

Chapter I

Introduction

Motivation, Objective and Scope

Glass is usually fabricated by melting. But if complicated shapes or new composite materials of glass are made, monolithic glass pieces can also be fabricated by viscous sintering from glass powders. These glasses are ground to small size which can be formed to any shape with an appropriate binder. Commercial glass is a basic type used in this study, because the composition of this glass is available at constant composition, and it is very stable against crystallization.

The objectives for the research are: preparation of monolithic glass pieces by viscous sintering, construction of a long - range dilatometer for on - line study of the sintering. The long - range dilatometer is built for sintering specimens in the range of T_M (the dilatometric softening temperature) and T_L (the Littleton softening temperature) because the ordinary dilatometer used in laboratory can't detect the shrinkage in this range. In this thesis, the viscosity level is used instead of temperature because the viscosity is the primary variable to calculate the time for sintering and it is used to compare the behavior of shrinkage of different glasses. The viscosity level can be indicated in terms of $\log \eta$, η in dPas, by giving the temperature at which a specific value of η is reached. This is done in the format $T(n.n)$, T in $^{\circ}\text{C}$, value $n.n$ is the numeric value of $\log \eta$.

There are two parts in this thesis, theoretical part and experimental part. The theoretical part is to study the viscous sintering and how to construct and know the principle of the long - range dilatometer while the experimental part is to prepare the specimens and study the sintering of them. This thesis work is illustrated in the flow chart below.



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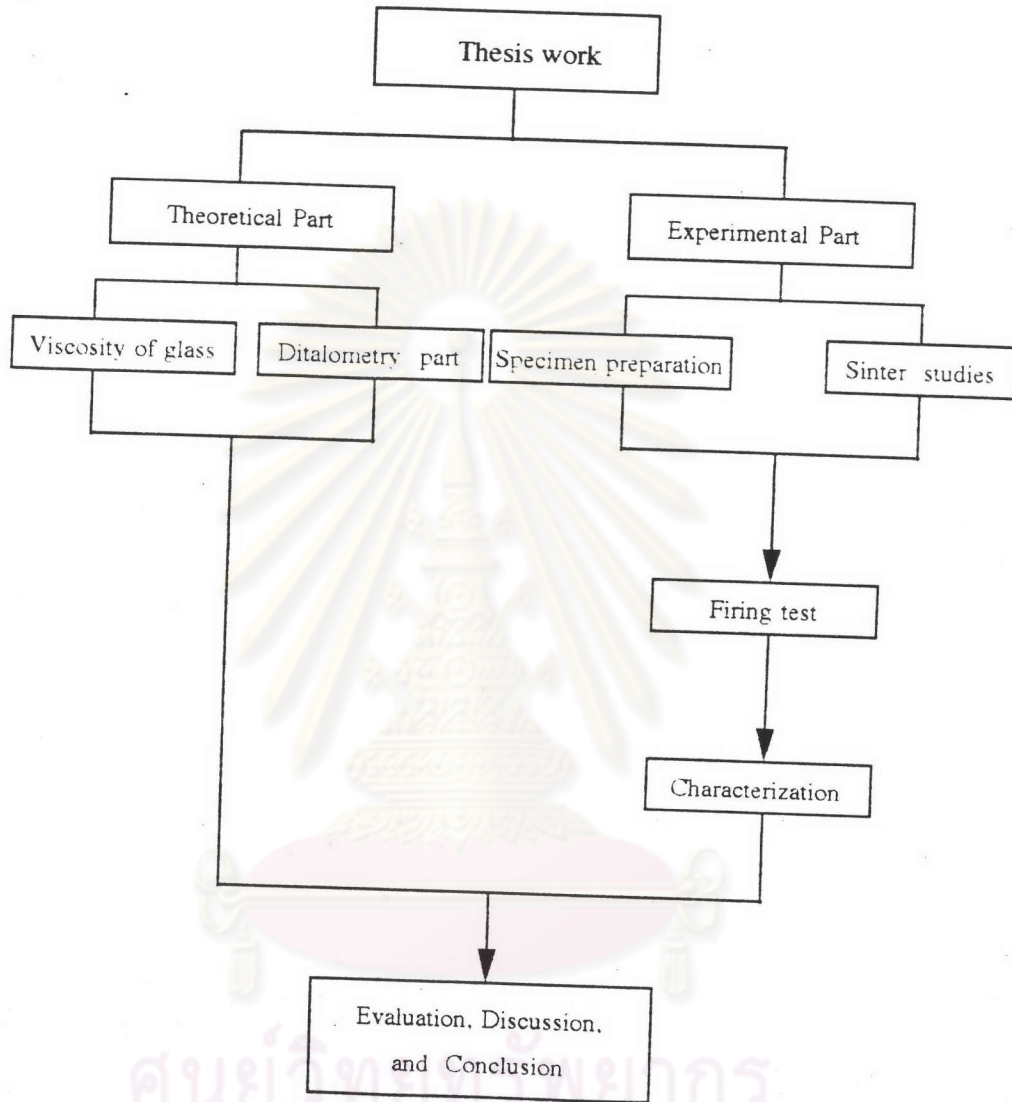


