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Chapter III

Experimental Part

Computer facilities

The fast automatic recording is performed by a computer. The experiments use a micro computer with CPU No. 80386 SX and a hard disk of 130 MB size. The read access memory installed in this computer was 8 megabytes. Since the amount of data from each experiment is very large, high memory will improve the speed for data analysis. The main board of this computer has six 16 bits slots and two 8 bits slots. The two 16 bits slots are used to install the AT I/O card and monitor card. This is the standard electronics used in every conventional IBM compatible microcomputer. One 8 bit slot is used to install an analog to digital conversion card (A/D card).

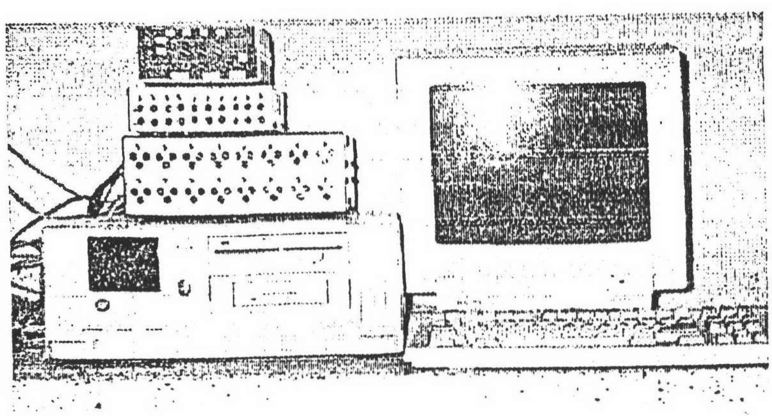

Figure 3.1 The computer that used in the experiments.

Analog to Digital card (A/D card)

The analog to digital card (code name:PCL 812 PG) is very useful to convert the analog signal from thermocouple and electrode to digital signals which can be processed by the computer. The card has 16 single-ended analog input channels with 12 bits resolution (12 bits can present 2^{12} = 4196 as the largest numerical; data with 12 bits resolution are precise to 3 decimals at best). A single-ended configuration has only 1 signal wire for each channel. Thus, the voltage to be measured is a DC voltage referred to a common ground. The highest conversion speed of this card is 30 KHz with 0.015% accuracy. The range of DC signal that the A/D card detects is (bipolar) $\pm 5V$, $\pm 2.5V$, $\pm 1.25V$, $\pm 0.625V$, $\pm 0.3125V$.

For this A/D card, a powerful and easy-to-use software package PC-LABDAS (1991) is available by which the individual parameter can be set by basic call statements.

The limitation of this card is the low resolution. Beside this, the input voltage cannot exceed 5 V and must be DC voltage. As to the resolution, an amplifier with multiplexer[1] board can correct this problem. As to the DC voltage, a simple electric circuit can convert the AC voltage from the resistivity probe into a DC signal.

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[1] A multiplexer is an electronic "switch" ("bus") which manages and operates many input signals sequentially (in a well-defined rythm) over one and the same amplifier unit.

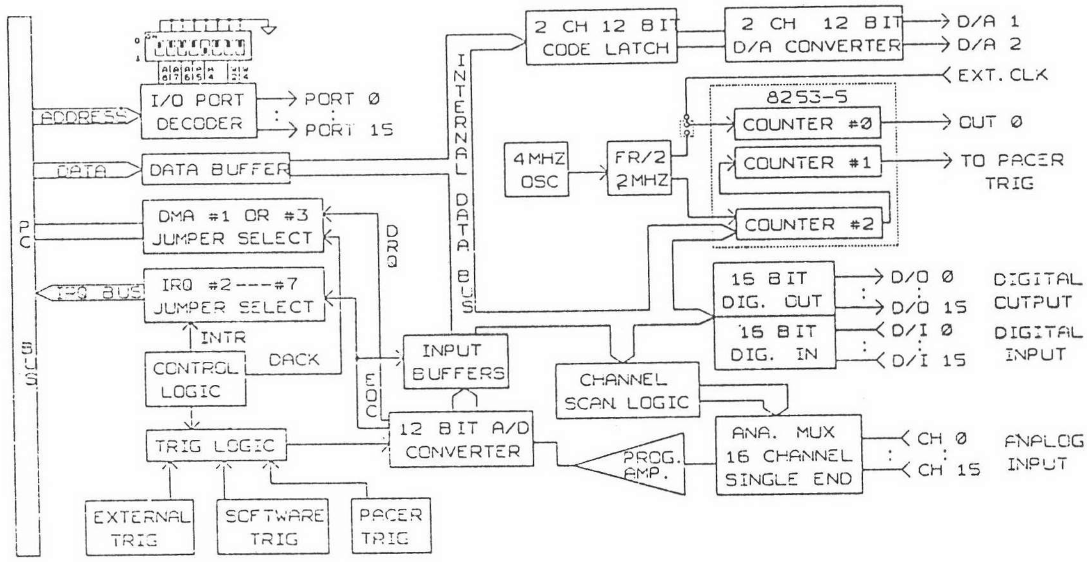

Figure 3.2 Block diagram of the analog to digital card

A. Setting the A/D card address

The A/D card has to be given an unambiguous address by which the computer can contact it. In these experiments, the address of A/D card was selected to be 220H.

