# Experiment No. 1

Objective - To study the cooling curve of a given Pb - Sn alloy and determine its composition.

Theory -
The knowledge of phase diagrams and accompanying solid state transformations is essential to synthesize alloys of variable compositions and desired properties. A simple technique of understanding phase diagrams is to study time vs. temperature curve of an alloy undergoing cooling with a uniform rate. The resulting thermal curve reveals internal energy changes during cooling.

Due to the release of internal energy difference of two phases (latent heat), the temperature does not fall during solidification and a horizontal part in the thermal curve is obtained. When one of the constituents of the alloy undergoes a first order thermodynamic transition (namely freezing) and the other constituent remains as such, the temperature of the alloy will exhibit a change from original slope depending on the relative percentage of the two constituents. Below is graph in temperature (°C) and time (sec).

When temperature of the allow falls to a value at which second constituent is also undergoing first order changes, then thermal curve will again exhibit horizontal slope, or change in slope depending upon whether in the meanwhile first component has, or, has not transformed its structure completely. Hence study of thermal curve will reflect the phase transformations of different constituents.

All phase transformations in alloys follows Gibbs phase rule.

For a binary alloy: degree of freedom = 4 − p. Where 'p' denotes the no. of phases. Degree of freedom is equal to the no. of variable parameters externally controllable - temperature, pressure and composition. Usually one degree of freedom namely pressure, is fixed during cooling as experiment is done at atmospheric pressure. The balance of degree of freedom are 3 − p, thus at point in phase diagram where three phases coexist the degree of freedom is zero (i.e. unique composition and temperature). Two phases coexist over a range of temperatures.

The temperatures corresponding to points where changes in slope occur in the cooling curve (Eg - points b, c, in Fig. 1) depend on the composition of the alloy. Given a known phase diagram and an experimentally obtained cooling curve these arrest points can be used to find composition of the alloy. Temperature of the allow is measured using a potentiometer attached to a copper-constantin thermocouple which must be kept embedded in the alloy.

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Procedure -

1. The unknown alloy is put in an alumina or porcelain crucible and placed into the center of the heating unit.
2. Before staring the heating run (temp) please ensure that the set temperature is set above 250 °C.
3. After the set temperature is reached (Alloy is melted), switch off the heating.
4. The temperature readings are taken every 15 - 20 seconds.

Result - The composition of Tin (Sn) and Lead (Pb) in the sample comes out to be -

Sn (Tin) : 33 % (approx)

Pb (lead) : 67 % (approx)

Precautions -

1. Do not touch solid Pb-Sn alloy.
2. Do not touch thermocouples.
3. Use stopwatch to count the time and temperature.
4. Note temperature carefully (without rounding off, only reading).

Teacher's Signature

Mahak

Aim - To study the cooling curve of a given Pb - Sn alloy and determine its composition.

Materials Required - Alloy, alumina or porcelain crucible, heating unit.

Observation Table -

| Time (sec) | Temp (°C) | Time (sec) | Temp (°C) |
|---|---|---|---|
| 0 | 280 | 190 | 270.1 |
| 10 | 280 | 200 | 269.5 |
| 20 | 279.8 | 210 | 268 |
| 30 | 279.4 | 220 | 267.1 |
| 40 | 279 | 230 | 266.5 |
| 50 | 278.7 | 240 | 265.5 |
| 60 | 278.5 | 250 | 264.4 |
| 70 | 278 | 260 | 263.7 |
| 80 | 277.6 | 270 | 263 |
| 90 | 277.2 | 280 | 262 |
| 100 | 276 | 290 | 261.3 |
| 110 | 275.2 | 300 | 260.4 |
| 120 | 274.8 | 310 | 259.7 |
| 130 | 274.2 | 320 | 258.9 |
| 140 | 273.6 | 330 | 258.2 |
| 150 | 272.9 | 340 | 257.3 |
| 160 | 272.2 | 350 | 256.8 |
| 170 | 271.5 | 360 | 255.7 |
| 180 | 270.9 | 370 | 254.8 |

