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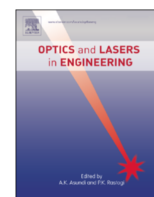

Localized rapid heating process for precision chalcogenide glass molding

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ABSTRACT

Precision glass molding is an important process for high volume optical fabrication. However, conventional glass molding is a bulk heating process that usually requires a long thermal cycle, where molding assembly and other mechanical parts are heated and cooled together. This often causes low efficiency and other heating and cooling related problems, such as large thermal expansion in both the molds and molded optics. To cope with this issue, we developed a localized rapid heating process to effectively heat only very small part of the glass. This localized rapid heating study utilized a fused silica wafer coated with a thin graphene layer to heat only the surface of the glass. The graphene coating functions as an electrical resistant heater when a power source was applied across the thin film coating, generating heat on and near the coating. The feasibility of this process was validated by both experiments and numerical simulation. To demonstrate the advantages of the localized rapid heating, both localized rapid heating process and bulk heating process were performed and carefully compared. The uniformity and quality of the molded sample by localized rapid heating process was also demonstrated. In summary, localized rapid heating process by using graphene coated fused silica wafer was characterized and can be readily implemented in replication of micro scale chalcogenide glasses.

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1. Introduction

Today micro scale optical components are gaining popularity for their wide applications in camera or sensor system for mobile devices and medical equipments. As early as in the 1980s, precision glass molding was first proposed for fabricating glass optical components with complicated surface geometries [9]. Since its introduction, micro scale glass molding has become a very important manufacturing technology for its low cost as well as high repeatability and reliability of the fabricated parts [4]. Conventional micro glass molding process also requires the heating of entire bulk glass blank above glass transition temperature (T_g) where the glass behaves like a viscoelastic material. In this arrangement, the glass mold, vacuum chamber where glass blank is placed and other mechanical components are heated up and then cooled down simultaneously. As such a long cooling cycle is generally required to ensure quality of the molded optics.

Among the drawbacks of conventional glass molding process, one major issue is its low energy efficiency due to long thermal cycles. In addition, bulk heating/cooling cycle can also cause problems ranging from thermal expansion of the molds, a shorter mold life, residual stresses inside the molded glass and refractive index variation in molded glass optics. To overcome these problems, numbers of experimental studies and numerical simulations have been devoted into in previous research works, including geometry compensation [2], evaluation of residual stresses [1,14], reducing refractive index variations [11,12] and applying protective coating on the mold surfaces [3]. More recently, precision glass molding has also even been tested for wafer level molding process demonstrated by Huenten et al. [7] to improve glass molding efficiency. However, wafer level glass molding in principle is still a form of conventional precision glass molding. Most of the aforementioned studies above failed to address the issues related to the prolonged and high degree of thermal cycles.

We have attempted to implement a graphene coating in precision glass molding in order to utilize its high hardness, low friction and high thermal conductivity [10]. For example, He et al. [5] conducted a comparative molding test between a silicon wafer mold with carbide-bonded graphene coated and a silicon wafer mold without graphene coating. Their investigation demonstrated that graphene coating enables silicon to be used as a mold material by preventing silicon to glass adhesions at elevated temperature. Additionally, graphene [13] or graphene based material [8], has gained more popularity for its combination of excellent thermal and electrical property and successfully applied in flexible electrothermal heating element. Sui et al. [13] annealed graphene oxide films at three different temperatures and

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revealed the electrothermal performances in diagram. They also investigated the mechanical stability and flexibility of these films by repeatedly bending to validate the heating function. Kang et al. [8] increased graphene resistance using lay-by-lay doping methods. However, according to their studies, neither graphene film heater from the two groups could reach 210 °C, which barely reaches the T_g for most common infrared glass materials.

In this research, we developed a graphene coated film heater that enables localized rapid heating of the shallow surface of glass that directly contacts with mold. This localized rapid heating mold was design to only elevate the temperature of area where micro features need to be replicated In this way, less thermal variation on mold and molding assembly is introduced comparing with conventional bulk heating glass molding equipment. This setup will reduce the undesired thermal expansion of the mold and molding assembly and thus will increase the life time of the molds and molding equipment. Moreover, the heating and cooling cycles will be significantly shortened since only the target area on the glass sample and its immediate surroundings will be heated above T_g.

In summary, the goal of this study was to implement a localized rapid heating process for micro scale precision glass molding using a carbide-bonded graphene coating [6] on the glass mold with micro features to address many of the aforementioned issues. A molding process of an infrared glass with relatively lower T_g than the oxide glass was investigated. The localized rapid heating was validated by both experiments and numerical simulation. To demonstrate its feasibility, this molding process was carefully compared with the conventional bulk heating process, which was performed on a commercial precision glass molding press. Finally, the uniformity and quality of the molded glass sample by localized rapid heating process were validated by molding large surface areas and the quality of the molded microstructures.

