Surface analysis and treatment of extruded fluoride phosphate glass preforms for optical fibre fabrication

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Abstract
Fabrication of fluoride phosphate glass optical fibres using the extrusion method for preform fabrication has been studied using the commercial Schott N-FK51A glass. The extrusion step was found to create a surface layer of differing composition from the bulk glass material, leading to defects drawn down onto the optical fibre surface during fibre fabrication, resulting in high loss and fragile fibres. Similar phenomena have also been observed in other fluoride-based glasses. Removal of this surface layer from preforms prior to fibre drawing was shown to improve optical fibre loss from >5 dB/m to 0.5 – 1.0 dB/m. The removal of this surface layer is therefore necessary to produce low-loss fluoride phosphate optical fibres.

Introduction

Fluoride phosphate (FP) glasses have various speciality optical fibre applications including mid-IR lasers,[1] UV transmission [2,3] and radiation dosimetry.[4,5] Being closely related to other fluoride glasses, FP glasses share similarities relating to preform and fibre fabrication, such as the high scattering loss and fragility associated with surface defects. In the literature, these surface defects in fluoride glasses are commonly attributed to crystallisation during the preform and fibre fabrication processes.[6–9] Here we show that a compositional change at the surface of the glass during preform fabrication is also a contributing factor to the poor surface quality observed on optical fibres fabricated from fluoride glasses.

During the fabrication of optical fibres from FP glass, changes to the surface layer of the glass can occur, resulting in a product with poor quality. Hence rather than a rough preform surface contributing to the high scattering loss and fragility of fibres, the problem might rather be due to a difference in softening point between the surface layer and internal material of a fibre preform. Here we observe that although the surface roughness is equal before and after removal of the surface layer, the fibre quality has significantly improved.

The fabrication of optical fibres from FP glasses with similar composition is not extensively reported in the literature, however several studies have demonstrated success.[10–12] Two different preform fabrications methods, extrusion and casting, have both been studied. Casting, a common method for preform fabrication using soft glasses, requires a skilled operator and is prone to producing bubbles within the preform.[9] In comparison the extrusion method does not introduce bubbles into the glass, the surface is smooth and it allows fine control over the preform geometry.

The extrusion method was demonstrated previously for fabrication of core/clad fluoride phosphate glass fibres.[10] Approximately 45 cm^3 volumes of glass were extruded through graphite components in a dry nitrogen atmosphere using a pressure of 95 bar. This previous work was done as a core/clad extrusion using stacked billets, where the core surface is not exposed to free atmosphere during extrusion and fibre drawing. However, for air-clad and microstructured fibres and for fibres made using the rod-in-tube technique, the core surface is exposed to atmosphere, thereby potentially allowing surface changes to occur.

In this paper, we examined extrusion of FP rods and drawing them into air-clad (i.e. unstructured) fibres to determine the impact on a fibre with a core exposed to free space. The initial fibres fabricated from FP glass had high loss and were fragile, prompting further investigation into the fabrication conditions for FP optical fibres. The effect of the extrusion process on the surface layer of the extruded preform was investigated using commercially acquired FP glass, followed by further preparation of the preform surface prior to fibre drawing in order to produce optical fibres with improved loss and mechanical strength.

Extrusion and Fibre Drawing Conditions

Glass Samples

Experiments shown here were all performed using a commercially acquired FP glass, product N-FK51A from Schott Glass Co. The composition of this material as determined using an electron microprobe is provided in Table 1. The material was procured as 30 mm diameter rods, the appropriate diameter for extrusion, then cut into 70 mm lengths, weighing approximately 180 g, and finally polished.

Extrusion

The extrusion method was used to produce 10 mm diameter fibre preforms, using the same method as has been described previously.[13, 14] After several trials to determine suitable temperature, force and die materials, the optimum parameters for the Schott N-FK51A glass was found to be extrusion at a speed of 0.05 mm/min at a temperature of 525   C, which requires a force of approximately 5 kN. The extrusion is performed in a nitrogen atmosphere and the die components are fabricated from stainless steel. Following extrusion, preforms are annealed at 450   C to relieve any stress remaining in the glass.

