Pre-polymerization fibres treatment with adhesion promoter

One mechanical drawback of hybrid polymer/silica fibre structures is lack of adhesion at the interface of the organic/inorganic materials. The active functional group of an acrylate monomer does not make a robust bond with silica. Therefore 3-TPM was used to overcome the bonding failure. Before commencing waveguide fabrication the fibre ends were cleaved and cleaned with acetone, then they were immersed in a plastic bottle filled of adhesion promoter in a refrigerator for different time durations (5 s. 20 s, 5 min, 20 min, and 1 hour) as shown in Fig. 1.

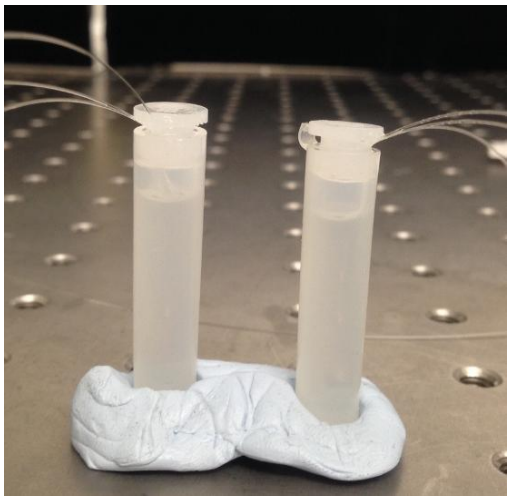

Fig. 1: Fibre ends are treated with 3-TPM by immersing the cleaved ends into the liquid for different time durations before waveguide fabrication.

Then the fibres were shaken well and dried by manual air duster. The ultimate goal of the treatment and a better adhesive joint is to produce an interface that is strong and durable. An excellent interface after treatment should acquire minimal imperfection to support a high quality polymer bridge.

Effect of treatment duration

Polymer bridges were fabricated by unidirectional illumination using the experimental set up shown in our previous paper*. The optical characterization was also done with same procedure. The experimental results show the substantial impact of the adhesion promoter, where uncured

liquid surrounding the bridges after polymerization was aggressively rinsed by pouring successive drops of ethanol. In contrast to the fabrication using untreated fibre ends the bridges formed after the pre-treatment showed no fragility as a result of rinsing. Moreover, waveguides were laterally misaligned to test the polymer/silica adherence. For each of the pre-treatment durations a set of three bridges with 200 µm length was fabricated and deflected transversely in 5 µm steps and the maximum deflection before breakage was recorded for each bridge. From Fig. 2, one can notice that for untreated fibres the bridge can be deflected to about 10 µm on average and on one occasion it did not survive rinsing. The maximum lateral deflection increases with increasing treatment duration, although the trend is not linear but there is a plateau region from 5min to an hour. A saturation like this is expected as the available reaction sites on the silica surface become filled. In general, 3-TPM was very effective, such that the treated fibres held polymer bridges for about 90 µm lateral shift.

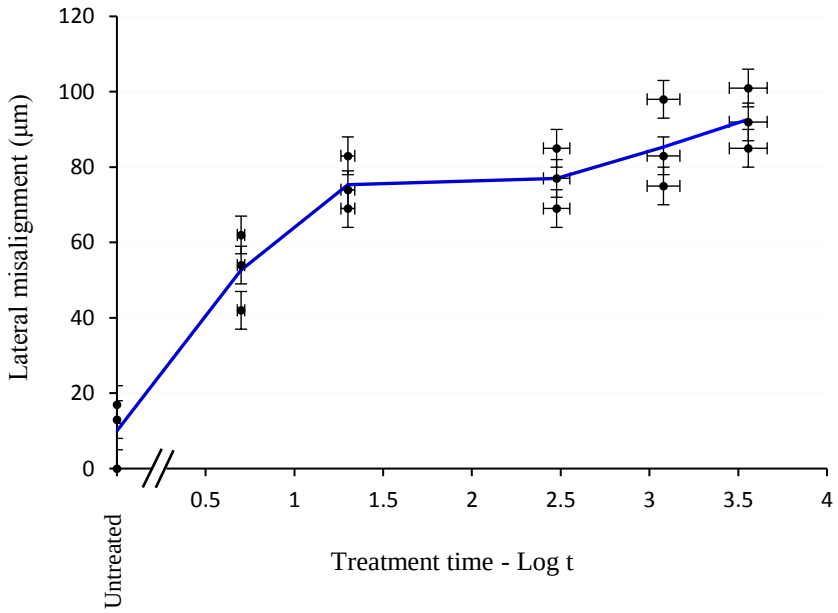

Fig. 2: Maximum misalignment before polymer detaches from fibre as a function of treatment time with 3-TPM. Points show three different repeats for each treatment, line shows the mean.