Mehek

| Time (sec) | Temp (°C) | Time (sec) | Temp (°C) |
|---|---|---|---|
| 380 | 254.1 | 640 | 234.3 |
| 390 | 253 | 650 | 233.4 |
| 400 | 252.3 | 660 | 232.9 |
| 410 | 251.5 | 670 | 232.4 |
| 420 | 250.7 | 680 | 231.8 |
| 430 | 250.2 | 690 | 231.4 |
| 440 | 250 | 700 | 230.7 |
| 450 | 249.4 | 710 | 230.2 |
| 460 | 248.5 | 720 | 229.9 |
| 470 | 247.6 | 730 | 229.3 |
| 480 | 246.6 | 740 | 228.6 |
| 490 | 246.1 | 750 | 228.1 |
| 500 | 245.3 | 760 | 227.6 |
| 510 | 244.6 | 770 | 226.9 |
| 520 | 243.8 | 780 | 226.4 |
| 530 | 242.9 | 790 | 225.8 |
| 540 | 241.9 | 800 | 225.2 |
| 550 | 241.1 | 810 | 224.7 |
| 560 | 240 | 820 | 224.1 |
| 570 | 239.3 | 830 | 223.4 |
| 580 | 238.6 | 840 | 222.7 |
| 590 | 238 | 850 | 222.2 |
| 600 | 237.1 | 860 | 221.6 |
| 610 | 236.2 | 870 | 221 |
| 620 | 235.6 | 880 | 220.4 |
| 630 | 235 | 890 | 219.7 |

Mahek

| Time (sec) | Temp (°C) | Time (sec) | Temp (°C) |
|---|---|---|---|
| 900 | 219.1 | 1160 | 199.4 |
| 910 | 218.5 | 1170 | 198.5 |
| 920 | 217.9 | 1180 | 197.7 |
| 930 | 216.9 | 1190 | 197 |
| 940 | 216.2 | 1200 | 196.2 |
| 950 | 215.5 | 1210 | 195.4 |
| 960 | 215 | 1220 | 194.7 |
| 970 | 214.1 | 1230 | 194 |
| 980 | 213.5 | 1240 | 193.3 |
| 990 | 212.7 | 1250 | 192.6 |
| 1000 | 211.9 | 1260 | 191.9 |
| 1010 | 211.4 | 1270 | 191.1 |
| 1020 | 210.5 | 1280 | 190.4 |
| 1030 | 210 | 1290 | 192.8 |
| 1040 | 209.2 | 1300 | 189.1 |
| 1050 | 208.8 | 1310 | 188.4 |
| 1060 | 207.7 | 1320 | 187.2 |
| 1070 | 206.3 | 1330 | 186.5 |
| 1080 | 205.6 | 1340 | 185.9 |
| 1090 | 204.9 | 1350 | 185.9 |
| 1100 | 204 | 1360 | 185.8 |
| 1110 | 203.1 | 1370 | 186 |
| 1120 | 202.3 | 1380 | 186.2 |
| 1130 | 201.4 | 1390 | 186 |
| 1140 | 200.8 | 1400 | 186.2 |
| 1150 | 200 | 1410 | 186.2 |

Mahek

| Time (sec) | Temp (°C) | Time (sec) | Temp (°C) |
|---|---|---|---|
| 1420 | 186.2 | 1680 | 186 |
| 1430 | 186.3 | 1690 | 185.9 |
| 1440 | 186.3 | 1700 | 186.1 |
| 1450 | 186.3 | 1710 | 186.1 |
| 1460 | 186.5 | 1720 | 186 |
| 1470 | 186.3 | 1730 | 185.9 |
| 1480 | 186.4 | 1740 | 185.9 |
| 1490 | 186.4 | 1750 | 185.9 |
| 1500 | 186.4 | 1760 | 185.9 |
| 1510 | 186.2 | 1770 | 185.9 |
| 1520 | 186.2 | 1780 | 185.8 |
| 1530 | 186.2 | 1790 | 185.8 |
| 1540 | 186.2 | 1800 | 185.6 |
| 1550 | 186.1 | 1810 | 185.3 |
| 1560 | 186.1 | 1820 | 185.3 |
| 1570 | 186.1 | 1830 | 185.3 |
| 1580 | 186.1 | 1840 | 185.2 |
| 1590 | 186.1 | 1850 | 185.1 |
| 1600 | 186 | 1860 | 185 |
| 1610 | 186 | 1870 | 184.7 |
| 1620 | 186 | 1880 | 184.2 |
| 1630 | 186 | 1890 | 183.6 |
| 1640 | 186 | 1900 | 183.1 |
| 1650 | 186 | 1910 | 181 |
| 1660 | 186 | 1920 | 179.3 |
| 1670 | 186 | 1930 | 177.6 |

