

## Chapter 3



### Experimental Part

#### Description and Scope

In accordance with its title, the long-term goal of the thesis work is the preparation of new glass composite materials. So, mixtures of agar gel (serving as diffusion medium for the precipitation process) and a commercial soda lime glass powder are prepared as matrix material. Since agar gel contains > 99 wt% water, it can be removed from the glass powder matrix, thereby leaving the precipitated structure in place. Description and scope of experiments are shown in Fig.3.1.

The experimental work consisted of preparation of preforms and of characterization. Preparation of the preforms comprised four steps. These are: glass powder preparation, gel preparation, precipitation tests, and heat treatment. An overview over the entire scope of experimental work is given in the flow chart in Fig.3.1. The specific difficulty of the preparation route lies in the fact that any individual problem solution had to stand the test of all consecutive step, too. Often, a problem solution which was specifically good for one individual step

of the preparation route had to be dismissed and rejected in a later stage where it caused unsolvable problems. This resulted in a problem solution strategy similar to the loop of a computer program. The preparation route had to be "looped through" many times until a suited preparation path was developed. Fig.3.1a describes in detail the progress of the preparation work. The first challenge was to overcome heterogeneous nucleation. HF etching was not successful, but silicone oil treatment solved the problem: Distinct precipitation patterns were obtained. This showed that hydrophobation of the glass grains was the appropriate answer. However, the problems caused by the silicone oil in the consecutive step were unsolvable. So the entire route had to be started all over again. Finally, a combined silane and paraffin treatment lead to the success.

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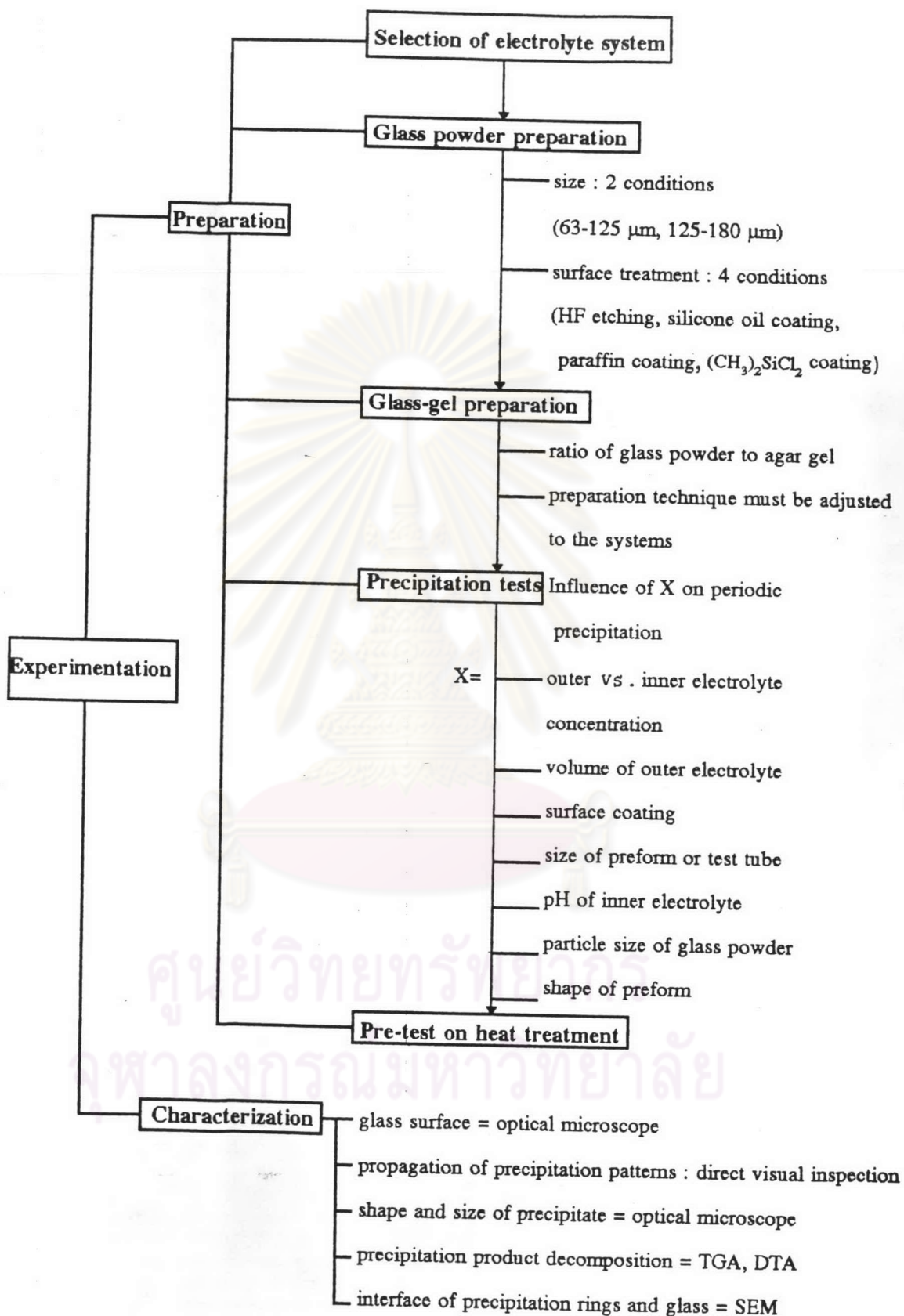