B. Calibration of the card

The card has to be calibrated before use. A calibration program (Qbasic language) is available. The card was adjusted by so-called jumpers and gain controllers. After calibration, it was checked by means of a normalized DC source.

Data acquisition software

Commercial software is used to read the signals that come from the A/D card. It can control channels, gains of the amplifier, and channels of the multiplexer. After reading the signals, it writes the data to a disk in ASCII format as a *.PRN file. This file can be processed by commercial table calculation software (e.g., LOTUS 123).

Amplifier with multiplexer board

The amplifier with multiplexer board (code name: PCLD 889) is a powerful programmable amplifier and channel multiplexer[2] preparing signals for the analog signal input channels of the A/D card. This board supports both +12 volt and +5 volt power supply and has a 8 bits programmable digital control.

This board manages 16 differential input channels into one analog output channel. The amplifier with multiplexer board allows to select the gains (i.e., amplifying factors) of 0.5, 1, 2, 10, 50, 100, 200 and 1,000. It also has built-in signal conditioning functions such as filtering, attenuation, and current shunt. The maximum input range is ±10 V DC and output current is 20 mA.

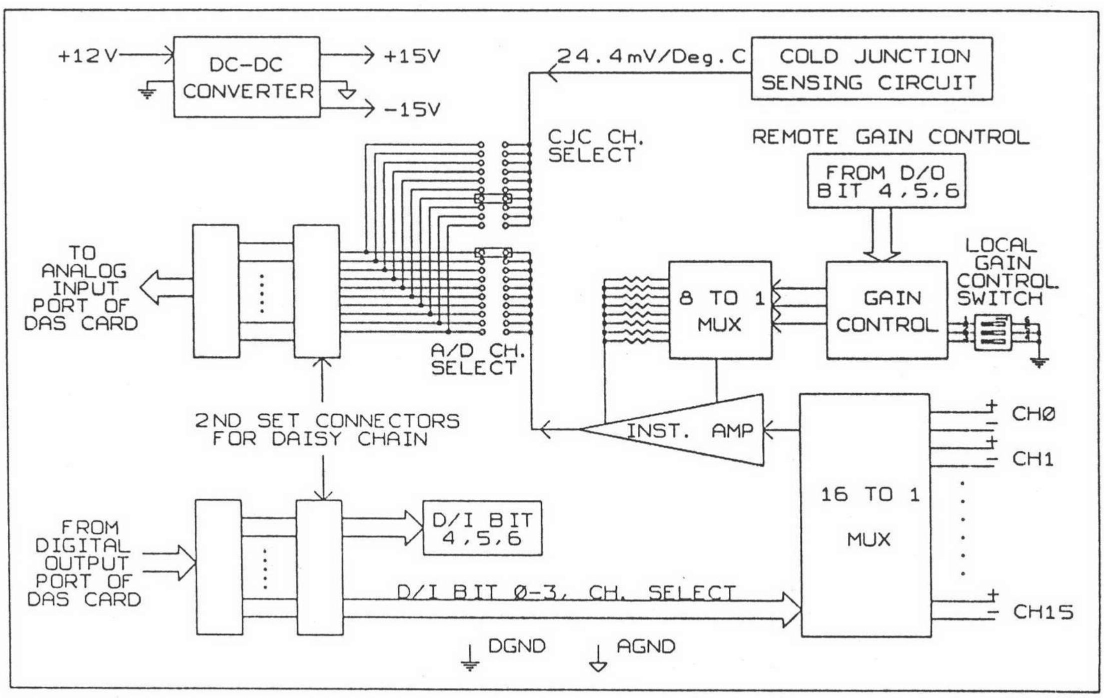

Figure 3.3 Block diagram of the amplifier and multiplexer board

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[2] Explanaion, see footnote on page 14

A. Floating, non-floating, and no ground test

1. Test with potentiometer

A standard 10 mV DC source was used. The source was connected like in figure 2.2, i.e. operating the floating, non-floating, and no ground modes. The results are presented to the graph in figure 4.1. This test can tell the importance of the setting the correct mode.

