Thermal analysis of cooling curves for phase diagram determination

1P6 Thermal Analysis

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Senior Demonstrator: Dr Enzo Liotti
Junior Demonstrator: Mr Marco Siniscalchi

Safety

Hazards	Control measures
High temperature molten metal	• Safety glasses and lab coat must be worn throughout entire time when in the lab. • Lab coat, safety glasses and visor, thermal resistant gloves, aprons and shoe protector are worn when working in the furnace room. • Follow instructions, training and supervision.
Incorrect operation of furnaces	• Follow instructions, training and supervision.

Safety Rules:

- Demonstrators **must** be present when you are working with molten metal.
- Request permission from the demonstrators before operating the furnaces.
- The furnaces and temperature inside the sample tube must not be allowed to go above 300°C.
- Listen and follow instructions carefully. If you do not understand instructions, then ask before you perform the tasks.

Contents

1. Objectives and Learning Outcomes

1.1. Objectives and Science

The overall objective of this practical is to demonstrate how a phase diagram of an alloy system can be obtained experimentally. The second objective is to learn how to use latest software techniques, such as LabView (Laboratory Virtual Instrument Engineering Workbench) and MATLAB (MATrix LABoratory), to collect, organise and analyse scientific data. This practical links with the lecture courses on thermodynamics and microstructures.

In this practical, an alloy system will be studied, by analyzing the cooling curves of samples with different composition, and its phase diagram determined. The cooling curves will reveal the beginning and end of solidification, pure metals and eutectics freeze at one temperature whereas other compositions within a range. The Phase Rule model predicts this phenomenon.

The practical involves the development of both practical and computing skills essential to become a successful material scientist.

1.2. Practical Skills

The work will involve the measurement, interpretation of cooling curves, and producing an alloy of uniform composition. It is expected that the practical will involve:

- Operate a simple tube furnace;
- Learn how to handle molten metals safely;
- Learn how to set up equipment to conduct simple measurements;
- Understand the basic working principles of thermocouples and use them to measure the temperature of a sample;

1.3. Computer Skills

- Use LabView to control hardware equipment and record temperatures;
- Use MATLAB to import datasets and develop a script that analyses the data.

2. Overview of the Practical

On Day 1 you will find eight tube furnaces loaded with prepared samples all from the same X-Y alloy system. During the practical you will prepare the experimental set up, test the eight samples and also be given the data of a ninth sample to analyse while the experiment is running. The nine prepared samples are: one of unknown pure element X, one of unknown pure element Y (not Yttrium) and the others are alloys with various compositions, as given in Table 1.

Sample	Composition (wt%)
A	100X
B	97X – 3Y
C	90X – 10Y
D	70X – 30Y
E	60X – 40Y
F	48X – 52Y
G	30X – 70Y
H	11X – 89Y
I	100Y

Table 1: Summary of all sample compositions.

Working as a group, you will record the heating and cooling curves of one of these prepared samples and share the data with the other groups. You will analyse the datasets (individually, not in group) using MATLAB and the results used to construct the phase diagram for the X-Y system.

In addition, on Day 1 each group will also choose a composition to make their own sample S_n, where n is your group number, which will be analysed on Day 2. The dataset (but not the compositions) will be shared with the other groups, and using the

phase diagram constructed from the results of the analysis of the samples in table 1, you will determine the composition of all the other groups' S_n alloys. The practical overall expected results are:

- X-Y phase diagram;
- Identification of the alloy system from the constructed phase diagram;
- Identification of all the S_n sample compositions;

2.1. Practical Timetable

Day 1:

1. Set up furnaces and measuring equipment;
2. Measurement of heating and cooling curves for the prepared sample;
3. Preparation of alloy S_n of composition ___%X and ___%Y;
4. Preliminary MATLAB data analysis of sample E.

Day 2:

1. Analysis of datasets A-I and S_n samples from other groups;
2. Determination of phase diagram and composition of S_n samples.