Fig.3.1 Flow chart of experimental work

Table 3.1 Flow chart of the problem solving process from the start to the final success

	step 1 Glass powder preparation	step 2 Gel preparation aq. solution, T=90 °C	step 3 Pre-test precipitation	step 4 Heat treatment T ≈ 500-700 °C
first start → use original glass	++	++	-	++
solve → treat surface with HF (remove edges)	++	++	problem : no bands	++
solve → treat glass surface with silicone oil ( hydrophobation)	++	+	problem : no bands	-
solve → eliminate silicone oil before heat treatment by chemical means		(homogenization of preform is difficult)	++	problem : silicone oil can not be removed ; remains as SiO <sub>2</sub> or Si
new start → treat glass surface with (CH <sub>3</sub> ) <sub>2</sub> SiCl <sub>2</sub> (hydrophobation)	++	+	-	++
solve → treat glass surface with paraffin wax	-	(homogenization of preform is difficult)	problem : single band on no bands	++
solve** → control temp. during gel preparation (≤ 65 °C)		problem : very fast solidity of paraffin wax, coating non homogeneous	problem : little no. of ring, non straight bands	
solve*** → treat glass surface with (CH <sub>3</sub> ) <sub>2</sub> SiCl <sub>2</sub> first, then with paraffin wax	-	+	+	
solve* → develop coating method by using excess paraffin wax and eliminating the excess wax	+	+	increased no. of ring problem: non straight bands	++
continue → study other factor	Successful preparation of a suited preform			
	(concentration of electrolyte, volume of electrolyte, pH, size of glass, tube, shape of preform)			

-: failure ; +: good, but difficult to preform ; ++: very good, easy to make

## Apparatus and Instruments

1. test tube
2. beaker
3. stirring rod
4. dropper
5. spatula
6. wash bottle
7. funnel
8. stand
9. pipet
10. test tube rack
11. volumetric flask
12. porcelain crucible
13. thermometer
14. water bath
15. hot plate
16. oven
17. furnace
18. agate mortar
19. attrition mill
20. steel hammer
21. vibrator
22. sieve



23. ultrasonic bath
24. pH meter
25. optical microscope
26. microscope slide
27. thermal analyser (thermogravimetry and differential thermal analysis)

### Chemical reagents

Table 3.2 Chemical reagents used in the experiments

Chemical reagents	Formular	Specification	%Assay Source
Ammonium oxalate	$(\text{NH}_4)_2\text{C}_2\text{O}_4$	142.11 g/mol	>99.5 May Baker LTD
Cupric nitrate-Trihydrate	$\text{Cu N}_2\text{O}_6 \cdot 3\text{H}_2\text{O}$	241.60 g/mol	>98.0 Fluka Chemikals
Ammonium hydroxide	$\text{NH}_4\text{OH}$	$d^{20} 1.6695 \pm 0.006$	28.0 AJAX chemicals
Acetic acid	$\text{CH}_3\text{COOH}$	$d^{20} 0.89$	- Merk LTD.
Formadehyle	$\text{H}_2\text{CO}$	-	- Fluka chemicals
Silicone oil	$-(\text{CH}_3)_2\text{Si-O})_n$	-	- Union Carbide LTD.
Trichloroethylene	$\text{C}_2\text{HCl}_3$	131.40 g/mol	- Fluka Chemicals
Paraffins wax	$\text{C}_n\text{H}_{2n-2}$	$T_{\text{lig}} = 65^\circ\text{C}$	- Common
Dimethyldichlorosilane	$(\text{CH}_3)_2\text{SiCl}_2$	129.05 g/mol	99.5% Fluka Chemicals
Hydrofluoric acid	HF	20.01	48% Merck
Hydrochloric acid	HCl	36.46 g/mol	35.4% Merck
agar gel	-	-	- Common