2. Test with thermocouple

This test used thermocouple as a voltage source. Thermocouple type K (NiCr-Ni) was used in this test. After the thermocouple was prepared, the three modes were tested.

B. High pass and low pass

The meaning of high pass and low pass are high frequency pass and low frequency pass. This setting is a hardware setting. Resistor and capacitor are used in this application. We can set it by a jumper of each channel of the amplifier. The amplifier was set the low pass at 417 Hz by 2.4 kohm of resistor and a 1μF capacitor. From the equation below, we can calculate the pass frequency:

$$Hz = 1/(R \cdot C)$$

A frequency lower than this frequency can pass through the circuit and any frequency higher than this frequency will be cut off. The experiment was performed to check for the effect of a pass on the collected data.

Data processing

The raw data in the experiments must be analyzed, Corrected for fluctuations, etc. We have many ways to solve this problem (See chapter 2, data processing). This experiment is done to know that what happens if data are integrated over periods of 10 seconds.

Probes (thermocouple and resistivity probe)

A. Thermocouple

The type K (NiCr-Ni) thermocouples with 0.6 mm wire diameter is selected in the experiments. The temperature probe can be made by inserting the thermocouples wire into sintered alumina tubes with 6 mm outer diameter and two 1.5 mm inner diameter bores, and twisting them together to make junctions. The icepoint measurements were performed to calibrate the thermocouple. The figure below show the sketch of icepoint measurement. The icepoint was 1.248 mV.

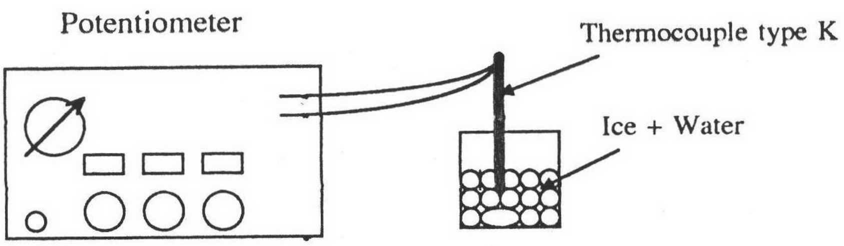

Figure 3.4 Sketch of the ice point measurement

The signal from thermocouple is a DC voltage in the range of 10 mV. It is necessary to convert the voltage signal to temperature. This can be done by a polynomial derived from the standard table of NiCr-Ni type K. The table is shown in the appendix A. The equation for the polynomial thus found is

$$T \text{ in } ^\circ C = -2.5 + 25.765X - 0.1045X^2 + 0.002X^3 \; ; \; X = mV$$

B. Resistivity probe

Resistivity probes are made from NiCr wires with 0.6 mm diameter. The ends of the NiCr wires are wound on a cylindrical rod of 5 mm diameter to make a coil-shape; 10 mm distance are left between two electrodes.

The resistivity probe should be connected to a circuit shown in figure 3.5. The circuit converts resistivity to DC voltage easily measured by the computer.

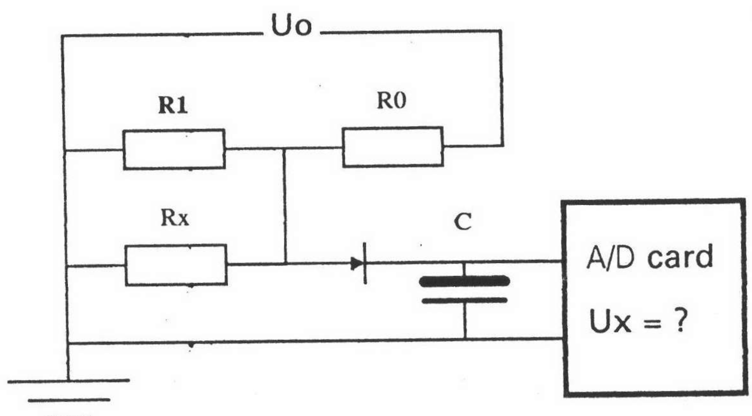

Figure 3.5 The resistivity measurement circuit with Uo = 3V AC, Ro = 10 kohm, R1 = 1 Mohm, C = 0.22 μF, Rx represents the resistance across 1 cm of batch (cross section: ≈ $1cm^2$).

