2-Component Solid/Liquid Phase Diagram

Introduction

In this experiment the phase diagram of a 2-component solid/liquid system will be constructed from cooling curves. A 2-component mixture of known composition is heated until it is completely melted. It is then cooled in an air bath, and the temperature is recorded at regular intervals. The temperature will drop steadily until the solid starts to separate out; then the heat of fusion will then tend to cause the temperature to level off. Two cases are possible and are shown in figures 1 and 2. In the case of a pure substance or a eutectic mixture the temperature will remain constant until all of the liquid has solidified. For mixtures other than eutectic mixtures, the temperature will drop much more slowly after the solid begins to separate out, but it will continue to drop until the eutectic temperature is reached. The temperature will then remain constant until all of the liquid has solidified; it will then again drop rapidly.

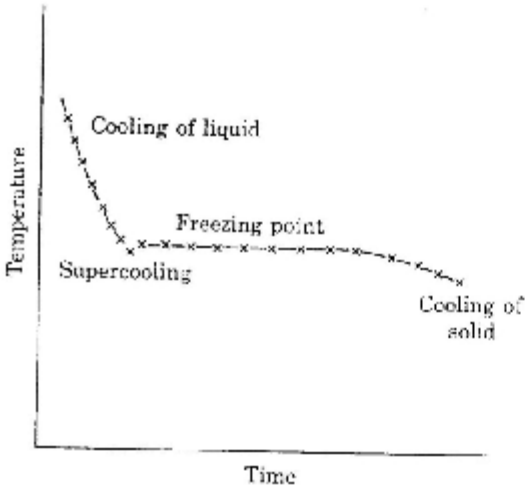

Figure 1
Cooling curve for a pure substance

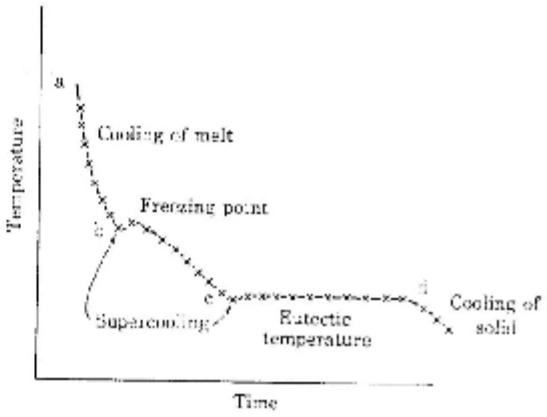

Figure 2
Cooling curve for a two component system

By putting together the information obtained from the cooling curves for mixtures of different compositions, a phase diagram can be constructed. (See Atkins, page 189 in Chapter 6, 8th edition) In a phase diagram the temperature is plotted against the total composition of the corresponding mixture. A curve is drawn from the melting point of the pure substance through the freezing points of the mixtures to the eutectic point. There will be two of these lines for each eutectic.

For the simple system that we are investigating in this lab, each solid separates out as a different solid phase. (With metals we have alloy formation to worry about.) The area below the freezing point line, but above the eutectic temperature, represents a mixture of liquid melt and the pure-solid substance whose composition lies on the same side of the eutectic composition. The area below the eutectic temperature and lying between the composition of any two pure substances represents a solid mixture of these two solid substances.

In this experiment the 2-component system of naphthalene and para-dichlorobenzene will be investigated. This exercise should give you the ability to understand and interpret simple 2-component solid/liquid phase diagrams. Although the main application and significance of these diagrams is in the area of alloys and metal melts, the temperature range of these two organics makes the system much easier to study than metals and provides a good model.

Procedure

You should add your material to an approximately 20 x 150 mm test tube. About 5 g of total material will give a satisfactory depth with this size of test tube. Melt the material by putting the test tube in a beaker of hot water (under boiling), or alternatively, heat the test tube with a Bunsen burner until the sample is completely melted. Then place the test tube into a larger test tube (about 25 x 100 mm) and then place the entire assembly in a beaker of water at about 20°C as in Figure 3. In obtaining data for the cooling curve, the temperature of the water in the large beaker should be kept at least 5 degrees below the temperature of the melt. This differential can be maintained by adding crushed ice to the water if needed.

Record the temperature every 30 seconds (more often during the first couple of minutes), using an ordinary 110°C thermometer. Stir thoroughly, using a wire stirrer or the thermometer, to ensure equilibrium. In each case continue cooling until the mixture completely solidifies and the temperature again begins to drop. After you complete your trial, reheat to melt the mixture and pour the used material into the waste container. The test tube can be washed if needed with a little acetone and dried to prepare for the next mixture.

Obtain cooling curves for mixtures of naphthalene and p-dichlorobenzene containing 0, 15, 30, 45, 60, 75, 90 and 100 percent p-dichlorobenzene by weight.

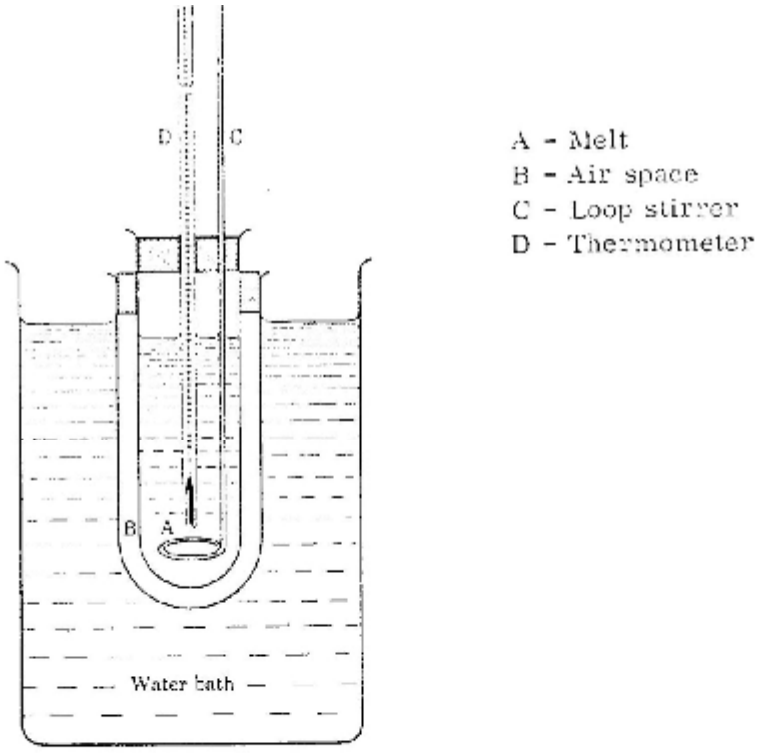

Figure 3. Apparatus for cooling curve.

Calculations

Draw the cooling curves (temperature vs. time) for each of the mixtures. From these cooling curves determine the freezing and eutectic temperatures. Draw and label the phase diagram, using weight percentages of p-dichlorobenzene on the concentration axis. No propagated error analysis is needed on this experiment.

Safety Precautions

This lab involves the use of a chlorinated hydrocarbon, para-dichlorobenzene. It is advised that women who are pregnant or who feel they might be should not handle this substance. All others should do all experimentation after weighing out the substances under the hood. Gloves should be used at all times.