1 316-2 Laboratories

1.1 Laboratory 1: Nucleation and Solidification in a Binary Eutectic Salt System

1.1.1 Laboratory Objectives:

To observe phase transformations in a binary eutectic system and the formation and subsequent coarsening of dendrites.

1.1.2 Learning Outcomes:

Upon completing the lab exercise, students should be able to:

- Explain the observation of birefringence.
- Discuss undercooling and observations related to cooling rate.
- Quantify coarsening using measured secondary arm spacing and surface area to volume ratios. Predict how S_v will change with coarsening.
- Estimate uncertainty in measured values (temperature and dimension).

1.1.3 General Instructions:

Read through the lab before you begin. It is possible to acquire data for parts II and III during the same runs, but you must anticipate when to pause the temperature on the hotstage and when to acquire images on the printer. Parts I and II are measurements of phase transitions and dendrite growth rates and initial secondary arm spacing at a constant cooling rate. Part III is a measure of dendrite evolution as a function of time at a constant temperature. Therefore, you could pause the temperature of the system as soon as dendrites appear. The initial progression of the dendrite across the screen will provide you with the rate for part II; the subsequent evolution of the structure at a constant temperature will provide the data for part III. Note: setup a video file and record time / temperature / scale bar on the images.

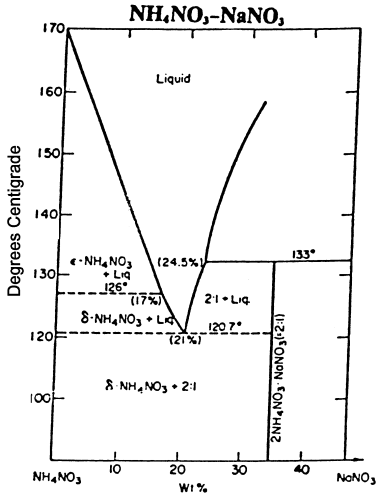

Figure 1.1: Phase diagram for the ammonium nitrate/sodium nitrate system.

Part I: Solidification Use the hotstage and transmission microscope to observe melting and solidification of ∼10wt% NaNO3 in the above salt system. Record transition temperatures on heating and cooling, and carefully note which transitions they correspond to. Fill in the table, below.

Table 1.1: Tempeartures of phase transformations at different cooling rates.

Rate		T(eut)	T($\delta - \varepsilon$)	T(liq)
	Heating			
	Cooling			
	Heating			
	Cooling			
	Heating			
	Cooling			

Birefringence: Are any of the phases observed during heating and cooling birefringent? Which? Label the phase diagram.

Part II: Observing the Microstructure as a Function of Constant Cooling Rate: A. Cool the sample from the melt at a rate between 1 deg/ minute and 15 degrees per minute. Watch the melt (from the microscope – it has a larger area of view than the camera) until you observe the primary solidification begin, then "hold" the temperature and start recording the video image. The actual distance can be scaled based on the magnification, and the dendrite growth velocity calculated from these results. The information for Part III may be obtained by continuing to record as the microstructure evolves with time at constant temperature.

B. Repeat (A) for two additional cooling rates.

Table 1.2: Data collection table for constant cooling rate.

Rate	T (dendrite formation)	Dendrite growth rate*	*Initial* secondary arm spacing *

* Correct for magnification.

Magnification : _____

Part III: Observing the Microstructure as a Function of Time at Constant Temperature (Isothermal Experiments) Repeat (A), but when solidification begins, pause the hotstage. Note the temperature. Capture images as a function of time (isothermal this time). Note: We will pause the temperature when we observe the dendrite formation. So the initial dendrite velocity, as well as the initial dendrite secondary arm spacing is a function of constant cooling rate; subsequent dendrite coarsening (secondary arm spacing) is a function of time at a constant temperature.

Table 1.3: Measured dendrite properties for isothermal experiments. Paused temperature = _____.