## Procedure

### 3.1 Selection of electrolyte systems

When selecting electrolyte systems for experimentation, the aspect of potentially new functions (high electric conductivity and high optical transparency) was kept in mind. In fact, the systems had to pass three criteria. These are criteria related to:

- function : Does the selected system have the potential to accomplish a material with an interesting function ?
- precipitation : Can the system be used to bring about a distinct unisotropic distribution (periodic precipitation) of a desired compound in agar gel mixed with glass powder matrix ?
- materials preparation : Does the applied system not impair

the transparency or other properties of the glass when passing the drying and sinter step ?

3.1.1. According to function : Recent papers have shown that even randomly introduced phases in silica glass have the potential to accomplish interesting and promising functions. Some target compounds investigated were AgCl (unpublished), CuCl (35), CdS (36). It has also been demonstrated just lately (23) how the otherwise exclusive functions of high optical transparency and electric conductivity can be united within the same material by an unisotropic distribution of an alien compound in a polymer matrix.

In this study, electrical conductivity and of glass is the property envisaged on a long-term basis. So Cu was given a closer look as function carrier element.

3.1.2 According to precipitation : Table 3.3 gives an overview over the dissociation steps and solubilities of Cu compounds. Table 3.4 complements the selection with a number of counter-electrolytes with high solubility in water, each bearing only one ion of a



certain target precipitate.

Table 3.3 Solubility limits (18 to 25 °C) of potentially suited precipitates.

precipitate	cation	anion	log $K_s$
$\text{CuCrO}_4$	$\text{Cu}^{2+}$	$\text{CrO}_4^{2-}$	-5.40
$\text{CuC}_2\text{O}_4$	$\text{Cu}^{2+}$	$\text{C}_2\text{O}_4^{2-}$	-7.54
$\text{FePO}_4$	$\text{Fe}^{3+}$	$\text{PO}_4^{3-}$	-21.89
$\text{FeC}_2\text{O}_4$	$\text{Fe}^{3+}$	$\text{C}_2\text{O}_4^{2-}$	-6.49
$\text{Fe(OH)}_3$	$\text{Fe}^{3+}$	$\text{OH}^-$	-38.27
	$\text{FeOH}^{2+}$	$\text{OH}^-$	-26.82
	$\text{Fe(OH)}_2^+$	$\text{OH}^-$	-16.38
	$\text{Fe(OH)}_3$		-6.52

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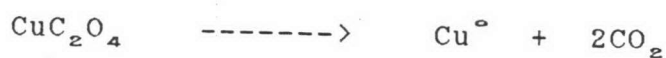
Table 3.4 Solubility limits (30 °C) of highly soluble electrolytes carrying ions of potential target precipitates, in g per 100 g H<sub>2</sub>O

compound	g/100 g
CuSO <sub>4</sub>	37.8
CuNO <sub>3</sub>	156.0
(NH <sub>4</sub> ) <sub>2</sub> CrO <sub>4</sub>	39.3
(NH <sub>4</sub> ) <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	6.1

From table 3.3 and 3.4 target precipitates with low solubility product and very soluble carrier partners can be chosen.

### 3.1.3 According to materials preparation

The target compounds containing Cu were chosen in such a way that the anion hopefully would be removable from the glass matrix during a heat treatment step, thereby reducing the copper. Copper oxalate (CuC<sub>2</sub>O<sub>4</sub>) matched this requirement well. Upon heat treatment in a non-oxidizing atmosphere, a dissociation like



can be expected to occur, leaving metallic copper behind. As co-anion of the Cu bearing reservoir electrolyte, nitrate were chosen because it has high solubility in water. For the counter-cation, alkali or alkaline earth could not be used since this would impair glass properties during a later sintering step. This left little choice, e.g.,  $\text{NH}_4^+$ .

