



Chapter III

Experiment

3.1 Description and scope

3.1.1 Description of experiment

An experiment was designed using a simple sample geometry instead of fibres. The experiment can be classified into 5 steps: materials preparation, sample preparation, sample characterization corrosion tests, and corrosion characterization. The following flow chart shows the details.

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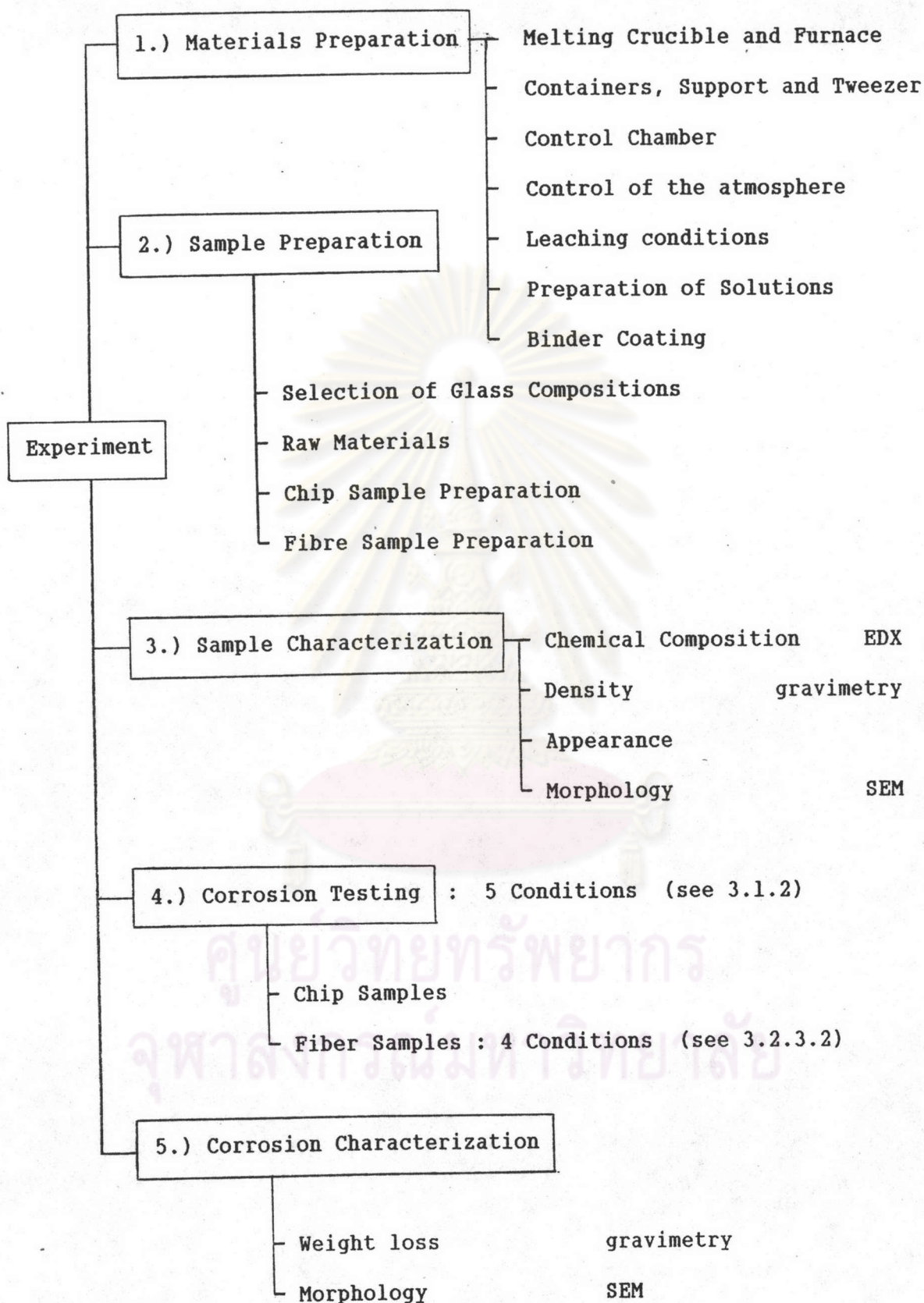
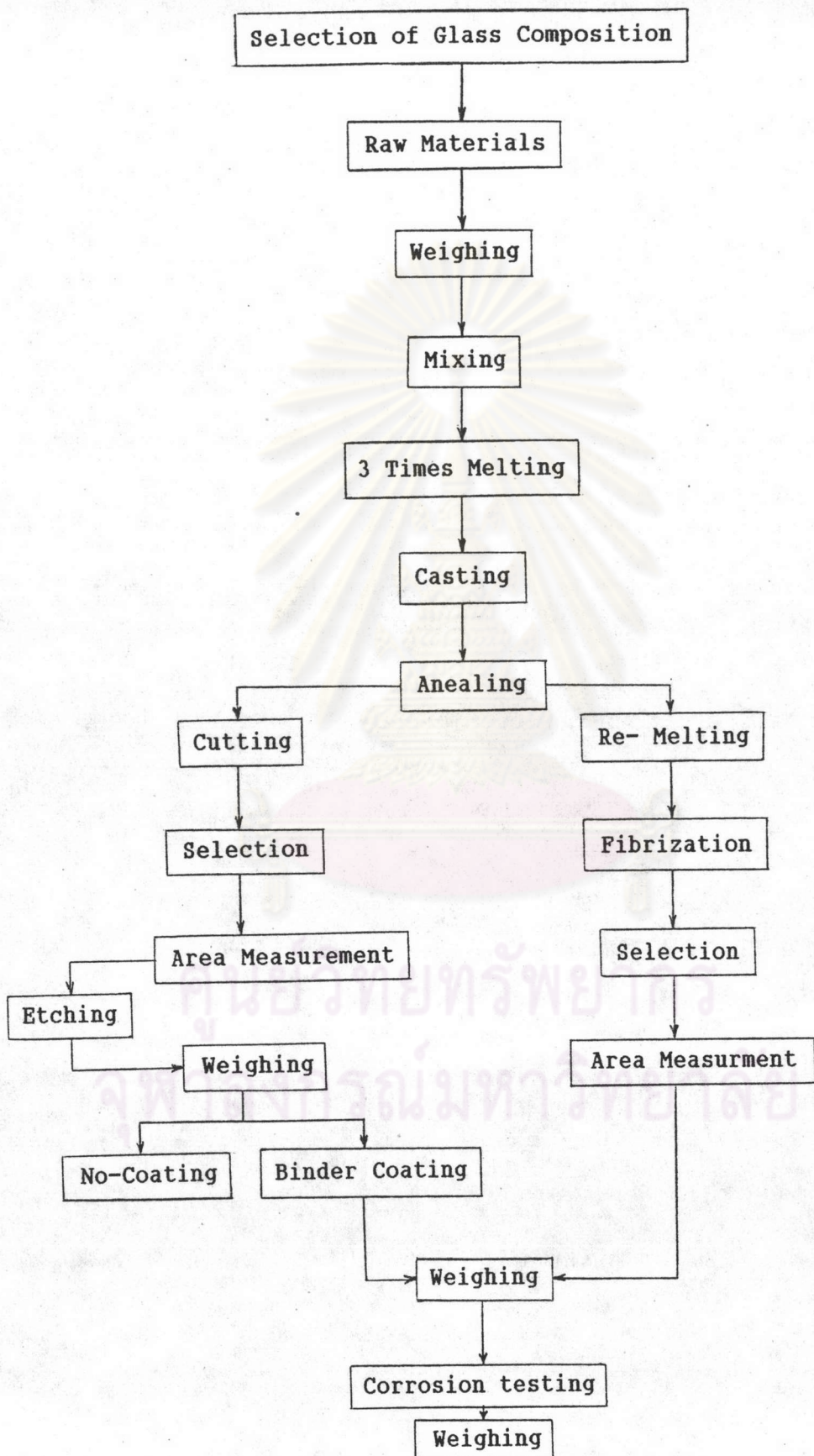


Fig 3.1 Experimental description flow chart

Fig 3.2 Flow chart of sample preparation and corrosion tests



3.1.2 Scope of Experimental Work

3.1.2.1 Chip Sample Tests

The number of individual tests was determined as follows:

- 8 glass compositions,
- 2 tests for each glass composition (double test)
- 4 time intervals,
- 5 corrosion conditions, i.e.,
 - original solution, sample without binder;
 - original solution, sample with binder;
 - solution bubbled with N_2 , sample without binder;
 - solution buffered at $pH \sim 5$, sample without binder;
 - phosphate-free solution, sample without binder;

This requires a total of 320 individual tests. Assuming a failure rate of 20 % a respective number of samples has to be prepared. Eight different glass batches were molten. The chemical composition was determined by XFA, and the density by gravimetry.