Mahek

| Time (sec) | Temp (°C) |
|---|---|
| 1940 | 176 |
| 1950 | 174.3 |
| 1960 | 172.8 |
| 1970 | 171.2 |
| 1980 | 176.9 |
| 1990 | 168.3 |
| 2000 | 166.7 |
| 2010 | 165.4 |
| 2020 | 164 |
| 2030 | 162.7 |
| 2040 | 161.4 |
| 2050 | 160.2 |
| 2060 | 159.9 |
| 2070 | 156.8 |
| 2080 | 155.8 |
| 2090 | 155 |
| 2100 | 154.1 |
| 2110 | 153.4 |
| 2120 | 152.7 |
| 2130 | 152.2 |
| 2140 | 151.9 |
| 2150 | 151.3 |
| 2160 | 150.9 |
| 2170 | 150.4 |
| 2180 | 159.8 |

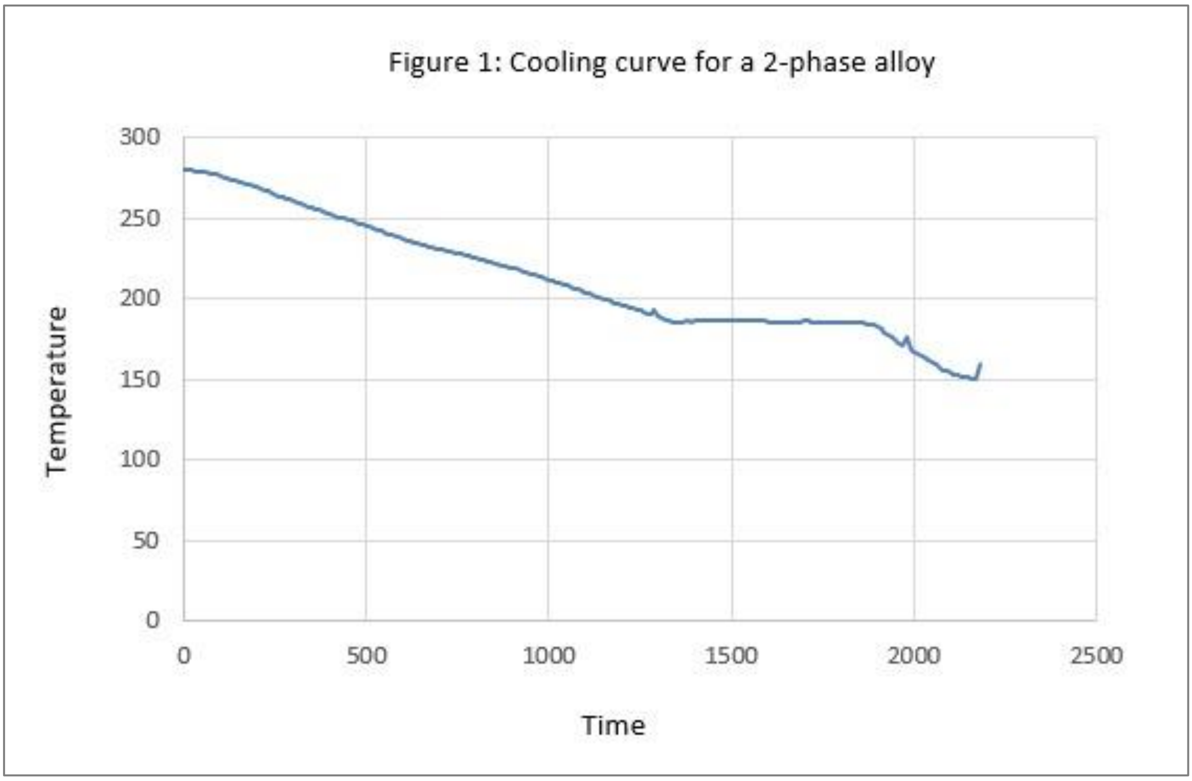


Figure 1: Cooling curve for a 2-phase alloy
Temperature
Time
300
250
200
150
100
50
0
500
1000
1500
2000
2500

