Chapter III

Experimental Part

3.1 Pre-tests

3.1.1 Research furnace

The furnace used in the pretest is an electrical furnace which was constructed using light alumina brick (C-2 type with 64 mm thickness) and Khanthal wire (Ni-Cr, 1.2 mm in diameter) as a heating coil. Total resistance for the heating coil is about 7 to 7.5 ohm and consumes 2.8 to 3.0 kW input power when supplied with 20 A AC. The time demand to yield 900 ^{o}C is approximately 1 Hrs.

3.1.2 Thermocouples and resistivity probes

Type K thermocouples (NiCr-Ni) with 0.6 mm wire diameter are used in this experiments. The temperature probe can be easily made by inserting thermocouple wires into sintered alumina tubes with 4 mm outer diameter and two 1 mm inner diameter bores, and twisting them together to make a junction. Calibration was performed by measuring the ice point at room temperature (-1.248 mV in our experiments). The details of calculation and the formulae used to convert voltage to temperature in ^{o}C are given in the appendix A. The resistivity probes are made of Khanthal wire with 1.2 mm in diameter, wrapped on a cylindrical rod of 5 mm diameter to make solinoid (to increase contacted surface) at the end of electrodes. 10 mm distance was left between the

two electrodes.

3.1.3 Materials and procedures

Raw materials and the ratio used in a combination of selected raw materials are listed in the table 3.1. Total amount of the mixtures is 100 g.

Tab. 3.1 The ratios and raw materials used in a pre-test; total amount is 100 g.

raw materials	ratios
soda ash + limestone	3:1 2:1 1:1 1:2 1:3
soda ash + dolomite	2:1 1:1 1:2
soda ash + sand	1:1 1:2 1:3
soda ash + feldspar	3:1 2:1 1:1 1:2 1:3
soda ash + limestone + sand	23:15:52*

* the unit is given in g (this results in a 74-10-16 wt. % oxide glass).

When preparing the mixture as listed in the table 3.1 already, a small amount of distilled water is added into the mixture. The electrodes are connected to a simple circuit (see figure 3.1 a-b) to convert AC to DC voltage. This is very important, because, if a DC voltage source were applied, polarization phenomena would occur. So, an output voltage value measured may be higher than the input. The lefts proceed as follows: low AC voltage source (approx. 11 V when no load connected)

of 50 Hz frequency was supplied to the electrodes, connect the power supply to a furnace coil and turn on the pen recorder to record both temperature and resistivity (in terms of voltage) simultaneously. Wait until the temperature reached to 900 ^{o}C then stop all procedures.

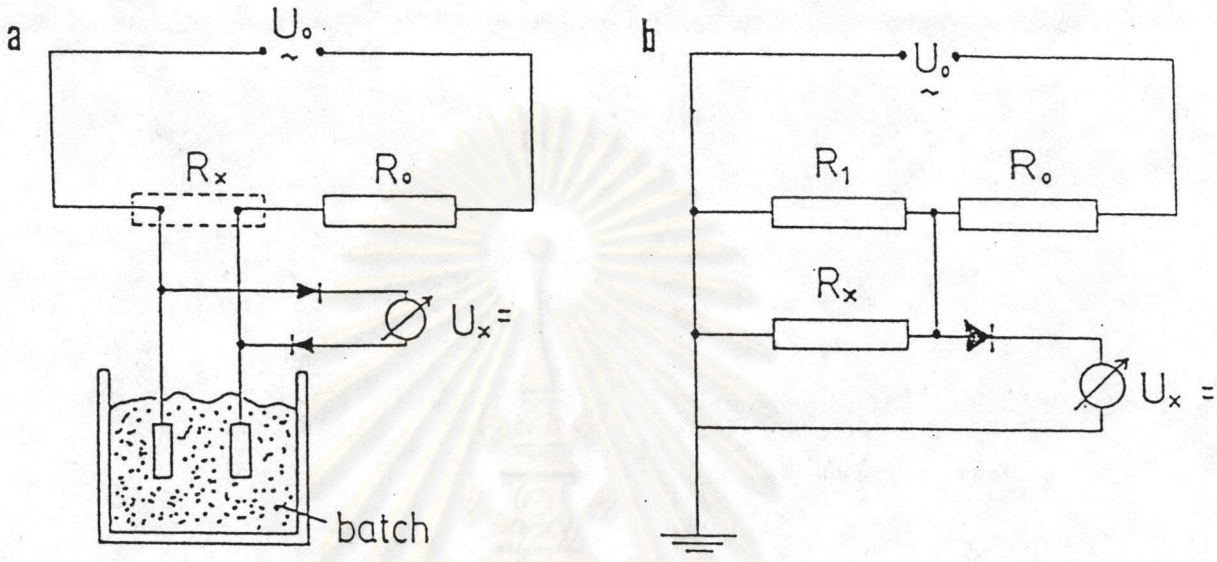

Fig. 3.1 a-b Circuit diagrams used for measuring the resistivity in terms of voltage, U_x; (a) initial concept (b) final realization with R_o = 10 K. and R_x = 1 M.