The required number of samples was cut by a low-speed saw ($2 \times 2.5 \times 0.2 \text{ cm}^3$) and pre-treated. With a sample approximate surface area of $10\text{--}12 \text{ cm}^2$ and a ratio of sample surface to leachant volume $s = 0.1 \text{ cm}^{-1}$, 100–120 ml of solution were required for each individual test. The leachate was renewed every 7 days.

In previous tests, dissolution velocities at 37°C ranged from 0.2 to 220 nm/d. With a density of $\rho \sim 2.5 \text{ g/cm}^3$ and a sample surface of $10\text{--}12 \text{ cm}^2$, mass losses ranging from 0.6 to 60 $\mu\text{g/d}$ were expected. In order to resolve the extremely low mass losses, more than two parallel tests were required for shorter time intervals.

3.1.2.2 Fibre Sample Tests.

The number of individual test was determined as follows : 4 glass compositions, one time interval, 4 corrosion conditions; original solution, N₂-bubble, pH 5 and phosphate-free solution.

Monosize fibres were prepared by remelting bulk glass in an alumina crucible which had a small hole $\phi = 0.5$ cm. The fibres were drawn with a rubber roller device.

With the same ratio of $s = 0.1 \text{ cm}^{-1}$, two types of experimental were designed (see 3.2.3.2), The corrosion time was only 7 days for studying the geometric effect.

3.2 Procedure and Equipment

3.2.1 Materials Preparation

3.2.1.1 Melting Crucible and Furnace

Before 8 glass compositions were molten, melting crucibles were prepared and a melting furnace was modified. Several of the glasses, such as borosilicate glass, basalt and slag, required melting temperatures of more than 1400°C. It was very difficult to find a big enough crucible to melt 1 kg batch at 1400°C. High alumina crucibles could not be used because of their poor thermoshock resistance. Graphite crucibles could not be used because of their influence on the redox state of the glass. So, 4 sizes of clay crucible were designed as shown in the following figure.



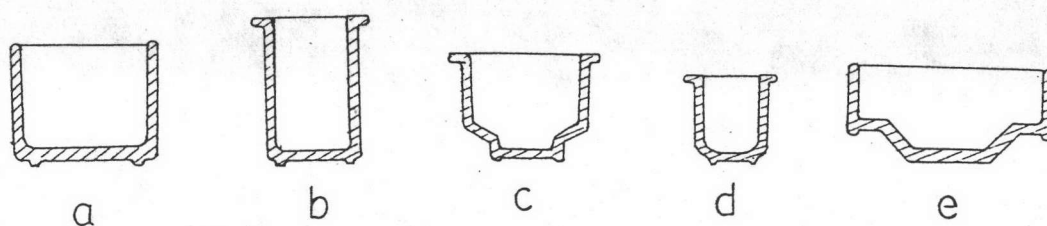


Fig 3.3 4 sizes of clay crucible (a, b, c and d) compared with commercial crucible (e).

Firstly, 2 compositions were selected as a trial and error. (see table 3.1). They were ground in porcelain dish and passed through sieve no. 120 mesh (125 μ m). After that slip body was prepared and cast into plaster mold which was prepared by using a plaster of paris at 75 consistency. The clay crucibles were dried in air for 2 days, then in the oven at 120°C. Bisquit fired at 850°C.

Table 3.1 Batch composition of clay crucible.

batch composition	I(%)	II(%)	Oxides	I(wt%)	II(wt%)
Kaolin (Ranong)	30	30	SiO ₂	31.8	37.7
Ball clay	15	20	Al ₂ O ₃	59.0	51.6
Feldspar	15	20	CaO	0.3	0.4
Al ₂ O ₃	40	30	Na ₂ O	1.1	1.5
			K ₂ O	1.7	2.0
			Fe ₂ O ₃	0.5	0.5
			MgO	0.1	0.1
			LOI	7.3	8.0

For checking a reasonable use, both these bisquit crucibles and commercial crucibles were used to melt glass batch at 1400°C for 5 h. The result show that crucible type II was easy to form and stronger than type I crucible. However, some crucibles broke and glass melt strongly damaged the furnace. Commercial crucibles always broke. So, type II crucibles were used; In order to avoid damage of the furnace, they were placed into a commercial crucible which served as emergency reservoir. However, slow heating rate between $500\text{--}800^{\circ}\text{C}$ was required to decrease the effect from quatz inversion at 573°C .

Fig. 3.4 Clay crucible put on the top of commercial crucible.

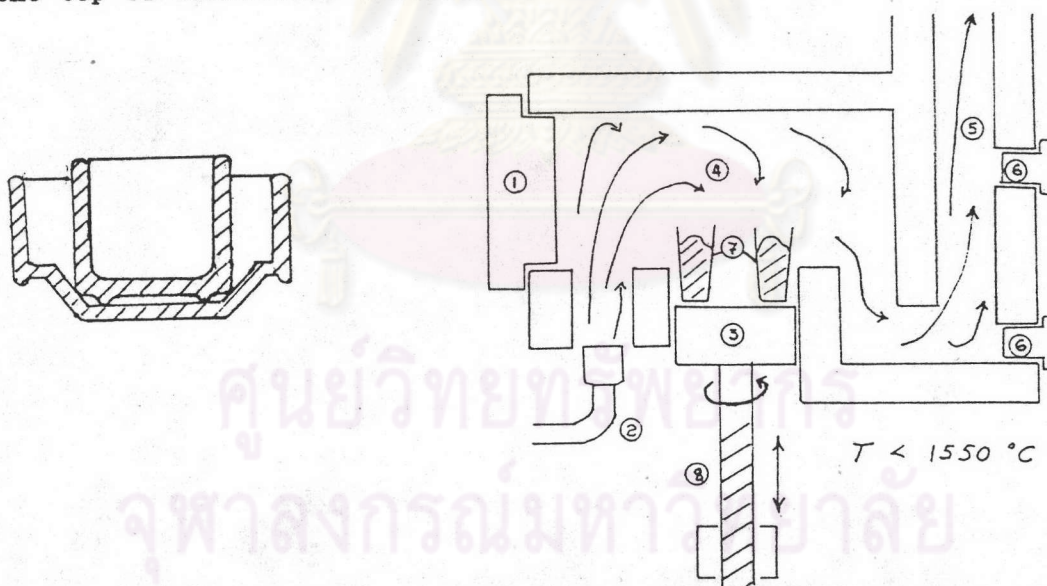


Fig. 3.5 High temperature gas furnace;

- | | |
|---------------------|----------------------------------|
| 1: front door | 2: venturi burner |
| 3: crucible support | 4: fire chamber |
| 5: flue gas chanel | 6: shutter |
| 7: crucibles | 8: turning and lifting mechanism |

After the pre-tests, a sufficient number of clay crucibles type II were fabricated by weighing, mixing, grinding in ball mill for 10 h, slip making, casting, drying and bisquit firing at 850°C.

