In this paper, to extend the application of this technique onto glass micro-embossing where the operating temperature is much higher, the CBG-coated silicon mold did not contact the glass sample when the coating was electrically charged in our modified process. The heat from mold to glass mainly transfers through radiation and gap conduction during heating stage, in turn, it requires a more careful temperature control. The paper is mainly organized as follows: the principle of Joule heat generated by carbide-bonded graphene (CBG) on silicon wafer is first explained. Then the schematic diagram of the newly CBG-based hot embossing is presented. To demonstrate the process feasibility, embossing attempt on N-BK7 glass is conducted to replicate a closely packed microlens array. The details on the mold fabrication, coating deposition and experimental apparatus are provided. Finally, the heating performance of CBG coating and the replication fidelity of microlens array are presented.

2. Principle and scheme of hot embossing based on localized rapid heating of CBG

2.1 Principle of Joule heat generated by CBG

As shown in Figure 1, when an electrically conductive material is applied with a voltage V, the resultant current *I* generates a heat flow $\dot{Q}$ mainly by the Joule effect dependent on the electrical resistance *R as given by* $\dot{Q} = I^2R$. CBG coating exhibits superior electrical (as high as $1.98 \times 10^4\ S/m$ [5]) and thermal conductivity, besides low glass-adhesion and low friction coefficient. Herein, the silicon wafer mould is used as the substrate mainly for two purposes. Firstly, microstructures can be easily fabricated onto silicon via various methods, such as etching, milling and even machining. Secondly, when the silicon is heated up to a certain degree, its resistance drops drastically to a low value. Afterwards, both CBG coating and intrinsic silicon substrate contribute to the total resistance, thus a considerable power can be obtained without too high electrical current.

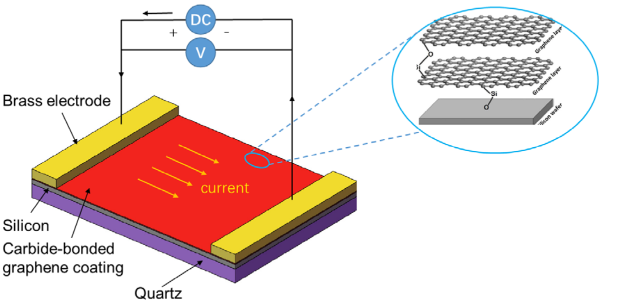

Figure 1. Illustration of Joule heating assembly using CBG-coated silicon

2.2 Scheme of CBG-based hot embossing

The CBG-embossed hot embossing includes typical heating, embossing, cooling and demolding steps (Figure 2). The distance between the CBG coated silicon mold and the glass sample should be small enough so that the glass sample can be heated effectively mainly through thermal gap conductance and radiation. The reason why the silicon mold doesn't contact the glass at evaluated temperature, is that when electrical current flows through hot glass surface above the transition temperature, bubbles would arise and accumulate within glass, thus result in a non-acceptable quality defect. Vacuum is necessarily desired to improve the heating rate. Since the glass's surface temperature is hard to measure, the temperature of silicon mold commonly should reach 50-80 ℃ above the softening point T_s of the specific glass before embossing. Once the desired temperature is reached, the current turns off and then the silicon mold is moved down to compress the softened surface layer within seconds. Afterwards the embossing position is held still for dozens of seconds to avoid immediate springback of the embossed structures. When the temperature of silicon mold drops below glass transition temperature, the embossing force is released but a small force to maintain the contact between glass and silicon mold in order to reduce temperature gradient. Once the temperature is below 200 ℃, nitrogen is purged to dismiss the vacuum condition.

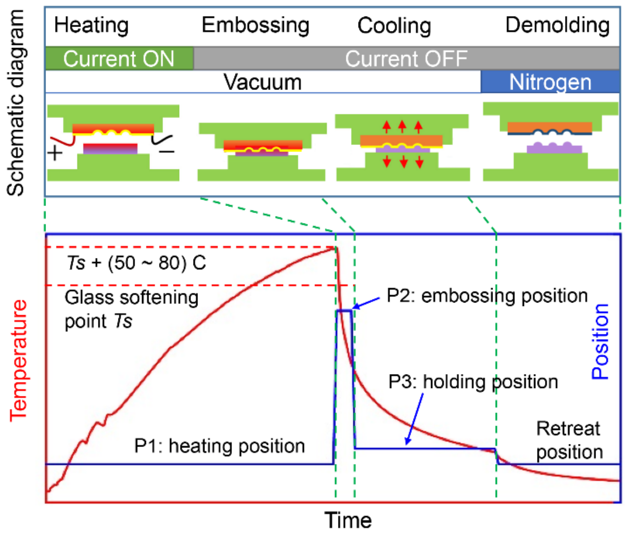

Figure 2. Schematic diagram of CBG-based hot embossing process, and time history plots of temperature on silicon mold and moving position