Fibre Drawing

Optical fibres were drawn into unstructured (air-clad) fibres with 160 μm diameter using an RF furnace with a graphite susceptor. Following several experimental trials adjusting draw speed and temperature, fibres were drawn at a temperature of 750   C and a preform feed rate of 2.5 mm/min. The drawing furnace was purged with N_2 during the heating and drawing stages. Following fibre drawing, fibres were tested for their loss using the standard cut-back method.[15]

Preform Analysis and Improvement

Preform Observation

Following initial extrusion and fibre drawing trials, fibres had high loss of approximately 5 - 15 dB/m and poor mechanical strength. Following optimisation of various extrusion parameters (speed, force, temperature and die material) and fibre drawing parameters (temperature, speed, tension), the fibre quality was marginally improved, but fibres still had high loss of approximately 5 - 10 dB/m and poor strength.

Observation of the neck-down region of the fibre drop showed the creation of a corrugated structure which was being drawn down on the surface of the optical fibres as shown in Figure 1 (a). The topography of this corrugated structure was measured using a Brooker GT-K Optical Profiler and is shown in Figure 1 (b). This corrugated structure has been observed previously in fluoride glass fibres, [16,17] fluoroindate glass fibres [8,9] and fluoride phosphate glass fibres.[18] For fluoroindate glass fibres, these defects on the fibre surface were found to cause both high scattering loss and increased fragility.[8]

Surface Layer Removal

Following this observation of the corrugated structure on the neck-down region and the high loss of these fibres, it was presumed a layer with a slightly different composition had formed on preforms during the extrusion process. Therefore, following another extrusion trial, the preform was treated by removal of the surface layer over part of its length. This surface layer was removed using a solution of 40 nm colloidal silica on a silk polishing pad, and an over-head rotational stirrer. The optical profiler was used to measure the surface roughness of the preform through the treatment process, this is shown in Figure 2. The initial as-extruded surface had a surface roughness of $R_a = 2 – 3$ nm, however as soon as surface layer removal began, the surface became more rough, with $R_a = 22$ nm. As the surface layer was further removed the surface roughness decreased back to the original value of $R_a = 2 – 3$ nm after approximately 90 - 120 minutes, this is indicative of a layer of varying composition and mechanical properties from the bulk material being broken and gradually removed. Measurement of the preform diameter before and after surface layer removal with callipers did not show a difference, indicating the amount of material removed was less than 10 μm.

Energy dispersive X-ray spectroscopy (EDX) was then performed to assess the composition of the as-extruded section to the surface treated section. The results are shown in Figure 3. A higher oxygen and lower fluorine content was found in the untreated, as-extruded layer of the preform compared with the treated section.

Fibre Surface Roughness and Loss Measurement

Following the surface analysis of the extruded glass, fibre preforms were treated to completely remove the surface layer prior to fibre drawing. The neck-down region of a fibre drawn from a treated preform can also be seen in Figure 1 (a) (right), showing a reduction in the amount of corrugation. Surface roughness of fibres was measured using the optical profiler. Fibres made from an untreated preform had a surface roughness of $R_a = 71.5 – 8$ nm, while fibres made from a treated preform had a reduced surface roughness of $R_a = 31.6 – 8$ nm. The loss of fibres both before and after treatment can be seen in Figure 4. The untreated preforms result in optical fibres with loss greater than 5 dB/m, while treated preforms produce fibres with reduced loss of 0.5 – 1 dB/m.

Discussion

Optical fibres fabricated from treated preforms with the surface layer removed were observed to have lower loss and higher mechanical strength, indicating the importance of this surface layer to be removed. The similar surface roughness between untreated and treated preforms implies it is the composition and properties of the surface layer itself which causes the neckdown corrugations, as no significant amount of surface defects were observed on the preform following extrusion. It is assumed the composition of the surface layer, containing a higher oxygen to fluorine content, in the untreated preforms led to more corrugations on the fibre neckdown, which resulted in higher surface roughness of the fibres and consequently high fibre loss. During the extrusion process, it is possible a small amount of fluorine is out-gassing from the surface layer. In addition, introduction of more oxygen into the glass from the atmosphere might be occurring at the extrusion temperature. Although there is a flow of nitrogen introduced from beneath which flows upward around the extrusion, there is no air-tight seal at the top of the extrusion; ambient atmosphere has an opportunity to come into contact with the glass. These two effects would explain the slight compositional difference between as-extruded and treated surfaces on the preform. This compositional difference causes a slight difference in thermal properties between the surface layer and the bulk material.