Fig. 1.1 Flow chart of thesis work

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Literature Survey

Kim and Ondracek (1995) studied the viscous sintering of glass powder. They studied with the commercial glass, borosilicate glass, soda-lime-silicate glass, and quartz glass, which was ground, and one fraction (100 - 120 μm) was selected. The cylindrical specimens (6 mm diameter, 25 mm length, $70 \pm 2\%$ theoretical density) were obtained by uniaxial cold pressing (135 Mpa) and drying at 120 $^{\circ}\text{C}$. The sintering temperature (T_{sin}) is calculate from the equation

$$T_{\text{sin}} \approx 0.8 T_L \text{ (in K)}$$

where T_L is the Littleton softening temperature

The shrinkage curve and viscosity as a function of temperature for the glasses studied is shown in the figure 1.2

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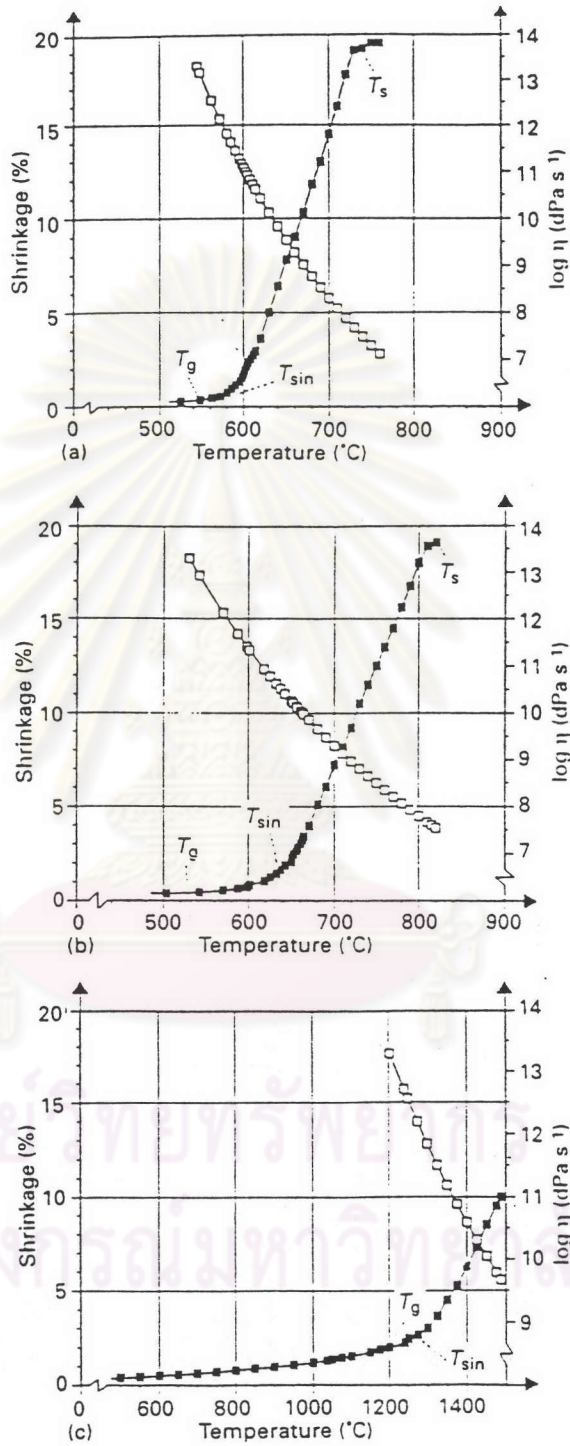


Fig. 1.2 (■) Linear shrinkage and (□) viscosity as a function of temperature for (a) soda-lime-silicate glass, (b) borosilicate glass, and (c) quartz glass.

Rahaman and Jonghe (1990) studied about sintering of glass powder under uniaxial stress. The sintering of spherical borosilicate glass powder under a uniaxial stress was studied at 800 °C. The experiments allowed the measurement of the kinetics of densification and creep. The viscosities for creep and bulk deformation, and the sintering stress were found to increase with density. The data show excellent qualitative agreement with Scherer's theory of viscous sintering (Scherer (1977)). In addition, the quantitative comparison between theory and experiment shows good agreement, the measured viscosity of the bulk glass was $\approx 1 \times 10^9$ dPas compared to $\approx 3 \times 10^9$ dPas obtained by fitting the data with Scherer's theory.

Clark and Reed (1986) studied the kinetics processes viscometry, crystal growth kinetics, and glass powder sintering were used to determine the activation energies of viscous flow, crystal growth, and sintering behavior of the standard glass, NBS 710, and two crystallizable glass powders in the calcia-aluminosilicate system. In a system which sintered successfully to zero porosity, the activation energy for sintering was comparable to the activation energy of viscous flow. When the apparent activation energy for the sintering process was lower than that of viscous flow, sintering was found to proceed at a slower rate because the precipitated crystalline phase retarded viscous flow and the shrinkage of the pores. Initial crystallization appeared to occur at the surfaces of particles in contact. A chemical treatment was partially successful in suppressing the onset of surface nucleation of the precursor powders.

Conradt (1994) studied the thermodynamic approach to viscosity in the glass transition. The glass transition is re-investigated by means of the formalism of linear nonequilibrium thermodynamics. The process is treated for melts cooled down below their liquidus temperatures in terms of three

events. These events are, in the order of decreasing temperature: by-passing of crystallization, freezing-in of stress relaxation, and freezing-in of near-range structural relaxation. Conditions for the viscosity levels are derived at which each of these events is accomplished. The model is tested against data of five one - component, three simple binary and ternary, and three multicomponent systems. Within the scope of this test, it is confirmed that the model correctly describes the viscosity levels typical of the glass transition, as well as their dependence on the cooling rate. An explanation for the 10^{13} dPas rule at T_g is included.



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