2.2. List of files

The practical involves the use of several files, which are found in the folder "Practical_1P6_Thermal analysis" on the desktop of the shared laptop connected to the furnaces. The folder contains the following files and subfolders:

- DataAcquisition_LabView: this folder contains all the necessary files to run the LabView user interface to record the heating and cooling curves;
- "NI USB 9211a manual.pdf": manual for the NI module;
- "s_ThermalAnalysis_1P6.m": Matlab script to be used to analyse the heating and cooling curves;
- Sample E data file;

3. Experimental Method

3.1. Measuring Heating and Cooling Curves

For this practical, each group is provided with one manually controlled tube furnace, a shared laptop running LabView for data acquisition and shared data logging hardware. There are four data logging units each of them connected to a laptop and recording the temperature of two experiments. The data logging is carried out using the National Instrument hardware and software: a NI 9211 thermocouple module, which read a voltage and convert it to temperature, is connected to a laptop via a cDAQ-9171 USB module. The module enables the communication between the thermocouple module and the computer. Each module can read 4 thermocouples simultaneously and it is controlled by LabView.

The temperatures will be recorded using thermocouples provided. Thermocouples are electrical device consisting of two dissimilar conductors forming electrical junctions at differing temperatures. A temperature-dependent voltage is produced as a result of the thermoelectric effect, and this voltage can be interpreted to measure temperature. There are several types of thermocouples differing in the combination of the alloys employed; in this experiment, we will use K-type thermocouples (chromel-alumen), which are well suited for measuring temperatures of non-reactive molten metals in the range −200 °C to +1350 °C.

3.2. Task 1

On Day 1, the specimens will have been made up already in crucibles and loaded into the tube furnace, but you will have to set up the data logging equipment and modify the given LabView code in order to be able to start the experiment.

As a first task connect the thermocouples to the module, remember that they have a positive and a negative end and that polarity is important so attach them the right way around! (**Hint**: thermocouples wire colors are standard, Google knows the answer, but you must ask the right question…).