### 3.2 Glass powder preparation

Glass powders were prepared from a commercial glass from Thai Glass Industry Co.,Ltd.. The chemical composition is given in table 3.5. Properties as calculated from UNIGLASS software are shown in table 3.6.

Table 3.5 Chemical composition of glass sample

chemical composition	wt. %
$\text{SiO}_2$	71.86
$\text{Al}_2\text{O}_3$	1.42
$\text{Fe}_2\text{O}_3$	0.026
$\text{TiO}_2$	0.032
$\text{CaO}$	7.51
$\text{MgO}$	4.56
$\text{K}_2\text{O}$	0.01
$\text{Na}_2\text{O}$	14.58

Table 3.6 The properties of a commercial glass sample.

symbol	meaning	value unit
M	molar mass	59.067 g/mol
q	density	2.492 g/cm <sup>3</sup>
$\alpha_{20-400}$	thermal expansion coefficient 20-400°C	$9.3 \times 10^{-6} \text{ K}^{-1}$
$T_{\text{liq}}$	liquidus temperature	1017 °C
$T_g$	glass transition temperature	544 °C
$T_M$	dilatometric softening point	584 °C
$T_L$	Little ton temperature	724 °C
$T_{\text{max}}$	crystallization maximum	960 °C
$G_{\text{dev}}$	Gibbs free energy of devitrification	7581 J/mol
$H_m$	enthalpy of melting	20,657 J/mol
G	shear modulus	27,387 J/mol
A	Vogel-Fulcher-Tammann constants	-1.8066
B		4,657.2
$T_0$		229

the viscosity at T (°C) is given by  $\log \eta = A + B/(T-T_0)$

### 3.2.1 Procedure of glass powder preparation

The first step of glass powder preparation consisted in crushing the glass sample manually with a steel hammer. The fraction smaller than  $180\ \mu\text{m}$  and bigger than  $63\ \mu\text{m}$  was separated by a sieve, another blow was applied, and so on. This procedure is known to avoid multiple fracturing of the particles which causes abundant fissures and sharp edges. The flow chart of glass powder preparation is given in Fig.3.2.



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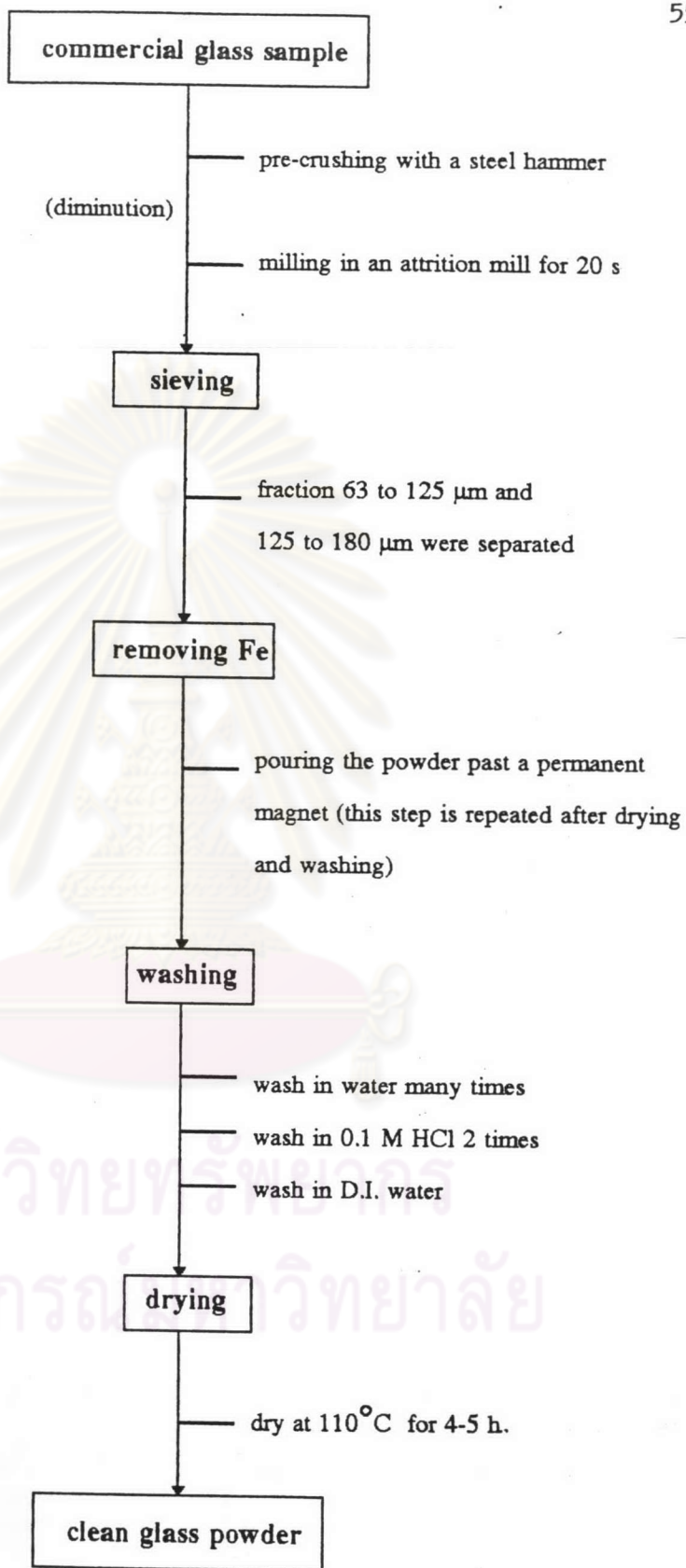


