

LABIV

PHASE TRANSFORMATIONS

Study Questions:

- 1. A 50wt% Pb-50wt% Sn alloy is cooled from 350°C to room temperature; at what temperature does the first solid appear ?
- 2. What is the composition of the first solid? How about that of the remaining liquid?
- 3. What is the composition of the last liquid to freeze when the temperature decreases to 183°C?
- 4. How much liquid is still left upon first reaching that temperature?
- 5. What would you see if you looked at the microstructure at room temperature?
- 6. Now consider a 95wt% Sn-5wt% Sb alloy at 300°C. What is the temperature at which the first solid appears? What is the composition of the first solid and of the remaining liquid?
- 7. What is the composition of the last liquid to freeze at about 232°C? How much liquid is left to solidify at that temperature, if any?
- 8. Now compare what you would see as microstructure for Sb-Sn and Pb-Sn at room temperature in terms of the phases present and their percent amount.
- 9. A steep liquidus line leads to a high constitutional supercooling, therefore to high instability ahead of a growing solid/liquid interface. Comparing the phase diagrams of Pb-Sn and Sb-Sn, which of the two alloys is expected to produce more dendrites?

All pure crystalline solids, including metals, have the property of melting and freezing at a single temperature. The metal's temperature decrease is due to the thermal conduction of the metal, both liquid and solid forms. At the melting/freezing point the heat loss continues, but the temperature stops dropping; the heat of fusion released by the solidifying metal exactly balancing the heat lost to the environment. This "thermal arrest" in the metal's cooling curve is exactly analogous to an ice bath; as long as there is metal in both the solid and liquid phases the temperature will remain constant at the metal's melting/freezing point. As soon as the last bit of liquid solidifies the temperature

drop will resume, although at a somewhat slower rate due to the lower thermal gradient and conductivity of the metal.

Some combinations of metals have a eutectic composition (Greek for "easy melting"), the eutectic being the alloy composition that has the lowest melting point (minimum in the liquidus curve), and the property of melting/freezing at a single temperature like a pure metal. Thus, the cooling curve of an alloy of eutectic composition will look like that of a pure metal.

It is also characteristic of a eutectic that it transforms from a single uniform liquid phase to as many solid phases as there are metallic components in the alloy. In the case of binary alloys, such as copper-silver, there will be two solid phases upon complete solidification of the eutectic composition. This typically results in a finely laminated "eutectic structure". A small amount of *supercooling* may occur just before solidification has a chance to begin (A eutectic should not be confused with an eutectoid; the eutectoid transforms from a single solid phase to as many different solid phases (distinct from the initial phase) as there are metallic components in the alloy and typically forms a finely laminated structure, but does so entirely in the solid state, starting and finishing as a solid.)

In alloy compositions other than the eutectic and the pure metal, different phases of the alloy melt/freeze at different temperatures, resulting in a "mushy" melting zone that spans a range of temperatures. Although the slope of the cooling curve decreases due to the heat released from the solidifying metal, there is no thermal arrest in this zone since there is no pure metal or eutectic to cause one. As one phase of the alloy solidifies preferentially, the composition of the solid and liquid change continuously with temperature. When the last bit of liquid has solidified the cooling curve becomes steeper since there is no longer any heat of fusion being released. The slope of the curve after solidification will be somewhat less steep than before solidification since the solid metal has a lower thermal conductivity and difference in temperature from the environment. For some alloys of a eutectic forming alloy system, the compositional shifts that occur during solidification will cause the last bit of liquid to solidify to reach eutectic composition. This liquid, being of eutectic composition, will behave like a eutectic and will cause a thermal arrest at the eutectic temperature until all of it has solidified

Laboratory Construction of a Eutectic Phase Diagram

For this lab you will construct a binary metallic phase diagram of tin and bismuth.

Samples

Eleven molten bismuth - tin alloys are located in the furnaces, the compositions are:

100%Bi
40 Bi - 60 Sn
90 Bi - 10 Sn
30 Bi - 70 Sn
80 Bi - 20 Sn
20 Bi - 80 Sn
20 Bi - 30 Sn
10 Bi - 90 Sn
60 Bi - 40 Sn
10.57 Bi - 43 Sn
11.100% Sn

Each lab session will determine the cooling curves for only two alloys. When all lab sessions are completed the data for all compositions will be posted.

Procedure

- 1. With your TA's help, calibrate the strip chart recorder and thermocouple.
- 2. Remove one crucible of molten alloy from the furnace and place it securely into the cooling unit.
- 3. Insert the thermocouple into the narrow protection tube immersed in the alloy. Turn on the strip chart recorder.
- 4. Allow the alloy to cool undisturbed. Note the changes in slope of the cooling curve as the temperature of the alloy slowly decreases.
- 5. Once the temperature drops below 110°C turn off the recorder.
- 6. Repeat steps 2 through 5 for your second alloy.

Note that the chart converting the thermocouple output in millivolts to temperature assumed a cold junction of 0° C. With your junction at room temperature a correction must be added. The correction can be done by either:

- 1. Adding 0.04 mV per °C above 0°C to each mV reading on the chart.
- 2. Adding the room temperature to the temperature obtained from the chart.

Data

- 1. In your note book, make an accurate copy of the temperature vs. time curve which were drawn by the recorder. Suitably label axes and points.
- Label on your curve the exact temperature where changes in slope occur and indicate what metallurgical changes were occurring. Label all phases occurring in each of the regions of the cooling curve.
- 3. Give the TA a table indicating the alloy composition and the temperatures where each alloy started and finished solidification.

4. After the last lab is finished, all collected data from each alloy will be posted to the course web site. From this data draw the liquidus and solidus lines for the Pb-Sn eutectic phase diagram.

Questions

These are just a few of the questions that should be incorporated into the write-up of your lab:

- 1. In the Bi-Sn binary phase diagram, at what compositions does thermal arrest occur?
- 2. What is Newton's law of cooling? What is *supercooling*?
- 3. Discuss each trend of the cooling curve at the eutectic point and 80Bi-20Sn composition.
- 4. What are the crystal structures of Bi and Pb?
- 5. Describe the microstructure that exists within each of the six distinct regions of the phase diagram and how they develop during solidification.
- 6. Discuss in detail the microstructural development of the: and 20Bi- 80Sn alloy compositions during cooling.
- 7. Discuss two reasons why you would you choose to specify a 19.2%Bi 61.9%Sn composition for a soldering application?