



Water corrosion of commercial neodymium-doped phosphate high-peak-power laser glass

Meng Li^{a,b}, Bin Wang^a, Xin Wang^a, Xu Yang^c, Danping Chen^{a,*}, Wei Chen^{a,*}, Lili Hu^a

^a Key Laboratory of Materials for High Power Laser, Shanghai Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Shanghai 201800, PR China

^b University of Chinese Academy of Sciences, Beijing 100049, PR China

^c Department of Physics and Astronomy, Ames Laboratory, Iowa State University, Ames, IA 50011, USA



ARTICLE INFO

Keywords:

Phosphate glass
Laser glass
Chemical durability
Surface corrosion

ABSTRACT

In this paper, the surface corrosion mechanisms of Chinese high-peak-power laser glass, the N31 glass, are investigated. Powder samples were immersed in deionized water for up to 72 days. The concentrations of ions in leachate solutions were measured by inductively coupled plasma optical emission spectroscopy, and the surface evolution during corrosion was characterized by X-ray photoelectron and attenuated total reflection infrared spectroscopies. The N31 glass leaches incongruently, and the normalized ionic release rate decreases with an increase of cation field strength. Depending on the oxygen-to-phosphorus molar ratio (O/P ratio) evolution on the glass surface, the surface corrosion process presents two stages, which can be indirectly supported by the attenuated total reflection infrared spectroscopy results. This study reveals composition and structure evolutions on the glass surface during water corrosion. In addition, our results suggest that the O/P ratio on glass surface could be used to characterize the surface corrosion degree and mechanisms.

1. Introduction

Thermonuclear ignition is a promising candidate to obtain carbon-free energy [1]. It is predicted that laser can be used to generate the necessary conditions to achieve fusion ignition, which is known as inertial confinement fusion (ICF) [2]. High-peak-power ICF facilities require strict technical standards for the gain medium, such as high gain, low nonlinear refractive index, excellent optical homogeneity, etc. [3–7] The current choice is Nd:phosphate laser glass, which satisfies all of the requirements of ICF facilities.

Commercial high-peak-power laser glasses lie near the metaphosphate composition range. The typical molar composition is around $60\text{P}_2\text{O}_5\text{-}10\text{Al}_2\text{O}_3\text{-}30\text{M}_2\text{O}/\text{MO}$ ($\text{M} = \text{K}, \text{Na}, \text{Li}, \text{Ba}, \text{Mg}$) [3,4,8]. Al_2O_3 is generally used in commercial phosphate laser glass [3–6,8]. Adding Al_2O_3 will improve the chemical durability, thermal conductivity, and decrease the thermal expansion coefficient of the Nd:phosphate glass by forming P–O–Al connections [3,4,8–10]. In addition, the Al_2O_3 content can decrease the Pt solubility, which is the major source of inclusion damage in phosphate laser glass [3,4,8,11,12].

The composition design of Nd:phosphate laser glass is a tradeoff between the optical and mechanical/chemical properties. Although the addition of Al_2O_3 can greatly improve the chemical durability of phosphate laser glass, this content should be minimized as it

deteriorates the optical properties of this glass [4]. Due to the natural low connectivity between phosphate tetrahedras arising from P=O double bonds, poor chemical durability has always been an obstacle for laser glass performance. During storage, transport, polish and service, water molecules will erode the laser glass surfaces and result in the accumulation of OH^- . When Nd glass is pumped as a laser gain medium, the OH^- on glass surface quenches excited Nd ions transition from $^4\text{F}_{3/2}$ to $^4\text{I}_{15/2}$, thus reducing its performance [3,4,13]. Moreover, water corrosion causes subcritical crack growth in this glass [14–16], reducing its resistance to laser-induced damage. Insights into the surface corrosion of Nd:phosphate laser glasses are essential in improving their performance for high-power-laser use.

Most theories regarding phosphate glass corrosion have been established by weight loss and ionic release experiments. Depending on the composition, phosphate glass exhibited congruent [10,17–19] or incongruent dissolution [19,20] behavior, and the glass weight loss was proportional to $t^{1/2}$ or t during the initial dissolution stage [17–19]. Structural characterization methods, such as Raman spectroscopy [21], infrared (IR) spectroscopy [20,22], nuclear magnetic resonance (NMR) [23], X-ray diffraction (XRD) [20,24], etc., have also been used to study the corrosion process. Although many useful structural details have been revealed, all these methods focused on bulk glass rather than its surface. As a powerful surface analysis method, X-ray photoelectron

* Corresponding authors.

E-mail addresses: d-chen@mail.siom.ac.cn (D. Chen), weichen@siom.ac.cn (W. Chen).

spectroscopy (XPS) has mostly been employed to determine surface composition [18]. A few studies have included further characterization of the eroded surface by fitting components [25,26], however, no successive structure and composition evolutions were provided to depict the overall surface corrosion process.

For laser glass community, the primary interest is the residual OH⁻ and corrosion-induced defects from nano- to micro-scale depths. In this work, our primary motivation is to study the surface corrosion mechanisms of the N31 glass, and to develop a method to evaluate the corrosion degree of Nd:phosphate glass surfaces. Surface analysis tools XPS and attenuated total reflection infrared spectroscopy (ATR-IR) were combined used for continuous characterizations, and based on these data the surface corrosion mechanisms of this glass were hypothesized. Besides, ionic concentrations in leachate solutions measured by inductively coupled plasma optical emission spectroscopy (ICP-OES) were also included in this study, in order to show the overall water durability characteristics of the N31 glass. This work may potentially improve the working performance of Nd:phosphate laser glass for high-power-laser facilities, and can be useful for the composition design of phosphate laser glass to enhance its water corrosion resistance.