Figs. 3 (a-e), show micrographs of 200 µm bridges associated with fibres which were untreated, treated for 5 s, 20 s, 5 min, and 20 min respectively with straight waveguide, maximum off set, and at fracture point. The comparison was done for polymer bridges that were made under the same physical and chemical circumstances for instance; curing power, exposure

time and bridge length. Also the fibre ends were fixed firmly in the v- grooves by a pair of strong magnets in order to prevent the fibres moving freely. Some bending of the fibres was still observed and so deflection was measured from the micrographs, not from the stage translators.

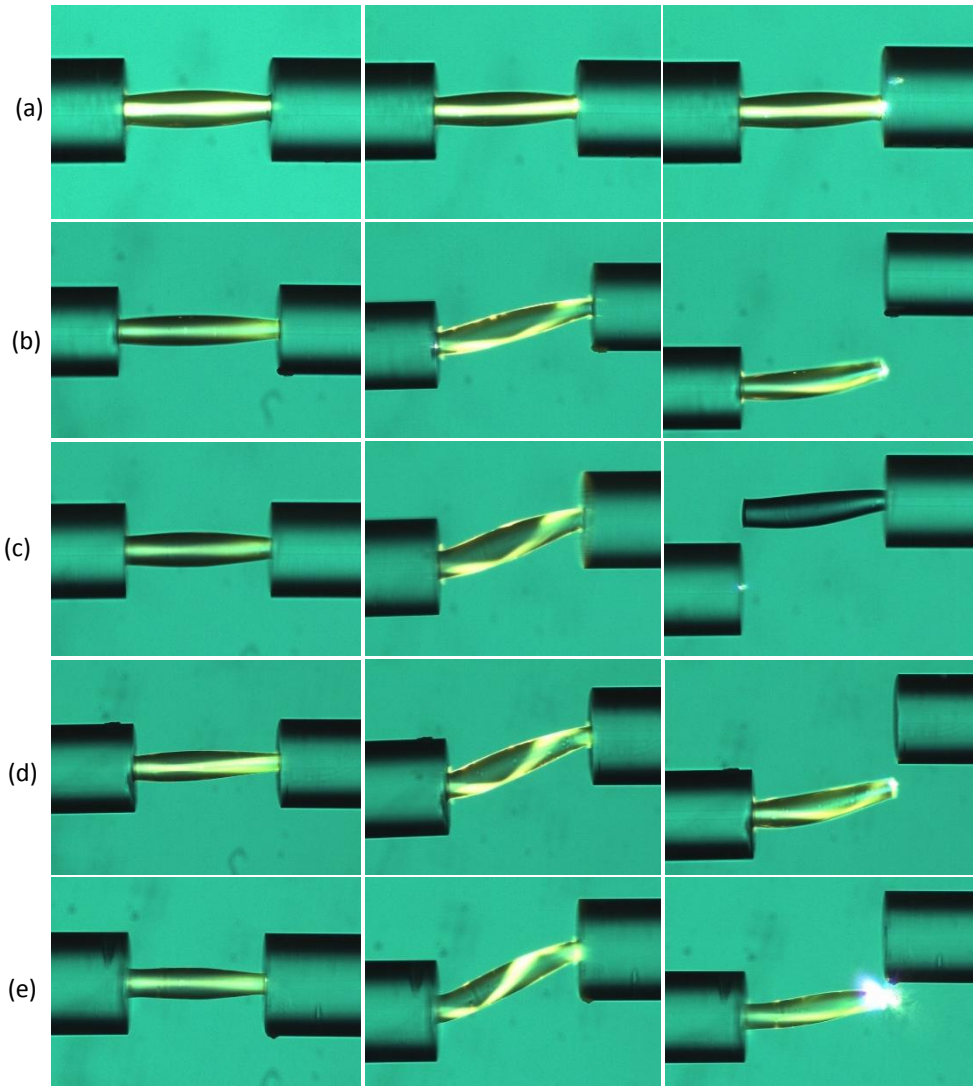

Fig. 3: Straight waveguide (left), then maximum lateral deflection (centre) and detaching (right) corresponding with a) Untreated fibres. b) Fibres were treated for 5 s, c) 20 s, d) 5 min and e) 20 min. Maximum deflection was measured from micrographs of the centre column. Bend in the fibres is evident from the fibres springing further apart when the bridge breaks (right).

