THERMAL FORMING PROCESS FOR PRECISION FREEFORM OPTICAL MIRRORS AND MICRO GLASS OPTICS

DISSERTATION

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By

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Glass thermal forming processes are an emerging industrial techniques that can be adopted for high volume manufacturing of common size spherical, aspherical, and freeform glass optics, as well as micro glass optical components. Thermal forming processes discussed in this dissertation include compression molding and thermal slumping. The thermal forming processes are net shape, environment friendly and high volume production manufacturing techniques. However, there are still quite a few technical challenges associated with these new processes which include proper curvature compensation, mold design and mold life issues, residual stresses in the molded lenses, and refractive index variation after molding. These difficulties must be overcome before glass thermal forming processes can be readily implemented in industry.

This dissertation research seeks a fundamental understanding of the thermal forming process for both freeform glass mirrors and glass micro optical lenses by adopting a combined experimental, analytical and numerical Finite Element Method (FEM) approach. Preliminary investigation was conducted on the optical design for beam shaping reflector and freeform two-stage solar concentrator. The freeform primary mirrors were used as thermal slumping test samples. Thermal slumping experiments were performed to determine the effects of different molding parameters i.e. the slumping
temperature, holding time and cooling rate on the final thermal formed glass mirrors’ quality. The surface roughness and contour error were evaluated based on the requirement of freeform solar concentrator. The manufacturing tolerance analysis of the freeform solar concentrator system was also performed. Numerical modeling was utilized to compensate the curvature deviation during a thermal forming process, and evaluated using experimental results with matching process conditions. Moreover, in compression molding of precision glass lens experiments were also performed to study the residual stresses under different cooling rate. An FEM simulation model was developed and predictions were compared with the actual experimental results. Based on the comparison, FEM simulation can be used to predict and optimize cooling rate in the thermal forming process.

Finally, compression molding experiments were performed to fabricate glass microlens arrays and diffractive optical elements (DOEs). The molded glass micro optical lenses were measured with AFM/SEM, and the optical performance of the molded lens was also evaluated by using a home-built optical metrology setup. Experimental results have showed that the thermal forming processes are capable of producing precision freeform glass mirrors and glass micro optics with shape and surface quality within the tolerance requirement.
DEDICATION

Dedicated to my beloved wife Wei Wang
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I would like to express my gratitude to my adviser, Prof. Allen Yi, for providing me with an opportunity to work under him and exposing me to a very exciting and futuristic field of precision optical engineering. I would like to thank Prof. Yi for his trust, guidance, enthusiasm and insight during my research. I also appreciate the suggestions, assistance and comments of other faculty members at OSU whom I had the opportunities to work with during the course of this research: Prof. Jose Castro, Prof. Blaine Lilly, Prof. James Lee and Prof. Rebecca Dupaix.

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CHAPTER 1: INTRODUCTION AND THE STATE OF THE ART

1.1 Thermal Formal Process

Glass thermal forming processes are an emerging industrial techniques that can be adopted for high volume manufacture of common size spherical, aspherical, and freeform optics, as well as micro glass optical components. Thermal forming processes discussed in this dissertation include compression molding process and thermal slumping process.

1.1.1 Precision Compression Molding of Aspherical Glass Lenses

Compression molding of precision glass lenses (glass molding thereafter) is one of the thermal forming techniques that can be adopted for high volume precision glass optical elements fabrication [Maschmeyer, 1983]. Precision glass molding was first developed by Kodak to fabricate both spherical and aspheric lenses used in various photographic systems [Pollicove, 1988]. Glass molding process is a hot forming method in which a heated raw glass gob or blank is pressed by optically polished molds to create the finished lens shape or designed surface with micro pattern. Controlled cooling (or annealing) of the molded glass component is carried out immediately after pressing to maintain a short production cycle while keeping the stress and thermal shrinkage level below a required value after the molding process. The molded glass lens is eventually
released at a temperature close to room temperature, as shown in figure 1.1. As compared to conventional glass fabrication technique (i.e., grinding, polishing, and lapping), glass compression molding is an environmentally conscious process since it is a near net-shape process and the use of polishing and grinding fluids needed for conventional method is also eliminated. Furthermore, compression molding process is a one-step fabrication process as compared with the more complicated traditional method, as shown in figure 1.2. Figure 1.3 shows the recorded temperature, load and position changes in a typical glass compression molding process, as described before.

![Figure 1.1 Schematic illustration of the glass compression molding machine and a complete molding cycle [Jain, 2006].](image)

In the past years, some work has been done to investigate the precision glass compression molding process and its application. Glass compression molding can be used to fabricate aspherical glass lenses and freeform glass lenses which can be used in different precision optical application [Yi, 2005; Yi, 2006b; Vogt, 2007]. Furthermore, compression molding process can also be used to fabricate microlens array and diffractive optical
elements that can find application in optical data storage, optical communication and
digital displays [Firestone, 2005b; Yi, 2006; Chen, 2008]. Although glass molding is a
promising new optical manufacturing process, there are still quite a few technical
challenges involved in this new process which include curvature compensation, mold
design and mold life, residual stresses inside of the molded lenses, and refractive index
deviation after molding process. These difficulties must be overcome before glass
molding process can be readily implemented in industry.

![Figure 1.2 Conventional abrasive based glass optical lens manufacturing process](image)

![Figure 1.3 Schematic diagram of a process sheet showing the variation of temperature
and mold position during a complete molding cycle](image)
1.1.2 Thermal Slumping of Glass Mirror

Glass thermal slumping was initially developed to produce the Schmidt corrector plates and then was applied to fabricate ophthalmic optical components [Smith, 1988]. Glass thermal slumping utilizes similar process conditions as compression molding, *e.g.* heating, molding, and cooling. The only difference is on the pressing force in the molding stage. In glass thermal slumping process, the raw glass sheet workpiece and mold are heated up to the working temperature (or soaking temperature) and then slumped by its own weight or (negative) vacuum pressure. Controlled cooling of the slumped glass mirror is carried out immediately after slumping is completed to keep the thermal shrinkage and residual stresses below required levels after the molding process. As compared to conventional abrasive based process, thermal slumping is also a high volume, low cost and one-step fabrication process. More recently, glass thermal slumping process has even been used to fabricate the segment of X-ray telescope mirrors and other extremely high precision glass optics [Jimenez-Garate, 2003; Zhang, 2003]. Based on these successful applications, glass thermal slumping process is considered to be a promising new method for fabricating precision optical components at an affordable cost.

The characteristics of low cost and high volume make the glass thermal slumping very suitable for fabricating optical components for solar energy system. Solar power is a clean alternative renewable energy to fossil fuel generated electricity. Solar power system can produce electricity without creating harmful emission during operation [Stine, 1985]. A solar energy system normally uses two typical designs, *e.g.* non-concentrating flat plates, and concentrating solar thermal or photovoltaic. Concentrating collectors could
reduce the area of the receiver by reflecting or refracting the light incident on a large area onto a small absorber area. The economics of photovoltaic conversion can improve significantly with intermediate levels of concentration. At high flux levels, efficient generation of electricity is technically feasible. Therefore, there is general agreement that some degree of concentration will be desirable for most applications [Luque, 2006]. However, the cost and complexity associated with optical concentration outweigh its advantages. Therefore, the glass thermal slumping process will be a potential method to fabricate solar optical components with an affordable cost.

1.2 Glass Rheology

A glass can be defined as “an amorphous solid completely lacking in long range, periodic atomic structure, and exhibiting a region of glass transformation behavior” [Shelby, 2005]. Glass plays an essential role in the optical system due to its great transparency, stability, and resistance at extreme conditions. The workability and parameters of glass around glass transition temperature is an important topic because of its relevance to the thermal forming process.

1.2.1 Viscosity

The viscosity of glass is a strong temperature dependent property, which determines the glass processing conditions in thermal forming such as: melting condition, working condition, and annealing condition [Shelby, 2005]. There are several different models to describe the temperature dependence of viscosity. In these models, Arrhenius equation
and Vogel-Fulcher-Tamman (VFT) equation are two of most commonly used. The Arrhenius equation is given by [Scherer, 1986]:

\[ \eta = \eta_0 \exp \left( \frac{\Delta H}{RT} \right) \]  

(1.1)

Where \( \eta_0 \) is a constant, \( \Delta H \) is the activation energy for viscous flow, \( R \) is a gas constant and \( T \) is the working temperature. Arrhenius equation can provide a good fit in the transition temperature range. Arrhenius equation is widely used to calculate glass structural relaxation in the cooling stage. The VFT equation can provide better fit when temperature is higher than the glass transition range, which can be expressed as [Fulcher, 1925]:

\[ \log \eta = -A + \frac{B}{T - T_0} \]  

(1.2)

where \( A, B \) and \( T_0 \) are the fitting constant with the value of \( T_0 \) being considerably lower than the \( T_g \) of the given glass.

**Figure 1.4** Calculated viscosity vs. Temperature curve for soda-lime glass and several important temperature points.
Figure 1.4 shows the viscosity curve of a commercial soda-lime glass curve fitted from VFT equation whose parameters come from the glass vendor [Howard, 2008]. In this figure, there are a series of reference points that characterize the viscosity temperature curve. The working point of glass is the temperature with a viscosity of $10^3$ Pa-s. At this temperature, glass can be formed or sealed for glass bottle or container. At the viscosity of $10^{6.6}$ Pa-s, this temperature is called softening point of glass. The viscosity corresponding to glass transition temperature ($T_g$) for common glasses has an average value $10^{11.3}$ Pa-s. The annealing point is a temperature at the viscosity of $10^{12.4}$ Pa-s, at which the glass is soft enough for the stresses relaxation within several minutes or smaller. The strain point is a temperature at which the viscosity of glass is $10^{13.6}$ Pa-s. Typically, in glass thermal slumping process, the viscosities are in order of $10^9$~$10^{10}$ Pa-s. In addition, for precision compression molding process, the viscosities are in order of $10^7$ Pa-s.

### 1.2.2 Glass Transition Region

Glass transition is a region of temperature in which molecular rearrangements occur on a scale of minutes or hours, so that the properties of glass change at a rate that is easily observed. Glass transition temperature ($T_g$) is a point in the center of the transition region. There is no very clear definition for glass transition temperature. Normally, glass transition temperature is seen as a temperature in glass transition region defined by changes in either thermal analysis curves or thermal expansion curves. The transition temperature of a glass material is determined by the chemistry composite of this glass. Different glass materials may have different transition temperatures. For a given glass material, the transition temperature is determined by the different cooling rates and
different measurement methods. When the glass material is cooled at higher cooling rate, it has higher glass transition temperature. Higher cooling rate causes departure from equilibrium at higher temperature, and then cause higher transition temperature. These material properties of glass show a sudden change at the glass transition temperature. Therefore, we can get the glass transition temperature by measuring these properties during a heating and cooling process. Two most widely used methods to determine glass transition temperature are measuring heat capacity and thermal expansion. Normally, the glass transition temperature is determined by the cooling rate of 10K/min.

**Figure 1.5** The strain $\varepsilon(t)$ change of glass when a constant uniaxial stress $\sigma_0$ at $t_0$

In the glass transition region, if a mechanical stress is applied to glass material, a time-dependent change in dimensions results occurs, as shown in figure 1.5. This process is called stress relaxation. When a constant uniaxial stress $\sigma_0$ is applied, the strain responds
in three components: an instantaneous elastic strain $\varepsilon = \sigma_0/E$, a delay elastic strain and viscous flow at the rate $\sigma_0/3\eta$. This behavior can be described by using viscoelastic model or simplified Newtonian fluid.

![Figure 1.6](image)

**Figure 1.6** The property change when a sudden change in temperature in glass transition region

In the glass transition region, if the glass is subjected to a sudden change in temperature, a time-dependent change in property occurs, as shown in figure 1.6. This process is called structural relaxation. There are several different methods to model the structural relaxation of glass in cooling process. One of the most widely used is Tool-Narayanaswamy model [Tool, 1948; Narayanaswamy, 1971].
1.2.3 Viscoelastic Model

At the thermal forming temperature, the glass material shows a strong viscoelastic behavior and thermally or externally applied stresses in glass will relax rather quickly. Viscoelastic behavior is a time dependent response of a material to either stress or strain. For a typical viscoelastic material, such as glass around the transition temperature, the application of a (constant) load will lead to material deformation, which consists of instantaneous deformation (elastic effect) and continuous deformation over time (viscoelastic), as shown in figure 1.5. The viscoelastic property will cause decay of the applied load and this decay is called stress relaxation. To describe the viscoelastic behavior, a generalized Maxwell model, as shown in Figure 1.7, can be used to model the stress relaxation of glass at transition temperature [Scherer, 1986]. The multi-Maxwell model consists of a series of springs with shear modulus $G_i$ and dashpots with viscosity $\eta_i$. The stress relaxation modulus $G(t)$ and the stress relaxation function $\psi(t)$ for this parallel model can be represented by:

$$ G(t) = 2G \sum_{i=1}^{n} w_i \exp(-t / \tau_i) \tag{1.3} $$

$$ \psi(t) = \frac{G(t)}{G(0)} = \sum_{i=1}^{n} w_i \exp(-t / \tau_i) \tag{1.4} $$

At time constant $t = 0$, $G(0) = 2G$. $\tau_i$ are the shear stress relaxation times, calculated by using equations $\eta_i/G_i$. $w_i$ are the corresponding weighting factors and related by:

$$ \sum_{i=1}^{n} w_i = 1 \tag{1.5} $$
The relaxation times $\tau_i$ and their corresponding weighting factors $w_i$ can be obtained by compression-relaxation test on the molding machine. Based on these equations, a viscoelastic counterpart of the elastic-thermal analysis equation is given [Soules, 1987]:

$$\Delta \sigma = D_E [\Delta \varepsilon - \Delta \varepsilon^{th}] - \sum_{i=1}^{n} (1 - e^{-\Delta t/\tau_i}) \sigma_i (t - \Delta t)$$

(1.6)

where $\sigma$ is stress tensor, $D_E$ is the elastic modulus matrix, $\varepsilon$ and $\varepsilon^{th}$ are the actual and thermal strain tensor respectively and $\Delta t$ is a small time step. The portion of the stresses that relaxed during the small time step and the change in the thermally induced stresses are converted to nodal forces and subtracted from the external forces in the model. Changes in displacement, strains and stresses can be calculated in the same way as in a simple elastic analysis. Besides Maxwell model, there are some another approaches that can be used to describe viscoelastic properties, such as, Voigt model, Burger model [Scherer, 1984].

### 1.2.4 Newtonian Fluid Model

Besides viscoelastic model, glass can also be considered as a Newtonian fluid under thermal forming conditions except at high stresses [Jain, 2006]. Since, the strain change
caused by elastic can be neglected if the total strain change is dominated by viscous flow. In that case, shear stress is proportional to the rate of deformation, which increases with the increase in molding velocity in the experiments. If the velocity of fluid flow varies directly with the applied shear force, the viscosity is independent of the force and the liquid is said to behave as a Newtonian fluid. And if the viscosity decreased at high shear stresses, this type was called non-Newtonian behavior, which is an important phenomenon in high shear rate forming processes. However, the stress level when non-Newtonian behavior in oxides glass occurs is so high that the phenomenon is rarely observed in most glass thermal forming processes. Since glass material at forming temperature can be considered as incompressible flow of a Newtonian fluid, the behavior of glass can be described using a simplified Navier-Stokes equation [Loch, 2002]:

\[ \rho \left( \frac{\partial \mathbf{u}}{\partial t} + \mathbf{u} \cdot \nabla \mathbf{u} \right) = -\nabla p + \nabla \cdot (\mu \nabla \mathbf{u}) + \mathbf{f} \]  

(1.7)

where \( \mathbf{f} \) represents the body force, \( \mu \) is the dynamic viscosity, \( \mathbf{u} \) is the velocity and \( \nabla p \) is the divergence of pressure. Under the assumption of incompressible fluid, density of glass is a constant and it follows the mass continuity equation as:

\[ \nabla \cdot \mathbf{u} = 0 \]  

(1.8)

Normally, in the thermal forming process, inertia part on the left side of equation can be neglected. In the thermal slumping process, the only body force of glass workpiece is the gravity. Therefore, the Navier-Stokes equation can be further simplified as:

\[ -\nabla p + \nabla \cdot (\mu \nabla \mathbf{u}) - \rho g \mathbf{k} = 0 \]  

(1.9)

The glass material can also be defined as a 3D Newtonian fluid by using rigid-viscoplastic model and the material behavior can then be described with equation 1.10
based on 3D Newtonian constitutive law where stresses $\tau_{ij}$ are a function of strain rate $\dot{\varepsilon}$ and viscosity $\eta$ related in the following equation:

$$\tau_{ij} = 2\eta(T)\dot{\varepsilon}_{ij} \quad (1.10)$$

1.2.5 Structural Relaxation

During cooling stage, glass material undergoes structural relaxation due to temperature change. Structural relaxation is a non-linear phenomenon since it not only depends on the current temperature but also on the history and direction of the temperature change, as shown in figure 1.6. Due to the structural relaxation properties, residual stresses will occur caused by non-uniform cooling of the glass workpiece. Residual stresses will result in an inhomogeneous refractive index and a curvature deviation, which both will deteriorate the optical performance of thermally formed glass components.

![Figure 1.8](image)

Figure 1.8 variation of specific volume during cooling of a glass-forming and a non-glass forming liquid [Scherer, 1986]
Structural relaxation refers to a time dependent response of glass material to a change in temperature. Figure 1.8 shows a plot of temperature dependent specific volume of a liquid being cooled at a given rate. In the liquid state the viscosity of glass is so low that the structure changes in step with temperature and the equilibrium structure is attained almost instantaneously. As the temperature is lowered further the viscosity of the liquid gradually increases along with the time required to attain a new equilibrium configuration. This results in the deviation of the cooling curve from the equilibrium line until the structure is finally frozen in a fixed configuration. This state is called the glass state since it possesses the rigidity of a solid but has a liquid like internal structure. During the cooling stage, because of the heat transfer inside the glass blank, the structural change of the surface will precede that of the interior. Thus temporary differences must exist between the specific volumes of layers of different parts of glass components. This difference in specific volumes produces strains and hence stresses. Finally, because these different parts reached the structure equilibrium in sequence, the stresses were frozen into glass component. The surface cools down more rapidly than the inside of the glass. Then the surface contracts more rapidly than the core of the plate. The mid-plane is in compression and by equilibrium, the surface is in tension.

Again during cooling, a response function $M_v(t)$ can be used to describe the change of a glass property in the transition region due to temperature drop:

$$M_v(t) = \frac{p(t) - p_2(\infty)}{p_2(0) - p_2(\infty)} = \frac{T_1(t) - T_2}{T_1 - T_2}$$

(1.11)
This response function represents the fraction of the property change that has yet occurred. The subscripts 0 and ∞ represent the instantaneous and steady state values of property $p$ of the glass material. The response function can also be described using an exponential function:

$$M_v(t) = \exp \left[ -\left( \frac{t}{\tau_v} \right)^b \right]$$ (1.12)

where, $\tau_v$ is called the structural relaxation time, and $b$ is a phenomenological parameter that was adopted to fit the response curve. The value of $b$ lies between 0 and 1.

Alternatively in the finite element method application, the experimental formula shown in equation 1.12 is also used to fit the experimental data:

$$M_v(t) = \sum_{i=1}^{n} \left( w_g \right)_i \exp \left( -\frac{t}{\tau_{vi}} \right)$$ (1.13)

where, $(w_g)_i$ are called weighting factors and $\tau_{vi}$ are the associated structural relaxation times. The structural relaxation times are strongly temperature dependent and can be calculated using the Narayanaswamy model expressed in equation 1.14 for a given temperature [Narayanaswamy, 1973]:

$$\tau_v = \tau_{v,ref} \exp \left( \frac{H}{R} \left[ \frac{1}{T_{ref}} - \frac{x}{T} - \frac{(1-x)}{T_f} \right] \right)$$ (1.14)

where $\tau_{v,ref}$ is structural relaxation time at reference temperature $T_{ref}$, which is known. $H$ is an activation energy and $R$ is ideal gas constant. $T_f$ is fictive temperature, and $x$ is a fraction parameter with value between 0 and 1. The fictive temperature $T_f$ can be obtained from equation 1.11 by using Boltzmann superposition principle and integrating over the
thermal history of the sample by equation 1.15 or using an efficient algorithm provided by Markovsky and Soules [Soules, 1984]:

\[
T_f(t) = T(t) - \int_0^t M_p[\xi(t) - \xi(t')] \frac{dT(t')}{dt'} dt'
\] (1.15)

Once the fictive temperature \(T_f\) is derived, the property of glass at a given time can be calculated using the following equation:

\[
\frac{1}{p(0)} \frac{dp(t)}{dp} = \alpha_l(T) + [\alpha_l(T_f) - \alpha_s(T_f)] \left(\frac{dT_f}{dT}\right)
\] (1.16)

where \(\alpha_l\) and \(\alpha_s\) are thermal expansion coefficients of liquid and solid glass material respectively. Then the linear thermal strain \(\varepsilon_{th}\) is given equation 1.17 for stress analysis during cooling stage in a FEM model.

\[
\varepsilon_{th} = \frac{1}{3} \frac{\Delta V}{V(0)}
\] (1.17)

Where, \(\Delta V\) is the specific volume change due to the temperature change in transition range, which can be calculated from equation 1.16. From these equations, the residual stresses frozen inside the glass sample when it gets through the transition range due to structural relaxation could be calculated by these governing equations and equation 1.6.

1.3 Numerical Simulation of Glass Thermal Forming Process

With the progresses made in numerical simulation capabilities and computing technology in recent years, finite element methods (FEMs) have been utilized extensively to assist in fabrication process analysis and optimization. Some of the major issues related to modeling glass thermal forming include: large free surface deformation, complicate
contact problem, non-linear material properties, and heat transfer between glass workpiece and molds. Due to these issues, glass molding at glass transition temperature ($T_g$) involves viscoelastic effects and structural relaxation, and thus modeling of material behavior around $T_g$ can be quite complicated [Scherer, 1986; Loch, 2002].

Cesar de Sa used an in-house FEM code to simulate the beverage container forming process [Cesar de Sa, 1986]. He used the Newtonian fluid model to simulate the glass molten process and the appropriate non-linear thermal boundary condition and contact were also taken in account. Hyre used the FEM program POLYFLOW for the numerical simulation of different stages of glass container manufacturing process [Hyre, 2002]. He modeled the glass material as an incompressible fluid, and also included the effects of viscoelasticity, non-Newtonian behavior, surface tension, and time-varying heat transfer. Druma et al demonstrated that the capability of FEM software DEFORM in glass forming simulation [Druma, 2004]. They simulated the pressing and cooling process of TV panel forming by coupling temperature simulation and structural calculation. Tsai et al used an elasto-viscoplastic model for glass material to research the deformation behavior at the thermal forming temperature [Tsai, 2008]. Zhou et al tried to simulate the behavior of glass material at forming temperature by using several different viscoelastic models and compared with experiment results [Zhou, 2009].