Fig.3.2 Flow chart of glass powder preparation



### 3.2.2 Surface treatment of clean glass powder

This step is the most difficult one because if the coated glass surface passes the step of gel preparation but does not succeed in the step of precipitation, this step must be repeated under varied conditions.

#### 3.2.2.1 Treatment by etching with HF and $\text{HNO}_3$

The first treatment chosen is hydrofluoric acid (HF) by immersing the clean glass powder in 5% HF mixed with 5%  $\text{HNO}_3$  for 30 sec., then in 5%  $\text{HNO}_3$  for 15 sec. and 2% HF mixed with 5%  $\text{HNO}_3$  for 120 sec.. Then the powder was washed with water for 15 sec. and immersed in ethanol solution for 10 sec.. HF was applied with the purpose to remove any sharp corners and edges. This step aimed at producing a powder with a possibly lowest number of surface flaws so as to reduce the number of sites able to initiate heterogeneous nucleation in the consecutive precipitation test. But in this thesis it turned out that HF is of no help to prevent heterogeneous nucleation and to support periodic precipitation (see table

4.1, test no. 4)

### 3.2.2.2 Treatment with silicone oil

After the failure in 3.2.2.1, we needed to find another coating or method to treat the glass powder surface, and silicone oil was chosen because it was an agent suited to render the powder surface hydrophobic. It indeed proved to be a suitable measure to by-pass the problem of heterogeneous nucleation (see table 4.1, test no. 5-7). The procedure for treatment is immersion of the cleaned glass powder in the solution of a silicone oil diluted with trichloroethylene ( $C_2HCl_3$ ). The ratio of silicone oil: trichloroethylene was 1:3. The surface bonding was established by subsequent heating, when a thin film of silicone oil bonded to the glass surface by baking at approximate  $300^\circ C$  for an hour. Unfortunately, the high oxygen deficiency of the silicone coating spoiled the glass properties during heat treatment. That is why the main test series, an alternative hydrophobic agent with a less high oxygen deficiency had to be applied.



### 3.2.2.3 Treatment with Dimethyldichlorosilanes

Dimethyldichlorosilane ( $(\text{CH}_3)_2\text{SiCl}_2$ ) is used as an hydrophobic agent and can be processed into a film by the same method as described in 3.2.2.2 (see table 4.1, no. test 10-11).

### 3.2.2.4 Treatment with paraffin wax

For the main test series, paraffin wax coating was used. Paraffin wax has the advantage that it can be removed by physical evaporation. However, the bond to the glass surface is of mere physical nature only. In using the paraffin wax, many problems had to be solved and a procedure had to be developed until the coating was uniform. First the paraffin wax was molten and mixed with the glass powder. This method of mixing was not good. The film on the glass surface was not uniform. Rather, quite large agglomerates occurred. So a new method was developed using a high excess of molten paraffin wax and eliminating the excess of paraffin wax. In the new method, the paraffin wax is molten, poured to the glass powder, then stirred until the powder is well dispersed. The excess is removed through filter paper.