From figure 3.5, the use of a AC source is very important because the AC voltage source will avoid polarization + migration phenomena in the batch. But, an output DC value will be higher than the nominal (or effective) input AC signal. The output signal should be divided by $\sqrt{2}$ to correct the value. From the circuit we will find Rx by the equations that show below.

$$\frac{Ux}{Ry} = \frac{Uo}{(Ro+Ry)}$$

Ry = R1•Rx/(R1+Rx)

Ux can convert into Rx :

$$Rx = R1 \cdot R0 \cdot Ux/[R1 \cdot (Uo-Ux)-R0 \cdot Ux]$$

Rx is converted to conductivity by :

$$\kappa = d/(A \cdot Rx)$$

with d = distance between the electrodes (1 cm), A = effective area of the electrodes (1 cm^2).

C. Probe

The probe is a combination of thermocouple and electrode. Figure 3.6 show the model of the probe.

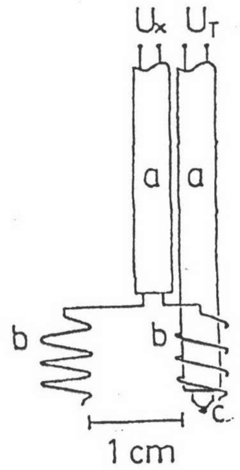

Figure 3.6 sketch of probe; a = alumina tube, b = NiCr wire, c = junction

An alternative design of the probe is known in figure 3.7. It uses Ni as the reference wire for both thermocouple and electrodes.

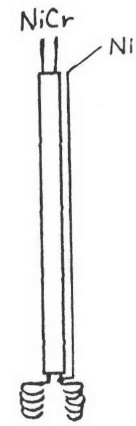

Figure 3.7 Sketch of new probe in small scale test

This new probe is used in the small scale tests. The advantages of new probe are use only 3 wires and 1 alumina tube in 1 test. The disadvantages of new probe were requirement of an absolutely ground for the test. If the ground is not an absolutely ground, noise from the electrode will interfere with the thermocouple, mode of the thermocouple has to be changed to non-floating mode.

Test in small scale

Small scale tests are important because the new instruments used have to be tested for the accuracy. These tests are easily to prepare.

A. Research furnace

An electric furnace is used in these small scale experiments. The volume is about 3 dm^3. It is constructed by a light alumina brick and has Kanthal wire (NiCr) with 1.5 mm diameter as heating elements. The power is 3 kVA at 22 A. A digital temperature controller is used to control the furnace temperature.

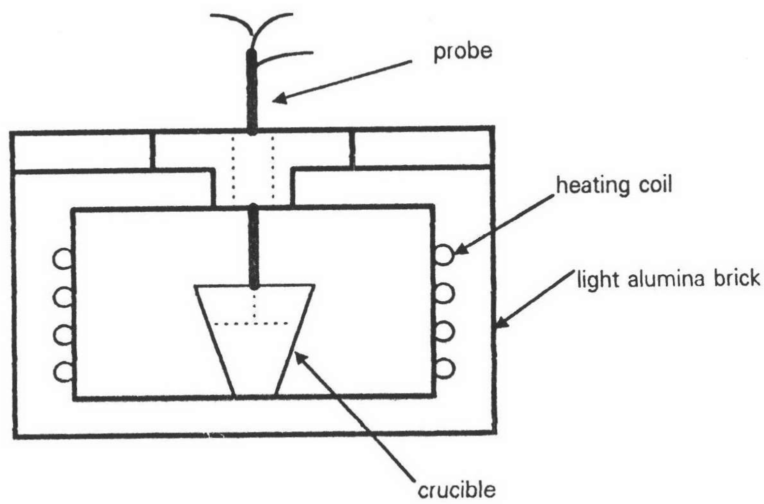

Figure 3.8 The sketch of the furnace and test.

B. Test with one component

This test was the first test of the fast automatic data recording instruments. Soda ash is selected for the experiment. Soda ash is suited to check the accuracy of the instruments because its melting point is well investigated: 858°C 120 g of soda ash was put into a small crucible with a probe. The batch was heated from room temperature to 1100 °C. The phase diagrams are shown in the appendix B.

C. Test with two components

After the test with one component, two component tests were performed to check whether or not the instruments can detect the melting behavior of more complicated systems, too. The two components selected in these experiments were soda ash and sand. These two materials were selected, mixed together at a ratio 1:1 by weight. The total amount of mixture was 120 g. The mixture and probe were positioned in the small crucible. The probe was immersed

4 cm deep in the batch. The crucible and probe were put it to the furnace and connected to the computer.

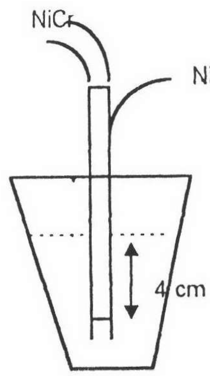

Figure 3.9 Sketch of small crucible with probe

The batch was heated from room temperature to 1100°C. The data were read at intervals of 1 s.