Soules et al used an FEM program MARC to study the stresses inside glass components under different molding conditions in case of a simple sandwich seal and a bead seal [Soules, 1987]. Stresses were generated inside glass workpiece due to the thermal expansion mismatch between different materials. The residual stresses inside tempered
glass were studied by using structural relaxation theory. A good agreement between the predicted stresses and the experimental results was obtained. Carre and Daudeville simulated the residual stresses and transition stresses in tempering soda-lime glass plate by using FEM software MAS/MARC [Carre, 1996]. The viscoelastic behavior and structural relaxation of glass during cooling process were considered into the model by using Narayanaswamy model. The numerical simulation results were in good agreement with their experimental results. Dang et al calculated the residual stresses during annealing process of glass bottles according to Narayanaswamy model by using FEM code ANSYS [Dang, 2005]. The simulation results were used to find the critical cooling zone and optimize cooling stage. Na et al also investigated the residual stresses and birefringence distribution by using a commercial FEM program ABAQUS [Na, 2007].

Wang et al investigated the curvature compensation in glass compression molding process with an iterative algorithm by using FEM software ANSYS [Wang, 2008]. They used multi-Maxwell elements to simulate the stress relaxation of viscoelastic model. After the curvature compensation by numerical simulation, the final curvature deviation from original design was less 2μm compared with 12μm before compensation. Sellier et al also developed an iterative algorithm to optimize mold design with FEM program ABAQUS by simulating glass molding process [Sellier, 2007].

Gaylord et al measured the parameters of glass material used in the numerical simulation of structural relaxation phenomenon including viscosity, friction coefficient, and structural relaxation parameters, and then established an FEM model to predict the final shape of molded glass lens [Gaylord, 2008]. Yan et al measured the glass viscosity near
the softening point and simulated the behavior by using FEM software DEFORM™-3D [Yan, 2009].

Tuck and Stokes et al developed the numerical simulation strategy for glass thermal slumping process [Tuck, 1997, Stokes, 2000; Agnon, 2005]. Hunt also studied the thin viscous sheets under gravity sagging by using numerical simulation [Hunt, 2002]. They used the simplified Navier-Stokes equation and in-house FEM code to describe the glass behavior at slumping temperature, and compensate the curvature deviation between upper free surface and mold surface.

In recently, our group also used the FEM program MSC/MARC to research the glass lens deformation, glass viscosity, and index deviation in the glass molding process [Yi, 2005; Jain, 2005; Jain, 2005b; Jain, 2006; Jain, 2006b; Jain, 2006c; Su, 2008; Zhao, 2009; Zhao, 2009b]. Jain used the MSC/MARC and DEFORM™-3D to simulate the precision compression molding process by using Newtonian fluid model in molding stage and Narayanaswamy model in cooling stage. He also measured the viscosity and elastic modulus of the molded glass material and compared the experimental results with numerical simulation. Su and Zhao simulated the refractive index deviation inside the molded glass lens and compared the simulation with measured results.
CHAPTER 2: RESEARCH OBJECTIVES

Glass thermal forming offers a promising approach for high volume, low cost glass optical production from freeform optical mirror to micro glass optics, which are difficult to make using conventional abrasive fabrication techniques. However, there are still quite a few technical challenges involved in the new process which include thermal expansion of the molds, mold life, optical components curve shift and residual stresses inside of the formed glass optics. These difficulties must be overcome before glass thermal forming process can be readily implemented in industry.

This dissertation research is mainly focused on determining the feasibility and performance of glass compression molding and thermal slumping for production of precision freeform mirrors and micro optics. The study involves extensive experiments and FEM simulation is used as a tool to develop a fundamental understanding of the process. Customized experiments will also be designed and conducted to measure residual stresses frozen in glass optical components in thermal forming process and curvature deviation after thermal treatment. Specifically, the research on these topics enhances our understanding of the cooling process and also provides a methodology for optical manufacturers to identify and optimize their process capabilities at a minimal cost. Diffractive optical elements and microlens arrays will be fabricated with precision.
compression molding process. The different molding parameters will be compared to evaluate their effect on glass diffractive optics molding process. Furthermore, a thermal reflow method will be introduced to fabricate glass microlens array. The goals of this part of the research are to use the standard lithography techniques to produce molds for glass diffractive optics and microlens arrays in order to identify proper manufacturing conditions.

The specific objectives of the proposed research are to:

i. Perform an optical design for freeform beam shaping reflector and freeform two-stage concentrator, and perform ray-tracing simulation to evaluate the designed optical components. Develop an iterative algorithm for freeform two-stage concentrator design. (Chapter 3)
ii. Study and optimize glass thermal slumping conditions to find a high volume, low cost fabrication process for the designed freeform concentrating mirrors. Measure the surface geometrical curvature and surface roughness to optimize the thermal slumping process. (Chapter 4)

iii. Implement a numerical simulation model to predict curvature change of glass mirror after thermal forming process. Different model will be compared. The results will be used to compensate the curvature deviation. (Chapter 5)

iv. Perform an FEM modeling of a simple cylindrical compression and cooling test and use the measured glass molding parameters as an input to numerical simulation and to compare the experimental measurement and predicted residual stresses in the simple glass cylinders at different cooling rates. The results will be used for curvature compensation of thermal slumping process of freeform glass mirrors. (Chapter 6)

v. Characterize the manufacturing error and surface finishing of slumped freeform glass mirror at different slumping parameters and molding conditions, and minimize the manufacturing error and the surface roughness to satisfy the designed tolerances requirement. (Chapter 7)

vi. Develop a method to fabricate glass diffractive optical elements and microlens arrays using glass precision compression molding process with glassy carbon, quartz glass, or fused silica as the mold materials. Perform optical measurement for fabricated glass precision optics. (Chapter 8)
vii. Investigate a real three-dimensional mold fabrication method based on combination of traditional lithography-etching technique and ultra-precision diamond machining process. The mold with real three-dimensional microstructure will be used for precision glass molding of glass diffractive optical elements. (Appendix A)

The relation of these research objectives is shown in figure 2.1. In summary, the main purpose of this dissertation research is to find a completed methodology of thermal forming process for freeform optics and micro optics, from optical design to thermal forming process, numerical simulation and manufacturing tolerance analysis.
CHAPTER 3: FREEFORM OPTICAL DESIGN FOR CONCENTRATING OPTICS

3.1 Freeform optical design for beam shaping

3.1.1 Theory calculation

In many engineering problems, traditional rotational spherical or aspherical optical surfaces are not sufficient to satisfy the requirement of reshaping and re-direction, such as beam shaping, illumination, and solar concentrating. In these cases, the purpose of optical components is not to image the object but to produce a prescribed irradiance distribution on the receiver or target screen. Such an optical system is called non-imaging optical system, which has been widely used in illumination and concentrating system. A freeform optical surface is a non-rotationally symmetric surface or a symmetrical surface that is rotated about any axis that is not its axis of symmetry. Freeform surface has started being often used in imaging and nonimaging optical design [Yi, 2006b; Michaelis, 2008]. The purpose of freeform optical design in this dissertation is for redirecting and reshaping the irradiance distribution from the light source over a pre-described target by using glass mirror, which can be fabricated using thermal slumping process, one of the thermal forming processes.
3.1.1.1 Refraction and reflection in vector form

To describe ray travelling in an optical system more efficiently, vector form is often used in the geometrical approach for freeform optical system design. When the incident ray is travelling in a direction, that direction can be defined by unit vector $\mathbf{i}$, and the reflected/refracted ray direction can be defined as vector $\mathbf{r}$ ($\mathbf{r}_x$, $\mathbf{r}_R$ respectively), as shown in figure 3.1. The direction of the ray travelling is related to the normal direction ($\mathbf{n}$) of surface and refractive index, described by Snell’s law:

$$n_1 \sin \alpha_1 = n_2 \sin \alpha_2 \quad (3.1)$$

where, $n_1$, $n_2$ are the refractive index of the medium of incident light and reflective (refractive) light space, respectively. In addition, $\alpha_1$ and $\alpha_2$ are incident angle and reflective (refractive) angle respectively. In a vector form, the Snell’s law can be expressed as:

$$n_1 \mathbf{i} \times \mathbf{n} = n_2 \mathbf{r} \times \mathbf{n} \quad (3.2)$$

![Figure 3.1 Snell’s Law geometry for light incident on a medium of higher refractive index](image)

25
From this equation, the direction of the refracted or reflected rays can be obtained by a linear combination of the incident direction $i$ and normal $n$ to the surface as:

$$n_2 r = n_1 i - (n_1 i \cdot n) n + (n_2 r \cdot n)n$$

(3.3)

In reflection case, $n_1 = n_2$, we can obtain $\alpha_1 = \alpha_2$, and this equation can be expressed as:

$$r = i - 2(i \cdot n)n$$

(3.4)

These two equations of vector rays’ direction are very useful for freeform optical design.

### 3.1.1.2 Aplanatic surface

Aplanatic surface is an optical surface in which the sum of the optical path to two fixed points in space has a constant value. This surface does not contribute spherical aberration to the image. The aplanatic surface is sometime used in nonimaging and freeform optics. Figure 3.2 shows three most often used aplanatic surfaces as reflector in the optical system design.

Parabola is one of the most common used aplanatic surfaces, which can transforms an incoming plane wave traveling along its axis into a spherical wave converging toward the focus, as shown in figure 3.2a. This property makes parabolic reflectors an ideal optic to collect and concentrate energy entering the reflector at a given angle to the focal point. In contrast with spherical reflectors, parabolic reflectors make no contribution in spherical aberration. However, parabolic reflectors still suffer severe off-axis aberration coma. In a polar coordinate system ($\rho, \varphi$), the profile of a parabola surface can be expressed as:

$$\rho = \frac{p}{1 - \cos \varphi}$$

(3.5)

where $p$ is the constant parameter to identify the parabola.
Ellipsoid is another aplanatic reflection surface, which can reflect a spherical wave coming from a focal point into another spherical wave converging toward the second (conjugate) focal point, as shown in figure 3.2b. Elliptical reflector is common used for illumination. In a polar coordinate system, the profile of an ellipse surface with one of the foci points at the origin point can be expressed as:

\[
\rho = \frac{\alpha(1 - \varepsilon^2)}{1 + \varepsilon \cos \varphi}
\]  

(3.6)

where \( \varepsilon \) is the eccentricity of the parameter and \( \alpha \) is the equatorial radius.

![Diagram of aplanatic surfaces](image)

**Figure 3.2** Three different aplanatic surface (a) Parabolic reflector, (b) Elliptical reflector, (c) Hyperbolic reflector

Hyperbolic surface can reflect the light rays coming from one of its focal points as if the rays are diverging from another focal point, as shown in figure 3.2c. Hyperbolic surface is widely used in telescope system, such as the secondary reflective mirror in a Cassegrain telescope. In a polar coordinate system that has its origin in a focus, the profile of the hyperbolic surface can be expressed as:

\[
\rho = \frac{\alpha(\varepsilon^2 - 1)}{1 + \varepsilon \cos \varphi}
\]  

(3.7)
Where $\varepsilon$ is the eccentricity of the parameter and $\alpha$ is the equatorial radii.

3.1.1.3 Geometrical Approach for Freeform Design

The purpose of this section is to describe a geometrical approach to design a freeform single reflector with a target in the near-field. This approach is based on the mathematical theory on the aplanatic surface and ray propagating in the vector form. This design approach does not use any rotational symmetry. Furthermore, this design will provide critical condition to avoid blockage between each reflective element.

Figure 3.3 The freeform reflective system in vector formulation

To start the freeform reflector design, we place a point source at the origin $O$, and define the flux pattern $J(m)$ as a function of direction $m$, which passes through the input aperture $D$ on the unit sphere centered at origin $O$. A reflective surface $R$ reflects all rays passing the input aperture $D$ to receiver surface $T$ following the Snell’s law. The reflective
surface can be expressed in a polar coordinate system as \( \rho(m) \) with \( m \in D \). The direction of reflected rays can be calculated by equation 3.4. Here, \( i \) is the direction of incident light along \( m \), and \( r \) is the direction of reflected ray. In the freeform optical reflector design problem, we provide the target map on the screen \( T \) on which the reflected rays must provide certain irradiance distribution, as shown in the vector form in figure 3.3. Obviously, according to the basic vector properties, the relationship between incident rays \( r(m) \), reflected rays \( u(m) \) and the target pattern vector \( v(m) \) can be expressed as:

\[
v(m) = r(m) + u(m)
\]  

(3.8)

**Figure 3.4** Geometrical layout for the freeform optical design for “OSU” logo

Furthermore, according to the conservation of energy, the input power inside the input aperture \( D \) will equal to the power received on the receiver screen \( T \) if neglecting the loss in reflection and scattering. Therefore, for any given incident light with irradiance map inside the input aperture \( D \) and target intensity distribution on the given screen \( T \), we can determine the reflective surface \( R \) by solving the equation 3.8.
In this section, we will describe an approach by using the reflective property of ellipsoid surface to build reflector. The target $T$ consists of a finite number of points. For example, the “OSU” logo shown in figure 3.4 includes 304 pixels. Our plan is to construct the freeform surface from a series of ellipsoid surface which have a common focus point at the origin $O$ and another one at the pre-described target $T$. Therefore, each point on the target $T$ corresponds a piece of the confocal ellipsoids of revolution defined as $E(v_i)$, shown in polar coordinate system as

$$\rho(m) = \frac{d}{1 - \epsilon m \cdot v} \quad (3.9)$$

In this equation, $d$ is the focal parameter determined by eccentricity $\epsilon$ and ellipsoid radii $a$. And $v$ is the vector directing to another focal point of the ellipsoid of revolution, which is also the pixel point on the target $T$ at the same time. In addition, the area of the reflector is determined by the input aperture $D$ and conservation of energy in equation 3.10 can be written as:

$$\int_D I(m)d\sigma(m) = \sum_{i=1}^{N} L_i \quad (3.10)$$

where $L_i$ equals to the output powers on the target screen required at points $v_i$, and $N$ is the total number of pixel on the target pattern. Therefore, for any pixel $v_i$ in the target plane, we have the energy balance equation as:

$$\int_{A_i} I(m)d\sigma(m) = L_i \quad (3.11)$$

where $A_i$ is the area of a piece of ellipsoid on reflector surface ($E_i$) corresponding to a segment of input aperture. This equation combined with equation 3.9 is the basic
conditions to build the freeform reflector surface. To make sure each ellipsoid of revolution $E_i$ established by focal parameter $d_i$ will contain target $T$ strictly inside, a constraint of all focal parameters must be satisfied as shown in equation 3.12 [Oliker, 2006; Oliker, 2006b]:

$$d_i \geq 2\omega$$

(3.12)

where $\omega$ is the maximal distance between any two points in target $T$ region.

To solve this problem, an iterative procedure is proposed and realized by using MATLAB program. For any given target pattern, we start the process with an initial ellipsoid reflector $E_1$ with the focal parameter $d_1 = \alpha M$. In this equation, $M$ is the maximal distance between origin point $O$ and the any pixel point in the target plane, and $\alpha$ is a positive number used to avoid self-blockage. When the $\alpha$ is larger enough, as shown in equation 3.13, no self-blockage can occur in the reflective system [Oliker, 2006; Oliker, 2006b].

$$\alpha \geq \frac{4\omega}{M(1 - \gamma)}$$

(3.13)

where $\gamma$ is the maximal angle between the vector from origin $O$ to target pixel and incident light rays. Once the focal parameter is determined, the eccentricity of the ellipsoid can be calculated from the basic properties of ellipsoid as:

$$\varepsilon = \sqrt{1 + \frac{d^2}{|v|^2} - \frac{d}{|v|}}$$

(3.14)

With the $d_i$ and $\varepsilon_i$, an initial reflector $E_i$ can be established. Right now, all light rays passing from the input aperture will be reflected by the reflector $E_i$ to point $v_i$. After that,
we begin to modify this initial reflector by changing other ellipsoid reflector. By decreasing the focal parameter $d_2$ of the ellipsoid $E_2$, part of $E_1$ surface will be blocked by the new ellipsoid. Continue reducing the $d_2$ until the area of $E_2$ satisfies the energy balance in equation 3.11. To find the focal parameter $d_i$ more accurate and efficient, a bisection method is used. Then, the same procedure is repeated for each of the remaining ellipsoids. By using this iterative algorithm, a freeform reflector including a series of ellipsoid segments can be established. A program written in MATLAB was used to complete the calculation.

To verify the design algorithm, two examples will be presented here. The schematically geometrical layout of the first example is shown in figure 3.4. The target pattern is an “OSU” logo, with 304 bright pixels under uniform flux distribution on each pixel of the pattern, as shown in figure 3.5a. The dimension of the freeform reflector is controlled by the input aperture, which is a circle with $5^\circ$ acceptance half angle. The distance between the light source and screen is defined as 300mm. The size of the screen is 50x50mm. The angle between the central incident ray ($m_i$) and the central screen vector ($v_i$) is 135°. Since there are total 304 pixels on the target, 304 pieces of ellipsoids of revolution will be established. For convenience of fabrication in next step, each ellipsoid part will be meshed into 100x100 small pieces with dimension less than 20x20μm. The coordinates of these small pieces will be used as machine tool path in future fabrication process and reflector piece coordinates in ray-tracing simulation. The program run-time is about 40min on a PC computer. The computation result of the freeform reflector is shown in figure 3.5b.
Figure 3.5 Freeform reflective mirror for an “OSU” logo pattern (a) the target pattern (b) the freeform mirror surface profile

The second example has the similar geometrical scheme as previous example. The target pattern is a President Lincoln’s portrait with 4,422 bright pixels under uniform irradiance distribution of each pixel on the pattern, as shown in figure 3.6a. The distance between the light source and screen is defined as 300mm. The size of the screen is 80x60mm. There are 4,422 pixels on the target, and 4,422 pieces of ellipsoids of revolution will be
established. Each ellipsoid part will be meshed into 30x30 small pieces with dimension less than 15x15μm. The program run-time is about 16 hours on a PC computer. The computation result of the freeform reflector is shown in figure 3.6b.

![Figure 3.6](image)

**Figure 3.6** Freeform reflective mirror for a Lincoln portrait (a) the target pattern (b) the freeform mirror surface profile
3.1.2 ZEMAX Ray Tracing Simulation

To evaluate the result of freeform optical design, non-sequential ray tracing is applied to trace the propagation of light rays after freeform reflector. Ray tracing is a method widely used in the optical design to describe the propagation of light rays through the optical system. Rays are random generated from the given light source and are transmitting to a random direction constrained by required flux distribution map. After reflection and/or refraction inside the optical system, the random rays will reach the final detector surface. If the number of rays is large enough, the radiation pattern on the surface of receiver can be used to evaluate the system performance. In the beam shaping or illumination design, ray tracing is a useful tool to verify the final irradiance distribution on target detector.

![Ray tracing result of the freeform optical reflector for “OSU” logo by using ZEMAX](image)

**Figure 3.7** Ray tracing result of the freeform optical reflector for “OSU” logo by using ZEMAX

Now most commercial optical design softwares such as Code-V, ZEMAX can perform ray tracing analysis. ZEMAX is used in this research. ZEMAX is commercial optical
design software that can be used for lens design, illumination, beam propagation, freeform optical design and many other applications [ZEMAX, 2009]. To build the freeform surface in the ZEMAX non-sequential mode, polygon object was used in this research. Polygon object is an efficient way for creating flexible user-defined objects in ZEMAX, which is composed of a collection of quadrangles whose vertices are imported from the calculated results. Any four neighboring points will form a small quadrangle part in the polygon object, which is defined as the reflective property. The file format can be created directly from MATLAB program into an ASCII textile file.

Figure 3.8 Ray tracing result of the freeform optical reflector for a Lincoln portrait by using ZEMAX

For the OSU logo freeform mirror, there are total 1900x1600 points in the meshed surface, and total 1899x1599 quadrangles defined for polygon object. For Lincoln
portrait freeform mirror, there are total 1980x2010 points in the meshed surface. Therefore, there are total 1979x2009 quadrangles defined for polygon object. Light source, detector screen, and freeform mirror are placed on the exact designed position. Total 100,000 rays are traced to simulate the optical system. Figure 3.7 shows the ray tracing result of the OSU logo freeform mirror. Figure 3.8 shows the ray tracing result of the Lincoln portrait freeform mirror. As shown in these results, the designed freeform mirror can realize the function of reshaping the light irradiance distribution on the target screen.

3.1.3 Fabrication and Optical Measurement

To demonstrate the optical design and ray-tracing simulation results, the freeform mirror for the OSU logo is manufactured. In our experiment, the freeform optical mirror was fabricated directly on an aluminum block surface by using the diamond turning process which was carried out on the Moore 350FG Ultraprecision Freeform Generator. This ultraprecision machine has been used to fabricate optical components directly with the optical surface quality [Li, 2006; Yi, 2005b]. The 350FG ultraprecision machine has three linear axes that are equipped with linear laserscales capable of resolving 8.6 nm while moving at a maximum speed of 1,800 mm/minute. The straightness on all slides is less than 250 nm. The work spindle is capable of reaching 6,000 rpm while maintaining axial and radial motion error of less than 25 nm. The work spindle can also maintain angular position to less than 5 arcs second in a modulated mode. The radius of machine tool is about 200 μm. The movement step of the machine tool is determined from the designed position matrix. For OSU logo freeform mirror, the step is less than 20μm and
for Lincoln portrait mirror, the step is less than 15\(\mu\text{m}\). Figure 3.9 shows the fabricated OSU logo freeform mirror on an aluminum block. After manufacture, the fabricated freeform mirror is measured by a coordinate measurement machine (CMM) and compared with the design shape, as shown in figure 3.10.