When all the thermocouples are connected, launch LabView and open the file "Thermocouple Data Acquisition_v2.vi", which is in the folder named "DataAcquisition_Labview" within the practical main folder. The program window is the front panel of the code, which was written for this practical (Fig. 1). Press Ctrl+E to open the underlying block diagram (Fig. 2).
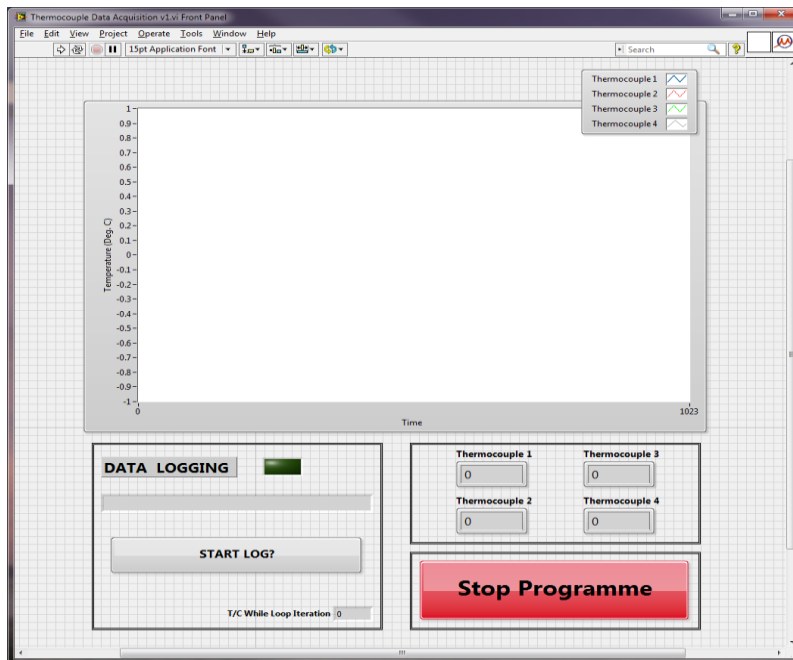
Fig. 1: Front panel of the LabView data logging program. Press Ctrl+E to see the block diagram. Press the run button (⇨) to launch the program.
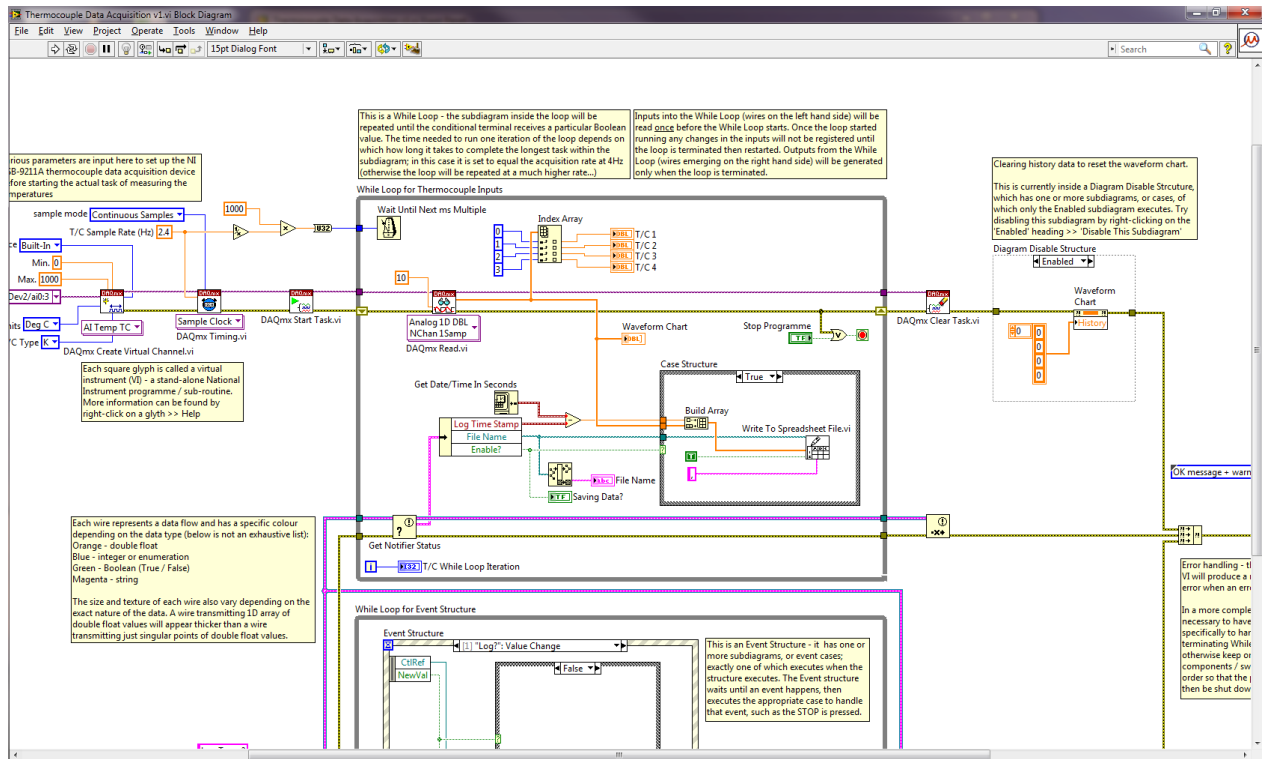
Fig. 2: Block diagram running behind the front panel.

3.3. Task 2

As a second task familiarize with the diagram and compare it with the front panel. Try to understand the functioning and discuss with your group the following questions:

- Where does the code begin?
- How many loops are there in the diagram?
- What is their role?
- How often do the loops run?

Once you think you understood the overall functioning of the diagram return to the front panel window and try to run it pressing the run button (⇨) on the top left to check if the thermocouples are connected. If there are no issues, you should be able to read the four temperatures in the central plot.

3.4. Task 3

If you get the program working, you will notice that the acquisition rate is very low. This was intentional and it is your next task to change it to a suitable value. To do so you need first to find out how to reset it and then to work out which would be a suitable acquisition rate.