From the circuit, the current passing through the resistors R_o and R_y must be equal (R_y is the total resistance of R_1 and R_x) then,

$$U_x/R_y = U_o/(R_o+R_y)$$

when

$$R_y = R_1.R_x/(R_1+R_x)$$

then the U_x read from the volt meter can be converted into R_x,

$$R_x = R_1.R_o.U_x/[R_1.(U_o-U_x)-R_o.R_x]$$

and into the electrical conductivity X of the batch blanket,

$$X = d/(A.R_x)$$

where d = distance between the electrodes and A = effective area of the electrodes.

3.2 Main-tests

3.1.1 Research furnace

figure 3.2 shows a versatile research furnace designed in the Dept. Mat. Sci. in the scope of a research project inspired by the furnace used by Bieler (1985) and Kröger (1960). The furnace is mainly constructed by a light alumina brick (C-2, 76 mm in thickness), also equipped with three burners with 3 cm inner diameter using butane gas as a fuel source. The melting chamber is large enough to melt 10 to 15 kg glass and ro receive rectangular crucibles of 22.5 cm x 26.5 cm x 13.5 cm (W x L x H) in dimension. The wall thickness of crucibles is approximate 1.5 cm.

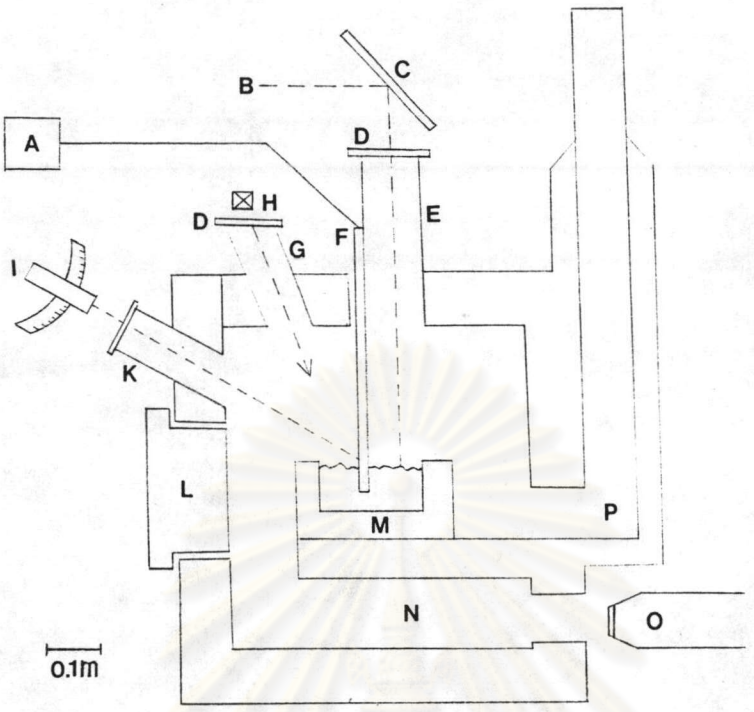

Fig. 3.2 The versatile melting furnace used in the main-tests.

A: recorder	B: observer; camera	C: mirror, fan-cooled
D: silica window	E: observation shaft	F: the sensors
G: illumination shaft	H: light source	I: goniometer optics
K: observation shaft	L: plug door	M: a crucible
N: burner channel	O: burners	P: exhaust

The heating characteristic of gas melting furnace before charging the batches is shown in a figure 3.3.