Large scale furnace

The furnace constructed for the large scale experiments. Consists of three major parts. This is, firstly, the lower furnace with the compartment for the cullet melt which can be heated electrically. This is, secondary, the upper furnace chamber heated by butane gas. This is, thirdly, the batch charging system.

A. The lower furnace

The electrical heater of the lower furnace is controlled by a digital temperature controller. The heating coil controls the temperature of the melt indirectly. It is positioned under the batch. The purpose of the electrical heats is to provide an independent heat source for the cullet melt, which is independent from the heat transfer in the atmosphere. The heating element of this furnace is made of

Kanthal wire with 2 mm diameter. The total resistance for the heating coil is about 10 ohm; the power is 3.0 kVA at 17.3 A. A sketch of the electrical part furnace is shown in figure 3.10.

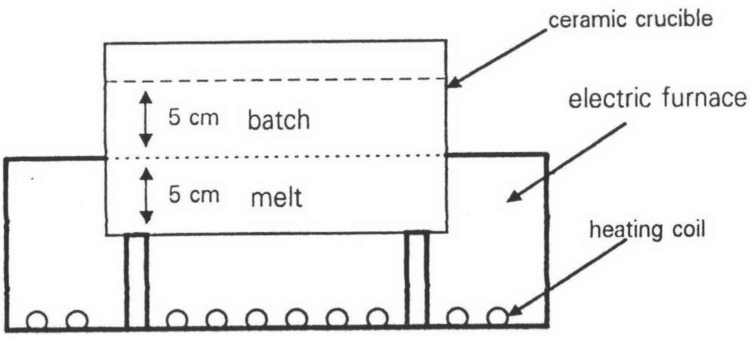

Figure 3.10 Sketch of the lower furnace with electrical heater

B. The butane gas furnace

This is the part used to control the temperature of the atmosphere. It uses butane gas with 3 burners. The burners are placed in front of the furnace and the flame rises along the front of the furnace to the top, flows past the upper side of the melting compartment, and enters the exhaust channel and exhaust tube (down dub). The exhaust tube is about 5 m long. This provides sufficient air dub. The temperature of the furnace can be controlled by adjusting the pressure of butane gas, and the air opening of the burners. The furnace lining is made of local light insulation fire bricks.

The melt compartments are rectangular ceramic crucibles made of fire clay. They are 26.5 cm long, 22.5 cm wide, and 13 cm deep (inner dimensions) and has a wall thickness of approximate 1.5 cm. The melting area thus is approx. 600 cm^2.

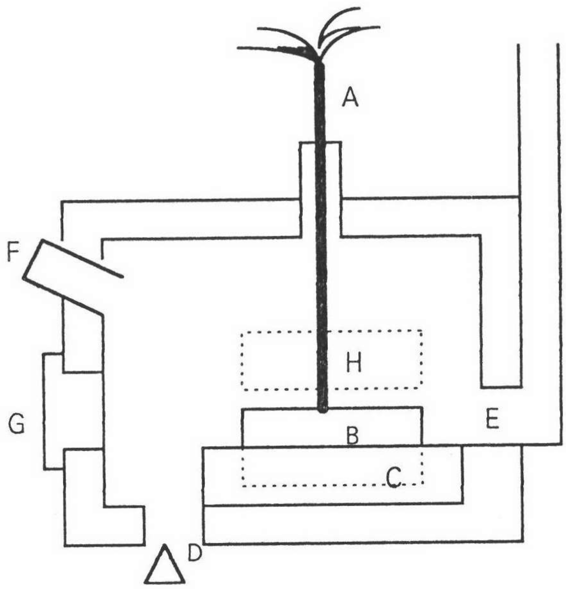

Figure 3.11 Sketch of the butane gas furnace

From figure 3.11, A = the sensors, B = the ceramic crucible, C = the electric furnace, D = burners, E = exhaust, F = observation shaft, G = plug door, H = loading part

C. Batch charging system

Fast and reproducible batch charging is mandatory for two reasons. Firstly, individual test can be compared only if they can be referred to an unambiguous zero time. Secondary, fast charging permits to investigate the very first minutes of batch melting. Faber et al. (1992) used a special metal container entered through the front door of the furnace and discharged above the melt. In previous tests in our lab, batch was charged through a metal tub, a procedure which required 4±1 min. Two metal containers (high temperature stainless steel) are used to load the batch by passing it through two side channels of the furnace. The metal container was basically a shovel with a pusher. The total time, of batch charging from opening the side channels to taking out the discharged containers was completed within about 30-45 seconds. This is a major improvement to previous work. The sketch of container is shown in figure 3.12.