A gas furnace suitable for 1400°C with big chamber and automatic up-down batch charging was selected as shown in Fig 3.5. In order to increase the heating rate two new burners (bigger than the old ones) were installed, and a new higher chimney was designed to increase the draft. This increased the heating rate; heating from room temperature to 1400°C required 3 to 4 h.

3.2.1.2 Container, Support and Tweezer

According to Scholze and Conrardt, 1987, the experiment system was designed by using polycarbonate materials and the fluid was never in contact with any metals which might have adversely influenced the results. In this experiment, all container, support and tweezers used high grade plastic materials. These materials also needed to be checked in pre-test. Three types of container; i.e, brown HDPE bottles, white HDPE bottles and white PP bottles were tested by measuring the weight loss after exposure with 1 molar NaCl at 90°C and 37°C for 7 days. The results showed that all types had very low loss (~ 0.05-0.1%). As the daylight had to be excluded in order to prevent the growth of algae, brown HDPE bottles were selected for this experiment.

Table 3.2 Pre-test of container

Types	loss at 37°C		loss at 90°C	
	Loss water%	Loss bottle%	Loss water%	Loss bottle%
1.) HDPE - Brown bottle	0.02	0.01	0.92	0.13
2.) HDPE - White bottle	0.02	0.09	-	-
3.) PP - White bottle	0.02	0.04	3.70	0.05

The support material for the sample was cut from medicine bottles from a dental clinic. This material was stable against solutions. A shape of support was designed which allowed good access of the solution from all sides of the sample. The support was put in a bottle as shown in the following figure.

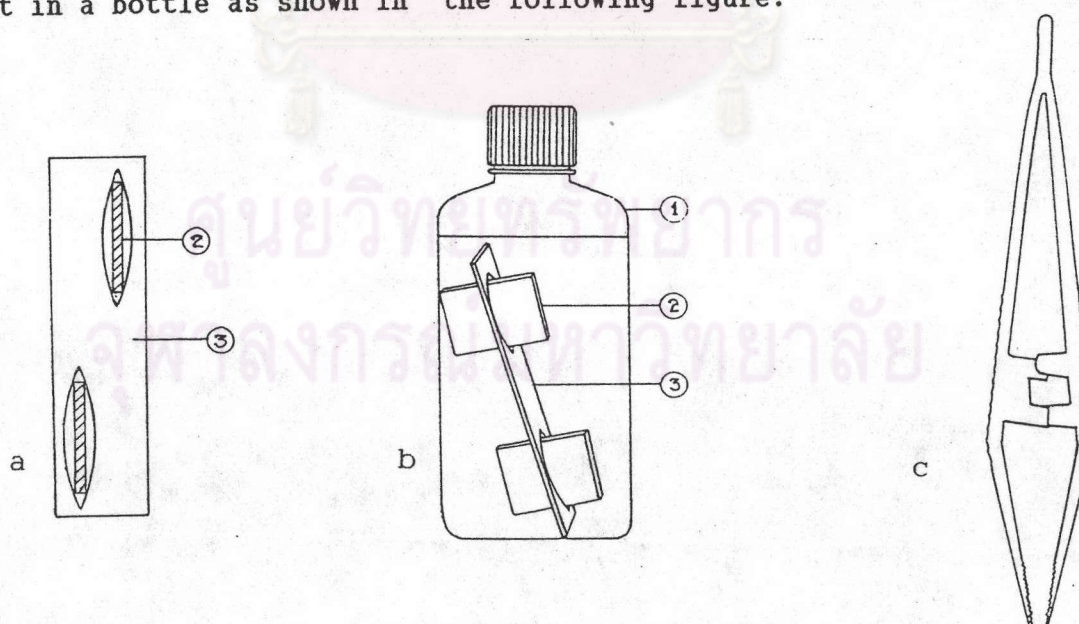


Fig. 3.6 Support(a), container(b) and tweezers(c)

1 = brown HDPE bottle

2 = glass sample

3 = support

3.2.1.3 Heating chamber

A big enough heating chamber was required to keep ~ 40 bottles at the same time at $37 \pm 1^\circ\text{C}$. So, a $1 \times 1.5 \times 0.7 \text{ m}^3$ chamber with blower was purchased. A cross section of this chamber is shown in the following figure.

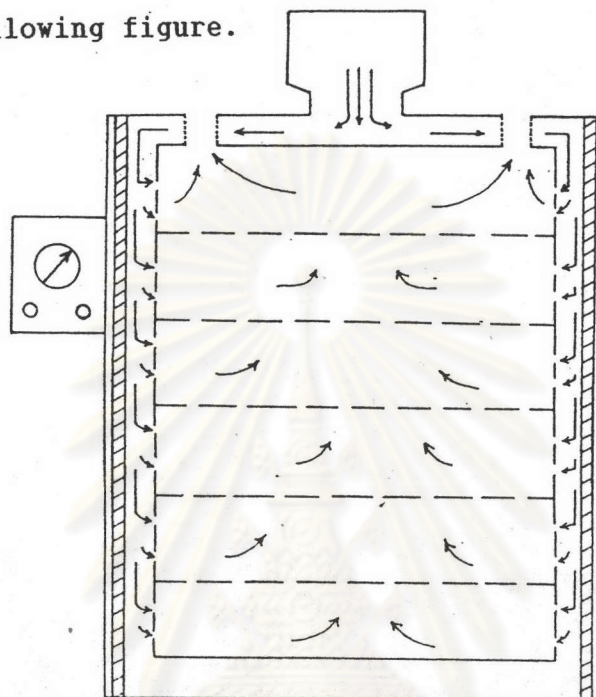




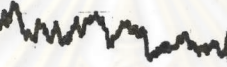

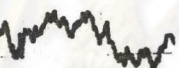

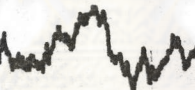
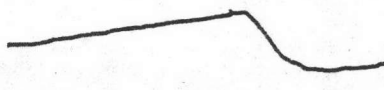




Fig. 3.7 Heating chamber.

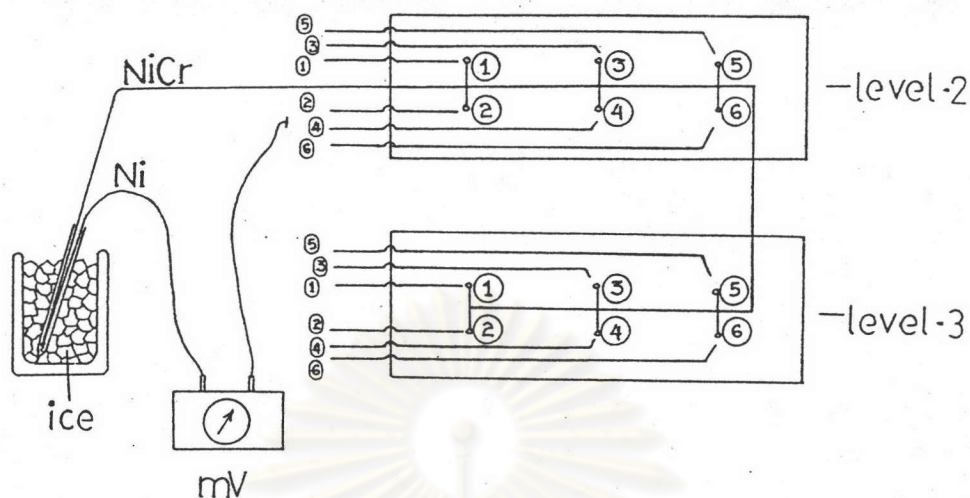
This chamber is equipped with 6 levels, a 3.5 blower, and maximum 9 kW power. For the experiments, a highly accurate long-term temperature control was required. Therefore, the commercial temperature controller was calibrated by using two-junctions of a NiCr-Ni type K thermocouple and corrected for systematic errors. Firstly, low temperature gradient was designed. The temperature at 6 levels were measured with the controller scale set to 100°C . The real temperatures were recorded by a pin recorder as a function of time for each level. The results showed that a high temperature gradient occurred. The situation was significantly improved after holes were cut into the support metal sheets of each level. The results are shown in table below.