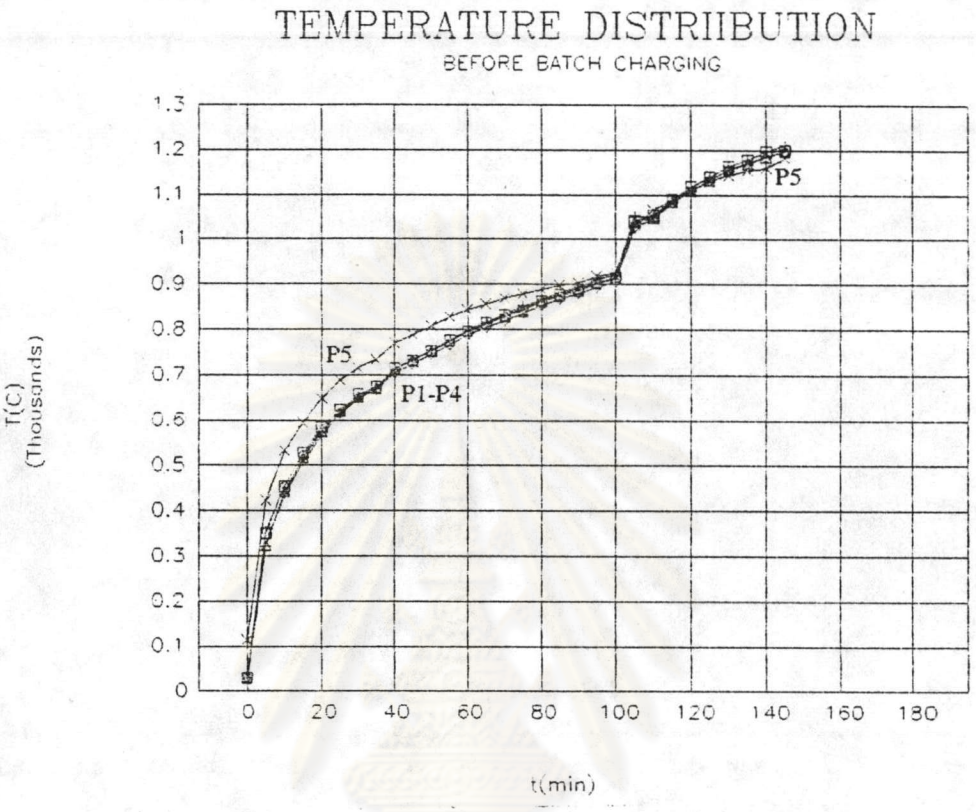

Fig. 3.3 The heating characteristic of the gas melting furnace used in the main-tests before charging the batches; P_1, P_2, P_3, and P_4 are the position at 0.0, 1.5, 3.0, 4.5 cm distance from the melt surface; P_5 is placed above the batch blanket.

3.1.2 Thermocouples and resistivity probes

Thermocouples type and wire size are the same as used in the pre-tests. The figure 3.4 shows an individual unit of thermocouple and resistivity probes arrangement (completed sensor is composed of 4 individual units). The insulators used for both thermocouples and resistivity probes are low temperature alumina and fused alumina tube (stand up to 1300 ^{o}C max. for low temperature alumina and 1900 ^{o}C for fused alumina tube). Lower sintering temperature alumina tubes are

used at the tip of the sensors in order to easily replace them when the batch composition is changed. It has 10 cm long, 3 mm outer diameter and two 0.75 mm inner diameter holes. The upper part of the sensors are made from fused alumina, 100 cm long, 4 mm outer diameter, and with two 1 mm inner diameter holes. The electrodes are made from NiCr wire with 0.6 mm diameter wrapped on a cylindrical rod with 5 mm diameter to make solinoid electrodes, the 5 mm long with approx. 1 cm free space between the two electrodes.

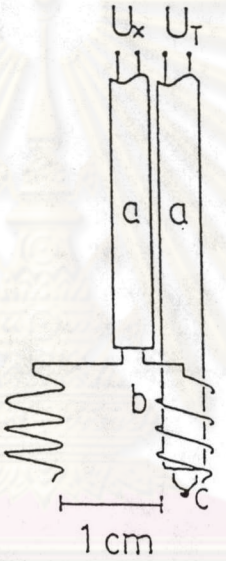

Fig. 3.4 A Sketch of an individual unit of temperature and resistivity probes.

3.1.3 Materials and procedures

The compositions of 12 individual batches are listed in table 3.2 and further batch properties are given in appendix C. All materials are used as received.

Tab. 3.2 Compositions of individual batches; S=sand, F=feldspar, D=dolomite, L=limestone, Sa=soda ash, Sc=Salt cake, Sp=salt peter, C=coal, and R=redox number. Amounts of raw materials are given in kg per 1 ton of batch.