3. Materials preparation and embossing experiment

3.1. Fabrication of microlens array on silicon mold insert

Single-point diamond broaching performed on Moore Nanotech 350FG, was employed to fabricate a closely packed rectangular plano-concave microlens array on a monocrystalline silicon wafer. The details on the dimensions of microlens array, broaching parameters and tool's parameters, are listed in Table 1. Herein a revised toolpath "S" toolpath is generated where the alternate lenslet is flipped up to avoid a sharp change at the apex running in opposite direction, so the resulting sine wave toolpath tends to run smoothly on Z-axis. By applying the new toolpath for the micro lens array machining, the fabricated microlens array has a form error less than 30 nm, and surface roughness of 2 nm in *Ra*.

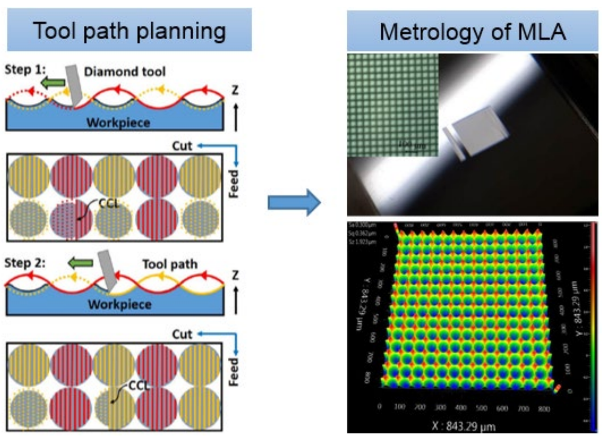

Figure 3. Illustration of the toolpath during machining; the image of fabricated microlens array on silicon mold, and microlens array's contour image measured using white-light interferometer Wyko NT8000.

Table 1 List of dimensions of microlens array, broaching parameters and tool's parameters

Parameters of microlens array	
Substrate material	Silicon (100)
Substrate dimensions	20 mm x 30 mm x 1 mm
Area of microlens array	3.0 mm x 3.0 mm
Pitch (μm)	60
Sag (μm)	0.7
Broaching parameters	
Depth of each cut (μm)	0.1
Cutting speed	60 mm/min for rough cut 10 mm/min for fine cut
Cutting tool parameters	
Material	Single-crystal diamond
Nose radius (mm)	0.5
Rake angle (°)	-25
Relief angle (°)	10

3.2. Deposition of CBG coating onto silicon mold

A thin layer of carbide-bonded coating deposited onto the silicon mold not only functions as the heating source, but also fulfils the purpose of anti-sticking between softened glass and silicon mold at evaluated temperature to ensure the surface quality of embossed elements. The deposition of CBG coating was realized by mainly referring the chemical vapour deposition (CVD) procedure developed in ref [5]. Regarding the complexity of preparation of GP-SO3H nanopapers, methane was selected as the main carbon source instead. Solid polydimethylsiloxane (PDMS) with a purity of 99.8% was chosen as the silicon source. The deposition equipment and conditions are presented in Figure 4. In this study, a thin layer of CBG coating with thickness of about 300 nm was deposited onto the silicon mold.

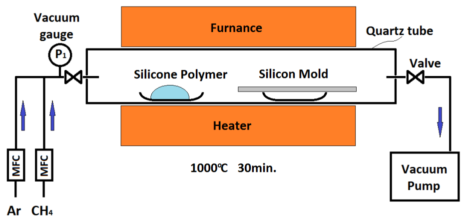

Figure 4. Illustration of the CVD conditions for CBG deposition.