Misalignment was performed by shifting the output fibre by 5 μm steps and for each step the bridge was pictured and the loss was measured at 1550 nm. Fig. 4 shows the effect of treatment

time on loss evolution against lateral misalignment. Generally the loss increases with deflecting polymer bridges laterally. The impact of treatment is clear from the optical transmission of the bridges in the first three steps up to 10 µm displacement. One can see that loss significantly decreases with increasing treatment time. The loss associated with the polymer waveguide fabricated between untreated fibres is about 14 dB when we deflect the output fibre 10 µm, while for the same misalignment the waveguide loss associated with modified fibres dropped to about 6 dB. The optical improvement can be attributed to the stronger adhesion between the polymer bridge and fibre end at interfaces. No further optical improvement was observed corresponding to the fibres treated for longer time durations.

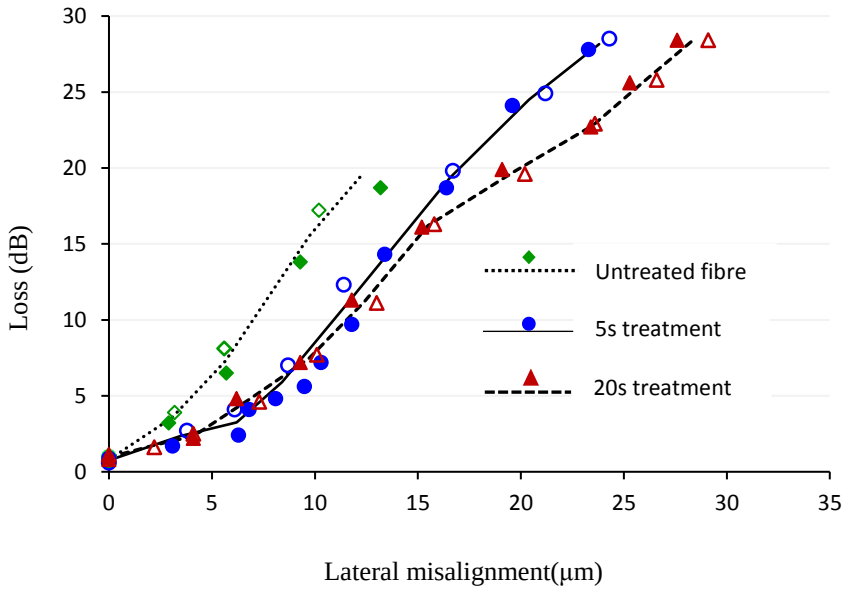

Fig. 4: Optical loss development against measured lateral misalignment associated with 200 µm polymer waveguide fabricated between fibres which have been treated for different time durations. Points show two different repeats for each treatment, lines are the mean.

Misalignment tolerance

The mechanical assessment was conducted by laterally displacing one of the fibres from original aligned position at 5 µm steps on the micrometre knob of the translation stages. Although, the 5 µm on the knob does not exactly mean 5 µm deflection of the waveguide, since fibres show some bending flexibility despite of they were fixed firmly on the translation stages.

Therefore, for accurate misalignment measurement Image J program was used for precisely measuring fibre deflection from micrographs. Fig. 2 (a-d) show the maximum lateral deflection of polymer bridges with different lengths (100 μm, 200 μm, 300 μm, and 400 μm).

The optical transmission variation against lateral misalignment and the data associated with various lengths of polymer waveguides is presented in excel file named (Loss vs lateral misalignment for (300, 400, 500) micron waveguides).

Polymer bridges under pure tensile stress

Further mechanical characterization was conducted by putting polymer waveguide under pure tensile strain by elongating polymer channel. Throughout this experiment the joint strength at polymer/fibre interfaces was evaluated by measuring breaking strain. The optical transmission was also investigated as a response to the tensile strain.

To investigate the effect of the polymer waveguide's size on its mechanical properties, three different sets of bridges were fabricated under the same experimental conditions, for instance curing power of 12.8 μW and PETA drop contains Eosin concentration of 0.5wt. %. Exposure time was the only parameter varied to obtain various bridge sizes with different cross-sectional areas as well as different joint areas at the polymer/fibre interfaces. The challenging point in this experiment was the drop's size and shape, since it is crucial in the polymer size through surrounding oxygenation. Short exposure time leads to thinner bridges and vice versa, as shown in Fig. 5 (a-c). The three columns are all 300 μm long waveguides which were cured for 5 s, 30 s and 60 s respectively. Then the polymer bridges were longitudinally extended and pictured at every 5 μm step before they detached from fibre.

