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# Influence of chemical polishing on fluorophosphate fiber preform

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**Abstract**

Surface treatment of optical glass fibers is an effective method to clean the impurities and remove the damage layer, such as scratches caused by the cold processing. Optical losses at the fiber core/cladding interface can thereby be decreased. Using X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM), we studied the surface composition and morphology of fluorophosphate (FP) glass after polishing and chemical etching. In addition, we investigated the etching mechanism and the optimal concentration of etching solution. Results show that the surface composition is very close to that of the bulk glass for some elements, and the root-mean-square (RMS) roughness is approximately 0.821 nm. A method including acid–alkali pretreatment and a second polishing step is a novel way to improve the smoothness of surface and to eliminate defects such as contaminants and scratches.


*Keywords:* B. Impurities; FP glass; Chemical polishing; Damager layer

## 1. Introduction

In the past few years, $Yb^{3+}$-doped single-frequency fiber lasers have been extensively investigated for many applications in science and industry, such as remote sensing technology and astronomy and meteorological observation [1,2]. Ring and linear cavities are the two types of resonant cavities in single-frequency fiber lasers, and the latter is much more stable than the former. Constrained by the low $Yb^{3+}$ solubility, the popular $Yb^{3+}$:silica fiber is not suitable for short-linear-cavity single-frequency fiber lasers with output over a hundred mW [3,4]. Fortunately, highly $Yb^{3+}$-doped phosphate glass fibers have proved to be a good candidates for such lasers [5–7]; a stable 400-mW single-frequency $Yb^{3+}$ laser was achieved with a 0.8-cm long $Yb^{3+}$:phosphate glass fiber. However, it is difficult to obtain laser output from $Yb^{3+}$:phosphate glass chips even at low temperature because narrow stark splitting of $Yb^{3+}$ in phosphate glass induces a series of shortcomings, such as low emission cross section at the lasing wavelength and thermal blocking problems [8]. However, a recent demonstration of a 1.16-W laser fabricated with $Yb^{3+}$: fluorophosphate (FP) glass [9] has shown that this material exhibits much better laser performance than $Yb^{3+}$:phosphate glass. This suggests that $Yb^{3+}$:FP glass fibers may be a good alternative for short-linear-cavity single-frequency fiber lasers.

For many applications, the raw glasses need to undergo extensive processing including cutting, grinding, and polishing. The fiber preform is fabricated by the rod-in-tube technique. After cold working, the surface of optical-fiber preforms will contain defects [10], such as contaminants, scratches and cracks. During the fiber drawing process, defects will end up in the core/cladding interface, and this will inevitably decrease the strength of optical fiber and increase optical losses in the fiber. It is known that acid treatment is an effective method to enhance the mechanical strength of optical-glass surface [11,12] and to eliminate micro cracks and surface defects [13–15]. Yang reported HCl etching combined with subsequent high-temperature treatment could improve surface quality of barium gallogermanate glass [16]. Mellott demonstrated melt aluminosilicate glass surface was fabricated that possessed a surface composition close to that of

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the bulk using chemical etchants (NaOH, $NH_4OH$ and HF) [17]. Barnes analyzed the surface composition of phosphate glass was varied with HCl, KOH and reverse osmosis water [18]. Although acid can improve the quality of surface, etch pits are produced, and the local refractive index changes. This leads to surface roughness at the micron scale when acid use is excessive. Thus, acid etching alone should not be used during the surface treatment of the optical preform. According to references [18,19], the alkaline solution 3 mol/L KOH is ideal for removing the damage layer without producing pits on the surface. Therefore, a method of successive acid–alkali treatments may etch the surface effectively without any pits.

In this study, we developed a chemical polishing method for FP glass in which acid–alkali steps were used. The surface composition and morphology of FP glass before and after treatment were investigated, and the root-mean-square (RMS) roughness [20] was used to evaluate the effect of the etching methods on FP glass. A new method of chemical etching and mechanical polishing is described to improve the quality of FP glass surface.