![Aluminum freeform mirror fabricated by diamond turning process](image)

**Figure 3.9** Aluminum freeform mirror fabricated by diamond turning process

After CMM measurement, an optical experiment was performed for the fabricated freeform mirror. The optical test setup was as same as the layout shown in figure 3.4. A 632nm He-Ne laser after 25 \(\mu\text{m}\) pinhole was used as the point light source with uniform output irradiance distribution at any direction. The result was projected to black screen with 50x50mm dimension. Figure 3.11 shows the result of illumination experiment. The distortion in measured image may come from the non-flatness of the screen and the fabrication error on the aluminum mirror.
3.2 Freeform optical design for concentrating photovoltaic

3.2.1 Basic concept for CPV optical design

One of the most important applications of freeform optics is the concentrating optical system for solar photovoltaic. Solar radiation incident on the earth is the primary energy
source for people’s life. Furthermore, solar energy is a clean alternative renewable energy to fossil fuel generated electricity. Solar energy system can produce electricity without creating harmful emission during operation [Stine, 1985]. Generally, we can assume the sun to be a spherical black body emitter [Winter, 1991]. Its radiant flux $\Phi_s$ can be expressed as:

$$\Phi_s = 4\pi R_s^2 \sigma T_s^4$$

(3.15)

Where $R_s$ is the sun’s radius, $T_s$ is its surface temperature and $\sigma$ is the Stefan-Boltzmann constant. By consequence of energy conservation, this flux passes through any external spherical surface concentric to the sun. Since radiation spreads out as the distance squared, by the time it travels to the earth, the radiant energy falling on the surface area can be calculated. Furthermore, according to the sun-earth geometry as presented in figure 3.12, the apparent angular size of the solar disc can be calculated from the diameter of the sun photosphere and the earth-sun distance. In general, the half apparent angle is 0.26° when the $D_{ES} = 1.496\times10^{11}$m and $R_s = 6.96\times10^8$m. Besides, in this condition, the flux density observed on earth surface is about 1,367 W/m$^2$, calculated from equation 3.15.

The solar energy system normally uses two typical designs, e.g. non-concentrating flat plates, and concentrating solar thermal or photovoltaic. Concentrating collectors could reduce the area of the receiver by reflecting or refracting the light incident on a large area onto a small absorber area. The economics of photovoltaic conversion can improve significantly with intermediate levels of concentration. At high flux levels, efficient
generation of electricity is technically feasible. Therefore, there is a general agreement that some degree of concentration will be desirable for more application [Luque, 2006].

![Figure 3.12 Sun-earth geometry](image)

There are several different types of concentrating optics designed for high concentration system. Parabolic mirror and Fresnel lens as the primary concentrator are two major approaches to concentrating solar irradiant, but not limited to these. Ries et al developed a two stage high concentration system using a large aperture parabolic mirror as the primary and the kaleidoscope as the secondary to uniform the irradiation distribution [Ries, 1997]. Johnston et al investigated a method which used the spherical reflecting elements with a parabolic orientation on a space frame dish structure as the high flux solar concentrator [Johnston, 2003]. They compared the optical performance and manufacturing feasibility of collectors having such as combination of surfaces. Feuermann and Gordon initialized a high concentration photovoltaic design based on miniature parabolic dishes [Feuermann, 2001]. They used a collection of miniature
paraboloidal dish (with a diameter of the order of 10 cm) that concentrated sunlight into a short glass rod, which was used the homogenizer to uniform light distribution.

Leutz *et al* initialized a method that used a nonimaging Fresnel lens for high concentration solar system [Leutz, 1999]. Ryu *et al* proposed a new configuration of solar concentration optics utilizing modularly faceted Fresnel lenses to achieve a uniform intensity on the absorber plane with a moderate concentration ratio, which can reach around 100X concentration ratio [Ryu, 2006]. Gleckman summarized the commercial used rooftop photovoltaic system using a Fresnel lens as the primary lens and a glass pyramid as the secondary [Gleckmann, 2007]. He also analyzed the manufacturing problem for primary Fresnel lens and secondary glass rod.

Besides traditional parabolic and Fresnel primary concentrator, several high concentration systems based on nonimaging optics have been developed recently. Cvetkovic *et al* developed a XR (reflective (X) – refractive (R)) nonimaging photovoltaic concentrator, which could reach the geometrical concentration of 800X [Cvetković, 2007]. The XR concentrator is a nonimaging lens-mirror combination composed by a primary rotational symmetric aspheric mirror and by rotational symmetric aspheric secondary lens which encloses the solar cell. The XR system designed with a 3D Kohler integrating lenticulations could provide homogenization in the absorber surface. Diaz *et al* investigated a high concentration optical system based on total internal reflection (TIR) [Diaz, 2007]. The theoretical geometrical concentration ratio of this system could be 1264X. Benitez *et al* proposed a novel two mirror high concentration nonimaging optic that shares the advantages of two mirror aplanatic imaging concentrators [Benitez, 2006].
This concentrator used the Kohler integrating system to get the uniform light distribution. Alvarez et al studied a nonimaging RXI concentrator, which has an acceptance half angle 1.5° and geometrical concentration 1300x [Alvarez, 1998].

In all the concentrating systems, there are two basic concerns that we need to consider when we design an optical concentrator optical system for concentrating photovoltaic system. First, the illumination distribution on the surface of receiver should be uniform. This one comes from the requirement of multi-junction photovoltaic solar cells whose performance will drop steeply if the radiation is not uniform on its surface [Luque, 2006]. Second, the optical system should accept all incident light rays within certain angle away from the normal direction, which is needed to eliminate the need for an extremely accurate normal incidence orientation and tracking system. This angle is called acceptance angle.

### 3.2.2 Optical design

In this chapter, the concentrating optical design is based on the freeform optics by using a geometrical approach described in previous section. To satisfy the requirement of uniform irradiance on receiver’s surface, a two-surface design method of freeform surface based on Kohler integrator arrays was proposed. In the Kohler integrator system, the rays emitted by one point of the source must illuminate the whole target [Cassarly, 2001; Benitez 2004]. For a solar concentration system, this means that the light rays impinging on the first primary surface have to be redirected to the entire receiving photovoltaic cell surface at any given incidence angle within the acceptance angle. In
addition, to achieve a uniform irradiance distribution, the rays arriving at any point on the target must come from every point of the light source.

Figure 3.13 The freeform secondary surface imaging the primary surface on the given target surface

The integrated concentrating Kohler optical design consists of two imaging optical reflective surfaces (primary and secondary). The secondary is placed at the focal plane of the primary, so that the primary surface images the sun on the secondary surface and then the secondary images the primary on the target cell surface. Since the incident light from sun can be treated as the paralleled rays with different incident angle, the primary surface was selected as parabolic segment to focus incident light to the secondary surface. To
ensure uniform light distribution, ellipsoid surface segments are used to image the primary surface on the target surface. Figure 3.13 illustrates the two-stage mirror surface system. The design algorithm detail is described below.

a. The starting points of the primary mirror and secondary mirror are given as the initial calculation start position as \( P_s (P_{sx}, P_{sy}, P_{sz}) \) and \( S_s (S_{sx}, S_{sy}, S_{sz}) \). At the same time, the position and size of the photovoltaic cell are also defined by a pair of pre-defined starting and end point \((C_s \text{ and } C_e)\).

b. According to the requirement of edge rays, an ellipsoid surface \((S_s-S_e)\) is initialized by using points \( P_s \) and \( C_e \) as the focal points and passing point \( S_s \). The size of this ellipsoid is controlled by the required acceptance angle at the primary surface, as shown as ray 2, 3 in figure 3.13. After being reflected by the ellipsoid surface, all incident rays within acceptance angle at point \( P_s \) are focused at target surface point \( C_e \), according to reflective property of the ellipsoid surface.

c. By defining that point \( S_s \) is passed through by the reflective line 1 from point \( P_s \) with maximum incident angle \(-\alpha\), the normal direction \( N_p \) of primary surface at point \( P_s \) can be calculated by the vector form Snell’s law as shown in equation 3.4. In that equation, incident direction \( i \) and reflective direction \( r \) both are known from the geometrical assumption, and then the normal direction \( n \) can be derived.

d. After getting the normal direction \( N_p \) at point \( P_s \), the reflective direction of the incident ray along normal direction can be calculated by using equation 3.4, shown as ray 2 in figure. The point of intersection between this reflective ray and the pre-
defined ellipsoid $S_e$ is defined as the focal point $S_f$ of the primary parabolic segment passing point $P_s$. The intersection point can be found by using the bi-section method. With the point $P_s$ and the focal point $S_f$, the primary parabolic segment profile can be derived as $P_s-P_e$.

e. According to the reflective pair line 2, the normal direction $N_s$ at the intersection point $S_f$ can be calculated by question 3.4. In addition, the reflective line of normal incident ray reaching the end point of the parabolic segment is reflected to the point $C_e$ by the ellipsoid surface at the point $S_f$. Therefore, by calculating the reflective direction of line $C_e-S_f$, the position of the end point $P_e$ of the parabolic surface can be determined by the bi-section method to find the intersection point between parabolic surface and ray 4.

f. By calculating the reflective ray with maximum incidence angle $\alpha$ at point $P_s$, the intersection point with the ellipsoid surface could be found, which was defined as the end point of the secondary segment.

g. Storing the parabolic and ellipsoid segment, and setting point $P_e$, $S_e$ as the new start points $P_s$, $S_s$, and then go to the step $a$ for a new loop until position of $P_e$ is on the left of the initial start point $S_s$.

h. Continuing the iterative algorithm until the calculated point $P_e$ is on the right of initial secondary starting point $S_s$ then the calculation is terminated. This critical condition is applied to avoid blockage of secondary on the primary mirror.
This design algorithm provides a process to design 2D two-stage concentrator to obtain uniform irradiance distribution. When the 2D results of primary and secondary mirror are expanded to a given width $W_p$ and $W_s$ respectively, the light distribution on the target surface is also turned to a related width $W_c$. Therefore, the 2D design turns to a pseudo-3D surface similar to a “stripe” pattern. Based on this idea, an array of the striped primary and secondary mirror is combined and modified to illuminate the desired target cell surface. Each stripe of primary and secondary combination is optimized by above listed algorithm. By using this method, a 3D profile is proposed. However, in this 3D design the integration is carried out in the radial direction, but not in the azimuthal direction. In addition, a merit function is applied to ensure the smoothness at the intersection of adjacent “stripes” by minimizing the “jumpiness” between any two neighbor “stripes”.

**Figure 3.14** (a) Primary freeform mirror based on previous stated algorithm (b) Designed secondary freeform mirror
A graphic interface execute program was programmed using MATLAB GUI, as shown in figure 3.15. As in this figure, we can adjust different parameters to obtain different primary-secondary pair. When the calculation is finished, we can output the surface information to ZEMAX for future ray-tracing verification or to data file for fabrication preparation. Finally, we estimate the total area concentrating ratio by using equation 3.16:

\[
C_g = \frac{A_{\text{Primary}}}{A_{\text{receiver}}} \tag{3.16}
\]
In this design, the receiver is an area with 5x5mm dimension, and the primary mirror is designed with dimension 100x80mm. Therefore, the final area concentrating ratio is about 320 in this design.

3.2.3 Ray-tracing simulation

After numerical calculation, a ray-tracing simulation was performed to verify the concentrating performance of designed primary-secondary pair. As presented in previous section, ZEMAX non-sequence part was used for ray-tracing. Primary and secondary mirrors were meshed into small segments, which were defined as quadrangle reflective surfaces in ZEMAX subroutine. A detector with 100 by 100 pixels was placed on the position of the solar photovoltaic cell. To simulate the incident light of sun, uniform collimated light with 0.26° half angle was introduced as the light source. We defined the ratio between the rays received by the detector and rays reaching the primary mirror surface as the transmission ratio. Transmission ratio is a critical condition to evaluate the acceptance angle of the concentrating system. Figure 3.16 shows the transmission ratio at different incident angles. As shown in this figure, when the tilted angle along x-axis of the incident ray is less than ±1°, there is no significant change in the transmission ratio. For the tilted angle along y-axis, that angle is ±1.2°. This result demonstrates that the freeform design satisfies the required condition of acceptance angle. An acceptance angle of ±1.2° is achieved according to the ray-tracing simulation result. The intensity distribution is also evaluated by the ray tracing simulation. Figure 3.17 shows the local irradiance distribution on the receiver surface when the incident light is along normal direction. The simulation shows that the freeform mirror can provide a vertical and
horizontal sharp cut-off gradient at the edge of solar cell receiver. Furthermore, the irradiance distribution inside the receiver border is uniform as shown in figure 3.17b. When the angle of the incident light is increased to 1°, this two-stage freeform concentrator can still provide sharp cut-off gradient at the edge, and the irradiance distribution is also uniform, as shown in figure 3.18. This simulation result demonstrates that this concentrator works well when the incident light angle is less than the acceptance angle, and can provide uniform irradiance distribution on the receiver surface.

![Graph showing transmission versus incidence angle]

**Figure 3.16** Ray-trace simulation of the freeform CPV design: transmission versus incidence angle
Figure 3.17 Ray-trace simulation result: (a) local light irradiance distribution on the cell when the sun is centered (b) Irradiance distribution on the cell along x-axis and y-axis respectively.
Figure 3.18 Ray-trace simulation result (a) local light irradiance distribution on the cell when the sun is off-axis 1 degree (b) Irradiance distribution on the cell along x-axis and y-axis respectively
3.2.4 Fabrication

The designed primary mirror of concentrator will be fabricated by glass workpiece using thermal forming process. We will discuss this process in next chapter. The secondary mirror can also be fabricated by using similar thermal slumping process. In this process, the secondary mirror was directly fabricated on an aluminum block using diamond turning process to show its surface profile. This arrangement allows us to focus exclusively on developing the thermal slumping process using the primary mirror as a test bed. The fabricated result of the aluminum secondary mirror is shown in figure 3.19.

![Secondary mirror of the concentrator fabricated by single point diamond turning process](image)

**Figure 3.19** Secondary mirror of the concentrator fabricated by single point diamond turning process
CHAPTER 4: THERMAL SLUMPING OF FREEFORM GLASS COMPONENTS

Thermal slumping process is a thermal deformation method to replicate the pre-fabricated mold surface profile to the slumped glass workpiece. Glass thermal slumping is a low cost, high volume, and environmentally conscience process for fabricating glass optical surfaces. The thermal slumping process has become a proven alternative for the ophthalmic application and x-ray telescope reflector [Zhang, 2003; Jimenez-Garate, 2003]. In glass thermal slumping process, the mold and glass workpiece are pre-heated to the slumping temperature until the glass became soft. Afterwards, the glass workpiece is deformed to the mold surface by gravity. After controlled cooling, the slumped glass workpiece is released at the room temperature. Figure 4.1 shows the major steps of the thermal slumping process.

Figure 4.1 thermal slumping process duplicates the surface curvature of ceramic mold to glass workpiece.
In this chapter, the thermal slumping process was used to fabricate glass mirror with parabolic of revolution surface, and also used to fabricate primary mirror of the freeform concentrator designed in previous chapter. An easy to machine MACOR® glass-ceramic was chosen as the mold material to reduce the total cost of fabrication process. Due to the limitation of experiment conditions, a 100 mm diameter mold and same size glass workpieces were used to demonstrate the thermal slumping process. This process can be easily scaled up to fabricate larger size glass primary mirrors for high concentration solar energy applications. Finally, different slumping parameters were applied to evaluate and optimize the performance of process. The slumped glass mirror was evaluated for curvature accuracy and surface quality by using coordinate measurement machine (CMM) and atomic force microscope (AFM). Surface roughness of the slumped glass mirror was also measured by using a profilometer.

4.1 Mold fabrication

The nature of glass thermal slumping process requires that the mold materials be able to work at high temperature with proper chemical and thermal stability. In addition, to reduce the total cost for primary concentrator mirror fabrication, the mold material needs to be fabricated easily using conventional machine tools such as a computer numerical controlled (CNC) lathe or milling machine. At typical glass slumping temperature, glass viscosities are in the order of $10^9 \sim 10^{10}$ Pa.s. For most glass materials used to fabricate optical components, maximum forming temperature will be over 600 °C. At this temperature, most mold material used in plastic molding process, such as aluminum, stainless steel, and nickel alloys start deteriorating and potentially can stick to glass
workpieces. Sometimes a thin layer material coating such as TiAlN/ZrN is applied to the mold surface to reduce sticking and mold wear [Hock, 2003]. However, coating process will increase the complexity and cost of the thermal slumping process.

Figure 4.2 Flowchart of the mold fabrication for freeform thermal slumping process

On the other hand, traditional glass compression molding insert materials such as cemented tungsten carbide (WC), silicon carbide (SiC), have good hardness, stability at high temperature and wear resistance, but the fabrication processes are designed primarily for high precision optical lense manufacturing therefore can be very expensive [Yi, 2005]. In this research, MACOR® (www.corning.com) ceramic was used as the
thermal slumping mold material for its excellent chemical stability at high temperature up to 1000 °C. MACOR® is a machinable glass-ceramic, which can be machined into any desired shape using standard metal working tools without any difficulty [MACOR, 2008].

According to our experiments, there are no significant sticking between MACOR® mold and soda lime glass mirror after thermal slumping process up to 660 °C. Furthermore, there are no visible wear and defects on MACOR® mold after thermal cycling. These properties make MACOR® ceramic suitable as a mold material for high volume glass thermal slumping.

Figure 4.2 shows the flowchart of mold design and fabrication process. To fabricate MACOR® ceramic mold, first of all, the mold profile was designed based on optical performance requirement, as presented in previous chapter. Then, an numerical simulation and finite element method were performed to compensate the error introduced in thermal slumping process. Mold design was modified by the compensation result. Based on the designed surface profile, a 3D part drawing was created using a modeling software, and then exported to CAE software FeatureCAM for CNC manufacture. Afterthat, MACOR® mold was fabricated using a CNC machine (Haas VF-3 CNC machining center) with a standard metal working carbide ballend mill. The fabricated molds were measured by using a coordinate measuring machine (CMM). Figure 4.3a shows the surface profile of MACOR® ceramic mold for freeform primary mirror, and figure 4.3b shows the deviation between measured results and designed surface profile. In addition, figure 4.4 shows the fabricated mold profile for parabolic surface. According to the measured result, the curvature deviation of fabricated MACOR® mold surface from
design is less than 40 μm, as shown in figure 4.3 and 4.4. In figure 4.3b, most curvature deviations are less than 40 μm except the edge of segments, which may come from the misalignment during CMM measurement.

Figure 4.3 MACOR® mold surface profile measured by CMM (a) surface profile (b) fabrication error compared with design surface
4.2 Slumping Process

All thermal slumping tests in this research were conducted on a custom home built hot glass-molding machine and thermal furnace (GRIEVE, BF-12128-HT thermal furnace). The details of the machine design and its performance have been included in references [Firestone, 2005]. This machine is capable of controlling temperature inside of the furnace over 800 °C. To control the thermal slumping formation and optimize process time and performance, the following process was used to make the glass mirror:

- A precut circular soda lime glass workpiece with 100 mm diameter and 1.1 mm thickness was placed on top of the MACOR® ceramic mold as shown in figure 4.1.
- The glass disk and MACOR® mold were loaded into the glass-molding machine.
• The furnace temperature $T$ was raised up to the strain point $T_{\text{strain}}$, and then the furnace temperature was held for a few minutes to ensure a uniform temperature distribution inside mold and glass workpiece.

• After temperature in the furnace reached a presumably uniform level, it was heated up again to soaking temperature at a lower heating rate.

• The furnace temperature reached soaking point and was held for a fixed amount of time to allow the glass workpiece to deform to the MACOR® mold surface.

• After soaking, the furnace was cooled down to room temperature, and the slumped glass mirror was removed.

• Figure 4.5 shows an example of temperature history collected by the thermal sensor inside the furnace during a cycle of thermal slumping process. The soaking temperature was 620 °C with duration of 120 minutes. Different soaking temperatures and different holding times were also performed.

![Temperature history of a selected thermal slumping process](image)

**Figure 4.5** Temperature history of a selected thermal slumping process
As stated in previous section, at soaking temperature glass viscosities are in the order of $10^9 - 10^{10}$ Pa.s. At this temperature, the viscosity is high enough that the thickness of glass workpiece does not change appreciably during forming while the surface tension can stretch the glass surface to desired surface roughness. On the other hand, glass viscosity is also low enough so it can slump to mold shape in finite working time. At a high temperature, more specifically, around glass transition temperature, glass exhibits viscoelastic behavior, which is strongly time and particularly temperature dependent.

When temperature goes much higher than $T_g$, viscosity of glass becomes extremely low that stress relaxation times are in the order of milliseconds or even shorter. These stress relaxation times are much shorter as compared to the soaking time thus the stresses introduced from loading are negligible. Therefore, the glass material can be modeled as a 3D Newtonian fluid and the material behavior can then be described using equation 4.4 based on 3D Newtonian constitutive law where stresses $\tau_{ij}$ are a function of strain rate $\dot{\varepsilon}$ and viscosity $\eta$ related in the following equation [Jain, 2006; Chen, 2008]:

$$\tau_{ij} = 2\eta(T)\dot{\varepsilon}_{ij}$$

(4.4)

The viscosity $\eta$ value of glass at different temperature is curve-fitted using the Vogel, Fulcher, and Tammann (VFT) equation [Fulcher, 1925] give by equation 4.5:

$$\log(\eta) = A + \frac{B}{T - T_0}$$

(4.5)

where $A$, $B$ and $T_0$ are constants that are usually obtained from the glass vendor. In this research, soda lime glass disks with near polished surface on both side with 100 mm diameter and 1.1 mm thickness ( $T_g = 557$ °C, S. I. Howard Glass Co. Inc, Worcester MA)
were used as the glass workpiece. The surface finish was created during the manufacturing process when the glass sheets were created from glass melt. No further polishing was performed on the glass samples. The viscosity curve for soda lime glass is shown in figure 4.6, calculated from equation 4.5. By using the exponential viscosity curve for the soda lime glass, glass viscosity at other temperatures in the molding range can be calculated for the experiment and the matching numerical simulation.

Figure 4.6 Viscosity versus temperature curve for the soda lime glass fitted by VFT equation

4.3 Measurement and Discussion

The inner surface contour of the slumped glass mirror was measured using a coordinate measurement machine (CMM, Sheffield Cordax RS-30 DCC) to evaluate the manufacture result. The results of CMM measurements of parabolic mirrors and freeform primary mirrors are shown in figure 4.7a and figure 4.8a. The measurement result is also compared with the pre-designed surface profile. Figure 4.7 shows the CMM measured results of slumped parabolic surface of resolution. As shown in the error map in figure
4.7b, the final curvature deviation is less than 40 \( \mu \text{m} \) when compared with original optical design. Figure 4.8 shows the CMM measured result of slumped freeform primary glass mirror of the concentrator. As shown in the error map in figure 4.8b, the maximum curvature deviation at the edge is less than 100 \( \mu \text{m} \), and at the maximum curvature deviation at the central area is less than 50 \( \mu \text{m} \). The detailed surface curvature compensation and manufacture tolerance analysis will be discussed in next two chapters.