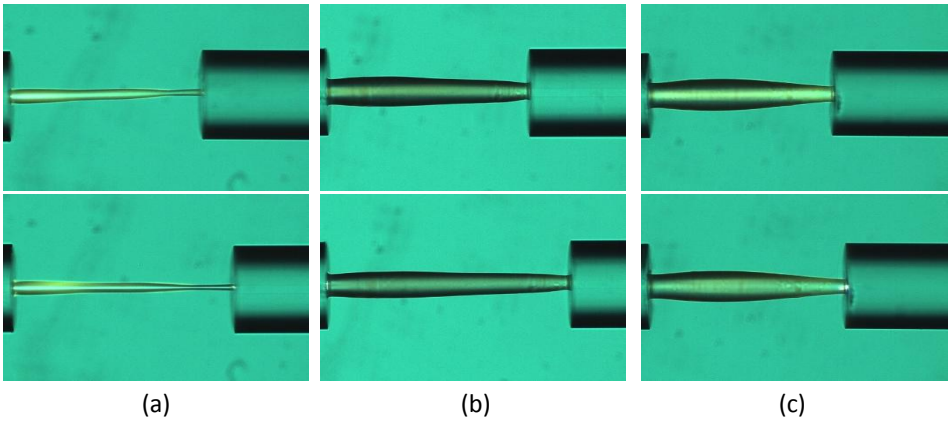

Fig. 5: Optical microscope images of 300 µm long polymer bridges fabricated with different exposures at different strain values. a) Thinner bridge cured for 5 s. b) Medium sized bridge cured for 30 s. c) Wider bridge cured for 60 s.

Different bridge sizes showed different maximum tensile strain values, which was not a monotonic trend; strain values were 13%, 23%, and 8% for thinner to wider bridges respectively, Fig. 6. Although the adhesion interaction area increases at the interfaces as exposure time is extended which in turn increases the adhesion between two materials, the polymer bulk size also enlarges which results in strengthening of the polymer bulk which results in less elasticity. Consequently, the resultant maximum strain before breakage will be determined by the competition between the adhesion joint strength, which cannot be much enlarged beyond some certain value, and the polymer structure size which is to a less extent limited. The bridge with medium structure size shows the largest strain value.

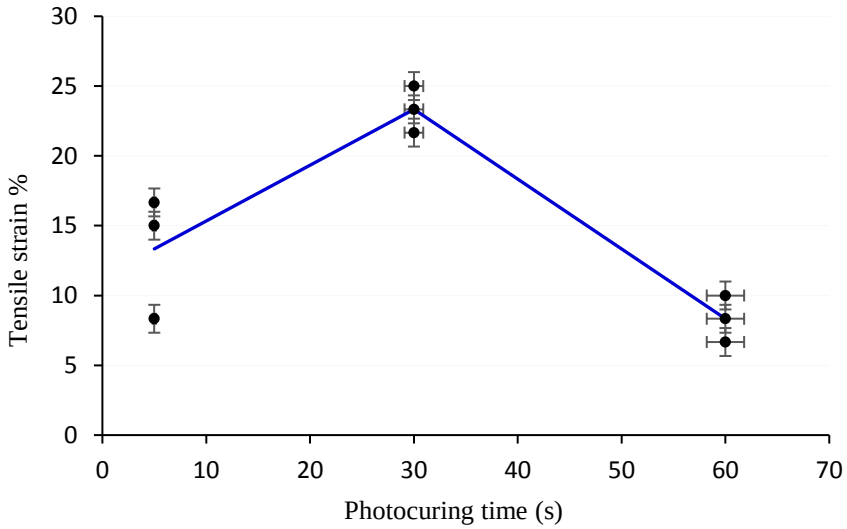

Fig. 6: Maximum strain against photocuring time corresponding to bleached 300 µm waveguides fabricated with the same beam power. Points show three different repeats for each treatment, the line is the mean.

A medium sized waveguide is not only the best mechanically but also optically more efficient. Fig. 7 shows the optical transmission under the strain was also examined. The result shows that average insertion loss decreases from 0.75 dB to 0.3 dB for tensile strain in the range between 4% and 7%, then it gradually increases but it does not return back to the value associated with 0% strain. Photobleaching immediately after rinsing results in local temperature raising by exciting the Eosin residuals remained in the waveguide structure, the high temperature elongates the waveguide and causes some bending. The initial tensile stress releases the bending and leads to loss reduction. Then after a certain strain value the applied pulling force starts to exert detaching force at the polymer/fibre interfaces and causes loss increasing again until the polymer detaches.