Fluoride phosphate glasses with a higher oxygen content have a higher glass transition and melting temperature,[19] hence during heating the surface layer would remain hard while the inside had softened. This would crack the surface layer and produce corrugations as it is drawn down, the fibre surface would therefore have many defects on the surface. This has been shown previously to cause fragility and high loss in fluoride and fluoroindate optical fibres.[8,16] Treated preforms which no longer have this surface layer show reduced corrugations on the neck-down region. Fibre loss and strength are both improved, which is attributed to a lower concentration of surface defects for the fibres. The loss in the 0.5 - 1 dB/m is equivalent to the bulk loss of the material,[20] hence any further improvement in fibre loss would need to come from the glass materials development. Fibre losses of 0.5 - 1 dB/m are similar to glasses of similar composition studied in the literature where loss in the 0.1 - 1 dB/m region was reported.[10, 11]

Conclusions

Fluoride phosphate glass fibres were fabricated from commercially acquired Schott N-FK51A fluoride phosphate glass. Initial fibre quality was poor, which is attributed to the formation of a surface layer with slightly different composition to the bulk glass formed during the extrusion step. Optical fibres fabricated from preforms following a surface layer removal step were found to have improved quality and reduced losses down to the material loss of 0.5 - 1 dB/m in the wavelength range of 400 – 1600 nm.
For fluoride and fluoride phosphate glasses, the effect of preform fabrication steps on the surface of the glass can significantly affect the quality of the optical fibre. This may be a contributing factor to the poor quality of fluoride glass optical fibres which have been fabricated without any preform surface treatment steps. Analysis of the glass surface layer for any compositional change, and subsequent processing, can significantly improve the optical fibre quality.

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Table 1: Composition of Schott N-FK51A FP glass as determined by analysis with an electron microprobe.

Element	Atomic Percentage
O	22.6
F	42.9
Na	0.1
Mg	2.4
Al	10.7
P	5.6
Ca	6.0
Sr	5.5
Ba	4.2