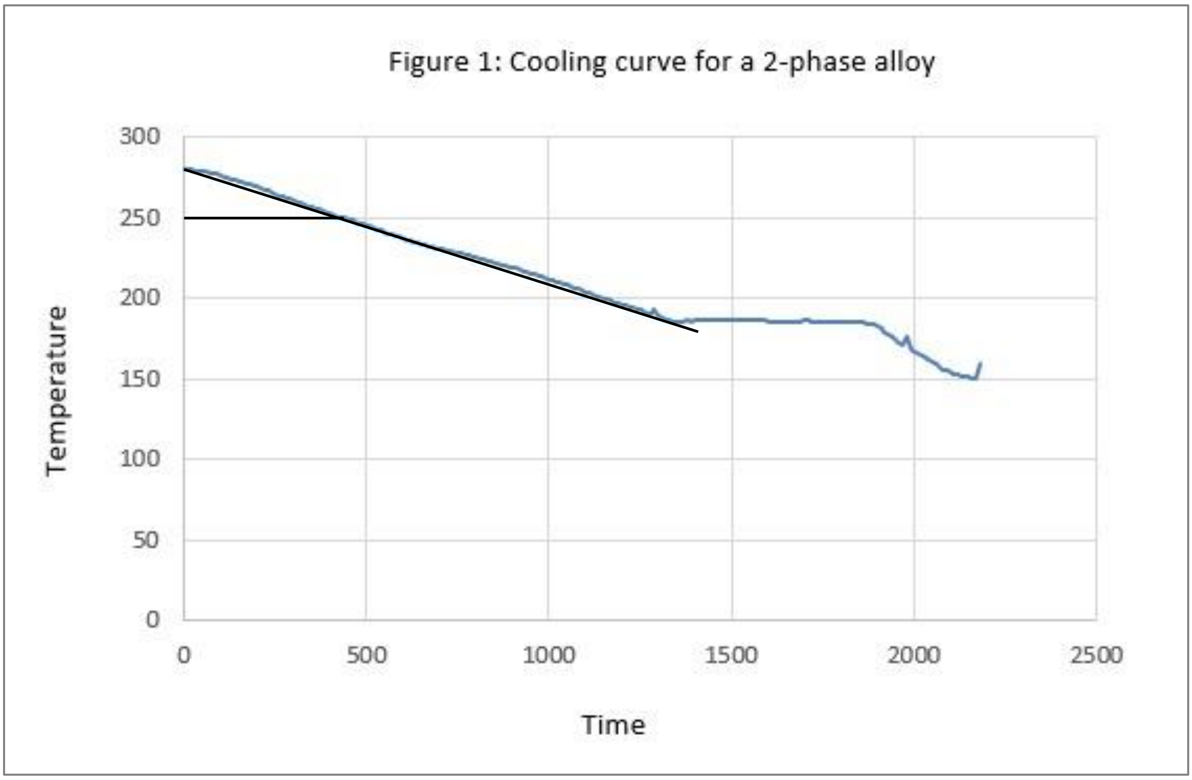
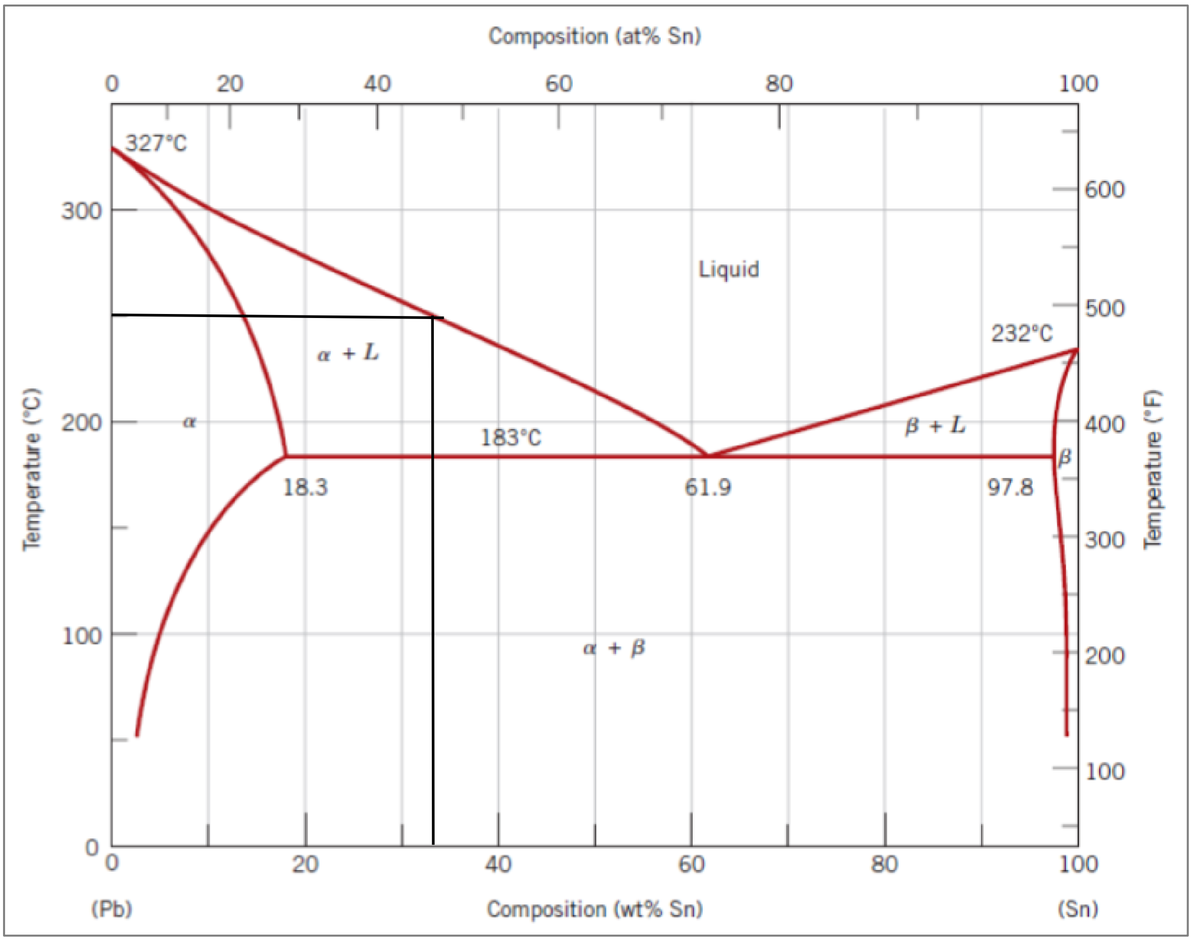


Figure 1: Cooling curve for a 2-phase alloy
Temperature
Time
300
250
200
150
100
50
0
500
1000
1500
2000
2500


Composition (at% Sn)
0
20
40
60
80
100
327°C
300
200
100
0
600
500
400
300
200
100
Liquid
232°C
α + L
α
183°C
β + L
β
18.3
61.9
97.8
α + β
Temperature (°C)
Temperature (°F)
0
20
40
60
80
100
(Pb)
Composition (wt% Sn)
(Sn)