2. Material and methods

2.1. Glass preparation

Commercial N31 phosphate laser glass manufactured by our laboratory was measured in the experiments. The composition was (50–60)P₂O₅-(8–12)Al₂O₃-(8–12)BaO-(10–14)K₂O-(1–3)Li₂O-(1–3)Na₂O determined by ICP-OES (ICAP6300, Thermo Fisher Scientific, United States) and X-ray fluorescence (ARL 9900, Thermo Fisher Scientific, Switzerland), and the Nd doping level was ~3.5 wt%. The bulk glass was polished into 40 × 40 × 10 mm pieces and ultrasonically cleaned in ethanol. Powder samples with particle sizes of 45–75, 75–355 and 355–900 μm were obtained by grinding and mesh sieving the glass pieces.

2.2. Leaching experiments

2.2.1. Long-term leaching experiment

The long-term leaching experiment was conducted to investigate the long-term ionic release behavior of the N31 glass. In order to accelerate the reaction rate, the powders with 45–75 μm particle sizes were used for this experiment, and 5 g of the powders was placed into a Teflon flask with 450 mL deionized water (DI) without a buffer (duplicate samples for ICP-OES and pH tests). The initial ratio of powder surface area to leachate volume (SA/V ratio) was 393 m⁻¹. The flasks were placed into an incubator at 80 °C for up to 72 days of reactions, and 5 mL of the leachate solution was taken from the two flasks each time for ICP-OES and pH tests. No additional water was added to avoid disturbance to the ionic concentration during the experiment. For the long-term leaching tests, the solution was stirred every 24 h to guarantee a homogeneous leaching process.

2.2.2. Short-term leaching experiment

The short-term leaching experiment was also conducted to investigate the initial dissolution behavior of the N31 glass. As the initial dissolution behavior of phosphate glass is often controlled by water diffusion into glass network, large size powders with 355–900 μm particle sizes were used for this experiment to alleviate the influences of fractures and dissolution products. 2 g of powders was placed into a Teflon flask with 450 mL DI water without a buffer (duplicate samples for ICP-OES and pH tests), and the initial SA/V ratio was 15 m⁻¹. The flasks were placed in an incubator at 80 °C for up to 72 h of reactions. The solution was stirred before 5 mL of the leachate was taken from the two flasks for the ICP-OES and pH tests. No DI water was added during

the whole experiment.

2.2.3. Samples for spectroscopic and morphologic characterizations

Powders with particle sizes of ~8 μm were used for the XPS and ATR-IR experiments. These samples were obtained by ball milling the powders with 75–355 μm particle sizes in a drying room (15.6 °C, 11.2% RH) for 3 h. The particle size was characterized by the laser particle size analyzer (Bettersize 2000, Better, China). Each 2-g sample was placed in a Teflon flask with 300 mL DI water at 80 °C, with a reaction duration from 0.5 h to 30 days. The initial SA/V ratio of this sample was 883 m⁻¹. The pH values were measured as presented above. The powders were rinsed with DI water, filtrated and dried in a desiccator after the leaching process.

Several 10 × 10 × 1 mm well-polished glass pieces were prepared for surface morphology characterizations. The glass pieces were suspended by Teflon thread in a Teflon container. After 2-g glass powders (~8 μm) and 300 mL DI water were added, the container was placed in an incubator at 80 °C for up to 5 days of reaction. One glass piece was taken at each setting time, rinsed with DI water and dried in a desiccator.

2.3. Scanning electron microscopy (SEM)

The surface morphologies of the glass pieces were measured by scanning electron microscopy (MERLIN Compact, ZEISS, Germany). The samples were coated with carbon before analyses.

2.4. XPS spectroscopy

The XPS measurements were conducted with a high-performance K-Alpha X-ray photoelectron spectrometer (Thermo Fisher Scientific, United States) in ultra-high vacuum. The glass powders were pressed into a copper holder to obtain a smooth surface [27]. Monochromatic Al Kα radiation ($h\nu = 1846.6$ eV) was used with 15 kV accelerating voltage. The survey scan was collected from 1250 to 0 eV, at 1 eV step size, 200 ms dwell time and 130 eV pass energy. C-1s, P-2p, O-1s, Al-2p, Na-1s, K-2p and Ba-3d narrow scans were collected at 0.1 eV step size, 120 ms dwell time and 20 eV pass energy. The positive charge on the glass surface was compensated by a low-energy electron flood (1 eV, 100 μA).

The surface composition of the leached powders was determined by XPS survey scan, and the relative sensitivity factor (RSF) of each element was provided by K-Alpha database.

Further spectra fitting analyses were performed using CasaXPS software. The C-1s and O-1s narrow scan spectra were smoothed and fitted with mixed Gaussian/Lorentzian line shapes. The high-resolution C-1s peak spectra were fitted into alkyl (C–H/C–C), alcoholic (C–OH), carbonyl (C=O), carboxyl (O–C=O), and carbonate (CO₃²⁻) components [28], and the binding energies of all elements were corrected with the adventitious alkyl (C–C) peak at 284.6 eV [27].

2.5. ATR-IR spectroscopy

The ATR-IR measurements were conducted with a Nicolet 6700 spectrometer (Thermo Fisher Scientific, United States), with Smart-ITR accessory equipped with a diamond crystal installed. The incident angle was 42°. A sensitive MCT/A detector was employed to detect the extremely weak signal. Each sample was measured in the region from 4000 to 650 cm⁻¹ for 64 rounds, with a resolution of 4 cm⁻¹.

3. Results

3.1. ICP-OES

The concentrations of ions leached into solution were normalized by the composition of the pristine glass determined by the ICP-OES

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