![Figure 4.7 CMM measurement results of slumped parabolic mirror (a) surface profile (b) error from the design profile](image)

The surface roughness of the slumped freeform mirror was also measured by using AFM, as shown in figure 4.9. Obviously, the surface roughness of the slumped glass mirror \((R_a = 3.8\text{nm})\) is much better than the surface roughness of MACOR® mold surface \((R_a = 1.4\mu \text{m})\), and can be used for optical purpose. According to these measurement results, glass thermal slumping process can be used to produce glass mirror with desired shape as a low cost one-step process.
Several important parameters were also investigated, including the relationship between surface contour error and thermal slumping temperature and holding time, and the relationship between surface roughness and thermal slumping temperature and holding time. The slumped freeform mirror is presented in this section. The surface contour error is drawn from the CMM measurement result. To evaluate the curvature error, the root
mean square (RMS) of the curvature deviation of every measurement is applied by using the equation 4.6:

\[
RMS = \sqrt{\frac{1}{n} \sum_{i=1}^{n} e_i^2} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} (x_i - x_i')^2}
\]

(4.6)

where \(n\) is the total measurement points, \(e_i\) is the deviation at each point, \(x_i\) is the designed position, and \(x_i'\) is the measured result from CMM.

**Figure 4.9** Surface roughness of slumped freeform primary mirror measured by AFM

Figure 4.10 shows the surface curvature deviation under different thermal slumping parameters. First, we keep the slumping holding time at constant at 120 minutes while changing the thermal slumping temperature from 600 °C to 660 °C every 10 °C, as shown in figure 4.10a. At higher temperature over 670 °C, the glass workpiece will stick to the MACOR® mold. From the measured result, no more improvement was seen when the slumping temperature was higher than 640 °C. We can consider that the glass
workpiece has made full contact with the MACOR® mold at and beyond this temperature. We also investigated the holding time changing from 30 minutes to 120 minutes at same thermal slumping temperature of 640 °C, as shown in figure 4.10b. Similarly, when the slumping time is larger than 60 minutes, the glass workpiece has made full contact with the MACOR® mold. Therefore, to secure full contact between the mold and glass workpiece, 640 °C slumping temperature and 90 min holding time will be used in future experiment.

![Figure 4.10](image)

**Figure 4.10** surface contour error of the slumped freeform primary mirror under different thermal slumping parameters (a) different thermal slumping temperature with 120 holding time (b) different holding time at 640 °C slumping temperature.

Figure 4.11 shows the surface roughness results under different thermal slumping parameters measured by Mitutoyo S-3000 profiler with the similar conditions as figure 4.10. As shown in this result, the surface roughness increases with the thermal slumping temperature and holding time. However, when compared with MACOR® mold ($R_a =$
1.4µm), the surface quality is still considered adequate for optical mirror applications when coated with reflective material such as aluminum film. The scattering test of different surfaced roughness will be presented in chapter 7.

![Graphs showing surface roughness at different temperatures and holding times.](image)

**Figure 4.11** surface roughness of the slumped freeform primary mirror under different thermal slumping parameters (a) different thermal slumping temperature with 120 holding time (b) different holding time at 640 °C slumping temperature.

One of the key limitations of the thermal slumping process is the ability to accommodate steep changes in curvature. According to previous research, the difficulty of replication steep curvature change can be expressed in equation 4.7 [Smith, 1988]:

\[ K = t \cdot \Delta P \]  

(4.7)

where \( K \) is the “degree of difficulty,” \( t \) is the thickness in millimeters of the glass workpiece to be slumped. In our research, \( t = 1.1 \text{mm} \). \( \Delta P \) is the parameter of curvature, and can be expression as:
\[ \Delta P = 5.30 \Delta (1/r) \]  \hspace{1cm} (4.8)

The factor \( \Delta (1/r) \) is the change of curvature of the surface acquired during replication. In practice, the value of \( K \) should not exceed 25 for replication to the highest accuracy [Smith, 1988]. In this thermal slumping experiment, for parabolic mirror, the maximum curvature change is determined by the designed curvature equation:

\[ Z = \frac{1}{525.8} r^2 \]  \hspace{1cm} (4.9)

The curvature of surface at any point on the parabolic surface is:

\[ \frac{1}{R} = \frac{p^2}{(p^2 + r^2)^{3/2}} \]  \hspace{1cm} (4.10)

Where \( p \) the parabolic parameter, and is 262.9 at this time. Therefore, the change of curvature at any neighbor position can be expressed as:

\[ \Delta \frac{1}{R} = \left| \frac{3p^2}{(p^2 + r^2)^{5/2}} r \right| \]  \hspace{1cm} (4.11)

For the glass workpiece with 100mm diameter, the maximum curvature is \( 8 \times 10^{-6} \) according to equation 4.11. The maximum degree of difficulty \( K \) is about \( 4.7 \times 10^{-5} \). Therefore, thermal slumping will be an efficient method to fabricate parabolic surfaces.

For the freeform primary mirror, there is no single equation that can be used to describe the surface contour. The curvature change can be calculated from the meshed point matrix by calculating any adjacent points. Figure 4.12 shows a calculated \( K \) value map over the whole designed freeform primary mirror. As shown in this figure, the degree of difficulty \( K \) is far less than 25 inside the small segment since they are part of parabolic segment. However, there are some large \( K \)-values at the joint edge of two segments due
to the change of curvature. Since the most area of primary mirror is still within the manufacture tolerance, we believe that the thermal slumping process is adequate for freeform optics. The detail of the manufacture tolerance analysis will be presented in chapter 7.

![K value map](image)

**Figure 4.12** The $K$ value map over the whole designed freeform primary mirror

### 4.4 Conclusion

A high volume low cost thermal slumping process was proposed to fabricate simple parabolic mirror and then the freeform glass primary mirror for high concentration solar energy. The surface profiles of the slumped glass mirrors were measured using a CMM and the surface quality was evaluated by AFM and profiler meter. Some of the key contributions of this section are summarized as follows:
1. MACOR®, as a machinable glass ceramic material, was used as mold material for its excellent mechanical and chemical stability at high temperature. All machining processes used in creating ceramic molds are widely available in machine shops in industry. No special tools were used. Furthermore, it was determined that no significant stickiness between the glass workpiece and the MACOR® mold occurred at the selected slumping temperature.

2. If slumping temperature profile were properly selected, the surface roughness of slumped glass mirror would remains unchanged after slumping because of surface tension. Higher the slumping temperature, shorter the slumping time. However, when slumping temperature is higher than 670 °C, the soda lime glass workpiece will stick to the MACOR® mold.
In thermal slumping and compression molding process, one of the most important design criteria is to satisfy the requirement of curvature of surface to sufficient optical precision. However, the transfer of even the basic former shape to glass is not exact. Geometrical deviation (or curve change as often referred to in industry) incurred during heating, molding and cooling process is a critically important manufacturing quality parameter. In glass compression molding and thermal slumping process, there are many factors that could lead to curve change in final products, such as thermal expansion, stress and structural relaxation and inhomogeneous temperature distribution inside the molding machine. In this chapter, we focus on the geometrical deviation caused by thermal expansion and free surface difference from the mold surface. The curvature change caused by residual stresses will be discussed in next chapter.

Mold design, manufacturing, coating and molding conditions are considered some of the most important factors for a successful process in compression molding of aspherical glass components and thermal slumping of freeform glass optical mirror. One of the most challenging jobs in mold design involves the compensation of curvature deviation, which is dependent on mold temperature, load, holding time and cooling rate. In a conventional
mold design process, experiments under different molding parameters are carried out in order to collect required data for mold compensation, which is time consuming and costly. As an alternative, numerical calculation and simulation including finite element method (FEM) has been extensively utilized to assist fabrication process analysis and optimization in manufacturing. In this chapter, different compensation methods were applied and compared with experimental results.

5.1 Zero Order Compensation

In the thermal slumping process, the final shape of the top free surface of glass workpiece after slumping stage is different from the mold surface. The relationship between the top free surface of the glass and the mold’s shape is very complicate and nonlinear, especial for the freeform optical contour. To compensate the geometry error introduced in this process by simplified method, we can start from an initial zero order compensation. The zero order compensation is based on the assumption that the glass forms a layer of uniform thickness $h$ over the mold surface at the end of the slumping process [Agnon, 2005]. Based on this assumption, for any designed freeform optical surface $K(r, \varphi)$, we can determine the compensated mold shape $Z(r, \varphi)$, as shown in figure 5.1. For any place on mold surface where the radius of curvature is $R_f$, the radius of curvature of the top glass surface would be $(R_f - h)$. For parabolic surface, we can easily calculate the zero order compensated mold surface from designed profile described in equation 4.10.

In the fabrication process for concentrating primary mirror, the desired shape of top glass surface is freeform profile without constant radius of curvature. To solve this problem,
we proposed a method in which the normal direction of every meshed point on the freeform optical surface was calculated based on the spline surface formed from neighboring points. Once the normal direction of each point on the designed surface is found, the compensated surface profile can be calculated using vector form calculation as shown in figure 5.2 and equation 5.1.

\[ \hat{r}_0 = \hat{r}_1 + \hat{n}_0 \hat{h} \]  

(5.1)

**Figure 5.1** Zero order compensation for thermal slumping process

**Figure 5.2** Vector form for calculating zero order compensation
Based on the calculation from zero order compensation, a new mold design can be created. To verify the zero order compensation result, estimation from previous experiment results can be made by using an inverse method. We can set the previous mold as the design without zero order compensation to directly compare with the slumped upper surface of the glass mirror. At the same time, we can also calculate the zero order compensated curvature of the mold surface as design to compare with real experimental results. Figure 5.3 shows the comparison with and without zero order compensation from the freeform glass slumping experiments. Obviously, after zero order compensation, we can get better experimental results with smaller error RMS value.

![Figure 5.3](image.jpg)

**Figure 5.3** Experimental results compared with compensated and non-compensated surface curvature

5.2 **Thermal Expansion Compensation**

Thermal expansion/shrinkage of the glass workpiece and mold material is another important impact factor for successful compensation of geometry contour, which is
highly dependent on glass and mold materials and slumping temperature. The different material properties between glass and mold material (MACOR®) is an important reason for the geometrical contour error after slumping process. Table 1 shows the thermal expansion coefficients of these two materials. The glass workpiece is slumped down to the mold surface at holding temperature at which the surface contour of MACOR® is slightly different from that of room temperature due to thermal expansion. At this temperature, if we let the glass replicate the mold surface under zero order compensation, the curvature will be different from the calculation at room temperature. At the same time, the glass workpiece will also expand and shrink with changing temperature. Therefore, to get more accurate surface contour result, the thermal expansion mismatching must be considered. To demonstrate the influence of thermal expansion, a simple model was applied. First, the thin sheet glass workpiece, which was used in our thermal slumping process with 1.1mm thickness and 100mm diameter, was placed on the MACOR® ceramic mold with freeform surface. Then, both glass and mold were heated up to the holding temperature. A linear thermal expansion equation was used to calculate the curvature change for both materials.

\[ \Delta L = \alpha L_0 \Delta T \]  \hspace{1cm} (5.2)

Where \( \Delta T \) is the temperature change, \( \alpha \) is the linear thermal expansion coefficient and \( L_0 \) is the relative position from origin point. Once the temperature reached the slumping condition, the expanded glass was slumped down to the expanded mold surface under gravity to replicate the expanded mold surface over the holding time. Afterward, the temperature was cooled down to the room temperature and both glass and mold shrunk.
back. By including the thermal expansion coefficient in consideration, the estimation of final shape can be improved. At the same time, zero order solution for thin sheet glass slumping process can also be applied in surface compensation combined with thermal expansion result together when the expanded glass workpiece replicates the expanded MACOR mold surface at slumping temperature. As shown in figure 5.4, more accurate prediction results are achieved when both factors are considered.

<table>
<thead>
<tr>
<th>Table 5.1: Thermal expansion coefficient of mold and glass</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Material</strong></td>
</tr>
<tr>
<td>MACOR</td>
</tr>
<tr>
<td>Soda Lime Glass</td>
</tr>
</tbody>
</table>

Figure 5.4 Comparison of three compensation methods.
5.3 FEM simulation

In the slumping process, the bottom boundary is continuously changing from free surface to a no-slip contact surface, which is a strong non-linear process. Besides, glass material also has very strong non-linear material properties, especially in the glass transition region. Therefore, the thermal slumping process is highly non-linear process, and zero order compensation and linear thermal expansion model is not enough in the slumping and cooling stage. The compensation process is more complicate than zero order error and linear thermal expansion. As an alternative, finite element method (FEM) has been extensively utilized to assist fabrication process analysis and optimization in manufacturing. More recently, new studies have also demonstrated that FEM assisted manufacturing approach can be a good alternative for solving problems in compression glass molding process and thermal slumping process [Sellier, 2007].

We and colleagues in IPT, Germany also established a systematical research together for geometrical deviation using experimental and numerical simulation for glass compression molding [Wang, 2008; Dambon, 2009]. In the first study, it was demonstrated that numerical simulation and experimental results showed a good agreement. Specifically, it was shown in our previous research that the amount of the curve change predicted using FEM simulation matches the measurements in compression molding experiments to less than 2 µm. Based on this result, compensation for the mold shape can be then made in order to produce the desired glass lens shape after molding or slumping without performing the initial molding test. Experiments in this research have shown that the molded or slumped glass lenses/mirrors using the compensated molds have a better
agreement with the design value as compared to the lenses molded using the original molds.

In this dissertation research, to compensate the curvature deviation in thermal slumping process, numerical simulations matching the experiment conditions are performed using MSC/MARC. Simulation of the molding process also includes three major steps: heating, molding (slumping) and cooling. The heating stage includes thermal and structural modeling to identify the temperature distribution and the corresponding thermal expansion, which are the important parameters for the following molding stage. During the molding/slumping stage, two different model methods can be used as Newtonian fluid model or viscoelastic model. According to our previous research, Newtonian fluid model can provide accurate enough result [Jain, 2006], and so it is used in this research. The resulting model is transferred to the cooling stage to obtain the final geometry. Structural
relaxation model was applied in the cooling stage. The geometrical deviation caused by structural relaxation under different cooling rates will be described in next chapter. In this chapter, only structural relaxation model under experimental cooling rate with uniform cooling temperature distribution was used. After the cooling stage, the final geometry was then compared with original glass lens design shape and the result was used as the feedback information for the new mold design. Figure 5.5 shows the three simulation stages in the glass thermal forming process. The flowchart of the integrated simulation and optimization procedures is shown in Figure 5.6. The integrated optimization procedures can improve the simulation process and provide more accurate simulation results.

As stated in previous section, in the thermal slumping process glass viscosities are in the order of $10^9$-10$^{10}$ Pa.s at soaking temperature. Glass material can be modeled as a 3D Newtonian fluid and the material behavior can then be described using 3D Newtonian constitutive law where stress tensor $\tau_{ij}$ is a function of strain rate $\dot{\varepsilon}$ and viscosity $\eta$ related as described in equation 4.4. The viscosity $\eta$ value of glass at different temperature is curve-fitted using the Vogel, Fulcher, and Tammann (VFT) equation given in equation 4.5 in chapter 4. $A$, $B$ and $\tau_0$ are obtained from the glass vendor. The viscosity curve for soda lime glass is shown in figure 4.6, calculated from Eq. 4.5. By using the exponential viscosity curve for the soda lime glass, glass viscosity at other temperatures in the molding range can be calculated for the experiment and the matching numerical simulation.
In this section, the parabolic glass mirror was first used as the example to demonstrate the FEM simulation process. Afterward, the freeform primary mirror was simulated using similar process in an approximate 2D condition. For parabolic mirror, due to the simplicity of the glass mirror geometry, 2D axisymmetric simulation of the glass thermal slumping process was performed using a commercial FEM code MSC/MARC. Figure 5.7(a) shows the meshed geometry of a glass workpiece and the MACOR® mold. The boundary conditions, including gravity, convection heat transfer, contact between glass
workpiece and MACOR® mold, were also shown in this figure. The contact between glass workpiece and MACOR mold was defined as Coulomb stick-slip model according to our previous research, and the shear friction coefficient is 1 in this simulation [Jain, 2006].

**Table 5.2: Mechanical and thermal properties of soda lime glass and MACOR® mold**

<table>
<thead>
<tr>
<th>Property</th>
<th>Soda Lime Glass</th>
<th>MACOR®</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastic modulus, $E$ [Mpa]</td>
<td>68500</td>
<td>66900</td>
</tr>
<tr>
<td>Poisson’s ratio, $v$</td>
<td>0.22</td>
<td>0.29</td>
</tr>
<tr>
<td>Density, $\rho$ [Kg/m$^3$]</td>
<td>2483</td>
<td>2520</td>
</tr>
<tr>
<td>Thermal conductivity, $k_c$ [W/m °C]</td>
<td>0.9</td>
<td>1.46</td>
</tr>
<tr>
<td>Specific heat, $C_p$ [J/Kg °C]</td>
<td>1046</td>
<td>790</td>
</tr>
<tr>
<td>Coefficient of thermal expansion, $\alpha_g$ [ºC]</td>
<td>$9 \times 10^{-6}$</td>
<td>$11.4 \times 10^{-6}$</td>
</tr>
<tr>
<td>Viscosity, $\eta$ [MPa-sec] (at 640°C)</td>
<td>202</td>
<td>N.A.</td>
</tr>
</tbody>
</table>

The simulation includes two major steps: 1) the glass workpiece was slumped down under its own weight at different viscosities from temperature change until cooling beginning; 2) the slumped glass mirror was cooled to room temperature under predetermined cooling rates. As mentioned before, the glass workpiece was defined as Newtonian fluid model in this simulation. Since the viscosity of soda lime glass is a function of temperature, it’s not accurate to use a single viscosity value to simulate the
entire slumping stage. A user defined subroutine was introduced to calculate the viscosities at different temperatures. MSC/MACR can load the calculation results and use them in the slumping step. Table 5.2 shows the thermal and mechanical properties of soda lime glass and MACOR® mold used in FEM simulation [Howard, 2008; MACOR, 2008].

Figure 5.7 (a) A cross sectional view showing the mechanic and thermal load in thermal slumping process (b) the final shape of glass workpiece after thermal slumping process
In the second step, a simple structural relaxation model with uniform cooling temperature distribution was selected in this research. Narayanaswamy model was used to approach the structural relaxation process. The structural relaxation parameters of soda lime glass are shown in Table 6.3. Figure 5.7b shows the slumped result simulated by MSC/MARC. The detailed structural relaxation model under different cooling conditions will be presented in next chapter.

![Diagram]

(a)

![Diagram]

(b)

**Figure 5.8** FEM simulation result for the slumped glass mirror shape before and after mold curvature compensation
Figure 5.8 shows the experimental measurement results of the slumped glass mirror using the mold design before and after compensation respectively. As shown in this figure, the glass mirror slumped using the compensated molds showed a much better agreement with designed value than the mirror molded without compensation. Therefore, new mold design was used in glass thermal slumping test in this research. The approach using numerical modeling provided an opportunity for optical manufacturers to achieve a lower production cost and a shorter cycle time.

We also performed FEM simulation for freeform primary mirror thermal slumping by using similar process as described before. Figure 5.9 shows the FEM model in the MSC/MARC before and after thermal slumping process. Due the complication of freeform primary mirror surface contour, only one single line along x-axis was drawn out to simulate the slumping process in a 2D plane strain situation. Figure 5.10 shows the comparison between FEM simulation and measured glass mirror surface from CMM machine. As shown in this figure, the FEM simulation can provide accurate estimation of final glass mirror shape with the error less than 40 μm, and the RMS in simulation is 0.208, which is better than the result from zero order compensation and thermal expansion compensation. The error may come from the measurement or misalignment between measuring and simulation. This comparison result demonstrates that the FEM simulation can be used as a useful for curvature compensation of thermal slumping process.
Figure 5.9 A cross sectional view showing FEM simulation of the freeform glass mirror thermal slumping before and after slumping process

Figure 5.10 FEM simulation result compared with measured glass mirror
6.1 Introduction

Residual stresses are important criteria for evaluating molded glass optical components. Residual stresses inside glass lenses can cause refractive index variation, unwanted light path deviation as well as intensity change that can result in image quality deterioration. Besides, residual stresses inside glass optical components can also cause geometrical change after the glass mirror being released from mold.

Different methods have been tested to measure residual stress distribution inside a glass workpiece. L.C. Shepard and his colleagues developed a method for internal residual stress measurement by measuring Rayleigh-scattered light propagating through glass sample at an oblique angle from a properly polarized laser beam [Shepard, 2003]. Brodland and Dolovich presented a method that used curved ray effects to investigate the stress profile in tempered glass [Brodland, 2000]. They measured laser beam curvature that it enters and exits on the same side to analysis and compute inside residual stress. Among these techniques, photoelasticity based method for residual stress analysis in glass or polymer components can be a useful tool to study the cooling process for glass molding. Birefringence is widely used for characterizing molecular orientation in
amorphous and semi-crystalline materials. It can provide general information of residual stress distribution inside the glass sample [Aben, 1993; Kuske, 1974].

The research in this chapter includes two parts. First, the residual stresses inside compression molded glass lens are measured and compared with numerical simulation result to provide a numerical simulation approach for controlling residual stresses. Second, the FEM simulation is applied to thermal slumping process to investigate the residual stresses and curvature deviation due to structural relaxation.

In compression molding test, a numerical simulation method was developed to model both the molding and cooling process. The residual stresses calculated using an FEM program were further employed to numerically calculate the birefringence inside the glass lens. These conditions were also used in experiments designed to study the BK7 glass lenses. The optical characteristics of the molded glass lenses were studied using a plane polariscope to evaluate the effect of the residual stresses. The simulation and experimental results showed a good agreement. Finally a critical cooling rate suitable for annealing process of molding glass optics was predicted based on simulation results. It is also demonstrated in this research that the parameter of annealing can be optimized using numerical simulation approach. It is shown through this study that numerical simulation plays an important role in glass molding research. Specifically it enhances our understanding of the cooling process and also provides a methodology for optical manufacturers to identify and optimize their process capabilities at a minimal cost.
In the second part, the FEM simulation was used to analysis the effect of residual stresses in geometry deviation of thermal slumping process. Different surface contour changes were investigated for different residual stress levels at different cooling rates.