Table 3.3 Optimizing the local and temporal temperature homogeneity of the heating chamber

Level from the top	Before cutting holes			After cutting holes		
	mV	°C	T signal	mV	°C	T signal
1	4.345	106		3.754	92.0	
2	5.687	139		3.974	97.0	
3	6.010	147		4.012	98.0	
4	6.281	154		4.020	98.5	
5	6.657	163		4.077	99.5	
6	6.579	161		3.911	95.5	
S.D. (N-1)	0.85	21.1		0.11	2.7	

After the holes were made, the temperature gradient was reduced by 8 times. According to the above data, levels 2 and 3 had the smallest effect. So, these two levels were selected. Secondly, the absolute calibration to a temperature of 37 ± 1 °C was performed by installing two junction thermocouples in the two selected levels as shown in the following figure.

Fig. 3.8 Temperature calibration system.



level 2		level 3	
Point °C	Point °C	Point °C	Point °C
1 37.1-37.8	4 37.1-37.6	1 36.5-37.5	4 36.8-37.4
2 37.7-37.6	5 37.3-38.1	2 36.6-37.1	5 37.3-37.8
3 37.2-37.7	6 37.1-37.6	3 36.8-37.5	6 37.8-37.5

The results show that each point at each level had a variation range of better than 37 ± 1 °C. To make double sure that this was the correct temperature, a medical thermometer (standard accuracy 0.1 K) was used.

3.2.1.4 N₂-CO₂ Gas Saturation

A N₂-CO₂ gas mixture in a ratio 95:5 by volume was prepared. The gas flow was bubbled into the containers, thus providing CO₂ saturation at an activity equivalent to 0.05 bar

CO₂ and a constant pH value of 7.6 ± 0.2 . In other experiments, only N₂ gas was bubbled into the containers, and the pH value assumed 8.5 ± 0.2 . The following figure shows the N₂-CO₂ flow system.

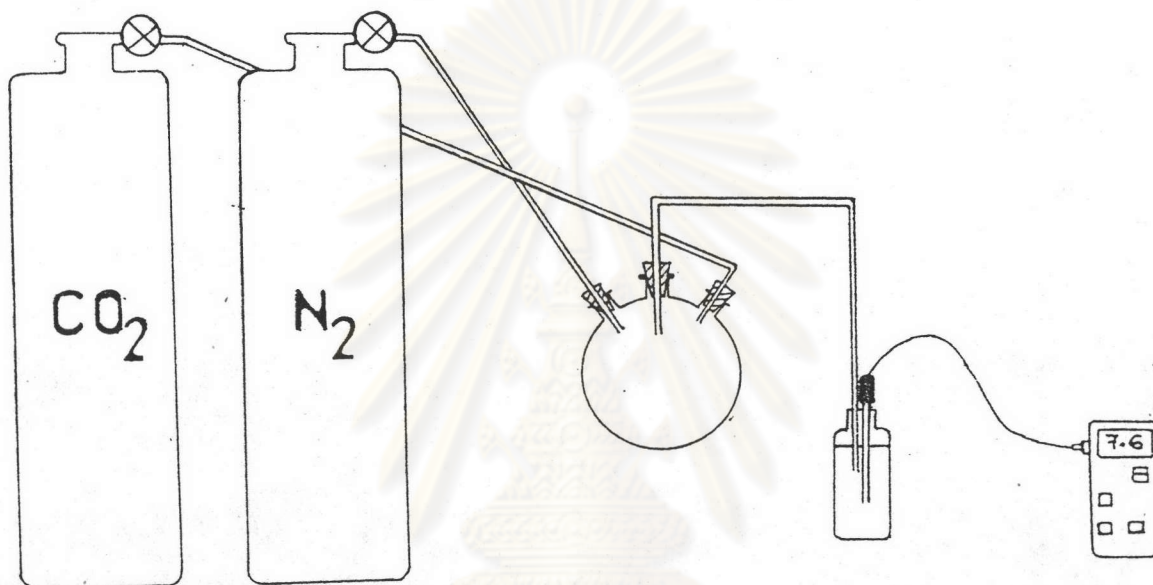


Fig. 3.9 N₂-CO₂ flow system.

In order to make the conditions comparable to former studies the time required for both CO₂ saturation and release had to be determined. The amount of gas had to be controlled carefully. Therefore a regulator valve with flow meter which can control small amounts of gas was selected. A N₂ : CO₂ ratio of 95:5 was set up. The following table shows the calibration data for the flow meters.

Table 3.4 Regulator calibration data.

metal ball on a middle of scale no.	volume (cm ³ /s)	
	CO ₂ regurator	N ₂ regurator
0.5	15	-
1.0	22	25
2.0	40	38
2.5	48	50
3.0	58	58

From the above table, when N₂ regulator opened at scale no.1, a 25 cm³/s of CO₂ gas was required. So it was very difficult to adjust the CO₂ regulator scale. So, N₂ regulator was set at scale no. 2 and the CO₂ regulator was opened until 20 cm³ per 10 seconds was received. The setting was re-checked every month. To find a time requirement for gas saturation, the pH decreasing with time was recorded as shown in the following figure.

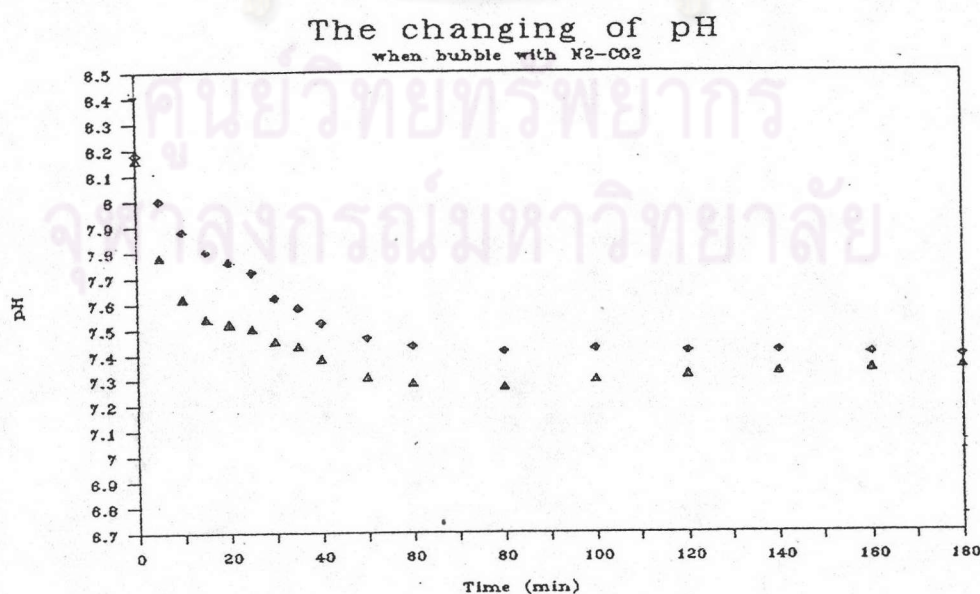


Fig. 3.10 Decreasing of pH with gas saturation time.