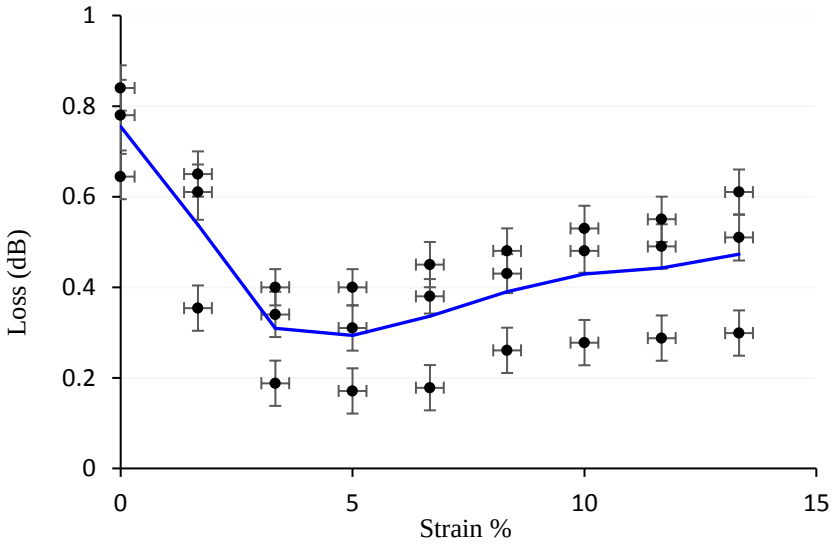

Fig. 7: Development of the insertion loss at 1550 nm of a 300 µm long waveguide fabricated with 30 s exposure as a function of tensile strain. Points show three different repeats for each treatment, the line is the mean.

The variation of insertion loss is not restricted to wavelengths where the waveguide is single moded (1550nm) but also it follows the same pattern of loss variation over the full spectral range (350 nm- 1750 nm), as shown in Fig. 8.

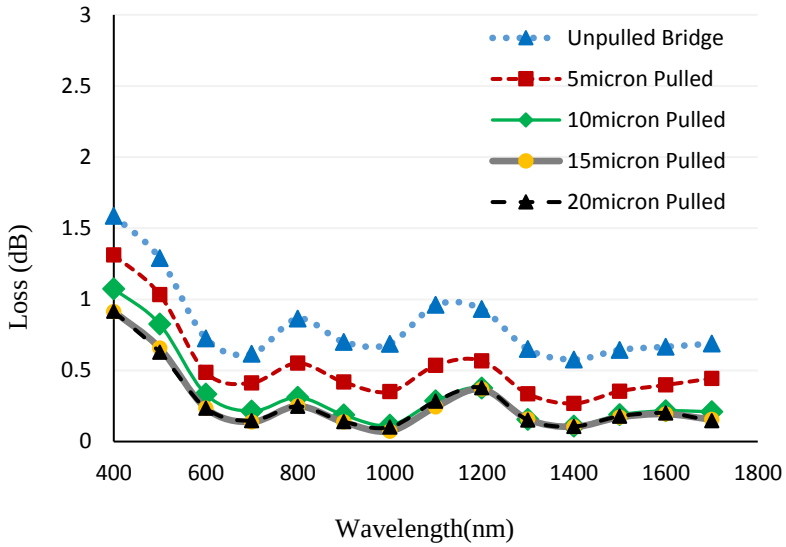

Fig. 8: Optical transmission variation of optimum size 300 µm long waveguide under waveguide elongation over a broad spectrum. The waveguide was fabricated from PETA droplet cured for 30 s with beam power of 12.8 µW.

Effect of photobleaching

To investigate the crosslinking degree and conversion rate of polymer structure on the maximum strain value, polymer waveguides were put under tensile strain immediately after rinsing. In the previous case the waveguide was photobleached until the florescence weakened then the stress applied, but in this experiment the beam was blocked after the rinsing. Waveguides of 300 µm long were fabricated, keeping the same optical and chemical parameters used for medium size waveguide in the previous case. Tensile stress was applied to the polymer bridges when crosslinking is expected to be low. Fig. 9 shows an unbleached 300 µm long waveguide under tensile strain. The bright fluorescence indicates that the waveguide is unbleached. The average of maximum tensile strain increased to 32% for three new waveguides, it has increased from 23% for bleached waveguides. The result verifies the postulation that the larger the degree of crosslinking and conversion rate the smaller the tensile strain.