6.2 Residual stress in molded glass lens

6.2.1 FEM simulation

In this research, molding of cylindrical BK-7 glass ($T_g = 557^\circ$C) lenses with both side initially optical polished flat were investigated using experimental and numerical simulation approach. Due to simplicity of the glass lens geometry, two-dimensional (2D) axisymmetric simulation of the glass molding process was performed using a commercial FEM code MSC/MARC. MSC/MARC is a general purpose FEM software package but particularly suitable for highly non-linear viscoelastic analysis. The upper and lower molds were 2 mm thick glassy carbon wafers that were simplified as rigid bodies in this simulation. The original glass lens blank was a 20 mm diameter and 10 mm thick double side polished cylinder, which was defined as the deformable part. Four-node isoparametric quadrilateral element was used to mesh the glass sample into 2500 elements. The simulation includes three major steps: 1) the glass lens blank and mold flats were heated to the forming temperature. 2) The glass lens blank was pressed into pre-specified thickness by closing the mold flats. 3) The molded glass lens was cooled to room temperature under one of the three pre-determined cooling rates.
Figure 6.1 Meshed numerical simulation model in glass lens forming. a) The meshed model before forming. b) The deformed model

Figure 6.1 shows the meshed geometry of a glass lens before and after step 2. The glass material was modeled as a Newtonian fluid using the relationship between stress and strain rate in previous chapter equation 4.4 to describe the glass behavior during pressing. During cooling, glass material was modeled as a viscoelastic material undergoing structural relaxation using the Narayanaswamy model. The behavior of Narayanaswamy model is discussed in chapter 1. The relative parameters were summarized in Table 6.1
and Table 2 respectively. The stress relaxation time ($\tau_s$) at reference temperature (685 °C for BK7) can be calculated by:

$$\tau_s = \frac{\eta}{G}$$  \hspace{1cm} (6.1)

where $G$ is shear modulus of the glass material, and $\eta$ is the viscosity at the working temperature. The structural relaxation time ($\tau_v$) at the reference temperature were calculated from stress relaxation time using the formula $\tau_v / \tau_s = 10.6$ for a similar glass [Scherer, 1986].

**Table 6.1: Mechanical and thermal properties of BK7 glass**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastic modulus, $E$ [Mpa]</td>
<td>82500</td>
</tr>
<tr>
<td>Poisson’s ratio, $\nu$</td>
<td>0.206</td>
</tr>
<tr>
<td>Density, $\rho$ [Kg/m$^3$]</td>
<td>2510</td>
</tr>
<tr>
<td>Thermal conductivity, $k_c$ [W/m °C]</td>
<td>1.1</td>
</tr>
<tr>
<td>Specific heat, $C_p$ [J/Kg °C]</td>
<td>858</td>
</tr>
<tr>
<td>Transition temperature, $T_g$ [°C]</td>
<td>557</td>
</tr>
<tr>
<td>Solid coefficient of thermal expansion, $a_s$ [°C]</td>
<td>5.6x10^{-6}</td>
</tr>
<tr>
<td>Liquid coefficient of thermal expansion, $a_l$ [°C]</td>
<td>1.68x10^{-5}</td>
</tr>
<tr>
<td>Viscosity, $\eta$ [MPa-sec] (at 685°C)</td>
<td>60</td>
</tr>
</tbody>
</table>

The displacement and thermal boundary conditions were obtained from the temperature and position data in the experiments. A simplified shear friction model was used to model glass-mold interface and a friction coefficient of 1 was used based on a previous research
Three different cooling rates were selected to study their influence on the molded samples. The temperature history curves of these three different conditions are shown in figure 6.2.

**Table 6.2:** Structural relaxation parameters of BK7 glass used in numerical simulation

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference Temperature, $T$ [°C]</td>
<td>685</td>
</tr>
<tr>
<td>Activation energy/gas constant, $\Delta H/R$ [°C]</td>
<td>47,750</td>
</tr>
<tr>
<td>Fraction parameter, $x$</td>
<td>0.45</td>
</tr>
<tr>
<td>Weighing factor, $w_g$</td>
<td>1</td>
</tr>
<tr>
<td>Structural relaxation time, $\tau_r$ [sec] (at 685°C)</td>
<td>0.019</td>
</tr>
<tr>
<td>Stress relaxation time, $\tau_s$ [sec] at 685°C</td>
<td>0.0018</td>
</tr>
</tbody>
</table>

**Figure 6.2** Temperature history of three different glass molding experiments
Under these simulation conditions, we can get some preliminary results for next step numerical simulation and comparison with experiment results. Figure 6.3 shows the predicted volume versus temperature curves using the structural relaxation model for three different cooling rates by FEM simulation. The influence of cooling rate on residual stress can be easily modeled based on the fictive temperature shift suggested by the Narayanaswamy model and the effect of cooling rate on volume is clearly illustrated in Figure 6.3.

**Figure 6.3** Predicted volume versus temperature curves by structural relaxation model for three different cooling rates

Figure 6.4 shows the simulated residual stress distribution of the molded glass lens at cooling rate of 1.24°C/sec if viewed in the direction normal to the cross section. The stress distribution is due to the temperature gradient in glass lens when glass went through the transition region during cooling. These thermal stresses were eventually frozen in the glass lens when the glass solidifies due to temperature drop. Based on the
stress distribution map, in the central region of the glass lens, $\sigma_{yy}$ and $\sigma_{zz}$ are dominant stress components. However, at the edge of the lens, stress $\sigma_{xx}$ and $\tau_{xy}$ are much higher. The stress distributions corresponding to this simulation were measured using polarimeter and will be discussed in next section.

**Figure 6.4** Simulated residual stress distribution in a molded glass lens using the structural relaxation model, cooling rate $q = 1.24 \, ^\circ\text{C/sec}$. (a) stress component $\sigma_{xx}$ (b) stress component $\sigma_{yy}$ (c) stress component $\tau_{xy}$ (d) stress component $\sigma_{zz}$
Figure 6.5 shows the simulated variation of stress component $\sigma_{yy}$ during cooling from molding temperature to room temperature at the center of the molded glass lens (node 26 as shown in Figure 6.1). Residual stresses for two different cooling rates ($q_1 = 1.24 \degree \text{C/sec}, q_2 = 0.43 \degree \text{C/sec}$) were compared. Higher residual stress was introduced by faster cooling rate because of a greater thermal gradient inside the glass lens.

The residual stresses in molded glass lenses incurred when the glass material goes through the transition region during cooling are a characteristic phenomenon of glass materials. Analysis of residual stresses after cooling has indicated that in order to precisely model the lens formation, the structural relaxation model is needed to predict the correct residual stresses distribution.

![Graph showing simulated variation of stress component $\sigma_{yy}$](image)

**Figure 6.5** Simulated variation of stress component $\sigma_{yy}$ during glass lens cooling from molding to room temperature at node 26 in Figure 6.1.
6.2.2 Experiment measurement

Study of residual stresses using birefringence is based on the photoelastic effect introduced by the test material when impinged with a polarized light beam. A stress free glass is amorphous without long-range order thus is isotropic, i.e., refractive index of a glass without stresses does not change with incident angle. However, when a glass component is under stresses, for example, a molded lens after cooling it will exhibit non-isotropic properties. This phenomenon can be utilized to reveal the status of the residual stresses inside a glass sample.

\[ n_1 - n_2 = C(\sigma_1 - \sigma_2) \]  \hspace{1cm} (6.2a)

**Figure 6.6** Transfer of the simulated stresses to principal stresses for molded glass lenses

The optical properties of a transparent material can be represented by a refractive index ellipsoid. If the principal refractive indices of a glass sample coincide with the principal stresses at a point in the sample, the following equations can be used to describe the relationship between the principal indices and the principal stresses, where, coefficient \( C \) is a material property called the stress-optic constant, \( n_i \) are the refractive indices along the principal axis and \( \sigma_i \) are the principal stresses.
\[ n_1 - n_3 = C(\sigma_1 - \sigma_3) \] (6.2b)

\[ n_2 - n_3 = C(\sigma_2 - \sigma_3) \] (6.2c)

The FEM simulation results have shown that all three normal stresses (\(\sigma_x\), \(\sigma_y\), \(\sigma_z\)) and two shear stresses (\(\tau_{xy}\), \(\tau_{yx}\)) exist inside the glass lens after cooling was completed. As shown in Figure 6.6, the calculated stresses from FEM simulation must be converted to principal stresses to apply the photoelasticity equations (2a-2c). The simulated principal stresses inside a glass lens can then be determined using the following equations [Douglas, 1963]:

\[ \sigma_1 = \frac{1}{2}(\sigma_x + \sigma_y - \sqrt{\sigma_x^2 + \sigma_y^2 - 2\sigma_x\sigma_y + 4\tau_{xy}^2}) \] (6.3a)

\[ \sigma_2 = \frac{1}{2}(\sigma_x + \sigma_y + \sqrt{\sigma_x^2 + \sigma_y^2 - 2\sigma_x\sigma_y + 4\tau_{xy}^2}) \] (6.3b)

\[ \sigma_3 = \sigma_z \] (6.3c)

\[ \tan 2\theta = \frac{\tau_{xy}}{(\sigma_x - \sigma_y)^2} \] (6.3d)

where \(\theta\) is the inclination angle between the incident light beam and the principal stresses \(\sigma_1\) when the incident polarized light was parallel to \(x\) direction as in Figure 6.6. When observed using a plane polariscope, the optical retardation \(\delta\) of a glass lens due to stresses can be calculated using the modified Wertheim law [Aben, 1993]:

\[ n_y - n_3 = (n_2 - n_3) + (n_1 - n_2)\sin^2 \theta \] (6.4a)

\[ n_y - n_3 = C(\sigma_1\sin^2 \theta + \sigma_2\cos^2 \theta - \sigma_3) \] (6.4b)

\[ \delta = (n_y - n_3)d = C(\sigma_1\sin^2 \theta + \sigma_2\cos^2 \theta - \sigma_3)d \] (6.4c)

The principles of a plane polarimeter are schematically illustrated in Figure 6.7. A typical plane polarimeter includes three major components, an illuminator or light source, a
polarizer, and an analyzer. The polarizer and analyzer are two identical plane polarizers. The light intensity (which can be displayed on a screen or viewed directly) behind the analyzer can be described using the following equation:

\[ I = I_0 \sin^2(2\phi) \sin^2(\frac{\Delta}{2}) \]  

(6.5)

where \( \phi \) is the inclination angle between the principal stress and axis of polarization for the analyzer. The phase difference \( \Delta \) is related to the wavelength \( \lambda \) of the light wave by:

\[ \Delta = \frac{\delta}{\lambda} 2\pi \]  

(6.6)

**Figure 6.7** Photoelastic model in a modified dark-field plane polariscope

In this experiment, the birefringence was measured using a PS-100-SF plane polarimeter (Strainoptics Inc. North Wales, Pennsylvania). The principles of the polarimeter were already explained in Figure 6.7. On the PS-100-SF polarimeter, the analyzer can be rotated around the central axis to adjust the fringe color of the point of interest. For the molded glass lenses, the directions of the two principal stresses at the edge are always
either parallel or perpendicular to the edge direction due to axisymmetricity. To measure the residual stresses, the glass lens was first placed in the optical system as shown in Figure 6.7 and rotated around the central axis until the point of interest was in the brightest region. The analyzer was then rotated to a position when the dark fringe appeared at the point of interest on the sample. The dark fringe appeared when the quarter wave plate introduced an equal amount of retardation at the point of interest on the sample. The reading off the marks on the quarter wave plate then provides quantitative information of the optical retardation of that point. Figure 6.8 plotted the images of molded lenses under three different cooling rates observed using the polarimeter.

![Figure 6.8](image)

**Figure 6.8** The images of three molded glass lenses observed using a polarimeter: (a) Cooling rate = 1.24°C/sec (b) cooling rate = 0.43°C/sec and (c) cooling rate = 0.13°C/sec

The birefringence of a thin slice removed using a thin diamond saw from the molded glass lens as shown in Figure 6.9 was also measured using the same polarimeter. The layer removal is a proven method in measuring residual stresses inside molded parts.
After the thin layer was removed from the sample, the residual stresses were kept in the same distributions as when the slice was in the original sample. Figure 6.10 shows the birefringence image of the sliced thin sample under the plane polariscope at two different rotational angles of the glass lens molded with a cooling rate of 1.24°C/sec (top view for \( \varphi = 0^\circ \) and bottom view for \( \varphi = 45^\circ \)).

![Figure 6.9 Layer removal method for birefringence measurement](image)

**Figure 6.9** Layer removal method for birefringence measurement

![Figure 6.10 Birefringence images of the sliced sample at different angle (top half \( \varphi = 0^\circ \) and bottom half \( \varphi = 45^\circ \))](image)

**Figure 6.10** Birefringence images of the sliced sample at different angle (top half \( \varphi = 0^\circ \) and bottom half \( \varphi = 45^\circ \))

From the FEM simulation results in previous section, it is seen that the residual stress distribution was axisymmetric. To calculate the total effect on residual stresses by the
entire thickness of the glass lens in the $x$ direction, retardation of each individual thin layer was calculated and then integrated over the thickness direction. Figure 6.11a shows the intensity distribution by using numerical simulation method with a monochromatic light of 565 nm wavelength. The dark area on the plate where the directions of the principal stresses are parallel to the axes of the polaroid is the isoclinics. The intensity change along the radius is caused by the relative optical retardation, known as isochromatics. Pseudo colors were used to show light intensity. Figure 6.11b shows the normalized intensity simulated using MSC/MARC along the direction of rotational angle $\phi = 45^\circ$ in the molded glass lens using three different cooling rates. Figure 6.11c plots the measured light intensity of three experimental lenses. The intensity distribution can be used to estimate the relative retardation and the residual stress distribution. The results from numerical simulation and experiments showed a very good agreement at cooling rate of 1.24°C/sec. At lower cooling rates, the predictions became less accurate. This is believed to be due to the fact that while stresses from structural relaxation diminished as predicted by the Narayanaswamy’s model, other parameters, such as friction, molding load and molding temperature began to contribute more to the residual stresses. The variations in these parameters can result in larger measurement errors for slower cooling as seen in Figure 6.11c and Figure 6.13 later.

Using the thin layer removal method, a complex 3D problem can be transformed to a simpler 2D problem. The thin layer removal method allows the residual stresses remain frozen in the sliced glass piece. The influence of stress component $\sigma_{zz}$ thus can be removed. Since the stress component $\sigma_{xx}$ is negligible as compared with stress component
Therefore, stress component $\sigma_{yy}$ can be directly measured in the case of 0° rotational angle. In Figure 6.12a, the profile of the intensity distribution is the same as the residual stress component $\sigma_{yy}$. For rotational angle of 45°, stress component $\tau_{xy}$ can be calculated, as shown in Figure 6.12b.

**Figure 6.11** (a) Simulated intensity distribution for cooling rate of 1.24 °C/sec, (b) The intensity distribution on a single line from center to edge along the 45° radius, (c) and measured results along the same direction in the molded glass lens using a plane polariscope under three different cooling rates.
(a)                                                 (b)

Figure 6.12 Simulated birefringence intensity distribution of the sliced sample at different rotational angle, (a) 0° (b) 45°

Figure 6.13 plots both the measured and the simulated fringe numbers at the points of interest (B, C, and D in Figure 8), which can be used to calculate the relative optical retardation and residual stresses. The relative optical retardation is calculated using equation 6.7:

\[ \delta = N\lambda \]  

(6.7)

where \( N \) is fractional fringe number and \( \lambda \) is the wavelength of the light used. Once the optical retardation was calculated then the residual stress component \( \sigma_{yy} \) of the sliced glass piece can be estimated as:

\[ \sigma_{yy} = \frac{\delta}{t_mC} \]  

(6.8)

where \( t_m \) is the slice thickness, and \( C \) is again the stress optic constant. For BK7 the \( C \) value is estimated as \( 3 \times 10^{-6} \text{ MPa}^{-1} \) as in reference [Vasudevan, 1972].

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Figure 6.13 Comparison between measured results of the fringe numbers using polariscope and the simulation results, (a) point A on the molded glass lens, (b) point B, C and D on the sliced thin glass layer.

Both numerical simulation and experiments have shown that residual stresses exist in molded glass lenses after cooling. To maintain the residual stresses at or below the level required for proper optical performance, post-molding annealing may become necessary. Annealing is interpreted in this study as a very slow cooling process (for example, many hours to complete a cycle). To find a critical cooling rate under which the required residual stresses level can be obtained, different annealing rates were tested using the MSC/MARC FEM package. The maximum residual stresses (as evaluated by Von Mises stress) in the molded glass lenses under these different annealing rates were shown in Figure 6.14a. This curve can be used to predict the critical annealing rate. As explained in Figure 6.14a, glasses annealed below this rate have very low stresses. For the BK 7 sample used in this study, when annealing was performed at the rate of less than 0.01°C/sec, no significant residual stresses were introduced. To optimize the annealing
process, a nonlinear annealing schedule could be used to save annealing time. The annealing process was separated to two fixed cooling rates (0.03°C/sec and 1°C/sec) with a break temperature ($T_b$). Break temperature can be defined as the temperature below which no significant stresses can be introduced due to increase of viscosity. A slow annealing rate can be applied above the break temperature, and a fast cooling rate below it. Figure 6.14b shows the simulated Von Mises stress as a function of temperature. As also shown in Figure 6.14b, there is no significant difference in the final residual stress due to cooling rate once the temperature drops below 520°C. The residual stresses are the same as in the lenses that were annealed using a single slow cooling rate. The frozen residual stresses were largely introduced by cooling above the break temperature. Therefore, a fast cooling rate can be used to save the cooling time once the break temperature is reached.

Figure 6.14 (a) Residual stress evaluated by Von Mises stress at node 1 versus annealing rate. (b) Residual stress versus temperature when the faster cooling starts at node 1 and node 26.
Based on the research in this section, some key conclusions can be drawn for next step investigation. FEM simulation can be used to predict the residual stresses of compression molded glass optical components. A reasonable agreement between numerical simulation and experiment results was demonstrated in this research. The methodology established in this research can be easily adapted to industrial problems. With birefringence and thin layer removal method, the residual stress distribution inside the glass lenses could be measured. The stress distribution can also be explained using numerical simulation. This result further justifies the validity of applying numerical modeling to glass cooling process. The residual stresses in a molded glass lens increase with cooling rate. The amount of residual stresses and their distribution can be predicted by FEM simulation using the Narayanaswamy model. If all other molding conditions were kept the same, faster cooling rate will result in higher residual stresses. There exists a critical cooling rate for a glass material, if the cooling rate can be kept below this critical level then the residual stresses inside the glass can be controlled to a level low enough for most applications. For a given glass material, residual stresses after molding will not change significantly below the break temperature regardless the rate of cooling used below the break temperature. For BK7 glass used in this research, this temperature is about 520 °C. After molding is completed, annealing maybe necessary to remove the residual stresses in the molded glass lenses for applications where certain stress levels are not tolerated. The Narayanaswamy model can also be used to design the annealing process. As suggested the critical cooling rate and the break temperature are different from glass to glass. However the numerical simulation model discussed in this research provides a clear
approach to solving such a problem for different glass materials without doing the actual experiments.

6.3 Residual Stresses for Curve Compensation

Structural relaxation of glass workpiece during cooling stage will not only cause the residual stresses and then birefringence (index deviation) in the molded glass lens, but also cause the geometry change of slumped glass mirror under different cooling conditions. In addition, when the slumped glass mirror is removed from the mold, the residual stresses will also cause the mirror to deform. Therefore, a critical cooling condition should be presented to satisfy the surface geometry requirement. As shown in previous result, FEM simulation is an effective method to predict residual stress distribution and to calculate the critical cooling condition.

In this part, FEM model will be built to simulate curvature deviation introduced in cooling stage at different cooling rates. To simplify the simulation process, a parabolic surface mold was applied to use the axisymmetric model in the FEM software. Thin sheet soda lime glass with 1.1mm thickness and 100mm diameter and MACOR® ceramic were used as glass material and mold material respectively. Detailed material properties can be found in previous chapters. As investigated in previous parts, residual stresses inside the molded glass components were only determined by cooling condition after slumping process. Since we focus on the curvature deviation caused by residual stresses in this part, the simulation will start from the end of slumping process and only cooling rates were changed in this FEM simulation to isolate other factors. When the slumped glass mirror
was cooled down to room temperature, it was removed from the mold surface as a free object only with a constraint at bottom center to limit its movement. The process is schematically shown in figure 6.15:

![Schematic diagram](image.png)

**Figure 6.15** Curvature deviation caused by residual stress inside slumped glass mirror

<table>
<thead>
<tr>
<th>Structural relaxation parameters of soda lime glass</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Reference Temperature,</strong> $T$ [°C]</td>
</tr>
<tr>
<td><strong>Activation energy/gas constant,</strong> $\Delta H/R$ [°C]</td>
</tr>
<tr>
<td><strong>Fraction parameter,</strong> $x^*$</td>
</tr>
<tr>
<td><strong>Weighting factor,</strong> $w_g$</td>
</tr>
<tr>
<td><strong>Structural relaxation time,</strong> $\tau_v$ [sec] at 640°C</td>
</tr>
<tr>
<td><strong>Stress relaxation time,</strong> $\tau_s$ [sec] at 640°C</td>
</tr>
</tbody>
</table>

* [Jain, 2006]

In the FEM simulation, cooling of slumped soda lime glass workpiece was defined as two-dimensional (2D) axisymmetric model due to simplicity of the mirror geometry using MSC/MARC. In this simulation process, the MACOR® mold was modeled as a rigid body in this simulation, and the glass work piece was defined as the deformable part.
and meshed with 3200 4-node isoparametric quadrilateral elements. Narayanaswamy model was used to simulate the structural relaxation. The material properties of soda lime glass required for structural relaxation was shown in table 6.3. The weighting factor and the structural relaxation time were obtained by fitting the data from equation 1.12 and 1.13. The structural relaxation time \( \tau_v \) at the reference temperature was calculated from stress relaxation time \( \tau_s \) and borrowing the ratio of \( \tau_v/\tau_s = 10.6 \) from reference [Jain, 2006]. The viscosity data was collected from glass vendor and calculated in previous chapter. The ratio, activation energy/gas constant (\( \Delta H/R \)) was calculated from viscosity curve and fitting the curve with Arrehenius equation as:

\[
\eta = \eta_0 \exp\left(\frac{\Delta H}{RT}\right)
\]  

(6.8)

where \( \eta_0 \) is a temperature indendent called the pre-exponential factor.

![Image](image.png)

Figure 6.16: Residual stress inside slumped glass mirror (a) Von Mises Stress with 20s cooling time (b) Von Mises Stress with 1000s cooling time.
The simulation started from the time that the glass began to cool down from 640 °C to room temperature 20 °C. Two load-case stages were applied for cooling stage and mold removed stage respectively. Several different cooling rates were used to investigate the affection of residual stresses for curvature deviation.

Figure 6.17 Curvature deviation under different conditions after cooling stage (a) FEM simulation result after cooling stage (b) Curvature change at the area I of glass mirror after cooling stage for two different cooling rates (c) Curvature deviation at the area I of glass mirror before and after removing from mold support
Figure 6.18 Displacement of edge points at slumped glass mirror under different cooling speeds.

Figure 6.19 Deviation at the edge point of the slumped glass mirror when it was removed from the mold support under different cooling rates.