3.3 Experiment equipment

As shown in Figure 5(a), a lab-built hot embossing apparatus was made, mainly composed of the core CBG-coated silicon mold and its fixture structure, working chamber, motion actuator, vacuum system and gauge, temperature control unit, force sensor, thermocouples and data logger. The main components inside of the working chamber is illustrated in Figure 5(b). The top and bottom mold assembly are aligned in vertical direction. A highly elastic silicone rubber is placed on the bottom, making the compression system flexible to avoid glass crack when an abrupt load is applied.

Figure 5(c) shows the mounting structures of the core CBG-coated mold assembly. Two copper ribbon leads are used to apply the voltage input. To be mentioned, a thick quartz plate together with two small quartz bars, is used to electrically and thermally isolate the silicon mold from the surrounding bodies.

Since the resistance of silicon is strongly dependent on temperature, the total resistance and the corresponding heating power of the system are intensely changed. To precisely control the heating performance, a programmable direct current (DC) powder supply is adopted and controlled by a segmental polynomial-sum power supply algorithm.

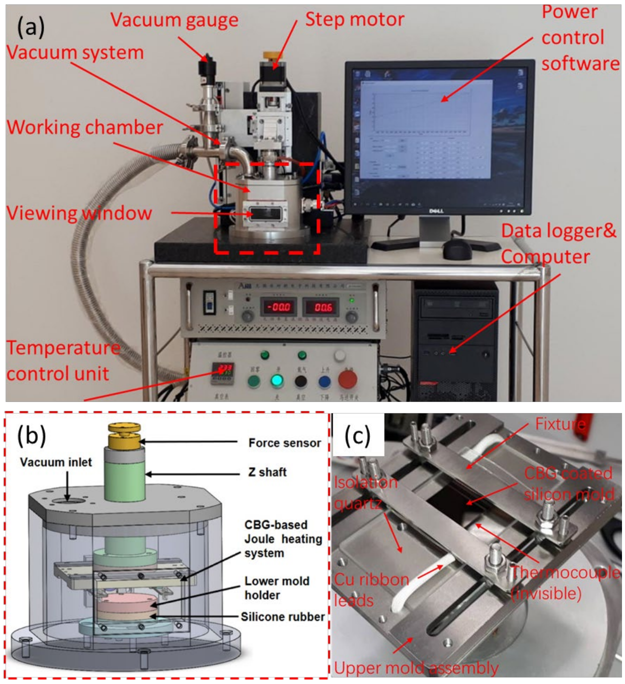

Figure 5. (a) The experiment apparatus and its main components, (b) 3D drawing of the working chamber, (c) the upper mold assembly equipped with the CBG-coated silicon mold.

3.4 Embossing experiment setup

The material of glass wafer is N-BK7 from Schott Inc., whose glass transition temperature T_g and softening point T_s are 557 °C and 719 °C respectively. The glass dimensions are 12.7 mm in diameter and 3.0 mm in thickness. According to the embossing temperature requirement, the maximum temperature on silicon is expected to be about 800 °C, which is considerably high enough to prove the superiority of the heating system. To effectively heat up the glass specimen through radiation and gap conduction, a small enough gap between glass and silicon mold is set as 0.1 mm before embossing. The preset heating powder linearly increases with time at a rate of 0.3 W/s during heating duration of 400 s, so the maximum heating power is 120 W. The required heating power to reach the expected embossing temperature, is determined by conducting trial experiments for now. The embossing process is finished within 20 seconds with a maximum compression force of about 150 N. The vacuum pressure of chamber can reach 5 Pa at steady state.

4. Results

4.1 Heating performance of CBG-coated silicon mold

The temperature of silicon mold measured by a thermocouple beneath is plotted in Figure 6(a). Besides, the applied voltage and current are plotted in Figure 6(b), and the equivalent resistance is calculated by using voltage divided by current. It is clearly seen that the resistance is about 400 Ω at low temperature, but continues decreasing sharply during the heating stage. At 800 ℃, the resistance drops down to almost 0.3 Ω. The drastic drop in resistance is caused by the negative temperature coefficient (NTC) behaviour of intrinsic silicon. In this study, when the heating power increases linearly with time, the temperature increasing rate of silicon mold is also almost linearly below 600 ℃, but after that it gradually becomes smooth because more energy is dissipated into the surroundings. Totally, the heating rate is approximately as fast as 2 °C/s, so the heating process only takes 400 s. Most importantly, the consumed heating energy is only 24 KJ, which is almost less than