Stop the program pressing the red button 'Stop Programme'. Do not stop it in any other way as it can cause problem of communication between the computer and the NI modules, in which case you might have to restart LabView.
Go to the block diagram and try to identify where the sampling rate is set. When you find it, try to reset it to another value and test if it works.

Now that you know how to correct the acquisition rate, you will then need to choose an appropriate value for the parameter (think carefully!). (**Hint**: you might find some help in the NI 9211 module manual).

3.5. Operating the Furnaces

Start the data log pressing 'Start Log?' and give the file a meaningful name. Remember that each computer is reading the temperature of two experiments. Thus, write down which channel correspond to your experiment in your lab-book.

The furnace is controlled by a Eurotherm temperature controller. Once you are ready to operate the furnace, it is **imperative** that a demonstrator supervises and provides explicit permission to switch on the furnaces.

Set the target temperature to 300°C and with the demonstrator's permission, switch on the controller. One group member must always remain by the equipment to monitor the sample's temperature. Once the temperature inside the sample tube has reached 300°C, notify the demonstrators and they will instruct you to switch off the furnace.

Two group members now prepare their own alloy, specimen S_n (see instructions below), which will be analysed by the staff before the beginning of Day 2. The

remaining group members relocate to the computer room to begin the MATLAB data processing (see Section 4.).

At the end of Day 1, check the temperature of the sample; if it is below 100°C, you can stop the data logging. However, remember that a laptop is recording data from two furnaces so check both temperatures. If the temperature is above 100°C, then notify the demonstrators. All data, including the S_n samples, will be collected at the beginning of Day 2. **It is your responsibility to collect the data.**

3.6. Preparing Sample S_n

In order to prepare specimen S_n, you must decide on its composition. Weigh the appropriate quantities of pure X and pure Y using the raw materials provided to give a total weight of 100g. You should take a note of the actual weights and equipment used (as this might be useful in your error discussion). Place X and Y together in the supplied crucible and let the Demonstrators know you are ready for further instructions. You must not enter the furnace room without supervision.

Under the supervision of the Demonstrators, put on protective clothing (visor, gloves, apron and shoe spats). With their guidance, place the crucible into the furnace pre-heated to 350°C using the long-handled tongs. Secure a stainless steel test-tube in a retort stand ready to receive the molten metal.

After 30 minutes, insert the thermocouple into the mold, remove the crucible containing the molten metal and stir with a graphite rod prior to pouring the molten metal into the steel test-tube. Allow the specimen to cool for 10 minutes. Specimen S_n is now ready for analysis.

4. Data Analysis

The analysis of the collected heating and cooling curves will be carried out using MATLAB to determine the temperatures where solidification starts and finishes and construct the phase diagram. You have been given a datasets for each of the nine composition in table 1 (sample A→I), and eight S_n sample datasets with unknown composition. **Working individually** you will analyse the data for sample A to I to construct the phase diagram, identify the alloy system and then use the phase diagram to identify the composition of the S_n samples. You should analyse each data set using the supplied MATLAB script titled 's_ThermalAnalysis_1P6.m'. The script will

guide you through the entire data analysis process and at the end you should be able to construct the phase diagram. Following the instructions, start to prepare a plot of the heating and cooling curves for this alloy, determining the temperatures where solidification starts and finishes. Consider how this information will contribute to your construction of the phase diagram. You will also be given a phase diagram from the literature of the alloy system, which you can use a comparison.

5. Lab notebook notes

For general good practice and advices about how to take notes in your lab notebook refer to `*Keeping a good Lab notebook: some advice and examples of good practice'* (Prof. Lozano-Perez).

For this specific practical, on top of the data analysis, results and conclusions the notebook should also include notes about the experimental set up. In particular, you should annotate all the information needed to replicate the experiments without duplicating the handout instructions. For instance, write down the file name and path were you saved the raw data and detailed instructions and information about the experimental set up. Using sketches and diagrams is encouraged as it improves clarity and helps to keep notes compact.