Figure 6.16 shows the residual stresses inside slumped glass mirror under two different cooling rates. As presented in previous section, faster cooling rate introduce larger residual stresses. The geometrical deviation during cooling stage is shown in figure 6.17a. To see the detail of curvature change, the area “I” was enlarged to shown the deviation at
different cooling conditions. First, we evaluated the curvature deviation for different cooling rates as shown in figure 6.17b. Obviously, due to the structural relaxation of the glass workpiece, different cooling rates will lead to different shapes. Figure 6.18 shows the displacement of a node at the edge of glass mirror along x-axis and y-axis under different cooling rates. To maintain the required curvature change for proper optical performance, a critical cooling rate has to be reached, which can be predicted from the curvature in figure 6.18. For soda lime glass sample used in this slumping test, when cooling time from 640 °C to 20 °C was greater than 200 seconds, curvature change can be considered as constant value. Once the glass workpieces were cooled down to room temperature, they were removed from mold surface. Figure 6.17c shows the curvature difference before and after this process. This type of curvature change is caused by the gravity load and residual stresses. We also simulate this type of curvature change under different cooling rates, shown in figure 6.17. Similar to previous results, when the cooling time was larger than 200 seconds, curvature change can be considered to be a constant value. As we presented before, at low cooling rate, the structure of slumped (molded) glass components would back to the original type as described with simple thermal expansion model. Therefore, for the soda lime glass used in this research, cooling rate of 3 °C/sec is a critical cooling rate for thermal slumping process. Furthermore, as previous research, a faster cooling rate can be applied after reaching the break temperature to save time. In our experiment setup, the cooling rate is far below the critical condition, so we can use uniform temperature distribution cooling model to estimate the curvature deviation in the thermal slumping process.
In addition, we also compared different simulation models in cooling stage with structural relaxation model. First, the linear thermal expansion model was applied with same thermal expansion coefficient used in structural relaxation model. As shown in figure 6.20a, the curvature deviation from structural relaxation result is larger than 60µm. Figure 6.20b shows the result of another approach with structural relaxation model. In this simulation, the entire glass workpiece was cooled down in a uniform temperature distribution instead of convection heat transfer boundary condition. That means no residual stresses will be generated during cooling stage, but the glass still undergo structural relaxation change. As shown in this figure, this approach resulted in error less than 0.05µm at low cooling rates. However, when the cooling rate turns higher, the error will also be larger. In our thermal slumping process, the cooling rate is low enough to use the uniform temperature cooling condition.

![Graphs showing deviation introduced from different cooling model](a) traditional linear thermal expansion model, (b) Uniform inside cooling distribution model
6.4 Conclusion

In this chapter, we evaluate the influence of glass structural relaxation in thermal forming process. First, compression molding of precision optical glass components was successfully modeled with a proper viscoelastic structural relaxation theory using MSC/MARC. The experiments under the same molding conditions were performed on a Toshiba GMP 211V machine at the Fraunhofer Institute for Production Technology in Germany to verify the simulation results. Furthermore, the residual stresses in molded glass lenses were carefully studied by performing both numerical simulation and measurements using birefringence method. In the second section, we investigated the residual stresses in the thermal slumping process by using FEM simulation approach. Different cooling rates were performed and different cooling models were studied. Some of the key contributions of this work are summarized below:

1) FEM simulation can be used to predict the residual stresses of compression molded glass optical components. A reasonable agreement between numerical simulation and experiment results was demonstrated in this research. Therefore the methodology established in this research can be easily adapted to industrial problems.

2) With birefringence and thin layer removal method, the residual stress distribution inside the glass lenses could be measured. The stress distribution can also be explained using numerical simulation. This result further justifies the validity of the numerical modeling for cooling process.

3) The residual stresses in a molded glass lens increase with cooling rate. The amount of
residual stresses and their distribution can be predicted by FEM simulation using the Narayanaswamy model. If all other molding conditions were kept the same, faster cooling rate will result in higher residual stresses. There exists a critical cooling rate for a glass material, if the cooling rate can be kept below this critical level then the residual stresses inside the glass can be controlled to a level low enough for most applications.

4) There exists a break temperature and residual stresses inside a molded glass lens will not change significantly regardless the rate of cooling below this temperature. For BK7 glass used in this research, this temperature is about 520 °C.

5) After compression molding is completed, annealing maybe necessary to remove the residual stresses in the molded glass lenses for applications where certain stress levels are not tolerated. The Narayanaswamy model can also be used to design the annealing process. As suggested the critical cooling rate and the break temperature point are different from glass to glass. However the numerical simulation model investigated in this research provide a clear approach to solving such a problem for different glass materials without doing the actual experiments.

6) By FEM simulation, the thermal slumping process for freeform primary mirror can be optimized. The geometrical deviation at different cooling rates was evaluated. The structural relaxation model can provide more accurate estimation for the curvature compensation of thermal slumping process.
In a freeform concentrating system with freeform optics, there are three different types of tolerance that need to be considered in both design and manufacturing, as shown in figure 7.1. The first one is the scattering of the incident light due to the surface roughness on the primary and secondary optical surface. The second one is the surface contour error of primary and secondary mirrors introduced in manufacturing process. This contour error between actual surface and the theoretical one will cause the angular deviation of reflective rays. The third one is the relative position error of primary, secondary mirror, and receiver during assembling process [Winston, 2005]. For a real concentrating system, to maintain the performance of the concentrators, all three errors need to be controlled within a specified range.

**Figure 7.1** Three different types of tolerance in concentrating system
In this research, since our focus is on the fabrication process of primary and secondary mirror, only the surface roughness and contour error introduced in the thermal slumping process will be investigated. According to the result analysis of surface roughness and surface contour error, the modified acceptance half angle will be presented.

7.1 Surface Roughness

7.1.1 Surface roughness at different temperatures

The surface roughness of the glass workpiece before and after slumping process was measured by AFM to evaluate the slumping results. To test the scattering performance of surface reflection, a thin layer reflective material will be coated on the slumped glass surface. In this research, an aluminum film with 100nm thickness was deposited on the slumped glass upper surface by using an e-beam evaporator (Denton Vacuum EVP501). After deposition, the surface roughness of the coated upper surface was also measured by AFM. Figure 7.2 shows the 3D surface texture of AFM measured result. The surface roughness is calculated by single line scan result (10 μm length) from AFM measurement. Figure 7.2a shows the AFM scanning result of blank glass workpiece before thermal slumping process. Figure 7.2b shows the AFM result of thermal slumped glass upper surface with 640 °C slumping temperature and 120 min holding time. The surface roughness values before and after thermal slumping process are 6.0 nm (R_a) and 19.4 nm (R_a), respectively. Obviously, the surface quality of glass workpiece becomes worse after thermal slumping process than before. However, after coating with 100nm aluminum film, the surface roughness value is improved to 6.9 nm (R_a) as shown in figure 7.2c.
since e-beam evaporation can improve small surface roughness. This surface roughness level is good enough to keep the surface scattering within limitation. The ability of surface reflection at different surface roughness will be measured below.

Figure 7.2 Surface roughness measured result by AFM (a) Original glass blank surface contour (b) thermal slumped glass mirror (c) thermal slumped glass mirror after coating with 100 nm aluminum film

According to previous result in figure 4.11 of chapter 4, whose surface roughness was measured by Mitutoyo S-300 surface profiler along a 10mm line scan, the surface
roughness of slumped glass mirror increased with thermal slumping temperature and holding time. Therefore, the surface roughness of slumped glass upper surface with smaller holding time or lower slumping temperature will be lower than this measured glass mirror. Furthermore, the surface roughness of the slumped glass mirror after coating can be identified as the same level as blank glass workpiece, if the thermal slumping temperature is lower than 640 °C or holding time is smaller than 120 minutes. To evaluate the relationship of surface roughness on final optical performance, surface reflection scattering will be discussed below.

7.1.2 Scattering measurement

When light is scattered from a rough surface, the statistical parameters of the distribution of scattered light may depend on those of the surface roughness [Welford, 1977]. In concentrating optical system, the extent of scattered light is an important parameter since it will affect the system performance. To evaluate the degradation of scattering, the intensity distribution at far field will be measured using an optical detector. The area of scattering surface involved is so large that the speckle pattern formed in coherent light has a Gaussian distribution of complex amplitude, as shown in figure 7.3.

To investigate the effects of different surface roughness on slumped glass mirrors, the optical scattering intensity distribution was measured by a home-built instrument, as shown in figure 7.4 [Li, 2009]. The glass sample is illuminated by a monochromatic He-Ne laser light source. The scattered light is detected by the optical detector (Edmond 53-357, surface area 5.1 mm²). The optical detector is place on a cantilever which is fixed
on the Aerotech ADRT-200 rotary stage so that the detector can rotate with the glass sample. We can adjust the position and direction of laser source and sample to place the laser beam directly over the axis of rotary stage. The sample is oriented so that the normal direction to the sample surface is at an incident angle of 5 degrees to the laser beam. In the measurement, the detector rotates with the sample position from 2 degrees from the sample surface normal to 20 degrees in 0.1 degree increments. The computer can record the angle of rotary stage and the current of detector.

**Figure 7.3** light reflective scattering from surface with certain surface roughness

In the application of freeform concentrator system, we only care about the intensity transform ability of slumped mirror. Therefore, only intensity distribution under different surface roughness was investigated. Once the intensity data at different angles was collected, they were curve fitted by using Gaussian distribution model. We can compare the intensity distribution by comparing the Gaussian parameters. Figure 7.5a shows a series of collected intensity data and the curve fitted Gaussian distribution. After shifting
the intensity distribution to the center, three different parameters were compared to evaluate the extent of intensity under different surface roughness levels e.g. the different thermal slumping conditions, as shown in figure 7.5b. FWHM (full width at half maximum) is an expression of the extent of the curve fitted Gaussian distribution function, at which the dependent variable is equal to half of its maximum value. At the same, the widths containing 99% and 99.9% intensity, which can also be used to estimate the extent of intensity distribution, were also collected from the measured intensity data. Slumped glass sample with different surface roughness under different slumping conditions were measured by using previous method. Measured and curve fitting results were shown in Figure 7.6.

Figure 7.4 Surface reflection scattering measurement setup [Li, 2009]
Figure 7.5 Intensity distribution of surface scattering (a) measured result and curve fitted Gaussian distribution (b) curve fitting result of different samples and three evaluation parameters of intensity extent

There is no significant change in intensity width of FWHM and 99% intensity width for the slumped glass freeform mirrors under different thermal slumping condition after coating from our experiment, as shown in figure 7.6a and 7.6b. The width containing 99.9% intensity distribution was increased steeply when the surface roughness increased.
to 0.13 μm corresponding 660 °C thermal slumping temperature and 120 minutes holing time. The glass workpiece with \( R_a = 0.02 \text{mm} \) surface roughness in this three figures is the blank glass piece directly coated with aluminum film without any thermal treatment. Therefore, we can state that there is no significant impact by scattering when the thermal slumping temperature is lower than 640 °C.

**Figure 7.6** The extent of light intensity distribution at different surface roughness (a) FWHM of curve fitted Gaussian distribution, (b) the width containing 99% intensity (c) the width containing 99.9% intensity.
According to these measurement results, no significant changes in the surface scattering of the glass workpiece before and after thermal slumping when the soaking temperature is controlled under a certain level (can be empirically determined and numerically modeled). When the soaking temperature is increased to 660 °C, the surface roughness of the upper free surface of slumped glass mirror is 0.13 μm ($R_a$) which will increase the extent of surface scattering intensity distribution. Therefore, to keep the surface scattering extent under the required level, the soaking temperature should be controlled below 660 °C. At the same time, the soaking temperature should be high enough to allow the glass workpiece slump down to the mold surface in a reasonably short amount of time for production, as presented in chapter 4.

7.2 Surface Contour Error

7.2.1 Position contour error and angular contour error

When an incident light ray is reaching the reflector, there are two types of contour error will affect the reflected direction: position contour error and angular contour error (slope error). For optical purpose, position error is irrelevant and only slope error should be considered. However, the position error can also be transmitted to an apparent slope error, which is comparable with the real slope error. Figure 7.7 shows the contour errors of a reflective surface including position error and slope error. In this figure, $D$ is the final deviation angle, $S$ is the slope error and $E$ is the position error. Obviously, the relationship between position error $E$ and slope error $S$ can be expressed as: $S(s) = \ldots$
\[ dE(s)/ds, \ s \text{ is the arc-length parameter of the theoretical profile.} \] By these relationships, we can obtain the total angular deviation expressed as [Winston, 2005]:

\[ D = m \left[ S - E \tan \theta_o \left( \frac{1}{R_c} + \frac{\cos \theta_i}{R_i} \right) \right] \tag{7.1} \]

Where \( \theta_i \) and \( \theta_o \) are angle of incidence and angle of reflection respectively, and \( R_i \) and \( R_c \) are the respective radii of curvature of wavefront of incidence and reflection light. \( m \) is a multiplier for the angular deviation, is given by:

\[ m = \begin{cases} 
2 & \text{(mirror)} \\
1 - \frac{\tan \theta_o}{\tan \theta_i} & \text{(dioptric)}
\end{cases} \tag{7.2} \]

\[ \text{Figure 7.7 Contour errors of a reflective surface} \]

For the freeform concentrator system in this design, the incident rays are coming from the sun, which can be considered as plane wavefront, therefore \( R_i \to \infty \) in this condition. Then, the equation 7.1 could be simplified as:

\[ D = m \left[ S - E \tan \theta_o \frac{1}{R_c} \right] = mS - mE \tan \theta_o \frac{1}{R_c} = mS - mS_E \tag{7.3} \]
Where the first part of the right hand side is the angular deviation introduced by slope error $S$, and the second part is the angular deviation introduced by position error $E$.

In the manufactured glass mirrors, the position error at each point can be measured using coordinate measurement machine (CMM). The measured values present a stochastic probability distribution. When the glass mirrors are slumped under different manufacturing conditions (temperatures, holding times), different error probability functions will be extracted from measurement results. In most cases, the probability distribution of position error and the slope error can be presented by Gaussian distribution (normal distribution), respectively. Figure 7.8a shows the position error of slumped glass with a 640 °C slumping temperature and 120 min holding time. Figure 7.8b shows histogram and curve fitted Gaussian distribution of the position error.

With the position error information, we can calculate the apparent slope error transmitted by these position errors using equation 7.4:

$$S_E = E \tan \theta_o \frac{1}{R_c}$$  \hspace{1cm} (7.4)

The $R_c$ at each position can be calculated from the freeform primary design, as well as the reflective angle $\theta_o$ of the primary exiting rays. The position error map between design and manufacturing result is obtained from measurement. When the manufacturing conditions consist of 640 °C slumping temperature and 2 hours holding time, the average position error is 0.000060mm and the standard deviation is 0.02881mm. The position errors can be curve fitted by a Gaussian distribution $N(0.000060, 0.028881^2)$. That means that for any point of the slumped glass mirror, which has a 90% chance, will have an
error within the region of ±1.65σ, or in the region of [-0.0474, 0.0476]mm. Therefore, for the angular deviation $S_E$ caused by position errors can also be presented by using a statistic distribution function.

Figure 7.8 Position error of slumped glass mirror (a) Position error map of slumped freeform glass primary mirror (b) histogram and curve fitted Gaussian distribution of position error
According to above calculation, the probability density function of angular deviation $S_e$ in mrad unit can be curve fitted by Gaussian distribution $N(0,0.0275^2)$. This means that for any point on the slumped glass mirror, with a 90% chance, will have a position error caused slope error is within the range of $\pm0.0454$ mrad or $\pm0.0026^\circ$. Compared with angular deviation introduced by slope error mentioned above, the position error can be neglected.

7.2.2 Find slope error

CMM can give the coordinates of each point on the slumped glass mirror surface. We can calculate the slope error by compare two normal direction of designed and measured surface curvature at each corresponding point pair. To find the normal direction of each point, two different methods can be applied. Linear interpolation method is easy and fast process, however the calculation error of this method is large. Since the slope error of the slumped glass is less than the level of milliradian, the linear interpolation method will introduce significant calculation error into the result.

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**Figure 7.9** Calculating normal direction by spline surface
In this research, a more accurate spline-surface method was employed to get the surface normal direction at each measured point, as shown in figure 7.9. The normal direction at point \( \theta \) was calculated from the spline fitted surface from neighbor points 1 to 8.

**Figure 7.10** Slope error of slumped glass mirror (a) slope error map of slumped freeform glass primary mirror (b) histogram and curve fitted Gaussian distribution of position error
Once the normal direction of each measured and designed position was found, the differences between them were the slope errors. Figure 7.10a shows the slope error map of slumped glass with 640 °C slumping temperature and 120 min holding time. Figure 7.8b shows histogram and curve fitted Gaussian distribution of the slope error. The slope errors can be curve fitted by a Gaussian distribution $N(-0.0181, 0.3063^2)$. It means that for any point on the slumped glass mirror, with a 90% chance, it has a slope error within the region of ±0.5054°.

### 7.2.3 Error transfer

For a single surface, the surface contour error can be characterized by the probability distribution function of the slope error and position error. For an optical system with two or more optical surfaces, there is no analytical theory available to quantity the effect of the errors. However, we can transfer the errors of one surface to another surface, leaving the first surface error free and increasing the surface errors of the next one. To transfer error from one surface to another surface, two restricted conditions must be satisfied [Winston, 2005]:

I: The deviation angle at each point of optical surface should be smaller than the acceptance angle at that point.

II: The deviation angle of the transmission ray between two surfaces should be small enough to make it at same level at central or at the edge ray.

If we define $D(SS)$ as the angle deviation caused by slope error at secondary mirror, $D(PP)$ as the angle deviation caused by slope error at primary mirror, and $D(PS)$ as the
transferred deviation angle from surface $S$ to surface $P$, which can be expressed as equation 7.5:

$$D(PS) = \frac{\delta_P}{\delta_S}D(SS)$$  \hspace{1cm} (7.5)

where $\delta_P$ and $\delta_S$ are angles from reflected ray to edge ray of surface $P$ and surface $S$ respectively. This equation means that the deviation angle at $S$ (secondary mirror) can be transferred as an apparent deviation angle at $P$ (primary mirror). Usually the ratio $\delta_P/\delta_S$ can be approximately estimated by $\beta_P/\beta_S$. $\beta_P$ and $\beta_S$ are the local acceptance angle of the concentrator bundle at a given point of primary and secondary mirror respectively. In this freeform concentrator design, the half acceptance angle of the primary mirror is designed as $1.2^\circ$, and the one of secondary can be calculated by geometry and relative position of primary and secondary as $3^\circ$. There the ratio $\delta_P/\delta_S$ is $2/5$ in this design. Then, the total angle deviation at surface $P$ can be expressed as the sum of local angle deviation and transferred angle deviation, as shown in equation 7.6:

$$D(P) = D(PP) + D(PS) = D(PP) + \frac{\delta_P}{\delta_S}D(SS)$$  \hspace{1cm} (7.6a)

$$D(P) \approx D(PP) + \frac{2}{5}D(SS)$$  \hspace{1cm} (7.6b)

Obviously, the manufacturing errors at the secondary mirror surface are attenuated when transferred to the primary surface. Assuming that the primary and secondary mirrors are fabricated using same slumping process and have same contour error probability distribution, the errors on the primary mirror will dominate the deviation of whole system.
7.2.4 Total contour error estimation

From previous calculation, we can state that the probability density function (pdf) of total angle deviation can be given by the convolution [Winter, 1991]:

\[ f(\theta) = f_{D(PP)}(\theta) \otimes f_{D(PS)}(\theta) \] (7.7)

So, the standard deviation of the total deviation angle can be estimated from the standard deviation of each surface:

\[ \sigma_{D(P)} = \sqrt{\frac{\sigma_{D(PP)}^2}{2}} \left( \frac{2}{5} \right)^2 \sigma_{D(SS)}^2 \] (7.8)

As we stated in the previous section, the slope errors of thermal slumped glass mirror has a Gaussian distribution with standard deviation \( \sigma_{S(P)} = 0.3063 \). Therefore, the standard deviation of angle deviation can be estimated by \( \sigma_{D(PP)} = m\sigma_{S(P)} = 0.6126 \). Hense, the total system standard deviation of angle deviation is 0.6598 calculated from equation 7.8.

After transferring all contour error to the primary surface, we can treat this angle deviation as the sun incident light coming in with an angle distribution to normal direction. Therefore, the angle deviation will reduce the designed acceptance angle. The modified acceptance angle can be derived from the cumulative distribution function of the Gaussian distribution of slope deviation, as shown in equation 7.9. Then, the ensemble average of the acceptance angle with errors with 90% transmission can be estimated from equation 7.10 [Tuma, 1979]:

\[ \Phi(x) = \frac{1}{\sigma_D \sqrt{2\pi}} \int_{-\infty}^{x} \exp\left(\frac{(t - E(\alpha))^2}{2\sigma_D^2}\right) dt \] (7.9)

\[ E(\alpha) = \alpha - 1.65\sigma_D \] (7.10)
The standard deviation of angle deviation in our designed concentrating system is 0.6598, and the designed half acceptance angle is 1.2°. Then the modified acceptance angle will be 0.1114°. This acceptance angle will require a very accurate assembly and tracking system. From figure 7.10a, we notice that the most large angle deviations are on the edge area of the slumped glass mirror, so we can cut edge from the manufactured glass mirror to get smaller standard deviation of slope error. Besides, we can improve the fabrication process to get more accurate surface and lower angle deviation by higher order curvature compensation. We can also modify our optical design with a larger acceptance angle and reduce the “jump” between small parabolic segments.
CHAPTER 8: PRECISION COMPRESSION MOLDING OF GLASS MICRO OPTICS

8.1 Introduction

8.1.1 Diffractive optical elements

Diffractive optical elements are increasingly playing an important role in high-speed data transmissions and other optical signals to electrical signals converting system [Wang, 1998]. The basic concept of a DOE relies on constructive and destructive interference of coherent light beams to produce the desired illumination geometry. For example, when two neighboring parallel slits are illuminated by a collimated light beam (e.g., planar wavefront of a laser) the slits become the sources of two parallel coherent beams. As the light propagates away from the slits, both act as independent light sources and the light from each slit propagates in a spherical shape. At a faraway distance, the two spherical waves interact with each other. In order to design the 2-dimensional diffractive patterns, an Inverse Fast Fourier Transforms (iFFT) of complex arrays is used. For example, in order to design a mask to produce a square pattern, the image is constructed as a bitmap graphics based on the intensity mapping of the image. The image is then inverted from the center to the corners, and an iFFT is performed on the inverted image based on equation 8.1 [O’Shea, 2004].
\[ F(f_x, f_y) = \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} f(x, y)e^{-j2\pi(f_xx+f_yy)}\,dx\,dy \]  

(8.1)

where \(f(x, y)\) is a function that includes the complex values of the original bitmap image (inverted) at the locations \(x\) and \(y\), and \(F(f_x, f_y)\) is the phase domain of the image (complex amplitudes) of the bitmap at locations \(x\) and \(y\). \(f_x\) and \(f_y\) are spatial frequencies. The basic setup for beam shaping with diffractive elements is shown in figure 8.1.