The entire procedure must be performed in a heating chamber of about 80 °C. Filtration was performed until no drops of paraffin wax dropped through the filter paper. By this method, the glass powder was coated uniformly and homogeneously and the amount of paraffin wax was kept very low, and did not cause excess porosity of glass perform during heat treatment. Glass powders pre-coated with dimethyldichlorosilane were coated with paraffin wax in the same way.

### 3.3 Glass-gel preparation

The glass-gel perform able to develop periodic precipitation must have a homogeneous dispersion. So the ratios of glass powder to agar gel and the method of preparation is important.

#### 3.3.1 Ratio of glass powder to agar gel

The importance of this step of work was the establishment of a preparation path yielding sufficiently densely packed glass powder samples embedded in an interconnected agar gel phase. Four different ratios of agar gel to glass powder were investigated. The reference ratio to start with is given by the ratio of void volume to weight of glass of a statistically packed powder.

### 3.3.2 The ratio of void volume to weight of glass of glass powder preform

The ratio of void volume to the weight of glass is found by weighing the empty test tube ( $W_1$ ), the test tube was filled with glass powder ( $W_2$ ) and then adding water was added to fill the voids ( $W_3$ ). water level reached the uppermost layer of powder. The weight of water in the voids ( $W_4$ ) is calculated by:

$$W_4 = W_3 - W_2$$

The density of water is approximated as 1 g/ml.. So the void volume ( $V_{\text{void}}$ ) is  $|W_4|$  ml. The ratio of void volume to weight of glass given in ml/100 g glass is

$$R = \frac{V_{\text{void}}}{W_2 - W_1}$$

The agar gel solution in the preform should be equal to the void volume of the densely packed powder. Lower and higher ratios were prepared, too. The best ratios resulting in dense packing yet good precipitation behavior were selected. The process of glass-gel preparation is shown in Fig.3.3.

### 3.3.3 Preparation of glass-agar gel preform

Agar gel solutions of 0.1 wt% using inner electrolyte as solvent were prepared. They were heated to dissolve the agar powder and boiled to prepare an agar sol. Then the glass powder was added to an excess amount of agar sol and stirred until the mixture was uniformly dispersed. The mixture was poured into a test tube (diameter 1.3 cm). Trapped air bubbles were removed by means of a vibration table. Then the excess amounts of agar sol were sucked off. The test tubes were closed with paraffin film and cooled down thus allowing the agar sol to form a gel. This step was performed very carefully because if the agar sol gels too early, the glass-gel preform is not uniform or contains residual bubbles. The density of glass powder preform was controlled by the masses of glass powder and heights in the test tubes.

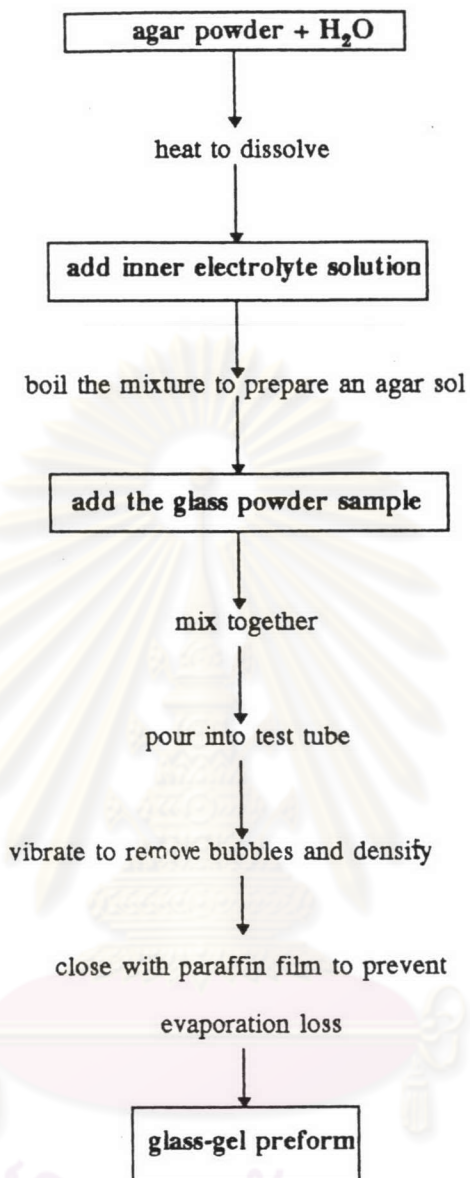


Fig.3.3 Flow chart of glass-gel preparation

Restriction in preparation of glass-gel preforms with hydrophobic glass powders required special care because the glass surface was not easy to wet and the sols tended to gel too fast. So a sufficiently high temperature and fast mixing process must be applied to comply with the short setting time from sol to gel. On the other hand, in the case of coating with paraffin wax, the temperature must not be too high in order to avoid destruction of the film of paraffin wax, which occurred above the melting point of paraffin wax ( 65 °C).

