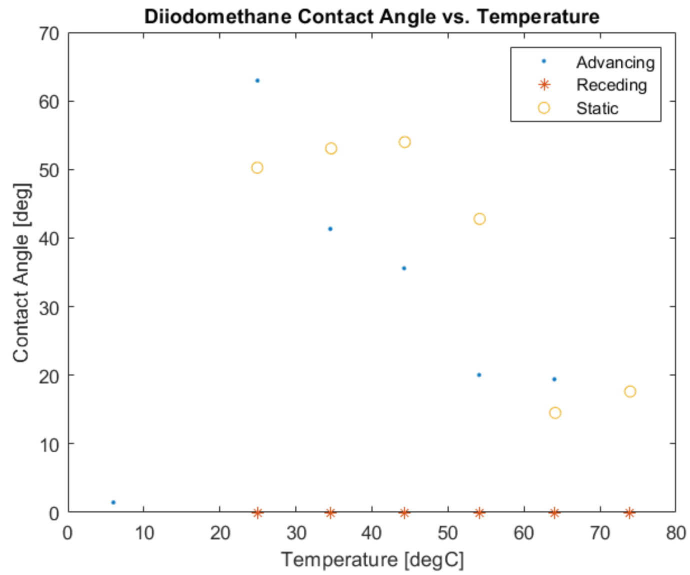

Figure 12. Contact Angle of Graphene in Diiodomethane

As shown in Figure 11, the experimental linear fit was approximate to the interfacial equation found online. Thus, I assumed the equation generated by this experimental fit was accurate. In Figure 12, the receding contact angle were zero, because diiodomethane has no polar component to its overall surface tension.

Future Steps

In the independent study for this semester, the design and fabrication of the medal droplet generator was almost completed. There are a few things need to be done before the apparatus is ready for testing. Firstly, the holes on Teflon blocks should be re-drilled to line up with the holes on the crucible. Additionally, the chamber need to be physically tested that there is no leaking under vacuum. Furthermore, a mount for the high speed camera should be made to make sure it can look through the viewpoint of the chamber. Also, the mirror assembly and IR camera need to be fixed onto the stage before use. If researchers are allowed to be back in lab, I am confident that all these work can be finished in two days and the apparatus will be successfully working in the near future.

There are a lot of things left for the data analysis part of the graphene project. The interfacial tension should be calculated for the other three liquids, and contact angle of graphene in each liquid should also be calculated. More importantly, error bars should be added based on factors such as the variation in temperature, the error in buoyancy force, the change in density with respect to temperature, etc. Hopefully, by this summer, a surface energy equation for graphene can be derived.

Reference

[1] Devin Williamson, *Metal Droplet Generator Apparatus for Study of Impact Dynamics and Cooling Rates Fall Rotation Research Report*

[2] KRUSS GmbH, *Wilhelmy Plate Method*, https://www.kruss-scientific.com/services/education-theory/glossary/wilhelmy-plate-methodAlireza Mo/

[3] The Engineering ToolBox, *Surface Tension*, https://www.engineeringtoolbox.com/surface-tension-d_962.html