Conclusively, time requirement for gas bubbling was ~ 30 -40 seconds. However, each bottle had not the same amount of solution. So, N_2 - CO_2 gas saturation was continued until the selected pH was reached. The other important thing was the change of pH with time due to the CO_2 release in the heating chamber at $37^\circ C$. This shown in the following figure.

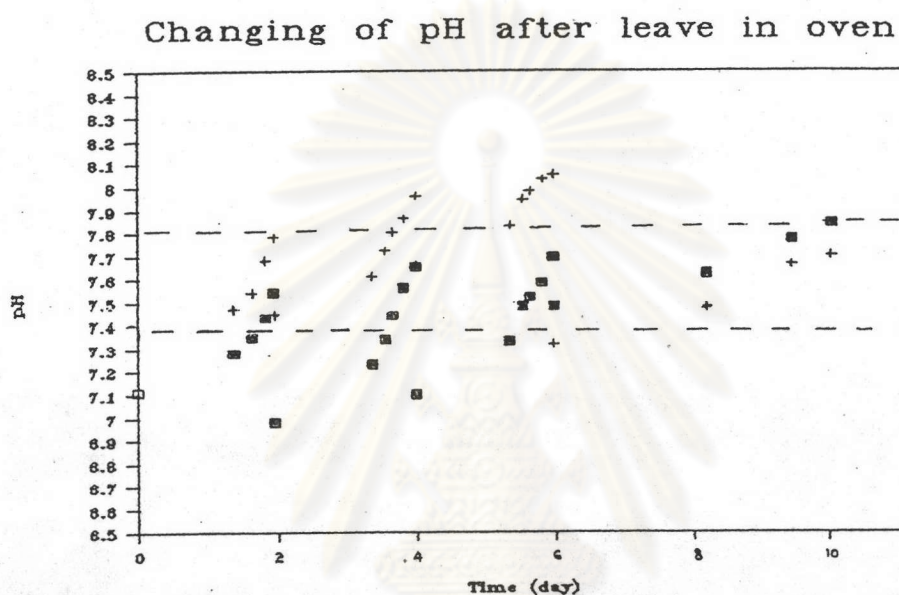


Fig. 3.11 Increase of pH after exposure in the heating chamber, re-bubble every 2 days.

From the above figure, the pH could be kept within a range of only 7.6 ± 0.2 for the original solution, and 8.5 ± 0.2 for N_2 saturation solution when gas bubbling was repeated every 2 days.

3.2.1.5 Etching Condition

Gunarathna and Robert, 1991, etched glass samples by using 2% HF plus 2% H_2SO_4 for 30s to remove the hydrated surface layer. For this experiment, HF and HNO_3 were used instead in order to avoid calcium sulphate precipitation on the surface of the Ca rich glasses. A new etching procedure was designed by etching in 3 different conditions with varied times. Each condition was

characterized by weigh loss, and morphology (by using a balance, optical microscope and SEM respectively). After that a suitable condition were selected.

The 3 different condition were :

1. 5% HF + 5% HNO₃ with 3 times interval 30s, 60s, 90s
2. 2% HF + 5% HNO₃ with 3 times interval 30s, 60s, 120s
3. 2% HF + 2% HNO₃ with 3 times interval 30s, 60s, 120s.

The weight loss upon dissolution and the morphology before and after etching was observed as shown in the table below. (JM glass)

Table 3.5 Etching condition data

condition	time (s)	%wt.loss (g/cm ²)x 10 ⁻⁵	optical morphology*
5% HF + 5% HNO ₃	30	44	x
5% HF + 5% HNO ₃	60	79	✓
5% HF + 5% HNO ₃	90	105	✓
2% HF + 5% HNO ₃	30	25	x
2% HF + 5% HNO ₃	60	39	✓
2% HF + 5% HNO ₃	120	72	✓
2% HF + 2% HNO ₃	30	21	x
2% HF + 2% HNO ₃	60	37	✓
2% HF + 2% HNO ₃	120	56	✓

*x = spot of light scattering can not be accepted when compared with untreated samples

✓ = acceptable

From the above table, some samples were selected for inspection by SEM. The results are shown in the figure below.