Batch	S	F	D	L	Sa	Sc	Sp	C	R
01	621.95	-	-	150.03	228.02	-	-	-	0
02	573.89	54.52	150.65	34.65	186.51	-	-	-	0
03	573.71	54.72	150.21	34.90	173.55	8.61	-	-	24.91
04	571.58	54.52	149.66	34.77	180.05	8.57	-	0.86	-0.07
05	571.61	54.52	149.66	34.77	180.06	8.72	-	0.66	4.95
06	572.64	54.62	149.93	34.83	178.95	8.59	-	0.43	10.04
07	573.51	54.70	150.16	34.89	177.79	8.72	-	0.23	15.00
08	574.54	54.80	150.43	34.95	176.67	8.62	-	-	20.10
09	571.37	54.50	149.60	34.76	179.98	8.71	-	1.09	-5.14
10	572.15	54.57	149.81	34.80	178.80	8.58	-	1.29	-10.12
11	571.11	54.47	149.53	34.74	179.90	8.74	-	1.51	-14.92
12	571.91	54.47	149.74	34.79	178.82	8.58	-	1.72	-20.20

For each investigation, 7 kg of cullet was charged into a crucibles and placed into the melting chamber of the cold melting furnace. Place The sensors was placed at the position as shown in the figure 3.5. The furnace was heated up until the temperature approached 1200 ^{o}C for all positions. The silica window from the observation shaft K was opened; 4 kg of dry batch pile (already pre-mixed, 1.4 - 1.5 g/cm^3 batch density) were charged through this shaft as quickly as posible (takes approx. 5 min. in our experiments), the silica window was

firmly closed and the temperatures and resistivities of all positions were recorded every 1 min. (using the same circuit as used before in the pre-tests for resistivity measurement). Recording was stopped when the temperature of P_5 had risen up to 1200 ^{o}C again and resistivities had reached the low values typical of primary liquid phase formation (eg., lower than 1 V). All sensors were immediately removed away from the blanket.

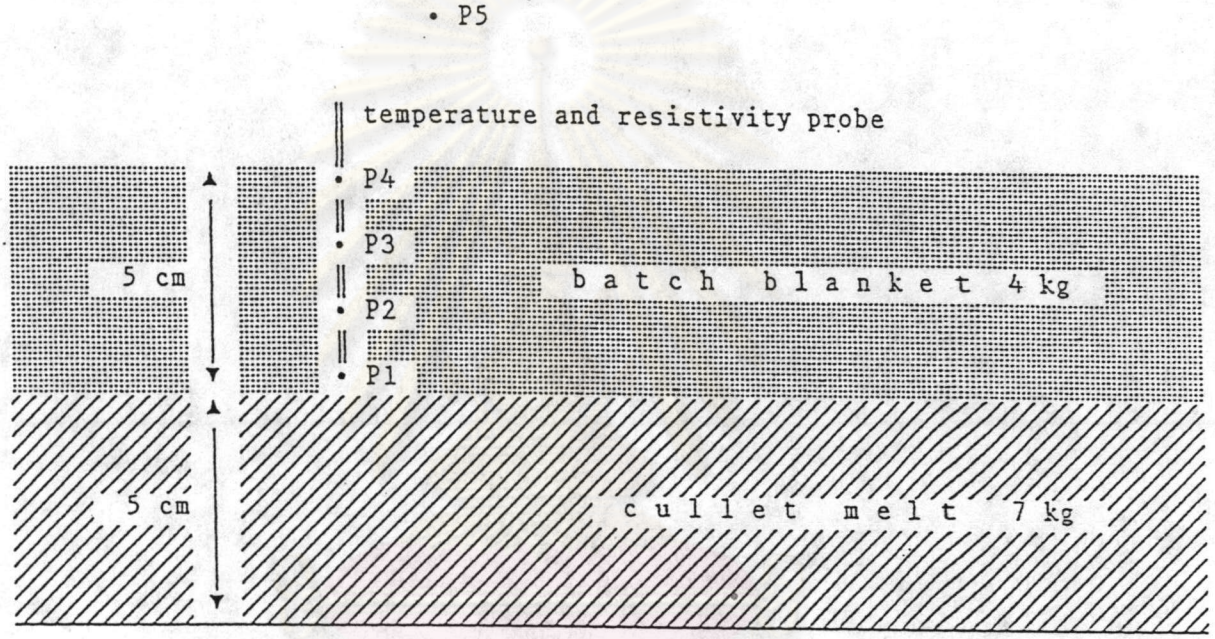

Fig. 3.5 Arrangement of the sensors in the melting furnace (the positions of P_1 to P_4 are the same as mentioned in figure 3.3). Note: P_1 does not touch the cullet melt.