**Figure 8.1** Optical performance test setup for glass diffractive optical elements

Figure 8.2a shows an 8-level 128x128 diffractive grating designed by using simulated annealing algorithm after \(10^6\) iterative with a home-build MATLAB program. The calculation can give the phase shift result at each small segment. By the refractive index information, we can easily get the depth distribution of this optical grating. Figure 8.2b shows the numerical simulated result of this diffractive grating by using equation 8.1. As shown in this figure, DOEs can realize the beam shaping purpose with pre-defined design.
Figure 8.2 (a) An 8-level 128x128 diffractive grating (b) numerical simulation result of far-field diffraction of the designed grating
One of the difficult issues related to DOE fabrication is manufacturing cost. Although direct fabrication using photolithography can be used to massively produce these elements, the cost remains high due to the complex procedures involved in the mask making and the following lithography process. Furthermore, there are concerns about the chemicals that are used in photolithography and about limitations on optical material selection due to strict requirements for cleanroom micromachining process. Injection molding of plastic optical elements allows substantial reduction in manufacturing cost but plastic optical elements lack in performance compared to glass optical elements.

Currently, glass diffractive optical elements are either fabricated using cleanroom micromachining technology or a direct ultraprecision machining process [Wang, 1998; Yi, 2003]; therefore, the manufacturing cost remains very high. The method discussed in this chapter is different from traditional fabrication processes, where instead of direct manufacturing; a replication method based on the compression glass molding will be presented. In previous publications, an experimental study and numerical simulation of compression glass molding process have been described [Yi, 2005]. It was shown that various sizes of diffraction limited aspherical glass lenses can be molded using compression molding technology. However, very limited attempts have been made toward making micro optical and diffractive optical elements where glass molding maybe an excellent choice.

The process reported in this chapter was focused on diffractive optical element fabrication but it has the potential to be adapted to a wide range of other applications requiring molding with features in the micro-scale. For example, more affordable
microfluidic devices made out of glass materials can be used when corrosive fluids are used and where neither plastic nor silicon is suitable. In such a situation, glass materials are a better option because glasses are naturally corrosion resistant. Furthermore, this process can be part of a manufacturing process for microelectronic devices where micro and diffractive optical elements can be directly fabricated and integrated into an electronic circuit thus lowering the overall manufacturing cost.

8.1.2 Microlens array

Microlens array is another micro-optical element which has wide application in optoelectronic and optical communication [Hutley, 1991]. Daly et al initialized a thermal reflow process to fabricate microlens arrays by melting photoresist cylindrical islands on the substrate [Daly, 1990]. The surface tension made the photoresist form spherical geometry of the microlens arrays when processing temperature reached a level above the transition temperature. Shen developed a novel method to microlens arrays mold insert by thermal reflow and electroforming [Shen, 2006]. This mold insert was used to fabricate polymer microlens arrays by hot embossing. Chang et al investigated a high volume roller embossing method to fabricate polymer microlens arrays [Chang, 2006]. They made the mold insert by electroforming process. These listed methods could be used to fabricate polymer microlens array at very low cost and high production rate. Compared with microlens arrays using polymer materials, glass material are more reliable due to its high transition temperature, chemical stability and good mechanical properties [Ottevaere, 2006]. Therefore, glass microlens arrays can be used in applications in harsh environments such as high temperature conditions. There are

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already many methods that had been developed to fabricate glass microlens arrays. Wakaki et al proposed excimer laser ablation method which could be used to fabricate microlens arrays directly on glass substrate [Wakaki, 1998]. Savander used the directly RIE etching process to fabricate microlens by glass and silicon materials [Savander, 1994]. However, the aforementioned processes are not yet efficiently mass manufacturing method for glass microlens. Compared with these fabrication processes, glass compression molding is an emerging technique that can be adopted for high volume fabrication of precision glass optical elements. This process is also suited for high transition temperature \( T_g \) glass microlens arrays.

Thermal reflow is a commonly used method for fabricating polymer microlens arrays due to its low cost nature, high productivity and simplicity [Daly, 1990]. In this process glass material is melted when processing temperature reaches a level above its transition temperature and surface tension of the molten material forces the formation of spherical geometry of the microlenses. In this chapter, a hybrid fabrication method for glass microlens arrays was developed by combining glass molding and thermal reflow process.

### 8.2 Fabrication Process

#### 8.2.1 Mold fabrication

Glassy carbon is used as glass mold material for its excellent chemical stability at high temperature and its optical polishability. The operating temperature of glassy carbon can reach up to 2,000 °C. In addition, glassy carbon can be easily fabricated using lithography and reactive ion etching (RIE) process. Furthermore, glassy carbon will not
stick with glass workpiece at high temperature without any coating. These properties make glassy carbon suitable as a mold material for fabricating DOE and microlens array. Besides glassy carbon, fused silica and quartz are also used as mold materials for glass compression molding of low $T_g$ glass.

The glassy carbon mold was fabricated by using cleanroom micro-fabrication process. To start, glassy carbon wafers of 2 mm thick, 50.8 mm diameter (manufactured by HTW Hochtemperatur-Werkstoffe GmbH, Gemeindewald 41, D-86672 Thierhaupten, Germany) were fabricated using micromachining process and followed with reactive ion etching as shown in Figure 8.3. The dark area was glassy carbon wafer. The photoresist (shown in shadow) was Shipley S1813 and was deposited with a thickness of 1.4 μm. The wafer
was then exposed to UV radiation for 3 seconds at a density of 15 mW/cm\(^2\) and 365 nm wavelengths, and then was developed for 1.5 minutes using MF319 developer and rinsed with water. After development, a Ti/Ni metal layer (20/50nm) was deposit on the glassy carbon wafer as the etching mask. Acetone was employed as the lift off material to remove photoresist layer unnecessary metal part. A bench top RIE(Reactive Ion Etching by Technics Micro-RIE Series 800II) unit was used for reactive ion etching of the wafer. The pressure during etching was 150 mTorr with an RF power of 250W. The oxygen flow rate during etching was 20 cm\(^3\)/minute. The etching rate is about 186 nm/min in this condition.

![Fabricated molds by micro-fabrication process with 10μm line width: (a) diffractive lines on glassy carbon mold measured by SEM, (b) diffractive lines on glassy carbon mold measured by AFM](image)

**Figure 8.4** Fabricated molds by micro-fabrication process with 10μm line width: (a) diffractive lines on glassy carbon mold measured by SEM, (b) diffractive lines on glassy carbon mold measured by AFM

Different etching rates can be achieved at different conditions. Different molds with several etching depth were applied for future compression molding test with different
etching time. For the DOEs compression test, glassy carbon was etched about 15min with 2.8μm depth by using listed etching condition, and for microlens array, the etching depth is about 10μm after 60min. The remaining metal mask was removed by acid solution after etching. Besides glassy carbon mold, fused silica wafer can be etched by using He/CHF3/CF4 (120/40/45 cm³/min) drying etching with 0.53cm gap, 600W RF power and 2 Torr pressure at a LAM490 dry etching machine. The etching rate at this condition is about 220nm/min.

![Etched patterns](image)

**Figure 8.5** Fabricated glassy carbon molds measured by SEM, (a) shows a pattern of the micro holes array with 100 μm diameter and 100 μm pitch distance, (b) shows an individual micro hole on the glassy carbon mold.

The glassy carbon molds were measured for geometric and surface finish. Figure 8.4 shows the SEM and AFM images of glassy carbon fabricated as the mold for diffractive optical elements. Figure 8.5 shows the SEM images of the glassy carbon molds for microlens array, and figure 8.6 is the cross sectional view of the glassy carbon mold.
measured by Taylor Hobson PGI 1250 profilometer along the diameter direction. To fabricate the microlens arrays used in this research, glassy carbon molds with 100 μm diameter micro holes were etched to 10 μm deep as shown in figure 8.6. The pitch distance was also 100 μm in this mold design.

![Graph showing height vs. radius](image)

**Figure 8.6** Glassy carbon molds measured by Taylor Hobson profilometer. The result shows that the diameter is 100 μm, and the distance is 10 μm.

### 8.2.2 Compression molding

After the glassy carbon molds were completed, the glass compression molding experiments for micro glass optical components were performed on a Toshiba GMP 211V machine at Fraunhofer Institute for Production Technology (IPT), Aachen, Germany. The schematic in figure 8.7 illustrates four major steps of the process. The glass blanks used in this experiment were optically polished on both sides. The shaded component in the figure is the glass blank.
The compression molding process began with heating of the entire mold assembly and the glass blank to the selected forming temperature (565 °C P-SK57, 685 °C for BK-7, 325 °C for K-PG325). The lower mold was then pushed upward at a velocity of 0.5 mm/min to the forming position. After reaching the molding temperature, the entire system was kept for 300 seconds (soaking time) to ensure uniform temperature distribution inside the machine. The molding force was kept constant at 2 kN for 40 seconds to ensure complete contact was made between the glassy carbon mold and the glass workpiece. A 200 N constant load was applied from start of the cooling stage. The applied load was removed after cooling for 40 seconds. Once the mold and glass temperature was lowered to approximately 200 °C, the glass workpiece was released and cooled to room temperature naturally. In precision compression molding test, different parameters were tested to optimize the compression molding including soaking temperature, holding time, and compression load.

Figure 8.7 Schematic illustration of glass compression molding process i.e., heating, molding, cooling, and release.
One example of process parameters were shown in figure 8.8 to show the temperature history and load for P-SK57 glass molding process. During this experiment, vacuum was applied at the beginning to ensure that no air remained in the gap between the mold and glass workpiece. This was immediately followed by nitrogen purge to further remove oxygen residual. During the cooling stage, the nitrogen flow rate was adjusted to control cooling speed.

![Molding temperature and press load time history for glass microlens array, with 565 °C molding temperature, 300 seconds soaking time, 2 kN molding force.](image)

**Figure 8.8** Molding temperature and press load time history for glass microlens array, with 565 °C molding temperature, 300 seconds soaking time, 2 kN molding force.

### 8.2.3 Thermal Reflow

To fabricate glass microlens array, after the molding process was completed, the glass workpiece with micro cylinders was re-heated to temperature that was higher than its glass transition temperature and was kept at this temperature for a pre-determined amount of time (400 seconds in the experiment performed at 600 °C) to achieve the desired surface curve shape as shown in figure 8.9. A controlled cooling was performed (same as
in molding process). At the soaking temperature, glass behaves as a viscoelastic material, which is strongly time and temperature dependent.

![Diagram of glass microlens array fabrication process]

**Figure 8.9** Illustration of the glass microlens array fabrication process, including three major steps i.e., fabrication of molds, glass compression molding, and reheating.

![Temperature time history graph]

**Figure 8.10** Temperature time history for reheating process with 600 ºC reflow temperature and 400 seconds holding time.

Microlenses with precise surface geometry could be created at different soaking temperatures (which affect surface tension) and soaking times. In this research, reheating
temperatures from 560 °C to 620 °C were selected and different soaking times were performed to evaluate the effect of process parameters. Figure 8.10 shows a temperature history for reheat thermal reflow process under 600 °C reheating temperature and 400 seconds soaking time.

8.3 Result and Discussion

8.3.1 Geometric measurement

8.3.1.1 Diffractive optical elements

The molded DOEs were measured using SEM and AFM, as shown in Figure 8.11. To make a valid comparison, AFM scans were performed on the same area on the glassy carbon mold and molded Bk7 glass DOEs. Figure 8.12 shows the comparison between the glassy carbon mold and the molded BK7 DOEs’ line scans under the molding condition. The results show good replicability between glassy carbon mold and molded. The surfaces of molded glass are smoother than glassy carbon due to the surface tension. This discrepancy could be controlled by changing molding conditions such as molding load, holding time and molding temperature. To study the influence of these parameters in glass DOEs molding, different conditions were tested.
Figure 8.11 Molded glass DOEs with 10μm line width: (a) diffractive lines on molded glass measured by SEM, (b) diffractive lines on molded glass measured by AFM

Figure 8.12 Curve comparison between mold and molded glass

The figure 8.13 shows the average of the peak-to-valley distance on the molded DOEs changed with different molding temperature, molding load, and different diffractive line dimensions. At certain conditions, increasing molding temperature or load could improve the replicability of molded glass. And obviously, replicability for larger diffractive line
dimension is much better than smaller one. However, higher temperature and load could introduce more serious mold wearing and sticking.

Figure 8.13 Molding results under different conditions: (a) holding temperatures, (b) loads, (c) and diffractive lines dimension
8.3.1.2 Microlens array

The glass microlens arrays fabricated using thermal reflow were measured by SEM and Veeco NT3300 optical profiler to evaluate fabrication results. To observe the surface morphology using SEM, a thin layer of gold was sputter coated on the glass substrate for 2 minutes under 20 mA current using Emitech K550X coater (Empdirect Products Inc., 704/17033 Butte Creek, Houston, TX 77090). Figure 8.14 shows the SEM images of the microlens array after the thermal reflow step.

Figure 8.15 shows the Veeco scan of the surface geometry for a microlens along the diameter direction after thermal reflow at 610 °C with 400 seconds soaking time. The dotted line is the curve measured in the experiment and the solid line is the results of curve fitting for a spherical shape of 830 μm radius. The microlens itself is about 150 μm in diameters and its height is about 2.89 μm.

![Figure 8.14 SEM images for fabricated microlens array on glass substrate, after thermal reflow at 610 °C with 400 seconds soaking time](image)

(a) (b)
Figure 8.15 Surface geometry of a microlens after thermal reflow (610 °C and 400 seconds). The circle dot line is the measured data from Veeco scan, and the solid line is the curve fitting for spherical shape of 830 μm radius.

Because the viscosity of glass material is strongly temperature dependent, different thermal reflow temperatures were tested to study the influence of temperature on surface curve change. Figure 8.16 shows the surface curve comparison of microlens arrays fabricated at different reflow temperatures with same soaking time. At a higher reheating temperature, glass material had a lower viscosity. Therefore a lower height of microlens array (sagittal value) was expected as in figure 8.17. When the reflow temperature was 560 °C or lower, there was almost no change in the surface shape from the molded glass micro cylinders. On the other hand, when the reflow temperature was more than 620 °C, the shape of the microlenses became effectively flat. Therefore, for P-SK57 glass, the reheating temperature for thermal reflow process should be controlled between 600 °C and 610 °C as shown in figure 8.17.
Figure 8.16 Surface geometry at different thermal reflow temperature. Four different results at different reflow temperatures are shown in this figure i.e., $T_1 = 560 \, ^\circ C$, $T_2 = 600 \, ^\circ C$, $T_3 = 610 \, ^\circ C$, $T_4 = 620 \, ^\circ C$.

Figure 8.17 Radius and height of microlenses at different thermal reflow temperature. With the increasing of reheating temperature, the radius of microlens increases and the height of microlens decreases.
8.3.2 Optical measurement

8.3.2.1 Diffractive optical elements

The experimental setup used to analyze the optical image formation was also similar to the system shown in figure 8.1. For this test, a more stable HP-5517B He-Ne laser was selected (Hewlett Packard, now Agilent Technologies, Inc., 395 Page Mill Road, Palo Alto, CA). The HP-5517B laser can provide a continuous beam of 1 mW peak power at wavelength of $\lambda = 632.99137$ nm. In this optical measurement, fused silica mold and molded glass DOEs with it were measured, since there are both transparent and have similar refractive index. Both the fused silica mold and the molded DOE were placed at about 350 mm away from the laser. The diffraction patterns were projected onto a Charge Coupled Device (CCD) line scan camera that was mounted 355 mm away from the DOE (Model 1000 line scan camera Entwicklungbüro, Reinholdstraße. 5 D-12051 Berlin, Germany). The 1000 model is equipped with a Sony line scan CCD sensor ILX553. The CCD sensor has 5,150 pixels, each pixel has a sensing area of 7x7 $\mu$m$^2$. Figure 8.18a is the schematic of the optical testing system. To scan an entire image, the rotary table (Soloist single-axis rotary stage by Aerotech, Inc., Pittsburgh, PA) was operated through a PC to move at a fixed speed. The onboard circuit of the CCD camera system allows a line scan to be acquired at each stoppage at 16 kHz and the results were uploaded to a PC as illustrated in figure 8.18b. In figure 8.19, the diffraction patterns from both the fused silica mold and glass DOE were plotted. For clarity only one line scan for each component was used. The dashed line represents the diffraction pattern from the DOE and solid line from the mold. A 15 point moving average was performed to both scans to
remove high frequency noise. It appears that about 20% less energy was projected by the molded DOE as compared to the fused silica mold.

**Figure 8.18** Schematic of optical performance test setup

**Figure 8.19** Optical performance test results for glass diffractive optical element and fused silica mold.
8.3.2.2 Microlens array

To investigate the performance of the fabricated microlens arrays, the focal lengths of the microlenses were measured by using a system shown in Figure 8.20. In the test setup, a He-Ne laser (wavelength 632.8 nm) was employed as the light source and a charge coupled device (CCD) was used as an image collector. The fabricated microlens array was placed on a precision stage, which can be moved along the optical axis direction. First, the microlens array was manually moved to a position where the focus was on the flat surface of the microlens array workpiece (Figure 8.21a). Then the stage was adjusted to move the microlens array away from the CCD camera until sharp focused spots were detected by the CCD camera (Figure 8.21b). Figure 8.20 shows the image and focused light spots detected by the CCD camera for one of the glass microlens arrays, which was fabricated using thermal reflow at 610 °C with 400 seconds holding time. The distance between these two positions gives the focal length of the microlens array.

**Figure 8.20** Optical test setup for measuring the focal length of a fabricated microlens array.
Figure 8.21 Images of light spots array produced by microlens array at focal plane. (a) is the image of CCD camera focused at microlens array surface, and (b) is the imaged focused at focal point. The distance between these two focal surfaces is the focal length of microlens.

The focal length of the microlens array can be determined approximately by using radius of curvature \( R \) and refractive index of P-SK57 glass with the equation below:

\[
f = \frac{R}{n-1}
\]  

(8.2)

The refractive index of P-SK57 for 632.8 nm wavelengths is 1.5849 according to the data sheet from Schott Glass [Schott, 2009]. The calculated radius of curvature after thermal reflow (610 °C and 400 seconds) is 830 μm according to the curve fitting results as shown in Figure 10. Therefore, the focal length of this microlens was 1.42 mm by using the equation 8.2. The measurement result of focal length using above method is 1.75 mm. Figure 8.22 shows comparison between the measurement results and theoretical calculation with equation 8.2 under different thermal reflow temperature. The difference
between measurement and theoretical calculation may come from measurement errors in focal length and geometrical curvature measurement.

![Figure 8.22](image)

Figure 8.22 The focal length comparison between optical measurement and theoretical calculation under different thermal reflow temperatures.

### 8.4 Numerical Simulation of Reflow Process

Reflow of a molten glass article can be driven by gravity and/or surface tension. The relative importance of each factor depends on the size of the flow domain. To compare the two effects, one may conduct an order of magnitude analysis of a fluid domain with a characteristic dimension of $D$. The work done by surface tension can be estimated to be

$$\Delta S = D^2 \gamma$$  \hspace{1cm} (8.3)

where $\gamma$ is surface tension, and the work done by gravity can be estimated to be

$$\Delta G = D^4 \rho g$$  \hspace{1cm} (8.4)
where \( \rho \) is density and \( g \) is gravitational acceleration. Therefore the relative importance of these effects can be compared using a dimensionless group defined as

\[
S_g = \frac{\Delta S}{\Delta G} = \frac{\gamma}{D^2 \rho g}
\]  (8.5)

In microlens molding, the characteristic dimension is on the order of 10 \( \mu \)m, resulting in an \( S_g \) on the order of \( 10^5 \). Therefore only surface tension needs to be considered in the reflow process for microlens.

Isothermal surface tension driven flow can be approximated using a creep flow model. In this case, the material is considered to be a purely viscous material, and compressibility is neglected. The simplified conservation equations are:

\[
\nabla \cdot \mathbf{v} = 0 \quad \text{(8.6a)}
\]

\[
-\nabla p + \nabla \cdot [\eta (\nabla \mathbf{v} + (\nabla \mathbf{v})^T)] = 0
\]  \( \text{(8.6b)} \)

where \( p \) is pressure, \( \eta \) is viscosity and \( \mathbf{v} \) is a velocity vector.

The geometry and the boundary conditions used in the reflow model are shown in Figure 8.23. It is assumed that the surface tension induced deformation is local to the microlens. The fluid domain (i.e., glass) is axisymmetric. Due to this axisymmetry, only half a cross-section is shown in the model. The boundary conditions on the symmetries are zero normal velocity and zero tangential stress, i.e., \( v_n = \hat{n} \cdot \mathbf{v} = 0 \) and \( f_s = \hat{n} \cdot \mathbf{\sigma} \cdot \hat{s} = 0 \), where \( \hat{n} \) and \( \hat{s} \) are unit normal and tangential vectors and \( \mathbf{\sigma} \) is the total stress tensor. On the free surface, surface tension causes a net normal stress on the surface, indicated as
\[ f_n = -\frac{\gamma}{\nabla \cdot \hat{n}} \] where \( \nabla \cdot \hat{n} \) is curvature. The governing equations and boundary conditions were implemented using Polyflow, a commercially available finite element analysis software package for polymer/glass molding and forming. The mesh size and time step used were 1 \( \mu \)m and 0.01 s. Further refinement of these parameters did not change the accuracy of the solution.

**Figure 8.23** Surface tension driven reflow model. In the model, the dimensions along the \( x \) and \( y \) axes have different scales; along the \( x \) axis, \( \text{FE} = 150 \mu \text{m} \), and along the \( y \) axis, \( \text{AF} = 20 \mu \text{m} \).

The dimensions of the compression molded micro cylinders, about 10 \( \mu \)m in height and 100 \( \mu \)m in diameter with a slope on the side, were taken as the dimensions for the initial geometry in the model. The typical surface tension of molten glass is approximately 0.3 N/m [Zhilin, 2003]. The viscosity of molten P-SK57 glass was previously characterized,
governed by the previous equation 4.4. Where \( A, B, \) and \( T_0 \) are model coefficients and for this material are -3.556, 2917.3, and 306.4, respectively. The calculated viscosity at 610 \( ^\circ C \) is \( 10^6 \) Pa-s. However, the simulation results with these parameters did not agree with the experimental observations. Instead, a good agreement was obtained at a slightly lower viscosity of \( 3 \times 10^5 \) Pa-s. The simulated reflow for \( \eta = 3 \times 10^5 \) Pa-s and \( \gamma = 0.3 \) N/m at different time is shown in Figure 8.24.