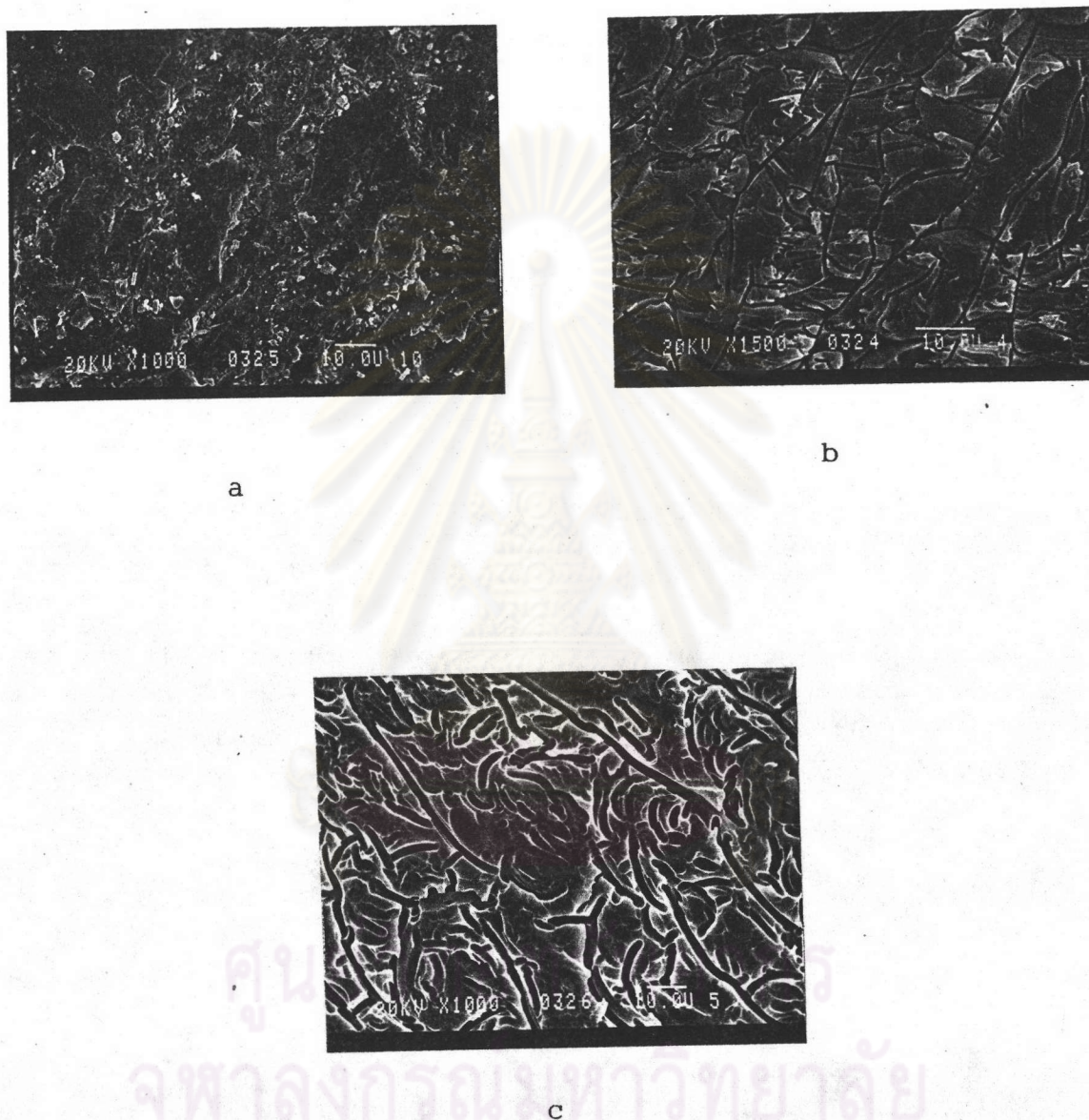
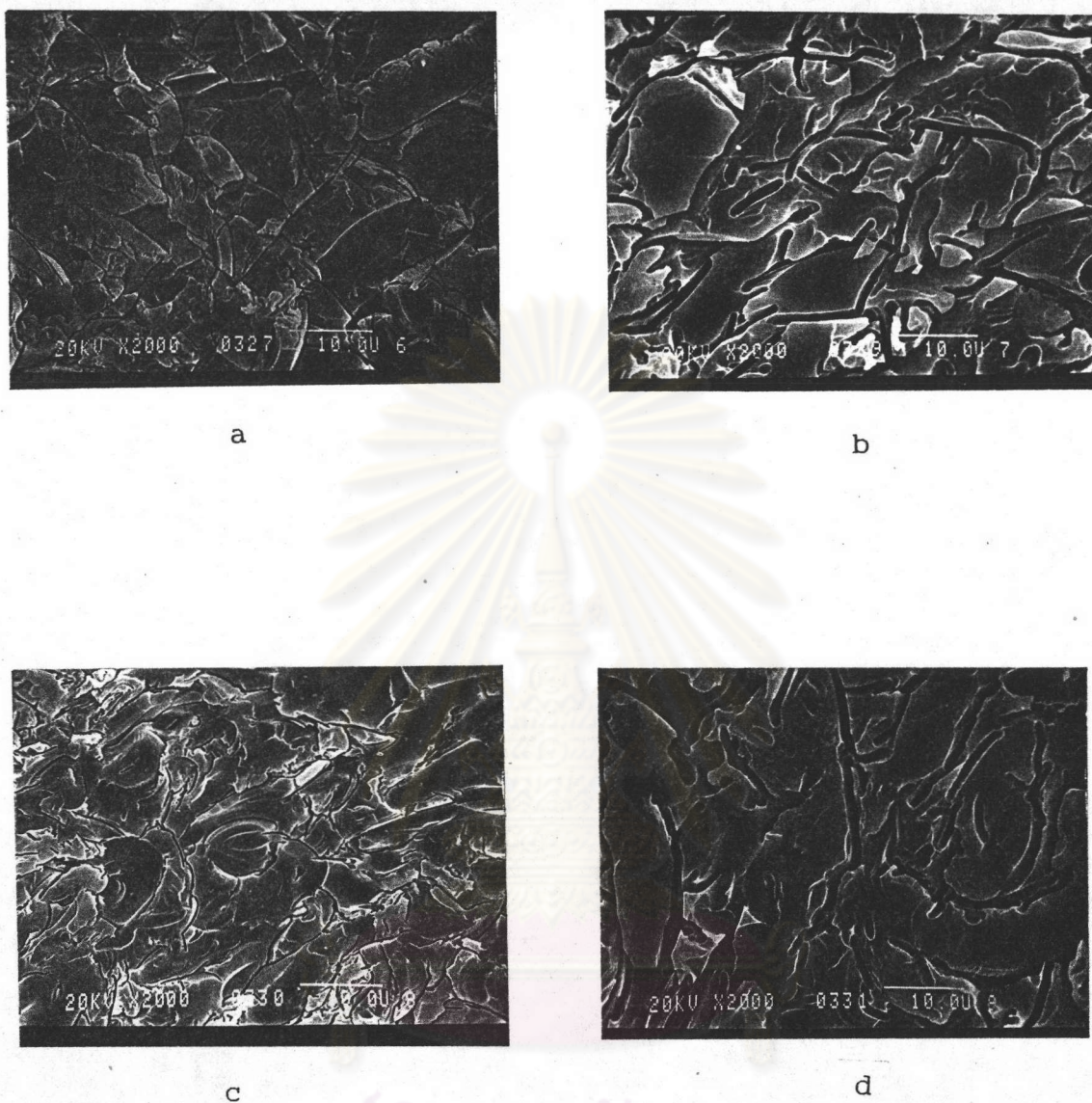


Fig. 3.12 SEM morphology of a) un-etching sample, b) etched with 5% HF + 5% HNO₃ (30s), c) etched with 5% HF + 5% HNO₃ (90s),

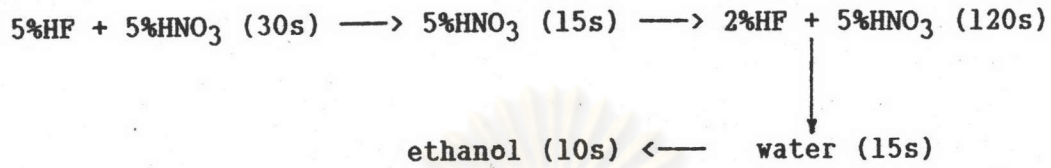


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Fig. 3.13 SEM morphology of etched sample;

- a) 2% HF + 5% HNO₃ (30s),
- b) 2% HF + 5% HNO₃ (120s),
- c) 2% HF + 2% HNO₃ (30s),
- d) 2% HF + 2% HNO₃ (120s).

The results shown that both conditions 1, 90s, and 2, 120s, can be used. Condition 3 can be used at 120s but this may take more time. So, strong etching followed by light etching was used. The etching step was selected like this:



Four types of sample which was already etched were inspected by SEM as shown in following figure.



Fig. 3.14 SEM morphology of etched sample with selective conditions;
a) JMI glass, b) B4 glass.



Fig. 3.15 SEM morphology of etched sample with selective conditions;
 a) Si glass, b) Et glass

The condition of the surface is not perfect; the geometrical surface is smaller than the surface actually exposed to the solution.

3.2.1.6 Preparation of Solution

A Gamble's solution as shown in the following table was prepared by weighing the substances with a precision 0.0001 g and mixing them in a brown bottle. Note that phosphate and carbonate were not mixed until the another compositions were mixed already. This is necessary to prevent precipitation in Gamble's solution.

Table 3.6 Composition of the simulated extracellular lung fluid used in the in vitro glass chip corrosion experiment in mg/l; CO₂ saturated at P(CO₂) = 0.05 bar.

Component	PH 7.6 ± 0.2	pH 5 ± 0.2*
MgCl ₂ .6H ₂ O	212	106
NaCl	6415	3208
CaCl ₂ .4H ₂ O	318	159
NaSO ₄ .10H ₂ O	179	90
Na ₂ HPO ₄	148	74
Na ₂ HCO ₃	2730	-
(Na ₂ tartrate).2H ₂ O	180	90
(Na ₃ citrate).5.5H ₂ O	186	5313
Na lactate	175	88
Glycine	118	59
Na pyruvate	172	86
HCl	-	44**

1 ml of formaldehyde per 1 l of solution was added in order to prevent the growth of algae and bacteria.

* = no CO₂ saturated with pH 5 solution

** = 12 ml of 1 M HCl per 1 l of solution was used.

3.2.1.7 Binder Coating

The conditions for coating the chip glass samples with binder were determined by experiment, too. A commercial binder from Microfibres Co, Ltd., was used. The composition is shown in the following table.

Table 3.7 Composition of binder.

component	amount
H ₂ O	1000 ml
Phenolic resin	25 g
(NH ₄) ₂ SO ₄	0.75 g
Silane	0.05 ml
Silicone	0.15 ml

These raw materials had to be kept in a refrigerator. Only phenolic resin had 2-3 weeks of life time.