## 2. Experimental

The formula for $Yb^{3+}$ doped FP glass is $10Al(PO_3)_3$-8Sr$(H_2PO_4)_2$-58$RF_2$-17$AlF_3$-7BaO-3$YbF_3$ (R = Mg, Sr, Ba). The glass was prepared by the conventional melting-quenching method. Raw materials were fully mixed and then melted at 1100 °C in a platinum crucible. After stirring and refining, the melt was cast into a steel mold and annealed at 480 °C. Then the annealed samples were cut to the size of $10 \times 10 \times 2$ $mm^3$ and polished with $CeO_2$ slurry for the etching experiments and optical measurements. The weight of each polished glass chip was 1.065 g, measured by analytical balance. And the surface area was 280 $mm^2$. We used three samples as the experimental subject to take comparison in each stage of experiment and all data had little difference in comparison analysis, so the results were obtained from their average. In this study, all experiments were performed at room temperature.

The glass was soaked in deionized water and then blown dry with nitrogen before all surface treatments and tests. The glass was suspended in a beaker, ensuring that all six faces were in contact with the chemical etchant. After etching, the changes in surface composition were probed using X-ray photoelectron spectroscopy (XPS) [21]. The dissolution rate $D_R$ ($g\ mm^{-2}\ h^{-1}$) describes the corrosion rate of the glass in the acid solution [16]. It is calculated as follows:

$$D_R = \frac{\Delta W}{t \cdot S} \tag{1}$$

where $\Delta W$ (g), $S$ ($mm^2$) and $t$ (h) are the weight difference of the glass before and after etching, the surface area of the glass, and the time of immersion in the acid solution, respectively.

The surface morphology was examined using an atomic force microscope (AFM). The root-mean-square (RMS) roughness $R_{RMS}$ is defined as the root of the average square of height deviations from the mean elevation plane, calculated from the relative heights of each pixel in the image:

$$R_{RMS} = \sqrt{\frac{1}{N}\sum_{i=1}^{N} Z_i^2} \tag{2}$$

where $z_i$ equals the difference in height from the mean plane for each point $i$, and $N$ equals the number of points measured.

The infrared transmission spectrum was measured by a Nexus FT-IR spectrometer (Thermo Nicolet) in a wavelength range of 2.5–4.5 μm. The elemental concentration of ions in solution was acquired using inductively coupled plasma atomic emission spectroscopy (ICP-AES). To facilitate interpretation of the data, the measured solution concentration of component $i$, $s_i$, was normalized to the concentration of that component in the glass. This normalized concentration of element $i$ ($[X_i]$ in $g/cm^2$) in solution at any time is calculated by [18]:

$$[X_i] = \frac{[s_i]}{SA/V} / [w_i] \tag{3}$$

where $[s_i]$ is the weight percent of component $i$ in solution (g/mL), SA is the surface area of the glass ($cm^2$), $V$ is the solution volume (mL), and $[w_i]$ is the weight percent of component $i$ in the bulk glass. The process of dissolution can be congruent (the same for each element released from the glass) or incongruent (dissolution accompanied by precipitation). In this case, where congruent dissolution occurs, $[X_i]$ provides a dissolution parameter for the glass through the use of:

$$D_i = \frac{[X_i]}{\rho} * 10^7 \tag{4}$$

where $D_i$ is the layer thickness of glass dissolved (nm), $[X_i]$ is the normalized concentration of component $i$ in solution ($g/cm^2$), and $\rho$ is the density (3.79 $g/cm^3$). The values of $D_i$ can also reflect the dissolution rate of glass in acid and alkaline solution.

## 3. Results and discussions

The structural model of FP glass can be described as $Al(F,O)_6$-octahedral chains bonded by mono- and diphosphate groups [22]. Phosphorus and oxygen form a $[PO_4]$ tetrahedral framework for the glass structure, and $Mg^{2+}$ and $Al^{3+}$ are considered network modifiers, encompassing network former. The electric-field distribution in the glass is asymmetrical because of the P=O bonds, resulting in thermodynamic instability. The strong ionic bonding character and covalent bonding character exist in the schema of structure together, by which FP glass is susceptible to surroundings. For example, FP glass reacts with molecular water in the air