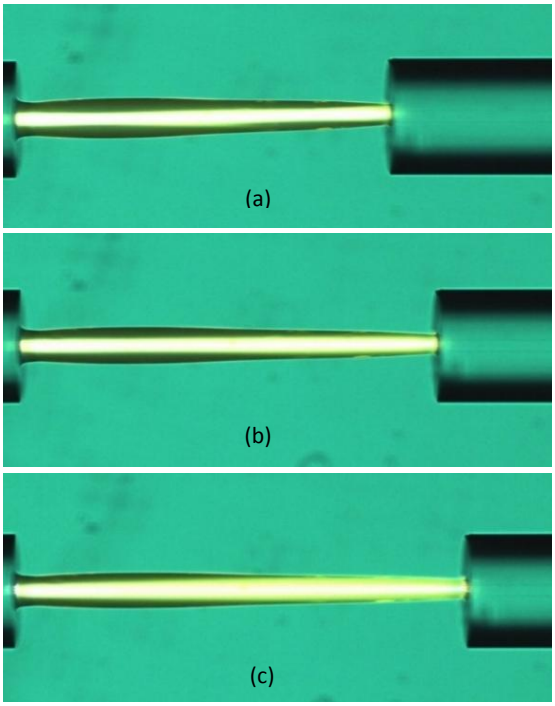

Fig. 9: Optical micrographs of 300 µm long waveguide under elongation immediately after rinsing. The waveguide was fabricated from PETA droplet cured for 30 s with beam power of 12.8 µW. a) The waveguide after rinsing. b) Elongated to 345 µm. c) Elongated to 390 µm.

The waveguide waist diameter reduces with tensile strain as illustrated in Figs. 9 and 10. The diameter decreases by 10 µm at the centre indicating polymer mechanical flexibility and large stress at interfaces. This phenomenon also shows the impact of adhesion promoter on the self-written polymer waveguide's mechanical and optical performance.

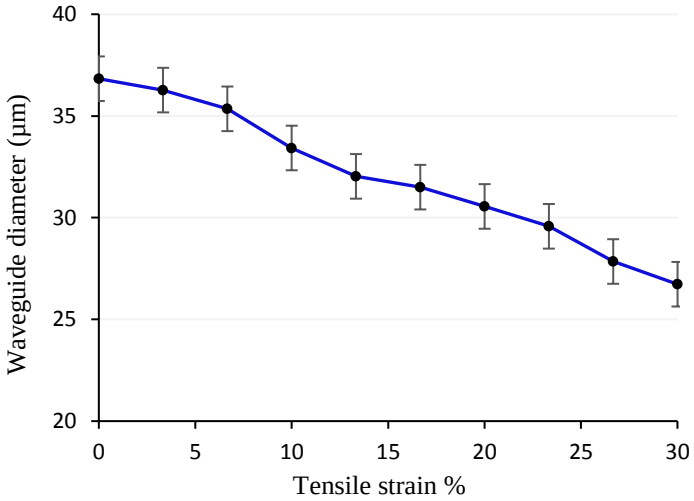

Fig. 5-20: The waist diameter reduction versus tensile strain corresponding to the unbleached 300 µm polymer waveguide shown in Fig. 9.

Long waveguides

In our previous paper we were able to construct polymer waveguides up to 600 μm, the limitation was mainly because of the size of the SMF28 fibres and the PETA viscosity were not allowed us to extend the liquid drop length beyond that limit. Also the intensity of the light emerging from fibre was not sufficient to cure the polymer very well beyond 400 μm. using bidirectional curing alongside with capillaries allowed us to fabricate polymer waveguides up to 1.5 mm, but they showed good optical transmission only up to 1.2 mm. The excel file (Loss vs waveguide length) presents the numerical detail of loss values associate with polymer bridges from (0.04 mm – 1.2 mm) and loss associated with airgap between aligned fibres for the same lengths. The detail of loss across the whole spectrum (350 nm-1750 nm) is presented in the excel file named (Loss for 1mm polymer waveguide). The data presented shows the transmission evolution immediately after rinsing and then after photobleaching until transmission reaches the maximum rate.

* Pshko A. Mohammed and William J. Wadsworth, "Long Free-Standing Polymer Waveguides Fabricated Between Single-Mode Optical Fiber Cores," J. Lightwave Technol. 33, 4384-4389 (2015).