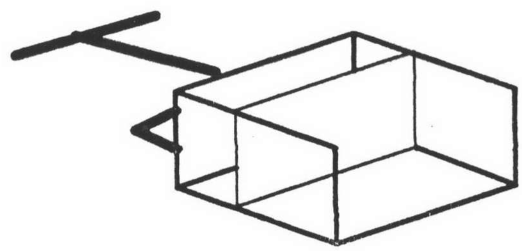

Figure 3.12 The special metal container

Test in large scale furnace

A commercial batch is selected in these experiments. It is loaded on the 7 kg melt in the ceramic pot when the atmosphere temperature has reached 1200 °C.

A. Thermocouple and resistivity probes

Thermocouple and resistivity probes are the same as used in the small scale experiments. But the large scale experiments used 4 individual sensors at 4 different levels, i.e., 0.5, 2, 3.5, and 5 cm in batch blankets over the melting surface. The arrangement of the probes in batch blankets are shown in figure 3.13.

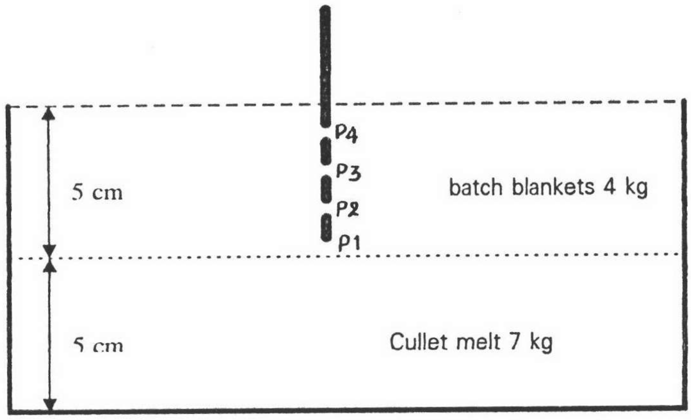

Figure 3.13 The arrangement of the probes in batch blankets

B. Heating-up characteristics

The furnace was heated up to 1,200 °C. A typical example of the heating-up characteristics is given in figure 3.14. Position 1 to 4 represent the temperatures at 0.5, 2, 3.5 and 5 cm respectively over the melt surface. Position 5 is the temperature of the butane gas atmosphere and position 6 is the temperature of the electric furnace beneath the ceramic pot.

After 800-900 °C, the cullet in the ceramic pot develop a melt with a smooth surface. When the temperature is heated up to over 1000-1100 °C, air bubbles eventually rise to the surface. The heating-up characteristics is shown in figure 3.14.