Time	Secondary Arm Spacing	Surface Area to Volume Counts

Table 1.4: Measured dendrite properties for isothermal experiments. Paused temperature = _____.

Time	Secondary Arm Spacing	Surface Area to Volume Counts

1.1.4 Lab Write Up:

Answer the following questions, using appropriate discussion of results, tables and plots. Neatness counts.

1. Describe what equipment was used in this lab and what measurements were made.
2. Make a table of your temperature results.
3. Is the temperature of the hotstage reliable? Why or why not? Be quantitative. Use your observations to substantiate your answer.
4. Did you observe birefringent phases? Which? Did you observe phases that were not birefringent? Which? What are the corresponding crystal structures?
5. Is there a relationship between the transition temperatures and the rate of cooling? What is it?
6. Does dendrite growth velocity depend on the cooling rate? What trend to you observe? Discuss.
7. From the constant cooling rate data, plot (initial) secondary arm spacing vs. cooling rate. Discuss.

8. Quantitatively, the secondary arm spacing is predicted to increase as a function of time to the 1/3 power. Plot the secondary arm spacing vs. time to determine if your data exhibits this behavior. Use the isothermal data.

9. The quantitative behavior of the surface area to volume ratio is predicted to change as time to the -1/3 power. What do your results indicate about coarsening? You can use overlays to determine the surface area per unit volume (# intersection of curved lines with phase / # points on overlay).

10. Choose a set of images, one taken at the initial dendrite formation, a second after some time has elapsed. Estimate the radius of curvature in each case. Assume that you had a uniform distribution of spheres, first with radius 1, coarsening to radius 2, using the two values above. Calculate the change in surface area to volume of such a change in radii. . Show your work. If you didn't measure these, try to choose reasonable values. Approximate the radius of curvature of the tip by half the distance across a secondary dendrite arm.

1.2 Laboratory 2: Age Hardening in Al Alloys

1.2.1 Objective:

To determine the hardness and conductivity versus aging time of 2024 (1″ wide x 1/8″ thick), 6061 (1/2″ wide x 1/8″ thick) and 7075 (1″ wide x ¼ " thick) aluminum alloys aged at temperatures of 25°C, 125°C, 225°C. Each lab section will be responsible for a single alloy. Aging will be conducted over the course of the week; team members should plan to make measurements throughout the week.

1.2.2 Final write-up:

Data from the three lab sections will be pooled. This must be submitted by the end of week 2. Group reports will need to include (discuss) ALL class data.

1.2.3 Procedure:

1. Measure as-received, un-solutionized samples.

Alloy	Hardness Scale	Hardness (5 values)	Conductivity

2. Solution-treat~ a set samples of each alloy at 500C for one hour. Quench in ice water.
3. Measure the hardness of each solutionized sample (5 measurements on each!); discard outliers. Record.
4. Measure conductivity. Note: the sample MUST be at room temp. (do this after hardness) Record. Store samples in ice water.

Table 1.5: Hardness and Conductivity of Various Samples

Sample #	**Hardness (5 values)**	**Conductivity**

5. Anneal at:
   (a) room temperature
   (b) 125 °C
   (c) 225°C

for times ranging from 10 minutes, ½ hour, 1 hour, 2 hours....64, 128, 264 hours....Note that data will be plotted on a logarithmic scale. Use this information to plan your measurements.

Table 1.6

Sample #	T (°C)	Date in	Time in	Date out	Time out	Anneal time (min)	Hardness (5 meas)	σ 5 meas)

1.2.4 Group report (informal)

First describe each alloy. (What do the 2XXX, 6XXX, and 7XXX designations indicate?) Give some examples of applications in each case. Indicate what the normal thermo-mechanical heat-treatments (tempers) would be for each alloy. Discuss your results in the context of these heat-treatments. (Include references.)