![Figure 8.24](image)

**Figure 8.24** Simulation results showing the evolution of the microlens geometry during surface tension driven reflow.

At these parameters, reflow would occur at a similar time scale as observed in the experiments, as shown in Figure 8.25 for a reflow process at 610 \(^\circ C\) and 400 seconds holding time. This discrepancy may be explained considering the deviation of the actual temperature inside the mold, i.e., the temperature measured outside the mold cavity could be significantly lower than the actual molding temperature. From equation 4.4 the
temperature corresponding to $\eta = 3 \times 10^5$ Pa-s is approximately 630 °C. The underestimated glass molding temperature predicted a higher viscosity and consequently a slower reflow process. Furthermore, the surface tension used in the simulation was an estimate from the literature and therefore could bring in additional calculation errors.

![Graph showing comparison between simulation and experimental results.](image)

**Figure 8.25** Comparison between simulation and experimental results.

### 8.5 Conclusion

Through design, fabrication and measurement of a compression molded glass DOEs, by using glassy carbon mold and fused silica mold, we have demonstrated that compression molding can be a promising process for high volume production of precision DOEs with micro and nano scale features. Geometrical and optical characterizations of the molded DOEs were demonstrated using different measurement techniques. The proposed strategy for DOE fabrication has several inherent advantages over existing technologies. First, it is flexible in that there are several hundred optical glasses available versus only a few
optical plastics. Second, the molded DOEs can preserve the optical quality of the mold surfaces; therefore, no post-molding processes are needed, i.e., it is a net shapemanufacturing process. Third, the compression molding process maybe integrated with other semiconductor processes due to its low molding temperature. Fourth, this method can be applied to fabricating many different optical components thus making it a very powerful tool to mass produce various optical elements at low cost, which opens the door for enhanced and more affordable optical systems. Finally, the compression molding process can be used to fabricate other components with micro and nano scale features at an affordable cost, e.g., microfluidic devices for biomedical applications.

Besides, from design, fabrication and measurement of P-SK57 glass microlens array, a new method for high volume production process was presented by combining glass compression molding and thermal reflow process. By controlling reheating temperature and soaking time, microlens array with different radii and focal lengths were fabricated. A numerical simulation was performed to evaluate and characterize the fabrication process. Furthermore, the quality of the glass surface geometry was measured using a mechanical stylus profilometer and the focal lengths of fabricated glass microlens arrays were measured using an optical setup. This method is based on glass compression molding therefore making it a very useful tool to mass produce various micro glass optical elements at a low cost.
A.1 Introduction

Accurate 3D microstructures on silicon or other wafer substrates are very important for optical, electronic and many other MEMS devices. Traditional cleanroom lithography and etching process are essentially a 2D method, although complicated procedures were designed to create some 3D microstructures. These processes however are mainly used to create planar features on silicon wafer substrate using bulk silicon machining technique [Kovacs, 1998]. In the past several decades, special techniques have been developed for 3D microstructure fabrication. For example, Beuret et al. [Beuret, 1994] used the multi-lithography step to obtain an inclined structure through two shifted masks inclined rotating exposures. Ghodssi’s [Watts, 2003; Morgan, 2004; Watts, 2005] group developed a method of gray-scale lithography for fabricating 3D silicon MEMS structures used for diffractive optics. Tostu et al. [Tostu, 2005] used a maskless method as part of the multi-layered exposure process to create 100μm diameter spherical and aspherical microlens arrays on silicon substrate. Bourouina et al created a new technique using the microloading effect in RIE for micromachining 3D structures with only one mask [Bourouina, 2004]. Zhang et al used a method based on micro-stereolithography process to build the 3D microstructures on polymer materials by laser-induced solidifying
technique [Zhang, 1999]. These methods, however, almost all involved in multi-exposure and precision alignment to create the multi-level profile in photoresist to simulate the continuous profile of a 3D structure. Therefore it is difficult to achieve high quality in a true 3D microstructured surface. Moreover, the complexity of these processes often becomes the sources for high manufacturing errors thus resulting in high fabrication cost and a prolonged cycle time. It is also very difficult to make changes to these manufacturing methods since each step is directly associated with the up and down steam processes.

In ultraprecision single point diamond turning process, real 3D freeform surface features with both geometric tolerance and surface finish required for image optics can be fabricated. In the past, ultraprecision diamond turning has been tested for directly fabricating microstructures [Kawai, 2001]. More recently, Yi et al. developed a simple slow tool servo (STS) process for microlens array and diffractive optical elements (DOEs) [Yi, 2005; Li, 2006]. Slow tool servo process is an ultraprecision fabrication method using single point diamond turning based on feedback control of the mechanical slides of the ultraprecision machine. By use of these methods, true 3D continuous microscale surfaces with optical surface finish can be fabricated without the expensive masks and complex multi-level photolithography set up, which can greatly reduce manufacturing cost and shorten production cycle time. Moreover, this technique can also be used to create microstructures on almost any surface profile other than a flat one that is required for lithography. There are important industrial applications that can benefit from this rather important and unique feature.
In this section, a new method that combines the ultraprecision machining process with the cleanroom RIE for 3D microstructure fabrication was investigated. Using a commercially available single point diamond turning machine and the STS process if necessary, true 3D microstructures were first created on a thin photoresist layer (10-20μm). These 3D microstructures were then transferred to a silicon or fused silica substrate using anisotropic RIE method. In a demonstration of its possible applications in industry, using a micro machined silicon wafer as a mold, 3D microstructures were duplicated onto a low transition temperature optical glass using precision compression molding technique, an emerging high volume precision optical manufacturing process.

A.2 Experiment

The method in our study includes three major steps. First the silicon wafer was cleaned using a sulphuric and hydrogen peroxide mix. Then a thick layer of SU-8 photoresist was deposited on the surface. After baking, the selected 3D microstructures were then fabricated by means of ultraprecision machining on the photoresist. The machined structures were then transferred to a silicon wafer surface by timed reactive ion etching (RIE). This process flow is graphically depicted in Figure A.1. Although the process was demonstrated on silicon and fused silica wafers in our investigation, the fundamental principle can be easily transferred to almost any material with arbitrary shapes. This approach is especially attractive for super hard materials such as silicon carbide, glassy carbon and other materials that cannot be diamond machined to optical surface quality. In our investigation, SU-8 was selected as the photoresist mainly because its availability and easiness to work with in the cleanroom. The Young’s modulus of SU-8 is about 4.5
GPa [Hopcroft, 2005; Yu, 2006]. SU-8 photoresist also has the appropriate mechanical properties for ultraprecision diamond machining process. The viscosity of SU-8 2025 photoresist (MICRO CHEM) is about 4500 cSt prior to baking. To prepare for the photoresist layer, 40μm thickness SU-8 was spin coated on the silicon wafer. This thickness is desired for the single point diamond turning fabrication process. The spin coater’s rotational speed was 1,000 rpm. After the spin coating, the wafers were baked 3min at 65°C and then 9min at 95°C on a soft bake oven. To improve the mechanical property of SU-8 photoresist, the wafers were put in a hard oven at 115°C with 20min. The complete details of ultraprecision machining, RIE and compression molding process are described below. As a demonstration, a high volume precision compression molding process was introduced to transfer the pattern from a silicon substrate to an optical glass after the micromachining steps were completed.

Figure A.1 Process flow for 3D microstructures on silicon substrate
A.1.1 Ultraprecision Machining 3D Microstructures on Photoresist

Since it is impossible to machine certain substrates such as glassy carbon or silicon carbide using a single point diamond tool, creating 3D microstructures on photoresist first then transferring it to the substrates becomes a necessary step. In micromachining of the photoresist layer, we have encountered several difficult issues. First, because the photoresist layer was spin coated on silicon or fused silica wafers surface its thickness could not be precisely controlled. Second, air bubbles can occur during the curing stage, thus the accuracy and surface finish of the fabricated structure maybe compromised. Third, photoresist can sometimes be accidentally peeled off from the wafer surface during the diamond turning process, especially when the remaining photoresist layer becomes very thin, e.g., several microns.

To solve these problems, several steps were used. First, part of the photoresist was removed by acetone so that a small portion of the wafer substrate was exposed and could be used as a reference. To accurately measure the photoresist thickness, this reference section should be as close to the section where the 3D microstructure will be machined as possible. Second, the photoresist was diamond turned to required thickness. In our experiments, the diamond turning process was carried out on the Moore 350FG Ultraprecision Freeform Generator. The 350 FG ultraprecision machine has three linear axes that are equipped with linear laserscales capable of resolving 8.6 nm while moving at a maximum speed of 1,800 mm/minute. The straightness on all slides is less than 250 nm. The work spindle is capable of reaching 6,000 rpm while maintaining axial and radial error motion to less than 25 nm. The work spindle can also maintain angular
position to less than 5 arc second in a modulated mode. The photoresist layer was rough machined from 40-45μm to 15-20μm, and then was finish machined to 10-15μm thick.

**Table A.1**: Ultraprecision machining process parameters

<table>
<thead>
<tr>
<th>Process</th>
<th>Tool radius (mm)</th>
<th>Spindle speed (rpm)</th>
<th>Feedrate (mm/min)</th>
<th>Depth of cut (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface rough turning</td>
<td>3.175</td>
<td>800-1,000</td>
<td>6-10</td>
<td>1-4</td>
</tr>
<tr>
<td>Surface finish turning</td>
<td>3.175</td>
<td>800-1,000</td>
<td>1-2</td>
<td>1</td>
</tr>
<tr>
<td>Structure rough turning</td>
<td>0.0025</td>
<td>800-1,000</td>
<td>5</td>
<td>1</td>
</tr>
<tr>
<td>Structure finish turning</td>
<td>0.0025</td>
<td>800-1,000</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>STS rough machining</td>
<td>0.254</td>
<td>Stationary</td>
<td>600</td>
<td>1</td>
</tr>
<tr>
<td>STS finish machining</td>
<td>0.254</td>
<td>Stationary</td>
<td>600</td>
<td>0.5</td>
</tr>
</tbody>
</table>

The machining process parameters are summarized in Table A.1. After each machining step, photoresist thickness was measured using an ultraprecision indicator (Federal Gage EHE-2056 with 100 nm repeatability and less than 4 gram measuring force) to ensure the remaining thickness is appropriate for microstructure fabrication. After the surface was diamond machined, a smooth and flat optical surface was obtained. The third step is to rough and finish machine the design pattern using the diamond turning process. For complicated 3D structures that are not axysymmetric, slow tool servo diamond turning process has to be used. In our study, a diffractive optic element was diamond turned and a
sine wave grating surface on photoresists was fabricated by use of the slow tool servo process.

A.1.2 Reactive Ion Etching (RIE)

The RIE was performed on the Technics Micro-RIE Series 800-II. The control of this machine can be easily adjusted to change the etching process conditions, such as gas flow rate, electrode power, chamber pressure and etch time. To replicate the 3D microstructure on the microstructured photoresist layer using single point diamond turning process, compensation in etch rate between that of silicon and photoresist is required due to the difference in photoresist and the silicon wafer (or other substrate material, e.g., fused silica). CF\textsubscript{4}-O\textsubscript{2} gas mixture was often used in plasma etching process for both silicon and photoresist [Rauf, 2002; White, 1985]. For example, in our first experiment, an even etch rate on both silicon and photoresist was identified to transfer the 3D photoresist microstructure to the silicon substrate by carefully changing the CF\textsubscript{4}-O\textsubscript{2} plasmas conditions, i.e. by varying the ratio of CF\textsubscript{4} and O\textsubscript{2}, supply power, flow rate and chamber pressure.

To achieve a higher production (etch) rate, the oxygen plasma was first used to etch the photoresist 3D structure until the lowest point of the structure reached the silicon substrate. At the end of the first step, a formula that can be used to etch both the photoresist and silicon at similar rate was selected through trial and error. The final conditions used in this paper are summarized in Table A.2 for three different etching conditions and materials. Using the conditions in Table A.2, microstructures fabricated
on photoresist layer by ultraprecision diamond turning were transferred to the silicon substrate. The ratio between the etch rate on photoresist and silicon wafer was also summarized in Table A.2. Figure A.2 shows the cross-sectional view and line scan image of a silicon blaze after etching with this formula. The measurements were acquired using the Veeco NT 3300 optical profilometer.

**Table A.2: RIE etch parameters**

<table>
<thead>
<tr>
<th>Etched Material</th>
<th>Silicon</th>
<th>Silicon</th>
<th>Fused Silica</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface Profile</td>
<td>Sine Wave</td>
<td>DOEs</td>
<td>Sine Wave</td>
</tr>
<tr>
<td>Gas Flow Rate (O&lt;sub&gt;2&lt;/sub&gt;):</td>
<td>20 sccm</td>
<td>30 sccm</td>
<td>20 sccm</td>
</tr>
<tr>
<td>Gas Flow Rate (CF&lt;sub&gt;4&lt;/sub&gt;):</td>
<td>20 sccm</td>
<td>20 sccm</td>
<td>20 sccm</td>
</tr>
<tr>
<td>Chamber Pressure:</td>
<td>270 mTorr</td>
<td>270 mTorr</td>
<td>270 mTorr</td>
</tr>
<tr>
<td>Electrode Power:</td>
<td>200 W</td>
<td>200 W</td>
<td>300 W</td>
</tr>
<tr>
<td>Etch Time:</td>
<td>~ 40 minute</td>
<td>~ 30 minute</td>
<td>~ 120 minute</td>
</tr>
<tr>
<td>Etch Rate Ratio (PR/Substrate)</td>
<td>1</td>
<td>1.5</td>
<td>4</td>
</tr>
</tbody>
</table>

To test this process on different materials, particularly on materials that cannot be diamond turned at all, e.g., ceramic and glass, a sine wave profile was also fabricated on the fused silica substrate. The parameters for machining fused silica were also listed in Table A.2.
A.1.3 Glass Molding Experiment

The glass molding experiment was performed on a Toshiba GMP 211V machine at the Fraunhofer Institute for Production Technology in Germany. The detailed descriptions of the experiment can be found from previous chapters. A low transition temperature glass ($T_g=285^\circ$C, K-PG325 from Sumita, Japan) was used as raw material. The refractive index
of this glass is 1.50679 and the thermal expansion coefficient (TEC) is $17.3\times10^{-6}\,\text{°C}^{-1}$. In this paper, our main goal is to study the new micromachining method to fabricate true 3D microstructure on hard and brittle materials, such as silicon, fused silica, and glassy carbon. These components can be used directly in micro device fabrication but are also suitable for use as mold/stamper for high volume production.

Glass samples detached from the silicon wafer mold right after the annealing process, indicating a non-sticking situation between the low $T_g$ glass and the micromachined silicon wafer mold. Since only a limited number of DOEs were molded, no reliable information on the tool wear was available. The silicon wafer mold was observed using an optical microscope under high intensity light after each cycle but no sign of wear was detected. For this glass type and the silicon wafer, the selected molding temperature was fairly low thus we would not expect substantial wear due to thermal loading. Alcohol was used to clean the samples prior to performing the surface measurements.

A.3 Results and Discussion

Single point diamond turning process was used in combination with reactive ion etching process to create accurate true 3D microstructures. To quantify the process capability, a 10mm diameter diffractive optical element was fabricated using a single point diamond turning machine on SU-8 2025 photoresist. The height of the finished microstructure $H(r)$ is a function of radius $r$ as in equation A.1:

$$H(r) = r^2 - 3n$$  \hspace{1cm} (A.1)
where, \( n \) is the nearest integers less than or equal to \( r^2/3 \). Surface measurements were performed to evaluate the difference between the design and machined profile on photoresist. Both the Veeco Dektak Series 3 stylus profiler system and the Veeco NT3300 optical profilometer were used to measure the photoresist profile. Figure A.3 shows both the diffractive optical element design and the measurement on photoresist after diamond turning and the scanning electron microscope (SEM) image of the diffractive optical element on the photoresist. The 3D structure profile is considerably better than the result with gray-scale lithography or other multi-level lithographic based method. As illustrated in Figure A.3, single point diamond turning process can produce continuous surface features as compared with many other techniques.

Figure A.3 (a) Profile of a diffractive optical element after diamond turning compared with design. The dashed line is the design curve, and the solid line is the diamond turned surface measured by a Veeco Dektak Series 3 stylus profiler system. (b) SEM image of the diffractive optical element on the SU8 2025 photoresist after diamond turning.
Figure A.4 (a) 3D image of a sine wave grating on SU-8 photoresist (b) 3D profile of the sine wave grating after etching measured by Veeco optical profilometer (c) Comparison of the profile of one period from the sine wave grating before and after etching

The 3D microstructures on photoresist were successfully transferred to silicon substrate by a carefully formulated RIE process. After the etching step was completed, a continuous 3D profile was created. The Veeco NT3300 optical profilometer was used to
measure the contour of the patterned photoresist and etched silicon substrate. As a further proof of the effectiveness of the process in creating true 3D microstructure, using the same fabrication technique, a sine wave grating was created. Because of the etch rate of the photoresist using the selected plasma etch recipe was the same as the silicon substrate for the plasma conditions that were used, the height of the sine wave grating after etching was the same as the photoresist after diamond turning. The contour of both photoresist and machined silicon substrate measured using Veeco optical profilometer are shown in Figure A.4. The profile of a selected peak from the sine wave grating on photoresist as well as on silicon wafer are plotted together in Figure A.4c for comparison.

The 3D analysis plot of the first two ridges of the etched diffractive optical element is shown in Figure A.5 measured on the Veeco NT3300 optical profilometer. The comparison between the photoresist profile and the etched profile on silicon wafer was also shown in Figure A.5b. Under the second etching conditions listed in Table A.2 (O₂ flow rate 30 sccm, CF₄ flow rate 20 sccm), the etch rate of SU-8 photoresist was about 1.5 times faster than that of silicon. Thus the error caused by the etch rate difference can be precisely compensated by offsetting the design curve. To obtain the proposed profile (the dash line), the photoresist was fabricated as the modified profile (the solid line) using diamond-turning process. After the etching process was completed, the final results were measured using the Veeco optical profilometer and plotted as the pink line. The small columnar structures at the center in Figure A.3a and Figure A.5 are fabrication error during the single point diamond turning process caused by the error of finding center. The final results showed a good agreement with the design profile. The surface roughness
was also studied by using an atomic force microscope (AFM). A surface roughness of $R_a = 88$ nm or arithmetic average was obtained after etching as shown in Figure A.6. This surface roughness increase was mainly due to the aggressiveness in reactive ion etching process. The same measurement on diamond machined photoresist indicated that the surface roughness was less than $R_a=10$ nm prior to etching. Although the microstructures fabricated in this study showed sub-mm scale resolution in lateral direction, there may exist certain optical applications that would require higher precision. We believe that both diamond turning and etching processes need to be improved to achieve higher lateral resolution. In addition, using a different masking layer other than the SU-8 photoresist may also provide a better result in lateral resolution as the etching of the SU-8 photoresist layer using the selected plasma might not have been a perfect anisotropic process.

Figure A.5 (a) First two ridges of an etched diffractive optical element (b) Comparison between photoresist profile (solid line) and etched profile (dashed line)
To illustrate the 3D microfabrication capability on non-diamond turnable materials, a thin layer of SU-8 photoresist was spin coated on a fused silica (amorphous) wafer. The same sine wave pattern that was machined on silicon wafer was also diamond turned on the photoresist. After the diamond turning process, the fused silica wafer was etched to replicate the pattern on the wafer substrate. Figure A.7 showed the result of fabricated fused silica. Unlike the etching process on silicon wafer, the etch rate for fused silica was much lower than that of SU-8 photoresist. Under this etching condition, the etch rate of SU-8 photoresist was determined to be four times of that of fused silica. Based on this result, a modified sine wave grating profile with a depth of 4 times that of design can be implemented in single point diamond turning since the total depth of etching was linearly proportional to etch time as indicated in Figure A.7.

![AFM image of an etched diffractive optical element surface acquired using the Veeco NanoMan Dimension 3000 SPM.](image)

**Figure A.6** AFM image of an etched diffractive optical element surface acquired using the Veeco NanoMan Dimension 3000 SPM.
Figure A.7 (a) Image of a sine wave grating after etching on fused silica substrate measured by Veeco optical profilometer (b) Profiles of one period from the sine wave grating before, after etching as compared to the design profile.

Figure A.8 (a) First two periods of a molded glass diffractive optical element. (b) Comparison between the silicon mold and the molded glass diffractive optical element.
The molded glass diffractive optical element was also measured using the 3D microstructure silicon mold by Veeco optical profilometer and the results were plotted in Figure A.8. The results demonstrated an excellent geometry accuracy and surface finish replication.

A.4 Conclusions

Ultraprecision single point diamond turning, in combination with slow tool servo process if necessary can be readily used to create accurate 3D microstructure with sub-micron features on SU-8 photoresist. This is possible due to the unique mechanical properties of the photoresist. The machined 3D microstructures on the photoresist layer can be etched into the substrate surface using timed reactive ion etching process. Unlike traditional silicon etching technique, an accurate controlled etch selectivity is necessary for transferring the fabricated pattern from photoresist onto silicon substrate. To do this, a carefully formulated CF$_4$-O$_2$ gas mixture plasma was identified experimentally to achieve pre-selected etching rates on both the SU-8 photoresist and the silicon wafer. The advantage of this method is the capability of manufacturing true 3D microstructures on almost any materials and any geometrical substrate without using a photo mask. This method is essentially a maskless process in that any patterns can be easily transferred from design to an ultraprecision machine then to the final substrates. It effectively eliminates multiple masks required for multiple exposure in some lithographic methods thus resulting in lower manufacturing cost and a shorter production cycle. This process also has less accumulation fabrication errors as compared to the previous processes where
multiple steps were involved. The fabricated micro components can be used directly in MEMS devices or as mold material in other high volume micro manufacturing processes. One example of the latter applications has been showcased in the compression molding of glass diffractive optical elements.

To improve the quality of the 3D microstructures, we would continue our investigation further in the following areas:

a) Precise control and measurement of the photoresist thickness for single point diamond machining process

b) Control the quality of the photoresist to improve uniformity in photoresist and obtain higher surface finish.

c) Improve diamond machining process to obtain a higher structure accuracy and surface finish.

d) Improve etching uniformity and identify a better etch selectivity.

e) Identify other etching methods such as ion beam milling process to improve the finished surface roughness.

f) Construct a fast tool servo to increase the fabrication rate.
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