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Precision Glass Molding of Fused Silica Optics

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Abstract
The range of applications for photonic systems, which includes industrial production as well as medicine technology, is still often limited by the employed beam guiding optics. Fused silica is an alternative optical material, providing better transmission properties and a higher resistance when it comes to high radiation intensities. However, currently there is no cost-efficient technique available for manufacturing complex-shaped fused silica optics.
On that account, this work focuses on precision glass molding of fused silica optics that allows the replicative manufacturing of high-quality optics in a one-cycle molding process. Due to the high molding temperature of up to 1400 °C, a crucial aspect of fused silica molding is to identify thermal resistant molding tool materials. For this, heating tests were performed at temperatures up to 1360 °C. Experimental results illustrate that precious metal as well nitride-based protective layers, established in molding common optical glass and deposited on SiC substrates, fail rapidly due to the high thermal load. Instead, first pre-tests demonstrate a promising thermal resistance of a carbon-based graphene protective layer.

Carbon, Coating, Material, Molding

1. Introduction

Optical components made of fused silica are essential due to the outstanding properties of this material. Fused silica is applicable for ultraviolet, visible and near infrared radiation based on its broad transmission range for electromagnetic radiation from 180 nm to 3.5 µm wavelengths. Additionally, the material has a high laser induced damage threshold (LIDT), which especially qualifies fused silica optics to be used for high-power lasers. Furthermore, the low coefficient of thermal expansion ($\approx 0.5 \cdot 10^{-6}\ K^{-1}$) and the high glass-transition temperature (≈ 1300 °C) facilitate the application of fused silica in high temperature environments.

In general, glass optics are traditionally manufactured by grinding and polishing processes. This manufacturing technology is well established and suitable for the production of planar and spherical shaped optical components. However, time-consuming direct manufacturing of complex-shaped fused silica optics (e.g. aspheres or diffractive optics) is neither practical nor economically viable. Instead, the replicative technology of precision glass molding is more convenient for the production of complex optics [1]. During the one-cycle molding process, a glass preform heated until the viscous state is molded to the desired shape using two ultra-precise molding tools. After cooling, the pressed glass optic can be incorporated in optical components without post-processing. However, durable molding tools are required to perform a cost effective manufacturing process. For this reason, several investigations focus on enhancing the molding tool lifetime using wear protective coatings [2-5]. Nowadays, three types of coatings are established for molding common optical glasses: precious metal coatings [3], nitride based coatings [4] and carbon based coatings [5].

Precision glass molding of fused silica, in comparison to the common optical glasses, is considerably more challenging caused by the high molding temperature of about 1360 °C, whereas the molding temperature of common optical glasses ranges from 400 °C to 700 °C. Due to high thermal loads caused by the high molding temperature, so far no suitable material could be qualified. For establishing precision glass molding of fused silica, the qualification of a thermal-resistant mold material is mandatory.

In this work, heating tests in a glass molding machine were carried out in order to investigate uncoated and coated silicon carbide substrates for fused silica molding. After heating tests, surface degradation mechanisms were determined by (white) light microscopy and scanning electron microscopy. The comparison of investigated silicon carbide substrates allows a summarizing statement about the potential of coated silicon carbides for fused silica molding.

2. Experimental Setup

2.1. Molding tools

Cylindrical molding tools of silicon carbide (SiC) with diameter 21.5 mm and height 3.5 mm were used. SiC is a convenient material for molding tools due to its high temperature resistance [6]. The plane mold surfaces were polished to optical quality (roughness Ra < 5 nm). Three different protective layers (platinum iridium PtIr, chromium aluminium nitride CrAlN and a graphene coating) as well as an uncoated sample were investigated. The PtIr [7] and CrAlN [8] protective layers were deposited by physical vapour deposition (PVD) at Fraunhofer IPT. The graphene layers were synthesized by chemical vapor deposition (CVD) at Ohio State University [9].

2.2. Fused silica

The glass blanks used were cylinders with a diameter of 10 mm and a height of 5 mm made of fused silica grade SQ1 (≈ 1200 ppm OH content, other elements ≤ 0.4 ppm).

2.3. Heating tests

Heating tests were performed on a commercial Toshiba Glass Molding Press GMP-207HV. The test process is isothermal due to the fact that the molding tools and the glass blank have the same temperature throughout the process.

Similar to an industrial molding process, a heating cycle lasts about 18 minutes (5 minutes heating, 8 minutes annealing and 5 minutes cooling). A specimen coated with PtIr, CrAlN as well as an uncoated SiC specimen were heated with a hung up fused silica glass blank to a heating temperature of 1360 °C. The graphene layers were tested in a first pre-test series up to 800 °C without any glass contact. All heating tests were carried out under a nitrogen atmosphere.