### 3.4 Pre-test on periodic precipitation

In this step, the outer electrolyte was filled on top of the glass-gel preforms into the test tubes. The tubes were closed with paraffin film to avoid evaporation and left in rest until the periodic precipitation was developed. Before the production of periodic precipitation of a metal compound of copper oxalate in glass-gel preforms was successful, many cases of pre-tests had to be studied (see table 4.1)

#### 3.4.1 Precipitation in pure agar gel

Pre-tests to produce the periodic

precipitation bands in pure agar gel containing  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  0.1 M as inner electrolyte solution and  $\text{Cu}(\text{NO}_3)_2$  0.2, 0.3, 0.5 and 1.0 M as outer electrolyte solution respectively, were performed.

#### 3.4.2 Precipitation in original glass-gel preforms

- Pre-test to produce the periodic precipitation bands in original glass-gel preforms contains  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  0.1 M as the inner electrolyte solution and  $\text{Cu}(\text{NO}_3)_2$  0.3, 0.5, 0.7 and 0.9 M as the outer electrolyte solution respectively, were performed. Based on these tests, the concentration of electrolyte suitable for precipitation was determined.

- Pre-test to produce the periodic precipitation bands in original glass-gel preforms with inversed electrolyte systems, i.e., with  $\text{Cu}(\text{NO}_3)_2$  0.2 M as the inner electrolyte solution and  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  0.2, 0.3 and 0.5 M as the outer electrolyte solution were performed. Based on these test, the effect of the best arrangement of the electrolyte solution on periodic precipitation formation was determined.

### 3.4.3 Precipitation in HF etched glass-gel preforms

Pre-test to develop the periodic precipitation in glass-gel preforms with HF etched glass surface were performed. The inner electrolyte solution was  $\text{Cu}(\text{NO}_3)_2$  0.2 M and the outer electrolyte solution is  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  0.2 and 0.3 M. In these pretest, the effect of the height of glass preforms was studied too.

### 3.4.4 Precipitation in hydrophobic glass (treated with silicone oil) -gel preforms

The pre-tests of section 3.4.2-3.4.3 were not successful. So alternative coatings were chosen to develop the periodic precipitation. In this step, silicone oil was used to make the surface of the glass hydrophobic. Precipitation was studied by using  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  0.1 M and 0.2 M as the inner electrolyte solution and  $\text{Cu}(\text{NO}_3)_2$  0.05, 0.1, 0.2, 0.3, 0.4 and 0.5 M as the outer electrolyte solution, respectively. The inverse arrangement of electrolyte solution by using  $\text{Cu}(\text{NO}_3)_2$  0.1, 0.2, 0.3 and 0.4 M as the inner electrolyte solution and 0.1 M  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  as the outer electrolyte was



applied, too. These pre-tests were very successful in developing periodic precipitation. But, unfortunately, in the heat treatment step severe problems occurred because the silicone oil is a very reducing agent, impairing the properties of the glass. From these tests, two essential things can be learned: Firstly, hydrophobic glass powder is suitable to allow periodic precipitation patterns to form. But, secondly, the coating agent must be removable at temperatures before the glass starts to sinter, i.e., preferentially below the  $T_g$  (544 °C).

#### 3.4.5 Precipitation in other hydrophobic glass-gel preforms

The best systems of concentrations found in section 3.4.4 were used to test the other coatings. The glass surface treated with dimethyldichlorosilane was studied first. The effect of pH of inner electrolyte solution and temperature was studied too. The result is not good (no precipitation patterns occurred), so paraffin wax was chosen as coating agent. Finally a combination of both dimethyldichlorosilane and paraffin wax was used for the coating of the glass surface.