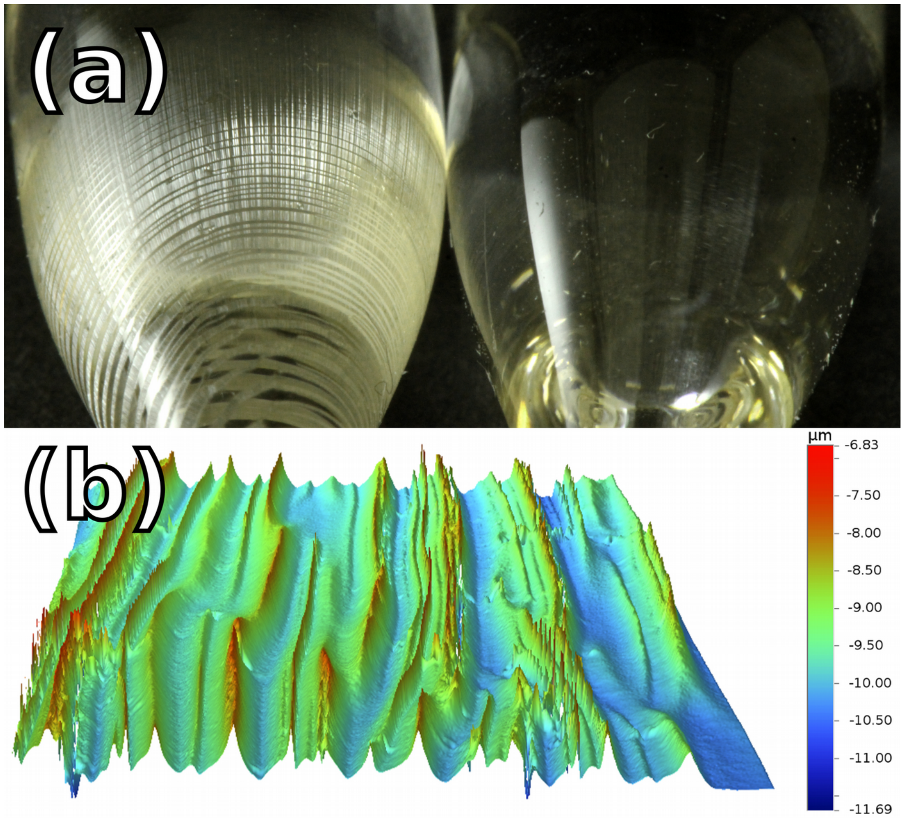

Figure 1: (a) Photograph of neckdown region of unpolished (left) and polished (right) preforms, all other fabrication conditions were constant. (b) Topography of the corrugations on the untreated preform neckdown measured with an optical profiler.

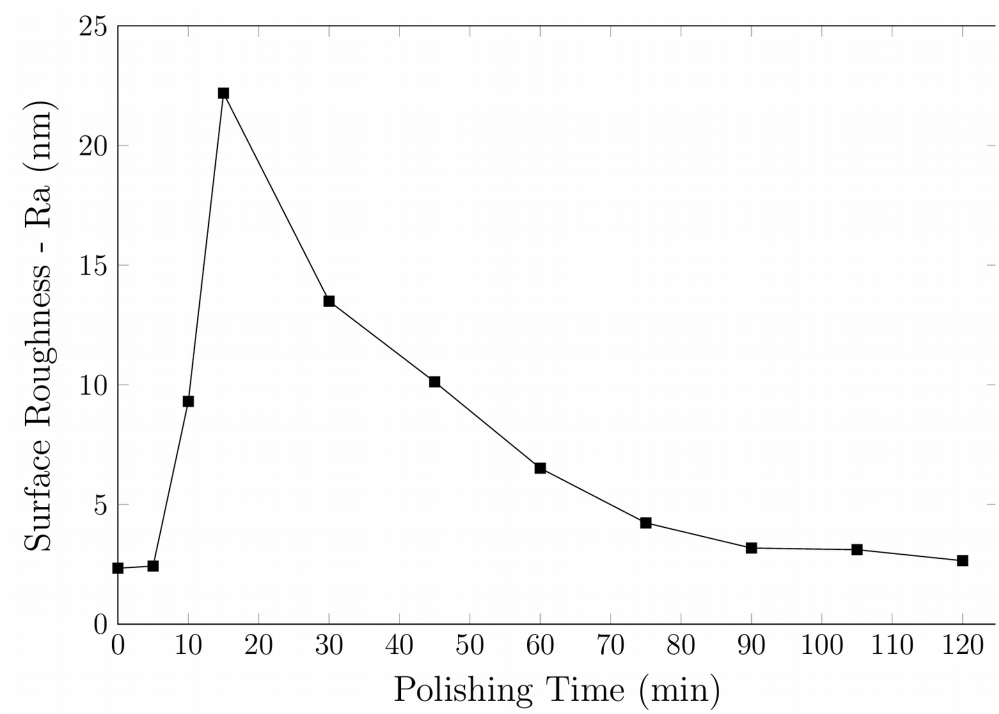

Figure 2: Surface roughness with respect to the surface layer removal time of an FP preform surface treated with colloidal silica. Lines are included only as a guide for the eye.