1. Plot hardness versus log-time for each of the temperatures measured using the pooled data.
2. Discuss the curves and their relative magnitudes and relative position on the time axis of any peaks observed, and label appropriately. In your own words, discuss in terms of the theory of precipitation hardening.
3. Plot conductivity vs. log-time. Correlate with hardness measurements. Why does the conductivity change?
4. Discuss uncertainties in your data. Include error bars on your figures. Use the correct number of significant figures. Assuming a normal (Gaussian) distribution of these values, you can find the uncertainty of the mean to within a 95% confidence level ,by determining 2X the standard error of the mean: 2x the standard deviation (STDEV in Excel) divided by the square root of the number of points measured (COUNT in Excel). There is a single command to do this: CONFIDENCE (.05, STDEV, COUNT). Indicate these values on your plots by adding error bars: left click the data set and choose Format data series > Y error bars. Note that you can use the Custom error bar command to add different values for each data point (i.e. each mean value of hardness for each individual sample).

Be sure to include the following:

1. Data from all alloys – not just the one measured by your group.
2. error bars on plots – see the handout.

3. results on as-received samples (Note that the 2024 samples had a **T4 temper**. The 6061 and 7075 samples had a **T6 temper**. Your discussion should define these, and indicate if they make sense, based on your aging results.)

4. results on solutionized samples.

References Porter and Easterling [**?**], pages 291-308. Matter program (on Macs). Rosen paper posted on Bb, others you might find.

1.3 Laboratory 3: Al-Si Alloy Solidification and Modification (not currently used)

1.3.1 Experiment:

Each lab group (2-3 individuals) will prepare a castable alloy of AlSi using Al and a "Master Alloy" of 50wt% Al/Si. One group in each section should choose a hypoeutectic composition, another the hypereutectic composition, a third, if there is one, the eutectic composition. Consult the Al-Si phase diagram. Total alloy mass should be~ 25 grams. Once cast, we will cut a portion of this sample to polish, and a portion to re-cast with a 10%Sr, 90%Al alloy to achieve Sr compositions between .05 and .2 wt% Sr. We will examine the microstructures of the polished alloys.

1.3.2 Write-up (memo style):

- Project objectives – brief summary. Why are Al-Si alloys of interest?
- Methods – briefly summarize procedures.
- Results and Discussion
  - Micrographs:
    - Hypo, hyper and (if done) eutectic unmodified samples
    - Modified samples – compare to unmodified.
  - Stereology
    - How do Image J results compare to predictions from phase diagram?
  - Details of microscopy – is Jackson criteria valid?
  - Other observations: porosity? Differences from center-to-edge of samples? What? Why?\

1.3.3 Experimental details (record here):

- Mass Al:
- Mass Al(50wt%)Si(50wt%) master alloy:
- Alloy composition (wt% Si):
- Mass of alloy used for modification:
- Mass of Al(90wt%)Sr(10wt%): Note: Target amount = .05 - .2 wt% Sr
- Other details, i.e furnace temperatures, materials, procedures used.

1.3.4 Short Answer Questions: due at the beginning of lab, week 2

1. We will be using the casting process in lab. What limitations or potential difficulties can you anticipate using this technique?
2. You have already used the AlSi phase diagram to predict the microstructure for each alloy. Can further detail be added? Can you predict any differences in primary phase formation? Use the Jackson criteria, discussed in P&E in section 3.4.6 (p. 170) to predict in detail what the shape of the primary phase in the hypo and hyper-eutectic alloys might be. Show your work. Lv, Si = 1788 kJ/kg; Lv, Al = 397 kJ/kg. Q – will the microstructures look like figure 3.65 (a) or (b) or neither?
3. How might the microstructure be further controlled by solidification conditions?

4. What might be the technological importance of Al-Si alloys?
5. What effect do modifiers (careful – "grain refiners" and "modifiers" play different roles) have on the microstructure?
6. Why is the modification in (6) desired?
7. A recent research summary by Napolitano *et al.* attributes the differences in morphology between un-modified and modified AlSi alloys to a variety of factors. Describe these factors.