### 3.4.6 selecting the most suitable coating and conditions of electrolyte

From the experiments in section 3.4.1-5 we get the suitable coating and the conditions for the development of periodic precipitation. The glass surface treated with dimethyldichlorosilane and paraffin wax was the best. And the outer and inner electrolyte solution was 0.3 M  $\text{Cu}(\text{NO}_3)_2$  and 0.1 M  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  respectively. So in the main tests series, this specific system was used.

### 3.5 Main tests on periodic precipitation

In the main tests, the glass was treated with dimethyldichlorosilane and paraffin wax as described above.

#### 3.5.1 Tests for the effect of outer electrolyte concentration

By using inner electrolyte solution of  $(\text{NH}_4)_2\text{C}_2\text{O}_4 = 0.1 \text{ M}$  and varying the concentration of outer electrolyte solution from 0.2 M to 0.3 M respectively the most suitable combination was determined.



### 3.5.2 Tests for the effect of inner electrolyte concentration

By varying the inner electrolyte concentration of  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  from 0.1 to 0.25 M respectively and using 0.3 M  $\text{Cu}(\text{NO}_3)_2$  as outer electrolyte the most suitable combination was determined.

### 3.5.3 Tests for the effect of the volume of the outer electrolyte

By using 0.3 M  $\text{Cu}(\text{NO}_3)_2$  as outer electrolyte solution and 0.25, 0.1 M  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  as inner electrolyte solution respectively and varying the volume of outer electrolyte (1.0 ml and 2.0 ml) the most suitable combination was determined.

### 3.5.4 Tests for the effect of pH of inner electrolyte

By using the system of 0.3 M  $\text{Cu}(\text{NO}_3)_2$  as outer electrolyte and 0.1 M  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  at pH 4.0, 6.3 and 9.4 as inner electrolyte solution the effect of the pH value on the precipitation process was determined.

### 3.5.5 Tests for the effect of the diameter of test tubes.

By using tubes with diameters of 1.05, 1.30, 1.60 and 2.40 cm in the system of 0.3 M  $\text{Cu}(\text{NO}_3)_2$  and 0.1 M  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  as outer and inner electrolyte solution, the effect of geometry on the precipitation process was studied.

### 3.5.6 Tests for the effect of particle size of the glass powder

The effect of fractions of glass powder from 63 to 125  $\mu\text{m}$  and 125 to 180  $\mu\text{m}$  using the same condition as in section 3.5.5 were compared.

### 3.5.7 Tests for the effect of temperature during precipitation

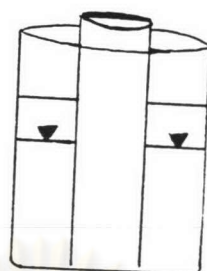
By compare two systems with different temperature-time programs, the effect of temperature was demonstrated. The first test was left for precipitation at 30 °C for 2 days, then kept at 2 °C for 4 days, and finally at 30 °C for 4 days. The latter test was kept at 30 °C for 2 days and then at 2 °C for 8 days. The sys-

tem of the electrolytes was the same as in section 3.5.6

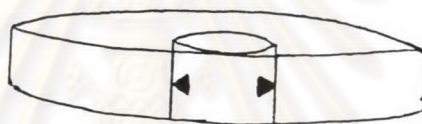
### 3.5.8 Pre-tests on other geometrical shapes of preforms

Besides the rod shapes, hollow rod and torus shaped preforms were studied. In the preparation of hollow rod preforms, the empty space between were used in big tubes (2.40 cm serving as outside diameter) and small tubes (1.05 cm serving as inside diameter) was filled by gel and outer electrolyte solution to generate a precipitation pattern of consecutive rings. From the point of view of electric conductivity, these rings have the potential function of inductive loops (see Fig.3.4 a). The torus preform was made by using a flat dish with diameter 9.50 cm. The diameter of the outer hole is 2.40 cm, filled with outer electrolyte. The result is a sequence of horizontal rings likewise having the potential of inductive loops (see Fig.3.4 b).

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a)



b)

Fig.3.4 Glass-gel preforms in shapes of

a) hollow rod

b) torus

### 3.6 Pre-tests on heat treatment

3.6.1 Pre-tests on drying by heating at  $250\text{ }^{\circ}\text{C}$  for 2-3 h were performed to check for the removal of water.

3.6.2 Pre-tests on heat treatment in the range of 500 to 700 °C for 2 h were performed to check for the complete removal of coating agents and for the behavior of precipitates within a sintering glass powder matrix.



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