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Numerical simulation and experimental study on crack self-healing in BK7 glass

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ABSTRACT

Crack self-healing is the result of a combination of multiple effects and involves many factors, such as mechanics, thermodynamics, physics, and chemistry. In this paper, a finite element simulation model and a modified crack-length-prediction model for crack self-healing in BK7 glass were proposed and verified experimentally. The simulation results showed a stress concentration at the tip of the crack at the initial stage of the crack self-healing process. The crack length decreased gradually, and the stress concentration area moved to the surface. The stress concentration area almost disappeared when the crack was healed completely. When the relative humidity was 64% and the compression was 5 MPa, under variable-temperature heating, the required time for a 19.8 μm pre-crack complete healing was 7.5 h. As the temperature increased, the required time for complete healing decreased, the contact state between the crack boundaries was improved, and thus the cracks were connected and healed.

1. Introduction

During the grinding, polishing, and finishing of brittle optical elements, defects such as breakages, scratches, cracks, dislocations, residual stresses, and impurities are generated in the material surface or subsurface [1]. These defects are precursors of laser damage to optical elements, and initial laser damage points are often formed in their vicinity [2]. Under high-power laser irradiation, the initial laser damage points of the optical elements are tens of microns, and the size of the damage increases exponentially with laser fluence [3]. To improve the laser-induced damage threshold (LIDT) of optical elements, pretreatment is attempted before use. The main pretreatment methods include chemical etching [4], ultraviolet and carbon dioxide laser irradiation [5,6], ion-beam polishing [7], MRF polishing [8], and high-temperature annealing [9]. Although these pretreatment methods have their own advantages, they exhibit some limitations. It is necessary to find a new pretreatment method to improve the LIDT of optical elements.

When brittle material anneals under appropriate conditions (temperature, humidity, pressure, and residual stress), material in the complex physical and chemical reactions will produce phenomena, such as softening, viscous flow, blunt crack tips, and crack self-healing, and these can mitigate crack [10–13]. Theoretically, this method improves the LIDT of optical elements used in high laser fluence. However, crack self-healing is not only crack closure, disappearance, or the recovery of material strength. The temperature, pressure, humidity and heat-treatment time need to be controlled accurately during the process. If the treatment transforms the material from the glass to the molten state, the surface roughness and shape error of the optical elements will be influenced, which results in the destruction of optical elements.

The temperature, pressure, and humidity are important external factors that affect the solid-phase reaction. Many scholars have conducted research on the influence of these factors on crack self-healing. Zhu et al. investigated the crack healing behavior of $(MoNb)Si_2$ materials with high-temperature oxidation in air. The material bending strength recovered significantly after heat treatment and the crack-healed samples exhibited higher bending strengths than the original level after treating in 1200 °C [14]. Doquet et al. found partial crack healing after annealing in air at 425 °C when a 20-MPa compressive stress was applied, and annealing at 450 °C under 20 MPa pressure led to a complete disappearance of the crack. The results also show that a high compressive stress can shorten the crack and promote healing [15]. Nam et al. investigated the crack healing behavior of SiC ceramics. The heat-treatment temperature has a significant influence on the healing process, and the optimum temperature for SiC healing is 1100 °C [16]. Xu et al. found that the effect of increasing the pressure of heating at low temperature was not obvious, and an unbalanced pressure can increase the time for complete healing [17].

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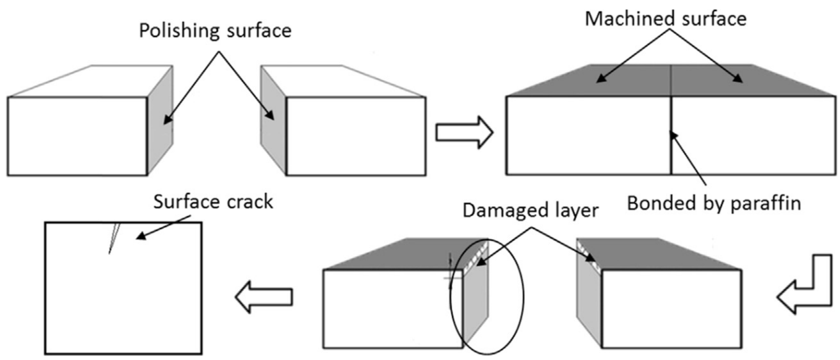

Fig. 1. Sample preparation process.

These investigations indicate that reaction and diffusion can be enhanced by an increase in temperature, and a high temperature with hot pressing can improve the contact state between crack boundaries and shorten the cracks significantly. Transmission between the same materials can cause crack healing and a restoration of material strength [14–17]. The glass viscosity is dependent on the temperature and water content. When a small amount of water is introduced into the melt glass, the material viscosity around the crack can be reduced by water dispersion and glass hydrolysis. Viscous creep flow causes crack healing and the recovery of material strength, and it blunts cracks, which mitigates the stress concentration at the crack tip, which improves the LIDT. However, cracks heal only when the temperature approaches the softening temperature (T_s), and the softening temperature is only tens of degrees lower than the glass transition temperature (T_g), so the temperature control must be very precise during the heat treatment [14–17].