3.2.2 Sample Preparation

3.2.2.1 Selection of Glass Composition

Four types of glass were selected corresponding to typical MMMF compositions investigated before. (Scholze and Conratt, 1992), i.e, JM glass, E glass, basalt and slag. For each type, two different compositions were molten. Variations in composition were chosen in such a way that significant effects could be demonstrated. Thermodynamic calculations (Conratt, 1992) had shown that the different effects of binder on the corrosion behavior of fibres are related to their MgO and CaO contents. Thus, a noticeable variation of the MgO and CaO content was envisaged.

The following page shows the 8 glass compositions which were selected.

JM1 = JM glass	JM2 = JM glass	} vary MgO and CaO content
E1 = E glass	E2 = E glass	
B4 = Basalt glass	B3 = Basalt glass	
S1 = Slag glass	S2 = Slag glass	

Table 3.8 Glass Composition in wt% oxides

oxides	JM1	JM2	E1	E2	S1	S2	B3	B4
SiO ₂	63.81	63.82	54.95	54.92	41.33	41.32	46.56	46.14
TiO ₂	0.06	0.06	0.04	0.04	0.70	0.70	2.91	2.93
Al ₂ O ₃	5.14	5.12	14.22	14.21	16.01	16.00	13.71	13.58
B ₂ O ₃	4.84	4.81	8.07	8.06	-	-	-	-
Fe ₂ O ₃	0.06	0.06	0.02	0.02	0.02	0.02	12.52	12.40
MgO	3.12	0.05	4.74	0.06	4.50	0.04	9.31	4.22
CaO	6.25	9.35	17.55	22.28	35.93	40.43	10.66	16.45
Na ₂ O	15.72	15.72	0.40	0.40	-	-	2.91	2.89
K ₂ O	1.01	1.00	-	-	1.50	1.50	1.41	1.39

3.2.2.2 Raw Materials

For the above 8 glass compositions, batch compositions were calculated. The batch compositions are showed in the following table. Along with this, the viscosity levels T_g, T(3.0), and T(1.5) for viscosities 10⁺¹³, 10⁺³, and 10^{+1.5} dPas, respectively, are given. These data are needed to properly process the glass melt.

Table 3.9 Batch Composition and Properties.

component	JM1	JM2	E1	E2	S1	S2	B3	B4
sand	405.21	408.82	415.57	419.86	292.25	294.92	364.52	244.16
feldspar	202.50	203.60	-	-	-	-	-	207.53
Al(OH) ₃	-	-	162.34	163.98	171.29	172.77	162.15	102.79
borax penta	84.59	84.82	-	-	-	-	-	-
dolomite	117.43	-	160.99	-	143.46	-	255.93	156.26
limestone	19.04	130.37	148.15	302.15	372.82	511.97	9.14	144.05
soda ash	160.06	161.50	5.18	5.19	15.48	-	38.76	5.49
potas	11.16	11.20	-	-	15.48	15.60	16.05	15.44
boric acid	-	-	107.78	107.78	-	-	-	-
titania	-	-	-	-	4.69	4.73	22.37	23.56
iron red	-	-	-	-	-	-	97.25	100.71
MgCO ₃	-	-	-	-	-	-	33.82	-
Tg	550.5	562.9	772.0	757.3	~790.0	~790.0	675.5	719.1
T(3.0)	1067.4	1043.6	1168.9	1181.7	1171.3	1198.3	1161.7	781.7
T(1.5)	1461.7	1440.9	1540.4	1557.0	1333.4	1337.0	1281.2	1160.3

3.2.2.3 Chip Sample Preparation

Procedure of glass melting

1st melt

1.5 kg of batch which was already weighed and mixed from the selected composition was charged into the crucible, then put into gas furnace and heated up step by step from room temperature to 1400 °C (time demand ~ 3 hrs), after that kept at

1400°C, for 1:30 h, cast into a copper mold, and cooled down in ambient air.

2nd melt

Glass sample from 1st melt was crushed ($\phi \sim 0.5-1.0$ cm), charged into medium size clay crucible and put into gas furnace, then heated up with the same condition as 1st melt, after that cast and cooled down in air.

3rd melt

Glass sample from 2nd melt was crushed ($\phi \sim 0.5-1.0$ cm), charged into small clay crucible, then put into an electric furnace and heated up according to the following scheme,

400 °C	20 min	900 °C	30 min
500 °C	30 min	1100 °C	60 min
600 °C	20 min	1400 °C	90 min
700 °C	30 min		



after that cast into copper-mold, anneal at $T_g + 50^\circ\text{C}$, (time demand 1 h per 1 cm thickness of sample), then cooled down in the annealing furnace over night.

Procedure of chip glass sample preparation

The glass from 3rd melt was cut into $2 \times 2.5 \times 0.2$ cm³ size by low speed saw, sharp edges removed by grinding paper or grinding wheel. After that, samples free of cracks, or bubbles were selected. Then the surface area of these sample was measured, and they were etched by HF and HNO₃ (see 3.2.1.5). The leachant had to be changed every 10 pieces of glass. Some samples were coated with binder. Some chip samples of each type of glass were selected for checking the chemical composition by EDX and morphology by SEM.

3.2.2.4 Fibre Sample Preparation

Three types of bulk glass, J1, E1 and B4, were molten again at approximately 1200°C, depending on each type of glass. The fibrization was done by using a pull machine at the speed ~ 50 rpm as shown in the following figure.

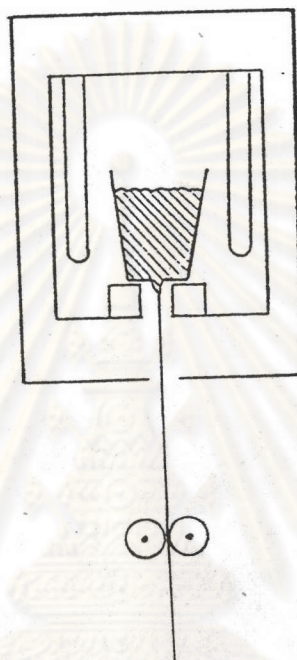


Fig. 3.16 Fibrization process with pull machine.

The fibrization temperature of J1, E1 and B4 glass was 1100°C, 1200°C and 1150°C respectively.

After fibrization, the appearance of fibres were investigated. Good fibres free of cracks or spots were chosen for corrosion testing. The surface area A of each individual fibre in cm^2 was calculated by the equation of $A = \pi dl$, d = diameter in μm , measured by using an optical scale microscope Nikon model, and l = length of fibre in cm.

3.2.3 Characterization of Prepared Samples

3.2.3.1 Appearance of glass melting.

Eight different glass types were compared in each step of melting, (chip samples and fibre samples). Color differences, homogeneity, amounts of bubble and the melting behavior in general were observed. This may help and give some clue to what happen during glass sample preparation.

3.2.3.2 Chemical Composition

Eight different glass compositions were molten and cut into chips form $2 \times 2.5 \times 0.2 \text{ cm}^3$ by a low speed saw. No further polishing was done. The respective number of samples were leached following the leaching condition as described in 3.2.1.5. After leaching, these samples were analysed chemically by XFA. Six standard glasses with already known chemical composition were used as standards to check the accuracy of the machine. For economic reasons, X-ray fluorescence analysis using the energy-dispersive discrimination mode (EDX) was used. The evaluation was based on the count per second (cps) of each element in each sample. The internal standards of the XFA apparatus as well as the sensitivity factors in the internal evaluation program are usually less well suited to analyze glassy materials.