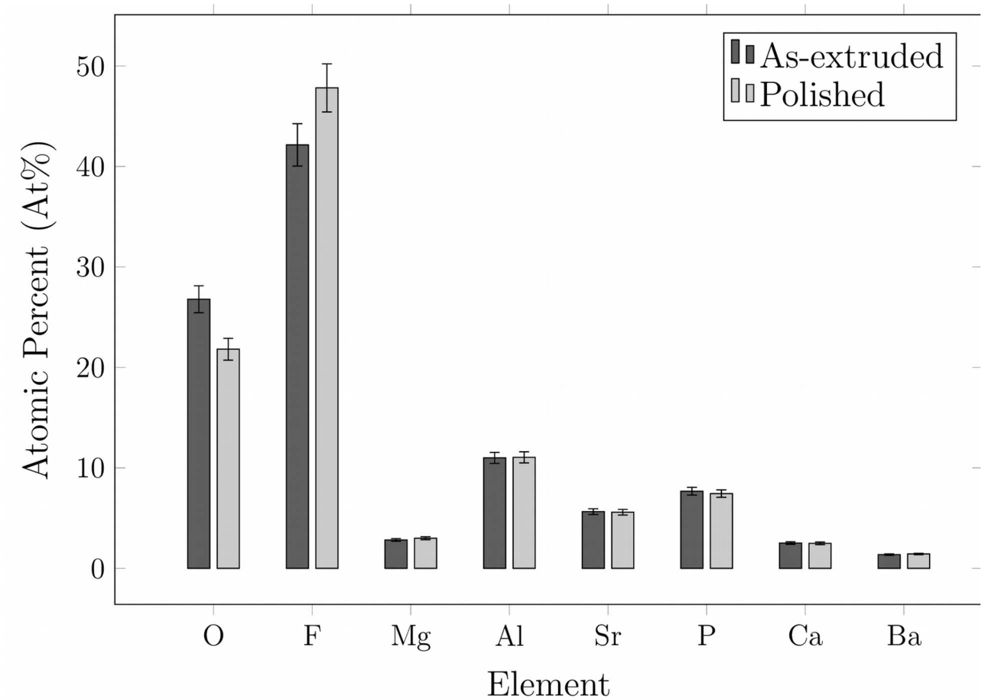

Figure 3: Comparison of the composition of untreated and treated preform surfaces, Composition of untreated and treated preform surfaces, measured using a scanning electron microscope in EDX mode.

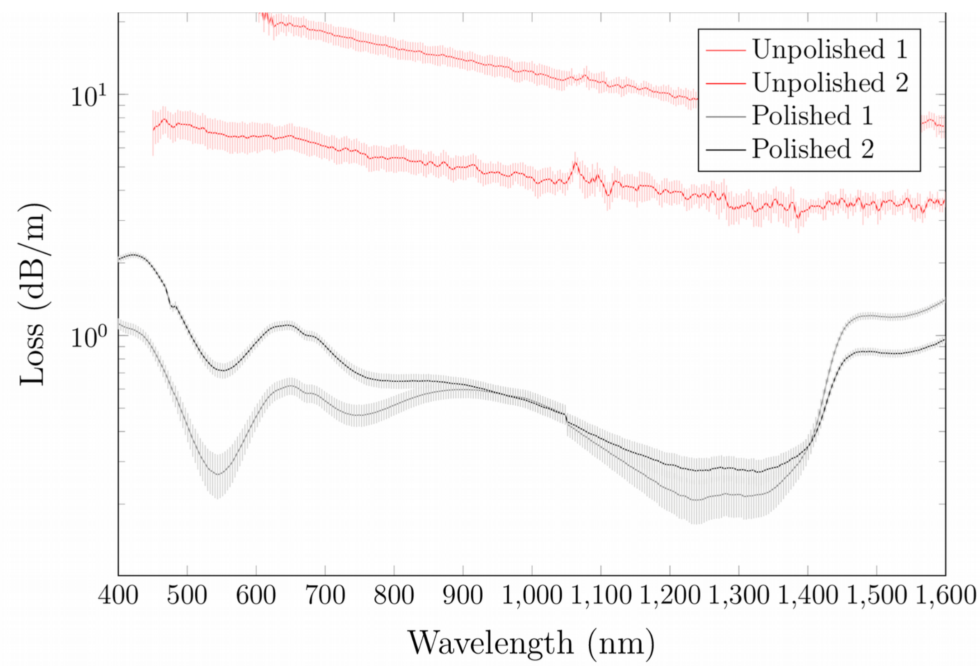

Figure 4: Loss spectra of fibres fabricated from untreated (red) and treated (black) extruded preforms.