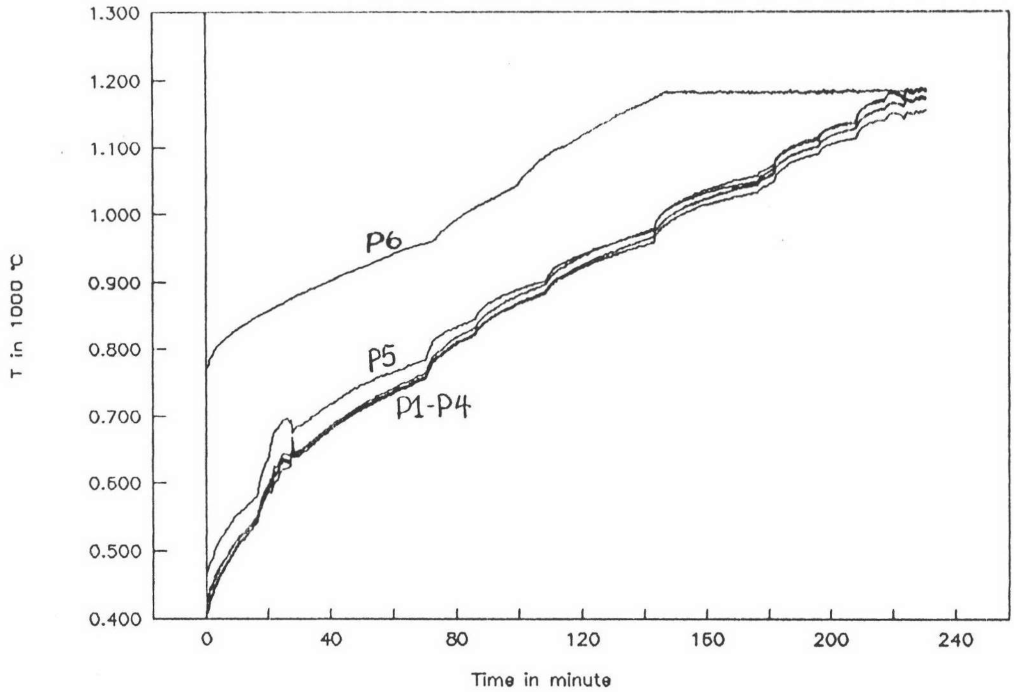

Figure 3.14 Heating-up characteristics of the research furnace

P1, P2, P3, P4 = position 0.5, 2, 3.5, 5 cm over the melt

P5 = position in the gas furnace atmosphere above the melt

P6 = position in the electric furnace beneath the ceramic pot

C. Batch calculation

A commercial glass batch is selected for these experiments; the amount of cullet in the batch is varied. For the batch calculation, the chemical composition of the raw materials has to be known. Table 3.1 shows the chemical composition of raw materials used in these experiments. Table 3.2 shows the amount of cullet in each batch.

Table 3.1 Chemical composition of raw material (wt. %)

	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O
sand	99.23	-	0.152	0.032	0.026	0.012	0.03	0.035
feldspar	69.0	-	18.20	0.24	0.40	0.14	10.50	0.11
dolomite	0.25	-	0.07	0.06	32.0	20.2	0.01	-
limestone	0.82	-	0.18	0.06	54.20	0.70	0.01	0.01
soda ash	-	-	-	-	-	-	58.20	-

Table 3.2 Amount of cullet in batch (by weight)

	B0%	B30%	B60%	B90%
% Batch	100	70	40	10
% Cullet	0	30	60	90

Table 3.3 shows the chemical composition of the commercial glass batch selected. The batch calculation is calculated by computer software UNIGLASS (1993). The calculation tells how much of each raw material is required in the batch. Table 3.4 shows the batch compositions.

Table 3.3 The chemical composition of the batch

SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	R
72.25	2.04	0.01	4.05	7.08	14.17	0.13	0.27	+10.05

R in table 3.3 is the redox number of the batch, adjusted by salt cake.

Table 3.4 Compositions of the batches

	B0%	B30%	B60%	B90%
Sand	554.46	388.12	221.78	55.45
Feldspar	83.51	58.46	33.40	8.35
Dolomite	164.28	115.00	65.71	16.43
Limestone	10.40	7.28	4.16	1.04
Soda ash	183.18	128.23	73.27	18.32
Salt cake	4.16	2.91	1.66	0.42
Cullet	0.00	300.00	600.00	900.00

D. Data recording

Automatic data management by a multi-channels analog to digital converter and computer were used. All thermocouples were connected to individual channels of the amplifier to amplify the signal, and all resistivity probes were connected to individual channels of the A/D card directly. Individual reading were taken every 1 second. The order of reading was: temperature of the atmosphere, temperature of the electric furnace, temperatures at position 1-4, Ux from resistivity probes at positions 1 - 4 respectively. The 3 V AC source applied the voltage to the electrodes simultaneously during the test.

E. First test with cullet free batch

Batch with 0% cullet was loaded on the melt. Both temperature of the atmosphere and electric furnace were 1,200 $^{\circ}$C. First, the 7 kg cullet was put to the ceramic pot when the furnace was still cold. When the cullet was molten and the temperature of the furnace was about 1,100 $^{\circ}$C, the probes were set to their right position. After waiting 30 minutes, until the temperature of atmosphere and the electric furnace started 1200 $^{\circ}$C, 4 kg batch were charged on top of the melt.

F. Solving the problem of electrodes interference

From the first test, the results was obtained that the data cannot be analyzed because the thermocouples had overflow signal, and the electrode had overvoltage signal. The problem source was readily identified as stray currents among the individual electrodes. So means were investigated to separate the individual levels of electrodes. Ways to electrically separate the electrodes were envisaged by:

1.) Increasing the distance between probes

This seemed to solve the problem, but it very difficult to operate.

2.) Changing the shape of the probes (ground coil shields inner coil)

The new probe shape is shown in figure 3.15. The new probes were tested.

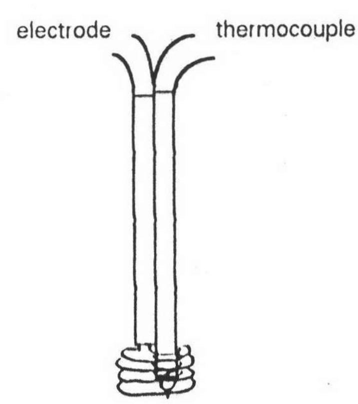

Figure 3.15 The individual new probe shape

3.) Making a new electrical circuit which provides a complete ("galvanic") separation among the probes.