2. Materials and molding process

2.1. Mold fabrication

Fused silica was selected as the mold material for its low thermal expansion coefficient, ability to withstand rapid temperature change without cracking and its high electrical resistance even at elevated temperature. Standard lithography techniques were employed to transfer micro patterns to glass mold by reactive ion etching. The starting material is a 50 mm diameter and 500 μm thick fused silica wafer. First this wafer was cleaned using acetone and isopropanol sequentially. YES 3 Hexamethyldisilazane (HMDS) prime oven was used to improve adhesion between photoresist and fused silica wafer. Afterwards, a layer of 1.4 μm thick S1813 photoresist was deposited on the fused silica wafer. The density of UV radiation for photoresist exposure was set at 15 mW/cm^2 with a duration of 2.4 s on an EV Group 620 advanced contact aligner. After the UV exposure, the wafer was developed for 2 min in MF-319 solvent and then rinsed in deionized water. Next the wafer was etched in Plasma Therm SLR-770 using CCl_4 at a flow rate of 20 cm^3/min. The two RF (radio frequency) power supplies were set at 29 W and 500 W, respectively. The pressure was set at 5 mTorr during the entire etching process. The etching rate was set at 0.1 μm/min. Finally the remaining photoresist was stripped and the wafer was cleaned in the ultrasonic bath. Fig. 1 (a) shows the finished wafer.

Generally speaking, weak van der Waals force limits the atomic layer graphene building, especially for two-dimensional cross-linked coatings such as graphene because it can be easily damaged or even wiped out. Therefore the carbide-bonded graphene method developed by Huang et al.[6] was employed to coat a much thicker graphene network on the fused silica wafer. To do this, a piece of P–SO_3H nanopaper and a piece of high temperature silicone rubber were placed in a quartz tube furnace where the fused silica wafer was placed. Initially, vacuum was created in the furnace while the temperature inside the furnace was rapidly increased from room temperature to 600 °C in 20 min. Next, after vacuum was established, the furnace was sealed and continuously heated up to 1000 °C in 10 min. During this stage, the pressure inside the furnace was gradually increased due to the decomposed gas from the nanopaper and the silicone rubber.

When the pressure reached atmosphere pressure, the vacuum valve was turned on under the protection of high purity nitrogen. At the elevated temperature, –Si or –SiO radicals are thermally generated by the silicone rubber. Simultaneously, the graphene sheets exfoliated from the nanopaper due to gas bubbles that were thermally decomposed from benzene sulfonic acid groups and reactively formed the carbon radicals at the edge. These graphene sheets were bonded either with themselves by C–Si and C–O–Si or on the fused silica wafer surface by C–O–Si and eventually formed a three-dimensional cross-linked graphene networks. The furnace was held at the highest temperature for 30 min before cooling to room temperature. Finally the fused silica wafer was rinsed with water and acetone and dried in a vacuum oven at 100 °C overnight. The resistance of this coating is subjected to the coating thickness and the coating electrical conductivity. The coating electrical resistance can be adjusted by changing the ratio of C and Si.

To connect the fused silica wafer with a power supply, two electrodes were fabricated as shown in Fig. 1 (b). 597-A electrically and thermally conductive adhesive (Aremco Products Inc.) was applied onto the two opposite areas using copper foils. After the electrodes were constructed, the wafer was dried at room temperature for 2 h, followed by baking in vacuum oven at 93 °C for 2 h.

2.2. Demonstration of localized heating

Localized heating demonstration was experimentally performed as shown in Fig. 2. In this experimental setup, a 3.8 mm thick infrared glass, arsenic trisulfide glass ($As_{40}S_{60}$) with T_g of 180 °C, was placed on a small fused silica wafer coated with the graphene layer. Three small K type thermocouples were used to monitor the temperature variation, as shown in Fig. 2, two (node 1 and node 2) mounted near the edge of the glass and one (node 3) at the middle of the cylindrical side face. A direct current (DC) was applied on two copper electrodes in ambient environment during the heating test. Fig. 3 plots the temperature measurements during a typical molding experiment. According to this figure, the middle layer of infrared glass was only heated up to 72.7 °C when the fused silica mold had reached 271 °C.

2.3. Molding experiments

Localized rapid heating glass molding was performed on a home-built machine (only the heating assembly is drawn) is shown in Fig. 4. A specially designed polymer base plate is placed under the graphene coated fused silica wafer and a K type thermocouple, mechanically mounted on the lower mold. The thermocouple was fixed on the fused silica wafer at the corner of the infrared glass without touching the glass. Two copper electrodes were sandwiched between the edge of the polymer base plate and a piece of metal shim stock and fastened using a bolt. A 3.8 mm thick arsenic trisulfide glass from the same batch was placed on the fused silica wafer. The lower mold was moved up by a linear drive until the top surface of the infrared glass touched the bottom of the tungsten carbide. The force on the glass sample was preloaded at 49 N before heating and the positions of the molds remained the same during the heating cycle.

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