2.4. Specimen characterization

In order to characterize the tool surfaces, light microscopy was used to make a first level assessment of the coating quality. The roughness parameter Ra of the specimens were measured by a white light interferometer (WLI) Wyko NT1100 produced by Veeco®. For evaluating the surface microstructure, scanning electron microscope (SEM) images were taken with a Zeiss Neon 40 EsB.

4. Results

The key results of the experimental series investigating a CrAlN, a PtIr and an uncoated specimen at a heating temperature of 1360 °C are summarized in Figure 1.

Already after one heating cycle, the CrAlN coated specimen showed an obvious discoloration in the area of glass contact. By means of scanning electron microscopy, extensive glass adhesion was demonstrable throughout the glass contact area, so that the surface roughness increased to a value of 61.9 nm.

The specimen coated with PtIr protective layer showed a slight discoloration and roughening (Ra = 22.8 nm) after 6 heating cycles. No glass adhesion could be observed. Instead, local coating delamination occurred.

The uncoated SiC specimen remained unchanged in color after 6 heating cycles. However, local macroscopic glass adhesion with diameter up to the millimeter scale covered the glass contact area.

The three investigated specimen already showed various surface failure (glass adhesion, coating delamination, roughening) after a low amount of heating cycles. In all, these material systems are not suitable for fused silica molding.

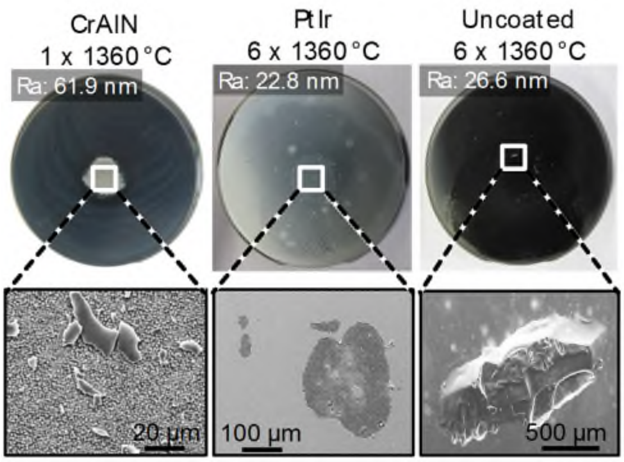

Figure 1. Photography and SEM image of the CrAlN coated specimen after 1 heating cycle at 1360 °C (left); photography and SEM image of the PtIr coated specimen after 6 heating cycles at 1360 °C (center); photography and SEM image of the of the uncoated specimen after 6 heating cycles at 1360 °C (right).

In Figure 2 the results of the first pre-test investigating the graphene coating on a SiC specimen without glass contact are summarized. Up to 800 °C, the maximal heating temperature during the pre-test, no surface failure was detectable. The graphene coating was unscathed without any cracks or delamination. The surface had still optical surface quality with a surface roughness about 2.5 nm.

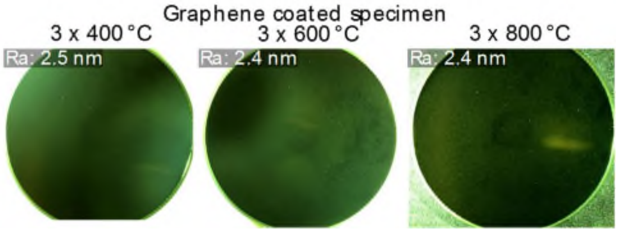

Figure 2. Graphene coated specimen after 3 heating cycle at 400 °C, 600 °C and 800 °C.

5. Summary, conclusion and future work

In this work, heating tests in a glass molding machine were carried out in order to investigate uncoated and coated silicon carbide substrates for fused silica molding. The following major conclusions were drawn about the suitability of coated and uncoated silicon carbide specimen for fused silica molding:

(1) Due to extensive glass adhesion CrAlN coating is not suitable.
(2) PtIr coating delaminates from SiC substrate due to high thermal stresses.
(3) Uncoated silicon carbide substrate shows local macroscopic glass adhesion, leading to a surface roughness Ra increase to values higher than 25 nm.
(4) Graphene coating surface remains undamaged and smooth (roughness Ra ≈ 2.5 nm) in heating tests up to 800 °C.

In consequences of the promising graphene coating results, further heating tests are planned with this material system. If the graphene coating also proves at heating temperatures up to 1360 °C, final tests in real molding processes are considered.

6. Acknowledgements

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