In previous work, manual switches had to be used for lack of a better recording method. It turned out that this primitive approach had the great advantage to sequentially address the electrodes. Thus the electrodes had been separated all the time, and the problem of interference had not been encountered in these previous tests at all. So we had to imitate and automatic the sequential. Automatic switches were used which were controlled (triggered) by the A/D card from the computer. This means: the two channels from the A/D card would operate a mechanical relay. The controlling programs are shown in table 3.5. It show how the 2 channels digital to analog control 4 channels of probes.

Table 3.5 Binary code set by the 2 channels D/A used to address 4 individual channels of the probes

	D/A channel 1	D/A channel 2
Probe 1	L	L
Probe 2	L	O
Probe 3	O	L
Probe 4	O	O

After the auto switch system was installed, a test run was performed.

4.) High noise peak from the furnace

The test with the auto switches could separate the probes from each other, but the measure did not solved all problems. High noise peak in the furnace had to be dealt with. High noise level disturbed the A/D card and made not detect it anything. To provide a cut-off the high peak, a Zener diode was used. A Zener diode (also called breakdown diode) permits only the voltage lower than the breakdown voltage to pass. Any voltage higher than this is shortened to ground. A 5 V Zener diode was used in the experiments. The new circuit used is shown in figure 3.16.

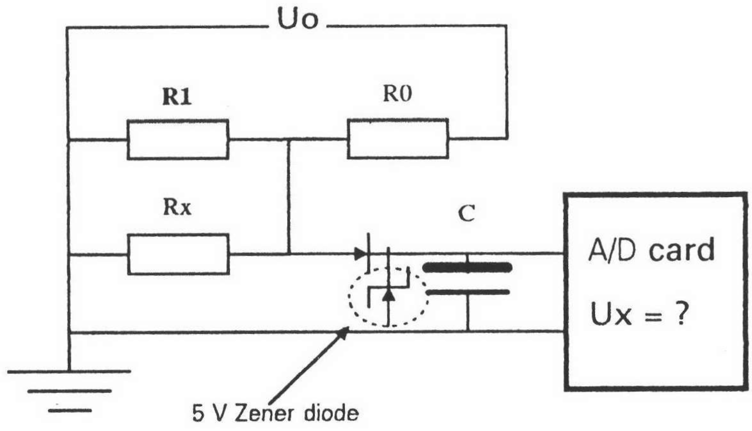

Figure 3.16 show the new circuit that use the Zener diode

The first test with the new circuit was operated in a different furnace. This furnace was an electrical type furnace using high current (about 40 A) This resulted in high noise peaks. So the furnace was the environment to test current-noise measures.

G. Solving the problem of the thermocouple

The problem of the thermocouple in the experiments was the high fluctuation of the signal.

1.) Testing commercial glass batch in a small scale test

This small scale test was performed to know whether or not the glass batch was the source of fluctuation. An amount of 120 g of cullet free batch was poured into the small fire clay crucible equipped with the probe already and heated up from room temperature to 1200 °C. The batch was not a cause of the thermocouple problem.

2.) Use of a capacitor to solve the problem

Since, the high value capacitors could integrate the fluctuation signal, capacitors with 2200 μF were tried to use in the high current furnace. The data were smooth. The high value capacitors seemed to solve this problem.

Additional test

A. Test the probes perpendicular and parallel to the gradient atmosphere

The ceramic crucible and probes were set like figure 3.17.

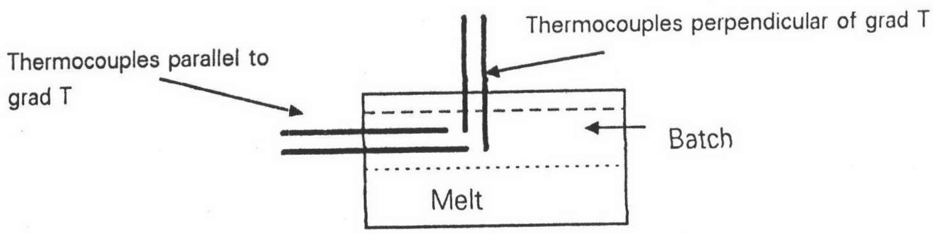

Figure 3.17 Sketch of thermocouple test in large scale experiment; the thermocouples were positioned in the direction or perpendicular to the temperature gradient in the batch

B. Test of melting temperature

The melting temperature when the batch was loading was measured directly in this experiment by the thermocouples.

Test of systems with varied amount of the cullet in the glass batch

The batch used in these experiments is shown in table 3.4. The cullet used in the experiments had 1-2 mm diameter. The cullet had the same target composition as the batch. The amount of cullet to the batch 0%, 30%, 60%, and 90%, respectively.

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