The finite element method is used to simulate crack self-healing, and the simulation results are intuitive with a low cost. Therefore, many scholars have tried to improve simulations of crack self-healing. Nielsen et al. developed and validated a three-dimensional model to simulate glass tempering, assembled from models of temperature-dependent viscoelasticity and structural relaxation. A prediction of transient and steady-state stresses in complex three-dimensional glass geometries was achieved, and convergence analysis and experimental verification were carried out [18]. Barth et al. used the finite element code Abaqus to simulate cooling of a bulk borosilicate glass. The influence of thermal gradient on the stress concentration and solidification process was analyzed. The dissipation of thermal strain during the transition from liquid to solid reduced the cracking phenomenon during cooling [19]. Nielsen, Barth et al. provided new ideas and reference data for the crack healing of brittle materials. Xu et al. used Abaqus to simulate SCN-1 glass crack closure and healing. The influence of stress, temperature, and crack morphology on the healing behavior of the glass were investigated and discussed. With an increase in temperature, the increased creep deformation and the grain diffusion promoted crack closure and healing [17]. Doquet et al. used the finite element method to simulate the effect of different constraints on the crack healing of inert borosilicate glass. The crack boundary was shortened with an increase in pressure at 400 °C annealing, and eventually the boundary was connected and healed. The tensile strength of the bridging layers was estimated to be 27–39 MPa after vacuum annealing at 400 °C [15].

These results indicate that crack self-healing involves many factors, such as mechanics, thermodynamics, physics, and chemistry, and is the result of multiple effects. Although different models have been proposed to analyze the crack variation at different stages during the healing process, it cannot be expressed by a single mechanism, and the results of a single crack model cannot be generalized to multiple cracks by simple linear superposition. Moreover, local variation at the crack boundary is usually unsynchronized and disordered, the crack healing path is unpredictable, and irregular secondary cracks are also involved [17]. To match the healing behavior of a single crack with the macro response, and to enable the self-healing method to be used in optical engineering, we need a finite element method model of the healing process that considers the influence of multiple physical fields and covers the entire framework.

BK7 glass material was used as the object of the research. Based on the creep characteristics of BK7 glass at a high temperature and the constitutive model of different creep stages, a finite element simulation model for crack self-healing was proposed and established. The influence of temperature and time on the healing process was analyzed. Finally, the feasibility and the correctness of the model were verified experimentally.

2. Sample preparation

BK7 glass surface microstructures that were introduced by grinding were observed through cross-sectional microscopy detection technology. [20] The surface crack morphologies were observed by atomic force microscopy (Nanite B, Nanosurf Ltd. contact mode, lateral resolution of 1.7 nm, axial resolution of 0.34 nm, and scanning area of 110 μm × 110 μm × 22 μm). The concrete steps were as follows:

(1) Two sides of the BK7 glass (30 mm × 15 mm × 6 mm) were polished (the CeO_2 particle diameter was 1.5 μm, the polishing solution concentration was 8 wt%, the polishing time was 4 h). (2) The two polished surface were bonded by melting paraffin, and the two parts were clamped by using a vice to provide a uniform and thin adhesive-layer thickness to prevent breakage during polishing, then the sample was placed at room temperature until the paraffin cooled. The principle is shown in Fig. 1. (3) The up-surface was lapped by using SiC of W28, with a lapping time of 1 h, and a lapping-fluid concentration of 8 wt%. (4) The BK7 glass was immersed in hot water to melt the paraffin, and was placed in an ultrasonic cleaner to remove impurities. The cleaned components were placed into a mixed solution (1.5% hydrofluoric acid and 1.5% NH_4F) for 30 s to remove the hydrolysis layer for crack exposure and then cleaned again by ultrasonic cleaner. (5) The cross-sectional morphology was studied by atomic force microscopy. The surface-crack morphologies and distribution were obtained.

Two obvious cracks on two specimens were selected for further simulation and heating experiments. The length of the first crack was 19.8 μm, its inclination angle was 85.1°, and its maximum crack width was 235 nm (Fig. 2(a)). The length of the other crack was 26.5 μm, its inclination angle was 86.8°, and its maximum width was 330 nm (Fig. 2(b)).

3. Heat healing experimental procedures and results

A heating experiment was carried out to verify the feasibility of the simulation method under variable-temperature heating conditions, and the morphologies of the BK7 glass surface cracks were obtained by optical microscopy.

The cracked sample (length of 19.8 μm) was heated in five steps under a 5-MPa pressure and at a 64% relative humidity in a vacuum