3.2.3.3 Density Determination.

The density is a key property to calculate the rate of corrosion (see 3.2.3). With an analytical balance, it can be determined to at most $2 \times 10^{-4} \text{ g}$ by underhook weighing. This method is based on Archimedes' principle. First, the balance was calibrated by using the manual in the annex. Then 0.5 l of D.I. water were adjusted to the temperature of the balance room. Eight

chip samples of glasses were submitted to at least 10 underhook weighing cycles per sample. A transparent plastic was used as air shield around the apparatus to prevent errors due to air movement. Finally, the densities were calculated by using the following equation.

$$\rho(\text{glass}) = \rho(\text{water})/[1-m_2/m_1]$$

for, m_1 : mass of glass in air (g)

m_2 : mass of glass in water (g)

$\rho(\text{water})$: density of water in (g/cm^3)

$\rho(\text{glass})$: density of glass in (g/cm^3)

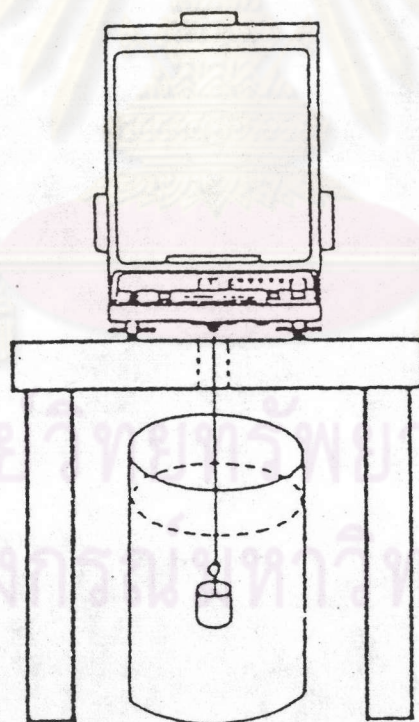


Fig. 3.17 Underhook weighing method.

3.2.4 Corrosion Tests

3.2.4.1 Chip Samples

1. The glass samples which were already leached were weighed by 5 decimals balance*.
2. A pieces of glass sample which was cleaned by ethanol was put into HDPE brown bottle.
3. The solution was put into the bottle by pipetts. The volume of solution in each bottle was limited by the factor of sample surface to leachant volume $s = 0.1 \text{ cm}^{-1}$.
4. The mixed gas $\text{N}_2:\text{CO}_2 \sim 95:5$ was bubbled into the bottle until the pH meter showed 7.6 for original solution and phosphate-free solution. For some tests, only N_2 gas was used; the pH would reach $8.5 + 0.2$. For solution buffered at pH 5, there was no need to bubble gas.
5. The bottles were kept at $37^\circ\text{C} \pm 1^\circ\text{C}$ for usually 2 months; for some tests, 4 months were required.
6. The temperature was checked by a medical thermometer every 2 days.
7. Gas was bubbled every 2 days, and the solution was renewed every 7 days.
8. Glass samples were weighed every 14, 28, 42 and 56 days. (112 days for some tests).

* 5 decimals balance : Weighing 5 times; the standart deviation should not be exceed $\pm 0.00002 \text{ g}$. The balance was calibrated every time befor use. To make sure that the balance was stable, the dispaly 0.00000 g had to prevail for 10s befor weighing. The sample weight was read 50s after placing the sample on the balance.

9. For studying the microstructure of glass surface, some samples was selected for SEM investigation.

10. The corrosion rate in each condition was calculate from the equation $R = w / A\rho t$ while w = weight loss in g, A = surface area in cm^2 , ρ = density in g/cm^3 and t = time in day.

3.2.4.2 Fibre Samples

Two types of experiment were designed as shown in the following figure.

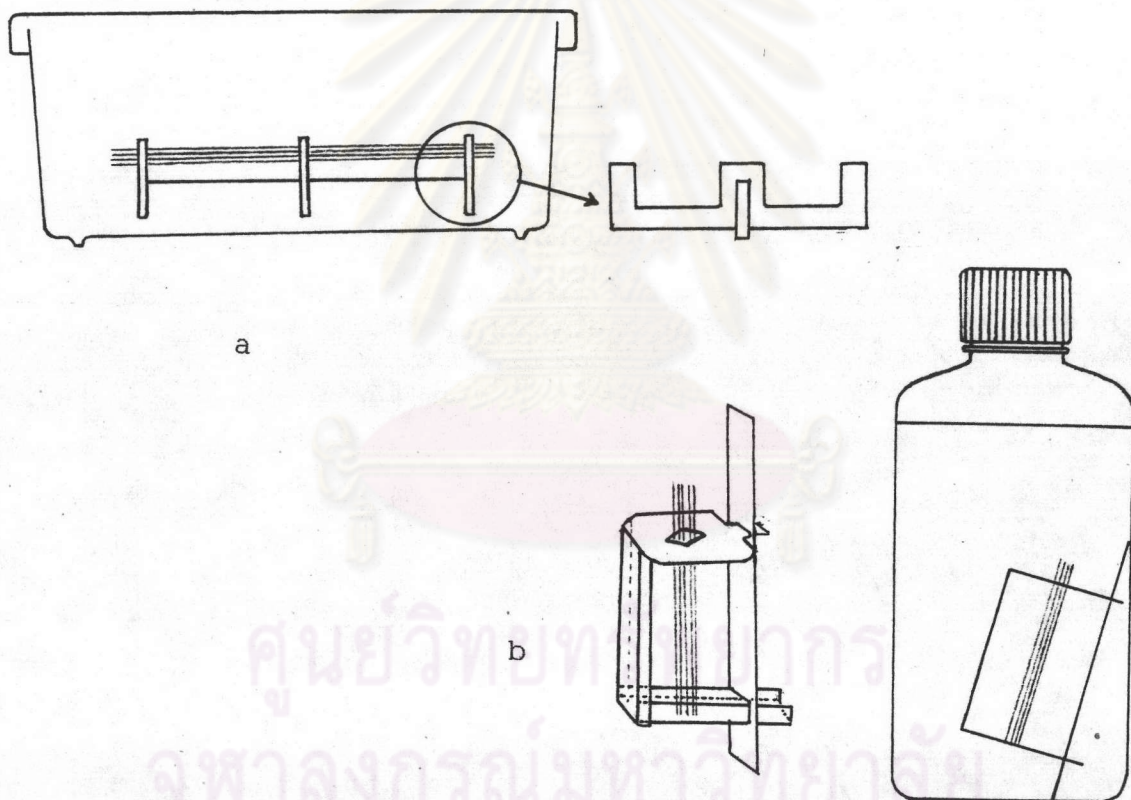


Fig. 3.18 Two types of experimental design

A number of fibres; (number depending on $s = 0.1 \text{ cm}^{-1}$) and $l = 5 \text{ cm}$, $V \sim 140 \text{ cm}^3$ for type a, $V \sim 160 \text{ cm}^3$ for type b; were weighed by 5 decimals balance, cleaned by ethanol and then put into HDPE brown bottle.

The experiment was performed for 7 days with 4 conditions, i.e. original solution, N₂ - bubbled solution, pH 5 and phosphate free solution. After the test, some types of fibre were selected for inspection by SEM.

3.2.5 Corrosion Characterization

3.2.5.4 Morphology

Optical microscope and scanning electron microscope (SEM) were used to inspect the surface of glasses. Firstly, this was used as a guide to selected the leaching condition. Secondly, it served as an illustration of what happen upon glass corrosion.

To observe the morphology by SEM, the chip samples were dried at 110°C for 1 hour, broken into small pieces, so as to make them fit into the SEM sample support, sputtered with gold and inspected by SEM.

3.2.5.2 Corrosion Rate Determination

Corrosion rates both of chips and fibres sample were determined from weight loss. This is the main purpose to re-check a predictive model. An effect of phosphate, carbonate, binder, buffer solution at pH 5 and geometry were also investigated.